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Elevated temperature tensile properties of powder metallurgy Ni_3Al alloyed with chromium and zirconium

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The tensile properties of hot extruded powders of Ni–24.1Al, Ni–19.1Al–8.5Cr, and Ni–17.4Al–7.9Cr–0.5Zr have been evaluated from room temperature to 1000° C. These powder metallurgy materials have a fine grain size that results in relatively little increase in yield stress with increasing temperature compared to coarse-grained or single-crystal materials. The alloy containing chromium and zirconium shows greatly reduced dynamic embrittlement in the temperature range 600 to 800° C where the unalloyed aluminide exhibits brittle behaviour. The Cr- and Cr + Zr-containing alloys deform superplastically above 900° C. The mechanism of superplastic deformation appears to be predominantly grain-boundary sliding.

1. Introduction

The recent interest in ductile intermetallic compounds has resulted in considerable development of alloys based on Ni_3Al. It is now well established that polycrystalline alloys with a substoichiometric aluminium content exhibit good room-temperature ductility when alloyed with 0.1 to 0.25 at % B [1–3]. It has been shown that solid solution strengthening with hafnium and zirconium can be effective in improving the mechanical properties of these alloys [4, 5]. Alloying with iron and chromium has been extensively studied, and improved strength and oxidation and sulphidation resistance are reported for alloys containing these elements [5–7]. Elevated temperature testing in air has shown that Ni_3Al is sensitive to dynamic oxygen embrittlement in the temperature range from 600 to 800° C; however, this effect can be minimized by reducing the aluminium content or alloying with about 8 wt % Cr [6, 8].

The effects of grain size on the mechanical properties of Ni_3Al have been extensively studied. Reducing the grain size has been found to significantly increase the room-temperature strength, and with relatively little effect on the ductility [3, 9]. Rapid solidification or powder metallurgy processing can be used to achieve refined microstructures and enhanced ductility [10, 11]. The powder metallurgy approach also offers the possibility of using near net shape fabrication techniques with the alloys based on Ni_3Al that are difficult to hot work from ingots. In particular, it has been shown that a Ni_3Al–Cr–Zr–B alloy that has been processed to have a fine grain size exhibits superplastic behaviour above 950° C [6].

This paper presents the results of a study of the elevated temperature tensile properties of powder metallurgy Ni_3Al and Ni_3Al alloyed with chromium and zirconium. The effect of the alloying additions on the reported superplastic behaviour of fine-grained aluminides was examined. The strain rate sensitivity has been measured as a function of temperature to determine the deformation mechanism that is responsible for superplasticity.

2. Procedure

Powders produced by vacuum gas atomization were obtained from Homogeneous Metals, Clayville, New York. The powders were sealed in evacuated steel cans and extruded at 1100° C. The chemical compositions of the three extruded materials are given in Table I. The unalloyed aluminide has an aluminium content of about 24.1 at %. Assuming that chromium occupies aluminium and nickel sites with equal frequency [12], the chromium-containing alloy has an aluminium equivalent of about 23.5 at %. Zirconium is expected to behave in a similar manner to hafnium, which has been shown to strongly prefer aluminium sites [13]. On this basis the Cr + Zr alloy can be considered to have an aluminium equivalent of about 22 at %.

Round tensile samples were machined from the Ni_3Al and Ni_3Al + Cr extrusions. The samples were annealed for 1 h at 1000° C prior to testing to produce an equiaxed microstructure with grain sizes of approximately 15 and 6 μm for the Ni_3Al and Ni_3Al + Cr, respectively. The Ni_3Al + Cr + Zr extrusion was sectioned into several longitudinal slices which were cold rolled with intermediate anneals to form thin sheet from which flat tensile samples were punched. The tensile samples were annealed for 1 h at 1000° C prior to testing to yield a final grain size of about 10 μm.

Tensile testing was done in laboratory air from room temperature to 1000° C. The Ni_3Al and Ni_3Al + Cr

TABLE I Chemical analysis of extruded powders (at %)

Material	Ni	Al	Cr	Zr	C	S	P	N	O	B
Ni_3Al	75.90	24.10	–	–	0.02	0.002	0.005	0.007	0.03	0.095
Ni_3Al + Cr	72.34	19.15	8.51	–	0.03	0.002	0.008	0.007	0.02	0.13
Ni_3Al + Cr + Zr	74.21	17.37	7.89	0.52	0.05	0.001	–	0.004	0.03	0.074

samples were tested to failure at a nominal strain rate of $5 \times 10^{-4}\,sec^{-1}$ and the Ni_3Al + Cr + Zr samples were tested at a nominal strain rate of $8 \times 10^{-4}\,sec^{-1}$. The strain rate sensitivity was measured as a function of temperature from tensile curves for samples which were pulled to failure at several strain rates, and, in some cases, using strain rate jump tests.

3. Experimental results

The Ni_3Al and Ni_3Al + Cr were both found to be ordered single phase after the final 1000° C heat treatment. This is consistent with published work on the solubility of chromium in Ni_3Al which indicates that the order–disorder phase boundary for 8% Cr is just above 1000° C [12]. The microstructure of the Ni_3Al + Cr + Zr alloy was found to consist of disordered islands within an ordered matrix after the final heat treatment.

At room temperature all of the materials exhibit large yield drops, and Lüders strains as large as 5%, before uniform elongation begins. The magnitude of the yield drops and Lüders strains both decrease as the test temperature is increased. The 0.2% offset yield stress of the extruded materials is shown as a function of the test temperature in Fig. 1. This method of specifying the yield stress was used to minimize the influence of the yield drops that were noted for the lower temperature tests. The corresponding total elongation values are shown in Fig. 2. It can be seen that alloying with chromium improves the strength and reduces the magnitude of dynamic embrittlement. The alloy which contains both chromium and zirconium shows some additional increase in strength and nearly complete supression of dynamic embrittlement.

The strain rate sensitivity exponent, m, defined by the relationship $\sigma = K\dot{\varepsilon}^m$ can be determined from the slope of a log stress/log strain rate plot. Stress–strain rate data for the extruded powder Ni_3Al and Ni_3Al + Cr are shown as a function of the test temperature in Fig. 3. It can be seen that there are generally two regimes in the log–log plots, with a change in slope at higher strain rates. If the slope between the two lowest strain rates is considered, it can be seen for the chromium-containing alloy that there is a monotonic increase in m as the temperature is increased. It was not possible to test the unalloyed material over the entire temperature range because of the sharply reduced ductility near 700° C. At 900° C and above the chromium-containing material has an m value of about 0.5 and the unalloyed aluminide has a value approaching 0.5. The alloy containing both chromium and zirconium exhibits behaviour very similar to the chromium-containing alloy at temperatures of 950° C and above [6].

The stress values shown in Fig. 3 were determined for temperatures below 800° C using strain rate jump tests. Above this temperature it was found that the low strain rate values were more consistent when measured from samples which were tested to failure. For these samples the values reported are the maximum stress, which occurs just after yielding. After a small amount of plastic strain (~ 1%), work softening occurred and the stress declined monotonically out to large strains. The strain rate sensitivity values

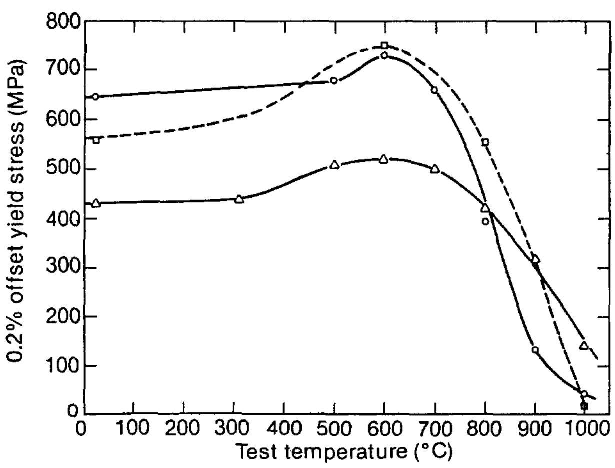

Figure 1 Yield stress as a function of test temperature. (△) Ni_3Al, (○) Ni_3Al + Cr, (□) Ni_3Al + Cr + Zr.

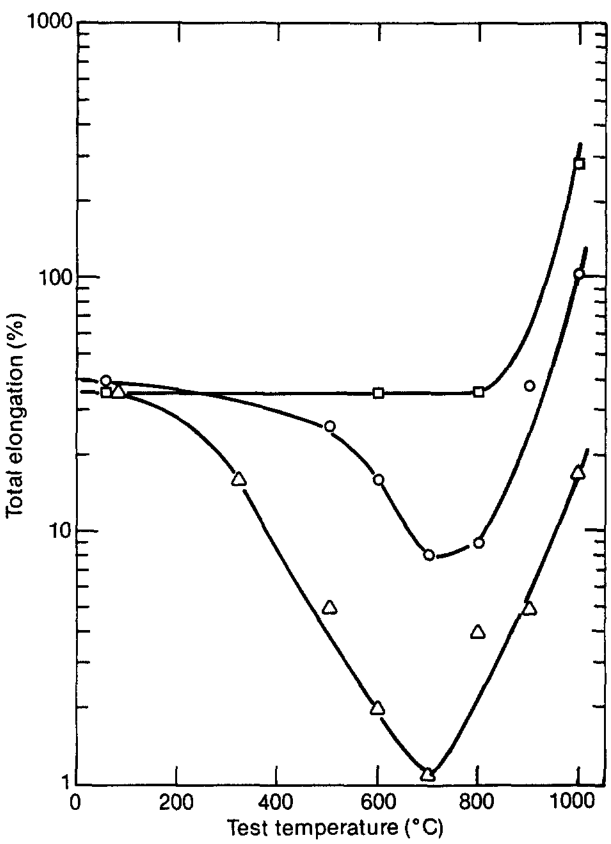

Figure 2 Total elongation as a function of test temperature. (△) Ni_3Al, (○) Ni_3Al + Cr, (□) Ni_3Al + Cr + Zr.

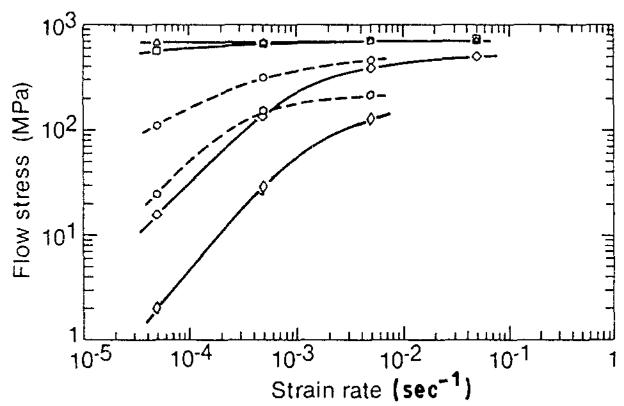

Figure 3 Log flow stress plotted against log strain rate as a function of test temperature for extruded VGA powders. (△) 25°C, (□) 700°C, (○, ◇) 900°C, (◊, ○) 1000°C. (——) Ni–18.4Al–8.3Cr, (– – –) Ni–22.6Al.

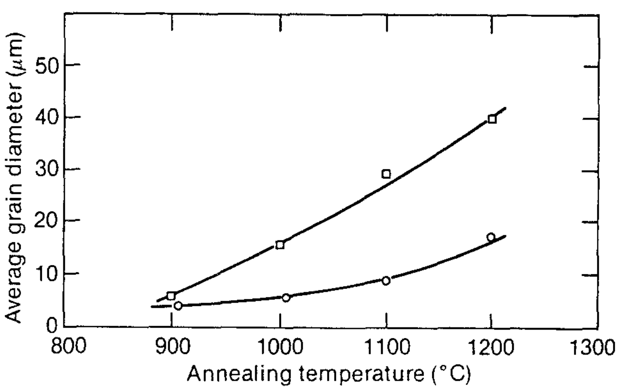

Figure 4 Grain size extruded powders as a function of temperature for 1 h anneals. (□) Ni–22.6Al, (○) Ni–18.4Al–8.3Cr.

measured by jump tests at high temperatures tended to be somewhat higher than those measured from samples tested to failure.

4. Discussion

The fine-grained material which results from extrusion of powders has good room-temperature strength compared to larger grained material which often results from casting, while retaining good ductility [2, 14]. The powder metallurgy materials tend to have a smaller increment of strengthening on increasing the test temperature compared to large-grained materials. In single-crystal Ni_3Al, for example, there is nearly a five-fold increase in the flow stress on raising the test temperature from room temperature to 700° C [15]. Experiments with samples of varying grain size from casting or recrystallization from single crystals indicate that in general the magnitude of the strength peak increases with increasing grain size [14, 15]. Alloying with chromium and zirconium results in considerable solid solution strengthening but does not appear to significantly influence the magnitude of the strength peak. The apparent strength of the zirconium-containing alloy shown in Fig. 1 might be slightly enhanced compared to the two other materials due to testing at slightly higher strain rate.

It is evident that the addition of about 8% Cr to Ni_3Al substantially improves the ductility between 600 and 800° C. The alloy containing about 8% Cr and 0.52% Zr shows no evidence of embrittlement in this temperature range. It is difficult to draw unambiguous conclusions about the effect of alloying additions because there is a difference in effective stoichiometry and microstructure between the zirconium-containing alloy and the other two materials. The zirconium-containing alloy is two-phase ordered plus disordered after the final heat treatment while the other alloys are single phase. It has been shown that the alloy stoichiometry has an influence on the degree of dynamic embrittlement [8]. Alloys with lower aluminium content were found to suffer less embrittlement.

Superplastic behaviour is usually observed in materials with fine stable grain size and high strain rate sensitivity [16]. The tensile ductilities shown in Fig. 2 suggest that the Cr and Cr + Zr alloys tend to behave superplastically above 900° C, while the unalloyed aluminide exhibits relatively poor ductility. The strain rate sensitivities of the alloyed materials are slightly higher than that of the unalloyed material; however, the data indicate that the unalloyed Ni_3Al has sufficiently high strain rate sensitivity to tend toward superplastic behaviour. Grain growth of the extruded materials was measured after 1 h annealing times at temperatures where superplastic behaviour is expected. These data are shown for Ni_3Al and the chromium-containing alloy in Fig. 4. It was difficult to quantitatively determine the grain size of the Cr + Zr alloy because of the very fine two-phase microstructure, but it appears to behave in a manner similar to the chromium-containing alloy. It can be seen from the figure that the unalloyed aluminide has a fine grain size initially and grain growth does not appear to be excessive in the temperature range from 900 to 1000° C for either alloy. It appears that the unalloyed material would behave superplastically except for the intervention of brittle fracture, possibly from oxygen embrittlement.

The strain rate sensitivity exponents for all three materials approach 0.5 for the lowest strain rates above 900° C. This value is usually taken to indicate that the dominant deformation mechanism is grain-boundary sliding [16]. Optical microscopy of polished cross-sections from samples deformed superplastically to large strain showed some wedge cracking at grain boundaries that appears to result from grain-boundary sliding. The overall shape of the strain rate curves is also normally described as showing a transition from grain-boundary sliding controlled deformation at low strain rates to dislocation flow at the higher strain rates [17].

5. Conclusions

The fine-grained materials from extruded aluminide powders have room-temperature strength above that which is usually obtainable with cast materials while retaining good ductility. The increment of increase in yield stress on increasing temperature is relatively small for the powder materials.

Alloying with chromium suppresses the amount of dynamic embrittlement observed near 700° C. The two-phase alloy containing chromium and zirconium shows very little evidence of dynamic oxygen embrittlement.

The alloys containing Cr and Cr + Zr exhibit superplastic behaviour above 900° C. The dominant mechanism of superplastic deformation is grain-boundary sliding. Unalloyed Ni_3Al has the characteristics necessary for superplasticity; however, oxygen embrittlement appears to prevent deformation to large plastic strains.

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