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Investigation on grain growth and strain rate sensitivity of a superplastic microduplex steel at 1000°C

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A microduplex stainless steel (25.7 wt % Cr–6.6 wt % Ni) was investigated to examine grain growth during static annealing and superplastic deformation at 1000° C. The grain size at a constant strain rate of 1×10^{-4} sec^{-1} increases according to $d \propto t^{0.49}$ where d is the grain size and t is the time (in min) involved in deformation. Under the present test condition, the contribution of both static (time, t_s) and dynamic (strain, ϵ) annealing appear to be significant and can be expressed by $d \propto t_s^{0.19} \epsilon^{0.29}$. While the exponent of the first term is constant, the exponent of the second term may depend on the strain rate. Strain rate sensitivities were evaluated from differential strain rate tests for different initial grain sizes. Both strain rate sensitivity and grain size were noticed to increase with deformation.

1. Introduction

The presence of fine equiaxed grains is one of the most important criterions of structural superplasticity. This condition is generally met in many heavily worked and annealed alloys of two or more phases because of the tendency for less mobility of the interphase boundaries than that of the grain boundaries. From static annealing data on grain growth, the growth laws, $d \propto t^{1/4}$ and $d \propto t^{1/2}$ (t being time), are seen to be obeyed in two-phase and one-phase systems, respectively [1]. In Pb–Sn alloys, a recent investigation [2] shows that diffusion coefficients during superplastic deformation are more than ten times higher than the diffusion coefficients in undeformed superplastic materials and more than 1000 times greater than those in non-superplastic alloys of the same composition. The existence of high diffusivity during superplastic deformation may then negate the advantage of lower grain growth kinetics gained by the prior static annealing of two-phase alloys. Investigations [3, 4] on the kinetics of grain growth also suggest that more grain growth occurs during superplastic deformation and the final grain size is seen to be larger the larger the degree of deformation.

For investigating superplastic behaviour, different initial grain sizes are obtained normally by static annealing of the mechanically worked materials. Most of the characterization of superplastic behaviour are then based upon the initial grain sizes obtained by static annealing. However, recently it has been demonstrated that grain growth during superplastic deformation may be significant enough to invalidate the use of initial grain sizes for satisfactory characterization of flow behaviour [5]. In this regard, evaluation of the correct value of strain rate sensitivity, m, has received much attention.

Superplasticity in a microduplex strainless steel (IN744) has been investigated in some detail [6, 7]. One of the interesting results from these investigations is that m increases with increasing strain. The information presented here indicates an anomalous effect of strain on both grain growth and strain rate sensitivity. The present work, on the microduplex stainless steel (25.7 wt % Cr–6.6 wt % Ni) was, therefore, undertaken with three objectives. First, to investigate grain growth under static annealing since time at high temperature during deformation also contributes to the resulting final grain size. Second, to investigate

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TABLE I Chemical composition (wt %) of the as-received material

C	Cr	Ni	Mn	Si	Ti	P	S	Fe
0.046	25.7	6.6	0.60	0.56	0.24	0.010	0.010	Balance

grain growth due to superplastic deformation by incorporating above data. Third, to correlate strain rate sensitivity with initial grain sizes.

2. Experimental procedure

The microduplex stainless steel (IN744) used in this study was obtained from INCO Research and Development Center in the form of 1.7 mm thick sheet. The chemical composition of this steel is given in Table I. Tensile specimens of 25 mm gauge length and 6.4 mm gauge width were machined from the as-received sheet. Tensile tests were conducted in argon atmosphere by a MTS machine which was programmed to run in a constant strain rate mode.

The heating condition for static annealing was simulated with the practice adopted here in tensile tests, which involved 25 min in heating and soaking time for stabilizing the temperature to an accuracy of ± 0.5° C. The annealing and tensile tests both were performed at a temperature of 1000° C. Metallographic specimens were prepared at selected conditions and micrographs of the longitudinal and transverse surfaces were obtained. Intercept lengths were then measured from the micrographs of both the surfaces and the mean of ~ 1000 intercept lengths is treated as a grain size (d) here. The error in the grain sizes ranged below 10% of the mean value at a confidence limit of 95%.

3. Experimental results

3.1. Static annealing

In order to study grain growth at 1000° C, several specimens of 4.8 μM initial grain size were annealed for times ranging from a few minutes to ~ 30 h. The grain size was seen to increase with increasing annealing time t_s and the time (in min) dependence of instantaneous grain size is shown in Fig. 1. The grain size obeys the time dependence law of the form

$$d \propto t_s^{0.19} \quad (1)$$

3.2. Dynamic annealing

For investigating grain growth during superplastic deformation at 1000° C, specimens of 4.8 μm initial grain size were deformed to different strain levels at a constant strain rate of 1×10^{-4} sec^{-1}. This strain rate was found to fall in region II of $\log \sigma - \log \dot{\epsilon}$ (σ is stress and $\dot{\epsilon}$ is the strain rate) plots for both initial and final grain sizes attained after deformation. The grain size was measured over the strain range of 0.04 to 1.20 (see Fig. 1). For the lowest strain ($\epsilon = 0.04$) the grain size after deformation was found to be smaller than both the initial grain size as well as the grain size attained after an equal period of static annealing (obtainable from the shoulder of the tensile specimen). On subsequent straining the grain size was found to increase with increasing strain. The time dependence of instantaneous grain size during the span of time, t_{sd}, involving up to the end of deformation is seen to obey a relation of the form

$$d \propto t_{sd}^{0.49} \quad (2)$$

From the grain size data at different strain levels a log–log plot of instantaneous grain size against true strain was made. This plot, shown in Fig. 2, has a slope of 0.29 and the strain dependence of grain size may be expressed by

$$d \propto \epsilon^{0.29} \quad (3)$$

3.3. Strain rate sensitivity

Tensile specimens of different grain sizes were obtained by static annealing at 1000° C for different periods of time. Differential strain rate tests were conducted on these specimens over the strain rate range of 1×10^{-5} to 2×10^{-3} sec^{-1} in the

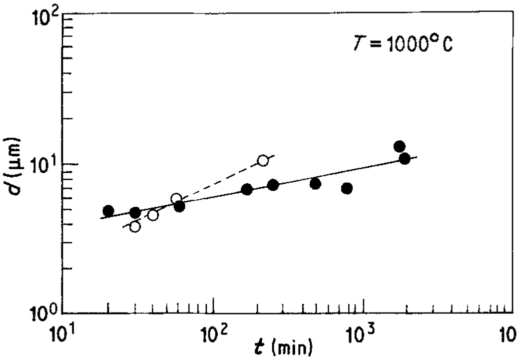


Figure 1 The time dependence of grain size at 1000° C during static (●) and dynamic (○) annealing ($\dot{\epsilon} = 1 \times 10^{-4}$ sec^{-1}).

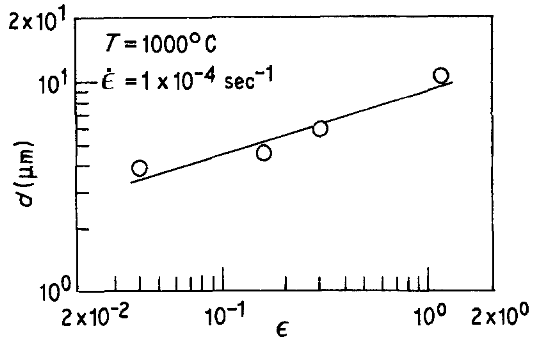


Figure 2 The strain dependence of grain size during superplastic deformation at a constant strain rate.

incremental order of strain rates. These tests were repeated twice on each specimen in order to obtain two cycles of load–elongation data with each specimen. The data corresponding to the first coverage of strain rates (1×10^{-5} to 2×10^{-3} sec^{-1}) are identified to represent cycle I behaviour while the repeated collection of load–elongation data are identified to represent cycle II. Generally, a strain of ~ 30% was involved in cycle I deformation and the cumulative strain after the end of cycle II was ~ 50%.

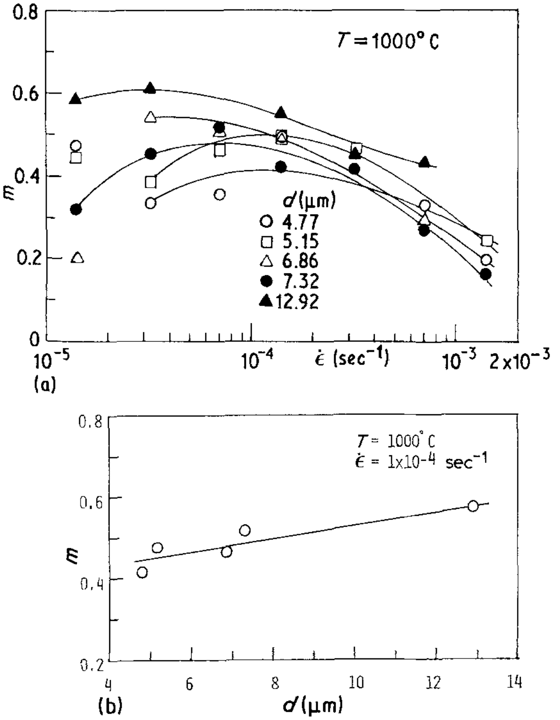
The load–elongation curves (recorded by a x–y chart recorder) at various strain rates involved in the differential strain rate tests were used to evaluate strain rate sensitivity by using

$$m = \frac{\log(p_2/p_1)}{\log(\dot{\epsilon}_2/\dot{\epsilon}_1)} \tag{4}$$

following the technique of Backofen *et al.* [8] but replacing cross-head velocity (V) by strain rate ($\dot{\epsilon}$). In Equation 4, P_2 and P_1 are the loads corresponding to the two strain rates $\dot{\epsilon}_2$ and $\dot{\epsilon}_1$ respectively. The strain rate sensitivity thus calculated corresponds to the strain rate which is a mean of the strain rates involved in its evaluation. Thus, m was obtained over the investigated range of strain rates for five initial grain sizes. The strain rate sensitivity data obtained from cycle I are shown in Fig. 3a for different grain sizes. The m against $\log \dot{\epsilon}$ curves for different grain sizes suggest the existence of maxima in the values of m. As the grain size becomes larger, the $\dot{\epsilon}$ at which the peak in the values of m occurs is seen to be lower. Within the strain rate range investigated, it appears that the maximum value of strain rate sensitivity is larger the larger the initial grain size. Thus, m against $\log \dot{\epsilon}$ curves shift along the direction of increasing m and decreasing strain rates as the grain size increases. Since maxima in m occur at different strain rates, the portion of the m against $\log \dot{\epsilon}$ curves in the vicinity of the maxima also have higher values of m for larger grain sizes. The values of m in this region were used to find their correlation with grain size at a constant strain rate of 1×10^{-4} sec^{-1}. The data are plotted in Fig. 3b and suggest the existence of a linear relationship of the type

$$m \propto k'd \tag{5}$$

where k' ($= 0.02$ at $\dot{\epsilon} = 1 \times 10^{-4}$ sec^{-1}) is a constant whose value depends on the strain rate.



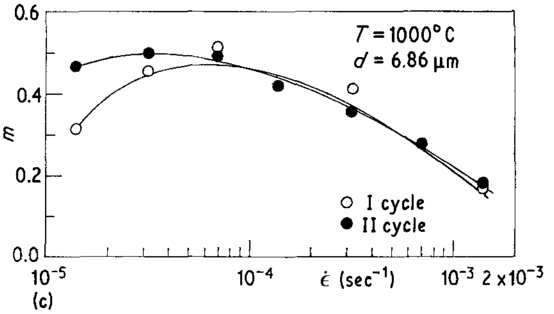


Figure 3 (a) The m against $\dot{\epsilon}$ plots for specimens having different initial grain sizes. (b) Variation of m with initial grain size at a strain rate of 1×10^{-4} sec^{-1}. (c) Effect of repeated strain rate cycling on the m against $\dot{\epsilon}$ data of a specimen having initial grain size of 6.86 μm.

The strain rate sensitivity from the cycles I and II of the differential strain rate tests were compared for each grain size. The data from cycle II (as compared to cycle I) showed an increase in the value of peak m and a decrease in the strain rate at which the peak occurs. A typical comparison of this nature is presented for the initial grain size of 6.9 μm in Fig. 3c.

Grain sizes of the tensile specimens having five different initial grain sizes were measured after the end of cycle II of the differential strain rate test. In all the cases significant grain growth was noticed and its extent could be as high as a 50% increase over the initial grain size.

4. Discussion

Static and dynamic annealing both contribute towards grain growth during superplastic deformation in the present investigation at 1000° C. The contribution of static annealing expressed by Equation 1 suggests significance of the time involved in testing. The contribution of dynamic annealing at $\dot{\epsilon} = 1 \times 10^{-4}\ \mathrm{sec}^{-1}$ expressed as a function of strain in Equation 3 appears to be more significant. The strain in Equation 3, however, has an inherently attached time term which depends on the strain rate under consideration. It is interesting to note that grain growth kinetics observed from the time dependence of grain size during straining up to different strain levels, Equation 2, is in agreement with the sum of the separately identified contributions of static and dynamic annealing, namely,

$$d \propto t^{0.49} \simeq t_s^{0.19} \dot{\epsilon}^{0.29} t_\epsilon^{0.29} \cong \dot{\epsilon}^{0.29} t^{0.48} \qquad (6)$$

since $\epsilon = \dot{\epsilon} t$. The subscript ϵ has been used to show that $t_\epsilon^{0.29}$ may vary according to the strain rate under consideration since the time exponent, n, may have different values.

Equation 6 suggests the possibility that at lower strain rates the grain growth will be dominated by static annealing whereas with increasing strain rates the contribution of static annealing will decrease proportionately. In a nickel base microduplex alloy the grain growth rate is reported [9] to be proportional to the 2/3 the power of the true strain rate. In the presence of comparable contributions of both static and dynamic annealing it may be possible to see a maximum in grain growth at some intermediate strain rate.

The value of the time exponent of 0.19 found in the case of static annealing is comparable to the value ($n = 0.25$) observed in typical microduplex alloys [1]. However, this value is less than that reported [3] for a superplastic Sn–1 wt% Bi alloy which has n of 0.46. The higher n value in Sn–1 wt% Bi alloy, instead, is comparable to the typical values of 0.5 in single phase materials [1], probably, because this alloy is more representative of a single phase system on the basis of its phase proportion. However, the exact values of n depend on the material, its purity and the temperature of annealing [10]. In superplastic Zn–Al eutectoid at 250° C [11] $n \simeq 0.1$ and in 7475 Al – alloy [12] $n \simeq 0.06$ to 0.12 were estimated from the reported grain size data. In Fe–Cr–Ni alloys a wide range of values of n, depending on composition and temperature, have been recently presented [13]. At 1000° C, the alloys containing few second phase particles showed a progressive increase in grain size with increasing time. In contrast, the alloys containing an appreciable amount of second phase particles indicated a much lower time dependence.

The grain growth investigated during high temperature deformation of superplastic materials shows the dominance of dynamic annealing over just static annealing. As a function of time involved in testing the final grain size is larger for higher strain rate in Ti–6Al–4V [14]. As a function of strain, the trend in grain growth is seen to be maximum at intermediate strain rate and decreases towards both lower and higher strain rates in Sn–1 wt% Bi alloy [3]. In Zn–22 wt% Al eutectoid microstructural analysis [11] showed that the grain growth due to deformation increases as the strain rate decreases. In Zn–0.4 wt% Al alloy [4] the grain size increases with the degree of deformation, reaching the maximum value at a low strain rate. For a constant strain, the time involved in deformation is greater at a lower strain rate. With this consideration, the rate of grain growth is reported to increase with increasing strain rate in a Ni base–38 wt% Cr–14 wt% Fe–1.7 wt% Ti–1.0 wt% Al [9] and Zn–0.4 wt% Al [4] alloys. This trend, also observed while evaluating the experimental results from the earlier investigations cited above, seems to apply in several superplastic materials. In this work the grain growth study has not been extended to different strain rate conditions. However, from our earlier observations [15] on microstructures after deformation at different strain rates the grain growth was maximum at lower strain rates.

Also, microstructural observations were made on specimens subjected to differential strain rate and constant strain rate tests. Grain sizes obtained after deformation by both the test techniques were more than those attained in static annealing for the period of testing. In addition, there existed a difference in grain sizes obtained after deformation by these two techniques. This therefore suggests that grain growth kinetic in this material may be sensitive to strain rate and the strain exponent in Equation 3 might depend on the applied strain rate.

In micrograined superplasticity, under steady state, the smaller the grain size the lower its strength and the higher the strain rates over which superplastic behaviour persists. For larger grain size the higher strain rate limit for operation of superplasticity is lowered. In the present investigation of strain rate sensitivity, grain sizes were noticed to increase due to deformation. This grain growth may then lead to strain hardening which may thereby influence the value of strain rate sensitivity. In comparison to the strain rate sensitivity for constant grain size, m for unstable microstructures of different initial grain sizes may be higher or lower [16]. The m against log $\dot{\epsilon}$ plots for different grain sizes in Ti–6Al–4V, in fact, show such behaviour and it also appears that the peak value of strain rate sensitivity for larger grain size may even be at a higher strain rate than that for smaller grain size [17]. This then violates the general trend of variation in m as a function of strain rate for different grain sizes [18]. Under the condition of microstructural instability, therefore, the strain rate sensitivity estimated may not apply to initial grain size.

The increase in m between cycles I and II of the differential strain rate tests may be the consequence of continuous grain growth since both m and grain growth have been noticed to be similarly influenced by strain. Under the condition of grain growth, the value of m evaluated by Equation 4 may also be larger (for a constant strain level) over some strain rate range for larger initial grain size. In the incremental strain rate change test this possibility arises since the smaller grains have a greater tendency for grain growth and the grain growth is seen generally to decrease with increasing strain rate (under combination of static and dynamic annealing). The increase in m with increasing grain size (see Fig. 3b) may therefore be anticipated under non-steady state deformation. In a detailed study [3] of grain growth in a superplastic Sn–1 wt% Bi alloy, in fact, the dynamic grain growth reaches a maximum at intermediate strain rate where the "m" also reaches a maximum value in several superplastic materials [18]. This observation therefore, probably, offers an explanation for the increase in m with increasing strain as reported in earlier investigation on this material [7]. From present results this may also be viewed in terms of following relation

$$\frac{\mathrm{d}m}{\mathrm{d}\epsilon} = \frac{\mathrm{d}m}{\mathrm{d}d}\frac{\mathrm{d}d}{\mathrm{d}\epsilon} \tag{7}$$

However, it may be possible that some other factor might also contribute to the variation of m. A change from one strain rate to the other strain rate generally leads a short transition (or a temporary non-steady) flow behaviour at the new strain rate and in some investigations [15, 19] strain hardening can only partly be accounted for by an increase in the grain size. In the case of strain softening, which results partly from breaking up of initially elongated grains into equiaxed grains, there also appears continuous a increase in m with increasing strain [6]. During strain softening some concurrent grain growth can also occur [19]. Analysis of microstructural data under such conditions becomes more complex and, in fact, illustrates the problem of associating the increase in m to any one particular type of microstructural change.

5. Conclusions

1. Both static and dynamic annealing contribute significantly to grain growth during superplastic deformation of the microduplex stainless steel at 1000° C.

2. Measurement of strain rate sensitivity may be affected by grain growth. Under the condition of non-steady state deformation, grain size and strain rate sensitivity both increase with increasing strain.

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