1300 K compressive properties of a reaction milled NiAl–AlN composite

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Cryomilling (high intensity mechanical ball milling in a liquid nitrogen bath) of the B2 crystal structure nickel aluminide leads to a NiAl composite containing about 10 vol.% of AlN particles. This is the result of a reaction milling process, where nitrogen incorporated into the matrix during cryomilling reacts with Al during subsequent thermomechanical processing to form the composite. Compressive testing at 1300 K of such materials densified by 1505 K extrusion or isostatic pressing at 1323 K or 1623 K indicated that strength at relatively fast strain rates ($>10^{-7}$ s^{-1}) is slightly dependent on the method of consolidation. At slower rates, however, no clear dependency on densification technique appears to exist, and four different consolidation methods possessed similar creep strengths. In all cases deformation at 1300 K occurred by two distinct mechanisms: at high strain rates the stress exponent is greater than 11 while at slower rates ($<10^{-7}$ s^{-1}) a much lower stress exponent ($\sim$6) was found. Comparison of density compensated creep strengths reveals that the properties of NiAl–AlN are similar to those of the single crystal Ni-base superalloy NASAIR 100.

I. INTRODUCTION

The high melting point, low density, and excellent oxidation/corrosion resistance of NiAl make this B2 cubic crystal structure intermetallic a possible candidate for high temperature structural purposes. Its poor elevated temperature mechanical properties,[1,2] however, indicate that use of the monolithic material is improbable. A number of attempts[3-8] have been made to introduce strength through solid solution alloying and/or precipitation hardening mechanisms. While this approach was generally successful for fast deformation rates, 1200–1400 K testing revealed that the stress exponents for creep are relatively low ($\sim$3), which leads to weak alloys under slow strain rate conditions.[3,5-8] A more promising approach currently under investigation involves "traditional" composite technology, and to date nickel aluminides strengthened with HfC dispersoids,[9] TiB$_2$ particles,[10] alumina whiskers,[11] and several different types of unidirectional fibers[12] have been studied.

Recently, preliminary mechanical property data were presented[13] for a nontraditional composite processing technique which yielded a strong, high temperature NiAl-based material. Specifically, it was found that cryomilling[14] of NiAl powder followed by hot extrusion resulted in a material that is at least six times stronger than the unprocessed nickel aluminide between 1200 and 1400 K. Apparently high intensity ball milling in

liquid nitrogen (cryomilling) of NiAl leads to the formation of a NiAl–AlN composite, where a thin mantle containing a high density of small ($<$50 nm) diameter AlN particles surrounds essentially particle-free grains. While the extraordinary strength of this composite probably derives from the inability of dislocations to move freely through the particle-rich regions, confirmation of this behavior awaits study by transmission electron microscopy.

The objectives of this paper are (1) to characterize the effects of cryomilling and consolidation on the NiAl powder and (2) to present additional elevated temperature mechanical property data on cryomilled NiAl densified by several techniques. It is believed that sufficient evidence exists to show that the NiAl–AlN composite is the result of a reaction milling process similar to that utilized by Jangg to produce Al$_4$C dispersed Al[15] and by Luton *et al.*[14] to produce carbonitride strengthened Al. Furthermore, creep data will be presented to demonstrate that NiAl–AlN composites have sufficient elevated temperature strength for consideration as a structural material.

II. EXPERIMENTAL PROCEDURES

Prealloyed, gas atomized Ni–51Al was cryomilled (ball milling in liquid nitrogen[14]) with a 0.5 wt.% Y$_2$O$_3$ addition in a modified Model 1-S Szegvari 10 liter attri-

tor, as the original intention of this work was to produce an yttria dispersion strengthened nickel aluminide.[13] After 16 h of milling, the charge was allowed to warm to room temperature under dry argon and subsequently handled in air. The cryomilled powder was then vacuum canned in steel and densified by (1) hot isostatically pressing at 1323 K, 207 MPa for 5 h or 1623 K, 207 MPa for 5 h, or (2) 1505 K extrusion at reduction ratios of 8:1, 12:1, or 16:1.

Cylindrical compression specimens, whose length was parallel to the extrusion direction for the extruded materials, were electro-discharge machined and ground to final size: ~5 mm in diameter by about 10 mm in length. Compressive stress-strain behavior was determined at 1300 K in air in a universal test machine running under constant velocity conditions at crosshead speeds ranging from 2.12×10^{-3} to 8.47×10^{-7} mm/s. The autographically recorded load-time charts were converted, assuming conservation of volume, to true compressive stresses, strains, and strain rates via the offset method. Constant load compressive creep testing at 1300 K in air was also undertaken to determine the long-term (up to 1000 h) strength of the cryomilled NiAl. In this case deformation was determined as a function of time by measuring the relative positions of ceramic push bars applying load to the specimen via an LVDT connected to a data acquisition system.[16] Such information was normalized with respect to the final specimen length and converted into true stress and strains by assuming volume conservation. Selected as-fabricated materials and compression tested specimens were microstructurally characterized by standard light optical microscopy, scanning electron microscopy (SEM), and x-ray procedures.

III. RESULTS AND DISCUSSION

A. Cryomilling of NiAl

The starting prealloyed gas atomized Ni–51Al powder [Fig. 1(a)] was spherical and uniform in size with an approximately 60 μm diameter, where the internal structure [Fig. 1(b)] of each particle was, in general, composed of several grains. After cryomilling the powder was reduced in size by about one order of magnitude and was no longer uniform in shape, ranging from small 1 μm flakes to 10+ μm chunks [Fig. 2(a)]. Examination of the cross section, however, indicated that many of the large cryomilled particles were, in reality, aggre-
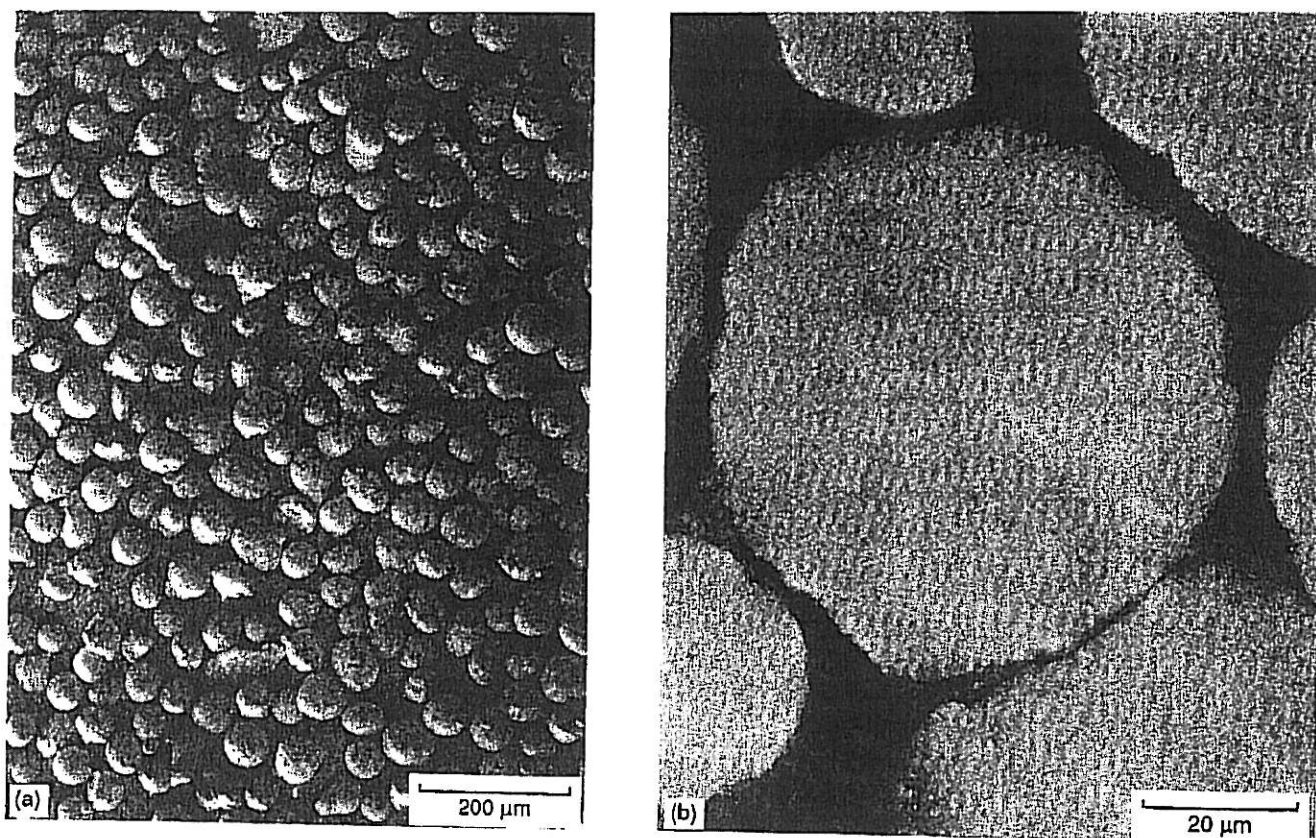

FIG. 1. Structure of as-gas atomized NiAl powder. (a) SEM photomicrograph and (b) light optical photomicrograph under differential interference conditions.
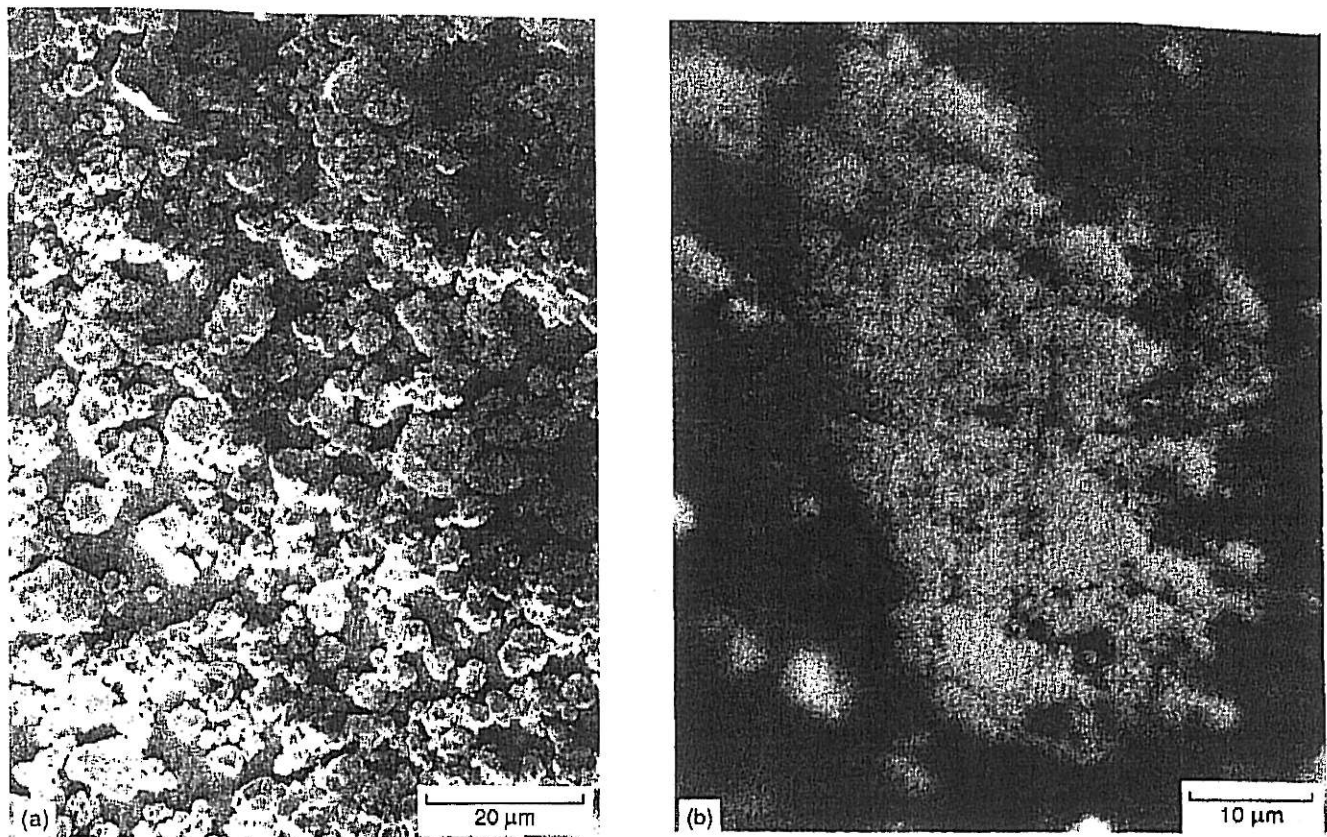
FIG. 2. Structure of cryomilled NiAl powder. (a) SEM photomicrograph and (b) light optical photomicrograph under differential interference conditions.

gates composed of the small flakes [Fig. 2(b)]. No second phase particles were visible on the surface or in the cross sections of either the as-gas atomized or as-cryomilled powder.

Wet chemical analysis of the as-received, gas atomized powder showed it to contain about 32.4 wt. % Al, 4 ppm N, and 135 ppm O. Spectrographic comparison of the Al levels of the cryomilled aluminide and the as-received powder revealed them to be nearly identical. On the other hand, the oxygen content of the cryomilled powder had increased to 0.58 wt. %, while the nitrogen level was 1.98 wt. %.

X-ray diffractometer scans[17] of the as-cryomilled powder revealed only a slight shifting to higher angles and peak broadening in comparison to those for the gas atomized powder. After 1505 K extrusion, however, distinct AlN peaks were found in the x-ray spectra.[13,17] Because AlN was not detected in the as-cryomilled powder but was readily visible after exposure to elevated temperature, we believe that the present materials are examples of the use of reaction milling to form a high temperature composite. That is, the nonequilibrium interstitial/solid solution alloying which occurs as a result of high energy mechanical milling leads to a precipitation reaction during subsequent thermomechanical processing. Furthermore, at least in this instance, reaction milling is capable of introducing relatively high volume fractions of refractory second phases. For example, based on the measured N_2 and O_2 contents, the assumption of negligible equilibrium solubilities for these elements in NiAl, handbook densities for AlN (3.27 Mg/m^3) and Al_2O_3 (3.97 Mg/m^3) and NiAl density as a function of composition data,[18] the present NiAl-AlN composites should contain about 1.3 vol. % alumina and 10.3 vol. % aluminum nitride in a Ni–47.3Al matrix. These estimates of AlN and Al content are in reasonable agreement with ones previously made on the basis of changes in the matrix lattice parameter.[13]

On a light optical level the microstructure of all consolidated materials appears as dark outlined "grains" (Fig. 3), where the outline is actually the AlN dispersed mantle surrounding particle-free regions.[13] Due to the nature of the structure, only the large particle-free grains could be characterized via a microscope; hence Table I presents a listing of the average dimensions for the largest particle-free features. In the case of the extruded materials, cigar-shaped grains [Fig. 3(a)] are observed, whose length increases and width decreases with increasing reduction ratio (Table I); presumably the aspect ratio (length/diameter) of all grains would be
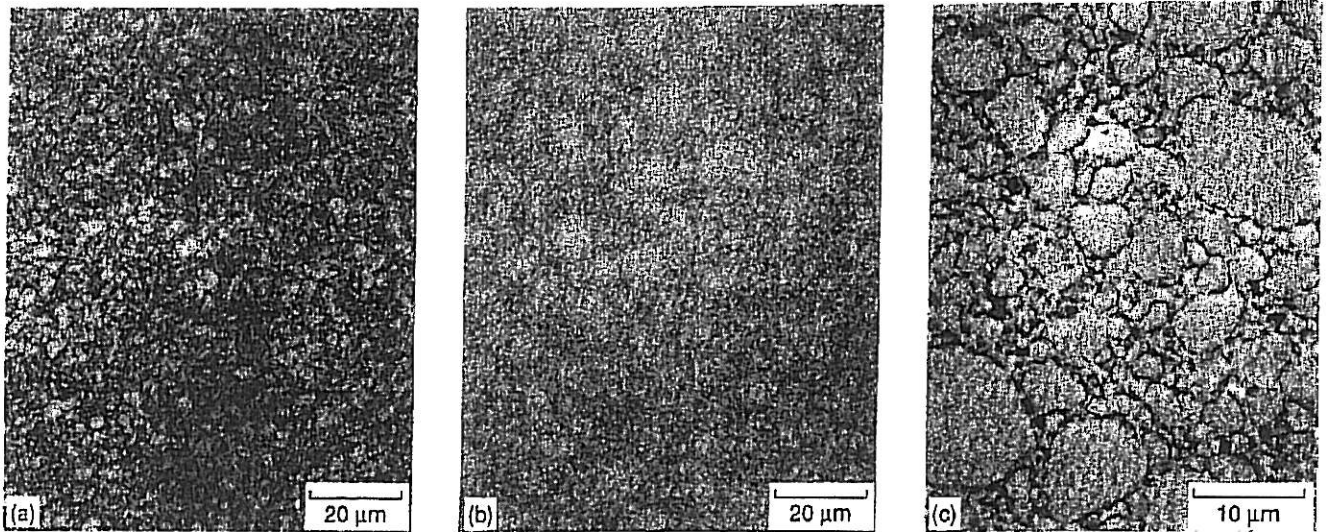
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FIG. 3. Structure of cryomilled NiAl after consolidation. (a) Light optical photomicrograph of cross section of 1505 K–12:1 extrusion, (b) light optical photomicrograph after hipping at 1323 K, 207 MPa, 5 h, and (c) SEM photomicrograph after hipping at 1623 K, 207 MPa, 5 h.

similar. The grain structure of the two hipped composites is similar, where both the 1323 K [Fig. 3(b)] and the 1623 K [Fig. 3(c)] pressing conditions yielded duplex morphologies with large (~7 μm) and small (~1 μm) approximately spherical AlN-free grains.

1. Stress-strain behavior

True 1300 K compressive stress-strain diagrams for the cryomilled Ni–51Al powder extruded at 1505 K with a 12:1 reduction ratio or hipped for 5 h at 1323 K and 207 MPa are presented in Fig. 4. Irrespective of the strain rate or densification procedure, rapid work hardening occurs over the first 1% of deformation. For the 12:1 extruded material [Fig. 4(a)], rapid strain hardening is succeeded by more gradual work hardening until either (1) a maximum stress followed by slow strain softening or (2) a constant flow stress is reached in the range of 2 to 4% deformation. The 8:1 extruded aluminide composite[19] tested at slower rates ($<10^{-5}$ s^{-1}) did

TABLE I. Characteristic dimensions of the larger particle free grains in NiAl–AlN.

Densification method	Diameter, μm	Length, μm	Length Diameter
1505 K extrusion at			
8:1	5	27	5
12:1	4	35	9
16:1	2	48	25
Hipping for 5 h @ 207 MPa and			
1323 K	7	...	...
1623 K	6	...	...

not follow this scheme; instead, the stress-strain diagrams illustrated continuous, albeit gradual, work hardening up to the end of the test (7 to 9% strain). Constant velocity testing of the NiAl–AlN composite which had been densified by 16:1 reduction at 1505 K[13] yielded true 1300 K stress-strain diagrams which are similar to those for the 12:1 extrusion [Fig. 4(a)]. In terms of resistance to deformation, the strength of the 16:1 material exceeded that of the lower reduction ratios by at least 5% at strain rates greater than 10^{-6} s^{-1}. In fact, at a deformation rate of 2×10^{-5} s^{-1}, the 16:1 processing yielded about 20% improvement (~35 MPa). Below 10^{-6} s^{-1}, the strengths of all three extruded materials became nominally equal.

The general behavior of the 1323 K hipped material [Fig. 4(b)] is similar to that of the 12:1 extruded stock [Fig. 4(a)]. However, the peak stress is reached in the range of 1 to 2% deformation [Fig. 4(b)], where the maximum occurs between 2 and 4% in the 12:1 extrusion [Fig. 4(a)]. While the 1300 K stress-strain response of the 1623 K hipped composite[19] is comparable to that of the 1323 K pressed material [Fig. 4(b)], the observed strain softening is much more severe after the higher temperature processing, and the 300 K difference in hipping temperature affected the strength. Comparison of the stress maxima shows that the 1323 K processed composite is about 20% stronger than the 1623 K hipped material at all measured deformation rates; furthermore, the strength properties after hipping at 1323 K slightly exceed those of the 1505 K, 16:1 extrusion.[13]

2. Constant load creep tests

True 1300 K compressive creep curves for NiAl–AlN composites densified by 12:1 extrusion at 1505 K
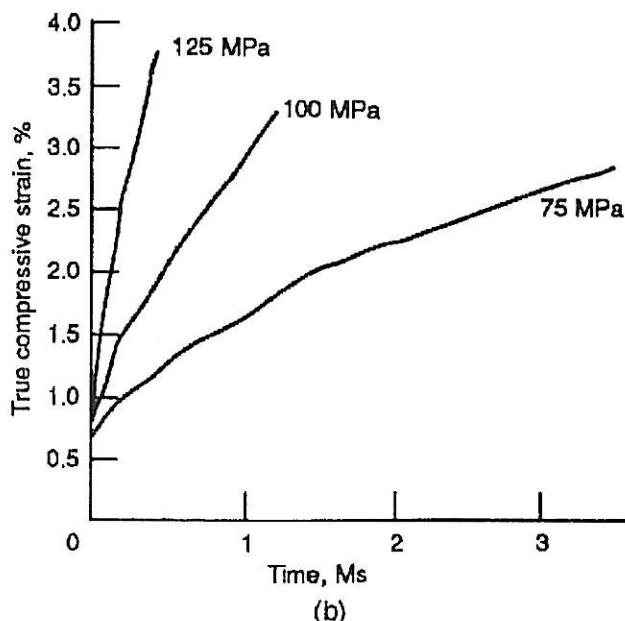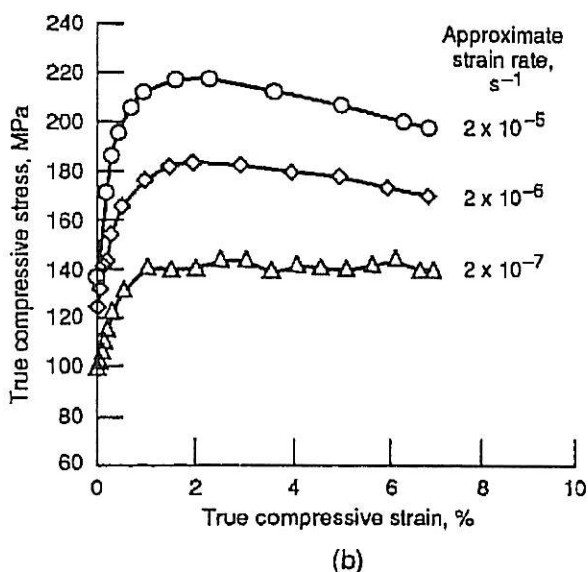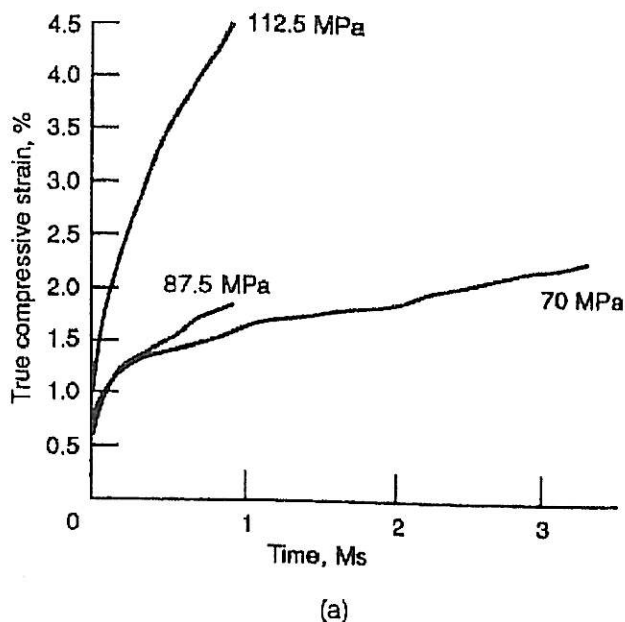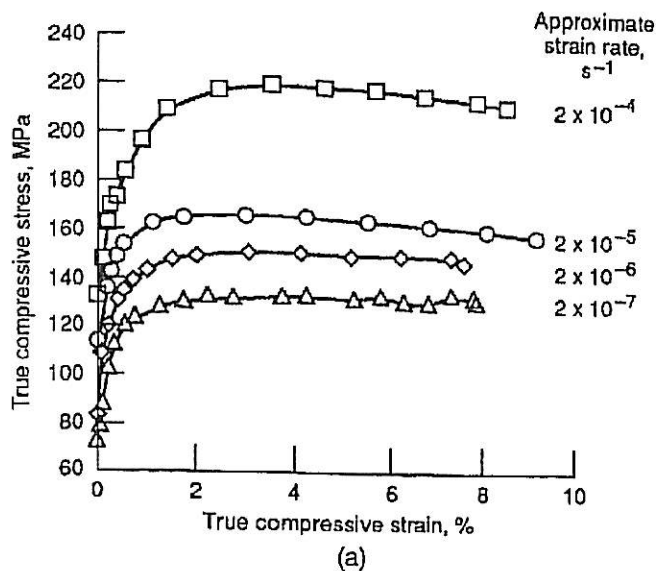
FIG. 4. True 1300 K compressive stress-strain curves for cryo-milled NiAl as a function of nominal strain rate after (a) extrusion at 1505 K–12:1 and (b) hipping at 1323 K, 207 MPa, 5 h.

FIG. 5. True compressive 1300 K creep curves for cryomilled NiAl as a function of applied stress after (a) extrusion at 1505 K–12:1 and (b) hipping at 1323 K, 207 MPa, 5 h.

and 1323 K hipping are presented in Figs. 5(a) and 5(b), respectively. In all cases the materials almost immediately (within the first few hours) deform about 1% with the application of stresses ranging from 70 to 125 MPa. Following this period of rapid deformation, work hardening was sufficient to cause normal transient creep behavior leading to rates which became, for all practical purposes, constant. While the applied stresses slightly differ for the extruded and hipped materials, comparison of the creep response in Fig. 5 indicates that both of these forms of NiAl–AlN have similar resistance to slow strain rate deformation. Compressive creep testing at 1300 K has also been undertaken on the 1505 K

−8:1 and −16:1 extruded and the 1623 K isostatically pressed composites.[19] These consolidation methods yielded similar behavior and strength levels to those shown in Fig. 5.
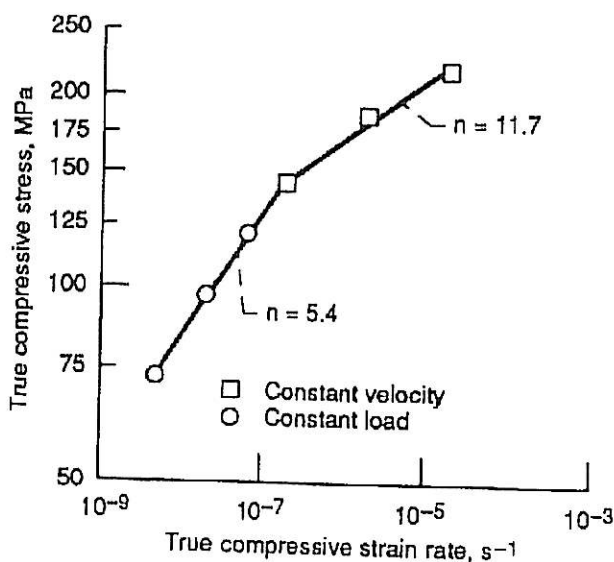
3. Flow stress–strain rate behavior

True compressive flow stress–strain rate data taken from the 1300 K constant velocity tests at 3% strain (Fig. 4) and the nominal steady state creep data from

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the 1300 K constant load tests (Fig. 5) are combined in Fig. 6. The values for the 1505 K, 12:1 material [Fig. 6(a)] and the 1323 K hipped composite [Fig. 6(b)] indicated that two deformation regimes exist with a boundary at a strain rate of ~10^{-7} s^{-1}; such behavior is in agreement with the results previously reported for NiAl–AlN densified at higher or lower extrusion ratios at 1505 K or hipped at 1623 K.[19] Following convention, the flow stresses σ and strain rates $\dot{\epsilon}$ for each regime were fitted to the standard power law equation,

$$\dot{\epsilon} = A\sigma^n, \tag{1}$$
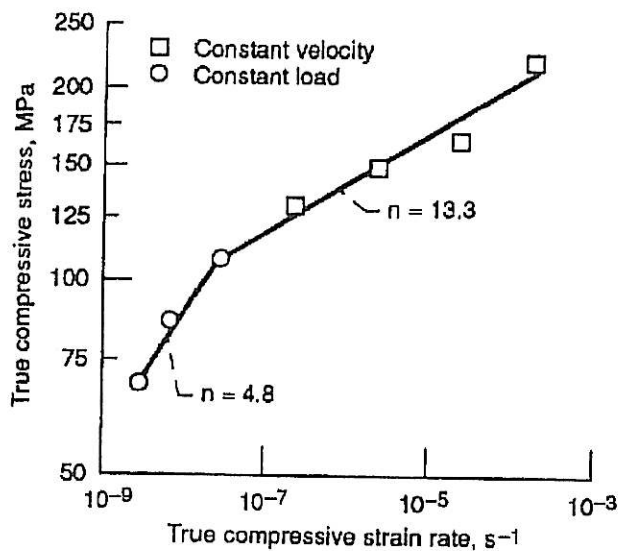
(a)

(b)

FIG. 6. True compressive 1300 K stress-strain rate behavior for cryomilled NiAl as functions of test and consolidation methods after (a) extrusion at 1505 K–12:1 and (b) hipping at 1323 K, 207 MPa, 5 h.

by linear regression techniques where A is a constant and n is the stress exponent. The graphical results of such fits are given in Fig. 6, and the values of the stress exponents, constants, and coefficients of determination, R_{d^2}, are presented in Table II.

While it could be argued that the separation into two deformation regimes shown in Fig. 6 is simply an artifact due to the change from constant velocity to constant load testing, we do not believe this to be the case. For example, a constant load data point clearly lies on the extrapolation based on constant velocity test results for the 12:1 extruded NiAl–AlN [Fig. 6(a)], and the opposite situation occurs for the 1323 K hipped material [Fig. 6(b)]. Furthermore, it was noted in Ref. 19 that several constant velocity data points deviate from the extrapolation of the high stress exponent region for the 1505 K–16:1 extruded material, and good agreement existed between both experimental procedures at a flow stress of ~125 MPa for the 1505 K–8:1 extruded material. Therefore, on the basis of the present results (Fig. 6 and Ref. 19), it appears that plastic flow in the NiAl–AlN composites at 1300 K can take place via two distinct mechanisms: under fast deformation conditions strain rate is highly dependent on stress (i.e., stress exponents >11 signifying a low temperature like behavior), while at slow strain rates ($\leq 10^{-7}$ s^{-1}) more normal high temperature creep processes ($n \approx 6$) in NiAl[1,2] seem to occur.

The densification technique does make a difference in terms of strength at fast strain rates [Fig. 7(a)], where for the present conditions two distinct groups exist. The cryomilled NiAl extruded at 1505 K–16:1 and hipped at 1323 K have nominally equal properties, while the assemblage of 1623 K hipped and 1505 K–8:1 and –12:1 extrusions is somewhat weaker. Based on the regression fits (Table II), it appears that the stress exponents for both sets of data are nominally equal. Hence the difference in deformation resistance is due to a change in the constant in Eq. (1). Below 10^{-7} s^{-1} in the low stress exponent regime, the composite flow stress seems to be less dependent on the method of consolidation [Fig. 7(b)], where all densification conditions except for the 1505 K–8:1 extrusion yield similar strengths. As was the case for fast deformation, the difference in strength at low strain rates between the 1505 K, 8:1 extruded stock and the other densification procedures appears to lie in the constant, not in the stress exponent (Table II).

B. Materialography

As the majority of the compression tested specimens are intended for transmission electron microscopy studies, only a few samples have been sacrificed for examination by light optical and SEM techniques. With only two exceptions little difference could be detected

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TABLE II. Power law fits of true compressive flow stress–strain rate data for NiAl composites.

Densification method	High stress exponent regime			Low stress exponent regime		
	A, s^{-1}	n	R_d^2	A, s^{-1}	n	R_d^2
1505 K extrusion at						
8:1	1.20×10^{-34}	13.0	0.975	2.14×10^{-18}	5.21	0.956
12:1	2.94×10^{-35}	13.3	0.971	4.94×10^{-16}	4.75	0.973
16:1	1.08×10^{-39}	14.9	0.996	9.91×10^{-23}	7.21	0.989
Hipping for 5 h @ 207 MPa and						
1323 K	1.02×10^{-32}	11.7	0.988	3.68×10^{-19}	5.40	0.993
1623 K	2.50×10^{-38}	14.7	0.994	...	...	...
Combined fits						
1323 K HIP and 16:1 ext.	1.82×10^{-37}	13.9	0.944	...	...	...
1623 K HIP, 8:1 and 12:1 ext.	2.69×10^{-35}	13.3	0.972	...	...	...
All except 8:1 ext.	...	...	...	5.19×10^{-20}	5.80	0.967

between as-densified (Fig. 3) and as-tested microstructures (Fig. 8). In general the composite/oxide interface was not smooth (Fig. 8) and contained numerous oxide protrusions into the matrix; however, no clear incidences of grain boundary oxidation were seen. Several examples of porosity were found near the air exposed surfaces both after relatively long-term [Fig. 8(a)] and short-

term[19] testing. Electron microprobe examination of the specimen shown in Fig. 8(a) revealed the existence of an Al concentration gradient of ~5 at.% between the oxide/matrix interface (low Al) and the sample interior; therefore it is possible that the pores are Kirkendall voids. These could result from the Al diffusion to the surface to re-form the alumina scale which is being con-
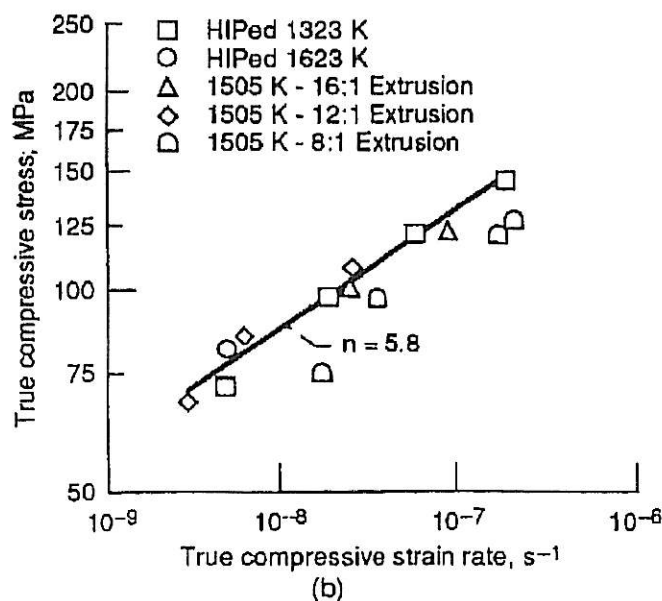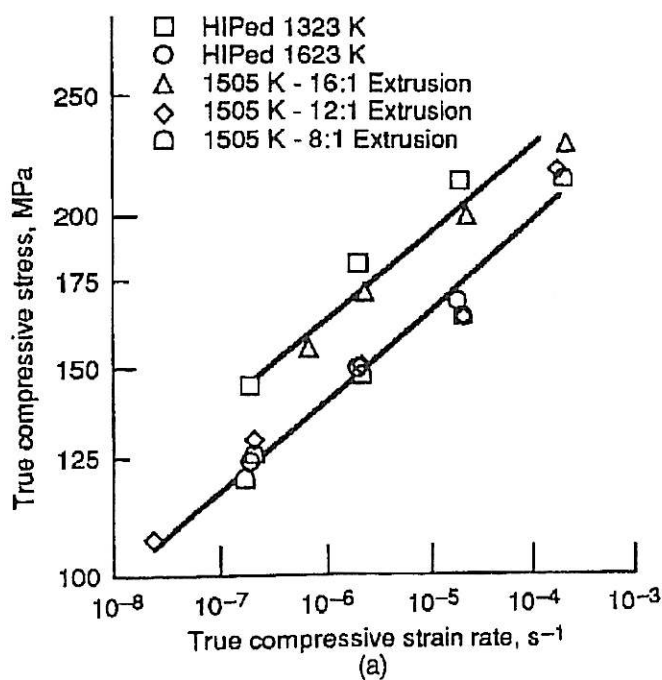

FIG. 7. True compressive 1300 K stress-strain rate behavior for cryomilled NiAl as functions of consolidation method (a) high stress exponent regime and (b) low stress exponent regime.
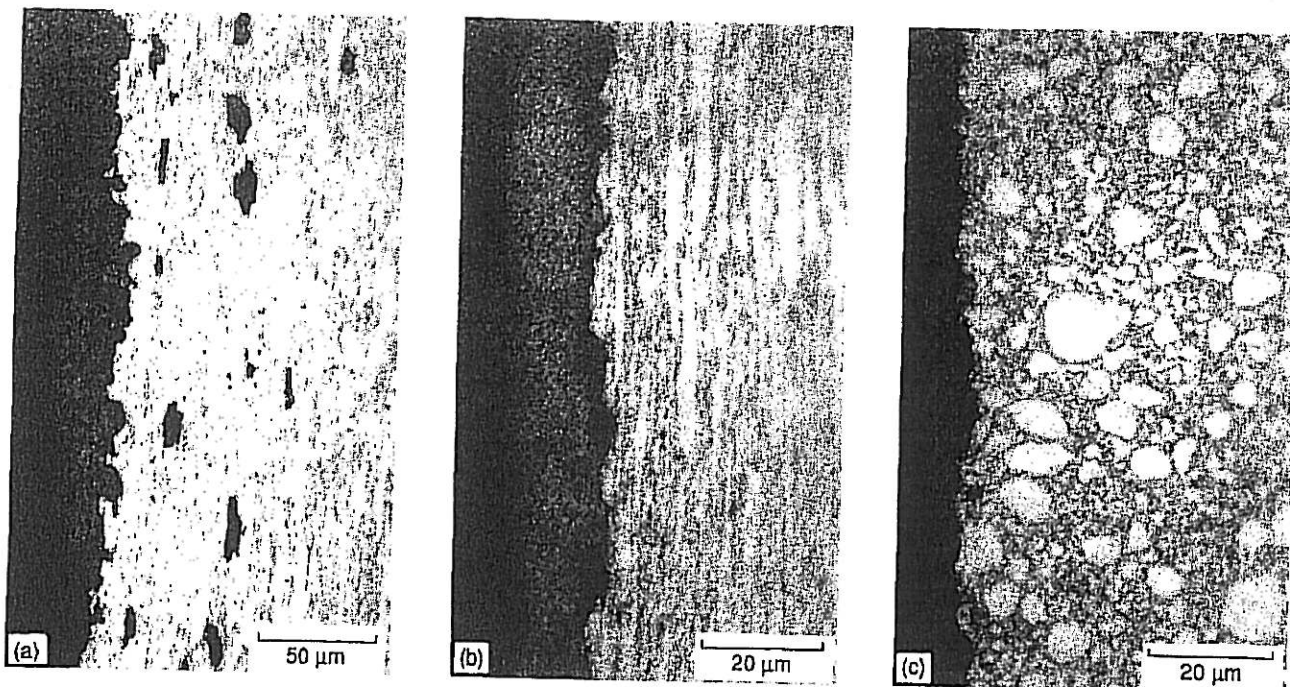
FIG. 8. Microstructure near air exposed surfaces for 1300 K creep tested NiAl–AlN composites. (a) Extruded at 1505 K–8:1 and tested at 100 MPa for 0.8 Ms to 4.8% strain, (b) extruded at 1505 K–12:1 and tested at 87.5 MPa for 0.8 Ms to 1.8% strain, and (c) hipped at 1323 K, 207 MPa, 5 h and tested at 100 MPa for 1.0 Ms to 3.2% strain. Compression axis is vertical.

tinuously cracked and spalled during compressive deformation. It should be noted, however, that this behavior was not consistent [Figs. 8(b) and 8(c)], as porosity was not observed in other long- and short-term specimens. Secondly, a few samples possessed cracks which appeared to start from the center of the specimen base, run parallel to the compression axis for a short distance (at most 2 mm), turn 90° (perpendicular to the stress), and either continue to the free surface or simply end; such cracks are likely due to friction between the specimen and the compression ram. Neither porosity nor cracking had been previously observed after compression testing of 1505 K, 16:1 extruded material.[13]

IV. CONCLUDING REMARKS

The results of more extensive 1300 K slow plastic strain rate testing of cryomilled NiAl indicate that NiAl–AlN does retain strength under creep conditions. However, there is a change in deformation mechanisms (Fig. 6) at about 10^{-7} s^{-1} from a high stress exponent process (~11) to a lower stress exponent process (~6). While strength is dependent on the method of densification at faster strain rates, Fig. 8(a), the nature of such dependency is unclear. It is difficult to envision deformation mechanisms where the dissimilar microstructures (Fig. 3, Table I, Ref. 19) produced by hipping (1323 K, 207 MPa, 5 h) and extrusion (1505 K–16:1) have approximately equal strengths which are, in turn, greater

than that produced by other hipping conditions (1623 K, 207 MPa, 5 h) or extrusion parameters (1505 K–8:1 or –12:1). Furthermore, the apparent lack of dependency of creep resistance on consolidation technique at low strain rates [Fig. 7(b)] is puzzling. Without extensive transmission electron microscopy study of tested specimens, we cannot make any meaningful speculation as to the governing dislocation mechanism in either regime or to the influence of densification method.

In spite of the transition to a lower stress exponent mechanism, NiAl–AlN is strong at low deformation rates. This can be visualized in Fig. 9, which contrasts the density compensated flow stress–strain rate behavior at 1300 K for several high temperature materials including single crystal Ni-base alloy NASAIR 100,[20] polycrystalline Ni-base B-1900,[21] polycrystalline Co-base MAR-M 509,[21] and NiAl.[1] On this basis the present composite has attractive properties, nearly identical to NASAIR 100 which is a first generation, rafting Ni-base superalloy designed for high temperature creep resistance. In addition to being strong, initial testing[22] indicates that the cyclic oxidation resistance of NiAl–AlN is much better than NiAl and is almost equal to the best known third element doped nickel aluminide. Such a combination of strength and oxidation resistance makes NiAl–AlN a worthy candidate for further study as a high temperature structural material both for monolithic and fiber composite applications.

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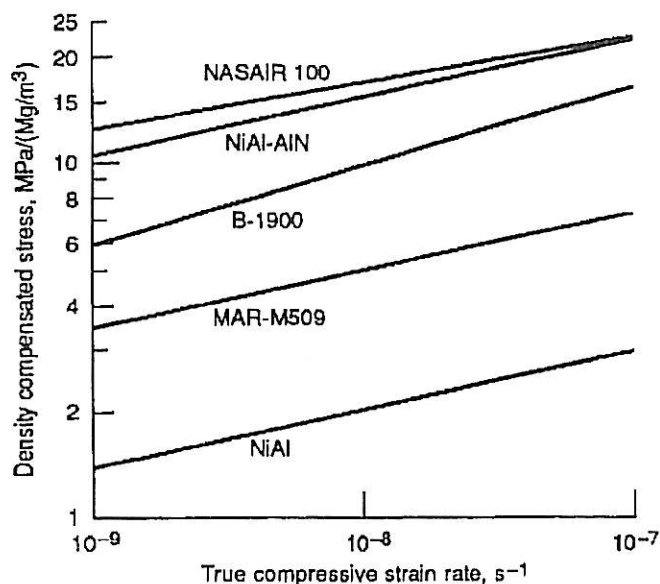J. Mater. Res., Vol. 5, No. 12, Dec 1990

FIG. 9. Comparison of the density compensated 1300 K creep strength of several high temperature materials as a function of strain rate. Flow stresses at 1300 K were normalized with respect to ambient temperature density.

V. CONCLUSIONS

Based on a study of the structure and chemistry of a cryomilled B2 crystal structure intermetallic NiAl, it is concluded that cryomilling leads to the production of a NiAl–AlN composite via a reaction milling process. Furthermore, 1300 K compressive creep testing of such composites consolidated by both hot extrusion and hot isostatic pressing indicated that density compensated strength is about equal to the Ni-base single crystal alloy NASAIR 100. This demonstrates that strong, creep resistant NiAl-base composite materials are possible.

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