MECHANICAL BEHAVIOUR OF FLEXIBLE POLYURETHANE FOAMS

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by

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Mechanical Behaviour of Flexible Polyurethane Foams

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Abstract

Dunlop Suspensions and Components manufacture microvon, a flexible polyurethane foam, which is used extensively in the automotive industry as spring aids for car suspension systems. The material is used because of its desirable non-linear elastic stress/stain characteristics, its quick recovery behaviour, and because it tends to produce little lateral expansion during compression.

The design of such spring aid components, however, remains something of a black art, since the behaviour of the material is not fully understood. Complications arise because the mechanical properties of the material are controlled by a large number of physical, chemical and processing effects.

The aim of the research has been to gain further understanding of the material, and its response under load, in order to be able to predict the compressive behaviour of the material. This has been achieved by combining microstructural observations of the deforming material with information obtained from detailed mechanical tests.

There have been many attempts by researchers to describe the behaviour of cellular materials. This has been done in a variety of ways, and approaches have included developing complicated strain energy functions or utilising simple models based on repeating cell units. However, a number of difficulties are encountered when applying these material models to microvon.

The microstructural observations and mechanical tests undertaken in this research have led to the development of new material models for cellular materials. Two types of model have been developed; a physical model and a series of phenomenological models. The physical model is a bi-linear relationship between stress, strain and density, in which the mechanisms of deformation are described. The phenomenological models have been developed using curve-fitting, to accurately predict the axial stress/strain and lateral/axial strain behaviour of microvon over a wide range of strain, density, temperature and strain rate.
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1. INTRODUCTION

Polyurethanes have been described as possessing so many outstanding physical properties that they are unmatched by any other single product for applications. The use and growth of polyurethane polymers has been spectacular, and they have gained the reputation of being one of the most versatile materials that can be tailored to an individual requirement.

The wide interest in polyurethanes is due to the diversity of products available. Depending upon the choice of starting components and the processing technique, it is possible to generate a wide variety of materials encompassing thermosetting plastics, thermoplastic rubbers, elastomeric rubbers, and flexible and rigid foams.

The basic research work on polyurethane elastomers was carried out by Dr. Otto Bayer and colleagues at Leverkusen in West Germany when they discovered the diisocyanate addition polymerisation process in 1937. During the early development of these polymers, the predominant chemical group present in the chain was the urethane group. However, with the rapid expansion of this class of material, polyurethanes now include those polymers which contain a significant number of urethane groups, together with a variety of other structurally important groups, such as ester, ether and urea. More recently, the term polyurethane has been used as a generic word to describe a wide range of polymeric materials, including not only foams and elastomers, but also surface coatings and adhesives.

The industrial breakthrough of polyurethanes occurred in the 1950s with the processing of consistently high quality elastomers, and with the introduction of the continuous flexible foaming process. This led to the dramatic rise in consumption of polyurethane in the 1960s which has approximately doubled each decade since then. During this early production period, the emphasis was on developing new polyurethane products, and the material soon found its place in the furniture and building industries. It was not until the 1970s that the material started to become more attractive to the automotive industry. In fact, polyurethane has been increasingly used in the automotive industry and now represents, on average, 30% of all plastic used in car manufacture.

Polyurethane foams have increasingly become a replacement for rubber materials such as natural rubber, styrene butadiene rubber (SBR) and nitrile rubber (NBR). This is because the material combines many of the desirable properties of both natural and synthetic rubbers, with certain characteristics of its own. Polyurethane foams tend to be light in weight, durable, resistant to a large range of organic solvents, have reduced lateral expansion and are inherent damping materials.

Foam systems have been developed to meet the needs of the automotive industry for such areas as recoverable shock absorption and reversible deformation in bumpers. Dunlop Suspensions and Components have developed a variety of polyurethane foam components for use in car suspension systems, these include; spring aids (or auxiliary springs), bump-stops (or jounces) and mounting bushes. Photographs of a selection of spring aid and bump-stop
components are shown in figure 1.1, while a typical vehicle suspension unit, with spring aid, is shown in figure 1.2.

_Microvon_, the trade name for the flexible polyurethane spring aids manufactured by Dunlop Suspensions and Components, is used extensively in vehicle suspensions to help in refining the quality of ride and reducing noise levels. The components can be made with complex internal and external shapes using a variety of different grades of material, providing complete flexibility in design. The difference in the grade being reflected in the combination of polymers used to make up the backbone of the material.

Many of the spring aid components are designed with a **buckling lip** which produces a smooth introduction to the stress/strain curve. This soft entry, combined with the large deflections that can be achieved using _Microvon_ instead of rubber, mean that the variable rate spring aid made from the foam can be added to a linear rate spring without creating harshness in the handling performance, thereby maintaining a comfortable ride. Appreciable benefits in cost and weight saving can be made over the traditional variable rate coil spring and bump-stop arrangement. The flexible polyurethane foams also have the advantage over rubber that longer spring aids can be made successfully from it, since the foam has reduced lateral expansion at large deflections.

_Microvon_ has been continually developed for more than twenty years and, within the last five years, has been fully accepted and used by car manufacturers world-wide. Although produced and manufactured successfully for a number of years, there still remains a great deal of uncertainty in the design of components. This is because the complicated behaviour of _Microvon_ is not fully understood, and there has been little study of the material. It is difficult to adequately explain, in simple terms, the mechanical behaviour of many cellular materials since their properties are controlled by a large number of physical, chemical and processing effects.

In an increasingly competitive industry, the need for greater control over the mechanical properties and a full understanding of the material is a necessity. This research has stemmed from the interests of Dunlop Suspensions and Components to stay ahead in the market and, since the company no longer has an active core research programme on the design of _Microvon_ components, a CASE award was arranged through the Engineering and Physical Sciences Research Council, formerly the Science and Engineering Research Council. The aim of the research was to gain greater understanding of the mechanical behaviour of the material and to provide some means of being able to predict the behaviour of _Microvon_ components.

This work has addressed these aims and also provides an in-depth study into the world of cellular materials and, in particular, flexible polyurethane foams.

The first couple of chapters of the thesis give an introduction into polyurethane foams, starting from an understanding of polyurethane polymers and leading on to the formation of polyurethane foam components. Chapter 2 discusses the properties of the type of polyurethane polymers that can be produced, and mainly concentrates on the structure and properties of polyurethane elastomers. These aspects of polyurethane elastomers are described in detail since
the elastomers are ideal for producing foams with properties which reflect the solid polymer.

Chapter 3 discusses the chemistry, manufacture and application of polyurethane foams. It introduces the complex chemistry of polyurethane foams and aims to give an outline of the reasons why material produced with the same ingredients may have different properties. Important factors such as environmental issues are also discussed. The properties of polyurethane foams are not usually affected by the chemistry alone, but also by the manufacturing processes involved. The foaming process, manufacturing techniques and production methods are all outlined in the second part of the chapter. The final part of the chapter discusses the properties and applications of the different types of polyurethane foam that can be produced.

The manufacturing process determines the morphology of the foam, which is discussed in chapter 4. The intent of the chapter is to explain how the different cellular structures are produced through the chemical and manufacturing processes. The need for the material to achieve certain configurations through natural forces is discussed with reference to the ideal cell structure. This then provides an introduction to the work undertaken on microstructural investigation in chapter 5, encompassing a comprehensive study of the as-received as well as the deformed material. The chapter looks at the variations in structure of the material, and incorporates the results of image analysis conducted on small samples. It also describes how the deforming microstructure was observed using a custom made compression rig which was fitted inside a scanning electron microscope.

Having made sufficient observations of the microstructure, a variety of tests were performed on samples of the material, the procedures for which are described in chapter 6. The tests included uniaxial compression, tension and shear, impact, compression set and water absorption. The testing has concentrated on compressive loading, since this is the mode of deformation most frequently encountered in the use of such cellular materials. However, since tensile and shear deformations may be encountered within the material during compression, it was necessary to gain some insight into the behaviour of the material under these modes of deformation. The results of the tests are presented in chapter 7.

One of the reasons for undertaking detailed microstructural examinations and conducting the mechanical tests was to provide information that could be used to develop an understanding of the mechanical behaviour of the material in order to be able to model it. A variety of models have been put forward in the past, ranging from complicated strain energy functions to models based on the mechanics of individual cells. Chapter 8 reviews the work done in this area and sets out to develop new material models to describe the mechanical behaviour of microvon.

Finally, the work on structural observations, material testing and modelling is discussed in chapter 9, and conclusions of the work are drawn out in chapter 10. This research, being the first to investigate the mechanical behaviour of the material microvon, has opened up a number of other areas of interest which have not been covered by the present work, these are then offered in chapter 11.
Figure 1.1: Selection of typical *microvon* components used in car suspension systems.

Figure 1.2: Photograph of a typical spring aid shown within a car suspension system.
2. POLYURETHANES

2.1 POLYMER TYPES

Polymers form the basis of all plastics, rubbers, fibres, adhesives and paints. They are widely used because they can operate in a variety of environments and have broad ranges of deformability and durability, which can be exploited by careful design. The range of properties of solid polymers is wide, with moduli ranging from below 1 MN m\(^{-2}\) to around 50 GN m\(^{-2}\).

Polyurethanes, also called polycarbamates, can be made with a number of desirable properties such as high hardness, creep resistance, high tensile strength (higher than many other rubbers), excellent tear resistance and abrasion resistance. They also have outstanding resistance to oxygen and ozone, although, they do exhibit low resilience and may decompose under prolonged exposure to water or steam.

The exact combination of properties such as stiffness, strength and hardness depend on parameters such as the chain length and character of the repeat unit in the polymer, the flexibility and character of the cross-linking agents, and the cross-link density. Not only can the character of the repeat unit be changed, but the distribution and type of active sites for cross-linking can also be varied. Some polymer foaming systems, particularly urethanes, will react with different types of chemical cross-linking agents to give polymers having very different mechanical, thermal, melt and corrosion resistance properties. The versatility of these systems makes it difficult to quote typical values of properties for a given type of polyurethane.

The polymer chains can be arranged in one of three different ways, forming a linear polymeric structure, a long chain network or a short chain network. In a linear structure the monomers are chemically joined end-to-end to produce a long linear chain-like polymer with many thousands of repeat units. Network polymers, however, are based on a type of monomer which can be chemically joined up, not only at each end, but also at other active sites. These monomers can therefore form covalently bonded three-dimensional networks, rather than linear long chains. If the links are widely separated, a flexible material will result; if close together, then the network will be more rigid. Short chain-branches tend to increase the modulus and tensile strength of polymers, and also have a large effect on the elongation, while the presence of long branches lowers the elongation and renders the polymer resistant to loading due to chain stiffening by bulky side chains.

There are few systems which admit so many variations, that different polymers in the same generic class can be either linear, network or a hybrid of the two, and polyurethane is one of them. This is because the two basic constituents of polyurethane can be chosen to contain long or short chains, each with many or few active sites.

The family of polyurethanes contains the three types of polymer used to make foams; linear thermoplastics, thermosets and elastomers. The typical structure of each of these
polymers is illustrated in figure 2.1.

Linear thermoplastics, like polyethylene, polystyrene, polyvinyl chloride and polytetrafluoroethylene, have long molecular structures, containing many thousands of monomers. In this type of polymer, the chains not only have a zigzag configuration but are randomly covalently cross-linked, so that the chains have a coiled-up configuration, with adjacent molecules severely entangled. Such a mass is usually isotropic and homogeneous, and the entanglement adds extra stiffness and strength to that provided by Van der Waals forces.

Thermoplastics tend to be amorphous, because of their lack of molecular order. On heating, they melt, and on cooling they will re-solidify and harden, a process which can be repeated over and over. The density of such linear polymers is nearly always near 1000 kg m$^{-3}$, because they comprise light elements loosely packed together; that of low density polyethylene is 920 kg m$^{-3}$, while that of polystyrene is 1050 kg m$^{-3}$. A small amount of crystallisation can give slightly better packing, and in the case of polyethylene, it increases the density to 970 kg m$^{-3}$, forming high density polyethylene, though polymers with heavier atoms, such as polytetrafluoroethylene, are denser still with densities that can be as high as 2200 kg m$^{-3}$.

Cross-linked thermosets, like epoxy and phenolic, have strong primary bonds linking the molecules to form a three-dimensional network of carbon-carbon bonds. They generally have a high cross-link density and are made up of rigid short groups between randomly distributed links. This gives many of the cross-linked thermosetting polymers a crystalline structure. These tightly cross-linked polymers are generally stronger at ambient, and especially at elevated temperatures. The density of thermosets is usually larger than that of thermoplastics, partly because most contain heavy oxygen or nitrogen atoms, and partly because the cross-linking itself increases the density by pulling the chains closer together. The density of such highly crystalline and rigid thermosetting plastics is typically between 1200 and 1500 kg m$^{-3}$. On heating, the polymers may soften slightly, but at higher temperatures they will char.

Elastomers, or rubbers, are a cross between thermoplastics and thermosetting plastics, since their structure is one which is partly amorphous and partly crystalline. Amorphous segments of the polymer tend to have a low modulus which directly reflects the stiffness of the Van der Waals bonds that join the molecules together. It is these, not the strong covalent carbon-carbon bonds, which stretch and deflect when the polymer is loaded. They can be thought of as linear chain polymers with a few widely spaced cross-links attaching each molecule to its neighbours. The links between these neighbouring atoms may be covalent or weak Van der Waals type bonds. The packing of the loosely tangled molecules is similar to that of linear polymers, so the density of most elastomers is like that of thermoplastics, about 1000 kg m$^{-3}$. Polymerisation and thermo-mechanical effects during processing cause the long-chain molecules of such polymers to arrange themselves in packs to form crystallites separated by amorphous areas.

All polymers can be destroyed by flame or excessive heat, although the rate of destruction depends on the type of polymer, the surface to volume ratio, the temperature and the duration of exposure to the heat source$^{23,34}$. The main differences between amorphous and partially
crystalline polymers, when heated from room temperature to their appropriate melt temperature, lie in the much bigger volume changes for the crystalline polymers. The thermal conductivity of amorphous polymers changes little over a wide range of temperature, but, for the partially crystalline polymers, there is a significant decrease in conductivity with increase in temperature until the melting point is reached.

In general, the usefulness of polymers is extremely limited, since many pure polymers cannot be processed into products having satisfactory properties. Thus compounds are often incorporated into the structure causing the chemistry to become more complex. Additions include antioxidants, anti-ozonants and ultra-violet light absorbers since the effects of oxygen, ozone and ultra-violet light lead to degradation of the polymer.

It may also be desirable to give the polymer higher strength and stiffness. There are several methods which can be applied to give stiffer polymers, this can be done by increasing the length of the polymer chain, preventing the chains from disentangling, making the chain itself from an inherently stiffer repeat unit, or by increasing the number of cross-links between chains. The reverse is true to make a polymer more flexible, thus, by varying the combinations of the techniques described, it is possible to alter and improve the properties of the polymers to suit the application.

Longer chains give rise to a more tangled structure, and thus the polymeric structure can have a higher strength. If further entanglement is required, the small hydrogens in the monomer can be replaced with a more bulky group, such as an oxygen. Organic polymers strengthened in this way restrict bending in the structure, however they are fairly easy to dissolve and are subject to swelling, since the bulky groups allow ready penetration of the system by solvents. A stiffer repeat unit can be achieved by introducing aromatic structures or other atoms along the main polymer chain.

Cross-linking is an effect that depends on a chemical reaction rather than on physical orientation of the chains. It is strongly accelerated by elevating the temperature, and is not reversible, because the strong and randomly located bonds connecting the chains do not merely softened, but breakdown altogether at temperatures high enough to melt it.

2.2 STRUCTURE OF POLYURETHANE ELASTOMERS

One of the first references to the physical structure of polyurethane elastomers was by Saunders & Frisch, who suggested that they could be considered as block copolymers, where the length and structure of each block could be controlled within certain limits. Their early investigation into the physical structure of these elastomers led to a general picture that the structure consisted of alternating hard and soft blocks or segments. In recent years, much work has been carried out to investigate the extent of the segmented structure, in particular, thermal analysis and X-ray scattering investigations have shown evidence for such a two phase structure from which polyurethane elastomers derive their high level of strength and flexibility.

The soft segments are typically made of low-molecular-weight polyols, and are usually
about 10-20 nm long. It is the soft amorphous segments which generally influence, not only the
elastic nature of the material, but also the properties at low temperatures.

The hard segments generally consist of aromatic diisocyanates, and are usually about 2.5-
5.0 nm in length. Other contributors to the hard crystalline segments are the relatively rigid
groups of urethane, urea, allophonate and biuret, which all have very little flexibility due to
their high aromatic content. These hard blocks can constitute between 30 and 30% by weight
of the total polymer.

The hard, rigid domains formed act as tie down points, and as filler particles, reinforcing the
soft segment matrix. These hard segments are held together through the action of Van der
Waals forces and hydrogen-bonded interactions. It is well known that the introduction of a
filler, such as carbon black into an amorphous vulcanised rubber, increases its hardness,
strength, hysteresis and abrasion resistance. It is also known that the properties improve as
the particle size decreases. The large increase in these properties in the case of polyurethane
must be due, therefore, to the very small size of the hard urethane segment in the chain. The
physical strength and elasticity of such elastomers at elevated temperatures are very dependent
on the structure of the hard segments and their ability to remain associated at these
temperatures. This hard segment structure can be varied by altering the structure of either the
isocyanate or any of the chain-extenders; variation having the greatest effect on the flow
behaviour of the material.

Interaction of attractive forces between hard segments are far greater than those present in
the soft segments, owing to the high concentration of polar groups and the possibility of
extensive hydrogen bonding. Hard segments significantly affect mechanical properties,
particularly modulus, hardness and tear strength. The modulus is seen to decrease and the
extensibility to increase with decreasing hard segment content in some elastomers. The
number of hard segments, and the cross-link density of the system, are also found to decrease
as the molecular weight of the soft segment is increased. Chemically cross-linking the
polyurethane appears to disrupt the molecular arrangement and, over a certain critical level,
significantly decreases the modulus and strength of the material. This is in contrast to other
hydrocarbon elastomers in which the opposite is true, and is possibly due to reduction in the
orientation of the chains, so that the polymer loses its crystallinity and becomes more
amorphous.

To obtain a high strength elastomeric polymer, a proper molecular arrangement of the hard
and soft segments must occur, in addition to the hydrogen bonds and other forces between the
hard segments. In general, if the hard, crystalline structure predominates, the material is
relatively strong, rigid and resistant to heat and dissolution, if the structure is amorphous, the
substance is soft, elastic, absorptive and permeable to fluids.

2.3 TRANSITION REGIMES OF POLYURETHANE ELASTOMERS

Elastomers are sensitive to temperature, and as such, they exhibit several transition regimes.
The transition temperatures are related to the soft and hard segments of the elastomers, and rely on the length of these segments. Although sensitive to segment length, they are usually unaffected by the overall molecular weight. Depending upon the temperature, the elastomer can be in any one of four different states, or regimes, corresponding to an increase in the free intermolecular volume with the temperature, and a decrease in the strength of the bonding forces. These four states, illustrated in figure 2.2, are: the glassy state, the transition state, the rubbery state and the fluid state.

Within the glassy state (<0.9 Tg), where Tg is the glass transition temperature of the material, if the polymer is kept very cold, no chains or parts of the molecule can move, and both the amorphous and crystalline segments are frozen into a rigid glassy state. Therefore, in this regime, the polyurethane elastomer tends to be stiff and brittle like any other glassy polymer.

Increasing the temperature within the glassy regime has two effects. First, thermal expansion increases the molecular separation and lowers the Van der Waals forces. Second, the thermal energy of the molecules permits small relative movements of side groups, or of short segments of chain, at loose sites in the structure and causes a small drop in the modulus. However, the slight increase in the mobility of the amorphous segments tends to be swamped by the reinforcing effect of the crystalline segments.

In the transition state (0.9-1.1 Tg) the amorphous segments start to become rubbery, but the crystalline segments still provide the polymer with substantial stiffness. The rubbery nature allows complete blocks or segments of the polymer chain to become free to move and rotate as local entities, causing a drop in the modulus by several orders of magnitude. Polymers in this regime are viscoelastic, and the modulus of the material is strongly dependent on the temperature and strain rate or time of loading. In general, the glass transition temperature increases with molecular weight and with the degree of cross-linking.

Once the glass transition temperature has been exceeded, the polymer chains tend to creep, just as metals and ceramics creep at temperatures above a third of their melting temperature.

In the rubbery state (1.1-1.4 Tg), the weak intermolecular bonds have melted while the strong covalent bonds, linking units in the polymer chain and forming the occasional cross-links, are still very intact. In this regime, the material softens with increase in temperature, as the matrix becomes more and more rubbery. The elastomers show slow, large recoverable deformations in this regime without yield or fracture. The rubbery modulus is approximately three times less than the glassy modulus and is a sensitive function of the cross-link density. Increasing the cross-linking density raises the modulus, so too does the combined effect of oxygen and sunlight, causing elastomers to turn hard and brittle.

In the fluid state (>1.4 Tg) the material transforms from a viscoelastic solid to a viscous and amorphous melt. Further heating will then lower the viscosity of the polymer, leaving the material in a fluid state.

Unlike thermoplastic and thermosetting polymers, the glass transition temperature of an
elastomer is well below room temperature. This means that, for elastomers, the rubbery regime spans room temperature and is often fairly wide. In this temperature range, the soft segments are sufficiently high above their second order transition temperature, or softening temperature, while the hard segments are below their second order transition point. This implies that, at room temperature, the secondary bonds have already melted, and the molecules can slide relative to each other with ease. Without the crystalline cross-links, which give the elastomer its mechanical stability, the material would be a viscous liquid.

Since polyurethane elastomers are generally within the rubbery regime, the materials can undergo enormous deformations; it is possible to stretch an elastomer repeatedly to five times its original length, completely reversibly\[^{25}\]. Up to extensions of about 200-300\%, the soft segment polymer chains undergo various extents of stretching and disentanglement, causing the rigid domains to lie in a distorted manner transverse to the direction of stretching. As the elastomer is further elongated, the orientation of soft segments improves only to a small extent, while the hard segments turn themselves with their longitudinal axes into the direction of elongation. This occurs since during the initial elongation the soft segments are stretched to different extents and are therefore placed under different loads. The locally varying forces in the structure then cause the transverse positioning of hard segments.

At this stage, the maximally loaded chains in the soft segment start to oppose any further elongation, so that extension above this point requires that sliding processes, between hard segments, take place. At about 500\% elongation, this restructuring process is virtually complete.

On unloading the thermal motion of the molecules, coupled with the memory built into the arrangement of the cross-links, causes the material to return to its original shape, though the restoring force is a small one. The soft segments disorientate almost completely, while the hard segments tend to remain in the oriented manner. This molecular organisation and restructuring on repeated extension provides a possible explanation of the stress-softening phenomena and high hysteresis in polyurethane elastomers\[^{45}\].

One of the most important requirements for polymeric materials is that they can be used over the desired range of temperatures without a significant change in properties. However, close to their transition temperatures the mechanical properties are likely to go through a radical change. This high sensitivity to temperature originates directly from the increasing rate of molecular rearrangement with temperature\[^{8}\].

In general, polyurethane elastomers are very suitable for the manufacture of expanded flexible foam, since the high strength of the solid polymer permits a foam of high strength to be produced. The chemistry and manufacturing techniques used in the production of polyurethane foam determine the mechanical properties of the cellular material produced. These aspects, therefore, are discussed in the following chapter.
Figure 2.1: Illustration of the difference in structure between the three main classes of polymer.

Figure 2.2: Graph representing the transition states of a segmented polyurethane elastomer.
3. POLYURETHANE FOAMS

This chapter examines the chemistry of polyurethane foams and discusses how the properties of the material can be altered according to the type of compounds employed. Although the structure of polyurethane foam is controlled, in the main, by the selection and ratio of the reactants used, this is not the only means of altering the structure. The way in which the polymer is blown and the techniques used for manufacturing and production are also crucial to the outcome of the foam, and these are also described.

The final polyurethane foam component, therefore, can be tailored to meet the requirements of the user. The last part of the chapter outlines some typical applications and uses for the different types of polyurethane foam than can be formed.

3.1 FOAM CHEMISTRY

3.1.1 Formation of polyurethane foams

Polyurethane foam formation involves many reactions and there are many complexities in their synthesis\(^3\)\(^{29}\). It has this complexity since the foams may contain, in addition to urethane groups, other complex groups such as urea, ether, ester, amide, allophanate and biuret. The final structure of the foam will contain repeating urethane groups regardless of raw material components or other groups which may appear in the polymer backbone structure as a result of any side reactions. An understanding of the formation of polyurethane foams involves consideration of the organic chemistry of the reactions leading to gas formation and molecular growth, the colloid chemistry of nucleation phenomena and bubble stability, and the rheology of the polymer system as it cures.

The whole macromolecular system in polyurethane foams can be designed so as to produce flexible, semi-rigid or rigid foams. This is achieved by altering the molecular flexibility or rigidity of the polymer chains by providing the necessary amount of chain-branching or cross-linking needed to give the properties required in the final structure. A number of other factors, such as the nature and ratio of the components in the polymer synthesis, the molecular weight, the level of intermolecular forces, the method of synthesis and the techniques of post-cure, also assist in determining the foam properties\(^3\)\(^{29}\).

In order to understand polyurethane foam chemistry, it is necessary to look at the way in which the urethane and polyurethane polymers are formed. The simplest way to produce urethanes is by the addition of hydroxyl compounds, primarily alcohol, to isocyanates, see equation (3.1).

\[
\text{R'N=C=O} + \text{R-OH} \rightarrow \text{R'-NH-C=O-R}
\]

Isocyanate Hydroxyl Urethane

\[
(3.1)
\]
Polyurethanes, on the other hand, can be synthesised by two principal methods: the reaction of bischloroformates with diamines, equation (3.2), and, more commonly, the reaction of diisocyanates with polymeric hydroxyl compounds, equation (3.3), usually in the presence of several catalysts.

\[
\begin{align*}
H_2N-R'NH_2 + Cl-CO-O-R-O-CO-Cl & \rightarrow [-NH-R'-NH-CO-O-R-O-CO-] + 2HCl \\
\text{Diamine} & \quad \text{Bischloroformate} & \quad \text{Polyurethane} (3.2) \\
O=CN-R'N=O + HO-R-OH & \rightarrow [-NH-R'-NH-CO-O-R-O-CO-] \\
\text{Diisocyanate} & \quad \text{Polymeric} & \quad \text{Polyurethane} (3.3) \\
& \quad \text{hydroxyl} & \\
\end{align*}
\]

Polyurethanes are not direct polymers of urethanes but are usually derived by polyaddition. It is common to see such reactions included under polyurethane foams even though no urethane chemistry is involved. This is because foam manufacture that utilises isocyanates tends to be grouped under urethane.

The types and concentrations of the two principal reactants, the isocyanate and polymeric hydroxyl (often referred to as the polyol), are crucial to the type of foam formed. The reaction of these two compounds is known as the gelling reaction because it is the primary means of polymerising the starting materials. When gelation occurs, the chains themselves become bonded or entangled to such an extent that a network of long chains are formed.

The isocyanate component commonly employed in urethane synthesis varies from aliphatic isocyanates such as 1,6 hexamethylene diisocyanate (HDI) to aromatic diisocyanates such as 1,5 naphthalene diisocyanate (NDI), 2,4 and 2,6 toluene diisocyanate (TDI), 4,4' diphenylmethane diisocyanate (MDI) or polymethylene polyphenyl isocyanate (PAPI).

The polymeric hydroxyl compounds generally used are linear or branched low-molecular-weight hydroxyl terminated polyesters or polyethers. A broader range of foam properties can be achieved by use of more than one polyol component, this not only allows the degree of chain-branching associated with the polyol component to be changed, but by varying the order of interaction of the polyols with the diisocyanate it is possible to vary processing factors such as temperature of reaction and viscosity.

Low-molecular-weight alcohols, such as glycerol or trimethylolpropane, are often added to the polyol mixture before reaction with the isocyanate to provide sites for chain-branching. Additions of such low-molecular-weight triols are one of the most effective means for increasing foam strength chemically, although this approach is not always desirable as it may lead to higher viscosity components. While additions of triols are commonplace, short chained diols, tetrols or aromatic diamines can also be used as chain-extenders and cross-linkers, and can help to control viscosity during foaming. Chain-extension with diol gives rise to a polyurethane foam with urethane linkages, whereas chain extension with diamine gives a foam with urea linkages.
When isocyanates react with water, a sequence of vigorous reactions takes place that generate heat which helps to drive the isocyanate-polyol reaction to completion. The isocyanate-water reaction is sometimes referred to as the blowing reaction, because of the carbon dioxide gas produced, and is primarily controlled by the water content of the formulation. The water present reacts with isocyanate to produce organic acids, equation (3.4) which can break down to form amines (or diamines) and carbon dioxide, equation (3.5), which both modify the structure. The amines, so formed, react with excess isocyanate to form urea bridges, equation (3.6). The more water that is present, the more gas is evolved, and the greater the number of active urea points for cross-linking. As well as forming urea, the isocyanate-water reaction may also form dimers and oligomers. It is possible, to a certain extent, to control the density of the foam by controlling the amount of water and diisocyanate used.

\[
\text{R-N=C=O} + \text{H}_2\text{O} \rightarrow \text{[R-NH-CO-O-H] (Isocyanate Water Carbamic acid)} \tag{3.4}
\]

\[
\text{[R-NH-CO-O-H]} \rightarrow \text{R-NH}_2 + \text{CO}_2 \quad \text{(Amine Carbon Dioxide)} \tag{3.5}
\]

\[
\text{R-N=C=O} + \text{H}_2\text{N-R} \rightarrow \text{R-NH-CO-NH-R (Amine Urea)} \tag{3.6}
\]

The water acts as a blowing agent, and is the most commonly used. The other main types of blowing agent are chlorofluorocarbons, such as trichlorofluoromethane (Freon 11), dichlorotrifluoroethane (HCFC), methylene chloride or halogenated paraffins. Although chlorofluorocarbons are generally considered chemically inert, they are unstable under certain conditions, and may react with primary and secondary alcohols to liberate aldehydes, ketones and hydrochloric acid. For these reasons, and because chlorofluorocarbon blowing agents are potentially hazardous to the environment, there has been considerable interest in polymer foams blown with alternative vapours, primarily water. One advantage in using chlorofluorocarbons over water, however, is that a more uniform distribution of cell sizes is produced.

During processing, the reaction rates of the blowing and gelling reactions must be balanced in order for the foam to reach the desired density before gelation. The gelling and blowing reaction rates are determined by the catalyst, which has a unique effect on each reaction. There are two main types of catalyst used; organo-metallic compounds and tertiary amines. The organo-metallics are usually organo-tin compounds, such as stannous octoate, dibutyl-tin diocotate and dibutyl-tin dilaurate. The stannous salts are the most powerful, and are used to facilitate the gelling reaction through polyaddition. The tertiary amines used, of which there are...
frequently two types, triethylene diamine and tetramethylbutane diamine, aid the blowing reaction.

Both types of catalyst, however, contribute to both reactions, and by varying the combinations of tin catalyst with a tertiary amine it is possible to produce highly active systems in which foaming and cross-linking reactions are properly balanced. The mixed catalyst system produces a means of eliminating such problems as foam shrinkage and void formation, which may be the result of an improper balance of gas evolution and polymer properties at the time of maximum gas evolution\textsuperscript{22}.

After fulfilling their role, the tertiary amines tend to evaporate from the foam while tin catalysts remain, although they may undergo some chemical change with time, for example, stannous tin may oxidise to form stannic tin.

Catalysts are not normally required for forming linear polyurethane foams, which are normally prepared in solution, because the polymer tends to dissociate into alcohol and isocyanate or to decompose into amines, olefins, and carbon dioxide at the high temperatures needed for melt polymerisation\textsuperscript{23}. The solubility of linear polyurethanes, in certain solvents, provides evidence of the essentially linear molecular structure of these materials. Polyurethanes are soluble in dioxane, tetrahydrofuran, dimethylformamide, dimethylsulfoxide, formic acid and sulphuric acid, and are generally insoluble in alcohols, water and hydrocarbons\textsuperscript{5}.

Organic acids can react with isocyanates to produce carbon dioxide and amides, equation (3.7). An excess of isocyanate leaves all the water and polyol reacted with still some isocyanate left over. Under these conditions, the urethane and urea can react with isocyanates through their active hydrogen atoms to produce aliphonate, equation (3.8), and biuret, equation (3.9), cross-links respectively. Although, since the urea link is more reactive than the urethane link, there is often a greater proportion of biuret cross-links. The formation of biuret and aliphonate usually occurs between 120 °C and 150 °C.

Some of the chemical reactions involved are reversible under the action of heat, and so introduce the possibility of molecular rearrangement during processing.

\begin{align*}
R'-N=\overset{\text{Isocyanate}}{\text{C}=O} & + \quad R-\overset{\text{Carboxylic Acid}}{\text{C}=O-H} \quad \rightarrow \quad R'-\overset{\text{Amide}}{\text{NH-C}=O-R} & + \quad \overset{\text{Carbon Dioxide}}{\text{CO}_2} & \quad (3.7) \\
R'-N=\overset{\text{Isocyanate}}{\text{C}=O} & + \quad R-\overset{\text{Urethane}}{\text{NH-C}=O-R''} \leftrightarrow \quad R-\overset{\text{Aliphonate}}{\text{NCOR''-C}=O-N\text{H}-R'} & & \quad (3.8) \\
R'-N=\overset{\text{Isocyanate}}{\text{C}=O} & + \quad R-\overset{\text{Urea}}{\text{NH-C}=O-N\text{H}-R''} \leftrightarrow \quad R-\overset{\text{Biuret}}{\text{NCOHNR''-C}=O-N\text{H}-R'} & & \quad (3.9)
\end{align*}
These latter secondary reactions can be useful for tailoring the mechanical properties of foams, since they eliminate a hydrogen atom previously capable of forming a hydrogen bond, and form a new, covalent, stabilising cross-link. Hydrogen bonding can occur between ester-urethane, ether-urethane and urethane-urethane groups, restricting polymer chain mobility. Similarly, Van der Waal’s forces operate between various parts of the polymer chain.

Silicone compounds are often incorporated into the formation of polyurethane foams in order to aid foam stability during reaction, and to control cell size and uniformity through reduced surface tension. These silicone compounds are termed surfactants, or foam stabilisers, and normally consist of polysiloxane derivatives, such as silicone oil or organo silicone copolymer. Other additives used in polyurethane foam synthesis may include emulsifiers, ultra-violet absorbers, heat stabiliser, anti-oxidants/ozonants, colour dyes and pigments, fillers and moisture absorbers.

Stored, pre-packed, polyurethane foam components are prone to chemical ageing which may jeopardise their intended function. As a consequence, many manufacturers of these chemicals will not guarantee their product for more than ninety days. This is for a variety of reasons, such as moisture attack on the isocyanates, blowing agent separation and general material instability, which leads to thermal degradation and incompatibility.

3.1.2 Altering the foam properties

The properties of polyurethane foams can be modified by altering the type and concentration of compounds used in their synthesis.

ISOCYANATES: 4,4' diphenylmethane diisocyanate is the compound used in the formulation of microvon and is used in preference to bulky 1,5 naphthalene diisocyanate because of its less hazardous nature, and because it gives rise to a more flexible molecular structure. Changing this component, therefore, alters the hard segment of the material and can lead to foams with higher or lower hardnesses. Diphenylmethane diisocyanate has a lower isocyanate content than toluene diisocyanate and has a more aromatic structure, is more viscous, and does not readily afford itself to low density foams. Asymmetrical molecules, like the 2,4/2,6 toluene diisocyanate combination, give materials of low modulus and hardness, while, in the case of symmetrical molecules, tensile strength and tear strength are shown to be greater, particularly those based on the 1,4 diphenylmethane diisocyanate substituted benzene ring system.

POLYOLS: Flexible foams are usually prepared from dihydroxy-polyols, whereas rigid foams are made with polyhydroxy-polyols. Polyether polyols generally give foams having a lower level of mechanical properties than the polyester-based material, owing to the weaker inter-chain attractive forces present. Polyether-based foams also show a higher level of crystalline order than polyester-based foams, presumably owing to the more regular chemical structure present, which tends to give them a less random cell structure. In general, very low-molecular-weight soft segments give poorly elastic, hard materials, whereas a high-molecular-
weight gives rise to soft materials having poorer mechanical properties.

CATALYSTS: The fundamental structure difference between using diol and diamine extended materials generally leads to differences in the mechanical properties of the foam. Diamine extended materials usually possess a higher level of mechanical properties owing to the strong hydrogen-bonded interaction of the urea group, while in the case of diol extended materials, a significant proportion of cross-linking is often introduced by the use of a triol to give improved properties. However, these materials are still of low hardness and strength compared with the diamine extended materials. Use of amine blends gives softer materials of lower modulus than those obtained using a single diamine, thought to be due to structural irregularities causing decreased intermolecular bonding.

BLOWING AGENTS: The thermal, and electrical, conductivity of foams can be profoundly influenced by the cell gas composition and the cell wall thickness. Thermal conductivities may be minimised, and densities increased, by letting closed-cell foams retain high-molecular-weight blowing agents, such as halo-carbons, which can insulate more effectively than air, and by reducing the formulated amount of blowing agent. If a proper choice of a low thermal conductivity gas is made for filling the foam, then, although air can still diffuse into the cells of the foam and cause an unwanted increase in thermal conductivity, an equilibrium is reached in which the constant value is still less than that for air alone because the mixture has a lower conductivity than air.

If water is used as the blowing agent, it is far more commonly encountered as a vapour than as a liquid, and in this state, tends to diffuse through solid membranes under the stimulus of partial pressure gradients. Once it has entered a closed-cell foam the cell vapour might condense at low temperature and accumulate, thereby, gradually increasing the weight of the foams and the overall thermal conductivity. However, use of chlorofluorocarbon blowing agents tends to have a better effect than using water as a blowing agent since it has an extremely slow rate of diffusion. Polymeric foams, therefore, tend to have lower thermal conductivities than any class of dense polymer.

3.1.3 Environmental issues

In recent years, public awareness of environmental issues has increased enormously, and as such, the pressure to be more environmentally friendly has become important. These pressures are having an effect on three main aspects of flexible polyurethane foam chemistry and manufacture. The first of these is the reduction, and eventual replacement of chlorofluorocarbon blowing agents, the second is the requirement of tighter fire ignition standards, and the third is the need for material recyclability.

Chlorofluorocarbons are volatile organic compounds which ultimately contaminate the atmosphere, and have been implicated in the destruction of the ozone layer. This has lead to renewed intent to search for an alternative, more acceptable blowing agent. One possibility is the use of water, although this is undesirable for a number of reasons, which have been
discussed above.

Since the discovery of polyurethanes and their acceptance in many important and varied applications, research has been carried out to improve the fire resistance of polyurethane foams. Although the term *polyurethane foam* may be a standard generic term, encompassing an entire family of products, there is nothing standard about the fire properties of these various materials. For example, it is known that diphenylmethane diisocyanate foams are better than toluene diisocyanate foams for fire ignition, however, both have short times to ignition.

There are some polyurethane foams which have remained essentially unchanged since the late 1960s and are potentially very dangerous, whereas, at the other extreme, there are foams which are extremely difficult to ignite under most realistic fire conditions, and do not readily propagate combustion.

The primary products of combustion of many foamed plastics are usually carbon monoxide and carbon dioxide, with smaller amounts of other substances. Such plastic foams are easily combustible because they are organic and contain flammable hydrocarbons, though their actual response to small ignition sources may vary. However, when polyurethane foams are burnt they give off smoke and toxic gases which may include, as well as carbon monoxide and carbon dioxide, nitrous oxides, hydrogen cyanide and isocyanate vapours. These combustion products are known to give rise to acute toxic effects such as asphyxia and narcosis.

Until the early 1980s the most popular approach to formulating *state of the art* urethanes, with a high level of fire resistance, was the incorporation of large quantities of generally inorganic materials into the formulation. Such additions included halogenated compounds for flame resistance, terephthalate-based polyester polyols and triazinic polyols for decreased flammability and smoke generation, aluminium hydrates for improved ignition resistance, and phosphorous for providing the foam with self-extinguishing properties. Many of these additions are effective, but, polyurethanes made with *melamine* technology show the most outstanding flammability properties, even under severe fire conditions.

Even though such additions usually involve only relatively small amounts of chemicals, they will drastically alter the flammability character of the foam, though, they are normally employed at some sacrifice to the other foam properties. Recently, much work has concentrated on modifying the structure of polyurethane itself, so that the polymer melts or decomposes below the ignition temperature.

The concept of plastics recycling and recyclability is still in its early stages. There are a number of methods available for recycling plastics, these include chemical recycling, physical re-bonding and pulverisation. However, the majority of polyurethanes are not suitable for such recycling because of the presence of unknown compounds in the foams, and the lack of markets for recycled products. Also, the economic and environmental costs of collection and separation of plastics would be considerably large. Despite this, there has been much research conducted with regards to plastics recycling, and this likely to increase with the pressure of making plastics more environmentally friendly.
3.2 FOAM MANUFACTURE

3.2.1 The foaming process

Polymeric foams can be blown in a number of different ways. Some foam manufacture uses compounds that change their state during processing, some rely on the use of chemical reactions, while others use a purely mechanical process. These three methods are referred to as physical, chemical and mechanical processes respectively.  

Physical foaming agents are compounds that change their physical state during cellular growth. For example, compressed gases expand when pressure is released and volatile liquids, the most important physical foaming agents, create cells when they change from liquid to a gas. The vaporisation of these liquid foaming agents is a reversible endothermic reaction and can serve as a useful means of controlling the process temperature. Aliphatic and halogenated hydrocarbons are the most widely used agents, although low boiling point alcohols, ethers, ketones, and aromatic hydrocarbons have been used.

Chemical blowing agents are materials that decompose under heat to at least one gaseous decomposition product. The most common chemical foaming agents are organic compounds that release nitrogen or carbon dioxide as the major component of the gaseous phase, and the most important property of such chemical agents is their decomposition temperature. In polyurethane foams, water and chlorofluorocarbons are generally used as chemical blowing agents, as discussed earlier.

The mechanical process involves vigorously agitating the molten mixture, containing the polyol and isocyanate, with dry, or inert, gas being forced through it so that the polymer is, essentially, whipped into a foam.

The tiny gas bubbles that are generated in the liquid polymer system by the methods described above, effect nucleation. After a short induction period, the gases evolved begin to diffuse into and enlarge the tiny nucleation bubbles, which causes a creamy appearance. The period of foam mixing to this point is known as the cream time, and is normally about six to fifteen seconds for flexible foams. As more vapours are generated, the foaming mixture continues to expand and becomes more viscous. The reduction of surface tension by the surfactant stabilises the foaming mixture and prevents coalescing of the bubbles.

The blowing reaction ceases about one to three minutes after mixing, but the gelling reaction continues and strengthens the polymer forming the cells. The period from foam mixing to full rise is known as rise time, during which the total number of bubbles remains constant. Polymerisation continues until the foam has gelled, which is usually between twenty seconds and two minutes after the rise time. The loss of surface tackiness which then occurs is known as tack-free time. Additional cure time is necessary to achieve optimum mechanical properties; it is dependant on temperature, and the required time may vary from hours to days. The final curing will be complete only when there are no isocyanate groups left unreacted.

For many microvon components, there are different stages of the curing process. In the pre-cure, the components are left to stand for approximately eight hours at room temperature, after
which time they are put in an oven at 95 °C for eighteen hours as a post-cure.

During the whole sequence of events from the initial reaction to final foam formation, it is possible to observe a change from the liquid system, first to a plastic gel, and then to a highly elastic polymeric structure.

If, during the formation of a closed-cell foam, an open-cell structure is required, the cellular material can undergo a thermal post-expansion treatment, known as reticulation, which ruptures the membranes between adjacent cells in the foam to produce an almost completely open-cell structure.

3.2.2 Manufacturing techniques

The manufacture of polyurethane foams involves controlling the introduction of the ingredients through careful metering of the mixing time, speed and temperature. Polyol, isocyanate and water must all be delivered at an accurate rate to maintain the desired stoichiometry. Usually, the lower viscosity isocyanates are dispersed into the more viscous polyols, which are then mixed together. Thorough mixing is critical because of the highly reactive nature of the components. Because of the rapidity of the reaction, it is difficult to control the physical properties, and if there is a slight change in concentration of the components, especially the catalyst, it can cause internal variations or eventual collapse of the foam structure. The timing is therefore critical and the mixing and metering equipment required is fairly sophisticated.

There are various techniques used commercially to manufacture polyurethane foam, the most common of which is reaction injection moulding (RIM). This process involves the rapid reaction of a mixture of compounds into a closed mould. The first stages in the RIM process involve preconditioning the liquid chemical components through temperature control, removal of entrained air, and adjustment of the level of dissolved gas. The chemicals are fed, at the required ratio, into one of two metering-mixing systems, at either low or high pressure. Low pressure systems use positive displacement pumps to deliver material to a mixing chamber with a mechanically driven impeller. In high pressure systems, precision high pressure pumps deliver material via flow-adjusting valves to a cylindrical impingement mixing chamber. In both cases, the chamber usually consists of a driven screw thread along which the components react. Once thoroughly agitated, the reaction mixture is injected into a temperature-controlled mould. The part is then left so that the reaction mixture can polymerise and cure sufficiently before demoulding occurs.

There are a number of variations in the RIM process, and also in the way the ingredients are fed into the system. There are three main techniques: the one shot process, the prepolymer process and the quasi-prepolymer process, all of which are shown schematically in figure 3.1.

In the one shot process foam ingredients are accurately weighed into a masterbatch which is thoroughly mixed and left to stand and de-aerate. The masterbatch usually contains a polyl
mix, water, an emulsifier, a structure modifier and paraffin oil which helps to control pore size and prevents splitting of the foam. Tin catalyst and silicone surfactant are then added to the masterbatch along with any additional components, and the mixture is thoroughly stirred to ensure sufficient emulsification of the foam ingredients. Once these ingredients have been well mixed, the isocyanate is added. The polymerisation reaction begins almost immediately, and as the polymer molecular weight increases by chain-extension, branching or cross-linking, the process of expansion is initiated, and the foam begins to rise due to the simultaneous formation of carbon dioxide. An inert, low-boiling halo-carbon (such as Freon) is usually added to augment the foaming action of the carbon dioxide. It is important that carbon dioxide evolved in the isocyanate-water reaction be allowed to escape, and also that the reaction is controlled, so that premature foaming does not occur. The reaction is carried out with slightly warmed components, and foaming is generally complete within a minute of the isocyanate being added. The polymerisation reaction increases the viscosity and sets the foam before it collapses. The temperature can continue to rise after the foam is fully expanded because of the continuing reaction of the isocyanate groups with water, amine and hydroxyl groups. Most polyurethane foams expand to between ten and forty times their initial volume during processing. A substantial exotherm is produced during the one shot process, which serves as post-cure for the expanded foam.

In the prepolymer process the isocyanate reacts with the polyol in a pre-reaction to form a low-molecular-weight isocyanate-terminated prepolymer. This is usually expanded with halo-carbon, or expanded and cross-linked with water to produce a foam. The advantages of the prepolymer process are mainly due to the reduction of exotherm in the early forming stage. This means that the possibility of inadvertent branching occurring in the system, which leads to the production of allophonates (isocyanates reacting with urethane groups) or biurets (isocyanates reacting with urea) and other reactions, is reduced because of the lower reaction temperatures. These unwanted side reactions are normally difficult to control in the one shot process. The disadvantages, though, are that prepolymer, being quite viscous, can present problems in maintaining accuracy when metering and mixing.

This type of process is the one currently used in the manufacture of microvon. The prepolymer is supplied by ICI Avalon and contains 18% pre-reacted diphenylmethane diisocyanate (MDI) with a mixture of two polyols; polyether and polyester. Diphenylmethane diisocyanate is used because it is available in a pure state and thus allows elastomeric products of high consistency and good stability to be manufactured. The resin mix, 90M808, also supplied by ICI Avalon, contains more of the polyether and polyester, as well as water, surfactant, pigments, catalysts and butane diol.

Prior to reacting, the two components are heated, the resin to approximately 50 °C and the prepolymer to 40 °C. The ingredients are then pumped to a mixing head where they are valve metered at a ratio of approximately 1:1. Reaction starts on the mixing screw and the temperature can rise to over 70 °C. During processing, further quantities of water, catalyst or surfactant may be added, as required, to alter the properties of the material, primarily the density of the foam.
The quasi-prepolymer process, which is intermediate between the prepolymer and one-shot processes, is useful where prepolymer is too viscous, where the resin does not easily adapt itself to one shot processes and when the equipment available is more suitable for two-part systems. In this process a part of the polyol is pre-reacted with the isocyanate to produce a prepolymer containing large excess of unreacted isocyanate. The resulting prepolymer is not as viscous as that normally encountered in the prepolymer process. The quasi-prepolymer is then reacted with an additional amount of polyol, which may contain the catalyst, blowing agent and other additives. Optimisation of the mixing and metering process employed in the application of the two component quasi-prepolymer system may be achieved by formulating each component to have equal viscosity. The system has the advantage of flexibility and of having low viscosity components, but as with one shot foams, there are problems with high exotherms and with high unreacted isocyanate content.

On a commercial scale polyurethane foams are produced by the one shot process or the prepolymer process. One shot polyether foams form the bulk of the flexible polyurethane foam now being manufactured. Quasi-prepolymer systems, also known as semi-prepolymer systems, are rarely used with flexible foam but are more important with the rigid and semi-rigid materials.

Another method for producing foams involves the thermally induced phase separation (TIPS) of polymer solutions. Foams formed in this way are usually termed microcellular because of their small pore diameters, typically 0.1 - 20 μm, compared with conventional closed-cell foams made by other methods using a blowing agent, typically 100 - 200 μm. In this technique a polymer and solvent are heated above their melting temperatures to achieve a homogeneous polymer solution. The solution is quenched in a controlled fashion in order to initiate thermally induced phase separation, either through liquid-liquid separation or polymer crystallisation. An isotropic morphology usually results from using liquid-liquid phase separation, whereas an anisotropic morphology results from liquid-solid phase separation via solvent crystallisation. Sometimes both processes occur during the thermal processing of a polymer solution. Once phase separation has occurred the solution can be frozen, which effectively terminates phase separation. The solvent is removed by sublimation of the frozen solvent (or by vacuum sublimation when a suitable solvent is used) or by liquid extraction of a resulting gel leaving behind a microcellular polymer foam. By controlling the phase separation during the quench, the foam density and cell size can be varied. However, foams made by phase separation generally display a range of complicated, ill-understood morphologies that are hard to describe.

3.2.3 Production methods

There are several methods of producing the final product; foams can be either formed into a continuous bun, or stock, spray-formed using a variety of techniques, or moulded. Each process requires that the mixing head delivers and dispenses the foam ingredients smoothly in order to minimise air entrainment or splashing. Figure 3.2 gives a summary of
some of these foam production methods.

A continuous foaming processes, known as slabstock, uses a reciprocating head that distributes freshly mixed liquid across the flat bed of a three-sided conveyer. In this process, mainly used for thermosets, foam expands freely and cures as it is carried away from the dispenser. The reaction is exothermic and the heat produced assists curing. The foam formed nearest the external surfaces generally experiences complex effects due to steep thermal gradients and the formation of natural skins. The regions of concentrated drag forces produce irregular cell orientation and abnormal cell elongation. Such anisotropy of structure is common in low density foams and is less pronounced in foams with finer cells and higher densities. Both polyester and polyether one shot foams are produced by the continuous foaming process, and are generally reacted with toluene diisocyanate.

Some foams can be formed by spraying techniques. Sprayed coatings of the reacting mixture, which are atomised in special mixing heads, are usually built up on a target surface by several successive passes, each laying a small amount of foam. Foams produced in this manner often encounter little resistance to the foaming process which occurs, since the foam is free to expand in all directions. If the foam ingredients and processing parameters are not metered accurately, this may result in an unstable foam being formed, which is likely to have a very non-homogeneous cellular structure.

The other main method for producing the final product is the foam moulding process. In many flexible foam moulding processes, after the ingredients are placed into a shaped cavity, the mould is closed, and the material foams expanding to fit the shape of the cavity. After curing, the mould is then opened, the foam part removed, and a fresh layer of silicone release sprayed on to the mould. This type of procedure is also encountered with foamed-in-place techniques.

Foamed-in-place horizontally (FIP-H) techniques seldomly lead to liquid being distributed perfectly within the mould. Cavities are therefore overfilled or packed to establish intimate contact with the top mould face, and some lateral flow results. For large parts, the mould will be filled from several nozzles carefully located to provide good distribution of the plastic and high quality surface finish. Foamed-in-place vertically (FIP-V) methods allow the foams to expand through approximately two-and-a-half metres or more of narrow passageway when filling high rise moulds. Frictional drag is therefore appreciable and the material flow characteristics usually change significantly during the rise.

Microvon components are produced by injecting the reacting mixture into a closed mould. This is usually achieved by having a large number of moulds on a rotating carousel passing a fixed injection point. Generally, urethane mixtures can be poured directly into a cavity or mould, whereupon the liquid flows into recesses and cracks, since the foam has good flow properties before curing, to form a strong seamless part. The polymer forms a dense skin in contact with the relatively cool mould walls at 45 °C, trapping gases in the foam centre. Surface depressions, or skin-holes, which are surface defects, may occur during thermal contraction. The parts produced by this process are entirely stress free and do not distort
during oven curing.

Differences in production process, formulation compounding equipment, foam stabilisation temperature and other, often uncontrollable factors, produce the considerable spread of characteristics of foam components. Many of the production methods described above tend to impose restrictions on the flow of the polymer, so that the cells become distorted. Variations in morphology within individual components is also common. The non-homogeneous morphology which occurs noticeably in the production of many foams poses problems with respect to material characterisation and ultimately in predicting part performance.

3.3 PROPERTIES AND APPLICATIONS

Cellular materials, in particular polyurethane foams, have been classified in a number of ways, either by their density, cell structure, average molecular weight between cross-links, or by the type of solid polymer. The dividing line between the groups, in each case, can be vague and hard to define. This is because, even though the physical properties of the various types can be very different, the chemistry involved is essentially the same. As discussed previously, these variations are not only derived from the type of chemistry employed, but also from the manufacturing technology and production methods used.

The two parts of a cellular material, the gas component and the solid component, contribute to the properties of the material in different ways. The gas component is of relatively low density, no direct strength and low thermal conductivity, and represents the major portion by volume. The solid component is of relatively high density, high strength and high thermal conductivity, and represents the major portion by weight. Essentially, the gas reduces product weight of a cellular material and resists the passage of heat, while the continuous solid provides rigidity and strength, and discourages internal fluid transfer.

This means that polyurethane foams can have many different properties, which makes them one of the most versatile of the foamed polymers. These properties include: low density, high strength to stiffness ratio, good energy absorption, high resilience, good adhesion, low thermal conductivity, superior durability, good ageing properties, and they also have high resistance to many organic solvents, rot and vermin. Polyurethane foams are also easy to fabricate, and they have the ability to be moulded, sewn, stitched, stapled and glued to numerous similar and dissimilar materials.

The low strength and large compressive strains that can be achieved with polyurethane foams makes them attractive for energy absorbing applications. It is the low stiffness of the foams that makes them ideal for a wide range of cushioning properties, and polyurethane foams have become the standard foam for seating. The low density of the foams permits design of light components, such as sandwich panels and large portable structures, and also allows effective buoyancy and floatation components to be produced.

The good insulating properties of cellular materials is due to their low density and correspondingly high gaseous volume content. In fact, many very low density polymeric foams,
have thermal conductivities similar to that of air. Conductivities may be even lower if the closed-cells contain some of the hydrocarbons, or other compounds that have a high molecular weight and low permeation coefficient, used for forming the bubbles since hydrocarbon vapour is of lower thermal conductivity than air. The low thermal conductivity allows cheap, reliable thermal insulation that can only be bettered by expensive vacuum based systems.

Although thermal conductivity through gases is much lower than through solids, the amount of heat transferred through the gas phase in a foam is usually the largest component of total heat transfer because of radiation through the large gas phase volume. Radiation heat transfer normally accounts for about a third of the total heat transfer in foams filled with chlorofluorocarbon.

Cellular materials have better acoustic, thermal and electrical insulation than solid polymers, and they also exhibit improved mechanical damping, dielectric properties, and usually higher flexibility. In the case of flexible foams, the properties are similar to those of natural rubbers and SBR foams, but polyurethane foams are also less flammable and have better resistance to ageing, oxygen and ozone. The latest flexible polyurethane foams are even offering competition in applications traditionally associated with latex foam rubber and flexible vinyl foams.

In general, the variety in properties of the different types of foam leads to a marked difference in their application. The flexible foams, because of their flexibility and high energy absorption, can be used as cushioning material, as safety padding and as spring aids. The uses of rigid foams depend on whether or not the foams are brittle or ductile. Brittle foams are normally used as thermal insulators, and ductile foams as construction materials.
Figure 3.1: A schematic representation of the three RIM manufacturing techniques.

Figure 3.2: Summary of foam application processes.
(FIP-H - foamed-in-place horizontally, FIP-V - foamed-in-place vertically)
4. CELLULAR MORPHOLOGY

4.1 ORIGINS IN MANUFACTURE

As discussed in the previous chapter, foams can be produced in a variety of ways. Their formation generally involves the production of gas bubbles in a liquid system which is being hardened by polymerisation, heat or catalytic actions. The process can be broken down into approximately three stages; nucleation of gas bubbles in a liquid polymer system, bubble growth, and stabilisation/solidification of the polymeric phase by cross-linking or cooling to give a structurally stable cellular system. These steps are represented schematically in figure 4.1.

Bubble nucleation has several sources: dissolved gases that are normally present in the polymer solution are forced into supersaturation by increasing temperature; low boiling-point liquids, that are incorporated into the system as blowing agents, are forced into the gas phase by increased temperature or decreased pressure; gases produced as blowing agents, for example, by the water-isocyanate reaction; chemical blowing agents that decompose thermally to form a gas. The cells, so formed, are usually composed of air, but they may contain other gases or vapours generated during foaming.

Physically, the effects of temperature, pressure and in some cases humidity are a number of conditions that can affect bubble nucleation. Other important, but less obvious, parameters are surface smoothness of the substrate, presence and concentration of certain surfactants and the rate of gas generation.

In order that the free energy of the system is a minimum, the initial bubble assumes a spherical shape on nucleation and grows as a result of the interaction of the differential pressure between the inside and outside of a cell and the interfacial surface tension. The pressure difference between the cells is related to their size difference, so that, in order to equalise the pressures, smaller bubbles tend to grow faster and coalesce with any neighbours. They contact other cells either by breaking the wall separating cells, or by diffusion of the cell fluid through the cell walls, from the small to the large cells. The points of contact of every bubble with others gives rise to thin plane surfaces (regarded as cell walls, faces or membranes), while the adjacent regions remain thickened (forming struts or ribs). When these boundaries reach other growing boundaries, edges and corners will develop. This tendency for cells to coalesce minimises the interfacial area, reducing the required free energy change, so that reduction of the surface area becomes the overriding factor in establishing the overall geometry.

As the cells grow in size and number (either by diffusion of gases coming out of solution into existing bubbles, coalescence of smaller bubbles, or by thermal effects of internal bubble pressure) the liquid phase between them is stretched thinner. This effect is known as wall-thinning and causes liquid to drain from the cell walls, by gravitational and capillary forces, into the lines of cell intersections forming the struts. This accumulation of the solid in the cell
struts thickens them relative to the cell walls, so that the volume of the polymerising liquid between the bubbles becomes small in comparison to the bubble volume. This is true for closed-cell materials, in which it is rare that solid is uniformly distributed between the struts and the walls. In many closed-cell foams, the transverse dimensions of the cell edges are about two orders of magnitude larger than the membrane thickness, since the cell membranes, occupying a few weight percent of the foam, are relatively thin and fragile. If, however, the foam is open-cell, all the material is drawn into the struts which form the cell edges and no cell walls are present. Therefore, in order to produce low density closed-cell foams, there must be careful control of the factors that influence membrane thinning.

The extent of the liquid drainage from walls to struts is generally less for thermoplastic than for thermosetting materials and less for hard than for soft polymers. A high viscosity limits this drainage, and can generate forces which are larger than those of surface tension giving structures which are not at equilibrium. Such an increase in viscosity may be caused by chemical reactions which increase molecular weight through polymerisation, cross-linking or by temperature reduction of high-molecular-weight thermoplastics.

The rate of growth of the cell appears to depend on such factors as the viscoelastic nature of the polymer phase, the blowing agent pressure, the external pressure on the foam and the permeation rate of blowing agent through the polymer phase.

Towards the final stages of bubble growth, the cell wall membrane thinning can continue to the point where the cell walls may incur damage. This damage can occur because of a combination of either drainage, high interfacial surface tension, low polymer strength or attractive Van der Waals forces promoting further thinning across the membranes. Therefore, if the structure is not stabilised, further drainage results in the rupture of cell membranes, which may be arrested at the edges to produce open-cells, or which may progress further to produce large voids or even cause collapse of the foam. The rupturing of membranes is typically marked by a sigh, or slight contraction of the foam, as the internal foaming pressure is relieved. This rupturing becomes a very important characteristic of most polymer foams and affects properties such as thermal conductivity, moisture absorption, breathability and load-bearing capabilities.

Bubble stability during growth is a function of the type and concentration of surfactant, rate of bubble growth, viscosity of the fluid medium, differential pressure variations, and pressure of cell-disrupting agents such as liquids or gases. Although, ultimate stabilisation occurs as a result of either chemical reaction continuing to the point of complete gelation or the physical effect of cooling below the second order transition point to prevent polymer flow.

As the final solidification is approached the previously formed bubbles may be distorted by mechanical restraints as the foam expands and grows freely along lines of minimum local resistance. Although these lines tend to follow the nominal bulk foam rise direction, cells are commonly elongated and oriented quite differently in various sections of a product. The extent of such distortion seems to be a complex function of mechanical forces on expansion, both internal and external, which can be due to mould shape, foam density, flow characteristics, and
also viscosity, surface tension and adhesion. These mechanisms suggest that vertical and horizontal cell walls and edges may contain unequal thicknesses of the polymer in the cells. Most foams are, to a greater or lesser extent, anisotropic, because of cell elongation in the rise direction, and in some cases they can be twice as stiff in the rise direction as they are in the plane normal to it. This, along with the fact that cell elongation is most clearly visible from the side, emphasises the importance of studying structures from several directions. The effect of anisotropy must, therefore, be considered when evaluating physical properties of cellular foams by obtaining samples oriented in specific directions to the flow process. Such anisotropy of structure is common in low density foams, because of the high gas volume, and is less pronounced in foams with finer cells and higher densities.

Closed-cell foams are usually produced where some pressure is maintained during the cell formation process, whereas open-cell foams are formed during a free expansion. Since closed-cell foams have cells with membranous walls from which most of the liquid can be drawn into the cell edges they have been known to behave like open-cell foams. The properties of any cellular solid, therefore, depend on the amount and distribution of polymer in space.

The final distribution of the polymer between walls and struts is determined by the gravitational and capillary forces causing the drainage, the viscous forces and surface area opposing the drainage, and the gas pressure exerted on the cellular structure before its stabilisation in the expanded state. It is these principles of bubble nucleation, growth and stabilisation that anticipate the foam structure.

4.2 CELL TOPOLOGY

The shape of a cell depends on the packing arrangement of the bubbles before expansion, see figure 4.2. In principle the simplest form for a foam structure will contain identical spheres uniformly distributed in a solid material, i.e. a mono-dispersion of spheres. Since spheres of identical size can be packed rigidly in at least five different regular patterns, not counting random arrangements, no unique configuration can be obtained in this manner unless some restrictions, other than density, are imposed on the system. Face-centred-cubic and hexagonal-close-packed configurations both have a packing density of approximately 74%, the maximum possible density of packing uniform spheres. A body-centred-cubic arrangement has a packing density of approximately 68%. These small packing densities are in contrast to values of 95% to 97% observed in actual low density foams.

Densities can be made to drop much further if smaller spheres are introduced along-side the larger ones, i.e. a poly-dispersion of spheres. However, this configuration is very unstable since adjacent bubbles coalesce more readily as their size ratio increases. Consequently, neither the mono-dispersion or poly-dispersion structure represents a typical foam.

More plausible values of packing density are obtained if the initially spherical bubbles in the foam are allowed to expand, so that they slide against each other to form a more or less closely
packed geometry. The contacting surfaces between bubbles mutually deform and become flat and tangential to the point of contact. In the final expansion, as work is expended to remove liquid from the close-packed system, the spherical bubble loses its shape to become a polyhedron. The structure consists of relatively thick ribs forming the boundary of the polygon for each of the faces, with very thin membranes stretched across them. A hexagonal-close-packed arrangement will turn each sphere into a trapezo-rhombic dodecahedron with six equilateral trapezoids and six congruent rhombi, while the face-centred-cubic format will transform each sphere into a rhombic dodecahedron, the twelve faces of which are congruent rhombic. Plateau identified the cell shape of a foam as being a rhombic dodecahedron, while assumed that the morphology of a foam could be represented by the interaction of both the rhombic and trapezo-rhombic dodecahedra, though his model has been regarded by some as unrealistic. Observations by Gibson & Ashby suggest that real cells often have between twelve and sixteen faces.

Theoretically, a large variety of cell shapes are possible in foams, all of which pack to fill space. The triangular, rhombic and hexagonal prisms, the rhombic dodecahedron and trapezo-rhombic dodecahedron, and the tetrahexahedron (truncated octahedron) are true space filling bodies. All of these have been suggested, at some stage, as idealisations for the shape of cells in foams, along with others which do not pack properly by themselves unless distorted: the tetrahedron, the icosahedron, and the pentagonal dodecahedron. Illustrations of some of these shapes are presented in figure 4.3.

In discussion of shape, certain topological principles must be applied. From a geometric point of view, a cellular structure can be thought of in terms of corners (or vertices), joined by edges, which surround faces, which enclose cells. Those geometric properties of a structure, which may be classed as topological, include any property which is unchanged when the structure is stretched, distorted, or twisted without limit, so long as it is neither torn nor joined to itself during deformation. Euler first expressed the important relationship between the number of corners, edges and faces on a simple polyhedron. Euler's law, which is derived from the mathematics of space, is independent of the curvature of the faces and edges. For other topological aspects of cell shape, such as edge and face connectivity and number of contact neighbours, Lewis's rule and the Aboav-Weaire law can be used. These laws imply that efficient space filling is not the only factor which influences cell shape.

The rationalisation of cell shape in most foam systems is based on the interplay of two main factors: the local geometric requirements resulting from the need to minimise the interface energy at the junction points, and the overall requirements of space-filling which limit the possible connections between the junctions.

Usually only three surfaces may meet at an edge. Assuming the pressure in all cells to be the same, local equilibrium requires that the surfaces should meet at mutual angles of 120°. It is conceivable, though, that four or more interfaces might meet at an edge, leading to other dihedral angles, but this is most unlikely. If the fraction of edges at which more than three faces intersect were significant, an inordinately large interfacial area per unit volume is implied.
and the equilibrium requirements would not be attained.

The edges themselves tend to meet symmetrically in groups of four so that the angle between each pair of edges is 109.471°. Three edges could achieve equilibrium only if they are coplanar, which would mean that no edge exists. Four is the number normally seen, although larger numbers have been observed. However, such connectivity of the edges and faces is harder to establish in closed-cell foams than in open-cell foams.

No single plane-faced polyhedron with flat faces and stable edge and corner configurations can conform to the geometrical requirements that all the faces should join at the set angle of 109.471°. A plane polygon would be required to have 5.1043 sides if the angles were of this value. A three-dimensional polyhedron constructed from such a corner would then have to have 13.394 faces, 34.195 edges and 22.789 corners, if all its faces were flat, its angles equal, and it could pack together to fill space. A pentagon is a good approximation to a polyhedron required to have 5.1043 edges, since each of its faces has 5.0 edges. The polyhedron obtained from a regular pentagon is the pentagonal dodecahedron. The corner angles of the pentagonal dodecahedron are 108° instead of 109.471°, and the surfaces meet at 116.57° instead of the 120° required for efficient packing. However, the differences and the resulting mismatch are small and the model can be used as a reasonable approximation.

In actual foams, while five-sided figures are found in abundance, four- and six-sided figures also occur with considerable frequency, though usually most cells have faces with five edges, no matter what the shape of the cell. If, for example, the cells are typically dodecahedra, the average is 5.14, and if they are icosahedra the average is 5.40. This means that frequent sightings of pentagonal faces in foams do not mean that the cells are pentagonal dodecahedra.

It should be noted, however, that no cell in a three-dimensional foam is a simple polyhedron with planar faces and straight edges, because a planar polygon cannot have all angles equal to the tetrahedral angle. The faces of real cells tend to be curved, so that uniform chemical potential may exist within the cell. The curvature is easily visualised as a consequence of the difference of pressure between the higher-pressure bubbles and their neighbours of lower pressure, which will cause transfer of gas by diffusion from the concave to the convex side of the cell membrane. The aim is to achieve uniform curvature and minimum area, subject to certain conditions imposed at the edges and corners. The uniform curvature of each face is governed by edge positions and the slope of the face at each edge, while the reduction of total interface area is obtained by sacrifice of cells having high curvature faces.

The most stable configuration is one devised by William Thompson (Lord Kelvin). The body is based upon the truncated octahedron, which, with six 4-sided and eight 6-sided faces, can stack on a body-centred-cubic lattice to fill space entirely. It has fourteen sides, which is the maximum number that any stackable space-filling body can have. Although the truncated octahedron meets all the compatibility requirements, it fails to satisfy the local geometry requirements because the vertex angles are 120°, 120°, and 90°. Surface tension equilibrium requires that they all join at 109.471°. Lord Kelvin showed that a simple distortion of the
truncated octahedron to what he called the minimum area tetrakaidecahedron will meet all the requirements. He also showed that, if the edges of such a polyhedron are curved so that each angle has the prescribed value of 109.471°, the compatibility conditions can be satisfied. This can be done by giving the hexagonal faces of the truncated octahedron a double curvature with zero mean curvature.

The Kelvin tetrakaidecahedron, or cubo-octahedron, is often considered to be the archetype of equilibrium cells and is a popular choice of cell shape because it has an average number of edges per face, and of faces per cell. This seems to match observations of grains in metals and cell shapes in some biological tissues; but the matter has not been thoroughly investigated. The shape of a tetrakaidecahedron, being irregular, is difficult to analyse. So for analytical treatment of foam properties, it is easier to analyse the pentagonal dodecahedron which has a surface-volume ratio close to that of the tetrakaidecahedron, and a simpler geometry. Although, even in an idealised structure consisting of dodecahedra stacked together (disregarding the topological incompatibility), an analysis relating load and elastic deformation would be complex and would probably require further simplifications.

Recently, a more effective packing system than the tetrakaidecahedron has been achieved with the Weaire-Phelan structure which has a repeating unit containing eight interlocking bubbles of two different shapes. The intricacies of this model, however, are likely to make it even less desirable for analytical treatment than either the pentagonal dodecahedron or the tetrakaidecahedron.

Due to the complications that arise when studying the types of cellular structure described above, many theoreticians utilise the essential simplicity of the Voronoi foam. If bubbles all nucleated randomly in space but at the same time, and all grew at the same linear rate, then the structure formed is a Voronoi foam. Figure 4.4 shows an illustration of a section through a typical Voronoi foam. The cells of such a foam fill space and are random, but they have curiously angular cells, and cells with, on average, 15.54 not 14 faces. However, real cells, if the shape is governed by surface tension, do approximate tetrakaidecahedra.

4.3 REAL CELLULAR STRUCTURES

Various researchers have studied the final shape of the cell edges that form in cellular systems. Christensen, in his examination of cell structures, assumed that the edges of cells were circular in cross-section, while Gibson & Ashby idealised them as being square, see figure 4.5. Other researchers have suggested that the struts may be triangular or represented by a thick-ended triangular bar. Observations of real struts typically show them to be concave, and the cross-section, while approximately triangular, is better described as a three-sided hypo-cycloid.

It is not only the cell edges which give rise to problems when trying to identify a particular shape, since in practice, the polyhedrons which form a cell may not necessarily be regular because their shape depends on their position in the cell. This can lead to the foam having a
Swiss cheese structure rather than a polyhedral structure, and makes analysis more difficult. Such foams do not have easily definable cells and the expression cell shape takes on new meaning. In these cases, a cell is an irregular void or gas volume surrounded by mass whose physical character and connectivity are neither easily definable nor necessarily regular.\textsuperscript{12}

Irregularities can occur in abundance, for instance, anisotropic cells and large voids are common place in many foam systems. These defects are generally caused by mechanical restraints imposed during the foaming process, and by problems encountered with the stabilisation of the foam system. This gives cellular materials a much more complex structure in reality, and describing these materials using a single unit cell, with specific strut shapes, may be an oversimplification of the macrostructure.

The cellular structure of microvon has been studied in some detail using scanning electron microscopy and image analysis techniques. The results of the work, both on the as-received and deformed material, are presented in the following chapter.
Figure 4.1: Diagrammatic representation of the steps involved in foam formation\textsuperscript{21}.

Figure 4.2: Illustration of (a) mono-dispersion, (b) poly-dispersion, (c) capillary flow and (d) polyhedral arrangement\textsuperscript{56}.
Figure 4.3: Table describing some of the cell shapes that have been suggested as a unit cell (the digits refer to the number of cell faces, edges and corners respectively).
Figure 4.4: Illustration of a section through a Voronoi foam. (The dots indicate the random points of nucleation)

Figure 4.5: Typical representations of the struts in cells.
5. MICROSTRUCTURAL INVESTIGATION

The type of flexible polyurethane foam examined throughout the microstructural investigation was *microvon* grade 90M808, a common grade of the foam, produced by Dunlop Suspensions and Components Ltd. The material was supplied in two forms; slabs (part PM10000) and cylinders (part MI118000). The slabs, measuring 168x104x10 mm, were supplied in three densities; 400, 520 and 650 kg m$^3$, while the cylinders, measuring Ø30x260 mm, were supplied in four different densities; 350, 530, 700 and 1050 kg m$^3$.

5.1 AS-RECEIVED MATERIAL

Extensive scanning electron microscope examination of the as-received cellular material has been conducted. This work was undertaken to study, compare and quantify the microstructure of the different density materials, thereby giving an indication of the variation in cellular structure of *microvon*.

5.1.1 Structural observations

The polyurethane foam was found to be a difficult material to cut accurately because of its flexibility. Initial attempts at cutting the material involved using a band-saw and also fracturing after immersion in liquid nitrogen. Both of these methods failed to produce a surface finish of high enough quality for scanning electron microscopy. A *cleaner* surface was achieved by using a surgical scalpel blade, which did not tear or rip the underlying cell structure. In order to observe the surfaces of the material using the scanning electron microscope the specimens had to be sputter-coated with gold before examination so that a clearer image, with a greater field of depth, could be achieved. Scanning electron micrographs (SEMs) showing the difference between cutting the material with a band-saw, fracturing it in liquid nitrogen and using a scalpel are presented in figures 5.1a, b and c respectively.

The SEMs in figure 5.2 compare *microvon* with different types of cellular structure. Figure 5.2a is a SEM of *microvon*, which has a closed-cell structure, while figures 5.2b and 5.2c show a typical open-cell and semi-closed-cell foam respectively, the formulation and density of which are unknown. Cellular materials such as these can have different amounts of material in the cell edges and/or faces; it is the relative contribution of these that aid in determining the properties of the foam.

A selection of specimens were removed from the 400, 520 and 650 kg m$^3$ slabs of the material in order to examine the cellular structure. The SEMs in figures 5.3 to 5.5 show pictures of the general structure of the various densities of material at two different magnifications. Further studies of the microstructure show it to be inhomogeneous; figures 5.6a, b and c show the type of anomalies that can occur within the microstructure of all three densities of material, such as, excessively large cells or voids.
Specimens were also cut to examine the depth of the surface skin region of the slab material. The SEMs in figure 5.7 show the change in structure that occurs near to the surface region of the material, in a sample with a density of 650 kg m\(^{-3}\). This is caused by the way in which the warm foamed polymer cools down on contact with the walls of the mould it is injected into. Another consequence of injecting a reacting mixture into a mould is that the material tends to flow non-uniformly and can lead to an anisotropic cell structure. The SEMs in figures 5.8a, b and c show that cell anisotropy can be present in microvon components, for material with a density of 400, 520 and 650 kg m\(^{-3}\) respectively.

Cylindrical foam specimens, of density 350, 530, 700 and 1050 kg m\(^{-3}\), were also examined under the scanning electron microscope; complete cross-sections of the four different density materials are shown in figures 5.9 to 5.12. SEMs of a higher magnification, taken from the previous montages, are shown in figures 5.13 to 5.16 respectively. The markings on the solid polymer in figure 5.16 are formed by the cutting process and are not features of the material.

5.1.2 Image analysis

In many cases the structure of cellular materials consist of a wide range of different cell sizes that makes determination of parameters to describe and characterise the cellular structure somewhat difficult. Because of this variation in individual cell size it is generally convenient to record the number of cells per unit length rather than the actual size. This is usually done by measuring the number of pores per linear twenty-five millimetre length, and gives rise to a measure known as the cell count\(^{23,24,29}\). In order to achieve this, scanning electron micrograph montages of approximately twenty-five millimetres were provided for both the slab and cylinder material, as shown in figures 5.17 and 5.18.

Since anisotropy tends to occur in many foams, mainly in the rise direction, it was necessary to obtain insight into the flow patterns existing in both the slab and cylinder moulds. If anisotropy were present in the material, it suggests that a difference may be noted in the cell count depending on the plane in which the foam is sectioned.

The final structure of the cellular material varies depending on whether the slab or cylinder material is examined, because of the different way in which the reacting foam ingredients flow into, and cure in, the mould. The technique for moulding the slabs and cylinders is the same, since both are formed by filling a mould vertically from an injection point near to the base of the mould. In the case of the slab mould a varying flow pattern is observed over the whole component, while, because of the geometry of the cylindrical moulds, the material sustains a uniform flow pattern as it rises, see figure 5.19. Therefore, sectioning the foam slabs in the vertical and horizontal planes can give rise to uncertain orientations of cell with respect to the rise direction. However, the flow pattern formed within the cylindrical foam mould means that if the material is sectioned in the axial direction, some estimation of the cell count in the rise direction can be determined. If the foam is then sectioned in the circumferencial direction, perpendicular to the rise direction, a different cell count may be achieved.
Thus, the cylindrical specimens have been studied in two planes; axially and circumferentially, or transversely, to investigate the possibility of anisotropy within the material, while the slab material has been investigated purely in-plane since the orientation of cells obtained during sectioning of the material remains unknown. A graph has been plotted, figure 5.20, to show the variation in cell count with material obtained from the slab and cylinder moulds.

Visual counting along a length of twenty-five millimetres is very difficult, because the cells are not on a string. This means that, since the cells are not located in horizontal layers after cutting the foam, the newly cut surfaces will always show missing struts, different levels and orientations of cells, and intermediate small pores. A cell count, therefore, can only provide a measure of the coarseness of a foam.

An image analysis system, Quantimet 500+ (version 2.0a), was used on SEMs of the different density material in order to provide some statistical information on the different cell structures. The SEMs that were analysed are shown in figures 5.21 to 5.27, along with bar charts which show the size distribution of cells recorded using the Quantimet software. The SEMs and graphs are shown in order of increasing specimen density, from 350 to 1050 kg m\(^{-3}\).

The information obtained from the image analysis is summarised in figures 5.28 to 5.31. The graphs have been plotted to show the relationship between certain aspects of the structure and specimen density. The graphs in the figures show how the relationship between percentage area of voids, cell count per square millimetre, mean cell area, and mean equivalent cell diameter all vary with specimen density. The mean equivalent cell diameter is defined as the diameter of a circle having the same area as the feature. The graph in figure 5.32 shows the variation in aspect ratio for cells at different densities, and indicates the range and deviation from the mean.

The image analysis undertaken has provided some useful observations of the general structure of different densities of microvon. However, one of the main problems of all cell size determinations is that they are applied to a population of data which is statistically insignificant when related to the volume of the material.

5.1.3 Cell wall thickness

The cell wall thickness of the material could not be determined directly using the image analysis system, so a number of simple models, relating the cell wall thickness to mean cell diameter and volume fraction of the solid material, have been derived, see Appendix I.1, and are given below.

\[
\begin{align*}
t_s &= \frac{d}{2} \left( \frac{1}{\sqrt{1 - V_r}} - 1 \right) \\
t_{sw} &= \frac{d}{4} \left( \frac{V_r}{1 - V_r} \right) \\
t_{sm} &= \frac{d\sqrt{3}}{8} \left( \frac{V_r}{1 - V_r} \right)
\end{align*}
\]
where \( t_1 \), \( t_2 \), and \( t_3 \) represent the cell wall thicknesses based on the geometries shown in figure 5.33, \( d \) is the cell diameter and \( V_f \) the volume fraction of solid material.

Information about the mean cell diameter and percentage of voids, taken from the image analysis, were then used to estimate values of cell wall thickness by applying the data to the models described above. A graph of the results is shown in figure 5.34. The values of cell wall thickness plotted are actually twice those determined from the equations since they represent half the solid material between two cells. These data were then used to calculate new values of the cell count per linear twenty-five millimetre length, using a cell count based on a repeating factor of mean cell diameter and cell wall thickness as indicated in figure 5.35. The results of the cell count per linear twenty-five millimetre length have been replotted in figure 5.36, and compared with the results determined using equation (5.1). Since the models given in equations (5.1), (5.2) and (5.3) gave similar values for the cell wall thickness, only the model given in equation (5.1) has been plotted against the actual results.

5.2 DEFORMED MATERIAL

Some scanning electron microscopy has also been conducted to study the structure of the deformed material. This work was undertaken to try to visualise the deformation modes encountered within the structure during compression. The deformed material was studied in two ways; statically using a miniature vice, and dynamically using a specially designed miniature compression rig.

5.2.1 Static deformation

SEMs have been taken of a wedge section of polyurethane foam, of density 520 kg m\(^{-3}\), which has been clamped and compressed as shown in figure 5.37. This was done in order to visualise what happens to the microstructure when compressed to different amounts of strain. Close-up photographs of the wedge, compressed to approximately 40\% and 50\%, are shown in figures 5.38a and b respectively. These SEMs indicate that the cellular structure of the material has begun to collapse along localised bands. A montage of the full cross-section of the deformed wedge is shown in figure 5.39.

A number of compression set tests were performed, discussed in the following chapter, and the permanently deformed material has been sectioned and examined. Photographs showing the permanent set sustained after holding different densities of material at 75\% compressive strain for a period of twenty-four hours at approximately 80 °C, are shown in figures 5.40 to 5.43. The photographs in these figures, which show the 350, 530, 700 and 1050 kg m\(^{-3}\) material respectively, indicate how the compression set test, under certain conditions, has caused permanent rearrangement of the cellular structure. Each of the figures shows the shape of the permanently deformed sample along with two SEMs; the first showing the collapsed structure near to the edge of the sample, and the second showing the state of the structure in the bulk of the material. The SEMs presented for the 1050 kg m\(^{-3}\) material appear to show very few, if
any, unclosed cells within the structure. This may be as a result of complete densification, where the gas has diffused out of the cells, or it may be due to the sputter-coating filling in the existing cells.

5.2.2 Scanning electron microscope compression rig

A compression rig, for use inside a scanning electron microscope, has been designed in order to observe the microstructural changes that occur during deformation. A schematic and photograph of the rig are shown in figures 5.44 and 5.45 respectively, while design drawings of the device are shown in Appendix II.1. The idea behind the device is that, a specimen placed between the two compression plates in the rig will deform such that the point of focus will remain central.

The specimen was held between two compression plates which were attached to two sets of ball screw nuts. The compression plates, in turn, were joined to two right-hand or two left-hand ball screw nuts fitted on to two shafts. Each shaft consisted of half right and half left-hand threads, corresponding to the associated ball screw nut, and were supported at their ends by bearings set into end plates. At one end of the shafts, sprockets and a chain were attached in order to link the two threaded shafts together. A third sprocket was also used to allow removal of slack from the chain.

Since the inside of the scanning electron microscope had to be under vacuum the equipment had to be free from all substances that could be drawn off by the vacuum, therefore ball screw nuts were used instead of standard screw threads since they required no grease. The chain and sprockets, used to drive the device, were also grease free.

Some means of driving the device externally, whilst maintaining a vacuum, had to be derived. This was achieved by modifying one of the spare vacuum sealed port plates to allow a square drive to pass through the side of the vacuum chamber. The drive was then attached to the compression rig via a flexible coupling and stud, bonded to one of the shafts. A handle was attached to the sealed port plate so that a means of turning the shaft, thereby compressing the specimen, was available. A calibrated dial was also used in conjunction with the handle so that the amount of compressive displacement could be measured.

Cubic specimens, approximately 15x15x15 mm, taken from the 350 and 700 kg m$^{-3}$ material, were examined in the compression rig. Figures 5.46a and b show photographs of the 350 kg m$^{-3}$ material in the device, undeformed and compressed to approximately 50% respectively. The microscope was focused on a single cell at the centre of the specimen and the handle turned in steps, a millimetre at a time, so that the structure could be photographed at intervals.

Initially, specimens were sputter-coated with gold, but deformation of the material caused cracking of the coating, giving rise to an unrepresentative image. Figures 5.47a, b and c show a coated 530 kg m$^{-3}$ density sample compressed to approximately 5, 20 and 35% respectively. Further specimens were therefore placed into the scanning electron microscope uncoated.
SEM sequences of the deformation of single cells, from 15x15x15 mm samples of the 350 and 700 kg m\(^3\) material, with the corresponding amount of deformation, are shown in figures 5.48 and 5.49. Some image analysis was conducted on the SEMs of the single cells to investigate the change in cell area during compression, and also to see the change in aspect ratio. The results obtained are plotted in the two graphs shown in figures 5.50 and 5.51.

Information from mechanical tests conducted on similar 15x15x15 mm cube samples, were then used to produce the plot shown in figure 5.52. The figure shows the load/deflection curves corresponding to compression of the specimens used for the montages in the two previous figures. The points on each curve represent the stage at which SEMs of the cell were taken.

Subsequent to the microstructural investigations, an extensive testing programme was undertaken to study the behaviour of microven under various loading and environmental conditions. This work has mainly concentrated on compressive loading, but has also focused on tensile and shear deformation. All of the mechanical tests that have been performed are detailed in the following chapter.
Figure 5.1: SEMs of microvortex cut using several techniques; (a) band-saw, (b) liquid nitrogen and (c) scalpel.
Figure 5.2: SEMs showing the different types of cellular structure; (a) closed-cell, (b) open-cell and (c) semi closed cell.
Figure 5.3: SEMs showing the general structure of the 400 kg m$^3$ slab material.
Figure 5.4: SEMs showing the general structure of the 520 kg m$^3$ slab material.
Figure 5.5: SEMs showing the general structure of the 650 kg m$^3$ slab material.
Figure 5.6: SEMs showing the variation in cell size for (a) 400 kg m$^3$, (b) 520 kg m$^3$ and (c) 650 kg m$^3$ density material.
Figure 5.7: SEMs showing the densification of the material near to the skin region for a 650 kg m$^3$ density material.
Figure 5.8: SEMs showing the occurrence of anisotropy for a (a) 400 kg m$^3$, (b) 520 kg m$^3$ and (c) 650 kg m$^3$ density material.
Figure 5.9: Cross-sections of a 350 kg m$^3$ cylindrical specimen.

Figure 5.10: Cross-sections of a 530 kg m$^3$ cylindrical specimen.
Figure 5.11: Cross-sections of a 700 kg m$^3$ cylindrical specimen.

Figure 5.12: Cross-sections of a 1050 kg m$^3$ cylindrical specimen.
Figure 5.13: Higher magnification SEM of the 350 kg m\(^3\) montage shown in figure 5.9.

Figure 5.14: Higher magnification SEM of the 530 kg m\(^3\) montage shown in figure 5.10.
Figure 5.15: Higher magnification SEM of the 700 kg m\(^3\) montage shown in figure 5.11.

Figure 5.16: Higher magnification SEM of the 1050 kg m\(^3\) montage shown in figure 5.12.
Figure 5.17: Montages of all the slab material used in the cell counts.
Figure 5.18: Montages of the cylindrical specimens used in the axial and transverse cell counts.
Figure 5.19: Schematic of the flow patterns associated with the slab and cylinder material. Sizes are shown relative to one another, arrows indicate injection point.

Figure 5.20: Graph showing the change in the linear cell count with density.
Figure 5.21: SEM of the 350 kg m\(^3\) material used for image analysis, with a corresponding bar chart of cell size distribution.
Figure 5.22: SEM of the 400 kg m$^3$ material used for image analysis, with a corresponding bar chart of cell size distribution.
Figure 5.23: SEM of the 520 kg m$^{-3}$ material used for image analysis, with a corresponding bar chart of cell size distribution.
Figure 5.24: SEM of the 530 kg m$^{-3}$ material used for image analysis, with a corresponding bar chart of cell size distribution.
Figure 5.25: SEM of the 650 kg m$^3$ material used for image analysis, with a corresponding bar chart of cell size distribution.
Figure 5.26: SEM of the 700 kg m$^3$ material used for image analysis, with a corresponding bar chart of cell size distribution.
Figure 5.27: SEM of the 1050 kg m$^{-3}$ material used for image analysis, with a corresponding bar chart of cell size distribution.
Figure 5.28: Graph showing the variation of percentage of area of voids with specimen density.

Figure 5.29: Graph showing how the cell count per unit area varies with specimen density.
Figure 5.30: Graph showing the variation in mean cell area with specimen density.

Figure 5.31: Graph showing how the variation in mean equivalent cell diameter with density.
Figure 5.32: Graph showing the variation in cell aspect ratio with specimen density.

Figure 5.33: Schematic showing the geometry of the models used to estimate cell wall thickness.
Figure 5.34: Graph showing the variation in estimated cell wall thickness with density.

Figure 5.35: Schematic showing the geometry of the repeating unit used to estimate the linear cell count.
Figure 5.36: Graph showing the change in the linear cell count with density plotted with estimated values using three different models. Solid lines are based on experimental results, while the dashed line represents the results based on equation 5.1.
Figure 5.37: Schematic of the static compression of foam within a vice.

Figure 5.38: Close up SEMs showing the bands of folding within a 530 kg m$^{-3}$ material compressed to approximately (a) 40% and (b) 50%.
Figure 5.39: SEM montage of the 520 kg m$^3$ foam material compressed to a variety of strains along the length of the vice.
Figure 5.40: Pictures showing the permanently deformed structure of a 350 kg m$^3$ specimen compressed to 75\% at 80 °C; (a) specimen, (b) SEM near edge and (c) SEM in bulk material.
Figure 5.41: Pictures showing the permanently deformed structure of a 530 kg m$^3$ specimen compressed to 75% at 80 °C; (a) specimen, (b) SEM near edge and (c) SEM in bulk material.
Figure 5.42: Pictures showing the permanently deformed structure of a 700 kg m$^3$ specimen compressed to 75% at 80 °C; (a) specimen, (b) SEM near edge and (c) SEM in bulk material.
Figure 5.43: Pictures showing the permanently deformed structure of a 1050 kg m\(^3\) specimen compressed to 75% at 80 °C; (a) specimen, (b) SEM near edge and (c) SEM in bulk material.
(Note: strengthening plates removed)

Figure 5.44: Schematic of the SEM compression rig.

Figure 5.45: Photograph of the SEM compression rig.
Figure 5.46: Close up photographs of a 350 kg m$^3$ specimen in the SEM compression rig, (a) undeformed and (b) 50% compression.
Figure 5.47: SEMs showing the problems that occur when deforming a 350 kg m\(^3\) coated specimen, (a) after 5%, (b) after 20% and (c) after 35% compression.
Figure 5.48: Sequence of micrographs showing the deformation of a 350 kg m$^3$ density specimen.
Figure 5.49: Sequence of micrographs showing the deformation of a 700 kg m$^3$ density specimen.
Figure 5.50: Graph showing the change in cell area of the single cells with axial compression.

Figure 5.51: Graph showing the change aspect ratio of the single cells under axial compression.
Figure 5.52: Graphs showing the load/deflection curves corresponding to the sequences in figures 5.48 and 5.49, dots represent intervals when photographed.
6. MATERIAL TESTING METHODS

6.1 TEST SENSITIVITY

The aim of the experimental work was to study the mechanical behaviour of microvon under a variety of loading conditions, and in some cases, at various operating temperatures. Tests have included; uniaxial compression, tension, and shear as well as impact and compression set. The tests have mainly concentrated on the compressive behaviour of the material since this is the mode of deformation most commonly encountered with cellular foams. The information from this work was collected for subsequent analysis, with the aim of gaining a greater understanding of the mechanical behaviour of the material. The data have also been collected to provide a framework from which to develop a number of phenomenological models.

Tests conducted on flexible polyurethane foams can be sensitive to a number of factors such as the origin, shape and size of the sample, as well as the mode of deformation, strain amplitude, frequency, temperature and humidity of the test. All of these factors make conducting tests on flexible polyurethane foams difficult, and this is further complicated because the materials are also sensitive to their loading history.

The origin of the samples is important since it is known that variations in flow pattern can give rise to an inhomogeneous cell structure, which may contain some anisotropy. The variation in the material structure often leads to differences in properties between batches of the same material, which can give rise to large discrepancies when comparing experimental work with theory. In order to overcome the effects of cell size variation, and their random distribution throughout the material, it was necessary to use relatively thick test pieces. The test pieces were cut so that they were at least fifty times the average cell size, this minimised the effects of cell size variation, and also reduced any edge effects that might have been present.

Examination of the microstructure of microvon has shown that anisotropy may exist, to a certain extent, within the foam. Results, therefore, may be sensitive to the orientation of the foam, and for this reason, samples were tested consistently in the same direction during a set of tests.

It is not only the orientation of the material that is important, but also the size of the sample, and the way in which it has been cut, that can affect the outcome of a test, since it has been shown that moulded samples of foam give more reproducible results than cut samples. Thus, the applicability of data obtained from laboratory tests may be limited by the use of such cut specimens, since it involves removal of the dense skin region.

The testing of these flexible polyurethane foam specimens in the laboratory is expected to give some guidance about the likely performance of the material in service. Although, the most reliable guidance to performance is obtained by testing samples of the finished product, using procedures that simulate the loads and conditions of service. This however, does not generate
information based purely on the material behaviour since tests on the final product will invariably evaluate the design of the part.

Possible experimental uncertainties may exist due to the effect of displacement constraints at the loading surfaces. These constraints may restrict the admissible mechanisms of deformation, though it is difficult to assess the contribution of this effect on test results\(^{11}\). The mechanical behaviour of the material can also be strongly dependent on the mode of deformation imposed on the samples, for example, the response of polyurethane foam in compression is very different to its response in tension. In general, compressive tests are insensitive to defects, or flaws, such as the occasionally large cell. However, tensile tests are sensitive to these factors, and are also sensitive to eccentricity in loading, pre-damage and the manifestation of anisotropy within the test specimen\(^{12}\).

During the deformation, the variation in strain amplitude and frequency may become important since they can alter the behaviour of the material by damage to the component through over loading, or by high levels of heat build-up during cycling\(^{12}\). The sensitivity to temperature is due to the segmented structure of the polyurethane, which is responsible for the transition regimes that can occur over the working range of the material\(^{17}\). It was therefore necessary to keep a record of temperature readings during tests, and, since the effect of humidity on microvon was unknown, measurements of the relative humidity were also recorded.

For the reasons described above, it was necessary to carry out all the tests under carefully controlled conditions in order to obtain consistent, reproducible data\(^{14}\). Also, in order to overcome the problems of variability within the material, at least three identical tests for each loading and environmental condition have been undertaken, with the aim of providing reliable data.

For testing purposes, the slab and cylinder material supplied were cut into three different types of test specimen; cubes (10x10x10 mm), strips (80x15x10 mm) and cylinders (Ø30x30 mm), see figure 6.1. The slabs were cut into cubes and strips for the preliminary compression testing and tensile testing respectively, while the cylinders were cut into smaller sections and used throughout the rest of the tests.

6.2 COMPRESSION TESTING

Since polyurethane foam is used principally in compression, most of the experimentation was concentrated in this area. Tests were carried out on a variety of specimens, of different density, to study the effects of static and dynamic behaviour under various loading and temperature conditions.

The general layout of the test equipment used throughout compression testing is shown in figure 6.2. It comprises of a MAND testing frame and control unit linked to an IBM compatible personal computer through which data were sampled and subsequently stored.
6.2.1 Preliminary compressive tests

In order to test foam cubes in compression a low friction compression rig and 2 kN trapezoidal load cell were mounted into a MANDE servo-hydraulic testing machine. A 2 kN trapezoidal load cell was used in preference to the low-profile shear cell built into the machine, rated at 100 kN, in order to achieve greater precision over the testing range required. The mounting of the specimen is shown in figure 6.3, whilst a schematic and photograph of the apparatus are shown in figures 6.4 and 6.5.

Information about the axial deformation of the foam was collected using a clip gauge extensometer, placed along the length of the specimen with the aid of a lightweight block of aluminium, see figure 6.4. The block was bonded to the top of the foam cube in order to support the clip gauge and to prevent slippage during testing. The specimens were bonded with their skinned surfaces perpendicular to the direction of loading, and allowed to cure before testing.

The platens and aluminium block were designed to be at least twice the width of the specimen to ensure that the foam cubes did not deform around the platens when compressed. The specimen and aluminium block were then bonded to the lower plate of the compression rig, and the clip gauge attached.

The readings from the calibrated load cell and clip gauge were fed into an IBM compatible computer, along with position feedback data taken from the servo-hydraulic actuator. The information was sampled through a PC30D multi-function analogue-to-digital (A/D) input/output (I/O) card using a data acquisition package called Status20 (version 2.03), which allowed the information to be stored digitally on computer for subsequent analysis. This package sampled information between ±10V, and allowed up to 2048 samples to be recorded per channel.

In order to perform cyclic tests, a ramp generator, incorporated into the MANDE control unit was used, which supplied a triangular waveform. The form of the input wave is known to affect the hysteresis, a phenomenon that occurs in elastically retarded materials, since the unloading of the material progresses at a different rate from the loading. It is known that, compared with a sinusoidal input, a triangular wave produces a less significant change in the hysteresis than a square wave\(^\text{15}\). The MANDE control unit was adjusted so that the specimen would be compressed using the ramp generator, under displacement control to provide constant strain, by 5 mm, i.e. 50% compression.

Initially, three foam cube specimens of each density, 400, 520 and 650 kg m\(^{-3}\), were tested to study the recovery behaviour of the material in compression. Each specimen was cycled at 5.0 mm s\(^{-1}\) for a single cycle, and the information about the recovery recorded using the data acquisition apparatus.

After the initial static recovery tests, a programme of dynamic cyclic compression tests was performed. Foam cube specimens of each density were cycled for ten continuous cycles, at a cross-head rate of 0.5 mm s\(^{-1}\), the subsequent measurements of load and axial displacement
being recorded. The set of tests was then repeated, but at cross-head rates of 5.0 mm s⁻¹, in order to determine the effect of varying the strain rate by an order of magnitude.

To determine if there was any difference in the behaviour of the material if cycled continuously or intermittently, a set of intermittent recovery tests were performed. These tests followed the same procedure described above, cycling specimens at 0.5 and 5.0 mm s⁻¹ for ten cycles, except that a period of thirty seconds for recovery was allowed between each cycle.

All the tests were conducted at room temperature and humidity, and the results are shown in the following chapter. In all, 45 foam cube samples were tested in uniaxial compression, and a summary of the tests, test conditions and the sampling information is given in Appendix III.1.1.

6.2.2 Further compressive tests

Further compression tests were performed on cylindrical foam specimens, as described below. These were undertaken after initial analysis of the preliminary compression test data obtained from the foam cubes.

Room temperature tests

Some dynamic compressive tests were conducted on cylindrical foam specimens in uniaxial compression. A modified set-up to the one for testing foam cubes was developed, in order to obtain more detailed information. The set-up consisted of a MAND servo-hydraulic testing machine, a sindanyo environmental chamber, a 10 kN trapezoidal load cell, two alternating-current linear variable differential transformer (AC LVDT) displacement transducers and two direct-current linear variable differential transformer (DC LVDT) displacement transducers. Schematics of the specimen mounting and the test set-up are shown in figures 6.6 and 6.7 respectively, while photographs are shown in figures 6.8 and 6.9.

The two AC LVDT displacement transducers were mounted on to opposite sides of a 10 kN load cell, in order to measure axial displacement. Holes in the base of the environmental chamber allowed the cores of the transducers to enter the enclosed testing area, where the ends of the transducers were connected by a flat aluminium plate which rested on top of the foam specimen. The signals from the two transducers were fed into an amplifier and averaging unit, and the resulting signal was then fed to a computer.

The two DC LVDT displacement transducers were used to measure the lateral deformation of the specimen during testing. The transducers were attached to the frame of the testing machine, and slits were inserted into the edges of the environmental chamber to allow the cores of the transducers to contact the foam specimen. The tips of the two transducers had pivoted-pin systems attached to them, allowing them to be pushed into the centre of opposite sides of the specimen, and still being free to move in a vertical plane, see figure 6.6. These pins were glued into the two lower density specimens, 350 and 530 kg m⁻³, because of the way in which
the material deformed during testing. In the case of the 700 and 1050 kg m\(^{-3}\) specimens, the pins were pushed into the material without being bonded. The signals from the two probes were passed into an amplifier and summation unit, and again the single output was fed to the computer. All the signals were also grounded to avoid extraneous noise.

The information from the five transducers were sampled through a PC30AT multi-function A/D I/O card using a software package called Dash300 (version 2.1). This package sampled information between ±10V, and allowed up to 16000 samples to be taken, regardless of the number of channels used. This package replaced Status30 since it had the ability to store more information digitally during testing for further analysis at a later stage.

Initially, a number of tests were performed to study the effects of changing the end constraint. Specimens were tested with their ends either bonded (Loctite 406), lubricated (silicone oil FI 11/50) or left free-standing under dry friction. Each end condition was tested on cylindrical specimens from the four different densities of material, 350, 530, 700 and 1050 kg m\(^{-3}\). Specimens were cycled at a cross-head rate of 0.5 mm s\(^{-1}\) for ten continuous cycles, and compressed by 22.5 mm, i.e. 75% of their original height. For the 1050 kg m\(^{-3}\) specimens, the load exceeded the 10 kN cell range, thus requiring the use of the load cell built into the test machine, rated at either 25, 50 or 100 kN.

On initial examination of the information, little difference was noted in changing the end constraint, therefore, further tests were performed with the specimens free-standing under dry friction conditions, representative of the typical situation observed in practice.

A number of tests were then performed in which specimens were cycled for ten continuous cycles at rates of 0.5 mm s\(^{-1}\) and 5.0 mm s\(^{-1}\) at room temperature, using the apparatus described in figure 6.7. The subsequent information about the load, axial and lateral displacements were recorded using the data acquisition facility.

In total, 36 cylindrical foam samples were tested in compression at room temperature and humidity. The results of the tests are given in the following chapter, while a summary of the tests, test conditions and sampling information is given in Appendix III.1.2.

**High temperature tests**

High temperature tests, at approximately 80 °C, were simulated using a hot air-blower from which the hot air was ducted, using insulated piping to prevent heat loss, to two ports in the environmental chamber. A temperature probe, used to control the temperature of the air, was inserted into the environmental chamber, at a point furthest from the hot air inlets. The schematic layout of the apparatus was similar to that for room temperature tests in figure 6.7, while a photograph of the set-up is shown in figure 6.10. The platens and fittings used inside the environmental chamber were blacked to prevent deterioration under the hot and dry air conditions.

A number of tests were conducted using a NiCr/NiAl Chromel/Alumel (type k) thermocouple, inserted into the centre of the specimen, in order to determine the time
necessary to heat the specimen to the required temperature. This showed that the centre of the specimen achieved the temperature of the surrounding atmosphere, at 80 °C, approximately thirty minutes after the initial heating of the specimen. Therefore, for each test, the environmental chamber was heated at 80 °C for fifteen minutes prior to testing, at which point the specimen was inserted, and heated for a further thirty minutes in order to bring the specimen up to temperature.

Tests similar to the ones conducted at room temperature were performed on the specimens, namely, samples were cycled for ten continuous cycles at cross-head rates of 0.5 mm s⁻¹ and 5.0 mm s⁻¹. The information from the five transducers was again sampled through a computer via a PC30AT multi-function A/D I/O card using the data acquisition package Dash300.

In total, 24 cylindrical foam samples were tested in compression at high temperature. The results of the tests are given in the following chapter, and a summary of the tests, test conditions and sampling information is given in Appendix III.1.2.

**Low temperature tests**

Low temperature tests, at approximately -30 °C, were undertaken using a controlled liquid nitrogen supply. The liquid nitrogen was injected directly into one of the inlets on the side of the environmental chamber via insulated pipes. The other inlet, used for ducting warm air during the high temperature tests, was sealed off.

The schematic layout of the test set-up was similar to that for room temperature tests, shown in figure 6.7, while a photograph of the apparatus is shown in figure 6.11. A solenoid valve, which controlled the flow of liquid nitrogen into the chamber, was surrounded by sixteen gauge steel, in order to reduce the strong electric field it produced, which was found to affect the signal from the load cell.

A small number of tests were conducted using a NiCr/NiAl Chromel/Alumel (type k) thermocouple to determine the duration necessary to allow the centre of the specimen to drop to the required temperature. These preliminary tests showed that the centre of the specimen dropped to the temperature of the surrounding atmosphere approximately twenty minutes after initial cooling of the specimen. For each test, the environmental chamber was cooled to -30 °C for fifteen minutes, and, once the specimen had been inserted, it was cooled for a further twenty minutes to bring the temperature of the specimen down.

A set of tests, identical to those conducted on specimens at room and high temperature were performed. The information from the five transducers was sampled through the computer via a PC30AT multi-function A/D I/O card using the software package Dash300.

In total, 24 cylindrical foam samples were tested in compression at low temperature. The results of the tests are given in the following chapter, while a summary of the tests, test conditions and sampling information is given in Appendix III.1.2.
6.2.3 Compression set

A range of compression set tests were also performed on cylindrical samples of microvon. These were undertaken in order to study the effect of prolonged compression, of varying amounts, on recovery under different operating temperatures.

Since each compression test required that a specimen be compressed at constant deflection for twenty-four hours, a compression set rig was designed and built in order to speed up the testing process. The rig allowed up to nine specimens to be compressed simultaneously, three at 25%, three at 50% and three at 75% of their original height. A schematic and photograph of the rig are shown in figures 6.12 and 6.13, while design drawings of the apparatus are shown in Appendix I. The rig, made from aluminium piston alloy, incorporated supporting columns in order to maintain the two plates parallel to one another, and to ensure that the specimens were compressed by the correct amount.

Before testing, the specimens were weighed, and their mass and height recorded. The specimens were then placed between the compression platens and compressed using a Denison testing machine. Once the top plate had contacted the supporting columns, set at the appropriate height, the four bolts on the top plate were tightened using locking nuts. The rig, along with the compressed specimens, was then moved to a suitable environment. After twenty-four hours the rig was placed back into the Denison testing machine, the load released, and the top plate removed. The specimens were then allowed to relax for thirty minutes after removal of the load, prior to their height being re-measured.

Initial tests were conducted on cylindrical foam specimens of 350, 530, 700 and 1050 kg m\(^{-3}\) at room temperature. Nine specimens from each density were compressed in sets of three to 25%, 50% and 75% of their original height for twenty-four hours.

Similar sets of tests were then conducted on foam specimens under extremes of temperature. Specimens were compressed and left at -30 °C and at 80 °C for twenty-four hours, after which time, the load was released and the specimens allowed to stand at room temperature for thirty minutes, before their height was re-measured.

In total, 108 cylindrical foam samples were tested using the compression set rig, and the results are given in the following chapter. A summary of the tests and test conditions for the compression set tests is given in Appendix III.1.3.

6.2.4 Impact

A number of impact tests have been performed on cylindrical foam specimens using a drop weight machine to simulate compression tests at high strain rates. This test equipment was developed as part of a final-year undergraduate research project\(^{22}\). The mounting of the specimen and test set-up are shown in figures 6.14 and 6.15, while photographs of the apparatus are shown in figure 6.16. The set-up consists of a weight which can be dropped from a set height down a smooth guide cylinder. The falling weight, to which extra mass can be attached, is made to impact onto a transmitting cone, larger than the diameter of the sample,
which in turn, compresses the foam specimen.

Information on the load and displacement during testing was acquired by using a 35 kN force washer, linked to a charge amplifier, and an optical laser extensometer respectively. Both devices were calibrated and fed into a four channel digital oscilloscope.

Prior to testing, the specimens were scragged, that is, they were pre-deformed for approximately three cycles, in order to ensure that the response obtained during the impact was not characteristic of the high-energy-loss first cycle seen in previous compression tests.

In order to set up the test specimens, a winch was used to raise the guiding cylinder above the test area. The specimen was then placed on a metal plate and located in a shallow recess, this in turn was rigidly attached on top of the force washer. Once located, the transmitting cone, to which a target plate had been attached, was placed on top of the foam specimen and the optical laser extensometer aligned with the target plate. The guide cylinder was then lowered using the winch, so that the cylinder made contact with the inner base of the transmitting cone. This was done so that, during a test, when the mass was dropped, it would stay on the guide cylinder. The inner base of the transmitting cone was also lined with a rubber insert in an attempt to damp out excess noise and vibration on rebound.

Once the equipment had been set up, and the digital oscilloscope set to sample the incoming information, the mass was dropped from a set height. Once the falling weight made contact with the transmitting cone, the movement was sensed on the target plate by the laser extensometer. This then triggered the oscilloscope, which sampled both channels simultaneously.

The information stored on the oscilloscope was then downloaded to an IBM compatible computer, by linking an IEEE 488 communication bus interface between the oscilloscope and the computer, and using a program called DL1200. The oscilloscope was used in preference to the software package Dash300 since it had the ability to sample at high frequencies.

Initially, three cylindrical specimens of each density, 350, 530, 700 and 1050 kg m\(^{-3}\), were tested by dropping a mass of 10.125 kg from 1.0 m. The tests where then repeated using a mass of 20.25 kg dropped from 0.5 m. This was done in order to achieve the same impact energy, but a different strain rate.

All the tests were undertaken at room temperature and humidity, and the results are given in the following chapter. In all, 24 cylindrical foam samples were tested under impact loading conditions, and a summary of the tests, test conditions and sampling information is detailed in Appendix III.1.4.

6.3 FURTHER TESTING

6.3.1 Tension

Some experimental work has been conducted on strips of foam taken from the slab material, providing three densities of material, 400, 520 and 650 kg m\(^{-3}\), to study the static and dynamic
behaviour of microvon in uniaxial tension. Since this mode of deformation is rarely used in practice, the amount of testing conducted was limited, and only aims to give an insight into the mechanical behaviour of the material in tension. Some work was necessary, however, since a component under compression will have tensile forces induced perpendicular to the loading direction on bulging surfaces.

The set-up consisted of two tensile jaw clamps which were mounted into a MAND servo-hydraulic testing machine, along with a 100 N LVDT load cell. A schematic of the specimen mounting, described below, is shown in figure 6.17, while a schematic and photograph of the apparatus are shown in figures 6.18 and 6.19 respectively.

The test specimens were bonded into specially designed grips, using a primer and adhesive combination (Loctite Prism 757 and 406 respectively), which were then clamped together using hexagonal bolts, as shown in figure 6.17. The specimens were aligned so that their skinned surfaces lay along the long specimen surface, accurate alignment being achieved with the aid of a small mounting rig. Design drawings of both the grips and alignment device are shown in Appendix II.3. Once the specimen had been clamped in the aluminium grips, and the adhesive allowed to cure at room temperature, it was mounted in two tensile jaw clamps.

Fixing rods were inserted through the ends of the tensile grips, which located in grooves on each jaw clamp, to prevent the tensile grips from slipping during testing. In order to obtain a measure of lateral displacement two LVDT displacement transducers, with flat platens, were centrally aligned on either side of the specimen. The signals from the two probes, which were mounted on a frame, were passed through a summing amplifier so that a single reading of total lateral displacement was obtained. A clip gauge extensometer was also mounted on to the specimen in order to measure axial displacement during testing.

The signals from the calibrated clip gauge, load cell and lateral displacement transducers were fed into the rear expansion port of an IBM compatible computer, along with position feedback data taken from the servo-hydraulic actuator. The information was sampled through a PC30AT multi-function A/D I/O card using the data acquisition package Dash300.

The MAND control unit was adjusted so that the specimens could be extended, under displacement control, by 9 mm, i.e. 15% extension.

Initially, a number of specimens of each density were tested to study the recovery behaviour of the material over a period of time. In order to achieve this, a method of releasing the specimen at full extension was necessary. A simple solution, using a two-pin system to hold the bottom jaw clamp to the test machine, was used. Each specimen was ramped, at 5.0 mm s⁻¹, to 15% extension, at which point, the pins were removed simultaneously, allowing the specimen to relax back to its original state. The change in displacement was recorded using the clip gauge extensometer.

After the initial static tests, specimens of each density were tested under dynamic loading to study the cyclic behaviour of the material in tension. The specimens were cycled for ten continuous cycles at a cross-head rate of 0.5 mm s⁻¹. The same test procedure was then
repeated, cycling the specimens at 5.0 mm s⁻¹, one order of magnitude larger than the previous cycle rate.

All the tests were conducted at room temperature and humidity, and the results are shown in the following chapter. In all, 27 foam strip samples were tested in uniaxial tension, and a summary of the tests, test conditions and sampling information is given in Appendix III.2.1.

6.3.2 Shear

Since very little work has been done to investigate the effect of uniaxial shear on flexible cellular materials, a programme of shear tests was undertaken in order to aid the understanding of the material behaviour. A specially designed shear rig was used throughout the testing, design drawings for which are shown in Appendix II.4. The shear rig, see figure 6.20, was placed with a 2 kN trapezoidal load cell, into a Losenhausen servo-hydraulic testing machine.

A schematic and photograph of the apparatus are shown in figures 6.21 and 6.22 respectively. The Losenhausen was used in preference to the MAND because of the low noise levels of the actuator, necessary for the low level of forces obtained during testing.

The shear rig consisted of two parts, a top holder rigidly attached to the top head of the testing machine, and a bottom holder which could be positioned on the bottom head. Both holders had a 5 mm deep circular recess in them to support the specimen. Once the recesses in the holders were brought opposite one another, the specimen could be inserted into the top holder, so that when the bottom holder was positioned and clamped correctly, it supported the other end of the specimen. Initially the samples were glued in place, but the bond broke during testing giving extraneous results, therefore no glue was used during the actual tests, and the specimens were held in place by friction fitting.

Information from the load cell, and data taken from the position of the actuator, were fed into the rear expansion port of an IBM compatible personal computer. The information was sampled through a PC30AT multi-function A/D I/O card using the package Dash300.

Specimens were cycled for ten continuous cycles, at a cross-head rate of 0.5 mm s⁻¹, and were displaced axially by 6 mm, i.e. 30% shear. This amount of displacement was found to be adequate in order to obtain data, and not allow the specimen to free itself from the shear rig. The set of experiments was then repeated, but with a cross-head rate of 5.0 mm s⁻¹.

All tests were conducted at room temperature and humidity, and the results are given in the following chapter. In total, 24 cylindrical foam samples were tested in shear, and a summary of the tests, test conditions and sampling information is given in Appendix III.2.2.

6.3.3 Water absorption

A number of cylindrical foam specimens have been tested to determine the amount of water uptake in the material. This can be important in microwave components since the water absorbed can cause corrosion to the metal parts that the spring aid is commonly in contact
with. A schematic and photograph of the apparatus used are shown in figures 6.23 and 6.24 respectively.

Prior to testing, the specimens were weighed and their mass recorded. The test then involved placing the specimens in a bowl of water, whereupon a metal gauze and weight were used to hold them under the water. A lid was then sealed over the bowl and the air removed using a vacuum pump. The removal of the air from the enclosed environment was used to draw air out of the specimens, so that, when the vacuum was released, water would displace the air. The period of time for which the specimens were under vacuum could be adjusted to different durations, and was initially set for one and a half minutes. After this period, the vacuum was released and the specimens were left for a further three minutes before removing them from the water bowl. Once taken out of the water, the specimens were dabbed dry, re-weighed, and the amount of water uptake calculated.

Three cylindrical specimens from each density, 350, 530, 700 and 1050 kg m\(^{-3}\), were tested under vacuum for durations of 1½, 3 and 4½ minutes. These times were chosen since the industry standard is three minutes duration under vacuum, and it was intended to determine the effect of varying this time on the amount of water uptake.

The tests were carried out at room temperature, and the results are given in the following chapter. In all, 72 cylindrical foam samples were tested using the water absorption apparatus, and a summary of the tests and test conditions is given in Appendix III.2.3.
Figure 6.1: Photograph of the three types of foam specimen used throughout testing.

Figure 6.2: Photograph of the general layout of equipment used for compression testing.
Figure 6.3: Close-up schematic of the cubes used during preliminary compression tests.

Figure 6.4: Schematic set-up of the apparatus used for compression testing of foam cubes.
Figure 6.5: Photograph of the apparatus used for compression testing of foam cubes.
Figure 6.6: Close-up schematic of the cylindrical foam specimens tested in compression.

Figure 6.7: Schematic of the apparatus used for compression testing foam cylinders.
Figure 6.8: Photograph of the cylindrical foam specimens tested in compression.

Figure 6.9: Photograph of the apparatus used for compression testing foam cylinders at 23 °C.
Figure 6.10: Photograph of the test equipment used for compressing cylinders at 80 °C.

Figure 6.11: Photograph of the test equipment used for compressing cylinders at -30 °C.
Figure 6.12: Schematic of the compression set rig, (the percentages indicate the amount specimens in each section are compressed to).

Figure 6.13: Photograph of the compression set rig.
Figure 6.14: Close-up schematic set-up of the cylinders used for impact testing foam cylinders.

Figure 6.15: Schematic set-up of the apparatus used for impact testing foam cylinders.
Figure 6.16: Photographs of the apparatus used for impact testing foam cylinders.
Figure 6.17: Schematic diagrams of the tensile grips used to hold the tensile test specimens.

Figure 6.18: Schematic set-up of the apparatus used for tensile testing of foam strips.
Figure 6.19: Photograph of the apparatus used for tensile testing of foam strips.
Figure 6.20: Close-up schematic of the cylindrical foam specimens tested in shear.

Figure 6.21: Schematic of the apparatus used for shear testing foam cylinders.
Figure 6.22: Photograph of the apparatus used for shear testing foam cylinders.
Figure 6.23: Schematic of the apparatus used to perform water absorption tests.

Figure 6.24: Photograph of the apparatus used in the water absorption tests.
7. TEST RESULTS

7.1 DATA ANALYSIS

The raw data from the uniaxial compression, tension, shear and impact tests, stored digitally on computer, were processed using custom computer software, written in Fortran 77. This was necessary to convert the voltage measurements of the various load, axial displacement and lateral displacement readings obtained during testing, into the relevant engineering stress and strain values. The software was also used to determine the size of the hysteresis loops, and thus determine a measure of the energy loss for the various cyclic tests.

The way in which the two data acquisition packages Status30 and Dash300 sampled information made it necessary to develop two types of computer program. In addition to this, since the information sampled using Status30 had a limited number of data points, more sophisticated signal processing was required.

The two types of program developed are essentially the same, in that both convert the raw test data to absolute data and also calculate the area of the hysteresis loops produced during dynamic tests. The programs mainly differ in the way in which the signals are conditioned and in the method used to calculate the area of each hysteresis loop.

The program, used for converting information sampled using Status30, was based on the least squares method for curve fitting, and fits third order polynomials to the loading and unloading stress/strain curves. This was done in order to utilise the limited, and relatively noisy data available. The intersection of the loading and unloading curves were then determined and the area under each curve calculated by integrating the curves over their length. This information was then used to determine the area of each of the hysteresis loops. The graphs in figure 7.1 illustrate how the raw stress/strain information is fitted and smoothed using generated third order polynomial fits. This type of program was only used for the preliminary compression tests on foam cube samples.

The other type of program, used for converting information sampled using Dash300, incorporates a modified form of the trapezoidal rule to calculate the area of individual hysteresis loops. The area under each of the loading and unloading stress/strain curves was broken down into a large number of rectangular strips. The sum of the areas of each rectangle gives rise to the total area under each curve, and in this way the area of each hysteresis loop was calculated. This type of program was used for the further compression tests, tension and shear tests. Modified forms of the program were also used to convert the single unloading and loading data obtained from the compressive and tensile recovery tests as well as the impact tests. The difference in the programs incorporating the trapezoidal rule is mainly contained within the algorithms used for data conversion. The graphs in figure 7.2 show typical conversions of the axial stress/strain data sampled using Dash300.

In cases where DC LVDT displacement transducers were used to measure lateral displacement, the high noise levels meant that aggressive smoothing algorithms were required.
Figure 7.3 shows conversions of typical noisy lateral strain/axial strain data sampled in compression.

Listings of the two types of program, based on polynomial fitting and the trapezoidal rule, are given in Appendix IV.1 and IV.2 respectively. In all, seven programs were written to convert the dynamic test data from the particular test set-ups; \textit{Pcomp} (preliminary compression tests), \textit{Pend} (further compression testing with end constraints), \textit{Prt} (further compression testing at room temperature), \textit{Plt} (further compression testing at low temperature), \textit{Pht} (further compression testing at high temperature), \textit{Ptens} (tension tests) and \textit{Pshear} (shear tests).

A number of other programs, which did not require dynamic analysis, were written simply to convert and smooth the raw data. These programs were; \textit{Pcompr} (preliminary compression recovery tests), \textit{Ptensr} (tension recovery tests) and \textit{Pimpact} (impact tests).

\textit{Material variability}

Since identical tests were carried out, in most cases, on at least three identical specimens, a large amount of information was obtained during testing. Many of the plots described in this chapter, therefore, are representative plots indicating the typical values or shape of curves. Some of the graphs show averages of the data points, or averaged curves, in order to present as much of the information as possible.

Initially the variation in the material was investigated, since the mass of all the slab material and cylindrical specimens was recorded. The graph in figure 7.4 shows the variation in density of the slab material used for the cube and strip specimens used for preliminary compressive and tensile tests respectively. Figure 7.5 shows the variation in density of all the cylinder samples used throughout the rest of the testing.

7.2 COMPRESSION TEST RESULTS

7.2.1 Preliminary compression

Typical results of the recovery of foam cube specimens after a single cycle in compression are shown in figure 7.6. Each specimen was ramped at 5.0 mm s\(^{-1}\) to 50\% compression and the load removed at the same rate. The plots, each for a different density, show how the material recovers over the subsequent ten minutes. It can be seen from the graphs, however, that recovery is almost instantaneous.

The plots in figure 7.7 show how the various density specimens recover during, and after, the ten cycles. The graphs show recovery after cycling the specimens by 50\% compression at a cross-head rate of 5.0 mm s\(^{-1}\). Information on the recovery after a further eighteen minutes is also given.

Typical compressive stress/strain plots for foam cube specimens cycled for ten continuous cycles in compression are presented in figure 7.8. Different density specimens were cycled to
50% compression at rates of 0.5 mm s\(^{-1}\) and 5.0 mm s\(^{-1}\).

The graphs in figure 7.9 indicate how the strain energy per unit volume, or size of the hysteresis loop, varies over the number of cycles tested for all the foam cube specimens cycled for ten continuous cycles. The information is plotted for tests conducted at 0.5 mm s\(^{-1}\) and 5.0 mm s\(^{-1}\), increasing in the vertical direction.

Figures 7.10 to 7.12 use the same sequence of graphs as in figures 7.7 to 7.9, for specimens which were cycled for ten cycles with intermittent recovery between each cycle. The graphs in figures 7.7 and 7.10 compare well to show the effect of introducing a period of recovery between cycles.

7.2.2 Further compression

The graphs in figure 7.13 show compressive stress/strain curves of tests conducted on cylindrical specimens of the same density, 350 kg m\(^{-3}\), but with different constraints at the loading surfaces. Each test was performed on the specimens using three separate end constraints, in order to ascertain the effects of changing the boundary conditions. Figure 7.14 shows the variation in strain energy per unit volume for all twelve of the specimens tested using the different end conditions. All specimens were cycled for ten continuous cycles at cross-head rates of 0.5 mm s\(^{-1}\), and were compressed by 75%.

In order to give some idea of the effect specimen density has on the shape of the stress/strain curve in compression, the graphs in figure 7.15 are presented. The graphs are plotted for end condition tests undertaken using only the frictional constraint, and it should also be noted that the compressive stress axes are different in each case. The upper, isolated curve on each graph was measured for the first loading cycle on the virgin specimen.

Figures 7.16 to 7.18 show compressive stress/strain curves for cylindrical specimens tested at a variety of temperatures; -30 °C, 23 °C and 80 °C respectively. All specimens were compressed by 75% at the two cross-head rates. It should be noted that the compressive stress axes vary in scale, according to the density and temperature of the material. The graphs given in figures 7.19 to 7.21, taken from information in the three previous figures, show typical stress/strain curves for the first and tenth cycles of cylindrical specimens tested at -30 °C, 23 °C and 80 °C respectively. This has been done in order to visualise the reduction in the size of the hysteresis loop, as well as its shift in position, during cycling.

An overview of the compressive stress/strain behaviour of the tenth cycle for cylindrical specimens tested at different rates and at different temperatures is shown in figure 7.22. In each graph, the higher the loop on the stress axis the higher the density, and it should be noted that the graphs have different scales for compressive stress.

The graphs in figure 7.23 show the change in energy loss between the first and tenth cycle for varying specimen densities. Graphs are plotted for specimens, which have been compressed by 75%, and cycled at different rates and temperatures. Note the change in ordinate scale for the low temperature tests in the figure.
Information about the strain energy per unit volume for all cylindrical specimens tested, compressed by 75% at different temperatures and cross-head rates, is shown in figure 7.24. Since each test was performed on three identical specimens, the data appear in groups of three, and have not been averaged. In each case, the higher the density the higher the strain energy value. This has been done to show the relatively low scatter that can be obtained by careful and consistent testing of the material.

The graphs in figures 7.25 to 7.27 show lateral/axial strain curves of the behaviour of cylindrical specimens in the left-hand column of graphs, and Poisson's ratio/axial strain in the right-hand column of graphs. The samples were cycled to 75% compression at different cross-head rates and at various temperatures. The curves have been smoothed since information obtained from the lateral displacement transducers contained large amounts of noise, inherent in DC devices.

7.2.3 Compression set

Results of the compression set tests are shown in figures 7.28 and 7.29. The graphs in the left-hand column of figure 7.28 show how much recovery is achieved after compressing cylindrical specimens by different amounts at set temperatures of -30 °C, 23 °C and 80 °C, and being held there for twenty-four hours. Each data point on the graphs is an average of three nominally identical tests performed on different specimens. Similar results are shown in the right-hand column of the figure, but the graphs are plotted for constant percentages of compression, rather than at constant temperature. A photograph of the samples after being compression set at the various temperatures is shown in 7.29.

7.2.4 Impact

Figure 7.30 shows the response of the cylindrical foam specimens to different impact loading rates. The graphs have been plotted for each of the different density of specimen used, with the mass dropped from the two set heights. Three plots are shown in each case, showing the relatively good reproducibility of the results.

7.3 FURTHER TEST RESULTS

7.3.1 Tension

The measurement of recovery of the material in tension gave rise to a number of difficulties. Once the specimen was extended and then released, there was a tendency for oscillation to be produced in the system. On analysis of the results, it appears that this oscillation resulted in the tensile grips becoming loose in the jaw clamps. This gave rise to large discrepancies in the test data, and for this reason, the information has not been presented here. Also, the results obtained from measurements of lateral strain in the material during the tension tests have not been presented, since it was found that significant errors had occurred during sampling the
information.

Typical tensile stress/strain curves are shown in figure 7.31. The six graphs show the behaviour of foam strip specimens of different density, cycled at 0.5 mm s\(^{-1}\) and 5.0 mm s\(^{-1}\). Note that the extent of deformation is much less than was achieved in compression.

Figure 7.32 shows information about the strain energy per unit volume, obtained by cycling the foam strip specimens for ten continuous cycles in tension. Each curve on the graph is an average of the three identical tests performed on specimens of the same density.

7.3.2 Shear

Stress/strain curves of cylindrical specimens cycled in shear are shown in figure 7.33. Specimens from each density were cycled, at room temperature, by 30% shear for ten continuous cycles at cross-head rates of 0.5 mm s\(^{-1}\) and 5.0 mm s\(^{-1}\). The graphs have different stress axes, and the uppermost curve in each case is for the first cycle of the material.

The variation in strain energy per unit volume is presented in figure 7.34. Each curve on the graph is an average of the three specimens tested under identical conditions. Two graphs are plotted, one for each of the cycle rates at which the specimens were tested.

7.3.3 Water absorption

The plots in figure 7.35 show the effect of water uptake on cylindrical foam samples held under water for various durations. Four graphs, one for each density of sample tested, show the results of six identical tests performed at each of the three time intervals.
Figure 7.1: Graphs showing the raw converted data from Status30, the method of smoothing and the smoothed data respectively.
Figure 7.2: Graphs showing the raw converted stress/strain data obtained from Dash300, and the smoothed data respectively.

Figure 7.3: Graphs showing the raw converted lateral strain/axial strain data obtained from Dash300, and the partly smoothed data respectively.
Figure 7.4: Graph showing the variation in foam density for the foam slabs used to make the cube and strip specimens for the preliminary compression and tension tests respectively. The dotted line indicates the quoted density of the material.

Figure 7.5: Graph showing the variation in foam density for the cylindrical foam samples used throughout the rest of the testing. The dotted line indicates the quoted density of the material.
Figure 7.6: Graphs showing how foam cube specimens of different density recover over a period of ten minutes after being compressed by 50% at a rate of 5.0 mm s\(^{-1}\) for a single cycle.
End strain value from graph = 0.0269
Value after a further 18.07 mins = 0.0267

End strain value from graph = 0.0278
Value after a further 18.07 mins = 0.0273

End strain value from graph = 0.0491
Value after a further 18.07 mins = 0.0483

Figure 7.7: Graphs showing how foam cube specimens of different density recover during, and after, being compressed by 50% at a rate of 5.0 mm s\(^{-1}\) for ten continuous cycles.
Figure 7.8: Graphs showing typical stress/strain curves for foam cube specimens of different density cycled at different rates for ten continuous cycles in compression.
Figure 7.9: Graphs showing how the strain energy per unit volume varies with cycle number for all the foam cube specimens cycled for ten continuous cycles in compression.
Figure 7.10: Graphs showing how foam cube specimens of different density recover during, and after, being compressed by 50% at a rate of 5.0 mm s\(^{-1}\) for ten cycles with intermittent recovery.
Figure 7.11: Graphs showing the stress/strain curves for foam cube specimens of different density cycled at different rates for ten cycles in compression with intermittent recovery.
Figure 7.12: Graphs showing how the strain energy per unit volume varies with cycle number for all the foam cube specimens cycled for ten intermittent cycles in compression.
Figure 7.13: Graphs showing the shape of three stress/strain curves for 350 kg m$^{-3}$ cylindrical foam specimens cycled at 0.5 mm s$^{-1}$ in compression with different end conditions.
Figure 7.14: Graph showing how the strain energy per unit volume varies with cycle number for all end condition, cylindrical specimens tested in compression.
Figure 7.15: Graphs showing the stress/strain curves of different density cylindrical specimens cycled at 0.5 mm s⁻¹ with frictional constraints in compression.
Figure 7.16: Graphs showing the stress/strain curves for cylindrical specimens cycled for ten cycles in compression at approximately -30 °C.
Figure 7.17: Graphs showing the stress/strain curves for cylindrical specimens cycled for ten cycles in compression at approximately 23 °C.
Figure 7.18: Graphs showing the stress/strain curves for cylindrical specimens cycled for ten cycles in compression at approximately 80 °C.
Figure 7.19: Graphs showing the stress/strain curves of the first (dotted line) and tenth (solid line) cycles of cylindrical specimens tested in compression at approximately -30 °C.
Figure 7.20: Graphs showing the stress/strain curves of the first (dotted line) and tenth (solid line) cycles of cylindrical specimens tested in compression at approximately 23 °C.
Figure 7.21: Graphs showing the stress/strain curves of the first (dotted line) and tenth (solid line) cycles of cylindrical specimens tested in compression at approximately 80 °C.
Figure 7.22: Graphs showing typical compressive stress/strain curves of the tenth cycle for specimens cycled at different rates and in different environments, (higher density gives rise to higher stress values).
Figure 7.23: Graphs showing how the strain energy per unit volume varies between the first (dotted line) and tenth (solid line) cycles of cylindrical specimens cycled at different rates and temperatures in compression.
Figure 7.24: Graphs showing how the strain energy per unit volume varies with cycle number for all specimens cycled in compression, (note change of axis for the lowest temperature).
Figure 7.25: Graphs showing the variation in lateral strain and Poisson's ratio for all the specimens cycled at -30 °C in compression.
Figure 7.26: Graphs showing the variation in lateral strain and Poisson's ratio for all the specimens cycled at 23 °C in compression.
Figure 7.27: Graphs showing the variation in lateral strain and Poisson's ratio for all the specimens cycled at 80 °C in compression.
Figure 7.28: Graphs showing how the percentage of height recovery varies against density for cylindrical specimens compressed to different percentages of their original height at set temperatures (left-hand column), and different temperatures to set percentages of their original height (right-hand column).
Figure 7.29: Photograph of all the compression set specimens after testing.
Figure 7.30: Graphs showing the impact loaded stress/strain curves obtained from dropping masses from 0.5 m and 1.0 m for different density cylindrical specimens.
Figure 7.31: Graphs showing the stress/strain curves for foam strip specimens of different density cycled at different rates for ten continuous cycles in tension.
Figure 7.32: Graphs showing averaged curves of how the strain energy per unit volume varies with cycle number in tension for specimens of different density cycled at different rates.
Figure 7.33: Graphs showing the stress/strain curves for cylindrical foam specimens of different density cycled at different rates for ten continuous cycles in shear.
Figure 7.34: Graphs showing averaged curves of how the strain energy per unit volume varies with cycle number for cylindrical specimens tested in shear.
Figure 7.35: Graphs showing the amount of water uptake in grams for different density specimens held under vacuum for varying periods of time.
8. MODELLING THE MECHANICAL BEHAVIOUR

8.1 MODELLING REVIEW

The mechanical behaviour of cellular materials has generally been considered difficult to characterise in simple terms since their properties are controlled largely by a number of chemical, physical and processing effects. However, an equation that relates the stress to strain is a necessary tool for predicting the response of a material to a given loading. The need to develop such constitutive equations for cellular materials has led researchers to utilise the two fundamental scientific approaches that can be used to describe the mechanical behaviour of a material; these are known as physical and phenomenological approaches.

In a physical, or microstructural approach, the macroscopic mechanical properties are explained and predicted by applying the laws of mechanics to the geometrical morphology of the foam, and using the mechanical properties of the virgin unformed solid.

In a phenomenological, or empirical approach, the macroscopic properties are measured directly from simple tests, ignoring the detailed morphology, and the information related to response is embedded in the average properties.

8.1.1 Physical model

A number of microstructural models have been developed to describe the behaviour of cellular materials where the global deformation of the foam is predicted from the deformation of a single unit cell. Such models seek to identify and analyse the mechanisms by which the cell walls and struts deform under load.

8.1.1.1 Elastomeric foams

Comprehensive work by Gibson & Ashby suggests that, in general, the compressive stress/strain behaviour of an open-cell elastomeric material can be broken into three separate regions; an initial linear elastic region (typically up to only a few percent strain), a stress plateau, and a densification region, as shown in figure 8.1. A similar three-part curve is also observed if the material is closed-cell, figure 8.2. In this case, the separate regions have been defined as Stage I, Stage II and Stage III, since the deformation mechanisms that occur, particularly during stage II, are different. The schematics given in the two figures are broadly based on the idealised behaviour of open and closed-cell elastomeric materials, and have been used here as a basis for discussing previous work.

Stage I - Linear elasticity

Gent & Thomas developed the first theories of foam mechanics to describe the linear elastic behaviour of an open-cell foam when they determined a relationship between the elastic
modulus of an open-cell foam and the volume fraction of the solid material. The model they used was based on a unit cell in the form of a cubic lattice, see figure 8.3, in which the deformation was assumed to be uniform through the lattice members, a process termed \textit{membrane deformation}. This model was later generalised to a random arrangement of thin elastic threads that connected rigid spherical joints of dead volume\textsuperscript{138}. They assumed that these thin elastic strands were circular in cross-section and randomly entered each junction point.

The models put forward by \textit{Gent & Thomas} give a very idealised representation of an actual open-cell foam, which is far from homogeneous, since the threads and interstices are often made up of a wide range of shapes and sizes. It is difficult though, to take these variations into account in a simple theory. Even so, the models of \textit{Gent & Thomas} are still widely used for predicting the linear elastic behaviour of cellular materials.

Since the original work of \textit{Gent & Thomas}, a number of researchers have set out to extend their theories. \textit{Lederman}\textsuperscript{129} extended the analysis of \textit{Gent & Thomas} to account for variation in material distribution between the joints and connecting members, whereas \textit{Kanakkanatt}\textsuperscript{135} extended the model to account for structural anisotropy. The open-cell model of \textit{Gent & Thomas} was also accepted by \textit{Christensen}\textsuperscript{135} who developed a similar theory for a low density closed-cell foam consisting of a network of planar films of uniform thickness.

There has been a great deal of confusion about the linear elastic deformation mechanisms, and some researchers have questioned the elastic analysis of the cubic lattice put forward by \textit{Gent & Thomas, Warren & Kraynik}\textsuperscript{134} and \textit{Gustafson}\textsuperscript{97}, for instance, suggested that many of these early models incorrectly assume that the cell walls carry only axial loads, and that they ignore the contribution of bending of the cell walls. Thus, they suggest that the theories of \textit{Lederman, Kanakkanatt} and \textit{Christensen} are not rigorous mechanical analyses, despite their apparent success at correlating data and their popularity.

Many of the later studies recognised the important contributions of \textit{cell wall bending} to the mechanical properties. Initial attempts to incorporate cell wall bending were put forward by \textit{Chan & Nakamura}\textsuperscript{99} who suggested that the elastic deformation should be modelled by considering the deflection of initially bent cell walls, loaded axially. This though, leads to results that are dependant on the initial degree of bending.

Cell wall bending was identified by \textit{Menges & Knipschild}\textsuperscript{99} as giving rise to the linear response shown by cellular materials. Both \textit{Ko}\textsuperscript{95} and \textit{Warren & Kraynik}\textsuperscript{134,135} considered bending and stretching of the connecting material in a cellular structure, and \textit{Ko} identified cell wall bending, and axial and shear deformation as determining linear elastic response. \textit{Ko} chose a specific geometry for his unit cell, based on the combination of a rhombic and trapezohedral dodecahedron. He derived expressions for the elastic modulus of the foam as a function of the relative density. \textit{Patel & Finnie}\textsuperscript{99}, on the other hand, assumed a less complicated unit cell, based on a pentagonal dodecahedron, and similarly derived relationships between the foam strength and density. \textit{Gibson & Ashby}\textsuperscript{99} suggest that the confusion about the mechanisms of deformation during the linear elastic regime are due, in part, to the use of such specific cell shapes, and also to the geometric complexity of many of the models.
The contribution to the strength of the cellular material from the cell walls and cell struts has also proved to be an area over which there has been much discussion. Patel & Finnie suggested that the role played by the cell wall was not much different to that played by the struts. They pointed out that even thin faces contribute to the behaviour of the material since bending of cell edges puts some of the faces in tension, so that they can resist the bending of load-bearing struts. Brezny & Green considered the strength of open-cell foams and found that closed-cells within the structure were an important parameter in controlling the properties of the bulk cellular material, directly influencing the strength. They observed faces in cellular materials as thick as half the strut diameter. However, both Menges & Knipschild and Gibson & Ashby suggest that, because of surface tension, most of the solid material in a closed-cell foam is drawn to the edges, leaving the faces very thin. They conclude that these thin faces do not contribute significantly to the properties and, therefore the behaviour of closed-cell foams can be assumed to be similar to that of open-cell foams.

Gibson & Ashby chose a model, similar to the cubic model of Gent & Thomas, where the deformation is controlled by the bending of individual struts within the unit cell. Adjoining unit cells are connected to each other at the midpoint of the struts. They dismissed previous work which identified membrane deformation as being the dominant deformation mechanism in elastomeric cellular materials, in preference to cell wall bending. By assuming this they are consistent with Ko's work where bending is important. Christensen points out that, in some cases, the properties predicted by the two methods, membrane deformation and cell wall bending, can be drastically different. He suggests that it is quite likely that there are separate material classes of behaviour that conform to each of the two separate hypotheses of mechanical action. As an example of the current confusion, Green cites two sets of experimental data, one of which supports the membrane deformation hypothesis and the other which supports the cell wall bending hypothesis.

Gibson & Ashby have studied the mechanics of both open and closed-cell models for elastomeric cellular materials, see figure 8.4. The mechanisms of linear elasticity differ between the two types of cell model; in open-cell foams, the cell walls bend when load is applied, while in closed-cell foams the process is more complicated because of the contribution from the cell membranes and gas pressure. Gibson & Ashby suggest that cell wall bending in closed-cell foams is the preferred deformation mechanism for Stage I, and they have derived an equation for linear elasticity during this regime,

\[ E = E_0 \left[ \phi \left( \frac{P}{P_0} \right)^\gamma + \left(1 - \phi \right) \left( \frac{P}{P_0} \right)^\gamma \frac{P_0(1-2\nu)}{E_0(1-\nu/\nu)} \right] \]  

(8.1)

where \( E \), \( \rho \) and \( \nu \) are the modulus, density and Poisson's ratio of the foam, and \( E_0 \) and \( \rho_0 \) are the modulus and density of the solid polymer. \( \phi \) is the volume fraction of solid contained in the cell edges and \( P_0 \) is the internal gas pressure.
Stage II - Non-linear elasticity

A variety of models have also been developed to describe the non-linear elastic behaviour of cellular materials. Gent & Thomas\(^{11}\), for instance, realised that the non-linear elastic behaviour in cellular solids was the result of some members buckling elastically. Menges & Knipschild\(^{12}\) also implicated the buckling of the cell edges as giving rise to the non-linear behaviour of cellular materials. The work of Patel & Finnie\(^{13}\) points out that some of the characteristics of compressive deformation in a closed-cell foam cannot be described by consideration of the struts alone, and that the non-linear elastic behaviour involves both buckling of the cell walls and the edges.

Gibson & Ashby\(^{14}\) suggest that compression beyond Stage I causes further deformation in the form of elastic budding of the vertical cell walls, which, for an ideal open-cell structure, would occur at constant stress, giving a flat stress plateau during Stage II. The point at which this plateau begins occurs when a critical load is reached in a layer of cells spanning the cross-section, which initiates elastic collapse of the foam. They point out that since elastic buckling modes in foams have not been observed directly, unlike those for honeycombs, it is not known whether other elastic modes exist.

Stage II for a closed-cell material tends to be more gradual, however, Gibson & Ashby are not clear as to the causes of the rise in the stress/strain curve. It is thought, though, that it may be due to compression of the gas or membrane stresses in the foam. Compression of the gas can only occur in closed-cell foams, and the result is that of providing a restoring force to the deformation, while membrane stresses can arise because the cell membranes stretch during deformation so that their tensile stiffness contributes to the stiffness of the foam. Further work by Zhang & Ashby\(^{15}\) suggests that the rising part of the curve is primarily caused by gas compression, and that the membrane stresses are much less important. They point out that this may particularly be the case for low density foams.

Gibson & Ashby\(^{16}\) have developed a relationship, based on the way in which columns buckle, that attempts to predict the elastic collapse stress which distinguishes the end of Stage I from the start of Stage II for closed-cell materials,

\[
\sigma_n = 0.03E \left( \frac{p_g}{\rho_s} \right) \left( 1 + \frac{p}{\rho_s} \right)^{\frac{1}{2}} + (p_a - p) \quad (8.2)
\]

where \(E\), \(\rho_s\) and \(\rho\) are the modulus, solid polymer density and foam density respectively, and \(p_g\) and \(p_a\) are the internal gas pressure and atmospheric pressure respectively.

At the end of Stage II, the deformation may still be recoverable, but beyond this point, some permanent set may be observed upon release of the load.
Stage III - Densification

The end of Stage II is generally attributed to the point at which the collapsing cell walls begin to touch, whereupon further collapse of the material becomes difficult and the stress/strain curve rises steeply, leading to densification. The limiting strain might be expected to be that at which all the pore space has been squeezed out, however, observations by Gibson & Ashby\(^{20}\) indicate that the cell walls entangle at smaller strains than this. They have also attempted to estimate the strain at the onset of Stage III as,

\[
\varepsilon_d = 1 - \frac{\rho_s}{\rho_f} \tag{8.3}
\]

where \(\varepsilon_d\), \(\rho_s\), and \(\rho_f\) are the densification strain, and solid and foam densities respectively.

During Stage III, the stress/strain curve approaches a limiting value and tends towards the elastic modulus of the solid material.

Overview of elastomeric physical models

In reality, foams may be made up of a distribution of cell shapes and therefore their geometry can be oversimplified using a single repeating unit cell geometry\(^{20}\). As well as specific limitations for individual models, there are also a number of uncertainties that can arise within any of the models described. These uncertainties arise in estimating the cell wall properties, which are usually considered to be the same as those of the bulk solid, and no variation with density or cell size is considered\(^{177,178}\). Part of the difficulty arises because of the variation in solid polymer moduli with the frequency of cross-links, so that for one type of polymer a range of moduli are possible, and a further part arises from the foaming process itself, which can alter the structure and properties of the cell wall polymer.

The comprehensive work by Gibson & Ashby has been identified by some researchers as having a number of draw-backs\(^{79,107,50,135}\). The most important of these, with regards to microvon, are that the models they derive are only valid for relative densities up to 0.3, and the assumption that the cell walls are thin relative to the cell edges. For microvon, both of these assumptions limit the applicability of the models, since the relative densities are high and they have a substantial amount of the solid material contained in the cell walls, which will contribute significantly to the stiffness of the foam\(^{139}\).

This has led to the need for developing of a physical model capable of describing the stress/strain behaviour of microvon, which is discussed later in the chapter.

3.1.1.2 Non elastomeric foams

If the type of polymer in the foam is not elastomeric, but brittle or plastic, then the
stress/strain curve would also have three parts, but the mechanisms of deformation would be different.

Plastic foams, like flexible foams, are initially linear elastic to low strains. However, beyond the linear elastic regime they suffer plastic collapse which occurs when the force applied exceeds the full plastic moment, creating plastic hinges. When the material is compressed further, it collapses plastically and may be affected by stretching and bending of the cell walls, as well as the presence of the cell fluid, if the cells are closed. As plastic collapse continues, it gives rise to densification of the material, and the stress/strain curve rises steeply. Such plastic yielding of the material is irreversible and the material remains permanently deformed.

Brittle foams, too, show an initial linear elastic regime. The material collapses in a manner known as brittle crushing, in which the cell walls fail when the moment acting on them exceeds a critical point. When the stress is reached at which cell walls fracture, the stress/strain curve becomes irregular and the foam crushes. In brittle foams, upper and lower yield points may be observed when a cellular material is loaded in compression. The upper yield point corresponds to the stress required to cause the first row of cells to collapse, while subsequent rows of cells deform at lower values of stress, giving rise to a lower yield point. Further deformation of the material leads to a point at which the material becomes completely crushed. The brittle crushing that occurs is not recoverable on release of the applied load.

8.1.2 Phenomenological model

Many of the early material models, based on the phenomenological approach, were developed to predict elastic behaviour in rubber materials and concentrated on small deformations. The mathematical theory of such small elastic strains has been developed to a high degree of sophistication, and the relationships derived are, in effect, a generalisation of Hooke's law.

However, the deformations which cellular materials undergo are much too large to be included in the classical theory of small strains. Since about the 1930s a considerable amount of effort has been directed towards finding the constitutive relation for rubber and rubber-like materials with a non-linear stress/strain characteristic, and which experience large deformations under loading. Many of the models aimed to find the correct expression for the strain energy of a body subjected to homogeneous strains, and are usually defined in terms of strains and material constants, if the body is isotropic. For small deformations of an elastic body, within the scope of the classical mathematical theory, the energy stored is determined completely by the body's strain components. When the deformations are large, the energy stored in an element of the body is determined by the components of the pure, homogeneous strain, which, together with a pure rotation, constitutes the strain undergone by the element of the body. In the purely rotational part of the strain no energy is stored in the element considered.
The term **hyperelasticity** is given to materials for which there exists an elastic potential function or strain energy density function, whose derivatives with respect to strain determine the stress. Hyper-elastic constitutive relations are usually suitable for describing rubber-like material behaviour, and they possess the elastic characteristics of complete reversibility of the deformation, although they characterise a response which is independent of the deformation history.

Much work, initiated by the original efforts of Mooney and Rivlin, has been concerned with the form of the strain energy function that accounts for different types of behaviour of various rubber and rubber-like materials. A large variety of strain energy functions have been proposed in the past; neo-Hookean (statistical theory), classical Mooney-Rivlin, generalised Mooney-Rivlin, the three term theory, Rivlin-Saunders, Thomas' model, Biderman theory, Blatz-Ko, Hart-Smith, Alexander, Klosner-Segal and Swanson. The neo-Hookean, classical Mooney-Rivlin, three term, Biderman and Klosner-Segal models may all be obtained as special cases of the generalised Mooney-Rivlin equation. The Hart-Smith and Alexander models are based on exponential series rather than the polynomial form of the strain energy functions used in the other models. These latter models, tend to use non-integer powers, in an attempt to add more detail to the strain energy based models.

Using the strain energy function approach for practical applications normally relies on a lengthy process which involves experimental measurements and curve fits to estimate the various derivatives of the function. For many of the large elastic deformation theories the material is usually assumed to be isotropic, incompressible with the volume change, and with negligible hysteresis. Such assumptions have been used to simplify the mathematical form of the strain energy function.

Unlike solid rubbers, which can be considered incompressible when deformed to large strains, cellular materials change their volume dramatically when compressed. However, the form of many of the models can be altered to include the assumption of material compressibility, and few such models are known to be superior to the Ogden model. Ogden developed a model, for materials such as rubber, which incorporated compressibility and was more accurate and stable than the Mooney-Rivlin model over a large range of strain. The Ogden model is general in nature and for special cases can be reduced to the neo-Hookean or Mooney-Rivlin form of the strain energy function. The form of the model can be expressed as,

\[
U = \sum_{i=1}^{N} \frac{2\mu}{\alpha_i} \left( \lambda_i^{\alpha_i} + \frac{\alpha_i}{2} \lambda_i^{\alpha_i} - 1 \right) + \sum_{i=1}^{N} \frac{1}{D_i} \left( J_i - 1 \right)^{2N} \tag{8.4}
\]

where \(U\) is the strain energy, \(N\) is a material parameter and \(\mu\), \(\alpha_i\), and \(D_i\) are temperature dependent material parameters, \(\lambda_i\) are the principal stretches, and \(J_i\) is the mechanical elastic volume ratio.

More recently, however, a number of researchers have developed phenomenological models
that have moved away from the strain energy function for defining constitutive models for rubber-like materials. Many of these models are based on experimentally obtained functions which are used to relate the stress to strain.

8.2 FINITE ELEMENT ANALYSIS

The finite element method is an efficient technique for examining and improving different design concepts at an early stage. The essential feature of finite element analysis (FEA) is the representation of the geometry of a component by a large number of simple element shapes. A set of loading and boundary conditions can then be applied to these elements to simulate the constraints that the component has imposed upon it in practice. Finally, in order to undertake the analysis, the material of the component must be characterised. Once the material behaviour has been described, the result is the outcome of solving an array of simultaneous constitutive equations for the elements of the analysis.

Such a method of determination of the stress distribution throughout a component has been tried in the past and it has struggled to deal with highly compressible materials such as microvon. Recently however, a variation on the Ogden model known as the modified Hill strain energy function, has been incorporated into a finite element package, Abaqus (version 5.2), developed by Hibbitt, Karlsson and Sorenson Incorporated, which was released early in 1993. The Hill model has the ability to predict the behaviour of elastomeric foams to high compressive strains. In order to assess the validity and range of the model, a number of finite element runs have been undertaken.

The modified form of the Hill model requires information on axial stress, axial strain and lateral strain behaviour of the material in order to equate coefficients for use in its polynomial form of the strain energy function,

\[
U = \sum_{i=1}^{N} \frac{2 \mu_i}{\alpha_i} \left[ \lambda_i^{2\alpha_i} + \lambda_i^{2\alpha_i} + \lambda_i^{2\alpha_i} - 3 + \frac{1}{\beta_i} (J^{1/m_i} - 1) \right]
\]

(8.5)

where \(U\) is the strain energy, \(N\) is a material parameter and, \(\mu_i\), \(\alpha_i\), and \(\beta_i\) are temperature dependent material parameters, \(\lambda_i\) are the principal stretches, and \(J_m\) is the mechanical elastic volume ratio.

For each stress/strain pair, Abaqus generates an expression for the stress in terms of the stretches and the unknown material constants.

Test data, obtained from compression tests performed on cylinders of microvon, have been used in conjunction with the model. For the cylindrical specimens, the data were obtained from transducers during a compression test to 75% strain at room temperature and humidity. A sequence of photographs showing the deformation processes in a 350 and a 1050 kg m\(^3\) density specimen are shown in figure 8.5.
The geometry of the cylinders were modelled using the pre-processor Femgen (version 2.2), developed by FemSys Limited, using first order, three-noded axisymmetric elements on a quarter of the cylindrical section. Rigid surface elements were used to deform the top surface of the material, using a coefficient of friction between the two surfaces of 0.25, and to constrain the bottom surface. The top rigid surface was then stepped, using a displacement constraint, to compress the geometry by 75% engineering strain. The same input deck, except for the relevant test data, was used to model cylindrical specimens from the 350, 530, 700 and 1050 kg m$^3$ density groups.

The results of the finite element analysis conducted on specimens of density 350 and 530 kg m$^3$ are shown in figures 8.6 and 8.7. Figure 8.6 shows the initial mesh followed by a deformation sequence of the 350 and 530 kg m$^3$ cylinders, which reached 75% and 64% strain, respectively before the analysis stopped. Figure 8.7 shows the comparison of the experimental and predicted stress/strain behaviour for the two densities. The specimens from the two higher density groups, 700 and 1050 kg m$^3$, failed to simulate more than 10% axial compressive strain, and therefore have not been presented.

The results of the analyses on the foam cylinders suggest that as the density of the material increased, the amount of deformation at which the finite element solution terminated decreased. This can only relate to the sensitivity of the model to the set of test data used in the analysis, since all other parameters remained constant. Thus, an improvement in the Hill strain energy model would be needed before it could be used to cope with higher density elastomeric cellular materials. There is, though, a possibility that other models, based on the strain energy function, may work for higher density materials, but these have not been investigated here.

One other problem that arose from the finite element analysis of microvon components was that, since the deformations were so large, the mathematical stability of individual elements began to degenerate. Figure 8.8 shows the meshed geometry of two common features of many microvon components, a buckling lip and rib. These sites are designed as points at which collapse of the structure will occur preferentially. The schematics in the figure indicate how, for a typical buckling lip and rib, the mesh can become distorted giving rise to mathematical instabilities. The models of the buckling lip and rib ran to 32% and 16% strain respectively before the solution failed to converge.

An assessment has also been made of a full spring aid, used in the suspension of a BMW car. The component was tested experimentally and the deformation simulated using finite element analysis on an axisymmetric section of the component. A simple compression test was performed on the component to high strains and the deformation photographed, see figure 8.9. Axial stress, strain and lateral strain data, obtained from testing a foam cylinder of the same density, were used to model the component. A sequence of the predicted deformation is shown in figure 8.10. The component reached 21% strain before the solution no longer converged.

Two further components were also modelled, a spring aid for a Ford Mondeo and one for a Ford Fiesta. Unfortunately, the solutions failed to achieve more than a few percent compressive strain.
The results of the work suggest that the meshing of the geometry gives rise to complications when buckling of the structure occurs, and leads to mathematical instabilities in local areas. The high level of distortion that occurred during testing therefore, could not be modelled due to limitations in the finite element mesh.

8.3 DEVELOPMENT OF A NEW MATERIAL MODEL

In order to develop a model capable of describing microvon, at a variety of densities, and without the need for the repeated use of experimental data, information obtained from observation of the microstructure and from mechanical tests has been combined. An investigation into the development of such a new material model using this information is discussed in the following section.

8.3.1 Physical model

The linear elastic behaviour of microvon, like that of many other elastomeric foams, is due to the elastic bending of the cell walls and struts, see figure 8.11. To initiate such bending, a certain amount of stress is needed, but once initiated, the need for further deformation is reduced because the small bending moment across the cell walls and edges increases. The bending that occurs will produce tensile or compressive forces in some of the cell walls, depending on their orientation with respect to the applied load, while others may not see such loads until the surrounding struts have started to bend. The mechanism of elastic bending will continue until some of the cell walls and edges begin to buckle, causing the response to become non-linear and thus enter Stage II.

The transition from Stage I to Stage II appears to be a gradual process, since there is a large distribution of cell edge lengths and thicknesses within the material, see figure 8.12. This suggests that both elastic bending and buckling operate throughout the two stages. Thus, while bending of some of the cell edges is taking place, those cells with the longest, or most favourably oriented edges, may begin to buckle, see figure 8.13. A small increase in stress would then be needed to cause more cell edges, those which are slightly shorter, to buckle. Once the mechanism of elastic buckling is well established, the structure may begin to collapse in layers or bands within the material, see figure 8.14. This process has also been observed in other foams by various researchers.\textsuperscript{33,174,175}

The deformation of the foam during these two stages appears to occur with little change in lateral strain, suggesting that the axial strain is accommodated within the foam by cell wall bending and buckling. This microstructural accommodation of the material implies that the contribution from the build-up of gas pressure within the cells during these stages is not significant. Since the microvon foams studied here are of a relatively high density, it is reasonable to assume that the foam is made of a stiff material and that the contribution from the gas pressure is small. If, however, the microvon was of a relatively low density, then the contribution from the gas pressure would be greater and may play an important role in
determining the mechanical behaviour of the material.

The linear elastic region is of little interest when studying microvon, since the material is used because of its non-linear elastic and densification behaviour, typically exploited up to strains of about 80%. Since the transition from Stage I to Stage II appears to be small, it is reasonable to assume that the stress/strain behaviour can, to an approximation, be defined by a single curve up to the end of Stage II. A simple relationship, therefore, can be determined, in terms of density and strain, which predicts the stress in the material over this range,

$$\sigma_s = 1.9\rho^{2.2} \quad (8.6)$$

where $$\sigma_s$$ is the stress during Stage I & II, and $$\varepsilon$$ and $$\rho$$ are the axial strain and density of the foam respectively. The derivation for the equation is given in Appendix I.2.

Data collected and fitted by Gibson & Ashby suggest that, at the onset of Stage II, the data can be fitted by a relationship that is proportional to $$\rho^{3.2}$$, equation (8.4). The model chosen by Gibson & Ashby covers a wide range of material encompassing several orders of magnitude change in density, and with considerable scatter. Since the current work on microvon is limited to a small density range with just one material, it is possible that a more accurate value has been obtained for the material, in the form of $$\rho^{3.3}$$.

At the end of Stage II, a state is reached where-after there is little further scope for microstructural accommodation, since the majority of the cells will have collapsed. At these high strains, as well as some of the cell walls beginning to contact one another, there is likely to be some contribution from the gas pressure in the cells. The effect of the gas pressure may introduce a temperature dependence but no additional rate dependence. At this point, the deformation changes from microstructural accommodation to one of macrostructural accommodation, in which the material starts to barrel giving rise to Stage III. Once the macrostructural mechanism becomes dominant, then further deformation of the material will cause it to behave like that of the solid polymer, ultimately being constrained by the geometric limit of barrelling. It is therefore reasonable to assume that during Stage III the stress/strain curves for different densities of material will begin to run parallel to one another. This will give rise to a linear Hookean response, and the slope of the stress/strain curve will give a value for the elastic modulus of the solid material. An equation for the stress during Stage III can be presented as,

$$\sigma_s = 115(\varepsilon - \varepsilon_s) + \sigma_s \quad (8.7)$$

where, $$\varepsilon_s = 6\rho^{0.33} \quad (8.8)$$

in which, $$\sigma_s$$ and $$\varepsilon_s$$ are the stress and strain during Stage III, while $$\sigma_s$$ and $$\varepsilon_s$$ are the stress and
strain at which the two curves, from equation (8.6) and (8.7), intersect, and \( \rho \) is the foam density.

Equations (8.6) and (8.7) lead to the overall picture of the stress/strain curve of the material as a bi-linear function. The bi-linear model has been plotted against experimental data in figure 8.15, using equations (8.6), (8.7) and (8.8). It can be seen from the figure that the actual cross-over point will always occur at a higher stress level than the predicted value given by \( \sigma_c \), since in reality, there is a gradual transition from Stage II to Stage III, which is again due to the range of cell sizes and cell wall dimensions present in the material. It is during this transition that the mechanisms of microstructural and macrostructural accommodation overlap, shown schematically in figure 8.16.

An estimate of the actual strain values for densification can be derived once the relative density of the material has been determined. The graph in figure 8.17, taken from figure 5.28, has been extrapolated to estimate the density of the solid material, enabling values of relative density to be calculated. In practice, there is not a single value of density of the solid material, since variation in the amount of water used to blow the foam plays an important role in determining the final outcome of the polymer; its variation leads to slightly different solid densities. However, a reasonably representative range of values can be achieved by this extrapolation.

The values of relative density are found to vary between: 0.16 - 0.26, 0.24 - 0.39, 0.32 - 0.52, and 0.47 - 0.78 for the 350, 530, 700 and 1050 kg m\(^{-3}\) respectively. These values give an indication of the proportion of solid material within the foam. Thus, it is possible to estimate the strain at which the foam reduces to the purely solid material using the equation,

\[
\varepsilon_d = 1 - \rho,
\]

where \( \varepsilon_d \) is the densification strain, determined by this method, and \( \rho \) is the relative density of the material.

The values determined from equation (8.9) have been plotted, in figure 8.18, with the experimental data and model shown in figure 8.15. The schematic in figure 8.19 identifies the proportion of the deformation mechanisms which are involved during compression, i.e. microstructural and macrostructural accommodation, including the overlapping region determined by the range of possible densities of solid material, derived from equation (8.9).

The change from foam to purely solid, as given by equation (8.9), can be visualised by studying the deformation sequence in figure 8.5. In the case of the 1050 kg m\(^{-3}\) sample, the material can be seen to deform initially with relatively little lateral expansion. After approximately 25% strain the material begins to expand laterally, in a more marked fashion. The value of densification strain for the material can be determined to be between 22% and 53% compressive strain. Thus, once a value between the two is reached, it would be expected that the material would essentially become solid and begin barrelling. This can be observed in
the deformation sequence.

In the case of the 350 kg m² specimen, a further complication arises in the form of macrostructural budding of the material which induces creases in the structure. However, the sequence of photographs in figure 8.5 indicates that during the collapse of the material the lateral expansion remains relatively unchanged. The material only really starts to deform laterally at approximately 80% compressive strain. The estimated value of densification strain for the 350 kg m² material lies between 74% and 84% compressive strain, which suggests that equation (8.9) describes, reasonably well, the point of densification of the material in practice.

8.3.2 Phenomenological models

The physical model described in the previous section is not adequate enough to fully describe the behaviour of the material over the entire range of strain. This is due to the limited accuracy at, and around, the point of densification. The simple physical model put forward also excludes the effects of temperature and strain rate on the mechanical behaviour of the material. Thus further investigation into a more accurate model has been undertaken.

Initially, the test data were used to determine whether microvon could be described by the classical Arrhenius equation relating strain rate and temperature, for a thermally activated process. Information from stress/strain curves of the different density specimens cycled at different temperatures and at different rates were used to plot graphs of logarithmic strain rate versus stress for constant temperature, see figure 8.20. Data were then taken from these graphs, at constant stress, and the corresponding values of logarithmic strain rate were plotted against the inverse of temperature, see also figure 8.20. The slope of the graph, plotted at constant stress, was used to determine the apparent activation energy of the material at constant strain. The failure of the latter curves to be parallel and thus have single values for the activation energy, indicates that the Arrhenius equation does not work for this material, i.e. the effect of temperature and strain rate are not associated or interactive. The results also suggest that the material does not follow the classical Williams-Landel-Ferry superposition principle, which dictates that an increase in frequency is equivalent to a finite decrease in temperature.

Subsequent investigation of the strain rate behaviour of the material showed that the effect of a large variation in strain rate, by several orders of magnitude, had little effect on the stress/strain behaviour, see figure 8.21. Thus, the material can reasonably be regarded as strain rate insensitive.

8.3.2.1 Axial stress/strain model

In an attempt to develop relationships between compressive stress, compressive strain, density and temperature, detailed examination of the experimental data was necessary. Curve fitting procedures were used to determine possible relationships between the various parameters affecting the overall mechanical behaviour of the material. From analysis of the test
data the following expression was derived, see Appendix I.3,

\[ \sigma = 0.15\rho^{3.2}\exp\left(\frac{2000}{RT^{0.8}}\right) \quad (8.10) \]

where \( \sigma, \varepsilon, \) and \( T \) are the stress, strain and temperature of the material, while \( \rho \) and \( R \) are the foam density and universal gas constant respectively. \( \sigma \) (Pa) can be used for the ranges \( 350 \leq \rho \) (kg m\(^{-3}\)) \leq 1050, \( 243 \leq T \) (K) \leq 353, \( 0 \leq \varepsilon \leq 0.5 \).

The experimental data have been plotted against the results predicted using equation (8.10) in the left-hand column of graphs in figures 8.22 to 8.24, at the different temperatures.

Since equation (8.10) only describes the behaviour of the material up to strains of approximately 50%, a different form of the strain function has been derived which is able to predict the behaviour of the material up to strains of 70-80%. The expression can be presented as,

\[ \sigma = 0.08\varepsilon^{3.2}\exp\left(\frac{2000}{RT^{0.8}}\right) \quad (8.11) \]

where \( \varepsilon = 4e - 29e^2 + 145.5e^3 - 309e^4 + 235.5e^5 \) (8.12)

where \( \sigma, \varepsilon, \) and \( T \) are the stress, strain and temperature of the material, while \( \rho \) and \( R \) are the foam density and universal gas constant respectively. \( \sigma \) (Pa) can be used for the ranges \( 350 \leq \rho \) (kg m\(^{-3}\)) \leq 1050, \( 243 \leq T \) (K) \leq 353, \( 0 \leq \varepsilon \leq 0.75 \).

This form of the equation has been plotted against the experimental data in the right-hand column of graphs shown in figures 8.22 to 8.24. For room temperature the equation will simplify to,

\[ \sigma = 1.0\varepsilon^{3.2} \quad (8.13) \]

where \( \sigma \) and \( \rho \) are the stress and foam density, and \( \varepsilon \) is given by equation (8.12). \( \sigma \) (Pa) can be used for the ranges \( 350 \leq \rho \) (kg m\(^{-3}\)) \leq 1050, \( 0 \leq \varepsilon \leq 0.75 \).

This simple model allows the accurate prediction of the axial stress/strain behaviour of the material up to large strains and for different densities of material.

8.3.2.2 Lateral/axial strain model

Although an equation that enables the prediction of the axial stress in relation to the axial strain is very useful, one other function necessary to describe the full behaviour of the material
is that relating lateral to axial strains. This is vital for a material with such extended initial compressibility followed by barrelling. The ability to model the lateral expansion of the material has been investigated, and again, it has been assumed that, as with the previous equations for stress/strain, the effect of strain rate can be neglected.

The data from lateral strain measurements were acquired using DC displacement transducers during tests on foam cylinders, see chapter 6.2.2. These devices have high noise levels associated with them, and therefore, the data are of a poorer quality than those sampled for axial strain. Another contribution to the signal noise arose from the vibrations through the testing frame to which the devices were attached. Thus an equation for predicting the lateral strain cannot be as precise as those developed previously for axial strain.

Initial investigation shows that the effect of temperature can, to a first approximation, be neglected when modelling lateral strain in terms of axial strain and foam density, see figure 8.25. Such a result is expected, since the behaviour of the material is based on geometry rather than applied stress. This means that it is possible to obtain a simple equation relating lateral strain, $\varepsilon_L$, to axial strain and density only. Such a model, derived in Appendix 1.4, can be used to describe the behaviour up to approximately 50% axial strain,

$$\varepsilon_L = 2 \times 10^{-6} \rho^{1/4} \varepsilon$$  \hspace{1cm} (8.14)

where $\varepsilon_L$, $\varepsilon$ and $\rho$ are the lateral strain, axial strain and foam density respectively. $\varepsilon_L$ can be used for the ranges $350 \leq \rho \, (\text{kg m}^{-3}) \leq 1050, \, 0 \leq \varepsilon \leq 0.5$.

Simple rearrangement of equation (8.14) gives rise to an approximation for Poisson’s ratio in terms of density, in which the negative sign has been ignored,

$$\nu = \frac{\varepsilon_L}{\varepsilon} = 2 \times 10^{-6} \rho^{1/4}$$  \hspace{1cm} (8.15)

where $\nu$, $\varepsilon_L$, $\varepsilon$ and $\rho$ are the Poisson’s ratio, lateral strain, axial strain and foam density respectively. $\nu$ can be used for the range $350 \leq \rho \, (\text{kg m}^{-3}) \leq 1050, \, 0 \leq \varepsilon \leq 0.5$.

Equation (8.14) has been fitted to experimental data in the left-hand column of graphs in figures 8.26 to 8.28, while equation (8.15) has been plotted in the left-hand column of graphs in figures 8.29 to 8.31. The good fits for the three temperatures justifies the omission of a temperature term from equations (8.14) and (8.15).

As with equation (8.13) in the previous section, the axial strains commonly encountered are up to 80%, and therefore, a modified form of the lateral strain equation has been derived to account for the effects of densification,

$$\varepsilon_L = 0.7 \times 10^{-6} \rho^{1/4} (\varepsilon + 5\varepsilon^2)$$  \hspace{1cm} (8.16)
where $\varepsilon_L$, $\varepsilon$, and $\rho$ are the lateral strain, axial strain, and foam density respectively. $\varepsilon_L$ can be used for the ranges $350 \leq \rho$ (kg m$^{-3}$) $\leq 1050$, $0 \leq \varepsilon \leq 0.75$.

The values for the quadratic form of the lateral strain equation have been plotted in the right-hand column of graphs in figures 8.26 to 8.28. A value of Poisson's ratio, as a function of axial strain, cannot be derived from equation (8.16), so the data have been recalculated as,

$$v = 1.3 \times 10^{-6} \rho^{1/4} (1 - \varepsilon + 4.5 \varepsilon^2)$$

(8.17)

where $v$, $\varepsilon$, and $\rho$ are the Poisson's ratio, axial strain, and foam density respectively. $v$ can be used for the ranges $350 \leq \rho$ (kg m$^{-3}$) $\leq 1050$, $0 \leq \varepsilon \leq 0.75$.

This form of the equation has been plotted against the experimental data in the right-hand column of graphs shown in figures 8.29 to 8.31.

Thus, equations (8.11), (8.12), and (8.16) or (8.17) together can be used to describe the compressive behaviour of microvon for a wide range of densities to high strains over a range of temperatures and strain rates. The range of conditions over which the equations apply are those typically experienced by microvon automotive components.
Figure 8.1: Idealised representation of the compressive stress/strain behaviour of open-cell elastomeric cellular materials\textsuperscript{33}.

Figure 8.2: Idealised representation of the compressive stress/strain behaviour of closed-cell elastomeric cellular materials\textsuperscript{33}.
Figure 8.3: Showing the model of the unit cell put forward by Gent & Thomas\cite{129}.

Figure 8.4: Showing the models of the unit cells put forward by Gibson & Ashby\cite{33}.
Figure 8.5: Sequence of photographs showing the deformation of the 350 & 1050 kg m$^3$ foam specimens.
Figure 8.6: Schematic indicating the deformation of the 350 & 530 kg m³ cylinders used in the analysis.
Figure 8.7: Comparison of the theoretical and experimental stress/strain curves for the cylinders tested. The solid and dashed lines represent the experimental data and the predicted data (using the modified Hill equation (8.5)) respectively.
Figure 8.8: Schematics showing the deformation of a typical buckling lip and rib.
Figure 8.9: Sequence of photographs showing the deformation of a BMW component.
Figure 8.10: Schematic indicating the deformation of the BMW component used in the analysis.
Figure 8.11: SEMs showing the process of elastic bending in a 700 kg m$^3$ foam.
Figure 8.12: SEMs showing the variation of cell wall thickness in *microvon* for a 350 kg m\(^3\) and 650 kg m\(^3\) foam.
Figure 8.13: SEMs showing the process of elastic collapse in a 700 kg m$^3$ foam.
Figure 8.14: SEM showing the formation of buckling layers in the material.

Figure 8.15: Experimental and predicted values of stress/strain using the simple bi-linear model, equations (8.6) to (8.8).
Figure 8.16: Schematic illustrating the difference between the microstructural and macrostructural deformation that occurs in closed-cell materials.

Figure 8.17: Graph showing extrapolation to find the approximate density of the solid material.
Figure 8.18: Experimental and predicted values of stress/strain using the simple bi-linear model, equations (8.6) to (8.8), and the estimated densification strain, equation (8.9).

Figure 8.19: Schematic showing the proportion of the deformation mechanisms for each of the four densities of material.
Figure 8.20: Graphs of logarithmic strain rate versus compressive stress and \(1/T\), plotted at a constant strain rate of 0.5 mm s\(^{-1}\).

Figure 8.21: Graph showing the stress/strain curve of a 350 kg m\(^{-3}\) specimen cycled at various rates at room temperature.
Figure 8.22: Graph showing the linear and polynomial equations for axial stress/strain fitted to experimental data at a temperature of -30 °C.
Figure 8.23: Graph showing the linear and polynomial equations for axial stress/strain fitted to experimental data at a temperature of 23 °C.
Figure 8.24: Graph showing the linear and polynomial equations for axial stress/strain fitted to experimental data at a temperature of 80 °C.
Temperature
(a) -30°C
(b) 23°C
(c) 80°C

Figure 8.25: Graph showing the lateral/axial strain curve of a 530 kg m³ specimen cycled at various temperatures.
Figure 8.26: Graph showing the linear and quadratic equations for lateral/axial strain fitted to experimental data at a temperature of -30 °C.
Figure 8.27: Graph showing the linear and quadratic equations for lateral/axial strain fitted to experimental data at a temperature of 23 °C.
Figure 8.28: Graph showing the linear and quadratic equations for lateral/axial strain fitted to experimental data at a temperature of 80 °C.
Figure 8.29: Graph showing the linear and quadratic equations for Poisson's ratio fitted to experimental data at a temperature of -30 °C.
Figure 8.30: Graph showing the linear and quadratic equations for Poisson's ratio fitted to experimental data at a temperature of 23 °C.
Figure 8.31: Graph showing the linear and quadratic equations for Poisson's ratio fitted to experimental data at a temperature of 80 °C.
9. DISCUSSION

9.1 MICROSTRUCTURE

The microstructure of different densities of microvon were examined using scanning electron microscopy. The variation in cell structure through cross-sections of the 350, 530, 700 and 1050 kg m\(^{-3}\) foam cylinders can be seen in figures 5.9 to 5.12 respectively. Such variability in the cell structure suggests that the density of the material supplied, in both slab and cylinder form, seen in figures 7.4 and 7.5, can vary considerably from sample to sample. Analysis of the data showed that the standard deviations in foam density for the 350, 530, 700 and 1050 kg m\(^{-3}\) material were 22.4, 13.2, 9.0 and 18.0 respectively. Such variation is due to the problems associated with trying to produce a homogeneous cell structure by control of bubble nucleation and growth through the physical, chemical and manufacturing techniques involved. In practice a uniform cell structure cannot be produced consistently, and this gives rise to the variation in specimen density.

Image analysis of the material has provided a useful insight into an otherwise complicated microstructure. The cell size distributions for the various density samples, shown in figures 5.21 to 5.27, give a clear indication of the spread of cell sizes. By examining these plots it can be seen that material with a density of 350, 700 or 1050 kg m\(^{-3}\) gives rise to a wide band of cell sizes. For the materials between 400 and 650 kg m\(^{-3}\), the cells appear to be consistently small and are confined to a narrow distribution. It has been shown that a coarse cell size may result in loss of load bearing ability\(^4\), thus, since the cell structure within the foam cylinders is similar to that of microvon components, see figure 9.1, it is suggested that microvon spring aid components, manufactured from material within the latter range of densities, may have better mechanical properties.

It is reasonable to assume that as the density of the foam increases, so the percentage area of voids decreases. For cellular materials such as microvon, this is the case and can be seen in figure 5.28. However, it should be noted that there is no one single density for the solid polymer material within the foam. This is apparent from figure 5.28, which shows that the percentage of voids is the same for both the 530 and 700 kg m\(^{-3}\) material. There is only one way for the two different densities of material to have the same area of voids, and that is if there is a difference in the density of the solid polymer. This is plausible with such materials, and the cause can be traced back to the chemistry involved during manufacture; more water is used in lower density foams in order to make them expand more readily, consequently, the reaction kinetics will change. This may lead to a solid which is more dense in the lower density foams, although the change should remain a relatively minor one.

The mean cell aspect ratios, re-plotted here in figure 9.2, show that cells in the lowest density foam have a higher aspect ratio than those in the higher density foam. This can be emphasised by studying figure 9.3 which shows the roundness of the cells, obtained from image analysis of the material, and is defined as,
Roundness = \frac{p^2}{4\pi A_f} \quad (9.1)

where \( P, A \) and \( f \) are the perimeter, area and digitisation factor respectively. A minimum value of unity is obtained for a circle.

This is expected since, in the lower density foams, there is a higher number of bubble nucleation sites which leads to cells growing and interacting with one another, causing them to become distorted. In a high density foam, the growth of the cells is unhindered by neighbours because of the smaller number of nucleation sites. Observations of the cell aspect ratios by other researchers suggests that for foams, a typical value is 1.3 and that there is a tendency for this to increase with cell size and decrease with density. This can be seen to accord reasonably well with the results in figures 9.2 and 9.3.

In order to gain some measure of the coarseness of the foam, a number of cell counts per linear twenty-five millimetres have been performed, the results of which are shown in figure 5.20. The data indicate that a peak in the number of cells over this linear range occurs at around 600 kg m\(^{-3}\). Information determined in this manner is often prone to error since it relies on the interpretation of the observer. However, the variation in cell count per square millimetre, shown in figure 5.29, indicates the same general trend in results. The two graphs suggest that in the lowest, middle and highest densities of foam there are a medium, large and small number of cells respectively. These observations correspond well to the variation in mean cell area, see figure 5.30; in the lowest, middle and highest densities of foam there are medium, small and large mean cell areas respectively. Both graphs imply that there are distinct variations in the cell dimensions which vary non-linearly with density.

A number of models have been developed to estimate the cell wall thickness in different densities of microvon, see figure 5.33. Data taken from the percentage area of voids, figure 5.28, and from the mean equivalent cell diameter, figure 5.31, were used to plot the curves for the three models shown in figure 5.34. In order to assess the accuracy of the models, they have been used to estimate the cell count per linear twenty-five millimetres. This has been achieved using a combination of cell wall thicknesses and cell diameters as shown in figure 5.35. It can be seen that the models appear to work reasonably well when compared to values of the linear cell count obtained by observation, see figure 5.36. Although the values are higher, the general trend can be seen to follow observations, except at the lower densities. Confidence is gained in the results therefore, since the plot combines the operator dependant results of the linear cell count with those calculated from results obtained using image analysis techniques. The discrepancy in the results at the lower densities may be due to errors in obtaining the cell counts, or due to variability in the measurements of equivalent cell diameter.

The three different models used to determine the mean cell wall thickness give very little variation in the estimated values for the cell count per linear twenty-five millimetres. Thus, it is reasonable to assume that the plot of mean cell wall thickness, given in figure 5.34, is a good representation of the variation with density.
Work done by Guenther suggests that cell wall thickness is directly proportional to cell size. If the equivalent graph is plotted for microvon, see figure 9.4, the relationship appears to be more random. However, it is feasible that a linear relationship could be drawn.

One drawback of virtually all image analysis results is that they are generally obtained for a small area of a sample, and thus the full picture may not be observed. Extensive analysis is desirable but has not been pursued in this work since it is a time consuming process; work done by Chaffanjon suggests that information from four hundred or more cells represents an adequate analysis. Another common drawback with image analysis of micrographs is that they are limited to two-dimensions, and therefore do not truly represent the three-dimensional structure. Thus, in many cases, cells will not have been sectioned along an axis of symmetry so there will be a large range of cut segments of cells, which will give rise to a distorted representation of the microstructure. It is not possible to derive the volume of the cells from measurements made in two-dimensions, except on the assumption of a constant shape and size. From the observation of shapes and the distribution in sizes determined for the samples of microvon tested, this does not appear to be an appropriate method. The technique of counting the number of cells per linear twenty-five millimetres, discussed earlier, is aimed at avoiding the two-dimensional problem by providing an estimation of the coarseness of the material.

Work was also undertaken to observe the deformed structure of microvon. The results suggest that collapse of the cells within the material is a localised phenomenon occurring in bands, seen clearly in figure 5.38, and that the highest distortion occurs near to the point of loading, see figure 5.39. It is also interesting to note that while some cells are completely collapsed, others are either unchanged or are just beginning to deform. Although the deformation mechanism may be the same for individual cells, the variation in cell size and wall thickness suggests that the behaviour of the material is better described by looking at the whole microstructure rather than a single unit cell. This approach has been adopted using the models developed in the previous chapter, and is discussed later in section 9.3.

Comparison of the permanently deformed structures, formed during the compression set tests, indicates that compressing the 350 and 530 kg m³ material to 75% strain for twenty-four hours, at various temperatures, leaves the samples with a small permanent set and the formation of a kink or crease around the circumference, see figures 5.40a and 5.41a. Observations on the formation of such creases show that, in some cases, they may occur around only part of the circumference, and that this may not always be at the mid-point of the specimen. In the case of the 700 and 1050 kg m³ material the samples are seen to be permanently deformed to a higher degree, particularly at higher temperatures, and without the formation of such creases, see figures 5.42a and 5.43a.

The transition from kink formation, resulting in a dual barrel shape, to a single barrel when compressed, see figure 9.5, appears to be a function of the density of the material and can be seen to occur between 530 and 700 kg m³. It is suggested therefore that dual barrelling occurs in an environment where there is a large percentage area of voids so that microstructural
collapse can take place with relative ease, and without necessarily any increase in lateral strain. Single barrel rolling appears to occur where there is a relatively low percentage area of voids in the material. In this case, microstructural collapse is limited, since the cell walls tend to be much shorter and more stocky, so that further deformation must continue with some increase in lateral strain.

Closer inspection of the microstructure, which has been compression set at 80 °C, shows that in the bulk of the 350 and 530 kg m\(^{-3}\) material the cells are only partially deformed, see figures 5.40b, 5.40c, 5.41b and 5.41c. However, in the higher density materials the cells have sustained a more collapsed structure, see figures 5.42b, 5.42c, 5.43b and 5.43c. This occurs to such an extent in the 1050 kg m\(^{-3}\) material that it is virtually impossible to observe the cells.

Although in general, the permanent set which is obtained in cellular materials can occur without necessarily any visible degradation of the polymer phase, at high temperatures, the intermolecular attractive forces, such as hydrogen bonds and Van der Waals forces, tend to be weak and are more susceptible to rearrangement\(^{19}\). It is possible therefore, that the build-up of thermal energy at 80 °C is sufficient enough to allow the solid polymer to restructure permanently. This is likely to have greater effect on the higher density material because of the larger number of points for restructuring, and because of the smaller percentage of cell fluid which may provide a restoring force on release of the load.

Differential scanning calorimetry has been used to investigate the possible transition regions for microvon\(^{19}\); the results indicating that a transition can occur between 138 °C and 148 °C. However, since the equipment had a range limited to greater than 100 °C, it was not possible to determine whether a transition occurred below this. Analyses undertaken on similar MDI polyurethanes suggests that there are transitions at about 80 °C, 130-150 °C and 200 °C\(^{38,44,186,187}\). The transition at 80 °C is attributed to the disruption of the secondary hydrogen bonds in the polymer structure. This implies that the compression set tests at this temperature are strongly affected by the transition point, causing a change in the physical state of the structure. If the temperature is increased to around 140 °C, degradation of the segmented polyurethane structure begins, particularly of the inter-urethane hydrogen bonding. At this temperature, as well as hydrogen bond disruption, chain scission is also taking place in the urethane and urea linkages. If there is a high hard segment concentration this may lead to even more disruption since more hydrogen bonds are available to undergo alteration. The transition above 200 °C is due to the melting of the crystalline structure in samples with sufficiently long hard segments.

The deformation of microvon, observed using the scanning electron microscope compression rig had to be conducted using uncoated foam samples since the gold sputter coating cracked during deformation, see figure 5.47. Once this problem was overcome, the results obtained provided a valuable insight into the compressive deformation processes within the material.

The sequences of photographs in figures 5.48 and 5.49, show the deformation of a 350 and 700 kg m\(^{-3}\) specimen respectively, obtained using the scanning electron microscope.
The photographs show the deformation of single cells within the microstructure which have been compressed to strains of approximately 50% and 70% respectively, see figure 5.52. The two figures, 5.48 and 5.49, emphasise the mechanisms of deformation during compression; it can be seen, in both cases, that cell wall bending is the initial mechanism, and is followed by a combination of both cell wall bending and collapse. It is also noted from the sequence of photographs that during the deformation some cells remain relatively undeformed, indicating the need to model the microstructure of the material as a whole.

The graphs plotted using information obtained from image analysis conducted on single cells during compression show how the percentage cell area reduces as the axial deformation increases, see figure 5.50. This is accompanied by an increase in the cell aspect ratio as the cells become flattened out, figure 5.51. In the case of the 350 kg m\(^{-3}\) material, the change in cell area appears to deviate at approximately 4 mm. This could relate to the point at which the cell started to buckle or the sample began to crease, since beyond this point, the cell area appears to reduce in size at a faster rate. At about 11.5 mm axial compression, the curve begins to level off. It is suggested that at this high level of deformation, approximately 77% for the sample, the deformation on a microstructural level has begun to cease and continues on a macrostructural level. In the case of the 700 kg m\(^{-3}\) sequence, the change in cell area appears to be more constant, without any abrupt deviation in the percentage cell area reduction. This gives weight to the physical model developed in chapter 8.3 which suggests that the behaviour of the material during elastic bending and buckling varies linearly up to the point of densification. At about 7 mm, the rate of cell area reduction appears to decrease. This too correlates well with the change from microstructural deformation to one of macrostructural deformation.

Due to the limitations of the scanning electron microscope compression rig, it was not possible to take the samples to higher axial deformations than those indicated in the plots. Thus, unfortunately, observations on the extent to which the cells close at very high strains leading to densification could not be achieved.

Some researchers have studied the compressive strength of cellular materials and have shown it to decrease\(^{18,19}\) and increase with increase\(^{8}\) in cell size at constant density. Others have shown that there can be both an increase and a decrease with cell size\(^{10}\), or even that the compressive strength is independent of cell size\(^{19,31}\). A possible explanation for these contradicting observations may lie in the variation in the proportion of the polymer contained in the cell walls. These variations may include features, such as the presence of open or filled cells in the material. Since varying the cell size of microvoid involves variation in the density of the material, it has not been possible to determine the relationship between compressive strength and cell size for the material.

The relative contributions to the strength of the foam from the cell wall and edges has also been investigated by a number of researchers. Some have suggested that the mechanical behaviour of closed-cell systems can be described by open-cell foams, if the membranes are
thin\textsuperscript{19,20}. This is because, when they are made, the majority of the solid can be concentrated in the cell edges. However, Sumino\textit{tara}\textsuperscript{19} found that the mechanical properties of a polyurethane foam are affected by the cell membrane distribution, which may occupy only a few weight percent of the foam. He also found that the cell membrane distribution within a single piece of foam was so different that the foam could have various mechanical properties. \textit{Ashby}\textsuperscript{24} supports the contribution made by the cell membranes and suggests that closed-cell materials can have a modulus three times that of open-cell materials. In the case of microvon, it is suggested that since the cell membranes are relatively thick, they do contribute significantly to the strength of the material.

In general, it can be seen that the cellular structure of the material directly influences the way in which the material deforms on a macrostructural level. As a result of the investigation, it is easy to see that the variations in the behaviour of the material may be due, in part, to the variation in cellular structure and density both in terms of the foam and the solid material.

\section*{9.2 MECHANICAL BEHAVIOUR}
\subsection*{9.2.1 Compressive deformation}

Recovery of foam specimens, deformed for a single cycle to 50\% strain at a cross-head rate of 5.0 mm s\textsuperscript{-1} and at room temperature, appears to be almost instantaneous, see figure 7.6. Further tests, cycling the material for ten continuous cycles and for ten cycles with intermittent recovery, suggest that, when cycled continuously, although much of the height is regained after each cycle, the build-up of height loss gradually increases over a period of time, shown in figure 7.7. This effect is seen to be reduced if a small period of time is allowed to elapse between each cycle, figure 7.10, implying that a time duration allows the material to recover further.

Recovery is important with regards to microvon components, since the need for them to recover quickly after large deformations is essential if the spring aids are to work effectively. Thus, if the components do not recover quickly, and are to be used continuously under cyclic loading, the ability of the spring aid to perform will deteriorate. After use, however, the component should recover the majority of its original height provided the material has not undergone severe loading or encountered harsh environments which may damage the cellular structure permanently. In practice, microvon components will commonly encounter periods of time during travel when they are not loaded, so that recovery can take place. The results suggest that microvon is a quick recovery material, and as such, is good for cushioning and high resilience applications. Slow recovery materials are mainly used in low rebound, high energy absorbing applications\textsuperscript{19,21,22}.

During a complete loading and unloading cycle, polyurethane foams are capable of dissipating a large amount of energy due to their highly hysteretic nature. The size of the hysteresis loop observed in cellular materials tends to be substantially larger than that found in similar solid polymers\textsuperscript{19}. Such energy dissipation in a compressed closed-cell system is
generally due to a number of factors which include both the stress softening and viscoelastic behaviour of the solid material, as well as the variable heat transfer between the compressed gases and the solid phase.

Stress softening is caused by energy loss in straining the solid polymer and occurs through continuous rearrangement of the secondary bonds, by breakage or slippage within the polymer chain structure, from the original configuration, to an alignment which is preferred in the stressed condition. On removal of the stress, the polymer gradually returns to its initial configuration, although, some of the bonds developed in the compressed state may resist the restorative force, giving rise to energy loss within the material. Such stress softening is the dominant means of energy dissipation in elastomeric foams and tends to be a manifestation of primary and secondary creep, or the movement of molecules with respect to each other, in an effort to reduce the state of stress set-up by the imposed force.

Another form of energy dissipation occurs when the cellular structure itself is strained, so that once cells have begun to collapse, interactions occur as the cells try to deform past one another. This can cause friction within the material generating small amounts of heat, and thereby contributing to the size of the hysteresis loop. This may lead to further loss if some of the cells are open or torn, giving rise to viscous resistance of the fluid through the cells.

The unusual viscoelastic properties which many elastomeric polymers exhibit are strongly non-linear and as such are difficult to describe. In polyurethane foams, such properties are generally ascribed to the variety and incompatibility of the hard and soft segments in the structure. This viscoelastic behaviour in foams is of importance due to its relation to the recoverability in the materials shape and strength after it has been compressed. Another contributing factor to the amount of energy dissipated can be the proximity of the bulk material to its glass transition state, since in the rubbery and glassy states the properties are much less rate dependant.

If the cells are closed, and the foam is compressed, the cell volume progressively decreases, increasing the fluid pressure within the cell. This increase in pressure can increase the temperature of the gas, causing the heat of the trapped gas to transfer to the cooler solid phase in the foam. This energy transfer in closed-cell foams, gives rise to a thermal relaxation mechanism which may affect the mechanical behaviour of the material.

The stress/strain curves of cube specimens cycled for ten cycles, either continuously or with intermittent recovery, are shown in figures 7.8 and 7.11, while a summary of the stress/strain behaviour is shown in figures 9.6 and 9.7 respectively. The most noticeable feature in figures 7.8 and 7.11, since all the graphs have the same axes, is the variation in the stress/strain behaviour due to density. It can be seen throughout the tests performed that this has more effect on the behaviour of the material than any variations in loading condition or environmental changes. The stress/strain curves given in the figures show that the hysteresis loops shift during cycling in such a way as to suggest there is a decrease in the stiffness. It appears that the curves are very similar, however, the size of the hysteresis loops are difficult to assess from these graphs. Plotting the strain energy per unit volume with respect to the
number of cycles gives a clearer picture of the change in the energy lost, see figures 7.9 and 7.12. These plots indicate a sudden drop in the energy after the first cycle, and smaller amounts over subsequent cycles. This sudden loss in energy gives rise to the loss in load bearing capacity of the material seen in the stress/strain curves and can be ascribed to damage induced in the cellular material by breakdown or tearing of the cell walls and struts in the structure. Comparison of the graphs suggests that the strain energy per unit volume is fractionally higher for those specimens cycled for ten continuous cycles with intermittent recovery. This is expected since the material had more time to undergo rearrangement back to its original height after each cycle.

The results of the compressive tests conducted on cylindrical specimens using a variety of end constraints showed the effect to be negligible, since very little change in the stress/strain curve can be determined, see figure 7.13. This can be seen more clearly in figure 7.14, which shows the variation in strain energy per unit volume of all the specimens tested. The graph indicates that the loss in strain energy of the different density specimens is within a range comparable with variation in specimens tested under nominally the same test conditions, see figure 7.24. One feature that does occur, seen in figure 7.13, although not due to a particular end constraint, is the drop in stress during the first cycle at approximately 40% strain. This can be attributed to the point at which the test specimen buckles forming a crease around the circumference.

The effect of cycling different density cylindrical specimens for ten continuous cycles at a variety of different test temperatures and cross-head rates can be seen in figures 7.16 to 7.18, while a summary of the stress/strain behaviour is given in figure 9.8. The graphs show that as the temperature increases, so the stress needed to deform the material to 75% strain decreases, implying that the material becomes more compliant at higher temperatures. This is expected since the higher the temperature of the material, the greater the mobility of the polymeric molecular chains to rotate and rearrange, and the weaker are the secondary bonds. Observations in other cellular systems suggest that the temperature dependence of the solid polymer is the main source for the effect of temperature on the stress/strain behaviour of the foam.

The effect of varying the cross-head rate appears to have little effect on the behaviour of the material, except at low temperatures. It is suggested that at these low temperatures, a transition in the physical state of the material occurs, which causes the polymer chains to become sensitive to mobility. Thus, at the faster rate, the polymer chains will have less time to rearrange, which increases the amount of heat generated in the material during cycling.

Experiments conducted at these low temperatures showed that once cycling had started, the specimens quickly warmed up over the ten cycles. In the case of the 350 kg m\(^3\) material, this was found to be approximately 10 °C over the duration of the test. The increase in the size of the hysteresis loops for these low density materials is due to the high percentage of cell fluid. Unlike the case of the high density materials, where the heat generated during cycling can be dissipated quickly, this is not the case in the low density materials because the cell fluid acts as
a thermal insulator.

The variation in the size of the hysteresis loops, between the first and tenth cycles, can be seen more clearly in figures 7.19 to 7.21. The relative size of the loop appears to increase from the 350 to the 1050 kg m⁻² material, suggesting that there is an increase in energy loss per unit volume with specimen density. The graphs in figures 7.22 and 7.23 support this observation.

*Microvon* components are often pre-flexed for three cycles to overcome the sudden change in stress/strain behaviour associated with the first few cycles. This pre-flexing is usually referred to as *scragging* and leads to a pre-conditioning of the component. It can be seen from the plots in figure 7.24, which clearly show the variation in the energy loss over the ten cycles, that the dynamic properties reach equilibrium after a small number of cycles, and that the majority of the change in behaviour, therefore, occurs over a short time period.

If samples are vigorously flexed, under fatigue loading conditions, disruption of the secondary hydrogen bonds takes place, the extent of which is influenced by the severity of the loading conditions. Such hydrogen bond rearrangement has been implicated as being a major contributing factor to the fatigue performance of the material. Polyurethane foams contain a large percentage of groups which employ secondary bonds, all of which may be affected by fatigue. The disruption can be minimised by forming foam systems with minimal amounts of water, since this reduces the number of urea sites available for realignment. Other bonds within the polymer structure may also be disrupted if the loading is severe enough, for example, failure of a urethane group surrounded by long flexible chains could occur. This however, would have less of an affect on the fatigue performance than failure of a hard group, such as an aliphonate or biuret, with relatively short chains connected to it.

Since fatigue tests, because of their nature, are long term and time consuming, little work has been undertaken in this research project to study the fatigue behaviour of *microvon*. However, *Kane* has related short term compression set tests to long term fatigue tests. He observed that seventeen hours compression set is equivalent to dynamic fatigue after 75,000 cycles at 1 Hz to 75% deflection. He also found that, if a polyurethane foam is compressed to 75% deflection, the major loss occurs within the first seventeen hours of compression with only a small additional loss after this period.

Results of the compression set tests undertaken in this work are presented in figures 7.28 and 7.29. The graphs in figure 7.28 suggest that deforming any density of material to 25% compression, at any temperature, has very little effect on the residual height loss once the load is removed. As the amount by which the sample is compressed increases, it can be seen that the amount of permanent set increases, particularly with the higher densities of material. This is because the greater the amount of compression, the greater the deformed shape of the material and the longer it will take for the chains to restructure to recover its original height. It is also suggested that the higher densities of material incur a larger permanent set since there is little restoring force from the presence of the compressed cell fluid in the closed-cells, unlike that in the low density foams. Over time, however, the loss in height is usually regained, since the majority of the secondary bonds will realign to the original configuration. It is possible though,
that not all of the height loss is related to polymer creep during the test, since it is not known if there is also a cell structure component involved.

As well as simulating fatigue tests, some experiments were also undertaken to simulate high strain rate tests by studying the response of the material under impact loading, see figure 7.30. The effect of impact on different types of cellular material have been reported by a large number of researchers. The results presented in this work indicate that there is little difference between the stress/strain behaviour when testing the material at the two different rates. During these tests however, the strain rate throughout the impact was not constant, since the falling projectile was retarded during compression of the material. When comparisons are made with the dynamic compression tests performed on microvon, it can be seen that, to a reasonable approximation, the stress/strain behaviour of the material is independent of strain rate, see figure 8.21. This may be due, in part, to the highly closed cell nature of the material, not allowing any gas to escape from the cells, and thus preventing viscous flow.

One feature that occurs, particularly with the highest density material, is a rippling effect in the stress/strain curve. It is suggested that this is due to wave propagation effects resulting from inertia of the foam. Work done by Benning supports this view. He shows that low density foams, with large or strongly oriented cells, do not have the capacity to transmit such shock waves as well as higher density foams.

The discussion of compressive behaviour so far has concentrated on axial deformation of the material. However, lateral strain information was also collected during testing of cylindrical samples at different temperatures. The data, plotted in figures 7.25 to 7.27, gives an indication of the lateral/axial strain and Poisson's ratio/axial strain behaviour of the material. The series of graphs suggest that the lateral strain behaviour of the material is essentially independent of temperature. This can be seen more clearly in figures 9.9 and 9.10, which show typical curves for lateral strain and Poisson's ratio versus axial strain for each density at the relevant crosshead rates and temperatures.

From the graphs showing the variation in lateral to axial strain for the higher density materials, it appears that the behaviour is a simple one. However, for the lower density materials, the form of the curve becomes more complicated. This is inevitably due to the formation of creases in the lower density material. The shape of the Poisson's ratio plots reflects this circumferential kinking of the material, which can be seen to occur at about 40% axial strain. Since the lateral displacement data were sampled from the midpoint of the specimen, where creasing tended to manifest in the low density samples, its value can be seen to decrease around 40% strain. Observations of foams that exhibit a negative Poisson's ratio, termed auxetic foams, have been investigated by a number of researchers.

The lateral strain and Poisson's ratio data collected cannot strictly be used to describe the behaviour of the material, since the lateral strain was measured from the midpoint of the specimens. It would have been more desirable to have measured the profile of the specimen during compression to obtain a lateral strain distribution. The results, therefore, can only be used to give an indication of the lateral strain behaviour of the material structure.
A number of researchers have attempted to determine a relationship between void density and Poisson’s ratio; both Gent & Thomas and Ko had looked at Poisson’s ratio and found no systematic relation between it and the void density. Patel & Finnie reported the values of Poisson’s ratio for thirteen foams of various densities and found them to be randomly distributed, with an average value of 0.33. Observations by Gibson & Ashby suggest that Poisson’s ratio is independent of relative density at all densities. However, Rinde’s analysis suggests that Poisson’s ratio increases with temperature and appears to be density dependent. From these observations, it can be seen that there is some disagreement as to whether Poisson’s ratio is sensitive to density. The results of the work presented here suggest that Poisson’s ratio is related to density, and that it cannot be assumed constant.

9.2.2 Further testing

Although cellular materials such as microvon are predominantly used in compression, the tensile and shear behaviour of the material is also of some importance. This is because during compression, regions of microvon components may be subjected to tensile and shear deformation.

In order to evaluate the behaviour of microvon in tension, a number of tests were performed, the results of which are presented in figures 7.31 and 7.32. The graphs in figure 7.31 indicate that the behaviour of the material is initially linear elastic, like that observed in compression. This linear elasticity in tension is generally thought to be caused by cell wall bending. Due to the limitations of the test, it was not possible to extend the samples to high strains, thus the effect of the deformation beyond the linear elastic regime could not be investigated. However, observations by Gibson & Ashby suggest that, at higher strains, the response is dominated by cell wall stretching as the cell walls rotate to align themselves towards the tensile axis. There is little interference in the straining process, unlike that in the case of compression, where neighbouring cells cause significant obstruction, and the stiffness of the foam increases. The basic mechanisms for tensile deformation, therefore, are quite different from those seen in compression, since the buckling which gives non-linear elasticity in compression is not possible in tension.

At very high strains, closed-cell elastomeric foams tend to fail by cell wall rupture or tearing. Observations by Patel & Finnie suggest that once cell wall rupture occurs, the deformation process before and after differ very little, showing no abrupt change in the stress/strain curve. This implies that the stress concentration introduced by such a wall rupture does not affect the foam strength. A summary of the stress/strain behaviour of microvon in tension is given in figure 9.11.

Models for the tensile properties of foams have been developed by Gent & Thomas and Harding. They concluded that the tensile modulus is a function of the foam density, solid polymer density, and solid polymer modulus. They also point out that there is no dependence of the tensile behaviour of the material on a cell structure parameter. Lederman, however, suggested that there is such a parameter, which significantly influences the tensile modulus.
Brezny & Green\textsuperscript{39} found that tensile strength and elongation increased readily as the cell size decreased, since the gas component is made up of negligible weight and has no tensile strength. In contrast, Gent & Thomas\textsuperscript{40} and Chaffanjon\textsuperscript{41} both suggested that the strength decreases with decrease in cell size. These observations imply that the confusion over the effect of cell size on the strength of the foam, discussed earlier for compression, also occurs in determining the effect on the tensile strength of the material. In the current work, it has not been possible to determine such relationships between the tensile strength of microvon and the cell size.

Plastic and brittle cellular materials fail in tension by a different mechanism than that for elastomeric foams\textsuperscript{17,19,24,35}. Cellular solids made from materials that yield plastically show a yield stress in tension caused by plastic bending of the cell walls. Brittle materials on the other hand, fracture suddenly by fast crack propagation, since a flaw or crack contained in a brittle tensile specimen will give rise to stress concentrations which will induce fracture of cell walls locally. The flaw induced in the cell wall will then extend leading to sudden catastrophic failure.

The results of the shear tests conducted on microvon are shown in figure 7.33, while a summary of the shear stress/strain behaviour is shown in figure 9.12. The curves in the graphs suggest that the initial stress/strain behaviour is linear elastic like that in tension. Since a pure shear produces no volume change, there is no contribution from the cell fluid to the shear modulus, so that the energy lost is through straining the solid polymer. Thus shear deformations become more important as foam density increases. There is however, little work in the literature on the shear testing of flexible polyurethane foams with which to compare the test results.

Under typical conditions, microvon components are exposed to the atmosphere. One constituent of the atmosphere that has been found to affect the behaviour of cellular materials in particular is the presence of high humidity\textsuperscript{35,19,25,26}. There are two different mechanisms by which water can be absorbed into, or transmitted through, a cellular material\textsuperscript{26}. The first is by diffusion from one cell to another through cell walls, and the other is by liquid or gaseous flow through holes between cells. When all cells are closed, moisture must repeatedly dissolve in the solid, diffuse through the solid, re-vaporise, and diffuse across the cell's vapour phase to the next membrane.

The results of the water absorption tests conducted on microvon samples are shown in figure 7.35. The graphs show that the lowest density material absorbs large amounts of water compared with the other densities of material. It is also interesting to note that the amount of water uptake does not necessarily vary linearly with density or with the duration under vacuum. This, however, may be due to the presence of skinned regions around the circumference of the cylindrical specimens tested. During manufacture, this skin is subjected to silicone release agent which may affect the surface of the samples. Another contribution to the variation in results may arise from the fact that the specimens were cut at each end using a hot knife which may have sealed some of the surfaces. It is possible that, because of the reasons described above, some of the samples were more prone to water uptake than others.
Room temperature immersion studies by Harding\textsuperscript{26} indicate that no liquid enters a foam in which the closed-cells are strong enough to stand imposed hydrostatic pressure. If the molecules of the foam are very hydrophobic or tightly cross-linked, the permeability is reduced still further. However, if the temperature rises, the total quantity of water vapour capable of being carried increases, and the rate of diffusivity through the cells increases. In fact, increasing the temperature and the humidity of a test has been shown to be detrimental to the material, particularly under fatigue conditions\textsuperscript{10,26}.

9.3 MODELLING

A review of previous attempts at modelling the compressive behaviour of elastomeric cellular materials has been discussed fully in chapter 8. In the case of the physical models studied, a number of limitations were found when applying them to microvon. These were due to the use of specific cell geometries, which were analysed in full and used as a basis for describing the behaviour of the material as a whole. For microvon this assumption cannot be made since the material has a wide range of cell shapes, sizes and wall thicknesses. Also, the relatively high density of the material and the variation in solid polymer properties, meant that the behaviour could not be described by a number of models. Since a suitable model was not found, it was necessary to develop a new material model to predict the compressive stress/strain behaviour of microvon.

As discussed in section 9.1, observations of the deforming microstructure of microvon suggest that linear elastic bending is the initial mechanism of deformation, giving rise to Stage I, and is followed by elastic buckling, Stage II. The transition between the two processes was found to be negligible, and therefore both mechanisms may operate throughout Stage I and Stage II. This gradual transition suggests that the behaviour of the material, to an approximation, can be described by a single curve up to the beginning of densification, Stage III. This simple model for the microstructural deformation, given in equation (8.6), describes the compressive stress/strain behaviour of the material, up to densification, as a function of density.

The transition from non-linear elasticity to densification also appear to be a gradual one, that is, while some cells are buckling, others are beginning to come in contact with one another. As the deformation continues, a stage will be reached at which the deformation on a microstructural level will cease, and the material will begin to barrel. Once this macrostructural deformation mechanism becomes dominant, the stress/strain behaviour of the material will begin to parallel that of the solid polymer. Thus, the deformation that occurs during Stage III can be described by a function of the elastic modulus of the material, given in equation (8.7). The point at which the two equations, describing the deformation during Stage I & II and during Stage III, intersect is given by equation (8.8).

The combination of these three equations gives rise to a physical model which can be used to describe the compressive stress/strain behaviour of microvon over a large range of strain. The comparison of the model with experimental data, shown in figure 8.15, suggests that the
model adequately describes the behaviour of the material, except around the transition region from non-linear elasticity to densification.

There have been numerous attempts at developing phenomenological models based on the strain energy function as a means of describing the mechanical behaviour of cellular materials, see chapter 8. These models tend to be complicated and require a large number of parameters to be determined from experimental data. One such model, a modified form of the Hill strain energy potential, was incorporated into the finite element package Abaqus. This model, developed particularly for predicting the behaviour of elastomeric cellular materials in general, was used to simulate a number of compression tests on microvon components. The results of the finite element analysis showed that the model incorporated into the package cannot cope with the higher densities of microvon, see figure 8.6. Another drawback, which was due to the finite element package rather than the material model, was the inability to deform components by large amounts, see figures 8.8 and 8.10. This occurred because the buckling lips and ribs, incorporated into the design of the component, underwent large geometric changes, causing the elements within these regions to become highly deformed, and therefore lead to mathematical instabilities. This is a problem common to a large number of finite element packages at present, although it appears to be a feature that will be rectified in the near future with the use of adaptive meshing techniques.

In order to obtained a model which could predict the behaviour of microvon more accurately, particularly around the transition region from microstructural to macrostructural deformation, and for the high densities, the use of curve fitting techniques was applied to the experimental data to form phenomenological models. This was also undertaken with the aim of incorporating the effects of strain rate and temperature.

Initial investigation into the strain rate behaviour of the material suggests that microvon is essentially strain rate independent, see figure 8.27. Thus the assumption used in determining the physical models appears to be a valid one. Since the material can be assumed insensitive to strain rate, curve fitting techniques were then used to determine the stress/strain relationship of the material purely in terms of temperature and density. The model derived in this manner is given in equation (8.11), and incorporates a fifth order polynomial, the minimum order found to accurately fit the experimental data. The model can be seen to compare well with the experimental stress/strain data over the full range of strain, see figures 8.22 to 8.24.

The equations developed for the physical model (8.6 to 8.9) and curve-fitting model (8.13) have been plotted together, against experimental data in figure 9.13. It can be seen that for low strains both models fairly accurately predict the behaviour of the material, while, at the onset of densification, the physical model underestimates the stress, but equation (8.13) describes it more accurately. At high strains it would appear that both models follow the behaviour of the material closely, although the lack of data at strains above 80% makes this inconclusive.

In order to be able to predict the full compressive behaviour of the material, phenomenological relationships were also derived to determine variation in lateral strain, and therefore Poisson's ratio, with respect to axial strain. Initial investigation showed that the
lateral/axial strain behaviour could be assumed to be independent of temperature, see figure 8.26. This is expected since the lateral strain behaviour of the material is based on geometry rather than applied stress. Simple quadratic equations, describing the lateral strain and Poisson's ratio of the material in terms of axial strain and density, have been determined and are given in equations (8.16) and (8.17) respectively. The models have been plotted against experimental data in figures 8.27 to 8.32. It can be seen that the equations give a fairly accurate representation of the lateral strain behaviour of the material over the full range of axial strain. However, as noted earlier, the data on which the models have been based was taken from the midpoint of the samples. Thus, for the low density material, the models incorporate the effects of material creasing, while, for the higher density materials, the models represent the maximum lateral strain achieved during compression of the material.

The development of phenomenological models by curve-fitting appears to provide an accurate method of determining the behaviour of the material over the ranges and operating conditions outlined. The models have been developed on the assumption that the imposed loading is static and that the material has been scragged. The former assumption has been incorporated because the phenomenon of hysteresis, the loss of strain energy despite the full recovery of strain, constitutes a considerable problem in predicting the dynamic behaviour of cellular materials. The latter assumption has been used in order to avoid any information regarding the first cycle of the material, since it may give rise to uncharacteristic behaviour of the material. Such an accurate method is necessary if component designs are to be based on the understanding of the overall material behaviour.

Physical modelling, while less accurate, provides the opportunity for developing guidelines to deal with new materials. This method will also be more robust when extrapolated outside the ranges used within this study because of the insight into the deformation processes that occur during compression.
Figure 9.1: SEMs showing the similarity in the microstructure of the foam samples tested here (top) and the microstructure of typical spring aid components.
Figure 9.2: Graph showing the variation in cell aspect ratio with density.

Figure 9.3: Graph showing the variation in roundness with specimen density.
Figure 9.4: Graph showing the variation in cell wall thickness with mean equivalent cell diameter.

Dual barrel shape
(low density foams)

Single barrel shape
(high density foams)

Figure 9.5: Variation in shape for the low and high density foam cylinders.
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Figure 9.12: Graph showing the stress/strain curves of the tenth loading cycles of the different density cylindrical foam specimens cycled at different rates in shear.
Figure 9.13: Graph showing both the physical and curve-fitting models plotted against experimental data at room temperature for all densities and at a cross-head rate of 0.5 mm s⁻¹.
10. CONCLUSIONS

The aim of the work has been to gain a better understanding of how *microvon*, a flexible polyurethane foam developed by Dunlop Suspensions and Components Limited, behaves under realistic loading conditions and to attempt to predict its mechanical behaviour in compression. This has been achieved by two means: through observations of the microstructure and by means of mechanical tests conducted on the material.

Initial investigation into the microstructure of *microvon* using scanning electron microscopy showed the need for careful preparation of the specimens. Once this was achieved, the most noticeable feature of the microstructure was the non-uniformity of the cell sizes and cell wall thicknesses. This becomes even more pronounced where the effects of material flow and curing lead to distortion, large voids, densification and anisotropy near the skin region.

Cell counts were performed to determine the coarseness of the material so that comparisons between different densities of material could be made. These measurements of the microstructure were complemented by applying image analysis techniques to scanning electron micrographs of the material. The data obtained have provided qualitative information of the microstructure of the material in terms of percentage area of voids, cell count per square millimetre, mean cell area, mean equivalent cell diameter and cell aspect ratio.

The application of image analysis to the microstructure has emphasised the variation in cell size distribution within *microvon*. It is noted that material with a density between 400 and 650 kg m\(^{-3}\) has a more consistent, narrow band of cell sizes. It is suggested therefore that *microvon* components made using material within this density range will have better mechanical properties.

A number of models were developed to estimate the average cell wall thickness in different densities of the material. These were then applied to data obtained by image analysis to predict the number of cells per linear twenty-five millimetres. The models were found to agree closely with the experimental data. The equation for the cell wall thickness of the material, based on a spherical cell model, is given by,

\[
t = \frac{d}{2} \left( \frac{1}{\sqrt{1 - V_f}} - 1 \right)
\]

where \(t\) is the mean cell wall thickness, \(d\) is the mean equivalent cell diameter and \(V_f\) is the volume fraction of the solid material.

Observations of the deforming microstructure have indicated the way in which the cellular structure deforms under load. Initially, the material is seen to deform by elastic bending of the cell walls, giving rise to Stage I. Further compression of the material causes this behaviour to change to one of elastic collapse, Stage II. This non-linear elastic behaviour may give rise to
the formation of localised bands of collapsed cells in the foam. The transition between the two mechanisms does not occur at a single point, so that both mechanisms operate throughout Stages I and II. At the end of Stage II, deformation on a microstructural level ceases and densification begins, giving rise to Stage III. The transition from Stage II to Stage III is also a gradual process, and once densification becomes the dominant mechanism, the deformation continues on a macrostructural level.

Mechanical testing of the material has shown that microvon has the ability to recover quickly after release of the applied load, under typical loading conditions. This is important if the spring aid components, manufactured from microvon, are to work effectively. It was also found that the amount of compression set in the material increased with increase in the test temperature, amount of compression and the density of the foam.

Dynamic compression tests showed the need for scragging the material because of the loss in performance during the first few cycles. It was also noted that at low temperature, scragging for three cycles was insufficient to achieve a steady state in the behaviour because of the generation of heat within the material. Analysis of the results has also shown that axial compressive deformation of microvon is, to a first approximation, independent of strain rate. It is noted however, that the stress/strain characteristics of the 1050 kg m$^{-3}$ material deteriorate if used at high strain rates because of the way in which stress waves are propagated through the material. It was also found that the behaviour of the material, particularly for the low densities, became rate sensitive at low temperatures.

Observation of the lateral strain behaviour of the material has shown that during the compression of the 350 and 530 kg m$^{-3}$ material, kinks or creases are formed in the foam samples. However, for the 700 and 1050 kg m$^{-3}$ material no such creasing occurred. It is suggested that this behaviour is density related, and the transition from dual barrelling, caused by the creases, to single barrelling occurs at some density between 530 and 700 kg m$^{-3}$. Another feature of the lateral strain behaviour of the material was that it could be assumed independent of temperature. This is expected since the lateral/axial strain behaviour of the material is governed by geometry rather than applied stress.

Although microvon is principally used in compression, an understanding of the tensile and shear behaviour of the material is important since during deformation of typical components, part of the structure may be subjected to these modes of deformation. The tests conducted showed that the tensile and shear behaviour of the material is initially linear elastic, like that in compression.

Observations of the deforming structure, performed using scanning electron microscopy, and information obtained from the detailed compressive tests have been incorporated into two models for the compressive deformation behaviour: a physical model and a phenomenological model, both of which are able to predict the mechanical behaviour of the microvon, with varying degrees of accuracy, over a large range of strain.

The physical model describes the behaviour of the material as a bi-linear relationship: the two parts representing bending and buckling (Stage I & II) followed by densification (Stage
III) of the cellular structure. A series of equations has been produced to describe these regions,

\[ \sigma_x = 1.9p^{2.2} \]
\[ \sigma_x = 115(e - \varepsilon_x) + \sigma_x \]
\[ \varepsilon_x = 60^{\mathrm{a33}} \]

where \( \sigma_x \) is the stress during Stage I & II, \( \sigma_x \) is the stress during Stage III, \( e \) and \( p \) are the axial strain and density, while \( \varepsilon_x \) and \( \sigma_x \) are the stress and strain at which the two curves intersect.

The physical model, obtained from the equations above, although very useful for describing the behaviour of the material, cannot be used for accurate prediction of the material's response, particularly at the intersection of the two curves. Such high accuracy may be required of a model incorporated into a finite element analysis package for instance. For this reason, a number of phenomenological models have been produced based on curve-fitting of the experimental data. Two different models have been developed, one defining the axial stress with respect to the axial strain behaviour, while the other predicts the lateral strain, or Poisson's ratio, with respect to axial strain behaviour.

For the axial stress/strain behaviour the equation is,

\[ \sigma = 0.085p^{2.2}\exp\left(\frac{2000}{RT}\right) \]

where \( \sigma \) represents the compressive stress over the full range of strain, \( p \) and \( T \) are the density and temperature of the material, \( R \) is the universal gas constant, and \( \xi = 4e - 29e^2 + 145.5e^3 - 309e^4 + 235.5e^5 \). This equation can be used for the ranges 350 kg m\(^{-3}\) \( \leq p \leq 1050 \) kg m\(^{-3}\), 243 K \( \leq T \leq 353 \) K, 0 \( \leq e \leq 0.75 \).

The lateral/axial strain and Poisson's ratio/axial strain behaviour can be described by,

\[ \varepsilon_L = 0.7 \times 10^{-6}p^{1.5}(e + 5e^2) \]
\[ \nu = 1.3 \times 10^{-6}p^{1.5}(1 - e + 4.5e^2) \]

where \( \varepsilon_L \) and \( \nu \) represent the lateral strain and Poisson's ratio, \( p \) is the density of the material, and \( e \) is the axial strain. These equations can be used for the ranges 350 kg m\(^{-3}\) \( \leq p \leq 1050 \) kg m\(^{-3}\), 0 \( \leq e \leq 0.75 \).
Fatigue behaviour

One of the main areas of interest which has not been covered in the present work is that of fatigue. This is an important area since, within a batch of identical components, approximately 25% may fail standard tests under cyclic loading. Observations of components under test have shown that once the fatigue crack has initiated, it progresses rapidly destroying the component within a small number of cycles, see figure 11.1. The understanding of this behaviour is not fully understood, and although there has been some research done on the fatigue of foam \cite{fatigue Foam}, further work needs to be done. Gaining sufficient information of the failure process in the material may lead to the development of a failure criterion which could be used to predict the fatigue life of microvon components.

Some initial investigations, based on observation and finite element analysis, suggest that although the material is principally used in compression, regions of tensile/tensile stress within the component may actually give rise to initiation sites for failure. In order to study the dynamic behaviour of the material under tensile loading conditions, a fatigue rig was set up, a schematic and photograph of which are shown in figures 11.2 and 11.3 respectively. The apparatus, developed from a soil shaking rig, was designed to simulate the tensile stresses induced in components by performing a three-point bend test, under cyclic loading. The rig consisted of a motor that indirectly drove a crank system and reciprocating arm, which could cycle the test specimen up to approximately 6 Hz. The motor, controlled using an AC variable speed controller, drove the crank and reciprocating arm via a chain and sprocket arrangement. The number of cycles during the test were recorded using a mechanical cycle counter attached to one of the sprockets.

A beam specimen was placed on to the support frame, and the speed controller adjusted to cycle the specimen at 2 Hz, a typical standard test frequency for microvon components. The reciprocating arm had the ability to be excited by approximately ±10 mm at the point of contact with the specimen. Although the reciprocating arm cycled in an arc, the curvature was found to be minimal over the linear 20 mm range. To limit the effect further, a roller bearing was inserted into the loading head to allow the load to self-centralise. A linear actuator, which was used to support the test specimen and frame, could be used to adjust the height of the specimen, and thus the amount of pre-load applied to it.

A limited amount of testing was undertaken using the equipment, and few results were obtained. This was because a number of problems arose when trying to study the fatigue behaviour of the material. The main problem encountered was that the fatigue cracks did not form in the dense skin surface of the material\cite{skin Surface}. This could have been over come by removing the skin of the material prior to testing, although it may have led to unrealistic fatigue behaviour.

Recent work\cite{Recent Work} has shown that a skinned microvon component can undergo millions of
cycles without failure. If the skin is removed however, the fatigue life is significantly reduced. This suggests that failure of microvon components could be initiated by local mechanical abrasion, causing the surface skin to be removed. Initial observations of the loading constraints imposed on components indicate that this is a possible mechanism for failure.

In order to determine whether heat build-up inside the specimen during cyclic loading played a role in fatigue of the material, it was necessary to obtain some thermal measurements from within the bulk of the material. However, using thermocouples at various regions in and around the specimen had drawbacks, since such intrusion of the transducers may lead to initiation sites for failure. One possible alternative lies in the use of thermal imaging techniques, although this has not been tried in relation to such cellular materials.

The problems of fatigue, therefore, open up a number of areas which could be investigated and may lead to a further understanding of dynamic loading behaviour of microvon components.

**Finite element analysis**

Two problems have been encountered when using finite element analysis software to model flexible polyurethane foams: limitations of the mesh and poor material models.

At present, only a limited number of finite element analysis packages are able to overcome the problems associated with element instabilities due to high deformations of the mesh. This work, although still in its infancy, has been achieved by the use of techniques such as adaptive meshing. In the near future it may be possible to have meshed structures that can intelligently adapt, generating more elements where instabilities maybe forming and reducing the number of elements in regions of little activity.

The work conducted during this research has provided equations which accurately predict the compressive behaviour of microvon. Once the ability of finite element codes to cope with high deformations is achieved, there is then the possibility of implementing the models, developed in chapter 8, into such codes. This can be undertaken with relative ease, since many finite element packages have the option for a user defined material, which could be utilised to model microvon. This, together with the design optimisation facilities available with many packages, would provide a powerful design tool allowing analysis of components on computer, without the need for a multitude of prototypes to be manufactured. Trials with several finite element package suppliers are underway at Dunlop Suspensions and Components Limited.

**New material formulation**

ICI Avalon, which supplies the resin and prepolymer to manufacture microvon, is currently developing an improved formulation. The new material is intended to give the polyurethane foam components better resistance to fatigue and impact damage, lower heat build-up during cycling and less height loss after compression set. The chemistry of the new material is being
developed from basics, and may incorporate significant changes to many of the chemical constituents.

Once the material has been successfully developed, simple experimental tests should show if the existing models can be used to predict the behaviour of the new formulation of flexible polyurethane foam. If the equations are found not to work accurately, then it may lead to the need for modification of the existing models, developed in chapter 8. This will require some further experimentation to determine the coefficients of the equations. However, the approach taken, and the form of equations used in this work, will allow a much smaller amount of experimental work to be conducted to provide sufficient data.
Figure 11.1: Selection of *microvon* components that have failed under cyclic loading.

Figure 11.2: Schematic of the fatigue rig used to dynamically cycle foam beam specimens.
Figure 11.3: Photograph of the fatigue rig used to dynamically cycle foam beam specimens.
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APPENDIX I - MODELLING INFORMATION

The sections in this Appendix derive the equations for cell wall thickness, axial stress/strain and lateral/axial strain, as presented in chapters 5 and 8.

1.1 Derivation of cell wall thickness models

A number of simple models have been derived to estimate the cell wall thickness of microvone. The cell wall thickness, in each case, has been determined in terms of volume fraction of solid material, \( V_f \), and the cell diameter, \( d \).

The first model, shown in figure I.1, is based on a hexagonal cell structure, and is derived below. In the derivation, \( t \) represents the cell wall thickness and \( r \) represents the radius of the cell interior.

From the geometry,

\[
I = r \tan \theta
\]

Total area = \( \frac{1}{2} r^2 \tan \theta + r t \tan \theta \)

Area of wall = \( r t \tan \theta \)

If the cell structure is simply extrapolated out of the plane, then the area and volume fractions are the same.

\[
\therefore \text{Volume fraction of solid } V_f = \frac{\frac{r t \tan \theta}{r^2 \tan \theta + r t \tan \theta}}{\frac{r}{2} + t} = \frac{2 t}{r^2 + 2 t}
\]

\[ V_f, r + 2 V_f, t = 2 t = 0 \]

\[ t(2 - 2 V_f) = V_f, r \]

\[ t = \frac{r}{2} \left( \frac{V_f}{1 - V_f} \right) \]

\[ \therefore t = \frac{d}{4} \left( \frac{V_f}{1 - V_f} \right) \] (I.1)

Equation (I.1) represents the wall thickness in terms of the diameter of the cell and the volume fraction. A similar relationship, leading to an upper bound, can be derived if the hypotenuse of the triangle is assumed to be the radius of the body, and can be given as,
Another model has been derived, shown in figure 1.2, and is based on modelling the cells of the foam, not as hexagons, but as circles.

\[
\text{Total area} = \pi (r+t)^2
\]

\[
\text{Area of wall} = \pi (r+t)^2 - \pi r^2
\]

\[
\therefore \text{Volume fraction of solid} \ V_f = \frac{\pi (r+t)^2 - \pi r^2}{\pi (r+t)^2} = \frac{t^2 + 2rt}{(r+t)^2} = \frac{t^2 + 2rt + t^2}{r^2 + 2rt + t^2}
\]

\[
V_f + V_f + 2V_f r - t^2 - 2rt = 0
\]

\[
t^2(V_f - 1) + 2rt(V_f - 1) + V_f r^2 = 0
\]

\[
\therefore t^2 + 2rt + \frac{V_f r^2}{(V_f - 1)} = 0 \tag{1.3}
\]

Equation (1.3) forms a quadratic in terms of \( t \) and can be solved to find the positive solution of the cell wall thickness,

\[
t = \frac{-2r \pm \sqrt{4r^2 - 4V_f r^2/(V_f - 1)}}{2} = -r \pm r \sqrt{1 - \frac{V_f}{(V_f - 1)}} = -r \pm \frac{r}{\sqrt{1 - V_f}}
\]

\[
t = r \left( \frac{1}{\sqrt{1 - V_f} - 1} \right)
\]

\[
\therefore t = \frac{d}{2\sqrt{1 - V_f} - 1} \tag{1.4}
\]

I.2 Derivation of physical model

By plotting logarithmic compressive stress against logarithmic density a series of parallel lines of constant temperature are produced, indicating that a relationship exists between them, see figure I.3. The graph gives rise to the expression,

\[
\log \sigma = m \log \rho + \log A
\]
I.3 Derivation of axial stress/strain model

Using the data plotted to form the equation developed above, equation (1.5), it is possible, by plotting values of logarithmic $A$ versus the inverse of temperature, to determine the extent of the temperature component, see figure I.4. From the graph,

$$\log A = \frac{m}{T} + \log A'$$

$$A = A' \exp \left( \frac{m}{T} \right)$$

Combining the equations (1.5) and (1.6) gives the equation for compressive stress in terms of density, temperature and a constant.

$$\therefore \sigma = A' \rho^{2.2} \exp \left( \frac{109}{T} \right)$$

To verify equation (1.7), the alternative route can be taken, whereby logarithmic stress is plotted against the inverse of temperature. This gives rise to parallel lines of constant density, see figure I.5, and leads to the expression,

$$\log \sigma = \frac{n}{T} + \log B$$

$$\log \left( \frac{\sigma}{B} \right) = \frac{n}{T}$$

$$\sigma = B \exp \left( \frac{n}{T} \right)$$

$$\sigma = B \exp \left( \frac{112}{T} \right)$$

By plotting logarithmic values of $B$ against logarithmic values of density, an approximately linear relationship is produced, see figure I.6. From the graph,
Equations (1.8) and (1.9) can be combined to produce the equation,

\[ \sigma = B' \rho^{2.2} \exp\left(\frac{112}{T}\right) \]  

(Eq. 1.10)

Equations (1.7) and (1.10) can be averaged to form a single equation, (1.11), below. In order to compensate for the deviation from linearity in the relationship between compressive stress and temperature, the equation becomes,

\[ \sigma = C \rho^{2.2} \exp\left(\frac{2000}{RT^{0.5}}\right) \]  

(Eq. 1.11)

where \( R \) is the universal gas constant.

For strains up to approximately 50% the stress/strain behaviour in compression can be assumed to be approximately linear. Incorporating the strain component into the equation, the expression becomes one relating the compressive stress to density, temperature and strain,

\[ \sigma = 0.15 \epsilon \rho^{2.2} \exp\left(\frac{2000}{RT^{0.5}}\right) \]  

(Eq. 1.12)

1.4 Derivation of lateral/axial strain model

Observation shows that an approximately linear relationship can be achieved by drawing a logarithmic/logarithmic plot of lateral strain against density, see figure I.7. This gives rise to the expression,

\[ \log \varepsilon_2 = m \log \rho + \log A \]
\[ \log \varepsilon_2 = \log(\rho^n . A) \]
\[ \varepsilon_2 = A \rho^n \]
\[ \varepsilon_2 = A \rho^{1.6} \]  

(Eq. 1.13)

This equation relates the density of the material to the lateral strain. The relationship between lateral and axial strain, determined from this relationship, can be found to be approximately linear up to strains of about 50%, and is independent of temperature. Therefore
the equation becomes a simple one relating lateral strain to axial strain and density only,

\[ \varepsilon_r = 2 \times 10^{-3} \rho^{1/4} \varepsilon \]  

(I.14)
Figure I.1: Model based on hexagonal cell structure.

Figure I.2: Model based on circular cell wall structure.
Figure I.3: Log/log plot of compressive stress against density for constant strain and strain rate.

Figure I.4: Log/linear plot of A against the inverse of temperature for constant strain and strain rate.
Figure I.5: Log/linear plot of compressive stress against the inverse of temperature for constant strain and strain rate.

Figure I.6: Log/log plot of B against density for constant strain and strain rate.
Figure I.7: Log/log plot of lateral strain against density for constant strain, strain rate and temperature.
APPENDIX II - DESIGN DRAWINGS

A variety of test equipment was designed and built to aid the experiments conducted throughout the work. The figures in this Appendix show the design drawings for the main pieces of apparatus used and described in the relevant chapter. All the drawings are shown in third angle projection, and all the measurements are in millimetres, except where stated. A list of the drawings is given below,

SEM COMPRESSION RIG

II.1.1 Compression platens for SEM compression rig
II.1.2 End plates for SEM compression rig
II.1.3 Strengthening members for the SEM compression rig
II.1.4 Bottom plate of the SEM compression rig
II.1.5 Vacuum port used on the SEM
II.1.6 Threads and other components used with the SEM compression rig
II.1.7 Flexible coupling and stud for driving mechanism
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COMPRESSION SET RIG

II.2 Compression set rig

TENSILE TEST APPARATUS

II.3.1 Tensile test grips
II.3.2 Tensile test grip alignment and mounting rig

SHEAR RIG

II.4 Shear rig
Figure II.1.1: Compression platens for the SEM compression rig.

Figure II.1.2: End plates for the SEM compression rig.
Figure II.1.3: Strengthening members for the SEM compression rig.

Figure II.1.4: Bottom plate of the SEM compression rig.
Material: stainless steel
Scale 1:1

4mm wide, 2.8mm deep recess for O ring

Figure II.1.5: Vacuum port and dial for the SEM compression rig.

Material: mild steel
Scale 1:1

Bearsings (x4): EE2 - OD=19.05, ID=6.35, Width=5.56
Ball screw nuts (x4): OD=19.05, ID=9.52, Length=25.4
Sprockets (x3): 15 Teeth - OD=20.60, ID=4.0, Width=4.0

Figure II.1.6: Left and right hand threads used in conjunction with the SEM compression rig.
Figure II.1.7: Flexible coupling and stud for driving mechanism.

Figure II.1.8: Driving mechanism for the SEM compression rig.
4 threaded studs M12, tapped into base

8 bolts M6, 25 long

8 rods Ø8, tapped into base
6 from edge

Material: RR58 aluminium piston alloy

Scale 1:2

Figure II.2: Compression set rig.
Material: aluminium
Scale 2:1

Figure II.3.1: Tensile test grips.
Material: aluminium
Scale 1:2

Figure II.3.2: Tensile test grip alignment and mounting rig.
Material: mild steel
Scale 1:1

Figure II.4: Shear rig.
APPENDIX III - TEST INFORMATION

The sections in this Appendix summarise the testing conditions used to perform tests on microvon, as detailed in chapter 6.

III.1 COMPRESSION

III.1.1 Preliminary compression testing

Specimen type: Cube (10x10x10 mm)
Test temperature: 25±2 °C
Relative humidity: 46±4%
% compressed: 50%
Sampled signals: (1) Clip gauge
(2) 2 kN load cell
(3) Position feedback
Data acquisition: STATUS30 in conjunction with a PC30D card

<table>
<thead>
<tr>
<th>Test type</th>
<th>Cross-head Rate (mm s⁻¹)</th>
<th>No. of specimens</th>
<th>Sampling Information</th>
<th>Density (kg m⁻³)</th>
<th>Initial</th>
<th>Pause time (s)</th>
<th>Final</th>
</tr>
</thead>
<tbody>
<tr>
<td>Relaxation: Single cycle</td>
<td>5.0</td>
<td>3 3 3</td>
<td>2048 samples at 2Hz</td>
<td>400</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Dynamic loading: Ten cycles</td>
<td>0.5</td>
<td>3 3 3</td>
<td>2048 samples at 10Hz</td>
<td>520</td>
<td>90</td>
<td>2048 samples at 2Hz</td>
<td>2048 samples at 2Hz</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>256 samples at 10Hz</td>
<td>650</td>
<td>60</td>
<td>2048 samples at 2Hz</td>
<td>2048 samples at 2Hz</td>
</tr>
<tr>
<td>Dynamic loading with intermittent recovery: Ten cycles</td>
<td>0.5</td>
<td>3 3 3</td>
<td>1024 samples at 2Hz</td>
<td>400</td>
<td>90</td>
<td>2048 samples at 2Hz</td>
<td>2048 samples at 2Hz</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2048 samples at 5Hz</td>
<td>520</td>
<td>90</td>
<td>2048 samples at 2Hz</td>
<td>2048 samples at 2Hz</td>
</tr>
</tbody>
</table>
III.1.2 Further compression testing

Specimen type: Cylinder (\(\varnothing 30 \times 30\) mm)
Test temperature: see table
Relative humidity: see table
% compressed: 75%
Sampled signals: (1) Load cell (10, 25 or 50 kN)
(2) Dual AC axial displacement transducers
(3) Dual DC lateral displacement transducers
Data acquisition: DASH300 in conjunction with a PC30AT card

<table>
<thead>
<tr>
<th>Test type*</th>
<th>Test conditions (Temp &amp; RH)</th>
<th>Crosshead Rate (mm s⁻¹)</th>
<th>No. of specimens</th>
<th>Density (kg m⁻³)</th>
<th>Sampling Information</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constraint: Bonded</td>
<td>25±3 °C 47±2%</td>
<td>0.5</td>
<td>1 1 1 1⁺</td>
<td>350 530 700 1050</td>
<td>5333 samples at 16.6Hz</td>
</tr>
<tr>
<td>Constraint: Lubricated</td>
<td>+24±2 °C 48±3%</td>
<td>0.5</td>
<td>3 3 3 3⁺</td>
<td>5333 samples at 16.6Hz</td>
<td></td>
</tr>
<tr>
<td>Constraint: Dry friction</td>
<td>-29±4 °C 0%</td>
<td>0.5</td>
<td>3 3 3 3⁺</td>
<td>5333 samples at 16.6Hz</td>
<td></td>
</tr>
<tr>
<td>Room Temp</td>
<td>+80±3 °C 0%</td>
<td>0.5</td>
<td>3 3 3 3⁺</td>
<td>5333 samples at 16.6Hz</td>
<td></td>
</tr>
</tbody>
</table>

* All specimens cycled for ten continuous cycles
⁺ These specimens were not tested using the 10 kN cell but with the 25 kN cell
⁺⁺ These specimens were not tested using the 10 kN cell but with the 50 kN cell
### III.1.3 Compression set

**Specimen type:** Cylinder (Ø30x30 mm)  
**Test temperature:** see table  
**Relative humidity:** see table  
**% compressed:** see table

<table>
<thead>
<tr>
<th>Test conditions (Temp &amp; RH)</th>
<th>Percentage* compressed (%)</th>
<th>No. of specimens</th>
<th>Density (kg m⁻³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>COLD</td>
<td>25</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>-27±2°C</td>
<td>50</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>0%</td>
<td>75</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>ROOM</td>
<td>25</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>+23±2°C</td>
<td>50</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>40±3%</td>
<td>75</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>HOT</td>
<td>25</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>+80±2°C</td>
<td>50</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>0%</td>
<td>75</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

* Specimens compressed for twenty-four hours
III.1.4 Impact

Specimen type: Cylinder (Ø30x30 mm)
Test temperature: 25±2 °C
Relative humidity: 50±3%
Masses & heights: see table
Sampled signals: (1) Laser extensometer
(2) 35 kN force washer
Data acquisition: DL1200 in conjunction with an oscilloscope linked via an IEEE 488 communication bus

<table>
<thead>
<tr>
<th>Test type</th>
<th>Drop height (m) &amp; (Mass (kg))</th>
<th>No. of specimens</th>
<th>Sampling Information</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Density (kg m⁻³)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>350</td>
<td>530</td>
</tr>
<tr>
<td>Impact loading</td>
<td>0.5 (20.25)</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>1.0 (10.25)</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

10000 samples at 200 kHz
### III.2.1 Tension

#### Specimen type:
Strip (80x15x10 mm)

#### Test temperature:
24±2 °C

#### Relative humidity:
46±4%

#### % extended:
15%

#### Sampled signals:
1. Clip gauge
2. 100 N LVDT load cell
3. Position feedback
4. Dual LVDT lateral displacement transducers

#### Data acquisition:
DASH300 in conjunction with a PC30AT card

<table>
<thead>
<tr>
<th>Test type</th>
<th>Cross-head Rate (mm s⁻¹)</th>
<th>No. of specimens</th>
<th>Density (kg m⁻³)</th>
<th>Sampling Information</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>400</td>
<td>520</td>
</tr>
<tr>
<td>Relaxation:</td>
<td></td>
<td></td>
<td></td>
<td>Initial</td>
</tr>
<tr>
<td>Single cycle</td>
<td>5.0</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>at 13.3Hz</td>
</tr>
<tr>
<td>Dynamic loading:</td>
<td>0.5</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Ten cycles</td>
<td>5.0</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>
III.2.2 Shear

Specimen type: Cylinder (Ø30x30 mm)
Test temperature: 23±2 °C
Relative humidity: 46±4%
% sheared: 30%
Sampled signals: (1) 2 kN load cell
(2) Position feedback
Data acquisition: DASH300 in conjunction with a PC30AT card

<table>
<thead>
<tr>
<th>Test type</th>
<th>Cross-head Rate (mm s⁻¹)</th>
<th>No. of specimens Density (kg m⁻³)</th>
<th>Sampling Information</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dynamic loading: Ten cycles</td>
<td>0.5</td>
<td>3 3 3 3</td>
<td>5333 samples at 40Hz</td>
</tr>
<tr>
<td></td>
<td>5.0</td>
<td>3 3 3 3</td>
<td>5333 samples at 363.6Hz</td>
</tr>
</tbody>
</table>
### III.2.3 Water absorption

**Specimen type:** Cylinder (Ø30x30 mm)  
**Test temperature:** 25±2 °C  
**Relative humidity:** 100%

<table>
<thead>
<tr>
<th>Test type</th>
<th>Duration under vacuum (minutes)</th>
<th>No. of specimens</th>
<th>Density (kg m⁻³)</th>
<th>350</th>
<th>530</th>
<th>700</th>
<th>1050</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water absorption</td>
<td>1.5</td>
<td>6</td>
<td></td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>3.0</td>
<td>6</td>
<td></td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>4.5</td>
<td>6</td>
<td></td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
</tr>
</tbody>
</table>
APPENDIX IV - COMPUTER PROGRAMS

The two programs listed in this Appendix are based on the two types of program used to convert the test data sampled from the relevant data acquisition package. A breakdown of the steps involved in each program is given below, and is followed by a full listing of the two programs.

**Polynomial program for data sampled using Status30:**
- Data conversion and smoothing.
- Calculation of the first derivatives, in order to determine the peak and troughs in the data.
- 3rd order polynomials are fitted to the loading and unloading curves, and integration points determined.
- The area under each curve is then integrated, and the two values subtracted in order to determine the area of each hysteresis loop.
- The results are stored in two files, one containing the converted data and the other the values of energy loss.

**Trapezoidal program for data sampled using Dash300:**
- Data conversion and smoothing.
- Calculation of the first derivatives, in order to determine the peak and troughs in the data.
- A modified form of the trapezoidal rule is applied to both the loading and unloading curves.
- The area under the loading and unloading curves are subtracted in order to determine the area of each hysteresis loop.
- The results are stored in two files, one containing the converted data and the other the values of energy loss.
IV.1 Test data conversion program using polynomial curve fitting

C WRITTEN BY J.M. WILLIAMS
C DEVELOPED SEPT 92 - MAR 94.
C DEPARTMENT OF ENGINEERING, UNIVERSITY OF LEICESTER.
C
C**************************************************************************
C PROGRAM POLY
C
REAL T(10000),X(10000),Y(10000),Z(10000),PARRAY(25,15)
REAL XVAL(25,2),AREAS(25),CONS(15),SSERR(15),TMINRAW,TMAXRAW
REAL TMIN,TMAX,XMIN,XMAX,XMEAN,YMIN,YMAX,ZMIN,ZMAX,YP(10000)
INTEGER KCOUNT,M,M1,M2,N,NCONS,NPOLY

C WRITE (6,10)
10 FORMAT (/IX,'**************** POLY ')
C WRITE (6,20)
20 FORMAT (/IX,'************ TEST DATA CONVERSION PROGRAM ****' )
C WRITE (6,30)
30 FORMAT (IX,'**************************************',/) 
C CALL SUBROUTINE MAINPROG TO RUN THE DATA CONVERSION PROGRAM FOR
C CONVERTING THE COMPRESSION DATA SAMPLED USING STATUS3
C
CALL MAINPROG (T,X,Y,Z,YP,PARRAY,XVAL,AREAS,CONS,
,SSERR,TMINRAW,TMAXRAW,XMINRAW,YMINRAW,YMAXRAW,ZMINRAW,ZMAXRAW,
1ZMINRAW,ZMAXRAW,TMIN,TMAX,XMIN,XMAX,XMEAN,YMIN,YMAX,ZMIN,
1ZMAX,KCOUNT,M,M1,M2,N,NCONS,NPOLY)

C WRITE (6,50)
50 FORMAT (/IX,'OPERATION COMPLETE',/)
END

C**************************************************************************
C SUBROUTINE MAINPROG (T,X,Y,Z,YP,PARRAY,XVAL,AREAS,CONS,
,SSERR,TMINRAW,TMAXRAW,XMINRAW,YMINRAW,YMAXRAW,ZMINRAW,ZMAXRAW,
1ZMINRAW,ZMAXRAW,TMIN,TMAX,XMIN,XMAX,XMEAN,YMIN,YMAX,ZMIN,
1ZMAX,KCOUNT,M,M1,M2,N,NCONS,NPOLY)
C
CHARACTER FILENAME*20,FILENAME1*20,FILENAME2*20
C
DIMENSION MINMAX(3,50)
REAL T(10000),X(10000),Y(10000),Z(10000),YP(10000),PARRAY(25,15)
REAL XVAL(25,2),AREAS(25),CONS(15),SSERR(15),TMINRAW,TMAXRAW
REAL XMINRAW,XMAXRAW,YMINRAW,YMAXRAW,ZMINRAW,ZMAXRAW
REAL TMIN,TMAX,XMIN,XMAX,XMEAN,YMIN,YMAX,ZMIN,ZMAX
INTEGER I,J,KCOUNT,L,M,M1,M2,N,NCONS,NPOLY

C DEGREE OF POLYNOMIAL (BETWEEN 2 & 14)
C N=3
C
C CALL SUBROUTINE CONV TO CONVERT RAW DATA
CALL CONV (T,X,Y,ZM,TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,YMINRAW,
YMAXRAW,ZMINRAW,ZMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,YMAX,ZMIN,
ZMAX,XMEAN,FILENAME)

C
WRITE (6,10)
10 FORMAT (IX,'xxxxxxxxxxxxxxxxxxxxxxxxxxx',/)
WRITE (6,20) M,N
20 FORMAT (1X,I4,1X,'DATA POINTS TO BE FITTED WITH A',/,
I10,' ORDER POLYNOMIAL.',/)
C
C CALL SUBROUTINE DERIV TO CALCULATE THE DERIVATIVES OF 5-POINT GROUPS
C
CALL DERIV (M,X,XMAX,KCOUNTMINMAX)
C
C CREATE LOOP FOR WORKING BETWEEN EACH PEAK & TROUGH
C
NPOLY=0
DO 80 J=1,KCOUNT
DO 70 I=1,2
M1=MENMAX(N,I)
M2=MMMAX(N+1,I)
NPOLY=NPOLY-M
C
WRITE (6,30)
30 FORMAT (IX,'xxxxxxxxxxxxxxxxxxxxxxxxxxx',/)
WRITE (6,40) NPOLY
40 FORMAT (1X,'POLYNOMIAL NUMBER',1X,I2,/) 
WRITE (6,50) M1,M2
50 FORMAT (1X,'INTEGRATION LIMIT POSITIONS:',14,IX,14,/) 
C
C CALL SUBROUTINE POLYNOM TO FIT POLYNOMIALS TO THE DATA
C
CALL POLYNOM (M1,M2,NCONS,X,Y,CONS,SSERR,YP)
C
C PUT CONSTANTS OF ALL POLYNOMIALS INTO A 2-D ARRAY, PARRAY
C
DO 60 J=1,NCONS
PARRAY(NPOLY,J)=CONS(J)
60 CONTINUE
70 CONTINUE
80 CONTINUE
C
C CALL SUBROUTINE ENDPTS TO FIND THE MIN & MAX END PTS FOR INTEGRATION
C
CALL ENDPTS (KCOUNTMINMAX,NCONS,PARRAY,XAREAS,XVAL)
C
C WRITE MINS, MAXS & AREAS TO A FILE ???OUT
C
FILENAME1=FILENAME
L=INDEX(FILENAME1,'.dat')
FILENAME1(L:LW)='.out'
OPEN (UNIT=2,FILE=FILENAME1,STATUS='UNKNOWN')
WRITE (2,90) FILENAME1
90 FORMAT (1X,'FILE: 'A20)
C
WRITE (2,100) TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,YMINRAW,YMAXRAW,
ZMINRAW,ZMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,YMAX,ZMIN,ZMAX
100 FORMAT (U10,MAXS & MINS OF RAW DATA:/',1X,TMIN',F9.5,
1',TMAX',F9.5,',/1X,XMIN',F9.5,',/1X,XMAX',F9.5,',/1X,YMIN',
1',YMAX',F9.5,',/1X,ZMIN',F9.5,',/1X,ZMAX',F9.5,',/1X,TMIN',
1',TMAX',F9.5,',/1X,XMIN',F9.5,',/1X,XMAX',F9.5,',/1X,YMIN',
1',YMAX',F9.5,',/1X,ZMIN',F9.5,',/1X,ZMAX',F9.5,/)
C
WRITE (2,110)
110 FORMAT (1X,'AREAS OF HYSTERESIS LOOPS:')
DO 130 I=1,JCCOUNT*2,2
   WRITE (2,120) (I/2)+I/2,AREAS(I),AREAS(I+1)
120 FORMAT (1X,'AREA OF LOOP ',12,' IS : ' ,F8.5)
130 CONTINUE
WRITE (2,140)
140 FORMAT (//)
CLOSE(2)
C
C WRITE CONVERTED TEST DATA TO A FILE ???NEW
C
FILENAME2=FILENAME
L=INDEX(FILENAME2,'.dat')
FILENAME2(L:L+4)='.new'
OPEN (UNIT=3,EUNIT=FILENAME2,STATUS='UNKNOWN')
DO 160 K=1,M
   WRITE (3,150) T(K),Y(K),X(K),YP(K)
150 FORMAT (1X,F6.1,3(','E9.5))
160 CONTINUE
CLOSE(3)
C
WRITE (6,170)
170 FORMAT
WRITE (6,180)
180 FORMAT (1X,EOLY DATA CONVERSION COMPLETED',/)
END
SUBROUTINE CONV (TX,Y,ZM,TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,
1YMINRAW,YMAXRAW,ZMINRAW,ZMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,YMAX,
1ZMENXMAXXMEAN,FILENAME)
C
C SUBROUTINE TO CONVERT COMPRESSION DATA USING PC30-STAT30, CONVERTS
C TIME(V), DISPLACEMENT(V), LOAD(V) & POSITION (V) TO
C TIME(S), STRAIN, STRESS(MPA) & POSITION (MM)
C
REAL TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,
1YMINRAW,YMAXRAW,ZMINRAW,ZMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,YMAX,
1ZMIN,ZMAX,XMEAN,FILENAME
INTEGER KMJSTDATA
CHARACTER FELENAME*20
C
WRITE (6,10)
10 FORMAT (1X,'INPUT FILENAME: ',$) READ (5,20) FILENAME
20 FORMAT (A20)
C
C CALL SUBROUTINE INITIAL TO INITIALISE ALL MINS & MAXS
C
CALL INITIAL (TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,
1YMINRAW,YMAXRAW,ZMINRAW,ZMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,YMAX,
1ZMIN,ZMAX,XMEAN,FILENAME)
C
C OPEN RAW DATA FILE
C
NDATA=1
OPEN (UNIT=1,FILE=FILENAME,STATUS='OLD',ERR=40)
30 READ (1,*JEND=60) T(NDATA),X(NDATA),Y(NDATA)
C
C FIND MINS & MAXS OF RAW DATA
TMINRAW = MIN(TMINRAW, T(NDATA))
TMAXRAW = MAX(TMAXRAW, T(NDATA))
XMINRAW = MIN(XMINRAW, X(NDATA))
XMAXRAW = MAX(XMAXRAW, X(NDATA))
YMINRAW = MIN(YMINRAW, Y(NDATA))
YMAXRAW = MAX(YMAXRAW, Y(NDATA))
ZMINRAW = MIN(ZMINRAW, Z(NDATA))
ZMAXRAW = MAX(ZMAXRAW, Z(NDATA))

NDATA = NDATA + 1
GOTO 30

40 FORMAT (6,50)
50 FORMAT (/1X,'NO SUCH FD LE EXISTS.' ,/) STOP
60 CLOSE(1)

M = NDATA - 1

C CREATE ABSOLUTE VALUES OF THE DATA, THEN APPLY CALIBRATION FACTORS AND DATA CORRECTIONS TO ABSOLUTE DATA

DO 70 K = 1, M
   X(K) = X(K) - XMINRAW
   Y(K) = (ABS(Y(K))) + YMAXRAW
   Z(K) = Z(K) - ZMINRAW
70 CONTINUE

ClIP = 0.89
CELL = 1.0

DO 80 K = 1, M
   X(K) = (X(K) * CLIP) / 10.0
   Y(K) = (Y(K) * CELL) / 0.1
   Z(K) = Z(K) / 2.0
80 CONTINUE

C FIND MINS & MAXS OF CONVERTED DATA

TMIN = MIN(TMIN, T(K))
TMAX = MAX(TMAX, T(K))
XMIN = MIN(XMIN, X(K))
XMAX = MAX(XMAX, X(K))
YMIN = MIN(YMIN, Y(K))
YMAX = MAX(YMAX, Y(K))
ZMIN = MIN(ZMIN, Z(K))
ZMAX = MAX(ZMAX, Z(K))
XMEAN = XMEAN + X(K)

80 CONTINUE

XMEAN = XMEAN / FLOAT(M)

C PRINT OUT MINS & MAXS OF THE RAW DATA

WRITE (6,90)
90 FORMAT (/X, 'TM1N ', F9.5, ' TMAX ', F8.3, /
   'XMIN ', F9.5, ' XMAX ', F9.5, /
   'YMIN ', F9.5, ' YMAX ', F9.5, /
   'ZMIN ', F9.5, ' ZMAX ', F9.5)

C PRINT OUT MINS & MAXS OF THE CONVERTED DATA

WRITE (6,110)
SUBROUTINE INITIAL (TMINRAW, TMAXRAW, XMINRAW, XMAXRAW, YMINRAW, YMAXRAW, ZMINRAW, ZMAXRAW, TMIN, TMAX, XMIN, XMAX, YMIN, YMAX, ZMIN, ZMAX, XMEAN)

C SUBROUTINE TO INITIALISE ALL MAXS & MINS
C
REAL TMINRAW, TMAXRAW, XMINRAW, XMAXRAW, YMINRAW, YMAXRAW, ZMINRAW, ZMAXRAW, TMIN, TMAX, XMIN, XMAX, YMIN, YMAX, ZMIN, ZMAX, XMEAN

C
TMINRAW=10000.0
TMAXRAW=-10000.0
XMINRAW=10000.0
XMAXRAW=-10000.0
YMINRAW=10000.0
YMAXRAW=-10000.0
ZMINRAW=10000.0
ZMAXRAW=-10000.0

C
TMIN=10000.0
TMAX=-10000.0
XMIN=10000.0
XMAX=-10000.0
YMIN=10000.0
YMAX=-10000.0
ZMIN=10000.0
ZMAX=-10000.0
XMEAN=0.0
RETURN
END

C------------------------------------------------------------------------------------------------------------------

SUBROUTINE DERIV (M,X,XMAX, KCOUNT,MINMAX)
C
C THIS SUBROUTINE CALCULATES THE 1ST DERIVATIVES OF X(K)
C IN ORDER TO PRODUCE +VE & -VE SLOPES WHICH ARE USED TO
C DETERMINE THE PEAKS & TROUGHS IN X(K)
C
DIMENSION MINMAX(3,50)
REAL D(10000),X(10000),XMAX
INTEGER K,KCOUNT,M

C
C CALCULATE THE 1ST DERIVATIVES OF X(K), PUT INTO D(K)
C KCOUNT - THE NUMBER OF PEAKS IN X(K)
C
DO 10 K=3,M-2

C------------------------------------------------------------------------------------------------------------------
\begin{verbatim}
CHX=-(2*X(K-2)-X(K-1)+X(K+1)+2*X(K+2))
D(K)=CHX/10.0
KCOUNT=G
C FIND POINTS (PEAKS) WHERE D(K) CROSSES FROM +VE TO -VE.
PUT VALUES IN THE 2nd COLUMN OF MINMAX(I,J)
DO 20 K=1,M-1
IF ((D(IC).GT.(G.5G*XMAX)) .AND. (D(K-i-l).LE.(G.5G*XMAX))) THEN
KCOUNT=KCOUNT+1
MINMAX(2,KCOUNT)=K-i-1
ENDIF
20 CONTINUE
C FIND POINTS (1ST TROUGHS) WHERE D(K) IS -VE OR ZERO.
C PUT VALUES IN THE 1st COLUMN OF MINMAX(I,J)
DO 70 1=1,JCOUNT
DO 30 K=MINMAX(2,I)-1,-1
IF (D(I{).LE.(G.5G*XMAX)) THEN
MINMAX(1,I)=K-i-1
GOTO 40
ENDIF
30 CONTINUE
MINMAX(1,I)=0.0
C FIND POINTS (2ND TROUGHS) WHERE D(K) IS +VE.
C PUT VALUES IN THE 3rd COLUMN OF MINMAX(I,J)
40 DO 50 K=MINMAX(2,I),M
IF (D(K).GT.(G.5G*XMAX)) THEN
MINMAX(3,I)=K
GOTO 60
ENDIF
50 CONTINUE
MINMAX(3,I)=M
60 CONTINUE
70 CONTINUE
C LIMIT THE COUNT TO THE EXPECTED TEN CYCLES
IF (KCOUNT.GT.10) THEN
KCOUNT=10
ENDIF
C PRINT OUT POSITIONS OF THE TROUGHS & PEAKS IN D(K),
C & THE CORRESPONDING X(K) VALUES
WRITE (6,80) KCOUNT
80 FORMAT (IX,' PEAKS FOUND IN DATA FILE.')
WRITE (6,90)
90 FORMAT (1X,'TRough Peak Trough Peak Value',/)
DO 110 K=1,KCOUNT
WRITE (6,100) MINMAX(1,K),MINMAX(2,K),MINMAX(3,K),X(MINMAX(2,K))
110 CONTINUE
C WRITE (6,120)
120 FORMAT (1X,'',/)
WRITE (6,130)
C
xxxii
\end{verbatim}
SUBROUTINE POLYNOM (M1, M2, NCONS, X, Y, CONS, SSERR, YP)
C
C THIS SUBROUTINE FITS POLYNOMIALS TO A SET OF DATA, GIVING THE
C CONSTANTS IN AN ARRAY, & GIVING THE SUM OF SQUARED ERRORS
C
REAL X(10000), Y(10000), YP(10000), XCALC, YCALC, DYBYDC(15)
REAL CONS(15), MAT(15,15), VEC(15), DIFF, SSEOLD, SSERR
INTEGER I, J, K, M1, M2, NCONS, NCY
C
NCONS = N + 1
SSBOLD = 1.0E+30
DO 80 NCY = 1, 200
C
C THIS ROUTINE INITIALISES THE VEC VECTOR & MAT MATRIX
C
DO 20 K = 1, 15
VEC(K) = 0.0
DO 10 J = 1, NCONS
MAT(J, K) = 0.0
10 CONTINUE
20 CONTINUE
C
C INITIALISE SUM OF SQUARE ERRORS & LOOP BETWEEN LIMITING POINTS
C
SSERR = 0.0
DO 50 I = M1, M2
XCALC = X(I)
C
CALL SUBROUTINE YCALC TO FIND CALCULATED VALUES OF Y
C
CALL YCALC(DYBYDC, CONS, NCONS, XCALC, YCALC)
C
YP(I) = YCALC
C
CALCULATE THE DIFFERENCE (DIFF) BETWEEN CALCULATED Y & Y,
C & FIND THE SUM OF SQUARE ERRORS (SSERR)
C
DIFF = YCALC - Y(I)
SSERR = SSERR + DIFF*DIFF
C
C THIS ROUTINE CALCULATES THE VEC AND MAT FOR USE IN GAUSS
C
DO 40 J = 1, NCONS
VECU(J) = VECU(J) + DIFF*DYBYDC(J)
DO 30 K = 1, NCONS
MAT(J, K) = MAT(J, K) + DYBYDC(J)*DYBYDC(K)
30 CONTINUE
40 CONTINUE
50 CONTINUE
C FIND WHETHER THE SOLUTION IS DIVERGING OR CONVERGING
C
IF ((SSEOLD-SSERR).LT.(1.0E-10)) THEN
  WRITE (6,60) SSERR
  GOTO 90
ENDIF
C
SSEOLD=SSERR
C
C CALL SUBROUTINE GAUSS TO PERFORM GAUSS-JORDAN ELIMINATION TO
C GET CORRECTION FACTORS (IN VEC)
C
CALL GAUSS (MAT,NCONS , VEC)
C
C APPLY CORRECTION FACTORS TO CONS VECTOR
C
DO 70 I=1,NCONS
  CONS(I)=CONS(I)-VEC(I)
70 CONTINUE
80 CONTINUE
C
90 WRITE (6,100) NCY
100 FORMAT (1X,'AFTER',12,'ITERATIONS.')
C
C PRINT OUT POLYNOMIALS & THEIR CONSTANTS
C
DO 120 I=1,NCONS
  WRITE (6,110) I, CONS(I)
110 FORMAT (1X,'CONSTANT',12,1X,F10.5)
120 CONTINUE
C
C OPTION TO WRITE OUT PROGRESS OF EACH POLYNOMIAL ON SCREEN
C
C WRITE (6,130)
C 130 FORMAT (/)
C WRITE (6,140)
C 140 FORMAT (1X,'PRESS RETURN TO CONTINUE.....',$)
C READ (5,150)
C 150 FORMAT (Al)
RETURN
END

C XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX

SUBROUTINE YCAL (DYBYDC,CONS,NCONS,XCALC,YCALC)
C
C THIS SUBROUTINE PRODUCES VALUES OF CALCULATED Y BY DETERMINING
C THE VALUE OF THE POLYNOMIAL AT XCALC, AND THEN DIFFERENTIATING
C THE VALUE OF XCALC
C
REAL CONS(15),DYBYDC(15),XCALC,YCALC
INTEGER NCONS
C
YCALC=0.0
DO 10 I=1,NCONS
  YCALC=YCALC+CONS(I)*(XCALC**(I-1))
10 CONTINUE
C
DO 20 I=1,NCONS
  DYBYDC(I)=(XCALC**(I-1))
20 CONTINUE
SUBROUTINE GAUSS (MAT, NCONS, VEC)
C
C THIS SUBROUTINE PERFORMS GAUSS-JORDAN ELIMINATION
C
REAL MAT(15,15), VEC(15), Y
INTEGER I, J, K, NCONS
C
DO 30 I = 1, NCONS
DO 20 J = 1, NCONS
IF (I.EQ.J) GOTO 20
Y = MAT(J, J)/MAT(I, I)
VEC(J) = VEC(J) - VEC(I)*Y
n = N CONS
DO 10 K = n, NCONS
MAT(J, K) = MAT(J, K) - Y*MAT(I, K)
10 CONTINUE
20 CONTINUE
30 CONTINUE
C
DO 40 I = 1, NCONS
VEC(I) = VEC(I)/MAT(I, I)
40 CONTINUE
RETURN
END

SUBROUTINE ENDEPTS (KCOUNT, MINMAX, NCONS, ARRAY, X, AREAS, XVAL)
C
C THIS SUBROUTINE FINDS THE X VALUES NECESSARY FOR INTEGRATING
C THE POLYNOMIALS BETWEEN, i.e. A AND B IN THE INTEGRATION ROUTINE
C
DIMENSION MINMAX(3,50)
REAL AREAS(25), ARRAY(25,15), POLY1(15), POLY2(15), POLY3(15)
REAL XVAL(25,2), X(10000)
INTEGER FOUND, I, J
C
DO 20 J = 1, (KCOUNT*2), 2
DO 10 I = 1, NCONS
C
C DEFINE POLYNOMIAL ARRAYS
C
POLY1(I) = ARRAY(J, I)
POLY2(I) = ARRAY(J+1, I)
P OLY3(I) = POLY1(I) + POLY2(I)
10 CONTINUE
C
C INITIALISE VALUES
C
XVAL(I, 1) = 0.0001
XVAL(I+1, 1) = 0.0001
XVAL(I, 2) = 0.0001
C
C IF POLY1 CUTS X-AXIS TO LEFT OF ORIGIN SET XVAL TO ZERO
C
CALL NRVAL (FOUND, NCONS, POLY1, XVAL(I, 1))
XVAL(I, 1) = MAX(0.0, XVAL(I, 1))
IF (FOUND.EQ.0) THEN
  XVAL(J,1)=0.0
ENDIF

C IF POLY2 CUTS X-AXIS TO LEFT OF ORIGIN SET XVAL TO ZERO
C
CALL NRVAL (FOUND,NCONS,POLY2,XVAL(J+1,1))
XVAL(J+1,1)=MAX(0.0,XVAL(J+1,1))
IF (FOUND.EQ.0) THEN
  XVAL(J+1,1)=0.0
ENDIF

C IF POLY1 AND POLY2 INTERSECT BEFORE MOVING TO LEFT OF ORIGIN,
C & IS WITHIN 10% OF THE ORIGIN, THEN SET XVAL TO THAT
C CROSSING POINT VALUE, ELSE SET XVAL TO ZERO
C
CALL NRVAL (FOUND,NCONS,POLY3,XVAL(J,2))
IF((FOUND.EQ.1).AND.(XVAL(J,2).GT.0.0).AND.(XVAL(J,2).LT.(X(MINMAX(2,J/2+1))*0.1))) THEN
  XVAL(J,1)=XVAL(J,2)
  XVAL(J+1,1)=XVAL(J,2)
ELSE
  XVAL(J,1)=0.0
  XVAL(J+1,1)=0.0
ENDIF
XVAL(J,2)=X(MINMAX(2,J/2+1))

C IF POLY1 AND POLY2 INTERSECT SOMEWHERE BETWEEN 10% BELOW &
C 10% ABOVE XMAX FOR THAT LOOP, THEN SET XVAL TO THAT CROSSING POINT,
C ELSE SET XVAL TO XMAX OF THAT LOOP
C
CALL NRVAL (FOUND,NCONS,POLY3,XVAL(J,2))
IF((FOUND.EQ.1).AND.(XVAL(J,2).LT.(X(MINMAX(2,J/2+1))*1.1)).AND.(XVAL(J,2).GT.(X(MINMAX(2,J/2+1))*0.9))) THEN
  XVAL(J+1,2)=XVAL(J,2)
ELSE
  XVAL(J+1,2)=X(MINMAX(2,J/2+1))
  XVAL(J,2)=X(MINMAX(2,J/2+1))
ENDIF

C CALL SUBROUTINE INTEGR TO INTEGRATE THE POLYNOMIALS BETWEEN
C THE END VALUES DETERMINED
C
CALL INTEGR (NCONS,POLY1,XVAL(J,1),XVAL(J,2),AREAS(J))
CALL INTEGR (NCONS,POLY2,XVAL(J+1,1),XVAL(J+1,2),AREAS(J+1))
20 CONTINUE

C WRITE (6,30)
30 FORMAT (1X,ESTIMATED AREAS OF POLYNOMIALS'/)
WRITE (6,40)
40 FORMAT (1X,'AREA OF LOOP ',12,' IS = ',F9.5)

DO 80 I=1,KCOUNT*2,2
  WRITE(6,70) (I+1)/2,AREAS(I)-AREAS(I+1)
70 FORMAT(1X,'AREA OF LOOP ',12,' IS = ',F9.5)
80 CONTINUE

RETURN
END

Cxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx
C SUBROUTINE NRVAL (FOUND,NCONS,POLY,X)
C
C THIS SUBROUTINE INCORPORATES THE NEWTON RAPHSON METHOD IN ORDER TO
C DETERMINE THE INTERSECTION POINTS WHERE THE POLYNOMIALS CUT THE X-AXIS.
C THE NEWTON RAPHSON TECHNIQUE IS USED TO FIND ROOTS OF INTERSECTION.
C
REAL DFFN,FN,POLY(15),DFPOLY(15),OLDX,X
INTEGER FOUND,LL,NCONS,NCONDF
C
FOUND=1
NCONDF=NCONS-1
DO 10 I=1,NCONDF
C
C DIFFERENTIATE POLYNOMIALS TO FIND SLOPE
C
DFPOLY(I)=POLY(H-I)**(H-I)
10 CONTINUE
K=0
20 OLDX=X
K=K+1
C
C CALL SUBROUTINE NRVAL TO FIND THE VALUE FN AND
C DFFN FOR USE IN THE NEWTON RAPHSON METHOD
C
CALL FUNVAL(FN,NCONS,POLY,X)
CALL FUNVAL(DFFN,NCONS,POLY,X)
C
C NEWTON RAPHSON METHOD
C
X=X-(FN/DFFN)
C
C CHECK: TO SEE IF THE VALUE OF FN IS SMALL ENOUGH AND CONVERGING
C AFTER 1000 ITERATIONS
C
IF ((ABS(FN).GT.(1.0E-5)).AND.(K.LT.1000)) GOTO 20
IF (K.GE.1000) THEN
FOUND=0
ENDIF
RETURN
END
SUBROUTINE NRVAL (FN,NCONS,POLY,X)
C
C THIS SUBROUTINE FINDS THE VALUES OF FN & DFFN FOR USE IN THE
C NEWTON RAPHSON EXPRESSION
C
REAL P(15),X,F
INTEGER LN
C
F=0.0
DO 10 I=1,N
F=F+(P(I)**(X**(I-1)))
10 CONTINUE
RETURN
END
SUBROUTINE INTEGR (NCONS,CONS,A,B,AREA)
C
C THIS SUBROUTINE ESTIMATES THE AREA UNDER A POLYNOMIAL BY
C INTEGRATION
C
xxxvii
REAL AREA, A, B, CONS(15)
INTEGER I, NCONS
C
AREA=0.0
DO 10 I=1, NCONS
AREA=AREA+CONS(I)/FLOAT(I)*ABS((B**I)-(A**I))
10 CONTINUE
RETURN
END

C***************************************************************************
IV.2 Test data conversion program using modified trapezoidal rule

C
C WRITTEN BY J.M.WILLIAMS
C DEVELOPED SEPT 92 - MAR 94,
C DEPARTMENT OF ENGINEERING, UNIVERSITY OF LEICESTER.
C
C
PROGRAM TRAP
C
REAL T(10000),X(10000),Y(10000),W(10000)
REAL TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,YMINRAW,YMAXRAW
REAL WMINRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,YMAX
REAL WMIN,WMAX,XMEAN,NWMIN,NWMAX
INTEGER KCOUNTM
CHARACTER ANS*4
C
C WRITE (6,10)
10 FORMAT (IX, "**********************************")
C
C CHOOSE CONVERSION PROGRAM
C
50 WRITE (6,60)
60 FORMAT (IX,' CHOOSE CONVERSION PROGRAM: ',IX, 
1 '10kN CELL (10) OR 25kN CELL (25) ?',A4)
READ (5,70) ANS
70 FORMAT (A4)
C
IF (ANS.EQ.'10') THEN
GOTO 90
ENDIF
IF (ANS.EQ.'25') THEN
GOTO 100
ENDIF
C
C CALL SUBROUTINE MAIN10 TO RUN THE DATA CONVERSION PROGRAM FOR
C CONVERTING THE 10KN COMPRESSION DATA SAMPLED USING DASH300
C
90 CALL MAIN10 (T,X,Y,TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,
1 YMINRAW,YMAXRAW,WMINRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,
1 YMIN,YMAX,WMIN,WMAX,XMEAN,KCOUNTM,NWMIN,NWMAX)
GOTO 110
C
C CALL SUBROUTINE MAIN25 TO RUN THE DATA CONVERSION PROGRAM FOR
C CONVERTING THE 25KN COMPRESSION DATA SAMPLED USING DASH300
C
100 CALL MAIN25 (T,X,Y,W,TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,
 1YMINRAW,YMAXRAW,WMINRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,
 1YMIN,YMAX,WMIN,WMAX,XMEAN,KCOUNTM,NWMIN,NWMAX)
C
110 WRITE (6,120)
120 FORMAT(1X,'OPERATION COMPLETE',//)
END

SUBROUTINE MAIN25 (T,X,Y,W,TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,
 1YMINRAW,YMAXRAW,WMINRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,
 1YMAX,WMIN,WMAX,XMEAN,KCOUNTM,NWMIN,NWMAX)
C
DIMENSION MINMAX(3,50)
REAL T(10000),X(10000),Y(10000),W(10000),TMINRAW,TMAXRAW
REAL XMINRAW,XMAXRAW,YMINRAW,YMAXRAW,WMINRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,
 1YMAX,WMIN,WMAX,XMEAN,KCOUNTM,NWMIN,NWMAX)
C
WRITE (6,10)
10 FORMAT (/ix,'***' '''***********************' ''**********',/)
C
C CALL SUBROUTINE CONV10 TO CONVERT RAW DATA
C
CALL CONV10 (T,X,Y,W,FILENAME)
C
C CALL SUBROUTINE SMOOTH TO PERFORM 9 & 15 POINT SMOOTH ON Y & W
C
CALL SMOOTH (Y,W,M,SY,SW,NWMIN,NWMAX)
C
WRITE (6,20) NWMN,NWMAX
20 FORMAT (1X,'NWMIN',F9.6,' NWMAX',F9.6,/) 
WRITE (6,30)
30 FORMAT (1X,10*X,10*X,/) 
WRITE (6,40)
40 FORMAT (1X,'PRESS RETURN TO CONTINUE...',/) 
READ (5,50)
50 FORMAT (A1) 
WRITE (6,60)
60 FORMAT (1X,'**************************************',/) 
C
WRITE (6,70) M 
70 FORMAT (1X,14,IX,'DATA POINTS FOUND, AND',/) 
C
CALL SUBROUTINE DERIV TO CALCULATE THE 1st DERIVATIVES
C
CALL DERIV (M,XMAX,KCOUNT,MINMAX)
C
CALL SUBROUTINE AREA TO CALCULATE THE AREA OF EACH HYSTERESIS LOOP
C
CALL AREA (FILENAME,KCOUNT,MINMAX,X,Y,TMINRAW,TMAXRAW,XMINRAW,
1XMAXRAW,YMINRAW,YMAXRAW,WMINRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,
 1YMIN,YMAX,WMIN,WMAX,NWMIN,NWMAX)
C
C WRITE CONVERTED TEST DATA TO A FILE ???.NEW
C
FILENAME2=FILENAME
L=INDEX(FILENAME2,'.dat')
SUBROUTINE CONVIO (T,X,Y,W,M,TMINRAW,TMAXRAW,XMINRAW,
   XMAXRAW,YMINRAW,YMAXRAW,WMINRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,
   YMIN,YMAX,WMIN,WMAX,XMEAN,FILENAME)
C
C SUBROUTINE TO CONVERT 10KN COMPRESSION DATA USING DASH300, CONVERTS
C SAMPLE NO, LOAD(V), AXIAL DISPL(V) & LATERAL DISPL(V) TO
C SAMPLE NO, STRESS(MPA), AXIAL STRAIN & LATERAL STRAIN
C
REAL T(10000),X(10000),Y(10000),W(10000)ACTRANS,CELL,DCTRANS
REAL TMINRAW,TMAXRAWXMINRAW,XMAXRAW, YMINRAW, YMAXRAW,WMINRAW
   WMAXRAW REAL WMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,YMAX,WMIN,WMAX,XMEAN,PI
INTEGER KMJSIDATA
CHARACTER FILENAME*20
C
10 WRITE (6,20)
20 FORMAT (1X,'INPUT FILENAME: ',$)
READ (5,30) FILENAME
30 FORMAT (A20)
C
C CALL SUBROUTINE INITIAL TO INITIALISE ALL MINS & MAXS
C
CALL INITIAL (TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,YMINRAW,YMAXRAW,
   WMINRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,YMAX,WMIN,WMAX,XMEAN)
C
C OPEN RAW DATA FILE
C
NDATA=1
OPEN (UNIT=1,FILE=FILENAME,STATUS='OLD',ERR=50)
40 READ (1,*,END=70) T(NDATA),Y(NDATA),X(NDATA),W(NDATA)
50 CONTINUE
60 FORMAT (1X,'NO SUCH FILE EXISTS !',/$)
GOTO 10
C CREATE ABSOLUTE VALUES OF THE DATA, THEN APPLY
C CALIBRATION FACTORS & DATA CORRECTIONS TO ABSOLUTE DATA
C
DO 80 K=1,M
Y(K)=Y(K)-YMINRAW
X(K)=ABS(X(IC)-XMAXRAW)
W(IC)=W(R)-WMINRAW
80 CONTINUE
C
ACTRANS=3.95
CELL=1.0
DCTRANS=4.32
PI=(2*ASIN(1.0))
C
DO 90 IC=1,JVI
Y(IC)=((Y(K)/1000)*CELL)/(0.225*PI)
X(KM(X(K)/1000)*ACTRANS)/30.0
W(IC)=((W(R)/1000)*DCTRANS)/15.0
C
C FIND MINS & MAXS OF CONVERTED DATA
C
TMIN=MIN(TMIN,T(K))
TMAX=MAX(TMAX,T(K))
XMIN=MIN(XMIN,X(K))
XMAX=MAX(XMAX,X(K))
YMIN=MIN(YMIN,Y(K))
YMAX=MAX(YMAX,Y(K))
WMIN=MIN(WMIN,W(IC))
WMAX=MAX(WMAX,W(IC))
XMEAN=XMEAN+X(K)
C
90 CONTINUE
XMEAN=XMEAN/FLOAT(M)
C
C PRINT OUT MINS & MAXS OF THE RAW DATA
C
WRITE (6,100)
100 FORMAT (/1X,' MAXS & MINS OF RAW DATA:' )
WRITE (6,110) TMINRAW,TMAXRAW,YMINRAW,YMAXRAW,XMINRAW,XMAXRAW,
1WMINRAW,WMAXRAW
110 FORMAT (1X,' TMIN' E9.2,'  ' TMAX ' ,F9.2,/,1X,' YMIN' ,F9.5,
1Y MAX 'F9.5,/,lX,' XMIN ' ,F9.5,'  XMAX ' ,F9.5,' /,
1' WMIN' ,F9.5,'  WMAX' ,F9.5)
C
C PRINT OUT MINS & MAXS OF THE CONVERTED DATA
C
WRITE (6,120)
120 FORMAT (/1X,' MAXS & MINS OF CONVERTED DATA:' )
WRITE (6,130) TMIN,TMAX,YMIN,YMAX,XMIN,XMAX,WMIN,WMAX
130 FORMAT (1X,' TMIN' E9.5,'  ' TMAX ' ,F8.1,/,1X,' YMIN' ,F9.5,
1Y MAX 'F9.5,' /,XMIN','F9.5,'  XMAX ' ,F9.5,' /
1' WMIN' ,F9.5,'  WMAX' ,F9.5)
RETURN
END
DIMENSION MINMAX(3,50)
REAL T(10000),X(10000),Y(10000),W(10000),SY(10000),SW(10000)
REAL TMNRAW,TMAXRAW,XMINRAW,XMAXRAW,YMINRAW,YMAXRAW,XMEAN
REAL WMNRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,YMAX,WMIN,WMAX
REAL NWMIN,NWMAX
INTEGER L,KCOUNT,M
CHARACTER FELENAME*20,JFILENAME2*20

C
WRITE (6,10)
10 FORMAT (I1X,'********************************************',/)

C CALL SUBROUTINE CONV25 TO CONVERT RAW DATA
C
CALL CONV25 (TX,Y,W,M,TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,
1 YMINRAW,YMAXRAW,WMNRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,
1 YMAX,WMIN,WMAX,XMEAN,JFILENAME2)

C CALL SUBROUTINE SMOOTH TO PERFORM 9 & 15 POINT SMOOTH ON Y & W
C
CALL SMOOTH (Y,W,M,SY,SW,NWMIN,NWMAX)

WRITE (6,20) NWMIN,NWMAX
20 FORMAT (1X,' NWMIN',F9.5,'  NW MAX',F9.5,/) 
WRITE (6,30)
30 FORMAT (1X,'********************************************',/)
WRITE (6,40)
40 FORMAT (1X,'PRESS RETURN TO CONTINUE.....',$/)
READ (5,50)
50 FORMAT (A1)
WRITE (6,60)
60 FORMAT (1X,'********************************************',/)

C
WRITE (6,70) M 
70 FORMAT (1X,1I4,1X,DATA POINTS FOUND, AND') 

C CALL SUBROUTINE DERIV TO CALCULATE THE 1st DERIVATIVES
C
CALL DERIV (MX,XMAX,KCOUNT,MINMAX)

C CALL SUBROUTINE AREA TO CALCULATE THE AREA OF EACH HYSERESIS LOOP
C
CALL AREA (FILENAME,KCOUNT,MINMAX,X,Y,TMINRAW,TMAXRAW,XMINRAW,
1 XMAXRAW,YMINRAW,YMAXRAW,WMINRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,
1 YMIN,YMAX,WMIN,WMAX,NWMIN,NWMAX)

C
C WRITE CONVERTED TEST DATA TO A FILE ???,NEW
C
FILENAME2=FILENAME
L=INDEX(FILENAME2,'.dat')
FILENAME2(L:L+4)='.new'
OPEN (UNIT=3,FILE=FILENAME2,STATUS='UNKNOWN')
DO 90 K=1,M
WRITE (3,80) T(K),X(K),Y(K),W(K),SY(K),SW(K)
80 FORMAT (1X,F6.1,5(','F9.5))
90 CONTINUE
CLOSE(3)

C
WRITE (6,100)
100 FORMAT (I1X,'********************************************',/)
WRITE (6,110)
110 FORMAT (I1X,'TRAP(25) DATA CONVERSION COMPLETE',/)
RETURN
SUBROUTINE CONV25 (T,X,Y,W,M,TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,
YMINRAW,YMAXRAW,WMINRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,
YMAX,WMIN,WMAX,XMEAN,FILENAME)
C
C SUBROUTINE TO CONVERT 25KN COMPRESSION DATA USING DASH300, CONVERTS
C SAMPLE No, LOAD(V), AXIAL DISPL.(Y) & LATERAL DISPL.(V) TO
C SAMPLE No, STRESS(MPA), AXIAL STRAIN & LATERAL STRAIN
C
REAL T(10000),X(10000),Y(10000),W(10000),ACTRANS,DCTRANS
REAL TMINRAW, TMAXRAW, XMINRAW, XMAXRAW, YMINRAW, YMAXRAW
REAL WMINRAW, WMAXRAW, TMIN, TMAX, XMIN, XMAX, YMIN, YMAX
REAL XMEAN, PI, CELL
INTEGER K,M,NDATA
CHARACTER FILENAME*20
C
10 WRITE (6,20)
20 FORMAT (1X,'INPUT FILENAME: ',A)$
READ (5,30) FILENAME
30 FORMAT (A20)
C
C CALL SUBROUTINE INITIAL TO Initialise ALL MINS & MAXS
C
CALL INITIAL (TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,YMINRAW,YMAXRAW,
1WMINRAW,WMAXRAW,TMIN,TMAX,XMIN,XMAX,YMIN,YMAX,
1REAL XMEAN, PI, CELL
INTEGER K,M,NDATA
CHARACTER FILENAME*20
C
10 WRITE (6,20)
20 FORMAT (1X,'INPUT FILENAME: ',A)$
READ (5,30) FILENAME
30 FORMAT (A20)
C
C OPEN RAW DATA FILE
C
NDATA=1
OPEN (UNIT=1,FILE=FILENAME,STATUS='OLD',ERR=50)
40 READ (1,*END=70) T(NDATA),X(NDATA),W(NDATA),Y(NDATA)
C
C FIND MINS & MAXS OF RAW DATA
C
TMINRAW=MIN(TMINRAW,T(NDATA))
TMAXRAW=MAX(TMAXRAW,T(NDATA))
XMINRAW=MIN(XMINRAW,X(NDATA))
XMAXRAW=MAX(XMAXRAW,X(NDATA))
YMINRAW=MIN(YMINRAW,Y(NDATA))
YMAXRAW=MAX(YMAXRAW,Y(NDATA))
WMINRAW=MIN(WMINRAW,W(NDATA))
WMAXRAW=MAX(WMAXRAW,W(NDATA))
C
NDATA=NDATA+1
GOTO 40
50 WRITE (6,60)
60 FORMAT (I1,'NO SUCH FILE EXISTS !(,/,)
GOTO 10
70 CLOSE(1)
M=NDATA-1
C
C CREATE ABSOLUTE VALUES OF THE DATA, THEN APPLY CALIBRATION
C FACTORS AND DATA CORRECTIONS TO ABSOLUTE DATA
C
DO 80 K=1,M
Y(K)=ABS(Y(K)-YMAXRAW)
X(K)=ABS(X(K)-XMAXRAW)
W(K)=W(K)-WMINRAW
80 CONTINUE

ACTRANS=5.28

CELL=2.5

DCTRANS=4.32

PI=(2*ASIN(1.0))

DO 90 K=1,M

Y(K)=((Y(K)/1000)*CELL)/(0.225*PI)

X(K)=((XAG/1000)*ACTRANS)/30.0

W(K)=((W(K)/1000)*DCTRANS)/15.0

C FIND MINS & MAXS OF CONVERTED DATA

TMIN=MIN(TMIN,T(K))

TMAX=MAX(TMAX,T(K))

XMIN=MIN(XMIN,X(K))

XMAX=MAX(XMAX,X(K))

YMIN=MIN(YMIN,Y(K))

YMAX=MAX(YMAX,Y(K))

WMIN=MIN(WMIN,W(K))

WMAX=MAX(WMAX,W(K))

XMEAN=XMEAN+X(K)

CONTINUE

XMEAN=XMEAN/FLOAT(M)

C PRINT OUT MINS & MAXS OF THE RAW DATA

WRITE (6,100)


C PRINT OUT THE MINS & MAXS OF THE CONVERTED DATA

WRITE (6,120)

120 FORMAT (/,' TMIN ',F9.5,' TMAX ',F9.5,/,1X,'YMIN ',F9.5,' YMAX ',F9.5,/,1X,'XMIN ',F9.5,' XMAX ',F9.5,/,1X,'WMIN ',F9.5,' WMAX ',F9.5)

RETURN

END

SUBROUTINE INITIAL (TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,YMINRAW,YMAXRAW,WMINRAW,WMAXRAW)

C SUBROUTINE TO UNITIALISE ALL MINS & MAXS

REAL TMINRAW,TMAXRAW,XMINRAW,XMAXRAW,YMINRAW,YMAXRAW,WMINRAW,WMAXRAW

C

TMINRAW=10000.0

TMAXRAW=-10000.0

XMINRAW=10000.0

XMAXRAW=-10000.0

YMINRAW=10000.0

YMAXRAW=-10000.0

WMINRAW=10000.0

WMAXRAW=-10000.0

xlv
SUBROUTINE DERIV (M,X,XMAX,JCOUNT,MINMAX)

C THIS SUBROUTINE CALCULATES THE 1st DERIVATIVES OF X(K)
C IN ORDER TO PRODUCE +VE & -VE SLOPES WHICH ARE USED TO
C DETERMINE THE PEAKS & TROUGHS IN X(K)

DIMENSION MINMAX(3,50)
REAL D(10000),X(10000),XMAX
INTEGER K,JCOUNT,M

C CALCULATE THE 1st DERIVATES of X(K), PUT INTO D(K)

DO 10 K=3,M-2
    D(K)=(-2*X(K-2)-X(K-1)+X(K+1)+2*X(K+2))/10.0
10 CONTINUE

KCOUNT=0

C FIND POINTS (PEAKS) WHERE D(K) CROSSES FROM +VE TO -VE.
C PUT VALUES IN THE 2nd COLUMN OF MINMAX(I,J)

DO 70 I=1,KCOUNT
    DO 30 K=MINMAX(2,I)-1,0,-1
        IF (D(K).LE.0.0) THEN
            MINMAX(1,I)=K+1
            GOTO 40
        ENDIF
30 CONTINUE
    MINMAX(1,I)=0.0
70 CONTINUE

C FIND POINTS (1st TROUGHS) WHERE D(K) IS -VE OR ZERO.
C PUT VALUES IN THE 1st COLUMN OF MINMAX(I,J)

DO 20 J=1,M-1
    IF ((D(K),GT.0.0) AND (D(K+1),LE.0.0)) THEN
        IF (X(K),GT.(0.50*XMAX)) THEN
            KCOUNT=KCOUNT+1
            MINMAX(2,J)=K+1
        ENDIF
20 CONTINUE

C FIND POINTS (2nd TROUGHS) WHERE D(K) IS +VE.
C PUT VALUES IN THE 3rd COLUMN OF MINMAX(I,J)

DO 40 K=MINMAX(2,J),M
    GOTO 40
40 CONTINUE

C
IF (D(K).GT.0.0) THEN
MINMAX(3,J)=K
GOTO 60
ENDIF
50 CONTINUE
MINMAX(3,J)=M
60 CONTINUE
70 CONTINUE

C LIMir THE COUNT TO THE EXPECTED TEN CYCLES
C
IF (KCOUNT.GT.10) THEN
KCOUNT=10
ENDIF
C
C PRINT OUT POSITIONS OF THE TROUGHS & PEAKS IN D(K),
C & THE CORRESPONDING X(K) VALUES
C
WRITE (6,80) KCOUNT
80 FORMAT (1X,I2,' PEAKS FOUND IN DATA FILE.',/) WRITE (6,90)
90 FORMAT (1X,'TROUGH PEAK TROUGH PEAK VALUE')
DO 110 K=1,JCCOUNT
WRITE (6,100) MINMAX(1,J),MINMAX(2,K),MINMAX(3,K), X(MINMAX(2,K))
100 FORMAT (1X,3(I4,4X),F9.5)
110 CONTINUE
C
WRITE (6,120)
120 FORMAT (1X,,'**************************************************************')
WRITE (6,130)
130 FORMAT (1X,'PRESS RETURN TO CONTINUE....',$/)
READ (5,140)
140 FORMAT (Al)
WRITE (6,150)
150 FORMAT (1X,,'**************************************************************')
160 FORMAT (1X,'AREAS OF HYSTERESIS LOOPS',$/)
RETURN
END

SUBROUTINE AREA (FILENAME,JCCOUNT,MINMAXXY,TMINRAW,TMAXRAW,
1XMINRAW,XMAXRAW,YMINRAW,YMAXRAW,WMINRAW,WMAXRAW,TMIN,TMAX,
1XMIN,WMAX,WMIN,WMAX,NMIN,NMAX)
C
C SUBROUTINE THAT USES A MODIFIED FORM OF THE TRAPEZOIDAL RULE
C TO CALCULATE THE AREA UNDER EACH SET OF CURVES
C
DIMENSION MINMAX(3,50)
REAL X(10000),Y(10000),AREAS(50),SUM1, SUM2,NMIN,NMAX
INTEGER I,J,KCOUNT,L
CHARACTER FILENAME*20,FILNAME1*20
C
DO 40 L=1,JCCOUNT
C
SUM1=0.0
SUM2=0.0
C
DO 10 K=MINMAX(1,J),MINMAX(2,J)
SUM1=SUM1+((X(K+1)-X(K))*(Y(K)+Y(K+1)))/2.0
10 CONTINUE
C
DO 20 K=MINMAX(2,I),MINMAX(3,I)
SUM2=SUM2-((X(K+1)-X(K))*(Y(K)+Y(K+l))/2.0)
20 CONTINUE
C = AREAS(I)=SUM1-SUM2
WRITE (6,30) I,SUM1,SUM2,AREAS(I)
30 FORMAT (1X,'LOOP ',12,' : 'F7.4, ' -'E7.4,' ='F7.5)
40 CONTINUE
C = WRITE AREAS OF HYSTERESIS LOOPS TO A FILE ???.OUT
C = FILENAME1=FILENAME
L=INDEX(FILENAME1 ,'.dat')
FILENAME1(L:L+4)='.out'
OPEN (UNIT=2,FILE=FILENAME1,STATUS='UNKNOWN')
WRITE (2,50) FILENAME1
50 FORMAT (1X,EILE : 'A20)
C = PRINT OUT MINS & MAXS OF THE RAW DATA
C = WRITE (2,60)
60 FORMAT (/,1X,'MAXS & MINS OF RAW DATA:')
WRITE (2,70) TMINRAW,TMAXRAW,YMINRAW,YMAXRAW,XMINRAW,XMAXRAW,
1WMINRAW,WMAXRAW
70 FORMAT (1X,'TMIN',F9.2,' TMAX ',F9.2,/,1X,'YMIN',F9.2,' YMAX',E9.2,
/,1X,'XMIN',F9.2,' XMAX ',F9.2,/,1X,'WMIN ',F9.2,' WMAX',F9.2)
C = PRINT OUT MINS & MAXS OF THE CONVERTED DATA
C = WRITE (2,80)
80 FORMAT (/,1X,'MAXS & MINS OF CONVERTED DATA:')
WRITE (2,90) TMIN,TMAX,YMIN,YMAX,XMIN,XMAX,WMIN,WMAX,
1NWMIN,NWMAX
90 FORMAT (1X,TMIN',F9.5,' TMAX ',F8.1,/,1X,'XMIN',F9.5,' XMAX ',F9.5,
/,1X,'YMIN',F9.5,' YMAX ',F9.5,/,1X,'WMDS, JF9.5,' WMAX',F9.5,
/,1X,'NWMIN',F9.5,' NWMAX',F9.5)
C = WRITE (2,100)
100 FORMAT (1X,/,1X,'AREAS OF HYSTERESIS LOOPS:')
DO 120 I=I,Jccount
WRITE (2,110) AREAS(I)
110 FORMAT (1X,'AREA OF LOOP ',12,' IS : ',F8.5)
120 CONTINUE
WRITE (2,130)
130 FORMAT (1/) C = CLOSE(2)
RETURN
END
C SUBROUTINE SMOOTH (S9,S15,M,AS9,AS15,NWMIN,NWMAX)
C THIS SUBROUTINE SMOOTHS OUT THE Y(K) & W(K) DATA COLUMNS
C = REAL S9(10000),S15(10000),AS9(10000),AS15(10000),NWMIN,NWMAX
INTEGER I,L
C = NWMIN=10000.0
NWMAX=-10000.0
C
NINE POINT SMOOTHING ALGORITHM

\begin{align*}
\text{DO 10 } & \text{I}=5,6,\ldots,M-4 \\
AS9(I) &= (-21\cdot S9(I-4)+14\cdot S9(I-3)+39\cdot S9(I-2)+54\cdot S9(I-1)+59\cdot S9(I) \\
&+ 154\cdot S9(I+1)+39\cdot S9(I+2)+14\cdot S9(I+3)-21\cdot S9(I+4))/231.0 \\
10 \text{ CONTINUE}
\end{align*}

FIFTEEN POINT SMOOTHING ALGORITHM

\begin{align*}
\text{DO 20 } & \text{I}=9,10,\ldots,M-8 \\
AS15(I) &= (-78\cdot S15(I-7)+13\cdot S15(I-6)+42\cdot S15(I-5)+87\cdot S15(I-4) + \\
&+ 1122\cdot S15(I-3)+147\cdot S15(I-2)+162\cdot S15(I-1)+167\cdot S15(I)+162\cdot S15(I+1) + \\
&+ 1147\cdot S15(I+2)+122\cdot S15(I+3)+87\cdot S15(I+4)+42\cdot S15(I+5)+13\cdot S15(I+6)- \\
&- 178\cdot S15(I+7))/1105.0 \\
20 \text{ CONTINUE}
\end{align*}

NWMIN = \text{MIN}(NWMIN,AS15(I))

NWMAX = \text{MAX}(NWMAX,AS15(I))

\text{IF (NWMIN} \gt 0.0) \text{ THEN}

NWMIN = 0.000000

\text{ENDIF}

\text{RETURN}

END

----------------------------------------------------------------------------------------------------------------------