Discontinuously Reinforced Intermetallic Matrix Composites

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Intermetallic matrix composites have recently received considerable attention as potential candidates for high-temperature applications. A variety of matrices and reinforcements have been examined to date, and reinforcement type, volume fraction, size, shape, and distribution have been shown to affect microstructure and mechanical properties. Several innovative approaches have been devised to produce discontinuously reinforced composites, ranging from such conventional techniques as blending, mechanical alloying, and rapid solidification processing to more exotic techniques, such as reactive consolidation and XD™ technology, which depend on the exothermic heat of formation of compounds. Composite mechanical properties of interest typically include high-temperature strength, the strain-rate dependence of strength, and ambient-temperature toughness and/or ductility. Thermodynamic stability of the reinforcement, elastic modulus mismatch with the matrix, and differences in thermal expansion coefficient between the matrix and reinforcement influence these properties. This paper addresses recent advances in these areas.

KEY WORDS: intermetallic matrix; composites; processing; discontinuous reinforcement; mechanical properties; microstructure, compatibility.

1. Introduction

Ordered intermetallics, particularly aluminides and silicides, have been examined as candidate materials for high-temperature applications because of their high melting points and potentially good oxidation resistance. However, experimental evidence indicates that several of these compounds do not have adequate creep resistance at the intended use temperature and almost all have unacceptable damage tolerance at room temperature. Several approaches have been examined to overcome these drawbacks, including reinforcing the intermetallics with ceramic particles, whiskers, and short fibers to improve their creep resistance and ambient toughness. Alternately, ductile reinforcements have been used in creep-resistant intermetallics to improve their fracture toughness and ductility at low temperatures.

A multitude of techniques has been developed to fabricate these composites. Equally intensive efforts are under way in the area of continuously reinforced composites, including long-fiber materials and laminates. However, discontinuously reinforced composites are relatively easier to fabricate than long-fiber composites; further, they behave isotropically unless the short fibers or whiskers are intentionally aligned, and are not as sensitive as long-fiber composites to mismatch in the coefficient of thermal expansion between the matrix and the reinforcement.

Mechanical properties of such discontinuous composites depend on the shape, volume fraction, size, and distribution of the reinforcement. Since these materials experience high use temperatures for prolonged periods

of time, the thermodynamic and kinetic stability of the reinforcement phase in the intermetallic matrix is critical; interfacial reactions during either processing or use will progressively degrade the mechanical properties. Also, the interfacial bond strength influences creep resistance and ambient toughness.

This paper provides an overview of the various approaches for producing discontinuously reinforced intermetallic matrix composites. The microstructures and mechanical properties of various such intermetallic composites are evaluated, and their benefits and drawbacks are discussed.

2. Overview of Processing Approaches

Most discontinuously reinforced intermetallic matrix composites are produced *via* powder metallurgy techniques. The high melting points of these intermetallics deter ingot metallurgy approaches, in part because the high reactivity of the molten metal can lead to the dissolution of reinforcement particulates, and in part because the high viscosity associated with large volume fractions of reinforcement makes casting difficult. Nevertheless, there have been some studies on ingot metallurgy-processed intermetallic composites, which will be discussed later.

Among the powder processing routes, the simplest technique involves blending the intermetallic powder and the ceramic reinforcement and consolidating the blend to full density.¹⁾ This approach is problematic because the reinforcement distribution is limited typically by the size of the intermetallic powder particles, and the size of

the reinforcement depends on what is commercially available. Alternately, mechanical alloying has been successfully used to produce oxide dispersion-strengthened intermetallics,²⁻⁵⁾ where the oxide is incorporated as an addition prior to milling. A variation of this milling process is cryo-milling,⁶⁾ where AlN is incorporated into NiAl.

Rapid solidification techniques have also been used to produce fine dispersoids in intermetallic matrices—carbides and borides are dissolved in the molten metal and then reprecipitated during hot consolidation. This approach has been successful in reinforcing NiAl with carbides and borides⁷⁾ and Ti_3Al with rare earth oxides.⁸⁾ While these processes have the advantage of producing very fine reinforcements that be classified more as dispersoids than particulates, they impose limitations on the volume fraction of reinforcement that can be incorporated.

Collectively termed reactive consolidation,⁹⁾ several variants of reactive sintering, including reactive hot pressing and reactive hot isostatic pressing, have been used to produce intermetallic matrix composites. In all cases, the reinforcement particulates/fibers are mixed with elemental powders with or without some prealloyed powders. The exothermic reaction resulting from the conversion of the elemental powders to the matrix intermetallic generates sufficient heat to produce a transient liquid phase which accelerates densification and is consumed during the process.⁹⁾ Thus, full density can be attained at a lower-than-usual temperature. NiAl-, Ni_3Al -, and Al_3Nb -based composites have been produced by this process. Reactive consolidation has an advantage over rapid solidification in that there is no limitation on the volume fraction of reinforcement or on the type and shape of reinforcement. Since the matrix intermetallic is formed during the consolidation step, it is possible, at least in principle, for the reinforcement to materialize within the grains rather than just at the grain boundaries. However, as with other powder processes, the reinforcement size and shape are controlled by commercial availability.

This drawback is eliminated in the XD^{TM} process,¹⁰⁾ developed at Martin Marietta Laboratories, which is a method for forming the reinforcements *in-situ*. Therefore, their size, shape, and volume fraction can be influenced by close control of processing parameters. The XD^{TM} process has been successfully used to produce particulate-reinforced composites of NiAl, TiAl, CoAl, and $\text{Al}_{22}\text{Ti}_3\text{Fe}_3$ (i.e. $\text{Al}_{66}\text{Ti}_{25}\text{Fe}_9$ on an at% basis). Microstructures and mechanical properties obtained in these composites were recently reviewed in detail by Kumar and Whittenberger.¹¹⁾

3. Discontinuously Reinforced Nickel Aluminide Composites

The two most widely researched intermetallics in the aluminum–nickel binary system are Ni_3Al and NiAl. Both these intermetallics have been examined with respect to their potential as matrix material for particulate composites.

3.1. NiAl-based Composites

Seybolt³⁾ first examined the effect of oxide dispersions (Al_2O_3 , ThO_2 , and Y_2O_3) in NiAl on tensile ductility, high-temperature strength, and stress-rupture response. The composites were produced by mechanical alloying and the reinforcement volume fraction ranged from 0–6%. The addition of particulates decreased the ductility, although strength increased up to $\sim 1150\text{ K}$. At higher temperatures, the strengths converged. It was concluded that these composites were not competitive with existing high-temperature materials.

Jha and Ray⁷⁾ produced rapidly solidified NiAl with various types of dispersoids by melt-spinning and Whittenberger *et al.* examined these composites at 1300 K in compression over a range of strain rates.¹²⁾ They found that of the various dispersions incorporated, only HfC successfully strengthened the matrix. A high stress exponent was obtained ($n > 10$), and there was indication of a threshold stress for creep.

Reactive consolidation techniques have been used to produce NiAl– TiB_2 composites containing large volume fractions of reinforcement (up to 40 vol% TiB_2). The variation in compressive yield strength with temperature for various levels of TiB_2 reinforcement is shown in Fig. 1. While the strengths of these composites are substantially higher than that of the monolithic NiAl at ambient and warm temperatures, the strengths converge at temperatures around 1200 K , the intended use-temperature range for such materials. A limited number of tensile tests at high temperatures¹³⁾ showed that the particulate composites (20 vol% TiB_2) were not as ductile as their monolithic counterpart.

Extensive studies have been performed¹⁴⁻¹⁷⁾ on stoichiometric NiAl and Ni-rich NiAl containing various amounts of $1\text{--}3\text{ }\mu\text{m}$ TiB_2 particulates, produced by the XD^{TM} process. A typical micrograph of an NiAl composite containing TiB_2 particulates is shown in Fig. 2(a); an electron micrograph of the particulates in the NiAl reveals clean interfaces (Fig. 2(b)). As indicated by the plot of compressive flow stress at 1% strain vs. strain rates for various vol% TiB_2 at 1300 K (Fig. 3), the composite is stronger than the monolithic material over the entire strain rate regime¹⁴⁾; further, the stress exponent of the 20 vol% TiB_2 composite approaches 9 (vs. 6 for the monolithic material), suggesting a change in the deformation mode. Estimated activation energies

Fig. 1. Compressive flow stress vs. temperature for NiAl– TiB_2 composites produced by reactive consolidation.⁹⁾

Fig. 2. As-hot-pressed, XD^{TM}-processed NiAl-TiB₂ composites: (a) 20 vol% TiB₂, (b) 5 vol% TiB₂.

Fig. 3. Compressive flow stress at 1% strain vs. strain rate at 1300 K for NiAl-TiB₂ composites produced by the XD^{TM} process.

Table 1. Temperature-compensated power law coefficients for NiAl-TiB₂ composites.¹⁴⁾

TiB ₂ (vol%)	Temperature range (K)	B	n	Q (kJ/mol)	R _d ² *
0**	1200-1300	0.52	6.33	365.9	0.996
5.0	1200-1300	5.14×10^{-8}	10.30	400.9	0.988
7.5	1200-1300	6.85×10^{-8}	10.09	407.6	0.988
10.0	1200-1300	3.14×10^{-10}	11.63	426.8	0.980
20	1200-1300	1.07×10^{-7}	8.87	397.5	0.989
30	1200-1300	7.28×10^{-6}	7.97	408.6	0.973

* Coefficient of determination.

** Data for strain rates $\leq 2 \times 10^{-5} \text{ sec}^{-1}$.

are shown in Table 1 together with the stress exponents. The enhancement in high-temperature strength was attributed to a decreased subgrain size and the retention of a high dislocation density,¹⁴⁾ and was greater for the 20 vol% TiB₂ composite with stoichiometric NiAl than for that with Ni-rich NiAl.^{14,15)}

Fig. 4. A comparison of the tensile creep-rupture behavior of stoichiometric NiAl-20TiB₂ composite, single-crystal NiAl, and a commercial Rene 80 superalloy.¹⁷⁾

Fig. 5. Fracture toughness of stoichiometric NiAl containing 0, 10, and 20 vol% TiB₂.

Fig. 6. Compressive flow stress at 1% strain vs. strain rate at 1200 and 1300 K for NiAl + Ni₂AlTi with and without 20 vol% TiB₂.

Figure 4 shows the results of tensile creep-rupture tests on stoichiometric NiAl containing 20 vol% TiB₂ particulates,¹⁷⁾ single-crystal NiAl, and Rene 80 superalloy (Fig. 4; note: stresses are not normalized with respect to density). While the composite is superior to the single crystal, it still does not compare to the superalloy. The fracture toughness of stoichiometric NiAl (6 MPa√m) is not significantly affected by up to 20 vol% TiB₂ addition (Fig. 5) and fracture mode remains transgranular cleavage. This finding contradicts Rigney *et al.*,¹⁾ who showed that toughness was doubled by the addition of 10 vol% TiB₂. They offered no reasons for the observed improvement, although particle distribution may influence the toughness, as proposed by Faber and Evans¹⁸⁾ to explain crack-deflection toughening.

In another study,¹⁹⁾ it was shown that particulate reinforcement of a two-phase NiAl + Ni₂AlTi alloy produced *via* the XD^{TM} process leads to a decrease in high-temperature strength (1 200 and 1 300 K) at fast strain rates ($\sim 10^{-4} \text{ sec}^{-1}$); the composite regains marginal superiority at the slow ($\sim 10^{-7} \text{ sec}^{-1}$) strain rates (Fig. 6). This behavior was attributed to the activation of dislocations at the particle-matrix interface at a threshold stress, as previously proposed by Edwards *et al.*²⁰⁾ It should be noted that the addition of TiB₂ particulates refines grain size but does not beneficially alter the brittle characteristics of the two-phase NiAl + Ni₂AlTi material.

Recent results on AlN-reinforced NiAl, produced by cryo-milling, deserve mention.⁶⁾ These composites are obtained by mechanical milling of NiAl in liquid nitrogen, which incorporates nitrogen into the NiAl. Subsequent thermomechanical processing then causes the precipitation of very fine (<50-nm) particles of AlN reinforcement. The 1 300 K compressive flow stress at 1 % strain over a range of strain rates, as determined by Whittenberger,⁵⁾ showed that the material was superior to other NiAl composites, particularly at the slow strain rates.

Creep studies have also been conducted on stoichiometric and Ni-rich NiAl reinforced with Al₂O₃ whiskers and short fibers.^{15,21)} Figure 7 shows a plot of

Fig. 7. Compressive flow stress vs. strain rate at 1300 K for stoichiometric NiAl-Al₂O₃ composites.

Fig. 8. Predicted and experimental data for the 1200 K compressive creep strength of NiAl containing various amounts of Al₂O₃ whiskers.

compressive flow stress at 1 % strain versus strain rate at 1 300 K for stoichiometric NiAl reinforced with the whiskers. In contrast to the TiB₂ composites, the improvements per unit addition are not as significant, stress exponent (n) does not change with volume fraction of reinforcement, and the experimental results observed can be adequately explained using the Kelly and Street model.²¹⁾ Agreement between experimental and predicted results can be seen in Fig. 8; the model is not considered accurate for reinforcement volume fractions ≤ 0.05 . Incorporation of 15 vol% Al₂O₃ whiskers increases the fracture toughness of stoichiometric NiAl from 6 to 9 MPa $\sqrt{\text{m}}$ but the whiskers are randomly oriented. The addition of up to 15 vol% Al₂O₃ whiskers to Ni-rich NiAl (61.5 at% Ni) does not improve the fracture toughness and only marginally improves 1 300 K compressive creep resistance. Recently, Alman and Stoloff¹³⁾ used injection molding to align chopped Al₂O₃ fibers in NiAl, thereby hoping to improve its toughness. However, the resulting composite exhibited anisotropic properties.

While most of these approaches have been aimed primarily at improving the creep resistance of NiAl, some have also increased ambient toughness. In this regard, a material called microstructurally toughened composites has recently received attention.²²⁾ This approach, specifically intended to increase the energy absorption capability of NiAl, entails incorporation of tubes of 304 stainless steel into the NiAl matrix. Notched impact energies as high as 91 J/cm² were measured on the composite, compared with <1 J/cm² for the monolithic material. Although this composite is continuously reinforced, the novelty of the method and the encouraging results warrant mention.

3.2. Ni₃Al-based Composites

There has been very little published work on discontinuously reinforced Ni₃Al composites, due in part to the reactivity of Ni₃Al. It is claimed that SiC, B₄C, and TiB₂ react extensively with Ni₃Al while Al₂O₃ and TiC are believed to be stable.²³⁾ Jang and Koch²⁴⁾ produced Ni₃Al containing Al₂O₃ dispersoids *via* mechanical alloying and showed that Hall-Petch-type strengthening could be obtained, but at the expense of ductility. Subsequently, Fuchs²³⁾ produced Ni₃Al-based composites containing TiC reinforcement by blending Ni₃Al powders with 25 vol% TiC with an average size of 2.5 μm and consolidating the blend. Tensile studies in the temperature range 298–1 273 K showed the yield strength of the composite to be 10–20 % greater than that of the monolithic material for all the temperatures studied, although composite ductility was lower. Good matrix/particulate bonding was reported.

Rigney *et al.*¹⁾ tested composites of Ni₃Al (with and without B) containing 10 vol% TiB₂ particulates, produced by blending and vacuum hot pressing. They found that the notched three-point bend toughness of the composites was lower than that of the monolithic material, and attributed it to TiB₂ particulates clustering at the prior Ni₃Al particle boundaries. Although they suggested an interfacial reaction between Ni₃Al and

TiB₂, they failed to observe a distinct reaction layer. McKamey and Lee²⁵⁾ produced hot extruded blends of Ni₃Al powders containing 10–20 vol% Al₂O₃ chopped fibers and showed that the composite was stronger than the monolithic material at ambient temperature but weaker at 1 273 K. Likewise, Moore *et al.*²⁶⁾ showed that the room-temperature tensile yield strength of Ni₃Al + B can be improved by the addition of randomly oriented Al₂O₃ short fibers although this occurred at the expense of ductility. Brennan *et al.*²⁷⁾ examined composites of Ni₃Al containing 25 vol% Al₂O₃ particulates, obtained by blending, hot pressing, and hot extrusion of the powders. They performed room-temperature three-point bend tests after various high-temperature exposures, but did not compare their results to those for the monolithic material produced under similar conditions.

4. Discontinuously Reinforced Titanium Aluminide Composites

The materials reviewed in this section include discontinuously reinforced composites of Ti₃Al, near γ -alloys (TiAl + Ti₃Al), and Al₃Ti-based (L1₂ and D0₂₂) compounds.

4.1 Ti₃Al-based Composites

While there has been significant research on monolithic Ti₃Al, research on discontinuously reinforced composites of Ti₃Al has been scant—primarily because of the reactive nature of Ti₃Al and the difficulty of identifying stable reinforcements. There has been some work on dispersion-strengthened Ti₃Al, where the dispersoids are rare-earth oxides incorporated by rapid solidification processes. For example, Konitzer and Fraser⁸⁾ examined the coarsening behavior of Er₂O₃ in Ti₃Al. Similarly, Rowe and co-workers^{28,29)} performed bend test studies on Ti₃Al containing Er₂O₃, Ce₂S₃, and Ce₄O₄S₃ dispersoids and found that the dispersoids lowered ductility. High-energy-rate forming approaches have been used to produce Ti₃Al reinforced with AlN, TiB₂, and TiAl reinforcements.^{9,30)}

4.2. TiAl + Ti₃Al-based Composites

Alloys of TiAl containing small amounts of Ti₃Al have been viewed as promising candidates for high-temperature applications. A review of the microstructure, processing, and properties of these alloys was recently published.³¹⁾ Particulate and short-fiber composites of these alloys have been produced *via* the ingot metallurgy variation of the XD^{TM} process,^{32–35)} by directional solidification,³⁶⁾ and by other solidification routes.³⁷⁾

Appropriate thermomechanical processing of these two phase intermetallics, coupled with secondary heat treatments, can produce a variety of microstructures ranging from a lamellar γ/α_2 structure to fully equiaxed. Such microstructural differences significantly influence mechanical properties. For example, creep resistance and ambient ductility are enhanced by an equiaxed microstructure, whereas fracture toughness reaches a maximum with a lamellar microstructure.^{33,38)} The addition of particulates *via* the XD^{TM} process has been

Fig. 9. The effect of similar thermomechanical processing on the lamellar-to-equiaxed morphology transformation in near- γ TiAl⁴⁰⁾: (a) monolithic material and (b) TiB₂-containing composite produced by the XD^{TM} process.

shown to refine the cast grain size³⁹⁾ and thereby enhance the kinetics for the lamellar-to-equiaxed transformation, which is thought to occur discontinuously at grain boundaries. Additionally, the particle-matrix interfaces also serve as nucleation sites for this transformation. Representative micrographs of this transformation in a monolithic material and its composite counterpart containing ~7 vol% TiB₂ are shown in Fig. 9.

In one study,³⁴⁾ the fracture toughness of a Ti–48Al–2V alloy containing 7 vol% TiB₂ particulates was found to be 16.5 MPa $\sqrt{m}$ for the lamellar structure and 8.6 MPa $\sqrt{m}$ for the equiaxed microstructure. The higher toughness of the lamellar form was mainly due to crack deflection along the lamellar interfaces (Fig. 10). Pao *et al.*⁴¹⁾ measured the fracture toughness of binary monolithic Ti–46at%Al and Ti–47at%Al containing 7 vol% TiB₂, both in the as-extruded condition and annealed at 1 473 K for 16 hr. The fracture toughness of the composite was around 9 MPa $\sqrt{m}$ for both processing conditions, while values for the monolithic material were 19 MPa $\sqrt{m}$ in the extruded condition and 14 MPa $\sqrt{m}$ after annealing. They attributed the degradation in toughness of the composite to the presence of the TiB₂ particulates. Room-temperature fatigue crack-growth resistance of the composite was also shown to be inferior to that of the monolithic material.⁴¹⁾

Kampe *et al.*³⁵⁾ examined the creep behavior of several monolithic and particulate-reinforced composites of such γ/α_2 alloys (near- γ) as a function of starting microstructure, microstructural stability during deformation, grain size, interstitial content, and processing history. A plot of the 1 073 K steady-state creep rate as a function of initial stress for several microstructures

Fig. 10. Fracture surfaces in Ti-48Al-2V + 7vol% TiB₂XD™ composites³⁴⁾: (a) investment cast material with a lamellar microstructure and (b) isothermally forged material with an equiaxed microstructure.

Fig. 11. Steady-state creep rate at 1073 K for powder- and ingot (forged)-based monolithic and XD™-processed composite materials with and without heat treatment: (a) as a function of initial stress and (b) as a function of inverse temperature.³⁵⁾

(Fig. 11(a)) indicates that the powder-processed alloys exhibit the least creep resistance. Further, the creep resistance of the heat-treated materials, with their increased grain size, is superior to that of the as-forged material. Calculations based on inverse temperature vs. steady-state creep-rate profiles (Fig. 11(b)) for the various processing conditions for all the alloys yielded an activation energy for creep of 340 kJ/mol, suggesting that lattice diffusion was dominant.³⁵⁾ The mechanism governing creep was reported to be the same in the unreinforced and the composite materials. Likewise, in another study, Kampe *et al.*³³⁾ showed that short-fiber reinforcement led to improved high-temperature strength and creep resistance relative to equiaxed particles without loss of fracture toughness. Subsequently, they used directional solidification techniques to align the short fibers and further improved the composite's properties.³⁶⁾

Larsen *et al.*⁴²⁾ examined investment-cast microstructures of these titanium aluminides in monolithic and particulate-reinforced forms and confirmed that the microstructure of investment cast monolithic titanium aluminide (near- γ composition) consists of a columnar grain structure with both a large grain size and a large distribution in grain size—200 μ m to as large as 6 000 μ m. However, incorporation of 7 vol% TiB₂ particulates in this material breaks down the columnar structure, replacing it with equiaxed fine grains, and also induces a uniformity in grain size, independent of section thickness. Room-temperature mechanical properties of investment-cast, HIPed, and heat-treated materials with and without TiB₂ are shown in Table 2. Improvements in strength and modulus with no loss in ductility are attributed to grain refinement and particulate strengthening. This approach has been successfully used to investment cast low-pressure turbine blades to near-net shape (see Fig. 1 of Ref. 42))

Research efforts at the University of California, Santa Barbara, have focussed on producing carbide and boride reinforcements in TiAl *via* solidification techniques. These reinforcements have platelet⁴³⁾ or rod³⁷⁾ morphologies. Compressive creep studies of TiAl reinforced with Ti₂AlC at 1255 K indicated a loss of strength relative to the solid solution-strengthened alloy. A similar response was observed for TiAl reinforced with rod-shaped Ta monoboride. While the composite in this case was significantly stronger than the binary monolithic TiAl, it was only marginally superior to monolithic TiAl containing Ta in solid solution.³⁷⁾ This failure of discontinuous reinforcements to strengthen the interme-

Table 2. Room-temperature mechanical properties of investment cast Ti-48Al-2V-2Mn alloy with and without 7 vol% TiB₂.⁴²⁾

Alloy	Yield strength (MPa)	Ultimate tensile strength (MPa)	Plastic elongation (%)	Modulus (GPa)
Ti-48Al-2V-2Mn	400–428	538–573	1.1–1.2	159
XD™ Ti-48Al-2V-2Mn + 7 vol% TiB ₂	573–587	704–731	0.9–1.1	180

tallics at high temperatures was explained by Rosler *et al.*⁴³⁾ as follows: at high temperatures, under an applied stress, hydrostatic stress components drive a vacancy flux from one face of the precipitate to another, causing local deformation at a rate that is higher than dislocation creep in the matrix. Based on this mechanism, they recommended particle thicknesses larger than 10 μm at temperatures and strain rates of technological interest.

4.3. Al_3Ti (D0_{22} and Ternary L1_2)-based Composites

There has been no work reported to date on discontinuously reinforced binary Al_3Ti , although there have been some efforts to reinforce Al_3Nb and Al_3Ta with chopped, randomly oriented Al_2O_3 fibers and with continuous Al_2O_3 reinforcements.^{9,44)} These binary trialuminides are, however, extremely brittle and have resisted attempts to improve their ambient-temperature damage tolerance. The total lack of effort on binary Al_3Ti is also likely related to its lower melting point relative to Al_3Nb and Al_3Ta . The brittle characteristics of binary Al_3Ti are somewhat ameliorated when it is alloyed to transform its D0_{22} structure to the cubic L1_2 structure. Ternary L1_2 trialuminides such as $\text{Al}_{122}\text{Ti}_8\text{Fe}_3$ (i.e. $\text{Al}_{66}\text{Ti}_{25}\text{Fe}_9$ on an at% basis), $\text{Al}_{67}\text{Ti}_{25}\text{Cr}_8$, and $\text{Al}_{66}\text{Ti}_{25}\text{Mn}_9$ exhibit significant ambient compressive ductility, and $\text{Al}_{66}\text{Ti}_{25}\text{Mn}_9$ exhibits limited tensile plasticity.⁴⁵⁾

Composites made by the XD^{TM} process have been used to study the compression behavior of monolithic and TiB_2 particulate-reinforced composites of these L1_2 trialuminides.⁴⁶⁾ The variation in compressive yield strength with test temperature for $\text{Al}_{22}\text{Fe}_3\text{Ti}_8 + 2\text{at}\%$ Nb (i.e. $\text{Al}_{66}\text{Ti}_{23}\text{Nb}_2\text{Fe}_9$ on an at% basis) with and without TiB_2 reinforcement is shown in Fig. 12(a). Ambient- and warm-temperature strengths undergo remarkable improvements upon the addition of TiB_2 particulates; at higher temperatures, however, the composites are weaker than the monolithic material. The effect of strain rate on high-temperature compressive flow stress for various levels of TiB_2 loading is shown in Fig. 12(b). While the composites are weaker at the fast strain rates ($\sim 10^{-4} \text{ sec}^{-1}$), they are superior to the monolithic material at the slowest strain rates representative of creep conditions. Possible reasons for this response have been discussed in detail elsewhere^{11,47)} and are similar to the reasoning proposed by Edwards *et al.*²⁰⁾

5. Discontinuously Reinforced MoSi_2 Composites

Recently, significant research interest has been focussed on the intermetallic compound MoSi_2 because of its combination of a high melting point ($\sim 2300 \text{ K}$) and excellent high-temperature oxidation resistance. Its ductile-to-brittle transition temperature is, however, in the neighborhood of 1200–1300 K. For MoSi_2 to be a viable structural material for high-temperature applications, both its room-temperature damage tolerance and its high-temperature creep resistance need to be improved. Accordingly, various researchers have studied composites of MoSi_2 discontinuously reinforced with either refractory particulates or whiskers or, alternately, a ductile metallic reinforcement to enhance damage

Fig. 12. (a) Compressive yield strength vs. temperature for monolithic and TiB_2 particulate-reinforced composites of $\text{Al}_{22}\text{Fe}_3\text{Ti}_8 + 2\text{Nb}$ and (b) true compressive stress-strain rate behavior for these materials at 1200 K.

tolerance. Various techniques have also been used to produce these composites.

Gibbs, Petrovic, and Honnell⁴⁸⁾ compared the ambient fracture toughness and high-temperature strength of MoSi_2 with and without 20 vol% SiC whiskers and concluded that these whiskers toughen the MoSi_2 at room temperature where it is brittle and strengthen it at elevated temperatures where it is ductile. Toughness was reported to increase from 5.3 to 8.2 $\text{MPa}\sqrt{\text{m}}$,⁴⁹⁾ and crack-deflection toughening was shown to be an important toughening mechanism.⁵⁰⁾ Yang *et al.*⁵¹⁾ measured flexural strength and bend fracture toughness on MoSi_2 reinforced with 20 vol% TiC particulates (2–5 μm). They reported a loss in strength in the composite due to premature failure; the fracture toughness remained unaffected. No significant interfacial reaction between TiC and MoSi_2 was observed.

Petrovic and co-workers^{52,53)} tried to further improve the properties of MoSi_2 by alloying it with other silicides such as WSi_2 ,⁵²⁾ NbSi_2 , TaSi_2 , Mo_5Si_3 , W_5Si_3 , Nb_5Si_3 , and Ta_5Si_3 .⁵³⁾ The disilicides all formed solid solutions and the $\text{MoSi}_2/\text{TaSi}_2$ alloy reacted with SiC to form TaC . Oxidation response was degraded where any disilicide solid solution was present. The addition of 20 vol% SiC whiskers to $\text{MoSi}_2/\text{WSi}_2$ increased the yield strength at high temperatures over that obtained by adding 20 vol% SiC to binary MoSi_2 .⁵²⁾ The variation in yield strength with temperature, obtained from four-point bend tests is shown in Fig. 13. In a study of the reinforcement potential of ductile refractory metals such as Nb and Ta for MoSi_2 , Carter and Martin⁵⁴⁾ found Ta to be a better candidate

Fig. 13. Bend yield strength vs. temperature for monolithic MoSi_2 and $\text{MoSi}_2/\text{WSi}_2$ and composites containing 20 vol% SiC .⁵²⁾ (Reprinted by permission of the American Ceramic Society)

Fig. 14. Compressive yield strength vs. temperature for MoSi_2 reinforced with 30 vol% of various refractory phases by the XD^{TM} process.⁵⁵⁾

based on interfacial reaction considerations.

The XD^{TM} process has also been used to produce discontinuously reinforced composites of MoSi_2 .⁵⁵⁾ The variation in compressive yield strength with test temperature for the unreinforced and composite materials (30 vol% reinforcement), as shown in Fig. 14, indicates an improvement in strength and also a strength dependency on reinforcement type. While SiC and TiB_2 are equiaxed particulates, the ZrB_2 and HfB_2 reinforcements have a plate morphology with a low aspect ratio. Aikin, Jr.⁵⁵⁾ claims that it is the particle size and shape, and not the particle type, that influence the high-temperature strength.

6. Other Discontinuously Reinforced Intermetallic Composites

CoAl with a B2 crystal structure is similar to NiAl in density, melting point, and stoichiometric range of existence. However, CoAl has received very little attention, primarily because of its extremely brittle nature. Mannan *et al.*⁵⁶⁾ used the XD^{TM} process to incorporate TiB_2 particulates in CoAl in an attempt to reduce the grain size and improve the ductility. Upon evaluating the high-temperature strength of these CoAl-TiB_2 composites as a function of strain rate they

Table 3. Temperature-compensated power law coefficients for CoAl-TiB_2 composites.⁵⁶⁾

TiB_2 (vol%)	Temperature range (K)	B	n	Q (kJ/mol)	R_d^{2*}
0	1100–1300	1.79×10^{-15}	2.95	384.1	0.995
5	1100–1300	4.1×10^{-3}	3.88	395.9	0.988
10	1100–1300	240	4.30	394.2	0.999
20	1200–1300	2.81	5.14	411.3	0.979

* Coefficient of determination.

Fig. 15. Compressive yield strength vs. temperature for Nb-26Ti-48Al alloy with 10 vol% of various reinforcements, produced via the XD^{TM} process.⁶⁰⁾

found that, unlike in the NiAl-TiB_2 composites, the activation energy was relatively independent of the TiB_2 content (Table 3). The stress exponent " n " in NiAl changes from ~ 6 to ~ 9 (Table 1) with increasing amounts of TiB_2 , while that for CoAl-TiB_2 increases from 3 for the monolithic material to 5.14 for the 20 vol% TiB_2 composite. Thus, in both systems, the stress exponent of the aluminides increases by a factor of 1.5 upon the addition of 20 vol% TiB_2 , suggesting that the dominant strengthening mechanism by the second-phase particles is likely the same.

Other intermetallic systems, mostly those with a topologically close packed (TCP) crystal structure, have also been examined because of their high melting points. These studies, typically restricted to ductile phase toughening, have been conducted for the $\text{Cr}_2\text{Nb}/\text{Nb}$ and $\text{Nb}_3\text{Al}/\text{Nb}$ systems,⁵⁷⁾ the $\text{Cr}_3\text{Si}/\text{Cr}$ system,⁵⁸⁾ and the $\text{Nb}_5\text{Si}_3/\text{Nb}$ systems.⁵⁹⁾ Only hardness indentation responses have been reported for the first two, but some bend test results are available as a function of temperature for the others.

A ternary intermetallic with the nominal composition Nb-25Ti-50Al has received some attention recently. The composition corresponds to a two-phase alloy of $\gamma\text{-TiAl}$ (saturated with Nb) and $\sigma\text{-Nb}_2\text{Al}$ (saturated with Ti). This alloy forms a protective alumina scale at high temperatures and is therefore suitable for use in oxidizing environments. Aikin, Jr. *et al.*⁶⁰⁾ used the XD^{TM} process to reinforce this intermetallic with thermodynamically stable boride, carbide, nitride, and silicide particles. The variation in compressive yield stress with temperature for unreinforced and composite materials containing 10 vol% of the various reinforcements is shown in Fig. 15. The shape of the reinforcement varied from equiaxed particles (TiN , 2–8 μm diameter) to whiskers with an

aspect ratio of 50:1 ($\text{Nb}_{0.67}\text{Ti}_{0.33}\text{B}$). The nitride-reinforced alloy has the highest strength, although the strengths converge above 1073 K.

7. Discussion

The various processes available for producing discontinuously reinforced intermetallic matrix composites are mostly powder metallurgy processes. Therefore, the cleanliness of the final product relies on the starting purity of the powders and processing care. As a result, the role of interstitials and oxides resulting inadvertently from processing cannot be separated from the role of reinforcements in influencing properties. Very few ingot metallurgy processes are available for producing these composites and in those cases, melt viscosity limits the volume fraction reinforcement to less than 10 vol%. Sputter deposition-based approaches have been suggested for producing "clean" composites, but these are still a laboratory curiosity.

In several of the systems studied, not only did particulate reinforcements not increase high-temperature strength, they weakened the monolithic material. Thus, the common notion that discontinuous reinforcements will always enhance the high-temperature capabilities of a material is not valid. This weakening has been attributed²⁰⁾ to intensification of shear stresses at particle-matrix interfaces by the discontinuity in the elastic properties at these interfaces. At some threshold level, this stress concentration can generate interfacial dislocations which pile up at adjacent grain boundaries. The subsequent climb rate of these dislocations controls the deformation rate. This explanation suggests that the weakening might be prevented by a close match in modulus and, possibly, coefficient of thermal expansion (CTE) between the matrix and the reinforcement. Such a match would, however, eliminate the advantages in stiffness of these composites.

It is well known that the reinforcement and matrix in continuous fiber-reinforced intermetallic matrix composites must have similar CTEs to minimize/avoid matrix cracking during thermal cycling. However, most ceramic fibers tend to have a lower thermal expansion coefficient relative to intermetallic matrices. Particulate additions could be used to lower the intermetallic matrix expansion coefficient, enabling the particulate-reinforced material to be used as a "matrix" for long-fiber composites. The particulates in this case however would have to have a much lower CTE than the matrix, which could negatively affect the high-temperature strength of the composite. The degradation in high temperature strength could arise directly due to interfacial cracking or due to possible formation of fine recrystallized grains at the interface due to thermal cycling, as a consequence of dislocation accumulation from the CTE mismatch. It has also been suggested that reinforcements may not strengthen at high temperatures because hydrostatic stresses around the particle drive a vacancy flux from one face of the reinforcement to another, in effect, promoting local deformation around the particle. Based on this argument, Rosler *et al.*⁴³⁾ suggested using coarse

platelets to enhance creep in TiAl, although these may act as crack nucleation sites and impair toughness.

Various studies have shown that the fracture toughness of ceramics and brittle polymers can be improved with discontinuous reinforcements. The improvements are attributed to several mechanisms, including crack bowing, crack deflection, crack branching, and micro-cracking. In intermetallic composites, such improvements remain to be seen. The limited data available indicate little toughness enhancement over the monolithic materials. Theoretical requirements for maximizing toughness depend on the mechanism of fracture that dominates, and include non-uniform distributions of reinforcements,¹⁸⁾ reinforcements with large aspect ratios,¹⁸⁾ and a good match in elastic modulus and CTE between the reinforcement and the matrix.⁶¹⁾ A strong interface is necessary to promote crack bowing, but may not be necessary if a different mechanism dominates. At present, it does not appear possible to predict the dominant fracture mechanism and, therefore, the criteria for selection of a reinforcement and matrix with the properties necessary to enhance toughness. As a result, we cannot define the real benefits to ambient toughness and high-temperature strength of reinforcing intermetallics with particulates, whiskers, and short fibers. The properties required to simultaneously enhance both these properties also are ambiguous.

The mechanical property database for intermetallic composites is poor to date—only limited tensile strength or tensile creep data are available. It is also not clear whether compressive creep studies can be correlated with tensile creep because problems with creep cavitation manifest themselves strongly in tension but not in compression—particularly in discontinuous composites where the reinforcement-matrix interface is susceptible to cavitation. Part of the reason for the lack of such tensile data is the problem of producing good-quality, fully dense material in sufficient quantities, and the difficulty and cost involved in machining tensile specimens. Alternate specimen geometries and clever techniques requiring only small specimens would certainly help in generating this much needed information.

Among all the various techniques and systems evaluated to date, the XD^{TM} process seems to be the most versatile. It has certainly been applied with a good deal of success to various intermetallic matrices, particularly TiAl, from which several near-net shapes have been investment cast successfully. Whether such parts will be superior in service compared to those from monolithic material remains to be verified, but at least at this stage, this technique seems to be one of the few that can deliver large quantities of material. The overall question of the superiority of discontinuously reinforced intermetallic composites relative to the monolithic material is application-sensitive and still remains open.

This paper would be incomplete without a comment on studies of oxide dispersion-strengthened iron aluminides. A separate section was not devoted to this family of materials because of the relatively few studies to date. A recent update of progress in this field by Vedula and Strothers²⁾ indicates that almost all of the effort has

Fig. 16. A comparison of the 1100 K compressive creep behavior and tensile yield strength for various oxide dispersion-strengthened Fe-40Al-0.1Zr-0.2B alloys²⁾ (Reprinted with permission from The Minerals, Metals and Materials Society).

focused on the Fe-40Al composition. In their study, the authors compare the compressive creep response and tensile yield strength at 1100 K of an Fe-40Al-0.1Zr-0.2B alloy containing 1 vol% Y_2O_3 and show that the compressive creep strength, extrapolated to the strain-rate regime of the tensile tests, is significantly higher than the tensile yield strength (**Fig. 16**). This behavior is attributed to the differences in the mechanism of deformation in the two types of tests and, therefore, the microstructures developed during the tests. Specifically, in the compressive creep tests, the data correspond to steady-state creep during which the substructure reaches equilibrium; in the tensile test, the yield strength corresponds to the features of the initial microstructure (*i.e.*, the tensile tests would more or less correspond to the transient stage in the compression creep curves). The authors argue that because necking instability occurs rapidly in the tension tests, it is not possible to obtain a steady-state condition in these tests or, therefore, a clear indication of the tensile creep behavior.

8. Conclusions

Several processes are available to produce discontinuously reinforced intermetallic matrix composites. These composites have been evaluated with such reinforcements as ceramic particulates, whiskers, and short fibers or, alternately, ductile second phases. Discontinuous reinforcements significantly enhance the damage tolerance of ceramic materials, but not intermetallic composites. While ceramic reinforcements are expected to enhance high-temperature strength and creep resistance, this has not always been the case. In several instances, the composites are weaker than the monolithic material, and the maximum strengthening due to reinforcement occurs at ambient and warm temperatures, where the monolithic materials frequently are already sufficiently strong. The properties of the reinforcement, matrix, and interface needed to enhance high-temperature strength and ambient-temperature damage tolerance simultaneously are not as yet well understood.

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