*Materials Science and Engineering*, 38 (1979) 169 - 174



# Effect of Stress Reductions on the Creep Behaviour and Subgrain Size in Aluminum Deformed at 573 K

I. FERREIRA* and R. G. STANG

*Division of Metallurgical Engineering, University of Washington, Seattle, Wash. 98195 (U.S.A.)*

(Received August 31, 1978; in revised form October 14, 1978)

SUMMARY

The effect of stress reductions, during steady state creep deformation, on the strain–time and subgrain size behaviour in high purity aluminum was studied. Stress reductions during steady state creep at 573 K produce a reduced strain rate which slowly increases, approaching the steady state rate which would be obtained at the reduced stress. This change in strain rate is accompanied by an increase in subgrain size as measured by optical techniques. The subgrain size, after deformation at the reduced stress, approaches the steady state subgrain size at the reduced stress.

## 1. INTRODUCTION

The effect of stress $\sigma$ on the steady state strain rate $\dot{\epsilon}_S$ of well-annealed pure metal samples deformed under creep conditions at constant stress and temperature can be described by an equation of the form

$$\dot{\epsilon}_S|_T = A\sigma^n \quad (1)$$

where $A$ and $n$ are constants. For most pure metals $n \approx 5$ [1]. The microstructure of samples in this condition, deformed under creep conditions, has been observed to change significantly during the deformation process. Barrett *et al.* [2] have shown that load application causes a large increase in dislocation density. Later subgrains start to form in the primary region. At this point the dislocation density must be described by two quantities: the dislocation density in the subgrain wall, and the dislocation density that is not associated with subgrain boundaries and is sometimes called the free dislocation density. Barrett *et al.* [2] have shown that the free dislocation density decreases as subgrain formation occurs. The subgrain size and free dislocation density within subgrains have been studied as a function of stress [2 - 7], temperature [6, 7] and strain [2, 6, 8]. Generally, it has been observed for many materials that the average subgrain size and the free dislocation density (not associated with subgrains) remain constant during steady state creep.

Several investigators have shown that the average subgrain size $\lambda$ which develops during primary creep is related to the applied stress by a power law relation of the form

$$\lambda = B\sigma^{-m} \quad (2)$$

where $B$ and $m$ are constants [1, 9]. The value of $m$ is usually of the order of unity although other values have been reported [6, 10, 11]. Robinson and Sherby [12] have developed an empirical expression for the steady state strain rate which includes subgrain size as an important parameter. For creep deformation at constant temperature this expression has the form

$$\dot{\epsilon}_S = A\sigma^P\lambda^q \quad (3)$$

where $A$, $P$, and $q$ are constants. Young *et al.* [13] have shown that for high purity aluminum $P = 7$ and $q = 2$. These results are consistent with eqn. (1) when $m = 1$ in eqn. (2).

Sherby *et al.* [14] have shown that when a fine subgrain size is introduced by deformation at a high stress and a stress reduction is performed in the steady state region, a transient will result. The strain rate, in the transient period after the stress reduction,

*On leave from Instituto Energia Atomica, São Paulo, S.P., Brazil.

will be significantly lower than the expected steady state strain rate which would result for a well annealed sample deformed at the reduced stress, suggesting that the material has been hardened. The reduced strain rate gradually increases. Some workers [13 - 15] have implicitly assumed that this acceleration in strain rate is accompanied by an increase in subgrain size.

Although assumptions have been made concerning the transient period, very few microstructural observations have been made after stress changes. Mitra and McLean [16] reported that no change in subgrain size could be observed, but the samples were only strained 1% after stress reduction. Pontikis and Poirier [17], in a study of subgrain sizes after stress reductions in AgCl, reported that no subgrain growth occurred even in samples which were held at zero stress for as long as four days. Parker and Wilshire [18] reported that no change in subgrain size could be observed following stress reductions in copper samples which were deformed until steady state creep was obtained and then subjected to a stress drop.

The purpose of this paper is to present the results of a study of the effect of stress reductions, in the steady state region, on the strain rate and subgrain size in high purity aluminum.

## 2. EXPERIMENTAL

The specimens used in this study were fabricated from high purity aluminum (99.999%) purchased in ingot form. The material was reduced 40% in thickness and subjected to a recrystallization annealing treatment. This process was followed, until stock with a thickness of 1.3 mm (0.050 in) was obtained, because f.c.c. materials have been shown to develop a rolling texture when subjected to reductions in thickness greater than 40% [19]. Tensile samples with a gauge length of 3.81 cm (1.5 in) and a cross-section 1.3 mm (0.050 in) by 9.5 mm (0.375 in) were machined from this cold rolled strip. Machined specimens were subjected to an annealing treatment, carried out in air at 753 K before testing. The average grain size after the final annealing treatment was 0.5 mm.

Annealed specimens were tested in a creep machine equipped with an Andrade–Chalmers constant stress arm in air at stresses between 3.44 MPa (500 lbf $in^{-2}$) and 16.15 MPa (2340 lbf $in^{-2}$). The variation in sample temperature along the gauge length was ±0.5 K, and the temperature variation during a test was always < ±1.0 K. All tests described here were conducted at 573 K. Strain was measured by use of a shielded linear variable differential transformer (LVDT). With this equipment strains of the order of $5 \times 10^{-4}$ could be measured, and estimations to $5 \times 10^{-5}$ were possible. At any time the test could be interrupted, the furnace moved away from the specimen, and the sample water quenched to room temperature under load. The cooling time between 573 and 323 K was less than 2 min, which was considered sufficiently rapid to freeze the high temperature substructure to room temperature.

Stress reduction tests were conducted by deforming the samples at stresses of 15 and 6.23 MPa to a true strain of 0.16, which is well into the steady state region. At a strain of 0.16 the stress was reduced to 3.44 MPa by rapidly removing a portion of the load. At any point the specimen could be rapidly cooled.

Samples for microstructural observation were sheared from the gauge length of the deformed specimens. Three techniques were used to reveal the subgrain structure of the deformed samples. For the technique which utilized transmission electron microscopy (TEM) samples were prepared by use of the window technique. They were mechanically polished until a thickness of 0.2 mm was reached and then electropolished in an ethanol–perchloric acid solution at 243 K until perforation occurred. A small piece near the perforation was sliced off and examined by TEM.

Samples for optical microscopy were individually mounted in a holder and mechanically polished. The sample was removed and electropolished in an ethanol–perchloric acid solution. Two different techniques were used to reveal the microstructure. The first, described by Lacombe and Beaujard [20], required an etchant composed of $HNO_3$ (47 ml), HCl (50 ml), and 40% HF (3 ml) at 0 °C. Samples were etched by immersion for 40 s, and the subgrain structure was visible by use of incident light microscopy. The second technique, developed by Perryman [21], required electropolishing of the sample in a solution of $H_3PO_4$ (60 ml) and $H_2SO_4$ (40 ml) at 80 °C with a current density of 0.8 A $cm^{-2}$ at 18 VDC. The sample was then anodized in a solution of $CH_3OH$ (49 ml) and HF (2 ml) in water (49 ml). The anodizing treatment was performed at room temperature at 15 V for 2 min. Samples were observed in polarized light and the subgrain diameter determined by use of ASTM grain size specifications [22]. Both optical techniques revealed the same subgrain sizes. The average subgrain diameter and the 95% confidence limits, obtained by optical microscopy, are reported in the Section 3.

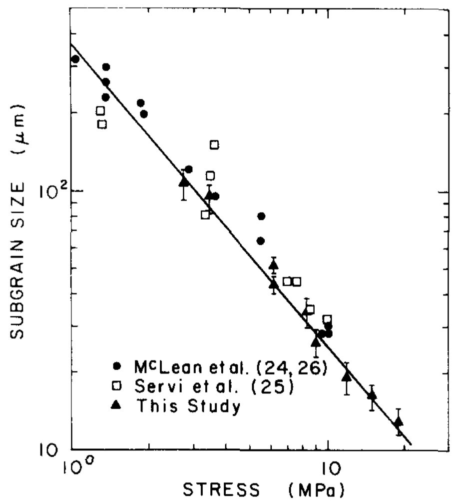


Fig. 2. The variation of subgrain size as a function of applied stress. The error bars shown for the data obtained in this study indicate 95% confidence limits.

3. RESULTS AND DISCUSSION

The effect of stress on the steady state strain rate at 573 K for the high purity aluminum used in this study is shown in Fig. 1. The results of Ahlquist and Nix [23] for 99.99% Al, obtained at the same temperature, are also shown, and excellent agreement is obtained. Although the strain rate at a given stress observed by Ahlquist and Nix [23] is lower than that observed in this study, the difference could be due, at least in part, to the higher impurity levels in the material they used. The data can be described by eqn. (1), for stresses below 8.35 MPa, with a stress exponent, which describes the stress dependence of the steady state strain rate, equal to 4.6. This value is in good agreement with those reported by other investigators [1, 9, 23]. Stresses greater than 8.35 MPa produce strain rates which are in the power law breakdown region described by Sherby and Burke [9].

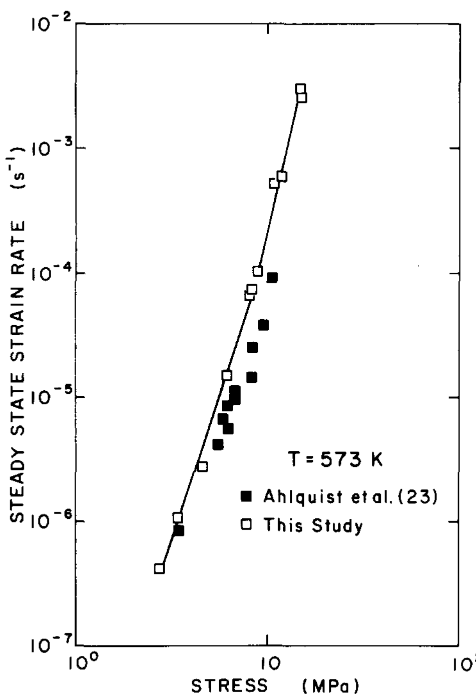


Fig. 1. The effect of stress on the steady state strain rate of 99.999% aluminum at 573 K. The data of Ahlquist and Nix [23] at 573 K for 99.99% aluminum are shown for comparison.

The effect of applied stress on the subgrain size developed during steady state creep in high purity aluminum is shown in Fig. 2. The data of refs. 24 - 26 are also shown, and there is good agreement. The results are in agreement with eqn. (2) which predicts that subgrain size decreases as stress increases. The stress dependence of the subgrain size was found to equal 1.1 ± 0.10 by a least squares fit of the experimental data to eqn. (2). This value is in good agreement with values found for aluminum and other metals [1, 9, 10, 14], but not with the value of 0.26 recently reported by Orlova *et al.* [6]. The subgrain size observed by Orlova *et al.* [6] in the range of stress used in this study is much smaller than those observed by others [1, 9, 10, 14, 24 - 26] and those found in this study by use of optical microscopy. It is possible that this disagreement occurs because different techniques were used for the measurement of subgrain sizes. Orlova *et al.* [6] used TEM exclusively, while a variety of techniques including optical microscopy, TEM, and X-ray microbeam were used in the other studies [1, 9, 10, 14, 24 - 26] ; only optical microscopy was used in the present study.

During the course of this investigation an attempt was made to use TEM for subgrain size determinations. The data obtained were in good agreement with those of Orlova *et al.* [6], but it was observed that the fine substructure was localized in certain regions of the foil. The morphology of this substructure was questioned by Ajaja [27]. Subsequently, very careful TEM specimen preparation revealed a much larger subgrain structure which had the characteristic dislocation configuration usually associated with subgrains formed during high temperature deformation. These larger subgrains were approximately the same size as those observed using the optical techniques. These subgrains were sufficiently large that preparation of a large number of foils for TEM observations would be required to produce statistically significant results; thus it was decided to focus on data collected by use of optical microscopy, even though the optical techniques have been criticized because of difficulty in distinguishing the smaller subgrains [11].

Typical strain–time curves, after stress reductions at a true strain of 0.16, from 15 to 3.44 MPa and 6.23 to 3.44 MPa are shown in Fig. 3. The strain rates as a function of time after the stress reduction, determined by measuring the slopes of the strain–time curves, are shown in Fig. 4. As can be seen, the strain rate after a stress reduction decreases initially from the value obtained immediately after the stress reduction to a minimum value. This effect is currently under study and will be the subject of a future paper. The strain rate data described in this paper will focus on the behaviour after this initial decrease.

Following the minimum, the strain rate gradually increases, approaching the steady state creep rate for the reduced stress. No necking was visible for total true strains below 0.28, but at strains of this magnitude the

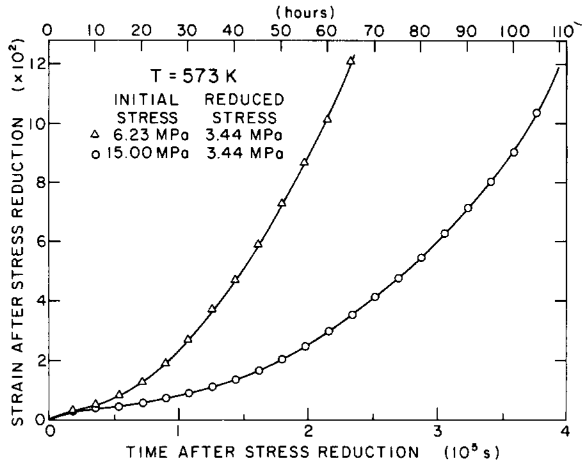


Fig. 3. Strain–time curves illustrating typical behaviour after stress reductions from 15 to 3.44 MPa, and 6.23 to 3.44 MPa. The stress was reduced at a true strain of 0.16 in each case.

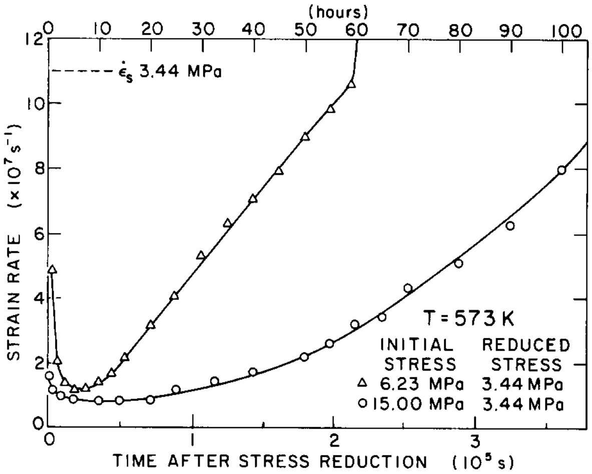


Fig. 4. The variation in strain rate as a function of time after a stress reduction for initial stresses of 15 and 6.23 MPa reduced to 3.44 MPa at a true strain of 0.16 in each case.

sample surface was rough and uneven making it extremely difficult to determine when necking occurred. For total true strains greater than 0.28 the measured strain rates at 3.44 MPa, for samples subjected to a stress reduction, were higher than the steady state strain rates for samples deformed at 3.44 MPa and not subjected to a stress reduction.

The variation in subgrain size as a function of time following stress reductions is shown in Fig. 5. These data clearly show that the subgrain size increases after a stress reduction approaching the subgrain size which would be obtained during steady state deformation at 3.44 MPa. This increase in subgrain diameter

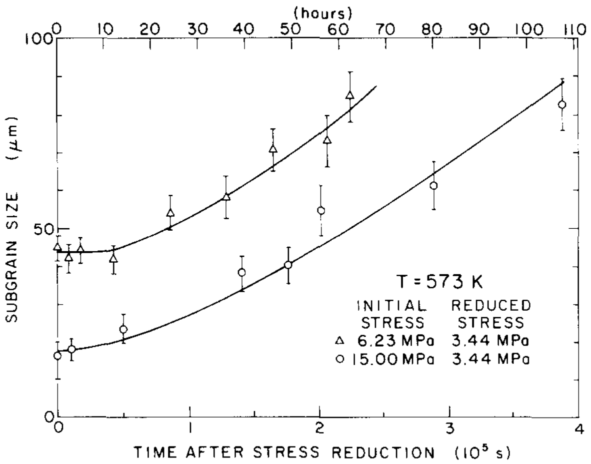


Fig. 5. The variation of subgrain size as a function of time for initial stresses of 15 MPa and 6.23 MPa reduced to 3.44 MPa at a true strain of 0.16. The error bars indicate 95% confidence limits.

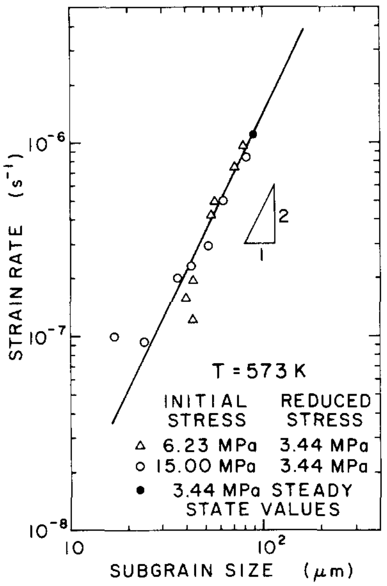


Fig. 6. Strain rate *vs.* subgrain size for samples which have been subjected to a stress reduction from 15 and 6.23 MPa to 3.44 MPa and allowed to deform at the reduced stress for differing time intervals. The strain rate is measured just before the test was terminated for subgrain size measurements. Data for a sample deformed well into the steady state region at 3.44 MPa and not subjected to a stress change are shown for comparison.

is accompanied by an increase in the creep rate. This effect is clearly illustrated in Fig. 6 which shows log (strain rate) *vs.* log (subgrain size) for samples which have been held at temperature for increasingly longer times after the stress reduction.

Our experiments clearly show that subgrain size in high purity aluminum increases after a stress reduction. This supports the assumptions made by Sherby and his coworkers [9, 12, 13, 14] but is in disagreement with the observations of Pontikis and Poirier [17] and Parker and Wilshire [18]. The present study shows that a significant amount of strain after the stress reduction is required for the subgrain size adjustment as suggested by Miller *et al.* [28]. The data of Pontikis and Poirier [17] indicates that they studied the strain–time–subgrain size behaviour, after stress reductions under load, for *ca.* 50 h and did not observe a change in subgrain size even though the strain rate increased. Perhaps ionic solids behave differently from metallic solids because of the complicated bonding and charge neutrality requirements, or possibly the complex thermomechanical history before final stress reduction might have affected the results. The data of Parker and Wilshire [18] indicate that strain–time–subgrain size measurements were conducted for *ca.* 3600 s after the stress reduction. This amounts to a strain of $< 0.5\%$ after stress reduction. Our study clearly shows that no change in subgrain size in pure aluminum would be observed after a strain of 0.5% after the stress reduction.

In addition, our experiments show that deformation at a higher stress definitely strengthens the material when the stress is reduced. This strengthening mechanism could be due to dislocation–subgrain interaction, but one must not reject the possibility that dislocation–dislocation interaction within individual subgrains might make significant contributions to the strengthening effect.

## 4. CONCLUSIONS

Stress reduction experiments in high purity aluminum in conjunction with subgrain size measurements lead to the following conclusions: (i) the subgrain size developed in high purity aluminum increases after reductions in stress; (ii) plastic deformation is necessary for subgrain growth after a stress reduction; and (iii) deformation after a stress reduction in the steady state creep region occurs at a lower rate. This could be due to dislocation–subgrain boundary interaction or dislocation interaction within subgrains.

ACKNOWLEDGMENTS

The authors wish to thank W. D. Nix for helpful discussions. This work was supported by the Graduate School Research Fund, the Office of Engineering Research of the University of Washington, and a grant-in-aid from Sigma Xi. One of the authors (I.F.) wishes to acknowledge financial support from Fundacao de Amparo a Pesquisa do E.S.P. and Instituto Energia Atomica, S.P., Brazil.

REFERENCES

1 J. E. Bird, A. K. Mukherjee and J. E. Dorn, Quantitative Relation Between Properties and Microstructure, Israel Univ. Press, Haifa, Israel, 1969, p. 255.
2 C. R. Barrett, W. D. Nix and O. D. Sherby, Trans. Am. Soc. Met., 59 (1966) 3.
3 L. J. Cuddy, Metall. Trans., 1 (1970) 395.
4 D. J. Mitchel, J. Moteff and A. J. Lovell, Acta Metall., 21 (1973) 1269.
5 S. Karashima, T. Iikubo and H. Oikawa, Trans. Jpn. Inst. Met., 13 (1972) 176.
6 A. Orlová, Z. Tobolová and J. Cadek, Philos. Mag., 26 (1972) 1263.
7 K. Challenger and J. Moteff, Metall. Trans., 4 (1973) 749.
8 S. Daily and C. N. Ahlquist, Scr. Metall., 6 (1972) 95.
9 O. D. Sherby and P. M. Burke, Prog. Mater. Sci., 13 (1968) 325.
10 A. Thompson, Metall. Trans., 8A (1977) 833.
11 J. J. Jonas, C. M. Sellars and W. J. McG. Tegart, Metall. Rev., 14 (1969) 1.
12 S. L. Robinson and O. D. Sherby, Acta Metall., 17 (1969) 109.
13 C. M. Young, S. L. Robinson and O. D. Sherby, Acta Metall., 23 (1974) 633.
14 O. D. Sherby, R. H. Klundt and A. K. Miller, Metall. Trans., 8A (1977) 843.
15 L. Raymond and J. E. Dorn, Trans. Metall. Soc. AIME, 230 (1964) 560.
16 S. K. Mitra and D. McLean, Met. Sci. J., 1 (1967) 192.
17 V. Pontikis and J. Poirier, Philos. Mag., 32 (1975) 577.
18 J. D. Parker and B. Wilshire, Philos. Mag., 34 (1976) 485.
19 I. L. Dillamore and W. T. Roberts, Metall. Rev., 10 (1965) 272.
20 P. Lacombe and L. Beaujard, J. Inst. Met., 74 (1948) 1.
21 E. C. W. Perryman, Acta Metall., 2 (1954) 26.
22 ASTM Designation E122 74, Annual Book of ASTM Standards, 1974, p. 207.
23 C. N. Ahlquist and W. D. Nix, Acta Metall., 19 (1971) 373.
24 D. McLean, J. Inst. Met., 81 (1952 - 53) 287.
25 I. S. Servi, J. T. Norton and N. J. Grant, Trans. Metall. Soc. AIME, 194 (1952) 965.
26 D. McLean and M. H. Farmer, J. Inst. Met., 85 (1956 - 57) 41.
27 O. Ajaja, personal communication, 1978.
28 A. K. Miller, S. L. Robinson and O. D. Sherby, Philos. Mag., 36 (1977) 757.