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SUPERPLASTIC BEHAVIOUR IN DYNAMICALLY RECRYSTALLISING ALUMINIUM ALLOYS

Abstract

Aluminium alloys can be thermomechanically processed to develop the grain fine microstructures required for superplasticity by either static recrystallisation prior to superplastic forming (SPF) or by dynamic recrystallisation during the early stages of deformation. The present work has examined and compared the superplastic behaviour and the microstructural evolution in Al-Li alloys (8090) sheet material processed by the second route for a wide range of temperatures and strain-rates. It was observed that the material showed a high potential for superplastic flow. Although significant superplasticity was observed at temperature of 400°C and below. The reasons for the high resistance of the material to strain localisation are discussed. It was noted that ductility enhancement could also be achieved by the control of the strain-rate path. A rapid pre-strain improved significantly the subsequent superplastic elongation to failure at optimum strain rate. Further enhancement has been obtained by pre-straining at constant velocity following by deformation to failure at lower constant velocity. The microstructure changes prior or during deformation were also examined.

***Keywords**: Superplasticity, Dynamic recrystallisation, Al-Li alloys.*

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Résumé

Un des pré-requis du comportement superplastique des matériaux est lié à une structure à grains fins et équiaxes. Cette structure peut être obtenue par recrystallisation statique (avant la déformation) ou par recristallisation continue dite dynamique (durant les premiers stades de déformation). Dans ce travail, nous avons examiné et comparé le comportement superplastique ainsi que l'évolution de la microstructure dans l'alliage Al-Li (8090) qui a été élaboré par le second procédé pour un large domaine de températures et de vitesses de déformation.

Le matériau étudié présente une superplasticité élevée, bien que cette dernière ait été observée à une température de 400°C et en dessous. Les raisons de cette grande résistance du matériau à la localisation de la déformation sont discutées. Il a été également remarqué que l'amélioration de la ductilité peut être obtenue par un contrôle du domaine de vitesse de déformation. Une pré-déformation rapide améliore suffisamment l'élongation superplastique à la rupture pour une vitesse de déformation optimale. D'autres améliorations peuvent être obtenues par une pré-déformation à une vitesse de déformation constante suivie d'une déformation à une vitesse inférieure. Les variations de la microstructure avant et durant la déformation sont aussi examinées.

***Mots clés**: Superplasticité, Recristallisation dynamique, Alliages* Al-Li.

ملخص

الشرط الاساسى للمادة حتى تكون ما فوق لدنة هو التبلور فى حبيبات صغيرة . يمكن الحصول عليه من خلال طريقتين, ألتبلور الستاتيكي أي قبل التشوه ما فوق اللدن, او التشوه الديناميكي أى الدي يحصل عليه خلال ألمراحل ألاولي للتشوه.

ألعمل الدي قمنا به هو دراسة و مقارنة التشوه و التبلور خلال التشوه ما فوق اللدن لالسبيكة الاليمنيوم من النوع Al-Li (8090) الدي حظر علي الطريقة الثانية من اجل مجال واسع في درجة الحرارة و سرعة التشوه

لقد لحض أن المادة المدروسة لها قبيلية كبيرة للتشوه ما فوق اللدن حتي درجة حرارة واطئة أي°400 و اقل. تشرح الاسباب في هده الدراسة . لقد تبين ايضا ان تحسين التشوه ما فوق اللدن ينكن الحصول عليه اما علي اجراء تشوه ابتدائى سريع او تشوه عن طريق سرعتين متتاليتين لقد درسنا ايضا التغير في تبلور المادة قبل و خلال التشوه.

***الكلمات المفتاحية** : المرونة الفائقة، التبلور الديناميكي، سبائك Al-Li*

Aluminium-lithium alloys are characterised by low density and high stiffness [1]. In addition various alloying elements are also present in order to improve mechanical properties such as ductility. The only agreement made so far is that superplastic deformation requires a fine stable grain size. Although zirconium is a minor alloy addition but it has been established that it causes a marked reduction in grain size [2]. In the United Kingdom, the development of Al-Li alloys has been concentrated on the Al-Li-Cu-Mg-Zr system [3-7]. The development has led to the designation of two alloys of nominal wt % compositions: 8090A (Al-2.5-Li-1.2Cu-0.7Mg-0.1Zr), 8090B (Al-2.6Li-1.9Cu-0.8Mg-0.1Zr). These alloys have many potential applications in aerospace industry, particularly the 8090A alloy, sometimes referred to as Lital A. It has been shown by Grimes [8] that these alloys in sheet form can be processed in order to develop superplastic behaviour. Following thermomechanical processing, superplastic deformation required microstructure, which can be achieved by either static recrystallisation or dynamic recrystallisation during the early stages of deformation.

Previous studies [9] identified the optimum conditions for superplastic flow for Al-Li 8090 processed by both routes and showed the statically material had a lower potential for SP behaviour, i.e. lower tensile ductility, higher flow stress and slower optimum deformation rates. It was shown, for both materials that SP flow could be markedly enhanced by control of the strain rate path. While there is a reasonable understanding of the mechanism of microstructural refinement by static recrystallisation, the way in which superplastic microstructure evolves by dynamic or continuous recrystallisation is less clear. Many attempts [10-14] based on observations have been made to explain the mechanism involved in materials which indergo continuous recrystallisation. It is believed that there is no reason to assume that there is a unique mechanism [10] during superplastic flow. The grain boundary sliding is maintained by an accommodation process which may involve grain boundary migration (GBM), or grain boundary rotation (GBR), or diffusion and /or dislocation process.

The aim of this work is to concentrate on dynamically recrystallising material and to investigate mechanical behaviour over a wide temperature range than was previously examined. The purpose of the work was to determine whether significant SP behaviour could be obtained in conventially processed materials at temperature appreciably below the previous determined optimum temperature of 530°C, without substantially raising the flow stress. The advantages of a lower forming temperature would be reduced grain growth and suppression of lithium loss from the surface of the alloy. The effects of a rapid pre-straining and the pre-annealing treatments on superplastic behaviour have also been investigated. In addition microstructural changes have been examined prior and during superplastic deformation.

EXPERIMENTAL PROCEDURE

The alloy examined was Lital A (8090) of nominal composition Al-2.5Li-1.2Cu-0.6Mg-0.1Zr, produced by Alcan. The material was obtained in the form of sheet of 3mm thickness. Tests were carried out in a three-zone split furnace, attached to the crosshead of an Instron machine interfaced with a computer. Tensile specimens of 10 mm gauge length and 5 mm gauge width were machined from the sheet provided, were strained to failure or to a pre-determined elongation at constant strain rates in the range of 10^{-3}-10^{-4} s^{-1} for temperatures between 300°C to 530°C.

Determination of m values (m = log σ/logε) was carried out using cross-head velocity cycling or jump strain rate testing, while in the strain hardening exponent, n value (n=logσ/logε), was calculated as function of strain from the stress-strain curves.

Microstructural changes were investigated using transmission electron microscopy (TEM). Thin foil were prepared from 3 mm discs removed from bulk material annealed to temperatures of 500°C to 530°C for 30 minutes, 60 minutes, 90 minutes. Samples were deformed to a nominal strain of 50% (0.4) and 100% (0.69) strains considerably less than the fracture strain, which appeared to be essentially convenient for microstructure studies. Tensile testing was then stopped, and specimens were then removed from the grips and quenched in cold water. TEM specimens were prepared from discs cut from the deformed region of specimens, in a twin jet electropolishing machine using 33% HNO_3 in methanol at –30°C and 18V and examined in a Philips 400T microscope.

RESULTS AND DISCUSSION

Strain to Failure

In order to examine the superplastic behaviour, specimens were strained to failure at temperatures from 300°C to 530°C (527-603K), at constant strain rates within the range 10^{-3}-10^{-4} s^{-1}. The tensile elongations to failure plotted against temperature for three strain rates are shown in figure 1. It is clear that superplastic behaviour is apparent at 300°C, but become appreciable, > 500 % elongation; at temperatures of 400°C and above. At 500°C and 530°C, tensile elongations > 900% were recorded for a strain rate of 5 x 10^{-4} s^{-1}. Stress-strain data for a strain rate of 2 x 10^{-4} s^{-1} for the range of temperatures investigated are seen in figure 2. It can be seen that the general level of flow stress decreases as temperature is raised. At temperature below 400°C, flow stress increases sharply.

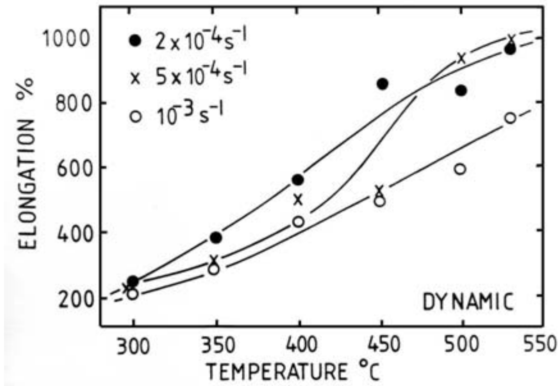

Figure 1: Elongation to failure versus temperature at various strain-rates.

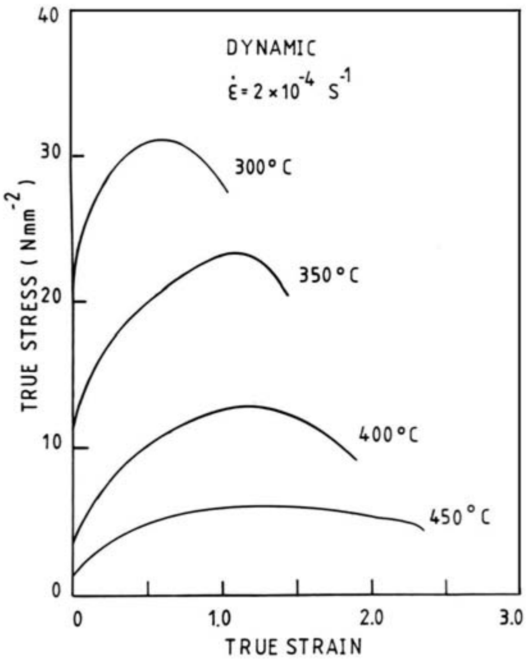

Figure 2 : Stress-strain curves at a fixed strain-rate of $2x10^{-4}s^{-1}$

This is consistent with the measurement of m and n values, which are plotted as a function of strain for temperatures of 530°C, 500°C, 400°C (fig.3).

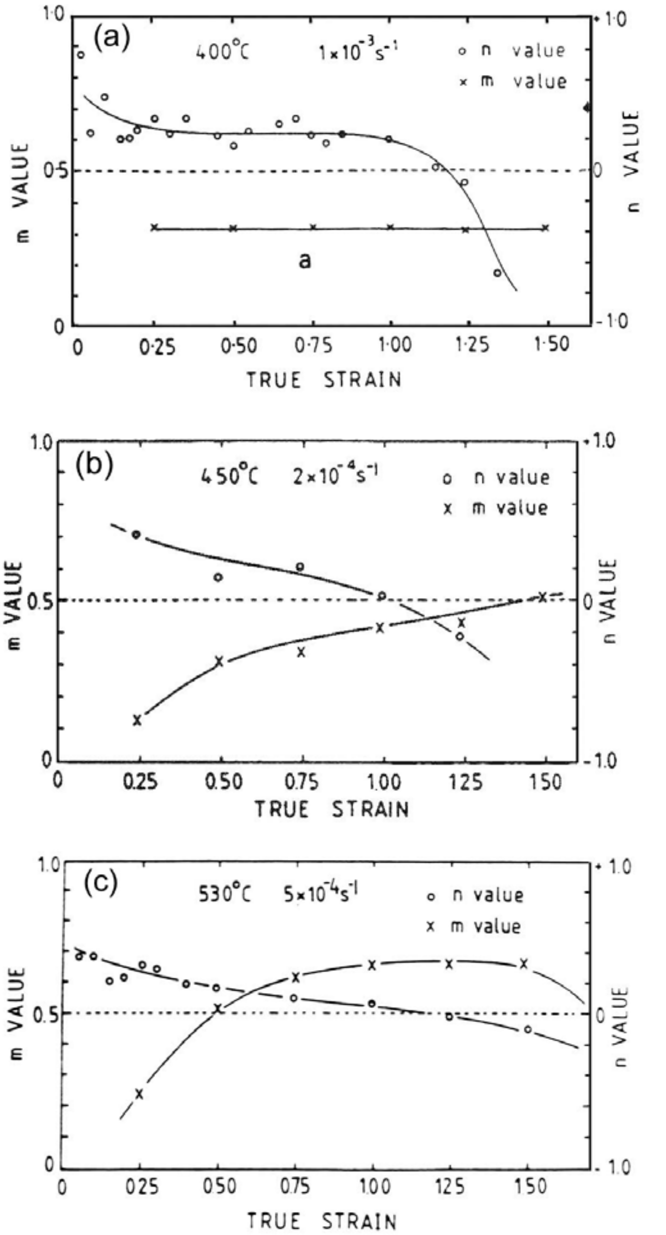

Figure 3: Variation of m and n at various temperatures and strain rates.

It can be seen at 530°C at the beginning of the deformation the value of m is too low, < 0.3, to maintain substantially tensile strain stability, but the n value is high, ≈ 0.4. Hence, without strain hardening the material would show early necking leading to a low tensile elongation. Similar behaviour is apparent at 450°C, but m increases more slowly. However, the high value of n in the early stages of deformation has inhibited mechanical instability until m becomes dominant leading to a substantial tensile strain to failure of 2.26 (860%). At 400°C, for the relatively high strain rate of 10^{-3} remains essentially constant with strain at ≈ 0.3, while n remains high before falling to zero at ≈ 1.15 (220%). Hence, tensile stability is attributed to the influence of both factors. It is interesting to note that while the value of m may be regarded as the most important characteristic for superplastic flow [15,16], it is sufficiently high to permit a further tensile elongation of 200%, leading to an elongation to failure of 400%. Similar experimental observations [5,17, 18] on tensile stability and necking in superplastic materials which consider both the strain rate sensitivity (m value) and the strain hardening coefficient (n value) have been made. It was show by Ash and Hamilton [5], and Ghosh and Hamilton [17] that strain hardening could make a significant contribution to the tensile stability of alloys as superplastic microstructure evolves by dynamic/strain enhanced recrystallisation.

EFFECT OF PRE-STRAINING ON SUPERPLASTIC DEFORMATION

a) Ductility enhancement

The high superplastic elongation to failure observed in the dynamically recrystallised material can be related to its high strain rate parameter (m value), which enables the material to resist necking. For Al-Li alloy at 530°C, it has been demonstrated [9] that the superplastic ductility can be further enhanced by the application of rapid pre-straining, following by a deformation at the optimum constant strain rate. It was also shown [9] that rapid pre-strain followed by constant velocity deformation led to a substantial enhancement in tensile ductility, i.e. from 900% elongation to 1800% as illustrated in figure 4. TEM observations showed that the rapid pre-strain rate $8.3 \times 10^{-3}\ s^{-1}$ gave a fully recrystallised (dislocation free) microstructure after a relatively small strain (0.4 corresponding to 50% elongation), whereas for deformation at the optimum constant strain rate of $5 \times 10^{-4}\ s^{-1}$ the SP microstructure was still evolving as shown in figure 5 (increasing m after a strain of 0.75, ≥ 100%, figure 5c).

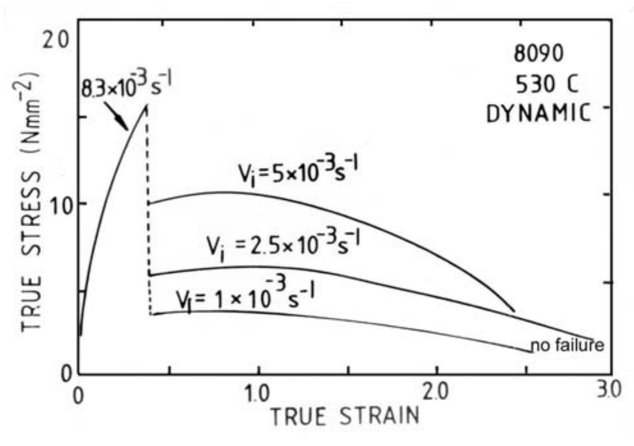

Figure 4: Effect of rapid pre-strain on strain to failure at constant velocity.

The effects of rapid pre-straining has been reported for several alloys in Pb-Sn alloys [19], and Supral 220 [6] and by Ash and Hamilton [5], and Ghosh and Hamilton [17] and Ghosh and Gandhi [7].

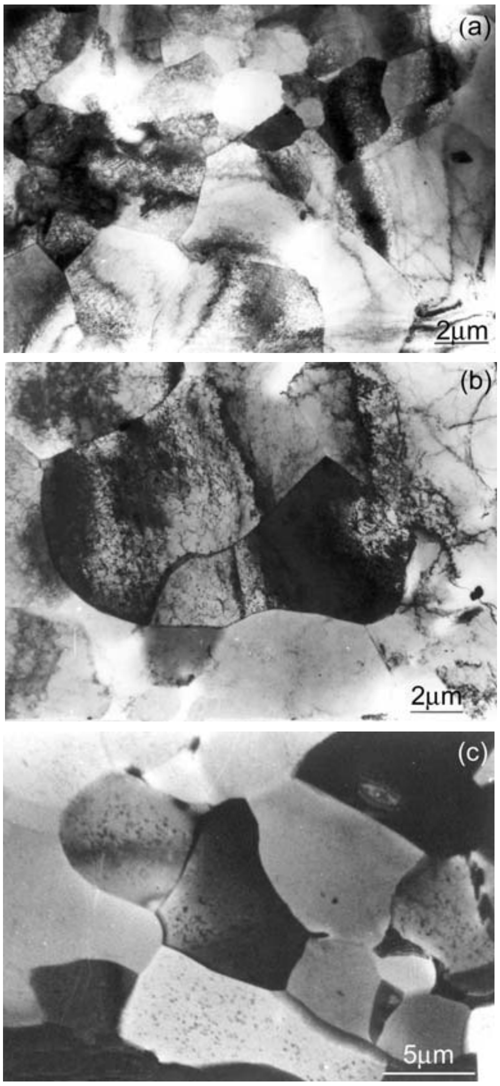

Figure 5: TEM micrographs showing the effect of pre-strain.
a) as-received, **b)** $\varepsilon = 0.4$ [50%], $\dot{\varepsilon} = 8.3\times10^{-3}\ s^{-1}$, **c)** $\varepsilon = 0.75$ [100%], $\dot{\varepsilon} = 5\times10^{-4}\ s^{-1}$.

The combined effects of strain hardening and strain rate sensitivity on tensile ductility have been considered by Caceras and Wilkinson [20] and Hamilton et al. [5] using an instability parameter, I, derived from earlier work by Hart [21] and Nichols [22] where $I = (1-\gamma-m)/m$. The greatest mechanical stability is associated with $I \leq 0$. Hence, substitution of measured values of m and γ for various rapid pre-strain rates in this relationship can enable the extent to which specimens can be deformed prior to the onset of excessive necking, to be predicted. Using this approach, it was apparent that at the temperature of interest, 450°C, it would not be possible to apply rapid strain rates much in excess of $10^{-3}\ s^{-1}$. Consequently, the effects of two pre-strain rates, $1 \times 10^{-3} s^{-1}$ and $2.5 \times 10^{-3}\ s^{-1}$, applied for a pre-strain of 0.4 (50% elongation) were examined. While the higher rate led to some enhancement of tensile ductility (≥ 600% el.), on subsequent deformation at $5 \times 10^{-4}\ s^{-1}$, it also caused an increase in the maximum flow stress from 11 $N.mm^{-2}$ to 13 $N.mm^{-2}$. However, the lower pre-strain rate led to enhancement of tensile ductility to a similar extent, but decreased the flow stress to ≈ 9 $N.mm^{-2}$ (fig. 6).

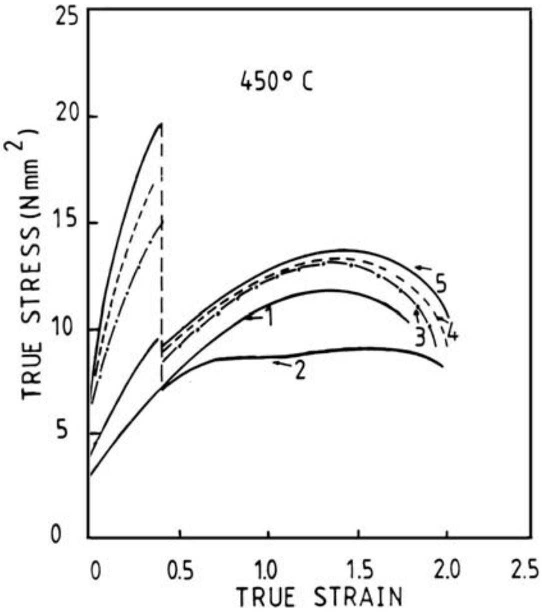

Figure 6: Effect of a rapid pre-strain on stress versus strain at $5\times10^{-4}\ s^{-1}$ and 450°C: (1) $\dot{\varepsilon} = 0$; (2) $\dot{\varepsilon} = 1\times10^{-3}\ s^{-1}$; (3) $\dot{\varepsilon} = 1.5\times10^{-3}\ s^{-1}$, (4) $\dot{\varepsilon} = 2.5\times10^{-3}\ s^{-1}$, (5) $\dot{\varepsilon} = 8.3\times10^{-3}\ s^{-1}$.

The effect of higher pre-strain of 0.7 (100%) for the pre-strain rate of $1 \times 10^{-3}\ s^{-1}$ shown in figure 6 led to a further enhancement of strain to failure and a reduction in flow stress to ≈ 8 $N.mm^{-2}$. Although these flow stress are higher than the corresponding value of ≈ 5 $N.mm^{-2}$ at 530°C without pre-straining, there would be beneficial effects of SPF at the lower temperature of 450°C, as outlined in the introduction. Alternatively, the application of a rapid pre-strain rate at 450°C prior to deformation at slower strain rate of $2 \times 10^{-4}\ s^{-1}$, would lead to an even lower flow stress than the maximum value of 6 $N.mm^{-2}$ as seen in figure 6, for no pre-strain. The considerable SP strains attainable at 450°C (≥ 800% el.) and the relatively low flow stresses involved should enable the effective use of back pressure to suppress cavitation, which is characteristic of SP flow in aluminium alloys, including Al-Li [23].

Piu and Huang [24] have recently outlined processing routes for the development of SP behaviour in an Al-Li alloy at relatively low temperatures, ≤ 450°C. While the material showed a reasonable SP response at 350°C, flow stresses were (≥ 20 $N.mm^{-2}$). However, at temperatures > 450°C, where the flow stress would be expected to be lower, the alloy showed a poor SP behaviour.

b) Structural changes during the early stages of superplastic deformation

It is worth noting there are few investigations dealing with the effect of pre-straining on microstructural evolution [25]. Investigations to date have mainly been studies on the effect of pre-strains on superplastic ductility and flow stress [5, 7, 26, 27].

In the present work, the effect of various pre-strains and pre-strain rates on the unrecrystallised alloy showed that superplastic microstructures were developed gradually by in-situ recrystallisation during the early stages of SP deformation. It was observed that under optimum deformation conditions, i.e. 530°C and 5 x 10^{-4} s^{-1}, the recrystallisation was not completed until 100% elongation (~0.7 true strain). Ricks et al [25] reported similar microstructural behaviour in Al-Li alloys (8090). Dislocations were evident within the structure at 50% elongation for the above deformation conditions. However, it has been observed in other alloys i.e. Al-Cu-Zr alloy, i.e. 50% superplastic elongation (0.4 true strain) is sufficient for dynamic recrystallisation to occur [25]. This may be the case for the investigated alloy if a rapid strain-rate is applied. A relatively uniform grain structure almost free from dislocations was developed at 50 % elongation at a strain rate of 8. 3 x 10^{-3} s^{-1}, as seen in figure 5. Matsuki et al [28] reported similar behaviour for a Al-Zn-Mg-Zr alloy where recrystallisation occurs during deformation at a rapid strain rate.

The continuous or in-situ recrystallisation, which occurs during the early stages of SP deformation, operates through a gradual conversion of subgrain boundaries into high angle boundaries. This can be seen in figure 5c, where grain boundary misorientation tends to increase with strain under optimum deformation conditions. Similar observations have been reported on others alloys such as Al-Zn-Mn-Zr alloys [29] and lately in Al-Li alloys [30, 31].

The mechanism behind the increase in boundary misorientation during the early stages of superplastic deformation (SPD) could involve sub-grain coalescence and/or strain-induced grain boundary rotation. The former has been suggested to operate during the initial stages of superplastic deformation of Al-Cu-Zr alloy [7], since examination of the microstructures showed that the sub-grain size increased with increasing strain. Whereas in Al-Li alloys [14], dislocations sliding in grain is suggested to be the main mechanism before the high angle boundary.

In the present study, it is suggested that the former mechanism is likely to play a role in the initial stages of recovery during pre-heat treatment, i.e. prior to superplastic deformation, where a recovered structure was develop [32]. However, the increase in the grainboundary misorientation may be associated with the latter i.e. strain induced boundary rotation, although subgrain coalescence may be also involved as the subgrain size tend to increase with increased strain. The increase in boundary misorientation can be explained as follows. During deformation the dislocation density in the sub-grains increases and matrix dislocations migrate to sub-grain boundaries. This is well illustrated in figure 5c, which shows dislocation interaction at the sub-grain boundaries, as well as in sub-grains, for a specimen deformed to 50% elongation under optimum conditions. The process continues until the misorientation builds up to a level (100% el.), sufficient to allow grain boundary sliding to accur. This change in the deformation mechanism during the early stages is consistent with the variation of the strain hardening and strain-rate sensitivity parameters, i. e. n and m values, with strains (fig.3a). It is clearly seen that the strain hardening is significant during the first stages of superplastic deformation for optimum deformation conditions. It is worth noting that dislocation activity within α-Al grains has also been reported by Ricks [25] and by Liu Quing et al [14] for Al-Li alloys, i.e. 8090 and Matsuki et al [32] for Al-Cu alloys, and by Hales et al [10] in Al-Mg alloy, during the early stages of deformation.

It appears therefore that the superplastic deformation (SPD) of dynamically recrystallising alloys involves conventional deformation mechanism i.e. dislocation/ diffusion creep or slip during the early stages of deformation in order to develop the microstructure required for SPD (i.e. high angle boundary).

EFFECTS OF PRE-ANNEALING TREATMENT

It is well established that superplasticity is observed in alloys that develop a fine grain structure. The present alloy has been thermomechanically processed to develop a micrograin structure during the early stages of deformation, i.e. dynamic recrystallisation. TEM examination of the as-received material revealed a high dislocation density, the presence of dislocation with the structure persists after static annealing at 500°C for 90 minutes and 530°C for 30 minutes [9]. However, a long time at a high temperature i.e. 530°C for 60 minutes, results in a well-defined sub-grain structure, substantially free from dislocations. Similar behaviour was observed by Liu Quing [14]. Thus the initial structure has been replaced by a polygonised structure. On subsequent deformation, under optimum conditions determined for the as-received material [9] (530°C; 5 x 10^{-4} s^{-1}). It was observed apart from small strains, the m values measured as a function of strains were lower than the m values for the as-received material.

As a consequence, the elongations to failure were reduced and the flow stress levels were increased on pre-annealing as seen in figure 7. The observations are essentially consistent with the earlier study [9], with showed that strain enhanced/dynamic recrystallisation resulted in superior superplasticity that associated with the static recrystallisation.

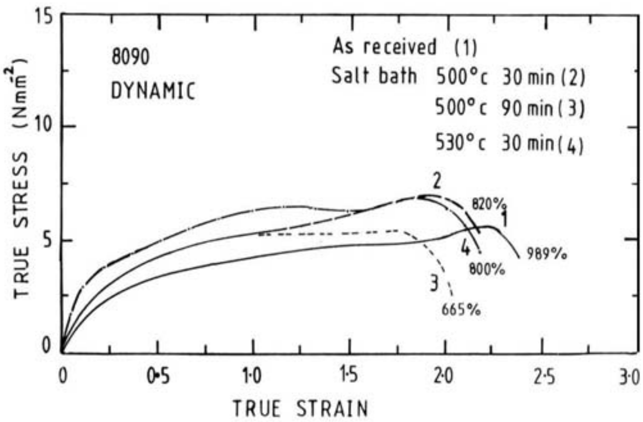

Figure 7: Effect of pre-annealing on stress-strain curves and tensile ductility at optimum strain-rate &= 5x10^{-4} s^{-1}.

CONCLUSION

1/ The high superplastic elongation to failure observed at relatively low temperatures in dynamically recrystallising alloy, was probably due to the relatively stable sub-structure resulting from the pinning action precipitates present in the alloy.
2/ The strain to failure could be enhanced by the application of a rapid pre-strain followed by deformation at lower constant strain rates. Further enhancement was achieved when the rapid pre-strain was followed by deformation under constant velocity conditions.
3/ Both strain-rate sensitivity and strain hardening parameters, i.e. m and n respectively, vary with deformation contributed to the high superplastic flow observed in the material.
4/ The microstructure required for superplastic deformation can be developed by either the application of a relatively high strain (ε = 0. 7 $\approx$ 100%) under optimum conditioned, or at a pre-straining at rates greater than the optimum. Although, long annealing treatments has even led to a well-developed structure.
5/ It has also been observed that the unrecrystallised material showed a resistance to static recrystallisation.

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