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The influence of grain size and composition on 1000 to 1400 K slow plastic flow properties of NiAl

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The compressive slow plastic flow behaviour of several B2 crystal structure NiAl intermetallics has been studied in air between 1000 and 1400 K ($\sim$0.53 to 0.74 T_M). Small grain-sized Ni–48.25 at % Al ($\approx$ 5 and 10 μm) was found to be stronger than the previously studied 17 μm diameter material. While grain refinement improved the strength at all test temperatures, the exact mechanism is not clear. Experiments at lower temperature revealed that composition as well as grain size can be an important factor, since Ni–49.2 Al was weaker than Ni–48.25 Al. Pronounced yield points were found during slow strain-rate testing at 1000 K; however, continued deformation appears to take place by the same mechanism(s) as found at high temperatures. Small changes in thermomechanical processing (TMP) schedules to fabricate Ni–49.2 Al indicated that basic deformation characteristics (stress exponent and activation energy) are not affected; however, the pre-exponential term could be modified if TMP alters the grain structure.

1. Introduction

A recent study [1] of the elevated-temperature slow plastic strain-rate properties of polycrystalline NiAl between 1200 and 1400 K indicated that strength was not dependent on grain size or chemistry when the average grain diameter was $\geqslant 15\,\mu$m. However, it was noted that it might be possible to strengthen this B2 crystal structure intermetallic by decreasing the grain size. This hypothesis was drawn from the observation that NiAl forms sub-boundaries during creep deformation. If a stable grain structure could be maintained that was smaller than the equilibrium substructure size, strengthening should take place. Indeed Vedula *et al.* [2] have already presented data that showed the 1300 K flow stress in Ni–50 at % Al for a strain rate of $\sim 2 \times 10^{-6}\,\text{sec}^{-1}$ more than doubled from 15 to 33 MPa when the grain size was decreased from 48 to 2 μm. This behaviour was not, unfortunately, consistent as they also found that similar changes in grain size had essentially no effect on the flow strength of either Ni–48 Al or Ni–52 Al.

In principle the transition from subgrain size control to grain-size domination should be easily detected from the stress–strain-rate behaviour, since the stress exponent for creep would increase in value from about 5 to approximately 8 [1]. Unfortunately small grain-size materials under elevated-temperature creep environments are apt to deform via diffusional creep as well as dislocation mechanisms. If the former make a significant contribution to the overall creep rate, the measured stress exponents could be misleading. The relative importance of diffusional mechanisms could be lessened, however, by lower temperature and/or faster strain-rate experiments.

This paper describes a study of the effects of small grain sizes ($\leqslant 15\,\mu$m) on the elevated-temperature slow plastic flow properties of Ni–48.25 at % Al between 1000 and 1400 K (approximately 0.53 to 0.74 of the solidus point). This work was undertaken to determine (i) if fine-grain microstructures can produce effective strengthening at high temperatures, and (ii) the mechanism(s) responsible for any improvement in properties. Materials were fabricated by hot extrusion of prealloyed powders. Both as-extruded and heat-treated aluminides were tested in air at strain rates ranging from 10^{-4} to $10^{-7}\,\text{sec}^{-1}$. Additional testing was also conducted on a Ni–49.2 at % Al aluminide produced by hot extrusion of blended prealloyed powder to examine if extrusion geometry affected the mechanical properties.

2. Experimental procedures

Heats of gas-atomized prealloyed powder having the compositions listed in Table I were procured from Alloy Metals Inc., Troy, Michigan. Fully dense materials, suitable for mechanical property testing, were fabricated by hot-extruding 76 mm outside diameter, 6.4 mm thick wall steel cans which had previously been filled with about 1200 g of powder and encapsulated under vacuum. Round-to-round extrusions were undertaken at a 16 : 1 reduction ratio and several temperatures (Table II). The higher processing temperature (1420 K) for Ni–49.2 Al, produced by the blending of appropriate amounts of Heats A12312 and A12316, was selected to agree with the method utilized for the similar composition alloy [1] extruded from 51 mm diameter tooling. The lower temperature was chosen to yield a smaller as-extruded grain size in

TABLE I Composition of NiAl aluminide powder

Heat No.	Composition (at %)					
	Al	Ni	C	H	N	O
A12312	52.7	Bal.	0.01	0.05	0.004	0.14
A12316	43.9	Bal.	0.011	0.02	0.004	0.09
10487	48.25	–	0.003	–	–	0.07

Ni–48.25 Al [3] but maintain the extrusion pressure beneath the press limit of 1310 MPa.

Cylindrical test specimens 10 mm long with a 5 mm diameter were prepared by electrodischarge machining and grinding with their length parallel to the extrusion direction. Samples were taken from both as-extruded aluminides and Ni–48.25 Al after being heat-treated for 16 h at 1390 K. Constant-velocity compression tests at speeds ranging from 2.12×10^{-3} to 2.12×10^{-6} mm sec^{-1} were conducted in a universal test machine to ~10% or more strain between 1000 and 1400 K in air. The autographically recorded load–time charts were converted to true compressive stresses, strains, and strain rates via the offset method and the assumption of conservation of volume. Additional details concerning specimen fabrication and test procedures can be found elsewhere [3, 4].

3. Results

3.1. Materials

Both visual and materialographic examination demonstrated that the as-extruded and heat-treated intermetallics were fully dense and crack-free. The equiaxed grain structure of Ni–49.2 Al (Table II) was slightly smaller than that found in the similar composition aluminide extruded from 51 mm tooling at 1420 K. The lower extrusion temperature for Ni–48.25 Al yielded a ~5 μm equiaxed grain size which was approximately doubled by the 16 h–1390 K anneal (Table II); the average diameter of these latter materials was significantly smaller than the previously tested 1505 K extruded Ni–48.25 Al [1]. X-ray diffractometer and (1 1 0) pole figures revealed the presence of minor (1 1 0) wire textures in both as-extruded intermetallics.

3. 2. Mechanical properties

3.2.1. Stress–strain

The true compressive stress–strain diagrams illustrating the 1000 K behaviour for the as-extruded and heat-treated aluminides are presented in Fig. 1. Two types of curves were observed: (i) yielding followed by flow at a constant or very slowly decreasing stress (Ni–49.2 Al and heat-treated Ni–48.25 Al, Figs 1a and c; and (ii) continued flow at a more or less steady stress (Ni–48.25 Al at an approximate strain rate of 2.2×10^{-6} sec^{-1}, Fig. 1b). These particular curves illustrate extremes in behaviour; testing between 1100 and 1400 K invariably yielded plastic deformation at a nearly constant stress level except for a few of

TABLE II Thermomechanical processing schedule and initial grain size of NiAl aluminides

Composition	Extrusion temperature (K)	Heat treatment	Equiaxed grain size (μm)
Ni–49.2 Al	1420	–	17
Ni–49.2 Al [1]	1420	–	14
Ni–48.25 Al	1100	–	5
Ni–48.25 Al	1100	16 h at 1390 K	9
Ni–48.25 Al [1]	1505	–	18

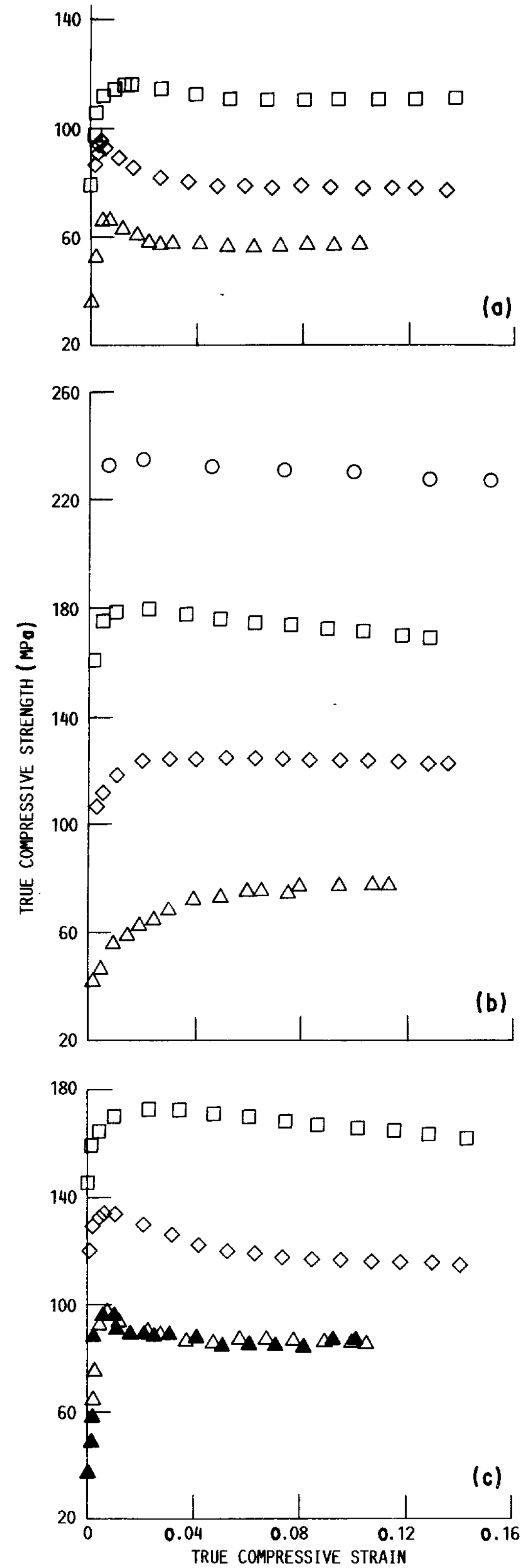

Figure 1 True compressive stress–strain curves for 1000 K-tested nickel aluminides: (a) 1420 K-extruded Ni–49.2 Al, initial grain size of 17 μm; (b) 1100 K-extruded Ni–48.25 Al, initial grain size of 5 μm; (c) heat-treated Ni–48.25 Al, initial grain size of 9 μm. Approximate strain rate (sec^{-1}): (○) 2.2×10^{-4}; (□) 2.2×10^{-5}; (◇) 2.2×10^{-6}; (▲, △) 2.2×10^{-7}.

the highest temperature, slowest strain-rate experiments. These strain-hardened, with the rates of work-hardening generally diminishing with deformation such that the stress required for further plastic flow only slowly increased after ~ 5% strain.

There does not seem to be any pattern to the various types of 1000 K stress–strain curves shown in Fig. 1 with respect to composition or grain size. While as extruded Ni–48.25 Al (Fig. 1b) basically deforms at a nearly constant flow stress for each imposed strain rate, the heat-treated version (Fig. 1c) and Ni–49.2 Al (Fig. 1a) possess yield points which for the slower rates of deformation are at least 10% higher than the stresses required for continued deformation. At faster strain rates, however, yielding appears to be much less pronounced.

Comparison of the 1000 K strengths of the various materials in Fig. 1 indicates that at each strain rate the smaller-grain-size Ni–48.25 Al aluminides are considerably stronger than the larger-grain-size Ni–49.2 Al. Although yielding somewhat confuses the 1000 K properties, as-extruded Ni–48.25 Al is slightly stronger than the heat-treated material at faster deformation rates; however, the opposite is true at the slower strain rates. These behaviours with respect to grain size and heat-treatment condition are basically sustained at the higher test temperatures.

3.2.2. Stress–strain rate

True compressive stress–strain-rate data for the aluminides are presented in Figs 2a, b and c where the flow stresses σ and strain rates $\dot{\varepsilon}$ were taken from the individual stress–strain diagrams. For the 1100 to 1400 K testing σ and $\dot{\varepsilon}$ are average values from the more or less constant flow regimes or are values taken at about 10% strain for continuous-work curves. These latter data points are designated by the half-filled symbols in this figure. Because of the diverse nature of some of the 1000 K stress–strain curves (Fig. 1), a range of data (stress and stain rate at yielding (filled circles) and at ~ 10% deformation (open circles)) is shown for this temperature, when necessary. Fig. 2d illustrates the behaviour of as-extruded Ni–48.25 Al which had been fabricated by 1505 K compaction and extrusion at 16 : 1 from 76 mm tooling [1].

The data in Fig. 2 reinforce the contention that the smaller-grain-size materials (Figs 2b and c) are stronger than the bigger-grain-diameter aluminides (Figs 2a and d) for all test conditions. Additionally, comparison of the behaviours of the 1100 K as-extruded (Fig. 2b) and the heat-treated (Fig. 2c) forms of Ni–48.25 Al illustrate the switching in strength for temperatures $\leqslant$ 1300 K where the as-extruded stock is slightly stronger at fast strain rates and somewhat weaker at the slower deformation rates. At 1400 K the latter two materials have equivalent properties, but they are considerably stronger than either Ni–49.2 Al or the 1505 K extruded Ni–48.25 Al.

The stress–strain-rate data in Fig. 2 were fitted to the standard power law and temperature-compensated power law rate expressions

$$\dot{\varepsilon} = A\sigma^n \qquad (1)$$

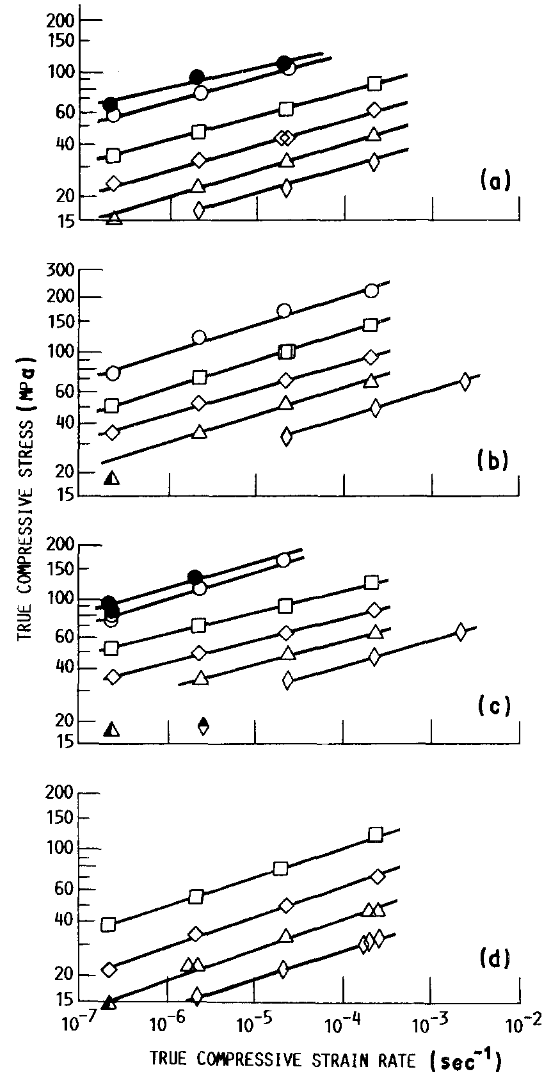

Figure 2 True compressive strain-rate–stress behaviour of nickel aluminides as a function of temperature: (a) 1420 K-extruded Ni–49.2 Al, initial grain size of 17 μm; (b) 1100 K-extruded Ni–48.25 Al, initial grain size of 5 μm; (c) heat-treated Ni–48.25 Al, initial grain size of 9 μm; (d) 1505 K-extruded Ni–48.25 Al, initial grain size of 18 μm. (●, ○) 1000 K; (□) 1100 K; (◇) 1200 K; (△) 1300 K; (◇) 1400 K. At 1000 K filled circles represent data taken from yield points while open circles indicate data after ~ 10% strain.

$$\dot{\varepsilon} = B\sigma^n \exp(-Q/RT) \qquad (2)$$

where A and B are constants, n is the stress exponent, Q is the activation energy for creep, R is the gas constant and T is the absolute temperature. Fits were accomplished by linear regression techniques, and the results of these calculations for A, B, n and Q, as well as the standard deviations δ_n and δ_Q for the stress exponent and activation energy, respectively, and the Coefficients of Determination R_d^2 are given in Table III. Due to yielding in Ni–49.2 Al and heat-treated Ni–48.25 Al, fits to Equation 1 at 1000 K were undertaken for both the yield point–yielding strain rate and stress and strain rate at ~ 10% deformation data. Also because of the uncertainty in the 1000 K behaviour, Equation 2 was only applied to the higher temperature data.

3.3. Post-test microstructures

Longitudinal sections of selected specimens were materialographically prepared and examined both in

TABLE III Summary of regression fits for aluminides

Identification	Temperature (K)	A (sec^{-1})	B (sec^{-1})	n	δ_n	Q (kJ mol^{-1})	δ_Q (kJ mol^{-1})	R_d^2
(a) Power law								
Ni–49.2 Al	1000*	8.16×10^{-22}		7.89	1.07			0.982
	1000†	2.23×10^{-19}		6.85	0.18			0.999
	1100	5.12×10^{-18}		6.95	0.08			1.00
	1200	1.15×10^{-16}		6.79	0.24			0.996
	1300	1.65×10^{-14}		6.04	0.28			0.996
	1400	5.34×10^{-15}		6.97	0.28			0.998
Ni–48.25 Al (extruded at 1100 K)	1000	1.02×10^{-18}		5.97	0.54			0.984
	1100	7.83×10^{-18}		6.17	0.04			1.00
	1200	1.31×10^{-17}		6.60	0.26			0.997
	1300	3.91×10^{-16}		6.32	0.45			0.995
	1400	4.18×10^{-15}		6.30	0.32			0.997
Ni–48.25 Al (16 h at 1390 K)	1000*	7.46×10^{-23}		7.78	0.43			0.994
	1000†	8.94×10^{-20}		6.45	0.33			0.995
	1100	2.02×10^{-20}		7.58	0.16			0.999
	1200	5.20×10^{-19}		7.47	0.21			0.998
	1300	2.22×10^{-17}		7.12	0.27			0.999
	1400	5.66×10^{-16}		6.87	< 0.01			1.00
(b) Temperature-compensated power law								
Ni–49.2 Al	1100 to 1400		9.62×10^{-4}	6.58	0.16	287.7	16.0	0.992
Ni–48.25 Al (1100 K-extruded)	1100 to 1400		1.47×10^{-4}	6.47	0.17	293.0	8.2	0.993
Ni–48.25 Al (16 h at 1390 K)	1100 to 1400		3.03×10^{-5}	7.41	0.12	313.0	5.4	0.998
Ni–48.25 Al (1505 K-extruded [1])	1100 to 1400		1.90×10^{-1}	5.80	0.09	318.5	5.9	0.996

*Fit of strain-rate–stress data from yield point.

†Fit of strain-rate–stress data from 10% strain.

the unetched and etched states. No evidence of grain-boundary cracking or integranular oxidation was observed; while most tested specimens had a more or less uniform oxide scale, the large-grain-size Ni–49.2 Al seemed to be less resistant at 1300 and 1400 K {for example: thicker scales and pitting} than the smaller-grain-diameter Ni–48.25 Al materials. Grain sizes were measured after deformation and are listed in Table IV. The grain diameters of Ni–49.2 Al and heat-treated Ni–48.25 Al were basically unchanged from the as-fabricated values after testing (Table I); however, some grain growth is taking place in the 1100 K as-extruded Ni–48.25 Al at 1300 K, especially at the slower strain rates, and at 1400 K. At this latter temperature both tested forms of Ni–48.25 Al possess equivalent grain sizes.

4. Discussion

While some increase in the average grain size was observed in the B2 nickel aluminides after testing at the higher test temperatures, these materials are basically resistant to grain growth (Table IV and [1, 2]). Hence improvement in elevated-temperature strength through grain refinement ($< 15\,\mu$m) seems to be a viable method to increase the slow plastic flow properties to at least 1200 K. Unfortunately, from the data in Table IV it is unlikely that very small grains ($< 5\mu$m) would be stable at higher temperatures; hence strengthening via thermomechanical processing to obtain an extremely fine microstructure could be somewhat limited. It should be noted, however, that a change in grain dimensions from about 18 μm (1505 K extruded Ni–48.25 Al) to 10 μm (1100 K extruded and heat-treated Ni–48.25 Al) still can result in significant strengthening even at 1400 K (Fig. 2); thus any decrease in sizing could be worthy of exploitation. Additionally a smaller grain size might have the secondary benefit of increasing the low-temperature ductility of NiAl [5].

The cause of the grain-size strengthening effect is unclear. Comparison of the activation energies and stress exponents of the Ni–48.25 Al materials (Table III) reveals that the Q values are nearly equal while differences in n exist, where the stress exponents for the smaller-grain-size forms are higher than that for the larger-grain-diameter material. While this was expected, the finding that the approximately 10 μm aluminide has greater stress exponents than the $\approx 5\,\mu$m intermetallic, irrespective of the test temperature or temperature range, is difficult to understand. Also the observation that the greatest effect of a decreasing grain size on strength occurs at the faster strain rates (Fig. 2) argues that a direct grain-size mechanism, such as the Hall–Petch seen in B2 FeAl [6], is taking place. Neither of these observations can be explained on the basis of a grain-size affected subgrain boundary creep mechanism in NiAl [1], since this model would predict that the stress exponent would raise from 5 to 8 as the grain size decreased, and the largest effect would be seen at the lowest strain rates.

Unfortunately the present data are insufficient to plainly define the role of grain size on the elevated-temperature slow plastic flow behaviour. It is quite probable that three grain-size-affected deformation mechanisms (Hall–Petch, subgrain formation and diffusional creep) are simultaneously operating in NiAl, and the competition among them leads to confusing behaviour.

The mechanical behaviour of the nickel aluminides at 1000 K seems to be different from that at higher temperatures. Previous 1100 to 1400 K testing of NiAl materials [1], as well as the current results, generally had stress–strain curves showing more or less constant

TABLE IV Grain size after testing

Test conditions		Average grain diameter (μm)		
Temperature (K)	Approximate strain rate (sec^{-1})	1420 K-extruded Ni–49.2 Al	1100 K-extruded Ni–48.25 Al	Heat-treated Ni–48.25 Al
1000	2.2×10^{-5}	15	–	8
	2.2×10^{-6}	18	5	9
	2.2×10^{-7}	15	5	9
1100	2.2×10^{-4}	14	4	9
	2.2×10^{-7}	15	6	9
1200	2.2×10^{-4}	14	5	8
	2.2×10^{-7}	18	6	9
1300	2.2×10^{-4}	17	8	9
	2.2×10^{-6}	18	11	10
	2.2×10^{-7}	18	12	11
1400	2.2×10^{-3}	18*	10	9
	2.2×10^{-5}	19	11	10
	2.2×10^{-6}	26	–	–

*Strain rate $\sim 2.2 \times 10^{-4}$ sec^{-1}.

flow stresses. While examples of yielding were also observed in a few fast-strain-rate experiments, the yield stresses were within a few MPa of that necessary for continued deformation. The yield phenomenon at 1000 K is much more pronounced, occurs at the lower strain rates and is reproducible (Figs 1a and c). On the basis of the present data it appears that yielding is not dependent on the Al/Ni ratio (it is not present in 1100 K extruded Ni–48.25 Al but was found in the heat-treated version of this material, Figs 1b and c); however it is possible that prior exposure to a temperature of the order of 1400 K could have precipitated a second phase or promoted a Cottrell–Jaswon type of locking mechanism which then led to the observed effect.

While the cause of yielding at 1000 K is not known, it is probable that continued slow plastic deformation at this temperature is controlled by the same mechanism(s) operating at the higher temperatures. This conclusion is based on the data in Table V where the extrapolated strengths at 1000 K, calculated from the temperature-compensated power law regression fits (Table III), are compared with the actual values. The closeness of the estimated data to the experimental quantities implies that deformation at 1000 K is an elevated-temperature process.

The previous study [1] of the 1200 to 1400 K mechanical properties of B2 NiAl materials concluded that strength was independent of composition for Al/Ni $\leqslant$ 1.03. The present results show that this statement is not correct at lower temperatures. For the similar large grain-size intermetallics (Table II), chemistry does influence strength at 1100 K where the 1505 K-extruded Ni–48.25Al (Fig. 2d) is stronger than 1420 K-extruded Ni–49.2 Al (Fig. 2a). Additionally the estimated strengths for 1505 K-extruded Ni–48.25 Al at 1000 K (Table V) indicate that it will be much stronger than Ni–49.2 Al at this temperature. These observations are in basic agreement with the findings of several others [7, 8] who noted that stoichiometric or near-stoichiometric nickel aluminides were the weakest at temperatures below 1200 K. While there can be differences in strength due to chemistry, such desparities are apparently minimized as the temperature is increased and/or the strain rate is decreased (Fig. 2 and Table V).

Finally, comparison of the flow stress–strain-rate data for the present Ni–49.2 Al aluminide, extruded from 76 mm tooling, and a previously tested Ni–49.2 Al material [1] extruded from a 51 mm diameter can, allows some estimate to be made about the influence of thermomechanical processing (TMP). While both billets were subjected to the same extrusion schedule (1420 K at 16:1 reduction ratio), there is a small

TABLE V Comparison of predicted* and actual flow strength at 1000 K

Material	Strength (MPa) at different strain rates					
	2.2×10^{-5} sec^{-1}		2.2×10^{-6} sec^{-1}		2.2×10^{-7} sec^{-1}	
	Predicted	Actual	Predicted	Actual	Predicted	Actual
1420 K-extruded Ni–49.2 Al	108	111	76	78	54	57
1100 K-extruded Ni–48.25 Al	173	178	121	124	85	77
Heat-treated Ni–48.25 Al	154	172	113	115	83	85
1505 K-extruded Ni–48.25 Al	155	–	104	–	70	–

*Predictions based on the temperature-compensated power law fits, Table III.

difference in the as-extruded grain sizes with the current intermetallic possessing a slightly larger structure (17 μm compared with 14 μm, Table II). Statistical testing of the data based on Equation 2 with a dummy variable for processing condition and processing-dependent stress exponents and activation energies revealed that there was a statistical disparity between the two TMP schedules. While n and Q were found to be independent of the billet size, the dummy variable proved to be significant (99% level). This was reflected in the pre-exponential term B, where it is somewhat greater for the 76 mm extrusion than the 51 mm one (0.004 compared with 0.003). Most likely this difference is simply a reflection of the initial average grain diameters; hence it appears that the mechanical properties are independent of thermomechanical processing unless it modifies the grain structure.

The results of this study suggest several areas for future work. The role of grain size in the elevated-temperature mechanical properties should be more completely investigated using secondary mechanical working, perhaps in combination with heat treatment, as a means to refine the grain structure. While lower extrusion temperatures would also result in smaller grain sizes, 1100 K represents the practical limit for the present steel cans and tool-steel extrusion punches; hence weaker canning materials or geometry would be necessary for lesser temperatures. Thermomechanical processing alone will, however, not be sufficient to prevent grain growth at elevated temperature; thus some mechanism to stabilize the grain structure will be required. While alloying to induce precipitation of a second phase might be useful, solubility at elevated temperatures could limit its applicability. Oxide dispersion strengthening, perhaps, would offer a more effective mechanism to maintain a fine-grain structure since the dispersoid is not soluble. In any case care must be taken that the strengthening effects of small grain size are not outweighed by diffusional creep.

5. Conclusions

Based on a study of the slow plastic compressive flow properties of several B2 NiAl intermetallics between 1000 and 1400 K, it is concluded that

(i) decreasing the grain size below $\approx 15\,\mu$m will increase the strength, and

(ii) chemistry does influence behaviour for temperatures $\leqslant 1100$ K.

References

1. J. D. WHITTENBERGER, *J. Mater. Sci.* **22** (1987) 394.
2. K. VEDULA, V. PATHARE, I. ASLANIDIS and R. H. TITRAN, in "High-Temperature Ordered Intermetallic Alloys", edited by C. C. Koch, C. T. Liu and N. S. Stoloff (Materials Research Society, Pittsburgh, 1984) pp. 411–421.
3. J. D. WHITTENBERGER, *Mater. Sci. Eng.* **57** (1983) 77.
4. *Idem, ibid.* **73** (1985) 87.
5. E. M. SCHULSON and D. R. BARKER, *Scripta Metall.* **17** (1883) 519.
6. J. D. WHITTENBERGER, *Mater. Sci. Eng.* **77** (1986) 103.
7. R. R. Van Der VOORT, A. K. MUKHERJEE and J. E. DORN, *Trans. ASM* **59** (1966) 930.
8. R. T. PASCOE and C. W. A. NEWEY, *Met. Sci. J.* **2** (1968) 138.

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