The role of microsuperplastic flow in high temperature fracture of AL-9021 mechanically alloyed aluminium

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A relatively new fracture mechanism, labelled microsuperplasticity, has been found to occur at elevated temperatures in some powder metallurgical aluminium alloys. Specifically it has been found to occur in mechanically alloyed aluminium AL-9021, as well as in a rapidly solidified aluminium alloy. Under load at high temperatures the material fractures with the formation of superplastic fibres. The mechanism responsible for this fibre formation on the fracture surface is thought to be due to the loose bonds of oxygen along the periphery of the grain and sub-grain boundaries. This behaviour has direct implications for the theories of superplasticity. It also helps in our understanding of mechanical alloys and powder metallurgical processing of aluminium alloys.

1. Introduction

Mechanical alloys (MAs) are a new class of materials that exhibit unusual properties and behaviour due to the special processing used to create the base material. Using this process it is possible to fabricate a material consisting of metal, ceramic and polymer, all existing simultaneously in a fairly stable state. These materials are made by using a controlled process of cold welding and fracturing on a very fine scale [1, 2]. The process uses high-energy attrition milling to produce ultra-fine powder. This grinding results in a mechanical mixing within the powder of various elements comprising the alloy. One of the last stages in processing this material is the consolidation of the powder under pressure and temperature similar to other powder metallurgical (PM) materials.

The material AL-9021 is chemically analogous to ingot metallurgy 2024 aluminium alloy except for the addition of 0.8% oxygen and 1.1% carbon. The carbon is introduced from a stearate used to control the cold-weld/fracture process. Recently, it has been found that the carbon exists within the material in the form of hydrocarbon [3]. The material has typically an ultra-fine grain size being $0.18 \, \mu m$ with a subgrain size of $0.02 \, \mu m$ [4]. The combination of strength and ductility of AL-9021-T452 exceeds that of 7075-T6 making it extremely useful as a high strength to weight ratio material.

It is generally believed that oxide and carbide dispersive strengthening are the major mechanisms imparting strength to these materials [5]. For oxides occurring in nickel base MAs, where yttrium oxide is introduced directly as one of the alloy elements at the time of processing, this might be true. However, in the aluminium MAs, the oxygen is introduced inadvertently from the atmosphere during processing.

It is now known that oxygen does not form a stable product of aluminium oxide in AL-9021 [6]. Rather than the usual strong bonds associated with aluminium oxide, a mechanical proximity reaction with very weak bonding occurs between the oxygen and the other metals present. In this material and other PM materials, true aluminium oxide has not formed on the particle surfaces during processing and hence cannot act as an oxide strengthening mechanism. It is possible and likely that the mechanical proximity reaction in which the oxygen participates in some manner with the aluminium does act to strengthen the material. An understanding of microsuperplasticity is based on this mechanical proximity reaction of oxygen in AL-9021 and other PM-produced aluminium.

Superplasticity is normally a macroscopic phenomenon and generally has practical applications in component forming. It is defined macroscopically as a localized neck-free elongation in excess of 100%. The general requirements for this behaviour are: first, a very fine and stable-grained structure with grain size less or equal to $10 \, \mu m$; second, operation at a temperature above 0.4 of the melting point temperature of the material; third, a high strain rate sensitivity factor greater than 0.3.

The phenomena of microsuperplasticity has been defined previously [7, 8]. It is the occurrence of superplastic behaviour on a microscopic scale during fracture of the component which results in a superplastic fibre formation on the fracture surfaces. More recently it has also been found to occur in a rapidly solidified aluminium alloy [9]. This means the mechanism is more broad than originally thought. It is therefore possible that a better understanding of its cause might be identified by the commonality in the processing methods of these two different materials.

2. Experimental details

This study of microsuperplasticity resulted from four different experimental programmes using AL-9021 material. The first programme was that of creep crack growth in small compact fracture toughness specimens. The second used heat-treated material exposed to temperatures approaching the melting point of the material. The third was tensile tests conducted at an elevated temperature and at various strain rate conditions. The fourth examined standard creep specimens held under constant load at constant temperature.

Creep crack growth tests were conducted on prefatigued components, 32 mm × 30 mm × 12.5 mm thick, with the initial presence of a sharp precrack. Tests were conducted at constant load conditions with pin displacement and time being monitored. Tests were run over the temperature range of 200 to 400° C. Crack growth occurred in the following stages; primary crack opening displacement (COD), secondary COD, component buckling, secondary COD, tertiary COD, and final fast overload fracture.

The heat-treated components, 12 mm × 20 mm × 8 mm thick, were solution treated at 595 to 620° C for various times followed by air cooling. The components were then sectioned and polished for both optical and electron microscopy. Sectioning was conducted using a slow speed diamond cut-off wheel. The specimens were then mechanically polished to 0.5 μm.

Constant strain rate tensile tests, using button head specimens, 17.5 mm gauge length and 4 mm diameter were conducted using a servo-controlled hydraulic material testing system, MTS model 810. A split furnace maintained a temperature to 400 ± 1° C through the duration of the tests. Recordings of load displacement were made which were later converted to stress–strain. The strain rates were varied over the range from 495 mm^{-1} min^{-1} to 5 × 10^{-5} mm^{-1} min^{-1}.

Standard creep tests were conducted on a Satec P5 constant load creep machine. Tests were carried out at a temperature of 400° C monitoring both time and extension. The specimens size was 5 mm diameter with a 27 mm gauge length and threaded ends.

Fracture morphology was determined using a Cambridge S100 scanning electron microscope (SEM).

3. Microsuperplasticity
3.1. Creep crack growth fracture morphology

The study of fracture morphology of creep crack growth specimens showed the formation of microsuperplasticity in all cases. Superplastic fibres were found to form at creep crack growth temperatures as low as 200° C, Fig. 1. This micrograph shows short fibre formation in specific locations, usually surrounding an intergranular failure region. Thus regions of intergranular fracture (brittle fracture) occur in cell-like arrangements surrounded by microsuperplastic failure (super ductile fracture). This type of mixed fracture behaviour is typical of most of the creep crack growth regions studied. The fracture behaviour of creep crack growth specimens operating at 400° C is shown in Fig. 2. Here it can be seen that a longer, finer and more distinct fibre formation occurs than at the
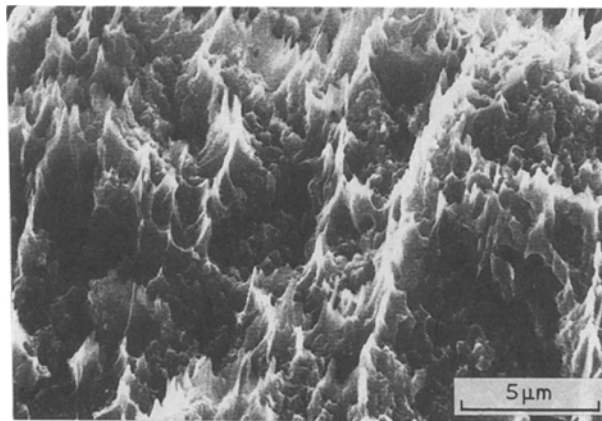

Figure 1 Fracture morphology, creep crack growth component, 200° C.

lower temperature condition. The higher temperature results in a greater amount and a more defined failure mechanism of microsuperplasticity as would be expected.

Examination of the base of the fibre formation shows that many grains appear to contribute to the formation of the fibre as seen in Fig. 3. Closer examination reveals flow lines with each line delineating the edge of a single fibre. This is better appreciated when viewing Fig. 4. This figure results from the formation of a bundle of fibres in a secondary crack that did not separate completely and later buckled when the load was relaxed. It can be seen that a number of fibres occur together with each fibre being approximately 0.1 μm diameter. Thus it becomes apparent that what is taken to be a single fibre in Fig. 2 is in most cases a bundle of fibres. The diameter of the individual fibres in Fig. 4 is approximately equal to half the grain size of the material. This magnitude of fibre size in relation to grain size means that the mechanism of grain-boundary sliding is not contributing in a major way to superplastic deformation in this material. Rather, general material flow is responsible for the ductility mechanism. This type of single-crystal flow mechanism has been found to occur before, during superplastic behaviour [10].

In the 400° C tested creep crack growth components, specific areas exhibit fracture behaviour quite different from that which normally occurs. One
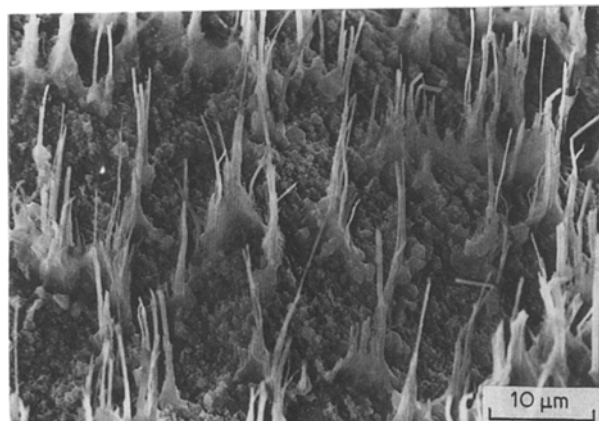

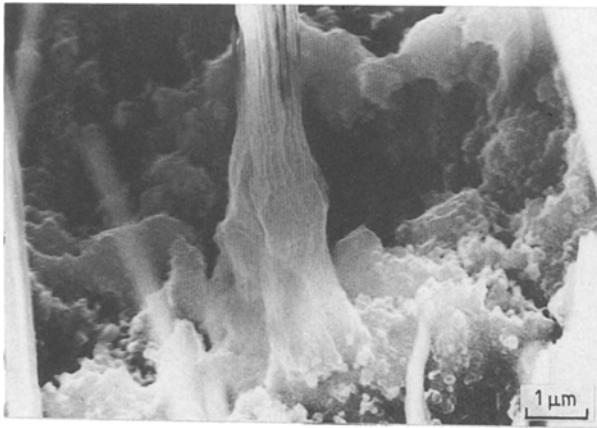Figure 2 Fracture morphology, creep crack growth component, 400° C.

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Figure 3 Base of fibre bundle, creep crack growth component, 400° C.

Figure 5 Formation process of fibres, creep crack growth component, 400° C.

of these areas is shown in Fig. 5. This picture is taken looking down into a secondary crack branch that formed off the primary crack surface. The process of fibre formation has been captured in this figure. The figure shows an area behind the secondary crack tip exhibiting various stages of microsuperplasticity. This means that the material provides resistance to crack propagation by refusing to fracture completely. The crack tip leaves local regions of unfractured material behind which act to retard the forward progress of the crack.

Fig. 6 shows the transition area between the fatigue precrack and the creep crack growth region. In this figure it can be seen that microsuperplastic fibre formation occurs initially at the prior crack tip itself. The general stretching as the crack tip opens becomes microsuperplastic formation. Thus the microsuperplastic mechanism can be active at the crack tip as well as behind it.

Another area that is not so typical is shown in Fig. 7. This region occurred when the specimen was reloaded after buckling had taken place. It can be seen that nearly 100% fibre formation occurs with a very small amount of intergranular cell formation. This indicates that strain rate is extremely important in the promotion of microsuperplasticity.

3.2. Heat-treatment behaviour and void formation
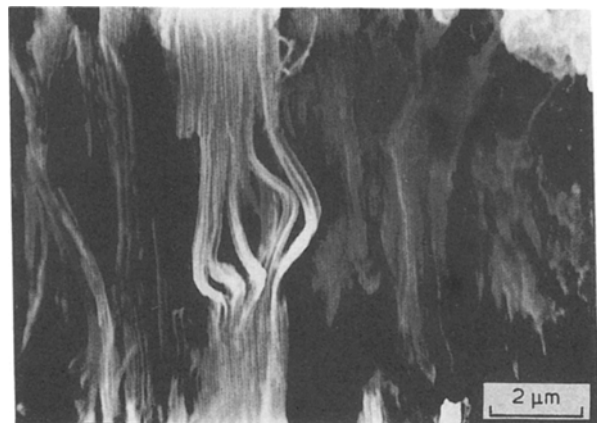By heating samples of AL-9021 in a vacuum environ-

ment, identification of the gas constituents evolving from the material can be determined. Fig. 8 shows the chromatograph gas analysis from material that was induction heated under vacuum for a period of approximately 20 sec. As can be seen, the quantity of oxygen is many times greater than either the hydrogen or the nitrogen content. This quantity is conservative, as some of the oxygen will remain trapped within voids in the material, and some oxygen will undergo a chemical reaction at the high temperature with the aluminium. If the material is heated for a long period of time using induction heating, or is heated in a furnace, then the oxygen that is evolved has ample opportunity to chemically combine with the aluminium and form aluminium oxide. This slow or incorrect heating technique will yield a gas analysis that indicates primarily nitrogen or hydrogen evolved out of the material. The quantity of hydrogen evolved will be almost identical to that measured using the correct technique and is many times lower in magnitude than that which occurs from the oxygen evolution.

Owing to the high reactivity of oxygen and aluminium at elevated temperatures, there is a very small but specific time duration of heating that exists in order to allow determination of the liberated free oxygen. Further description on the experimental details of the oxygen evolution experiments can be found in [6].

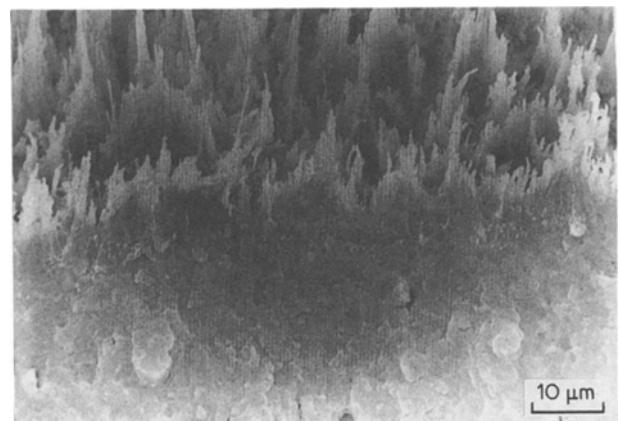Components heat treated at elevated temperatures form internal voids due to the evolution of free oxygen

Figure 4 Buckled fibre bundle, creep crack growth component, 400° C.

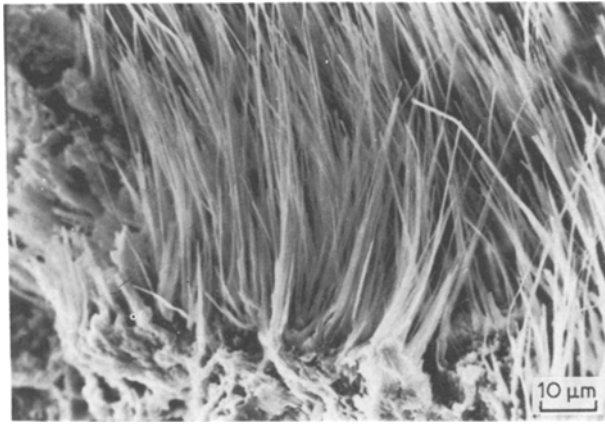Figure 6 Transition fatigue/creep crack growth, 400° C.

Figure 7 Fracture morphology near buckled region, creep crack growth component, 400° C.
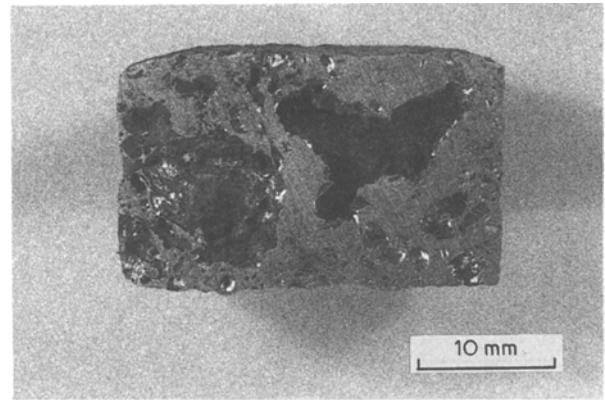

Figure 9 Section through heat-treated component, 620° C.

out of the material. The extent of the large void formation can be seen in a component section that was heat treated at 620° C as shown in Fig. 9. In addition to the large void formation, many smaller voids and internal cracking also form. The cracks and void size geometry are dependent upon the duration of heating. If the component is heated quickly, via instant immersion into a salt bath, then large cracks form, Fig. 10. However, if the component is heated slowly, as in a furnace, then small microcracks form, Fig. 11. These microcracks link together to provide passageways leading to void initiation as also shown in Fig. 11. The formation of voids results from the build-up of oxygen pressure within the material and subsequent fracturing or high-temperature deformation of the material. This type of void formation mechanism in aluminium alloys has likely been wrongly attributed to hydrogen [11]. Material which was heat treated at a temperature of 620° C by salt bath immersion was sectioned and examined. Large fracture voids are found as seen in Fig. 12. This figure also illustrates another mechanism that is occurring, termed stress striations [4]. If the area within this void is viewed, a coarse microsuperplastic behaviour is found. This is shown in Fig. 13. If the released oxygen is generated quickly at an elevated temperature, then areas within the material experience a sudden build-up of pressure. The material then deforms at some specific strain rate during the formation of a void. Fig. 13 suggests that the high tem-
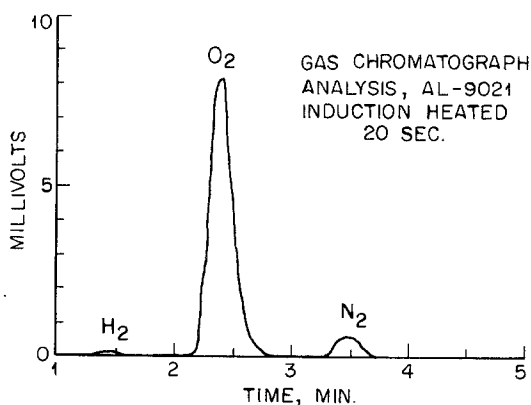
perature and strain rate conditions produced by the evolution of oxygen from the material due to elevated heating, are favourable to the formation of micro-superplasticity.

3.3. Tensile strain rate behaviour

Results of testing over the wide range of strain rates showed that AL-9021 just satisfied the definition of superplastic behaviour at a specific strain rate condition around $10 \, mm^{-1} \, min^{-1}$ which yielded a total elongation of 114%. The resulting fracture morphology associated with this macroscopic condition was mainly that of microvoid coalescence with some intergranular fracture.

The mechanism of microsuperplasticity was not found to occur until the strain rate was reduced to a very low value being approximately $5 \times 10^{-5} \, mm^{-1} \, min^{-1}$. Associated with this condition was a total elongation of only 2% which is in contrast to the previous superplastic condition. The fracture morphology for this slow strain rate condition is shown in Fig. 14. The start of microsuperplastic fibre formation has just begun. As is typical, the microsuperplastic behaviour can be seen to surround areas of intergranular fracture. The overall appearance is very similar to that found in the creep crack growth components tested at 200° C.

Thus the two conditions, microsuperplasticity and superplasticity are not synonymous; each is governed by its own unique parameters over the realm in which it operates.
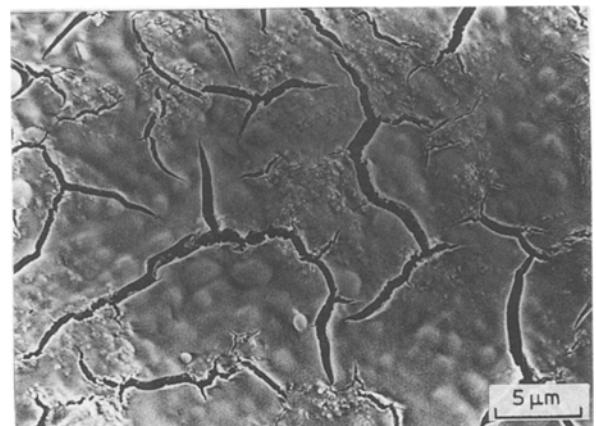

Figure 8 Chromatograph analysis of gas evolved from AL-9021, induction heated for 20 sec.

Figure 10 Internal cracking, fast heated component, 595° C.
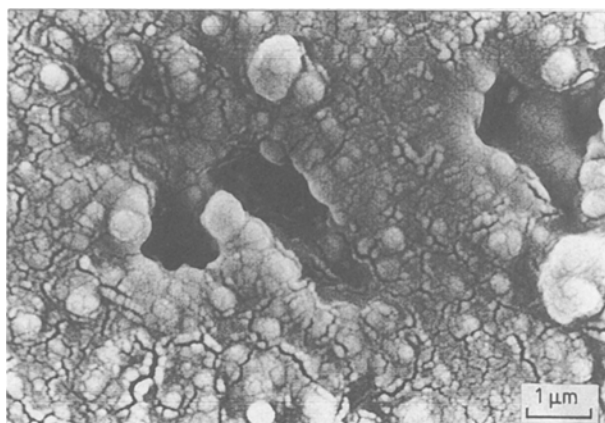
Figure 11 Microcracking and void formation, slow heated component, 595° C.

Figure 13 Microsuperplastic fibre formation within void, fast heated component, 620° C.

3.4. Standard creep specimen behaviour

The creep behaviour of AL-9021 material shows enormous creep resistance, approximately 300% improvement as compared to 2024 aluminium. The creep failure mechanism is not the usual type where damage occurs first in the middle of the specimen with the formation of an internal crack propagating outwards. Rather in this material the crack forms on the outside surface and propagates inward. It is interesting to note that fracture actually begins by a mechanism of intergranular failure. This, in turn, is followed fairly quickly by the establishment of the microsuperplastic mechanism as shown in Fig. 15. Thus even regular creep failure of an initially smooth AL-9021 specimen is governed by surface crack initiation and subsequent propagation.

Once the crack is initiated, the fracture behaviour of this type of specimen quickly establishes itself in the form of typical microsuperplasticity, Fig. 16. The microsuperplastic phenomena occurs in the same manner as in the previous crack propagation tests except that the microsuperplastic fibre formations on the whole are not as dense, and larger areas of intergranular fracture take place. This is likely a result of a lower stress intensity factor and a lower value of strain rate due to the geometry effect occurring in the standard type creep specimens.

4. The microsuperplastic mechanism
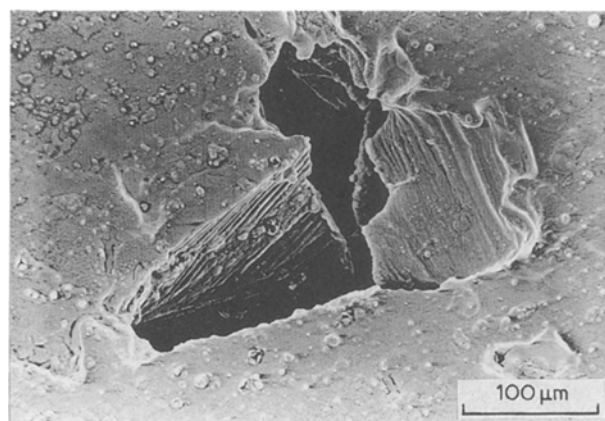
The results from the four experimental programmes

point out the various conditions that lead to the formation of microsuperplasticity. Some of the controlling parameters have been identified.

Low-temperature operation with AL-9021 is not detrimental as the oxygen is stable within the material. However, at higher temperatures, it has a strong tendency to be rejected by the structure. It was seen that the oxygen within the material plays the important role. The release of oxygen creates microcracks as well as voids. Thus, rather than forming aluminium oxides, the oxygen acts to induce fracture of the material when it is exposed to elevated temperatures. The intergranular fracture is attributed to oxygen release or planes of weakness at grain boundaries or subgrain boundaries due to the presence of loosely bound oxygen. The microcracking means that free surfaces are created within the material. These free surfaces promote local microscopic unfailed islands of plane stress. These areas then possess extra ductility that helps in the formation of microsuperplasticity.

It is apparent that the microsuperplastic behaviour in this material is extremely sensitive to strain rate, and once a low macroscopic value of strain rate occurs, this mechanism is promoted. The macroscopic strain rate of $5 \times 10^{-5} \, \text{mm}^{-1} \, \text{min}^{-1}$ becomes a value of $10 \, \text{mm}^{-1} \, \text{min}^{-1}$ when a local microscopic gauge length of $0.1 \, \mu\text{m}$ is used. Thus it appears that the superplastic behaviour of the material as a whole is identical for both macroscopic and microscopic size.

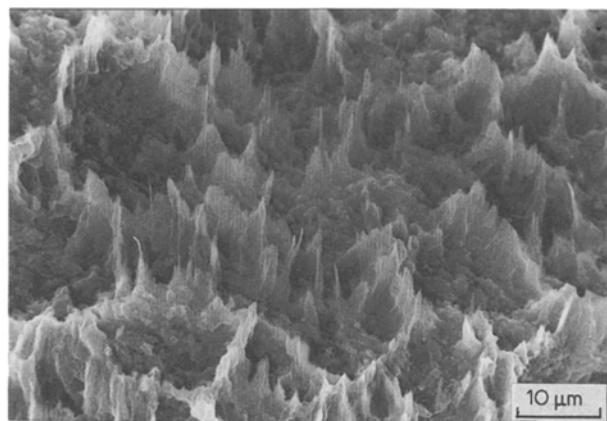As the temperature is increased the slow strain rate

Figure 12 Void formation and fracture, fast heated component, 620° C.

Figure 14 Fracture morphology, slow strain rate component, 400°C.

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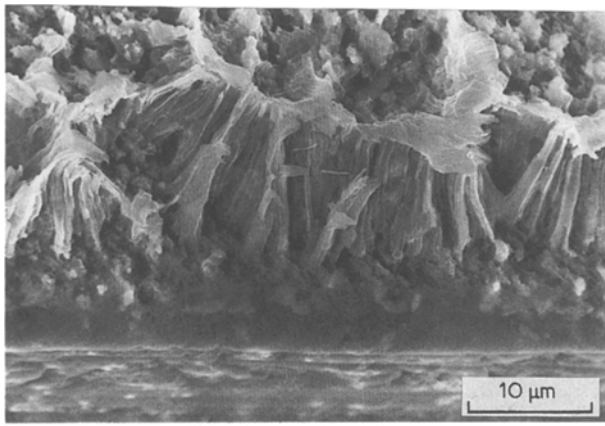*Figure 15* Crack initiation area, standard creep component, 400° C.

Figure 16 Fracture morphology, standard creep component, 400° C.

conditions required to promote microsuperplasticity are relaxed as found from the heat-treatment results. These results also indicate that it is not necessary for the material to be exposed to ambient air for the formation of microsuperplasticity because the mechanism occurred internally.

A question that cannot be answered at this time is whether the ambient environment promotes the formation of the superplastic behaviour or whether its promotion comes solely from within the material.

Because this same mechanism has been found in a rapidly solidified aluminium alloy [9], which is a totally different type of material, it becomes apparent that similarities in processing must hold the key. Both materials are exposed to oxygen in the atmosphere during processing. It is likely that other fine-grained aluminium PM materials will also be found to exhibit the same microsuperplastic mechanism, unless they are processed under inert conditions.

An energy dispersive X-ray analysis was conducted on both the superplastic fibres and the adjacent material that failed by intergranular fracture. No discernible difference was found. A more exhaustive study needs to be undertaken using electron diffraction and other techniques.

If the passive layer that formed on the surface of aluminium was a true chemical bond of aluminium oxide, then this phenomenon would not occur. Rather we are looking at a new type of reaction and we should re-examine our current theories of passivation, oxidation and general chemical reactions. Microsuperplasticity is one consequence of this mechanical proximity reaction.

5. Conclusions

1. The microsuperplastic behaviour is sensitive to both strain rate and temperature.

2. Internal oxygen appears to play a major role in the formation of microsuperplastic fibres.
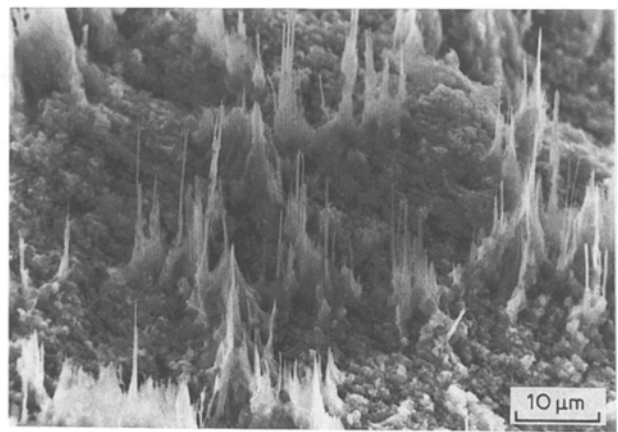
3. Generally, fibres are formed when local fracture surrounds an island of unfractured material; local fracture being caused by release of free oxygen or planes of weakness due to high oxygen content.

4. Microsuperplasticity can occur at the crack tip as well as behind the crack tip.

5. Fine-scale superplastic flow in this material is not a result of grain-boundary sliding but rather from general flow of the material.

6. Fracture mechanisms are completely different between superplasticity and microsuperplasticity.

7. Superplasticity, requires a high macroscopic strain rate while microsuperplasticity requires a low macroscopic strain rate. The superplastic flow of the material appears to be the governed by the same local value of strain rate.

Acknowledgements

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