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Effect of swaging on the 1000°C compressive slow plastic flow characteristics of the directionally solidified eutectic alloy γ/γ'–α

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Swaging between 750 and 1050° C has been investigated as a means to introduce work into the directionally solidified eutectic alloy γ/γ'–α (Ni–32.3 wt% Mo–6.3 wt% Al) and increase the elevated temperature creep strength. The 1000° C slow plastic compressive flow stress–strain rate properties in air of as-grown, annealed, and worked nominally 10 and 25% materials have been determined. Swaging did not improve the slow plastic behaviour. In fact large reductions tended to degrade the strength and produced a change in the deformation mechanism from uniform flow to one involving intense slip band formation. Comparison of 1000° C tensile and compressive strength–strain rate data reveals that deformation is independent of the stress state.

1. Introduction

In a recent study [1] of the elevated temperature tensile creep rupture strength of a simple directionally solidified eutectic (dse) γ/γ'–α alloy, we noted that decreasing the average interfibre spacing appears to be the only effective means of strengthening found to date. In particular alloying by either solute (molybdenum and/or aluminium) variation or multi-element additions [2, 3] has not yielded more creep resistant materials. While advanced growth techniques can be used to somewhat refine the interfibre spacing, the technical difficulties associated with maintaining a good, fault-free microstructure are considerable. Therefore other strengthening methods are of interest.

In 1972 Oblak and Owczarski [4] showed that elevated temperature thermomechanical processing could improve the creep resistance of a Ni-base superalloy (Udimet 700). The increased strength was due to a polygonized dislocation substructure. Such behaviour is consistent with the ideas of Sherby *et al.* [5] and Schmidt *et al.* [6] who have discussed and demonstrated the strengthening role of the substructure, in particular the subgrain size, on elevated temperature mechanical properties.

While a well developed substructure can lead to improved creep properties, non-equilibrium dislocation structures can also be beneficial. For example Harrigan *et al.* [7] found that cold rolling could improve the 825° C vacuum creep properties of a γ' strengthened Ni–18.6 wt% Cr–4.3 wt% Al alloy, Such materials contained intense slip bands which underwent some dislocation annihilation and rearrangement prior to testing at 825° C; however they did not form a subgrain boundary structure. More recently Marlin *et al.* [8] increased the 760° C creep strength of the Ni-base oxide dispersion strengthened alloy MA 754 through small amounts of predeformation at 760° C which simply increased the average dislocation density.

The above studies indicate that it could be possible to increase the elevated temperature creep resistance of dse alloys through working. In this aspect the γ/γ'–α system is ideal, because both the matrix and strengthening phases are ductile whereas most dse systems contain a brittle strong phase.

Hence rather large amounts of work should be possible without fracturing the α-Mo fibres, and one should be able to introduce dislocations into both constituents.

This study presents the results of part of a continuing effort to examine the potential of dse alloys for use in gas turbine engines. In this paper the slow plastic flow properties of as-grown and annealed γ/γ'–α are compared to those of γ/γ'–α which had been swaged at 750, 900 and 1050° C approximately 10 and 25% in a single pass. Swaging was selected for the working technique; because it results in relatively uniform deformation, maintains fibre alignment and simulated a reasonably quick, commercially feasible forming operation. Testing was accomplished in compression at 1000° C at strain rates ranging from about 2×10^{-4} to about 2×10^{-7} sec^{-1}. Standard materialographic procedures were used to assess the microstructure prior to and following testing. In addition the stress–strain rate behaviour determined in compression was compared to that previously measured in tension [1].

2. Experimental procedures

Approximately 300 mm long, 8 mm diameter γ/γ'–α bars of nominal composition Ni–32.3Mo–6.3Al (wt%) were grown at 17 mm h^{-1} by a modified Bridgeman technique. Each bar was microscopically examined for nonaligned regions and growth faults by grinding and polishing a narrow stripe along the bar length. About the centre 70% of each bar was well aligned and generally free of faults. Further details of the dse preparation methods can be found in [1].

Three bars without growth faults were selected for warm working by swaging. Each bar was cut into two nominally equal lengths which were then encapsulated in stainless steel. The pieces from each bar were placed into a furnace and heated for 1 h prior to swaging about 10 or 25% in a single pass. Swaging was accomplished at 750, 900 and 1050° C, and approximately one third of each piece was swaged.

Right cylindrical compression specimens 5 mm in diameter and 10 mm long were machined from both the swaged and unworked (annealed) regions of each piece and from an as-grown γ/γ'–α bar. Samples to assess the amount of work and quality of the microstructure were taken from the vicinity of the transition region between the swaged and unworked sections.

1000° C compression tests were conducted in air at constant velocities ranging from 2.12×10^{-3} to 2.12×10^{-5} mm sec^{-1} in an universal testing machine. During testing the temperature was maintained to 1° C, and the difference in temperature between the top and bottom of the compression specimens never exceeded 3° C. Both stresses and strains were calculated from load–time charts where strains were determined by an offset method [9] in combination with a normalization procedure based on the actual length change at the end of the test. Additional details concerning the testing technique are in [10].

3. Results

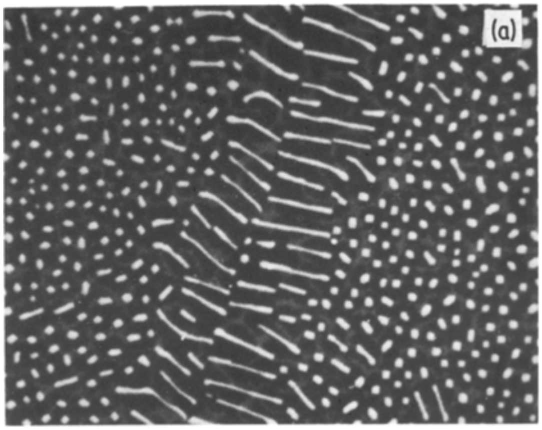
3.1. Effect of swaging on structure

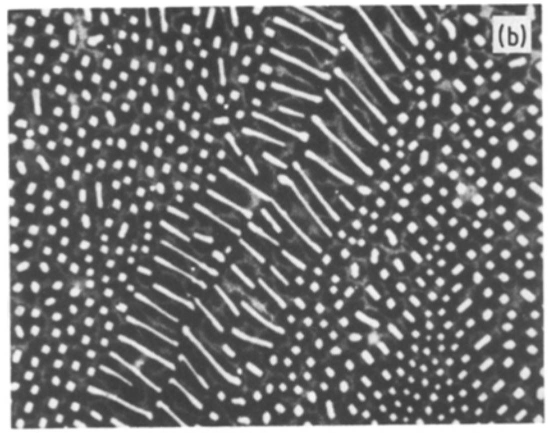
Based on the study of Oblak *et al.* [4] and the concepts of Sherby and co-workers [5, 6], swaging temperatures of 750, 900 and 1050° C were chosen as the best compromise to introduce both warm and cold work into γ/γ'–α. These represent homologous temperatures of 0.6 to 0.8 for the γ/γ' matrix (based on the melting point of Ni_3Al) and 0.35 to 0.45 for the α-fibres (based on the melting point of pure molybdenum). Ambient temperature swaging of γ/γ'–α was not undertaken because of the very high strength of this material.

While swaging bare γ/γ'–α was attempted between 750 and 1050° C, it was not successful. The stainless steel clad bars were, on the other hand, worked without difficulty. However the ends of the pieces subjected to the larger reductions were generally cracked. Also during machining it was found that the γ/γ'–α piece swaged about 25% at 900° C had a longitudinal crack running almost the entire length of the worked region; therefore, only one sound specimen was obtained for this condition.

Light and scanning electron microscopy (SEM) of the swaged, annealed and as-grown γ/γ'–α materials revealed little, if any, difference in microstructures with the exception of the size of the fibres and the interfibre spacing. In particular the α-fibres retained their generally square cross-section shape, and the grain boundaries maintained the zipper-like appearance even after 750° C swaging. Typical examples of worked microstructures are shown in Fig. 1. No evidence of cracking was found in the polished specimens.

The interfibre spacing λ for the various material conditions are given in Table I; these were calculated from





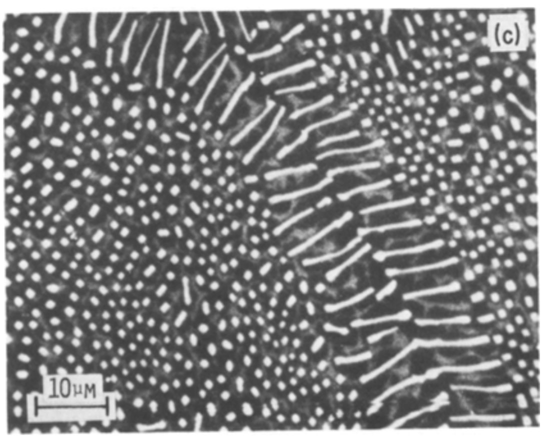


Figure 1 Transverse microstructure of $\gamma/\gamma'-\alpha$ in the vicinity of grain boundaries after swaging approximately 25% at (a) 750° C, (b) 900° C and (c) 1050° C.

$$\lambda = 1.075/N^{1/2} \quad (1)$$

where N is the number of fibres per unit area. The amount of work was calculated from the change in bar diameter and from the change in interfibre spacing where

$$\text{work} = 1 - \left(\frac{\lambda'}{\lambda^0}\right)^2 \quad (2)$$

and λ', λ^0 are the swaged and annealed interfibre spacings, respectively. As can be seen in Table II, the agreement between the two computation methods is, in general, only fair. The differences are probably due to local variations in the diameter and interfibre spacing along the length of each bar.

TABLE I Average interfibre spacings of as-grown and thermomechanically processed $\gamma/\gamma'-\alpha$*

Temperature	Spacing (μm)		
	Annealed	Larger swaging die	Smaller swaging die
As-grown	1.77	–	–
750° C	1.83	1.70	1.55
900° C	1.92	1.73	1.56
1050° C	1.81	1.65	1.58

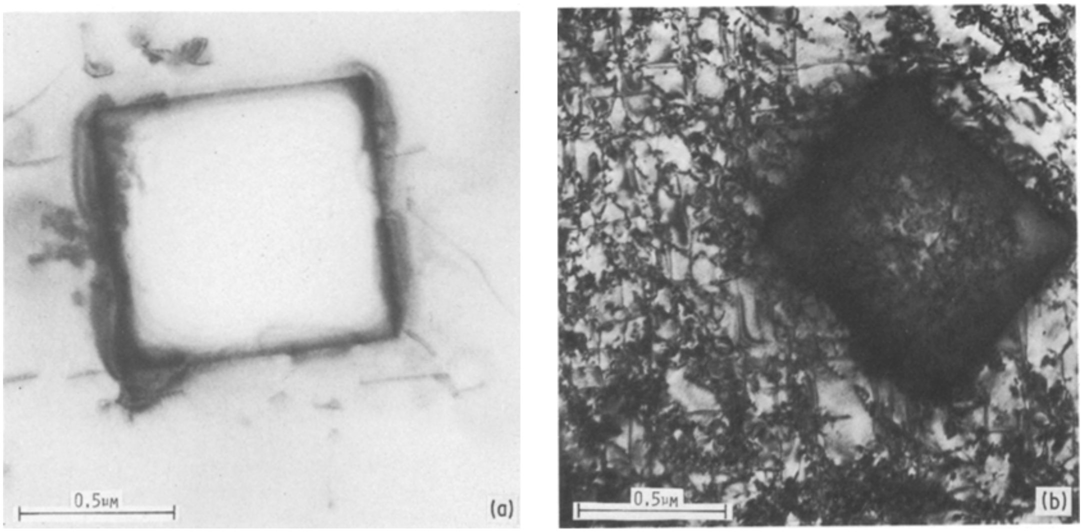
*Error estimated to be ± 0.03 μm.

Typical transmission electron (TEM) photomicrographs of as-grown and swaged $\gamma/\gamma'-\alpha$ are presented in Fig. 2. It is clear from these photographs that swaging introduces significant numbers of dislocations into both the γ/γ' matrix and α-fibres. In general the structure of the materials worked about 10% at 750, 900 and 1050° C are similar with the exception that the dislocation density in the fibres was less in the 750° C worked alloy than either the 900 or 1050° C swaged stock. Examination of $\gamma/\gamma'-\alpha$ swaged about 25% at 1050° C revealed that its structure (Fig. 2c) is like that of the materials swaged about 10% (Fig. 2b); except its dislocation density is higher. For these conditions neither dense dislocation pile-ups were

TABLE II Per cent work after elevated temperature swaging

Swaging temperature	Large die	Small die
*(a) Based on change in diameter**		
750° C	9	25
900° C	9	27
1050° C	10	30
(b) Based on change in interfibre spacing†		
750° C	14	28
900° C	19	34
1050° C	17	24

*Error estimated to be ± 3%.
†Error estimated to be ± 5%.



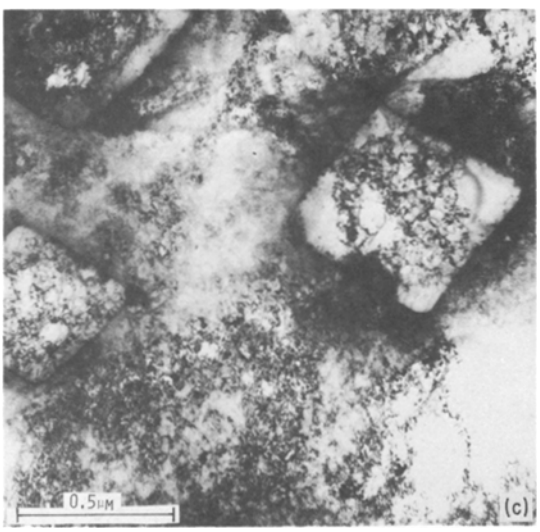


Figure 2 Typical transmission electron photomicrographs of γ/γ'–α (a) as grown, (b) swaged about 10% at 900° C and (c) swaged about 25% at 1050° C.

observed at the matrix/fibre interfaces nor were distinct subgrains formed. Swaging did, however, somewhat affect the cross-sectional shape of the α-Mo fibres where occasional rounding of the corners was evident after about 10% work and rounded corners coupled with the loss of straight edges after about 25% work.

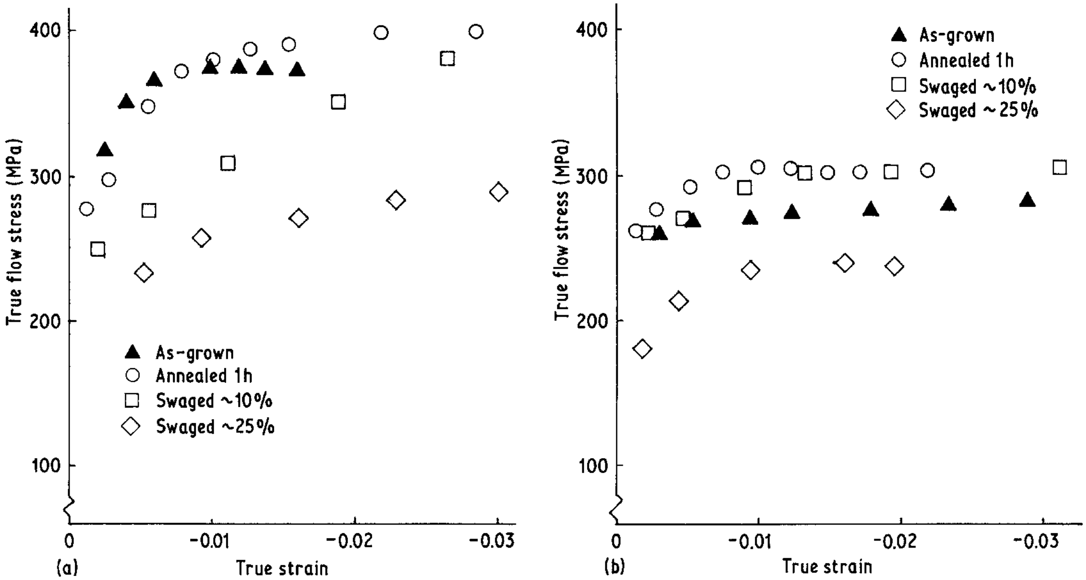
3.2. Mechanical properties

In this work compression testing at 1000° C was utilized, because compressive loading should insure more uniform deformation than tensile testing, and 1000° C is the highest use temperature envisaged for γ/γ'–α and should provide the severest test of dislocation stability. The true stress–true plastic strain behaviour was similar for all material conditions, and typical examples are given in Fig. 3. Here it can be seen that γ/γ'–α work hardens before reaching a more or less constant flow stress. The data also reveal that swaging did not improve the 1000° C strength. In several instances, in fact, swaging actually decreased the high temperature flow strength. These impressions are reinforced by the true stress–strain rate data in Fig. 4 for the as-grown and thermomechanically processed alloys. At best it appears that swaging 10% did not affect strength while higher amounts of work reduced the load bearing capacity, particularly at the slowest strain rate.

All the flow stress–strain rate data were statistically examined using linear regression techniques to fit

$$\dot{\epsilon} = A\,\sigma^n \tag{3}$$

where $\dot{\epsilon}$ is the strain rate, A is a constant, σ is the stress, and n is the stress exponent. This analysis indicated that the 1000° C properties of γ/γ'–α after annealing 1 h at either 750 or 1050° C are equivalent to those of the as-grown material. On the other hand 900° C annealing somewhat improved the 1000° C slow plastic behaviour in comparison to the characteristics of the as-grown alloy. Nominally 10% work was found to have no effect on flow stress–strain rate properties while higher amounts of work were generally deleterious.



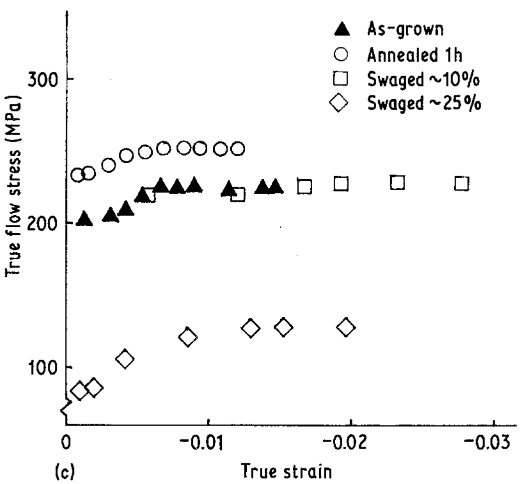


Figure 3 Typical compressive true stress–true strain diagrams for as-grown, annealed 1 h at 750° C, and hot worked at 750° C γ/γ'–α materials tested at 1000° C and nominal strain rates of (a) $2.1 \times 10^{-5}\,\mathrm{sec}^{-1}$, (b) $2.1 \times 10^{-6}\,\mathrm{sec}^{-1}$ and (c) $2.1 \times 10^{-7}\,\mathrm{sec}^{-1}$. The specimen swaged about 10% and tested at the strain rate of $2.1 \times 10^{-5}\,\mathrm{sec}^{-1}$ reached a constant flow stress of about 410 MPa after – 0.042 strain.

3.3. Post test microstructure

Most specimens tested at the two slower strain rates were examined by light optical and SEM techniques. Typical SEM photomicrographs are presented in Fig. 5. In general the microstructures of the as-grown, annealed and swaged 10% materials were similar. For these conditions some agglomeration of the molybdenum laminae occurred at the grain boundaries while individual fibres within grains retained their generally square cross-section (Fig. 5a). The microstructure of the materials worked about 25% also exhibited random agglomeration of grain boundary lamina; however the shape of the individual molybdenum fibres was degraded (Fig. 5b) and intense slip band formation was found (Figs. 5c, d and e) which was not present after 25% swaging (Fig. 1). Within the slip bands fibres were sheared into fragments which then coalesced and permitted cavitation to occur. As witnessed by Fig. 5d the intersection of a slip band with the specimen surface can result in large offsets. This photomicrograph also illustrates the oxidation processes in γ/γ'–α where a α-Mo fibre-free zone is formed at the outer surface.

4. Discussion

This attempt to improve the elevated temperature creep strength of γ/γ'–α through introduction of work by swaging was not successful. Since swaging resulted in alike TEM microstructures, and these are similar to those commonly found in cold worked materials; it seems that non-equilibrium dislocation configurations do not provide additional creep resistance. On the basis of the deformation behaviour of the more heavily worked materials, it is clear that at least some of the induced dislocation structure is retained during 1000° C compression testing and this can degrade the performance of the alloy. In particular heavy prior work produces a change in slow plastic deformation mechanism(s) from uniform flow to one involving

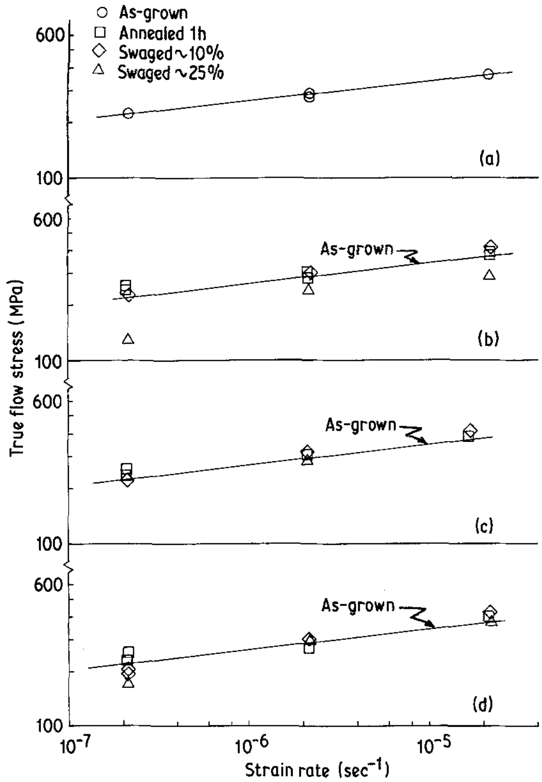


Figure 4 True compressive stress–strain rate behaviour at 1000° C for γ/γ'–α: (a) as-grown and thermomechanically processed at (b) 750° C, (c) 900° C and (d) 1050° C.

intense slip band formation which is detrimental to the strength under slow strain rate conditions. Due to the seeming inability to develop a stable subgrain boundary structure by swaging in a single pass, the effect of an equilibrium dislocation structure on the slow plastic strength of γ/γ'–α can not be answered by this study.

On the positive side the present results indicate that γ/γ'–α can be rapidly worked at least 10% between 750 and 1050° C without affecting the elevated temperature strength properties. Therefore, small shaping operations, such as forging or bending to provide appropriate contours or twists in gas turbine airfoils, should be possible without harm. Additionally because the induced dislocation structure after small amounts of work was essentially independent of the swaging temperature and similar in appearance to a cold worked alloy, it is likely that γ/γ'–α could be rapidly worked nominally 10% at any temperature and still maintain its elevated temperature slow plastic properties.

Combination of the compressive flow stress–strain rate data for as-grown and annealed 1 h at 750 or 1050° C material from this study with the 1000° C tensile creep stress–strain rate data from [1] allows one to evaluate the effect of the stress state on the plastic properties. Such a comparison is of interest since a recent study [11] of creep in lamellar Al–$CuAl_2$ composites reports that the stress exponent in compression is less than that in tension.

All appropriate strain rate–stress data for γ/γ'–α are presented in log–log format in Fig. 6. Treated as separate entities, the best regression fits of the data yields a stress exponent of 9 for compression and one of 13 for tension. While this difference in exponents appears to be large, the actual ability of the fit for tension to predict the behaviour under compression and vice versa is reasonable. The major difference in the individual regression equations lies in the constant term(s) in Equation 3 which vary by almost 10^{10}.

Since the factors affecting deformation at extremely slow strain rates and low stresses should be equal irrespective of the stress state, all data were fitted to

$$\dot{\epsilon} = B\,\sigma^{X_c n_c}\,\sigma^{X_T n_T} \qquad (4)$$

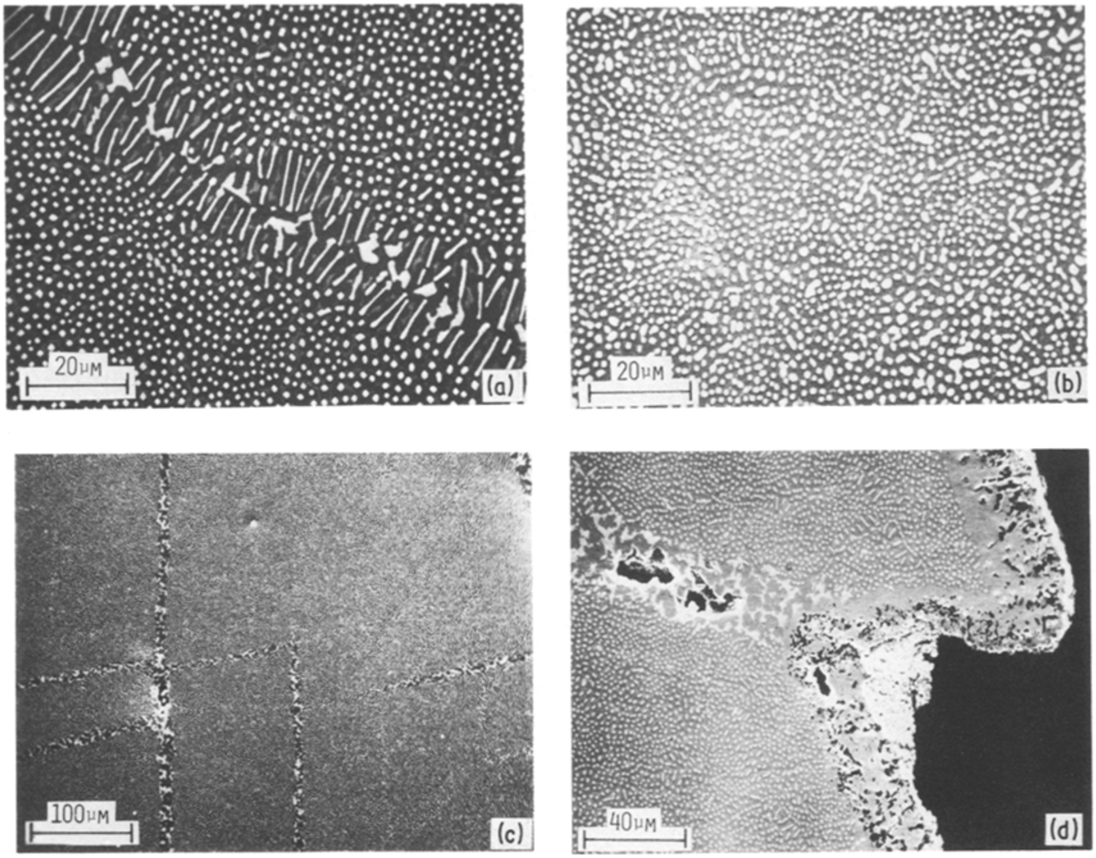
where X_c equals 1 for compression and 0 for tension; X_T equals 1 for tension and 0 for compression; and n_c and n_T are the stress exponents for compression and tension, respectively. Statistical testing of these two regression curves revealed that they were equivalent; hence the stress exponent for γ/γ'–α is not dependent on stress state. At 1000° C

$$\dot{\epsilon} = 1.8 \times 10^{-34}\,\sigma^{11.3} \qquad (5)$$

(standard deviation $n = 0.6$)

well describes the strain rate–stress behaviour of γ/γ'–α, as shown in Fig. 6 with a regression coefficient of 0.94 and a standard deviation of fit about the predicted strain rate of 2.4. Finally it should be noted that independence of the stress exponent and the state of stress validates compression testing at constant velocities as a means to determine the plastic characteristics of a material.

This study suggests two areas for future work. The small but positive effect of a 900° C heat treatment on the creep properties of γ/γ'–α deserves further effort. Most likely this strength improvement is due to precipitation of small molybdenum particles in the γ/γ' matrix [12]



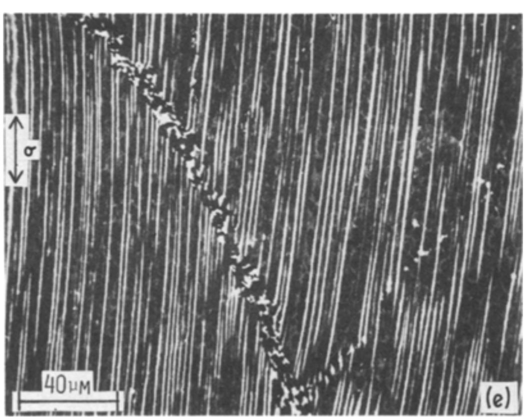


Figure 5 Typical scanning electron photomicrographs of $\gamma/\gamma'-\alpha$ tested at 1000° C in compression. (a) swaged about 10% at 750° C, $\dot{\epsilon} \sim 2.15 \times 10^{-7}\ \text{sec}^{-1}$ to 2.8% strain and $\sigma = 229$ MPa; (b) swaged about 25% at 1050° C, $\dot{\epsilon} \sim 2.1 \times 10^{-7}\ \text{sec}^{-1}$ to 1.4% strain and $\sigma = 168$ MPa; (c) and (d) swaged about 25% at 750° C, $\dot{\epsilon} \sim 2.1 \times 10^{-6}\ \text{sec}^{-1}$ to 10.2% strain and $\sigma = 240$ MPa; (e) swaged about 25% at 900° C, $\dot{\epsilon} \sim 2.1 \times 10^{-6}\ \text{sec}^{-1}$ to 10.7% strain and $\sigma = 280$ MPa. (a) through (d) are cross-sections and (e) is a longitudinal section.

which have been found in solution treated and aged materials. Secondly strain/anneal cycling should be investigated as a means to produce a stable subgrain boundary structure in $\gamma/\gamma'-\alpha$. If successful, the influence of subgrains on the creep behaviour of composites could be determined.

5. Summary of results

Based on a study of the effects of swaging in a single pass between 750 and 1050° C on the slow plastic strain rate properties of $\gamma/\gamma'-\alpha$ in compression, the following results were observed:

1. Small amounts of work (10%) can be introduced without deleterious effects on the high temperature creep characteristics.
2. Larger amounts of work (25%) produce changes in deformation mode and microstructural degradation and tend to reduce the creep strength.
3. Comparison of the compressive strain rate–stress data to those from tension testing reveals that plastic behaviour is not dependent on the stress state.

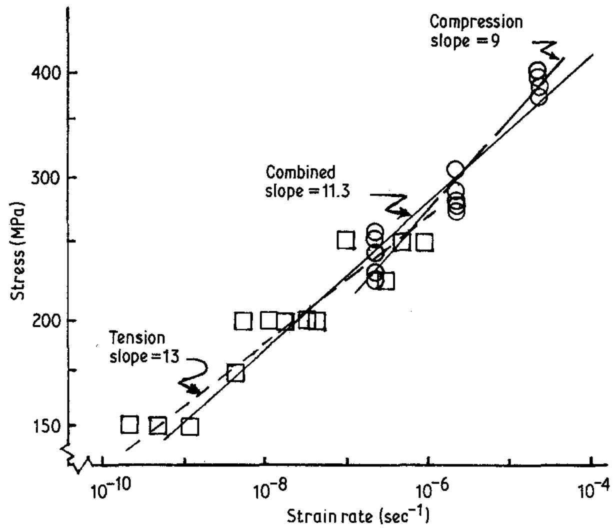


Figure 6 Compressive and tensile stress–strain rate behaviour for $\gamma/\gamma'-\alpha$ at 1000° C.

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