

Fracture of intermetallic Matrix Composites

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ABSTRACT

The relationship between powder processing techniques and resulting fracture behavior of intermetallic matrix composites is reviewed. Intermetallic alloys discussed include Ni_3Al , NiAl , TiAl and Ti_3Al . It is shown that appreciable strengthening can be achieved by filaments or plate-like reinforcements, although fracture ductility is usually not improved unless the reinforcement itself has high ductility. The role of fiber-matrix wetting and interfacial reactions in controlling fracture behavior is emphasized.

KEYWORDS

Composites; intermetallics; fracture; nickel; alumina; titanium diboride.

INTRODUCTION

Intermetallic compounds, especially the aluminides, offer a number of attractive properties as potential aerospace materials, as may be seen in Table 1. These alloys possess low density, high melting points and usually are alumina formers. However, they suffer from low (or no) ductility at ambient temperatures and many are relatively weak as binary alloys; this necessitates techniques designed to improve both low temperature toughness and high temperature creep resistance without sacrificing the desirable properties described above. Utilizing fibers or particles of higher melting ceramics or refractory metals, several techniques are being studied to produce new materials. The purpose of this paper is to review work in this field, especially in the area of the relationship between processing and fracture behavior.

PROCESSING APPROACHES

Intermetallic matrix composites based upon Ni_3Al , NiAl , Ti_3Al and TiAl are being produced by a variety of powder metallurgical and casting

Table 1. Properties of Intermetallic Compounds

Alloy	Structure	Young's Modulus (10 ⁶ psi)	T _m (°C)	T _c (°C)	Density (g/cc)
TiAl	L1 ₀	25.5	1460	1460	3.91
Ti ₃ Al	DO ₁₉	21.0	1600	1100	4.2
NiAl	B ₂	42.7	1640	1640	5.86
Ni ₃ Al	L1 ₂	25.9	1390	1390	7.50
FeAl	B ₂	37.8	1250-1400	1250-1400	5.56
Fe ₃ Al	DO ₃	20.4	1540	1540	6.72
CoAl	B ₂	42.7	1648	1648	6.14
Nb ₃ Al	A15	---	1963	1963	7.29
Nb ₂ Al	D8(σ)	---	1871	1871	6.87
NbAl ₃	DO ₂₂	---	1607	1607	---
Al ₃ Ti	DO ₂₂	---	1342	1342	---
AlTa ₂	D8(σ)	---	~2000	~2000	6.9
Al ₃ Ta	DO ₂₂	---	1627	1627	---
(Fe ₂₂ Co ₇₈) ₃ V	L1 ₂	---	1400	950	7.80
(Fe ₆₀ Ni ₄₀) ₃ (V ₉₆ Ti ₄)	L1 ₂	---	1400	680	7.60

processes. The following powder techniques and specific matrix-reinforcement combinations have been reported in the literature:

- reactive sintering or HIPping of elemental powders and short fibers, leading to a randomly oriented fibrous composite of Al₂O₃ or SiC in boron-doped Ni₃Al (Bose *et al.*, 1988; Moore *et al.*, 1988)
- injection molding of prealloyed Ni₃Al alloy powders to produce an aligned fibrous composite (Moore *et al.*, 1988)
- hot pressing of NiAl and TiAl powders made by the "XD" process, which results in the formation of a particulate-reinforced composite (Westwood, 1988; Mannan and Kumar, 1988)
- hot pressing of elemental or prealloyed powders mixed with TiB₂ or TiC powders to produce dispersion strengthened Ni₃Al+B (Rigney *et al.*; Fuchs, 1988) or NiAl (Rigney *et al.*, 1988)
- hot pressing of prealloyed Ni₃Al alloys with Al₂O₃ fibers (Povirk *et al.*, 1988)
- hot pressing of SiC reinforced Ti₃Al+Nb powder sheets (Brindley, 1987; Brindley *et al.*, 1988)
- high energy rate forming of TiAl/Ti₃Al composites (Marcus *et al.*, 1988)
- mixing of brittle TiAl with ductile Nb powders (Elliot *et al.*, 1988)
- hot pressing of Al₂O₃-reinforced Al₃Ta (Anton, 1988).

To date only Nourbakhsh *et al.*, (1988) have reported on the preparation of composites by a casting technique. Pressure casting was utilized to produce disks of Ni₃Al-Al₂O₃ composites. Significantly, no wetting of fibers by two alloy matrices (IC-50 and IC-218) was obtained unless a wetting agent such as Ti was used; however, bonding was weak. As will be shown below, the strength of the fiber-matrix interface in Ni₃Al-Al₂O₃ composites produced by reactive sintering or HIPping of either elemental powders or prealloyed powders has been extremely low, thereby leading to poor fracture behavior.

Potential processing problems apart from poor wetting are numerous, as listed in Table 2. Especially worrisome are porosity in the matrix

Table 2. Potential Processing Problems

Poor Wetting, Weak Bonding
Reactivity
Fiber Alignment
Thermal Expansion Mismatch
Porosity in matrix
Mechanical destruction of fibers

and degradation of fibers during processing or service.

To date only six composite alloys have been prepared in sufficient quantities to permit evaluation of tensile or bend properties and their publication in the open literature. The remainder of this paper will be concerned with these systems.

FRACTURE BEHAVIOR OF SPECIFIC ALLOY SYSTEMS

Ni₃Al+B/Al₂O₃

Processing Steps and Test Procedures. Two methods have been employed in our laboratory to produce Ni₃Al-base composites:

- reactive HIPping of Ni, Al and B powders with 3v% Dupont FP Al₂O₃ fibers (Bose *et al.*, 1988)
- reactive HIPping of prealloyed IC-218 with 5v% Al₂O₃ fibers (Moore *et al.*, 1988). Tensile samples were prepared from Ni₃Al+B and IC-218 base composites.

Reactive HIPping of the Ni₃Al+B with alumina fibers has been carried out at 800°C for 30 min at 104 MPa pressure. Reactive HIPping of monolithic Ni₃Al+B was done at 1100°C for 60 min at 172 MPa and also under the same conditions as that of the Ni₃Al+B with alumina fibers.

A micrograph of the cross section of as-HIPed Ni₃Al+B is shown in Fig. 1a); a composite sample is shown in Fig. 1b). Note the large particles in Fig. 1a) and the randomness of the fibers and the apparent lack of attack of the fibers by the matrix in Fig. 1b).

Prealloyed IC-218/Al₂O₃ mixtures were HIPed at 1100°C or 1150°C for 1 hr at a pressure of 172 MPa. Some samples were then heated to 1050°C for 1 hr, furnace cooled to 800°C, and held for 24 hr to remove second phase particles.

Typical microstructures of as HIPed and heat treated IC-218/Al₂O₃ are shown in Figs. 2a) and 2b), respectively. A second phase is known to exist in the matrix alloys (Liu, 1987). Note the lack of fiber alignment in these samples. A precipitate was noted at fiber/matrix interfaces, probably ZrO₂, according to (Povirk *et al.*, (1988).

Tensile tests on reactively HIPed or HIPed prealloyed powders were carried out at room temperature or at 600°C on cylindrical tensile samples. Crosshead rate was 8.5 x 10⁻³ mm⁻¹, corresponding to a strain rate of 7.25 x 10⁻⁴ s⁻¹. Room temperature tests were conducted in air, and

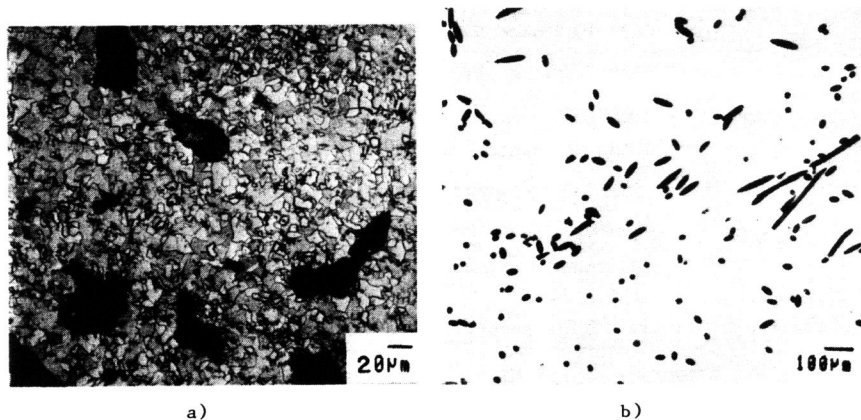


Fig. 1 Microstructures of reactively HIPed Ni₃Al alloys made from elemental powders a) Ni₃Al+B b) Ni₃Al_B/Al₂O₃ composite.

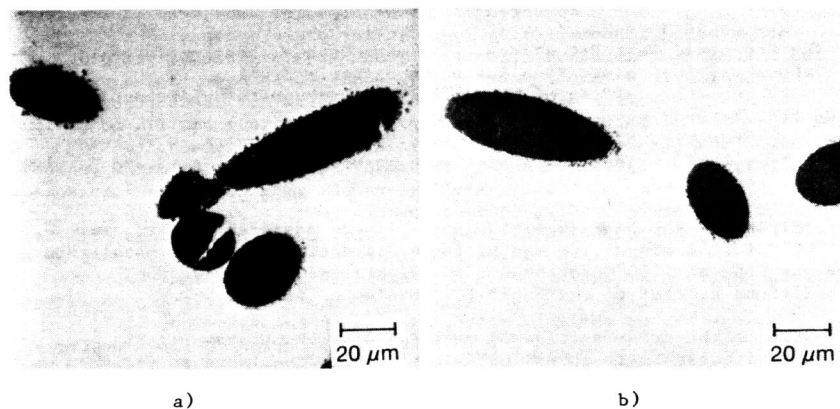


Fig. 2 a) Microstructures of HIPed IC-218/Al₂O₃ composites made from prealloyed powders, as HIP 1150°C. b) Precipitate at fiber-matrix interfaces in heat treated IC-218/Al₂O₃.

tests at 600°C in 10⁻⁵ torr vacuum.

Tensile Properties and Fracture Behavior. Tensile data for reactively sintered Ni₃Al-based composites and matrix alloys are listed in Table 3. Appreciable ductility was obtained at 25°C in Ni₃Al+B (HIPed at 800°C) and IC-218 (HIPed at 1100°C or 1150°C). The poor properties of Ni₃Al+B HIPed at 1100°C arise from the large second phase particles seen in Fig. 1a). These ductility values are far less than are usually observed in cast and wrought or conventional P/M alloys (Liu, 1987).

Table 3. Tensile Properties of Ni₃Al-Base Alloys.

Sample	Test Temp °C	σ _{YS} (MPa)	σ _{UTS} (MPa)	ε _F %	Remarks
Ni ₃ Al+B	25	286	759	14.8	As HIP, 800°C
Ni ₃ Al+B	25	494	677	2.1	As HIP 1100°C
IC-218	25	638	1380	19.8	As HIP 1100°C
IC-218	25	663	1400	23.5	As HIP 1150°C
IC-218/Al ₂ O ₃	25	663	890	3.5	As HIP 1150°C
Ni ₃ Al/Al ₂ O ₃	25	474	548	1.0	As HIP 800°C
Ni ₃ Al+B	25	591	828	5.2	Heat Treat, 1100°C-HIP
IC-218	25	535	1428	23.1	Heat Treat, 1100°C-HIP
IC-218	25	518	1421	21.5	Heat Treat, 1150°C-HIP
IC-218/Al ₂ O ₃	25	499	756	6.0	Heat Treat, 1150°C-HIP
IC-218	600	787	1070	15.9	As HIP 1100°C
IC-218	600	766	1049	15.2	As HIP 1500°C
IC-218/Al ₂ O ₃	600	814	869	1.0	As HIP 1150°C

Also, fibers produced no strengthening (based on yield stress) and actually caused a decrease in tensile strength due to the sharply lowered ductility of the composites. However, heat treated IC-218/Al₂O₃, tested at 25°C, did show 6% elongation. The ductility problem undoubtedly arises from the randomness of the fibers and relatively poor bonding, as well as their relatively low strength. It is hoped to solve the alignment problem by use of injection molding. Tensile properties as a function of temperature are shown in Fig. 3 for HIPed IC-218 and IC-218/Al₂O₃ composites. Note that there is little influence of fibers on yield stress, but UTS and ε_F are lower.

Fractographs from specimens of IC-218 and IC-218/Al₂O₃ tested at room temperature in air are shown in Figs. 4 and 5, respectively. Note the predominance of transgranular facets in as-HIPed IC-218, Fig. 4. Figs. 5a) and 5b) show that while matrix fracture is transgranular, bonding is poor between fibers and matrix for as HIP and HIP + heat treated samples, respectively. At 600°C in vacuum the as-HIPed IC-218 again showed predominantly transgranular fracture, see Fig. 6a). The IC-218/Al₂O₃ composite again displayed lack of bonding at the interface and the breakup of fibers, Fig. 6b).

The results reported in Table 3 may be compared with recent observations by Povirk *et al.*, (1988) on hot pressed IC-15 (Ni-24a%Al-0.24%B) and IC-218 alloys, Table 4. IC-15 was hot pressed at 1300-1350°C and IC-218 at 1250-1300°C. Micrographs of composites from the two alloys are shown in Figs. 7a) and b), respectively. All alloys were then annealed for 2 hr at 1000°C and 24 hr at 800°C and bend or tensile tested at 25°C. It appears that ductilities of Ni₃Al/Al₂O₃ are lower, and those of IC-218/Al₂O₃ are higher in the present work. Particularly noteworthy in the work of Povirk *et al.*, (1988) was the observation of good bonding between IC-15 and the Al₂O₃ fibers and the achievement of 10% ductility at room temperature. Both fracture of fibers, Figs. 8a) and debonding, Fig. 8b), were readily observable in this work. Hot pressing also produces stronger unreinforced IC-218, with a yield stress of 900 MPa and substantial ductility.

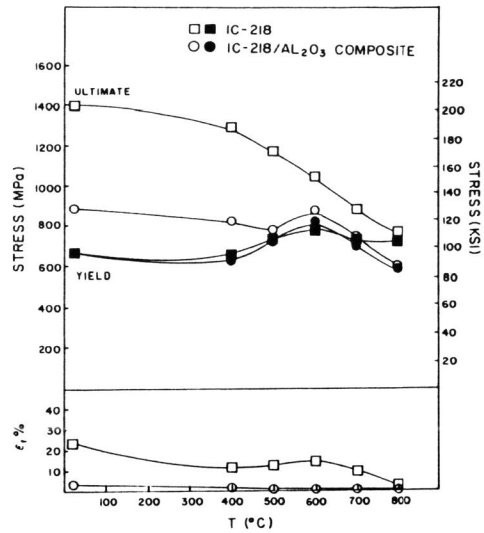


Fig. 3 Effect of temperature on tensile properties of IC-218 and IC-218/Al₂O₃ composites.

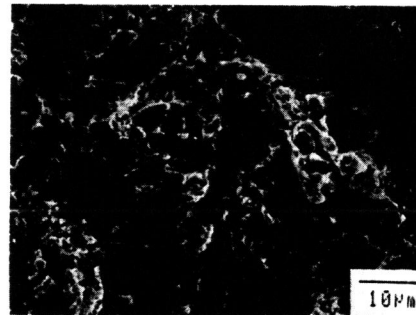
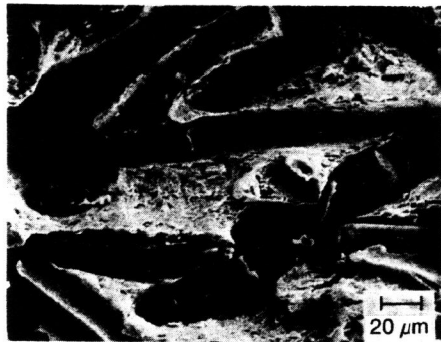
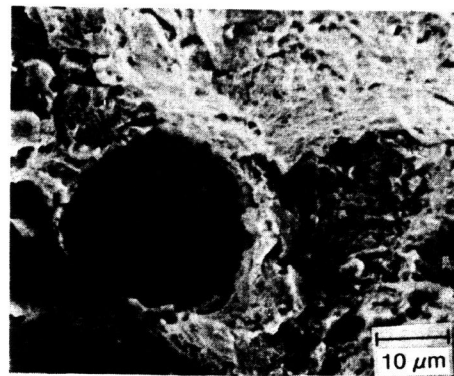


Fig. 4 Transgranular fracture in IC-218, HIPed at 1150°C, tensile tested at 25°C.

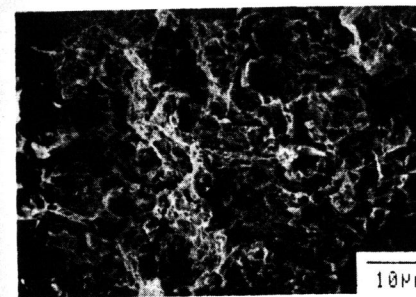


a)



b)

Fig. 5 Fracture surfaces at 25°C in IC-218/Al₂O₃ composites a) as HIP, 1150°C b) HIP + heat treated, 1050°C 1 hr.



a)

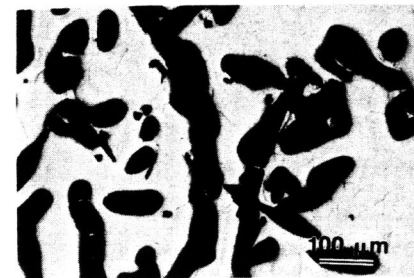


b)

Fig. 6 Fracture surfaces at 600°C, vacuum a) as HIP IC-218 b) IC-218/Al₂O₃.

Table 4. Tensile Properties of Hot Pressed Alloys (Povirk et al., 1988).

Alloy	g.s. μm	σ _{ys} (MPa)	σ _{UTS} (MPa)	ε _F %	Remarks
IC-15/Al ₂ O ₃	--	500	---	>5	Bend
IC-218	10	900	---	>5	Bend
IC-218/Al ₂ O ₃	7	---	170-200	<1	Bend
IC-15/Al ₂ O ₃	25μm	---	---	>10	Tensile



a)



b)

Fig. 7 Microstructure of hot pressed composites a) IC-15/Al₂O₃ b) IC-218/Al₂O₃ (Povirk et al., 1988).

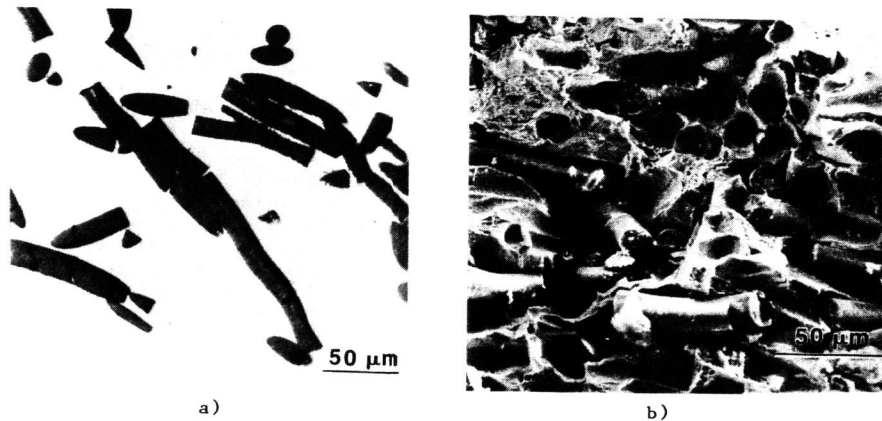


Fig. 8 Fracture of IC-215/Al₂O₃ tested in tension at room temperature; $\epsilon_f=10\%$ (Povirk, et al, 1988).

Ti₃Al+Nb/SiC[8,9]

Processing Steps. Matrix Ti-24a%Al-11%Nb was made from powder produced by the Plasma Rotating Electrode Process (PREP). Fibers were continuous 140μm diameter SiC (SCS-6). Composites with 40v% SiC were consolidated by hot pressing of powder cloth. Coupons were annealed in vacuum at 1250 and 1365°C for 1 to 100 hrs to determine the extent of fiber-matrix reaction.

Tensile Properties and Fracture Behavior. Tensile strengths vs temperature for the matrix, fibers and Ti₃Al+Nb/SiC (0° orientation) composites are shown in Fig. 9 (Brindley, 1987). Note that the tensile strength is generally near to that predicted from a rule of mixtures calculation, except at 25°C(298°K), where a sharp reduction in strength was noted. This was apparently due to impurity contamination exacerbating the very low ductility of both phases at room temperature (Brindley et al., 1988). SEM fractographs reveal no evidence of ductility in either phase at room temperature, see Fig. 10a). At higher temperatures increased fiber-matrix pullout and increased fiber-matrix debonding were observed, Figs. 10b-d). Increased debonding at elevated temperatures is very apparent in Figs. 11a)-c) (Brindley et al., 1988). At 23°C, Fig. 11a), the bond between fiber and interface appears to remain intact, perhaps due to both chemical and mechanical effects during processing. However, thermal cycling of this composite between 985°C and 23°C leads to cracking at the interface, probably due to thermal expansion mismatch (Brindley et al., 1988).

TiAl/TiB₂ and NiAl/TiB₂

Processing Steps and Fracture Behavior. A patented process for introducing single crystal platelets of TiB₂ into various metallic matrices has been developed by Martin Marietta Corp (Westwood, 1988). Up to 10v%

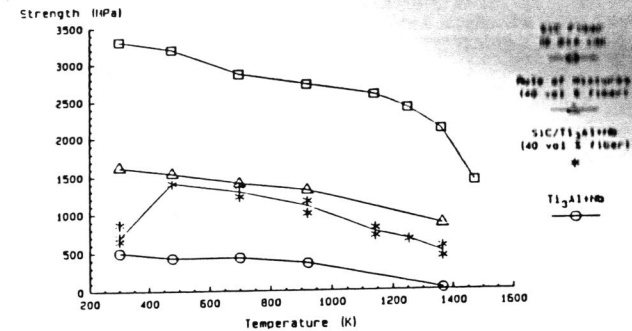


Fig. 9 Effect of temperature on tensile strength of Ti₃Al+Nb, SiC and Ti₃Al+Nb/SiC composites (Brindley, 1987).

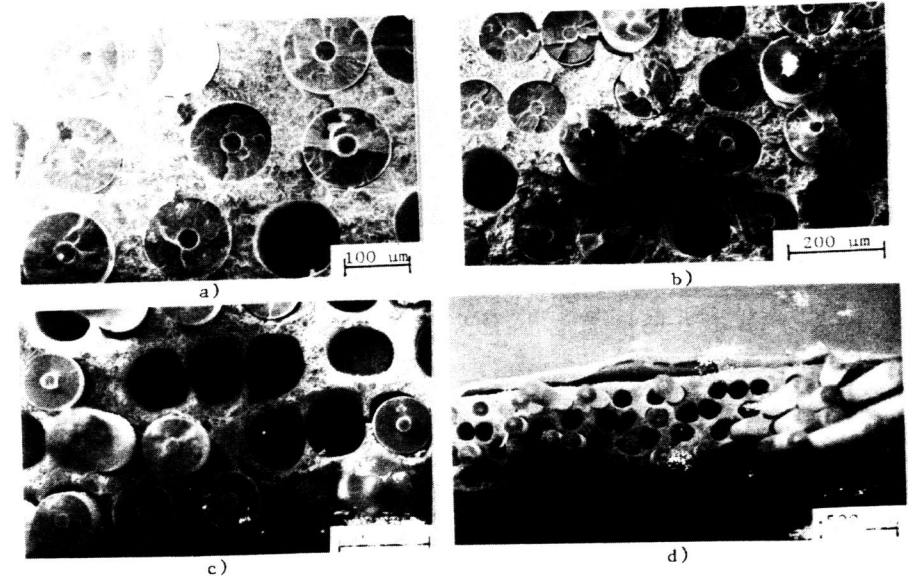


Fig. 10 Effect of temperature on fracture behavior of Ti₃Al+ Nb/SiC composites, showing increased fiber pullout and debonding with increasing temperature (Brindley et al, 1988) a) 23°C b) 425°C c) 875°C d) 1100°C.

of TiB₂ in γ TiAl has been obtained by hot pressing, extruding and rolling (Christodoulou et al., 1988). Only sketchy reports of fracture behavior exist. It appears that cleavage of the matrix, coupled with some pullout of TiB₂, is the fracture mode in reinforced γ tested in creep at 800°C. The introduction of TiB₂ into NiAl leaves nearly unchanged the low toughness of the matrix, (about 6 MPa/m) see Fig. 12 (Mannan and Kumar, 1988), while increasing strength substantially. Potentially, this process

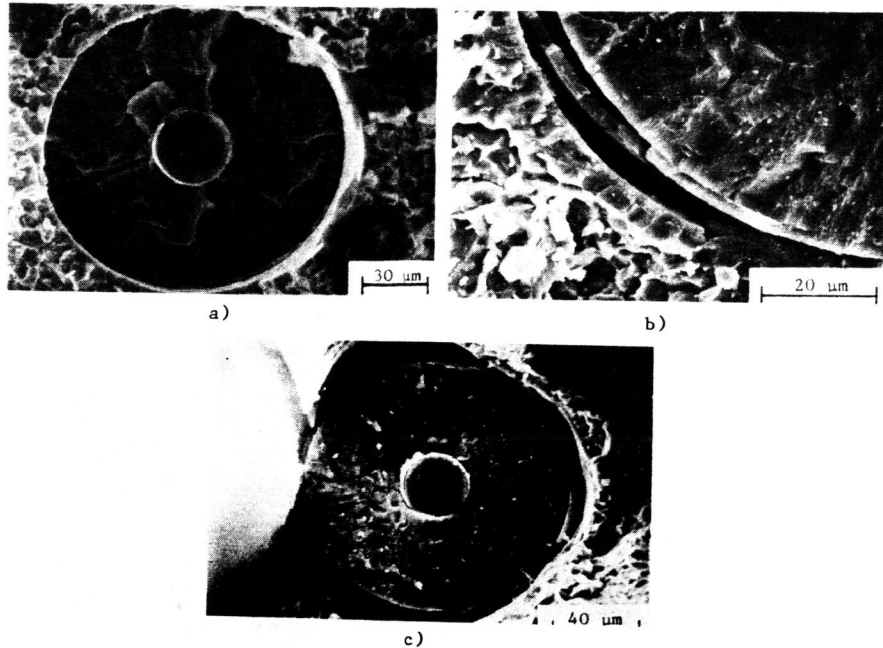


Fig. 11 Effect of temperature on debonding at fiber-matrix interface in $Ti_3Al+ Nb/SiC$ composites (Brindley et al, 1988) a) 23°C b) 650°C c) 1100°C.

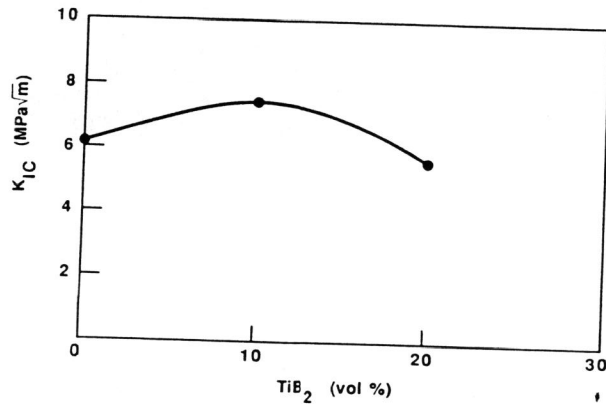


Fig. 12 Room temperature fracture toughness of $NiAl/TiB_2$ composites, (Mannan and Kumar, 1988).

may be applied to other matrices or, alternatively, different particles (carbides, borides, silicides and, nitrides) might be introduced.

TiAl/Nb

Considerable interest has been shown in the concept of bridging cracks in brittle matrices by the use of ductile particles to increase alloy toughness. Elliot et al., (1988) have reported that Nb powder, when mixed with TiAl and then rolled or extruded to elongate the ductile Nb particles, serves as an effective toughener. Both fracture toughness and the tendency for stable crack growth were reported to be improved. The pancake shaped Nb particles bridge cracks and decohere from the TiAl matrix.

Al₃Ta/Al₂O₃.

Anton (1988) has utilized reactive hot pressing of elemental Ta and Al powders and 35v% continuous or chopped FP Al_2O_3 fibers to form Al_3Ta/Al_2O_3 composites. Pressure was applied at 660°C, the melting point of aluminum. Although Al_3Ta is considered to be a line compound (DO_{22} structure), a nearly single phase matrix was obtained by annealing at 1200°C for four hours subsequent to consolidation.

Figure 13 shows the results of bend tests at room temperature in vacuum on monolithic Al_3Ta and the composites (Anton, 1988). Note the brittleness of Al_3Ta and the improved strength and toughness conferred by continuous fibers. The composite with chopped fibers, on the other hand, displayed lower modulus and tensile strength, accompanied by a high apparent plasticity resulting from fiber pull out.

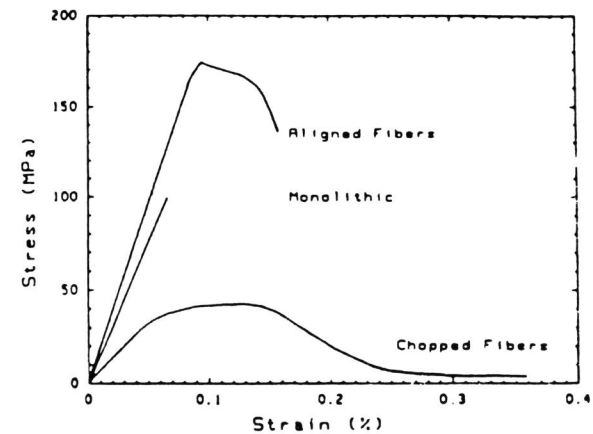


Fig. 13 Stress-strain curves from four-point bend tests of monolithic Al_3Ta and $Al_3Ta-Al_2O_3$ composites at room temperatures (Anton, 1988).

DISCUSSION

The key to improving strength and fracture resistance of intermetallic compounds with reinforcements such as filaments, whiskers or platelets lies in the processing techniques chosen for consolidation. Of the various aluminides utilized as a composite matrix to date, only Ni₃Al+B and related alloys containing Cr and Zr exhibit more than a few percent elongation in tension. Clearly, the introduction of non-aligned, poorly wetted fibers of Al₂O₃ into these alloys may severely degrade ductility with little or no improvement in yield or tensile strengths. Wetting of Al₂O₃ by cast Ni₃Al alloys is reported to be enhanced by the addition of titanium to the melt (Nourbakhsh *et al.*, 1988). Therefore, alignment is desirable, and this has been accomplished to some degree through utilization of injection molding with short fibers. However, in order for injection molding to succeed, several formidable problems must be overcome, to wit:

- a) removal of binder, usually an organic such as polyethylene
- b) use of powders of smaller diameter than that of the fibers
- c) successful post-molding consolidation, probably by HIPing, to achieve full density.

While work is underway on these problem areas, progress has been slow, and no fracture data on injection molded products have yet been obtained.

The other aluminides discussed in this paper: Ti₃Al+Nb, TiAl, Al₃Ta and NiAl, display at most 2-3% elongation in tension at room temperature. Several properties must, therefore, be improved simultaneously for composites based on these alloys to be useful: a) improved strength and creep resistance and b) improved ductility or toughness. While it has certainly been possible to markedly improve the tensile strength of Ti₃Al+Nb over a wide temperature range, Fig. 8, there has been no report of the influence of SiC on toughness at low temperatures, except for a reference to plasticity at temperatures of at least 200°C (Brindley *et al.*, 1988). In the case of TiB₂-reinforced NiAl made by the XD process improved toughness has not resulted (Mannan and Kumar, 1988).

While it has been universally recognized that good interfacial properties are central to a functioning composite system, it is by no means certain what type of interface is required for the best combination of strength and toughness. Certainly for brittle matrices such as ceramics and many of the intermetallics, crack deflection along weak interfaces is desirable to improve toughness. On the other hand, fiber pull out has long been cited as an important factor in determining toughness (Chawla, 1987). The length of fiber should be large but close to the critical transfer length, l_c , to maximize the work of pull-out and to prevent the composite from breaking into two halves. At the same time l cannot be less than l_c for this will not allow the fibers to reach their tensile strength and therefore the full strengthening potential of the fibers will not be reached. Generally, fiber pull out contributes more to toughness than does fiber-matrix debonding, and this is confirmed in the case of Al₃Ta-Al₂O₃ composites (Anton, 1988). Of course, debonding must precede pullout. For the case of Ni₃Al+B matrices with Al₂O₃ fibers the critical transfer length $l_c = 7.8 \times 10^{-3}$ cm, while for IC-218/Al₂O₃ composites $l_c = 4.2 \times 10^{-3}$ cm.

For both matrices the critical length is much less than the actual fiber lengths used, yet strengthening was minimal. The most likely explanation for this result is that:

- a) the fibers were not wetted by the matrix
- b) many interfaces in the random composite were acted upon by high tensile stresses, leading to early crack initiation along the interfaces. The high notch sensitivity of Ni₃Al has previously been reported (Stoloff *et al.*, 1987); therefore, cracks once initiated propagate readily to fracture.

There is general agreement that Al₂O₃ particles as well as fibers are compatible with Ni₃Al-type alloys. In fact, Povirk *et al.*, (1988) suggest that good bonding without appreciable reactivity was achieved in hot pressed Ni₃Al+B/Al₂O₃ and IC-218 Al₂O₃. Ni₃Al+B/Al₂O₃ exhibited 10% elongation at room temperature compared to about 2% in our own work.

In the hot pressed alloy, most fibers oriented transverse to the tensile axis debonded from the matrix, while fibers oriented parallel to the tensile axis tended to fracture; fibers inclined to the tensile axis showed both mechanisms (Povirk *et al.*, 1988), see Fig. 8. In reactively sintered or reactively HIPed composites, on the other hand, debonding was the principal failure mode at all fibers. It is possible that the very short reaction times (albeit at very high temperatures) during reactive processing are inadequate to produce sufficient bonding to force the fibers to fracture under stress. Hot pressed composites with the IC-218 matrix were brittle, probably due to the formation of zirconia particles at the interface. Although these particles were quite evident also in HIPed IC-218/Al₂O₃ (Fig. 2), ductility reached 6% at 25°C, Table 3.

An alternative approach to improving the toughness of a brittle matrix is to use ductile reinforcements. This concept has proven successful when Nb powders are mechanically worked to produce pancake shaped particles in TiAl (Elliot *et al.*, 1988). However, two other ductile Ti-base alloy powders mixed with TiAl were not effective in improving toughness due to the formation of a hard, brittle reaction zone between the two phases (Elliot *et al.*, 1988). It is clear, therefore, that lack of chemical or mechanical degradation of the reinforcement during processing and service is the paramount consideration. Even when that problem is overcome, however, thermal expansion mismatch may lead to premature failure, especially during thermal cycling (Brindley *et al.*, 1988).

SUMMARY

Reactive HIPing of elemental and normal HIPing or hot pressing of prealloyed powders with randomly oriented short Al₂O₃ fibers has successfully produced fully dense composites based on Ni₃Al+B. Although the yield strength of Ni₃Al+B, HIPed at 800°C, is increased by the presence of fibers, no such beneficial effect is found with IC-218. In all cases ductility is reduced relative to monolithic materials. However, IC-218/Al₂O₃ HIPed at 1150°C and then heat treated did exhibit 6% elongation at 25°C. Injection molding techniques have been developed to orient Al₂O₃ fibers. Ni₃Al+B/Al₂O₃ composites made by hot pressing exhibited higher ductilities and better bonding than reactively sintered or HIPed product. The properties of Ti₃Al/SiC, TiAl/TiB₂ and Al₃Ta/Al₂O₃ composites also are briefly reviewed. Al₃Ta reinforced with continuous aligned fibers of Al₂O₃ appears to have promise as a high temperature composite, in part due to the excellent oxidation resistance of the matrix.

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