INDUSTRIAL SCALE PROCESSING AND ELEVATED TEMPERATURE

PROPERTIES OF $\text{Ni}_3\text{Al-Cr-Zr-B}$ ALLOYS*

V. K. Sikka

Metals and Ceramics Division
Building 4508; MS 083
Oak Ridge National Laboratory
P. O. Box 2008
Oak Ridge, Tennessee 37831

E. A. Loria

Metallurgical Consultant
1828 Taper Drive
Pittsburgh, Pennsylvania 15241

Abstract

This study is the first to evaluate the elevated temperature properties of two of the strongest $\text{Ni}_3\text{Al-Cr-Zr}$ alloys when made in sufficient quantities and processed to products simulating industrial-scale operations. The technologies considered are powder processing, melting techniques, near-net-shape processes, and cold and hot working of cast products. The tensile properties from ambient to 1100°C and creep rupture properties in the range of 704 to 1093°C are presented. These properties are most sensitive to the grain size differences produced by the various processing techniques. The results compare favorably with those obtained on commercial superalloys and reveal good potential for elevated temperature structural use.

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Alloys based on the ductile-ordered intermetallic compound Ni_3Al are being considered for a range of structural applications (1-3). These include gas and steam turbines, automotive pistons, turbochargers and valves, heating elements for appliances, aircraft fasteners, gas and oil well tubular products, and components for corrosive environments. Acceptance will depend on the development of the processing technology and the mechanical properties of various products for these applications. With this in mind, a study was conducted on the effect of processing scaled up quantities of two $\text{Ni}_3\text{Al-Cr-Zr-B}$ alloys by six different methods and determining their elevated temperature tensile, creep, and rupture properties. The two alloys (IC-218 and IC-221) provide improved high-temperature strength via solid solution-hardening effects of 0.8 or 1.7 wt % Zr, while the 7.8 wt % Cr alleviates dynamic embrittlement by changing the oxidation process. To accommodate these elements, the aluminum content is reduced from 11.3 to 8.5 wt % and boron remains at 0.02 wt %. The IC-218 and IC-221 alloys are primarily ordered structure (γ' -phase) with small amounts of the disordered structure (γ -phase) at room temperature. The fraction of disordered structure in these alloys increases at high temperatures (greater than 1000°C). On the other hand, IC-50 (11.3Al-0.6Zr-0.02B) has only the ordered structure (γ' -phase) up to its melting point, but it lacks the high-temperature ductility of the chromium-containing alloys.

Materials and Processes

This study is the first to evaluate materials made in sufficient quantities and processed to products simulating industrial-scale operations. Because of expected restricted hot workability and its negative effect upon a number of possible applications, various processing techniques have been explored for the determination of elevated temperature properties. These include powder metallurgy (PM) techniques utilizing 125 to 250 kg of powder from two atomizing sources which were either hot isostatic pressed (HIP) or consolidated under atmospheric pressure (CAP) and then extruded to bar stock. Also, small isothermally forged disks and rapid omnidirectional compacted (ROC) disks were produced from the two PM processes. Melting and casting 50- to 250-kg air-induction melt (AIM) or vacuum-induction melt (VIM) heats as well as duplexing via electroslog remelting (ESR) provided cast billet and hot extruded products. Cast tube hollows of 125-mm OD and 25-mm wall were produced from argon-induction melting of 250-kg heats as well as 15-kg castings and twin roller cast sheet of 200- to 250-mm width and 1.5- to 2.0-mm thickness were produced from 50-kg AIM heats. Flowcharts describing the status of each of these processes investigated by the Oak Ridge National Laboratory (ORNL) in conjunction with industry have been presented (4).

For testing purposes, sheet specimens were used principally to reduce machining cost. This was the case for the VIM-extruded, ESR-extruded, AIM-extruded, cast billet-cold rolled, twin-roller sheet, and PM-extruded specimens. Although extruded products were sheet or round bar, sections from these were cold rolled and annealed to a thickness of 0.76 mm. Round specimens of 6.4-mm diam were prepared from cast billet as well as 3.2-mm-diam subsize rounds which were cut from an isothermal and an ROC disk prepared from the PM products. Tensile tests were conducted on four Instron machines, using sheet specimens of 0.76-mm thickness by 25-mm gage length. In all cases, a constant crosshead rate was used rather than strain rate. Most commonly used was $8.3 \times 10^{-4}/\text{s}$ (because of ASTM requirements), but $3.3 \times 10^{-3}/\text{s}$ was used on some tests. Several tests were conducted at a range of strain rates to explore superplasticity and effects on strength and ductility. All creep tests were constant load tests. Those on sheet specimens were conducted using dead-load creep machines. Creep tests on 6.4-mm-diam round

specimens were conducted using a lever-arm machine with lever-arm ratios of 12:1 or 20:1. For all sheet specimens, strain was measured by extension of the pull rod by a mechanical dial gage. For round specimens, an attached strain-averaging extensometer provided strain measurements. For all cases, strain-time data were computer plotted and analyzed for minimum creep rate and other quantities such as start of second-state creep, start of third-stage creep, etc. Ruptured specimens were measured for fracture strain and reduction of area.

Results

The grain size of six products of IC-218 vary between 9 and 21 μm for three different PM processes, cast and hot extruded and cast and cold-rolled sheet versus 727 μm in cast tube hollow, per Figure 1. The tensile

Figure 1 - Optical microstructure of IC-218 alloy specimens processed by six different processes. (a) Cast tube hollow, grain size = 727 μm ; (b) cast and hot extruded, grain size = 11 μm ; (c) cast and cold-rolled sheet, grain size = 16 μm ; (d) powder extruded, grain size = 16 μm ; (e) powder isothermal disk, grain size = 9 μm ; and (f) powder ROC disk, grain size = 21 μm . Magnification: 500 $\times$.

properties, plotted from ambient to 1100°C, show a band of values for 0.2% yield strength, ultimate strength, and total elongation, per Figure 2. Yield strength is one of the more fundamental properties of a structural alloy. It is a property to be maximized and, in the case of elevated temperature applications, to be invariant with temperature. The yield strength of the five fine-grain products remains stable within a band of 550 to 750 MPa up to 600°C, then declines gradually within 400 to 600 MPa at 800°C then declines sharply to below 100 MPa at 1100°C. The coarse-grain tube hollow has the lowest yield strength of all products at room temperature of 450 MPa, which rises gradually to the highest value of all products of 680 MPa at 800°C and then declines to still the highest value of 250 MPa at 1100°C. The ultimate strengths of the five fine-grain products decline gradually within a band of 1250 to 1500 MPa at room temperature to 520 to

650 MPa at 800°C, while the coarse-grain cast product which has a much lower strength of 750 MPa at room temperature maintains it to 800°C. All products then decline in the same manner to 1100°C with the latter still exhibiting

Figure 2 - Tensile properties comparison for IC-218 alloys processed by six different processes. All tests were conducted at a strain rate of 0.05/min (8.3×10^{-4} /s).

the highest strength. The total elongation of all six products is within 20 to 38% at room temperature and the range then widens between 7 and 35% at 800°C in five products, followed by a sharp rise to values exhibiting superplasticity at 1000°C. The 20% elongation of the cast tube hollow only declines to 15% at 800°C and shows no superplasticity at 1075°C. Unexpectedly, the 22% elongation in cast and cold-rolled sheet product, having a fine-grain size of 16 μ m, declines continuously to a nil ductility value at 800°C and then rises sharply to a superplastic value of 260% at 1000°C.

The IC-221 alloy was processed by similar techniques used for IC-218 alloy. The same trends in grain size and tensile were observed in IC-221. When compared under the same conditions with IC-218 (i.e., fine-grain PM-extruded product), the yield and tensile strength of IC-221 are higher than IC-218, see Figure 3. The IC-221 alloy maintains its yield strength of 635 MPa up to 800°C, while IC-218 declines slightly from 525 to 435 MPa at 800°C, then both decline rapidly to 100 MPa at 1000°C. Ultimate strength declines gradually from 1500 MPa for IC-221 versus 1400 MPa for IC-218 to 690 MPa versus 550 MPa at 800°C and then to the same 120 MPa at 1000°C. Elongation values are the same at 35% at room temperature, with a gradual decrease to a minimum of 11% at 800°C in IC-218 compared to an unexpected deep decline from 28% at 600°C to 5% at 800°C in IC-221. Then, there is the usual steep ascent at 1000 to 1100°C. It is noteworthy that the room temperature ductility of the various IC-218 and IC-221 products is sufficient for conventional mill material handling and shipping operations.

The effects of grain size on tensile properties of Ni₃Al produced in small (laboratory) quantities have been extensively studied (5-6). Reducing the grain size has been found to significantly increase the room-temperature strength with relatively little effect on the ductility. Our results on industrial-scale processing of larger quantities reveal superior yield and ultimate strength from ambient to 700°C for fine-grain products versus

coarse-grain cast products, but the latter remain stable and gradually increase to the highest strength values at 800°C. This change is brought about by the increase in lattice resistance to slip and the decrease in the effectiveness with which grain boundaries impede slip as temperature rises. Above 950°C, a fine-grain product produced by either extrusion of cast billet or extrusion of powder shows superplastic behavior, whereas coarse-grain cast product shows higher strength and low ductility (2-3).

Figure 3 - Comparison of elevated temperature tensile properties of IC-221 versus IC-218 extruded products prepared under the same conditions.

positive slope line. Also, it is apparent that fine-grain material from cast billets provides higher values than fine-grain material from powder billets so that separate lines are drawn differentiating these two fine-grain products. In general, the same trends are observed in comparing IC-218 fine-grain products with very coarse-grain (6600 μm) cast billet, per Figure 5. The plotting of the data for fine-grain products of IC-218 reveals that separate lines separating 14- to 16- μm grain size from 7- to 10- μm grain size are drawn which show that the former has a higher rupture strength but lower elongation and reduction of area than the latter at the same parameter.

Figure 4 - Larson-Miller parameter of creep rupture properties of IC-221.

From this analysis, the 100- and 1000-h rupture strengths at 649, 732, 816, and 982°C (1200, 1350, 1500, and 1800°F) are listed in Table I for the various products of IC-218 and IC-221 and compared with electrosag cast IC-50. Once again, it is obvious that a coarse-grain cast structure has much superior rupture strength than fine-grain wrought material. The diffusional creep mechanism operative in the test temperature range of this investigation is believed to be responsible for such a significant grain-size effect. The 100- and 1000-h creep rupture strength of the various IC-218 and IC-221 products are compared in Figure 6 with those published for A-286, N-155, V-57, Waspaloy, and IN-100 over the 649 to 982°C (1200 to 1800°F) range. Some IC-218 and IC-221 products are superior and the rest are closely comparable to A-286, V-57, and N-155. Some of our products are even comparable to Waspaloy within this range, but above 816°C (1500°F) where the above Fe-Ni-Cr alloys are not used and Ni_3Al alloys would not be recommended, the nickel-base superalloys (IN-100 and Waspaloy) provide superior creep rupture strength. Again, in all cases, cast products provide higher creep rupture strengths than wrought products.

The Arrhenius plot of the steady-state creep rate versus temperature, Figure 7, for several products of both alloys yields an activation energy for creep of 81.7 kcal. This value

Figure 5 - Larson-Miller parameter of creep rupture properties of IC-218.

offer the best fabrication possibility via the melting route for a variety of shapes. These processes offer the best chance of success for utilization of nickel aluminides in various applications suggested for them. Hot-working processes such as hot forging and hot rolling for the cast products are still not fully developed. Hence, cold processing followed by heat treatment currently has the best chance of success. Among the hot-processing methods, hot extrusion has been investigated most thoroughly and has immediate potential for commercialization.

is higher than the published values of self-diffusion of nickel (68 kcal) and γ -iron (70 kcal) but still suggests that the rate-controlling processing in high-temperature creep is diffusion controlled. Analysis of the creep data via the Nabarro-Herring creep mechanism, Figure 8, provides reasonable agreement between predicted and observed values for different grain size materials, and the grain size dependence of creep at high temperature is explained by the Nabarro-Herring creep mechanism which involves lattice diffusion under the action of applied stress.

Discussion

This paper has presented property data from scaled up quantities of two high-strength $\text{Ni}_3\text{Al-CrZr-B}$ alloys prepared by various techniques. The tensile and creep properties are sensitive to grain-size differences resulting from the various processing techniques, but the alloys still provide an attractive alternative to certain superalloys. The current status of the processes being explored at ORNL in conjunction with industry is that powder processing is closest to commercialization and ideal for fabricating complex shapes. Nickel aluminide can be melted within specifications by the simplest method such as AIM. Secondary melting such as electroslag remelting produces ingots of most desirable grain structure for forging and extruding and of excellent surface quality. Near-net-shape processes from molten metal

Figure 6 - Comparison of 100- and 1000-h creep rupture strength of IC-221 and IC-218 with A-286, N-155, V-57, IN-100, and Waspaloy.

Figure 7 - Arrhenius plot of steady-state creep rate versus temperature.

Figure 8 - Plot showing predicted and observed rupture strength of IC-218 alloy. Values for 15- μm grain size were predicted using Nabarro-Herring equation and data on 10- μm grain size material. Comparison is for data in the temperature range of 732 to 816°C and for 100- and 1000-h rupture strength.

Table I. Creep Rupture Strength of Three Nickel
Aluminides as Affected by Processing

Temperature (°C)	Time to Rupture (h)	Stress (MPa)					
		IC-50 Electroslag Cast	IC-218			IC-221	
			Cast	Wrought		Cast	Wrought
				7-10 μm	14-16 μm		
649	1000	---	---	427	---	---	655
732	100	310	586	255	538	614	379
732	1000	207	379	138	276	393	207
816	100	172	290	93	186	296	138
816	1000	124	193	48	103	193	69
982	100	37	69	---	23	67	16
982	1000	14	26	---	---	24	---

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