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Processing and properties of SiC-particulate reinforced titanium matrix composites by shock wave consolidation

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Résumé: En utilisant la méthode de consolidation par ondes de choc, des compacts de Ti, renforcés de particules de SiC, sans porosité, et ne présentant ni réaction interfaciale ni fissures macroscopique, ont été obtenus. Un recuit post-compactage approprié à également fait l'objet d'une étude de manière à améliorer la séparation des impuretés et par conséquent, la ductilité de la matrice. Des traitements thermiques additionnels du composite permettent de contrôler la taille des domaines de réactions interfaciales. Les résultats des tests de comportement mécanique suggèrent que la bonne ductilité du composite Ti-SiC est conservée pour des domaines de réactions interfaciales minimum ($<0,1 \mu\text{m}$) et qu'un domaine de réactions interfaciales de plus de $0,25 \mu\text{m}$ diminue la ductilité ainsi que la résistance du compact.

Abstract: Using shock wave consolidation, fully dense SiC-particulate reinforced Ti matrix composite compacts that are free from interfacial reactions and macroscopic cracks have been obtained. Proper post-consolidation annealing has been explored to improve impurity segregation and, thus, the ductility of the matrix. Various heat treatments of the composites produce controlled interfacial reaction zone sizes. Results of mechanical testing indicated that good ductility is retained in the Ti-SiC composites with a minimum interfacial reaction layer ($<0.1 \mu\text{m}$), while no interface reaction or an interfacial reaction layer of $0.25 \mu\text{m}$ thick or more is detrimental to the tensile ductility and strength of the composites.

1. INTRODUCTION

The extreme chemical reactivity of titanium provides a major challenge in selecting reinforcement materials and processing techniques for Ti matrix composite production [1]. While titanium matrix composites offer improved high temperature characteristics compared with aluminum matrix composites, avoiding detrimental interfacial chemical reactions during *fabrication* of titanium composites is crucial to the material performance. Continuous SiC fiber-reinforced titanium composites have been successfully produced with a $1\text{-}5 \mu\text{m}$ carbon-rich coating on the SiC fibers. Carbon coating is introduced to reduce and control the interfacial reaction during processing and service [2-4]. However, little work exists at present in exploring a similar interface-controlled toughening approach for *particulate*-reinforced

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titanium matrix composites. The standard processing procedures used in making particulate-reinforced composites, such as casting and hot pressing, often expose the composite to high temperatures for an extended time and are unsuitable for reactive composite systems. Diffusion barriers are difficult to produce on particles and the processing routes used for fiber-reinforcement titanium composite are usually not applicable to SiC particulate-reinforced titanium MMCs. Loretto and Konitzer [5] reported virtually no ductility and a low tensile strength of SiC particulate reinforced Ti-6Al-4V composites. Their composite compacts made by powder blending and hot pressing are fully dense but have thick reaction zones (4-5 μm).

Shock wave consolidation of powders has recently been explored for SiC particulate reinforced titanium composite by Christman *et al.* [6] and Tong *et al.* [7] and SiC particulate reinforced titanium-aluminide composites by Rhodes *et al.* [8]. The extremely high pressure and short process time (on the order of a microsecond) achieved during shock consolidation have made it feasible to obtain *fully dense compacts with a minimum of interfacial reactions*. Here we report our efforts in optimizing the processing procedure for fabrication of Ti-SiC composites through shock wave consolidation and results of mechanical testing of processed composites.

Fig. 1 Schematic of Caltech dynamic compactor.

2. SHOCK WAVE CONSOLIDATION OF TI AND TI-SiC POWDERS

In our present study, Ti-SiC metal matrix composites were fabricated with the Keck Dynamic Consolidation Facility. A schematic of the target fixture and propellant driven flyer plate assembly is shown in Fig. 1. A 31.5 mm diameter flyer plate is mounted onto a nylon sabot and accelerated by smokeless shotgun or pistol powder. The densification of the powders is accomplished by the passage of a strong shock wave generated upon the impact of flyer onto the green compact, the target powder [7-10].

Three titanium compacts (#150, #155, and #158) made from three types of unreinforced Ti powders and the three corresponding SiC particle reinforced composite compacts (#151, #152, and #154) were

Table 1 Results of Chemical Analysis of Titanium Powders (weight ppm)

Designation	O	H	C	N	Fe	O* atomic ppm ^c	Comments
Type I ^a	1250	42	189	52	71	465	99.9%,-100 mesh
Type II ^a	1740	92	159	53	98	602	99.5%,-325 mesh
Type III ^a	2300	17	354	1500	1810	1795	ball milled Type II powder for nine hours, -40 mesh
CP Titanium ^b	1800	150	1000	300	2000	1035	ASTM Grade 1, maximum impurity limit is listed

a—Teledyne Wah Chang Laboratories (Albany, Oregon);

b—ASM Metals Handbook [11];

c—Equivalent oxygen concentration [16].

obtained free of macroscopic defects. All composite compacts were reinforced with 10% by volume SiC. The chemical composition of three types of titanium powders is listed in Table 1. Also listed in Table 1 are the maximum impurity limits of commercially pure (CP) ingot processed Ti. A summary of our shock wave consolidation experiments is given in Table 2. The density of each compact was determined using the immersion method of weight measurements in air and toluene. Microhardness measurements were carried out using a Leitz Wetzlar Miniload hardness tester. X-ray diffraction (Inel CPS-120 diffractometer using Co K α radiation, $\lambda=1.7902$ Å) was used to verify the phases in the Ti compact. SEM (CamScan, at 15 keV) was used to study details of fracture surfaces, Ti-SiC interface zones, and Ti-Ti particle boundaries. 3-mm diameter TEM samples were prepared by ion milling and were examined using a Philips EM430. Specimens were fractured *in situ* under ultra high vacuum ($<3 \times 10^{-9}$ Torr) and the freshly created fracture surfaces were rapidly examined by high resolution scanning Auger electron microscopy (Perkin-Elmer).

Elastic moduli of both Ti and Ti-SiC compacts were determined by using the measurements of ultrasonic wave speeds. The compression tests were conducted using an Instron Universal Materials Testing System 4204 at a constant crosshead speed of 0.5 mm/min (the resulting strain rate was about 4×10^{-3} s $^{-1}$). Three-point bending tests were conducted to assess the tensile properties of the compacts. Analysis of linear elasticity and power-law plasticity was used to compute both the maximum stress and strain within the bending specimens (for details, see Tong *et al.* [7]).

Table 2 Summary of Shock Parameters for Matrix and Composite Compacts

Shot No.	Flyer Velocity (m/s)	Peak Pressure (GPa)	Shock Energy (kJ/kg)	Homogeneous Temperature (°C)	Initial Porosity	Powder Type	Mixing & Degassing*
150	1023	4.20	407.3	689	0.4571	Ti (Type I)	Schedule 1
151	1055	4.40	432.0	723	0.4600	Ti (Type I) + SiC (#600)	Schedule 1
155	939	3.42	356.2	617	0.4753	Ti (Type II)	Schedule 1
152	1095	4.61	464.5	766	0.4664	Ti (Type II) + SiC (#600)	Schedule 1
158	978	4.11	369.6	636	0.4382	Ti (Type III)	Schedule 2
154	963	4.07	357.6	619	0.4328	Ti (Type III) + SiC (#600)	Schedule 2

* Powder mixing and pre-compaction degassing schedules:

1. wet mixing in toluene, vacuum outgassing at 600°C for 6 hours;
2. ball milling for 9 hours in argon-filled vial, vacuum outgassing at 600°C for 12 hours.

3. EXPERIMENTAL RESULTS

3.1 Unreinforced Titanium Compacts

Typical results of mid-point deflection versus load in three-point bending tests are shown in Fig. 2 (A) for titanium compacts. For as-consolidated compacts, *all* bending specimens broke in the linear region which indicated a brittle type failure. An SEM observation of the fracture surface of the as-consolidated compact #150 indicated that the fracture occurred along the particle-particle boundaries in the bending specimen. Shock wave consolidated titanium and stainless steel compacts obtained by others have also shown very little tensile ductility in the as-consolidated state [12-13]. Failure at the particle-particle boundaries in bending tests indicates a weak and brittle bonding between particles. Brittleness and low strength of particle-particle boundaries are due to the localized deformation and the concentration of contaminants (*i.e.*, such as oxides and other impurities) around particle surfaces. To improve the ductility of shock consolidated compacts, proper annealing should thus be selected to reduce or eliminate prior particle-particle boundary effects in the compacts and to diffuse surface contaminants into particle interiors [14-15]. Annealed specimens of Ti compacts #150 and #155 indeed deformed plastically up to a maximum plastic strain of at least 10% *without* failure in bending tests (see Fig. 2A).

3.2 Ti-SiC Composites

No apparent reaction layer was observed at the Ti-SiC interface in any as-consolidated composite compacts using SEM and TEM. Some results of three-point bending tests for as-consolidated and annealed composite compacts are shown in Fig. 2 (B). The effects of interfacial reaction layers on the compressive and tensile properties of Ti-SiC composites are illustrated in Figs. 3 (A) and (B). As shown in Fig. 3 (A), the compressive strength of Ti-SiC composites increases with increasing annealing temperature (*i.e.* with interfacial reaction zone size). For specimens annealed at 850°C and 950°C, specimen cracking was visible at a compressive strain of 0.3-0.4 which indicated a somewhat less ductile

Fig. 2 Results of bending tests of as-consolidated (a) and annealed (b) specimens made from Ti compact #155 (A) and Ti-SiC compact #152 (B). The specimen was annealed at 650°C for 45 hours in vacuum. The load is normalized by the elastic limit load and the deflection is normalized by the corresponding elastic limit. Solid line — experimental; dashed line — analytical. The insert is the elastic-plastic stress-strain curve of Ti and Ti-SiC from the bending test.

titanium matrix. On the other hand, any significant amount of interfacial reaction (say $x \geq 0.25 \mu\text{m}$) in Ti-SiC composites degraded their tensile ductility, as shown in Fig. 3 (B). Virtually zero ductility was observed for as-consolidated samples (brittle Ti particle-particle boundaries and weak bonding between Ti and SiC particles) and annealed at 950°C for two hours (heavily reacted SiC particles). Plastic yielding was observed for samples annealed at 750°C and 850°C for two hours but their tensile ductility was still very poor ($\leq 1\%$). Good ductility ($>5\%$) was obtained only for the sample annealed at 650°C for 45 hours in which only a minimum of interfacial reaction occurred ($x \leq 0.1 \mu\text{m}$) and the impurities around titanium particles were fairly well diffused.

Fig. 3 Results of compression tests (A) and bending tests (B) of the Ti-SiC compact (#152) with a range of interfacial reaction zone thickness, x : (a) as-consolidated ($x \approx 0 \mu\text{m}$); (b) annealed at 650°C for 45 hours ($x \approx 0.05\text{--}0.1 \mu\text{m}$); (c) annealed at 750°C for two hours ($x \approx 0.25\text{--}0.35 \mu\text{m}$); (d) annealed at 850°C for two hours ($x \approx 0.85 \mu\text{m}$); (e) annealed at 950°C for two hours ($x \approx 3.9 \mu\text{m}$).

4. CONCLUSIONS

Shock wave consolidation has proved to be an effective technique to process SiC-particulate reinforced titanium metal matrix composites with 100% theoretical density, little or no interfacial reaction and free of macroscopic defects. Elimination of prior titanium particle-particle boundary effects in as-consolidated compacts by proper post-consolidation annealing results in a significant increase in tensile strength and ductility of both titanium and its composite compacts. Microcracking of large SiC particles during consolidation results in elastic moduli of composites lower than theoretical estimates. A ball-milling process is used to reduce the size of SiC particles, the resulting composites have elastic constants which are comparable to theoretical predictions. In comparison with the compacts made of regularly mixed powders, the yield strength of both titanium and its composites made of ball-milled powders is much higher. The ductility of the composite compact made by ball-milled powders, however, is very limited in both compression and bending tests. This brittle behavior is due to a much higher level of impurities (especially N and O) induced during ball-milling. Improved mechanical properties could be obtained by processing composite compacts using regularly mixed *micron-size* SiC and titanium powders.

Properly annealed 10% volume fraction SiC particulate reinforced titanium composites (#151 and #152) made of as-received powders show improved tensile strength over that of unreinforced Ti matrix while their tensile ductility remains excellent (>5%). In comparison, heavily reacted SiC reinforced titanium composites show degraded tensile strength and poor ductility ($\approx 0.16\%$) as reported by Loretto and Konitzer [5]. Interfacial reaction zone sizes from a fraction of a micron to several microns have been obtained for shock wave consolidated Ti-SiC composites by further post-consolidation annealing and the interfacial reaction rate is consistent with those reported in the literature [17-20]. Our results of mechanical testing indicated that good ductility is retained in SiC particulate-reinforced Ti metal matrix composites with a minimum interfacial reaction layer ($<0.1 \mu\text{m}$), and an interfacial reaction layer of $0.25 \mu\text{m}$ thick or more due to the reaction is detrimental to the tensile ductility and strength of the composites.

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REFERENCES

- [1] Loretto M.H. and Johnson T.P., *J. Microscopy* **169** (1993) 131-138.
- [2] Whatley W.J. and Wawner F.E., *J. Mater. Sci. Lett.* **4** (1985) 173-175.
- [3] Wei W., *J. Mat. Sci.* **27** (1992) 1801-1810.
- [4] Warriar S.G. and Lin R.Y., *JOM* **45** (1993) 24-27.
- [5] Loretto M.H. and Konitzer D.G., *Met. Trans.* **21A** (1990) 1579-1587.
- [6] Christman T. *et al.*, *Scripta Metall. et Mater.* **25** (1991) 631-636.
- [7] Tong W. *et al.*, submitted to *Acta Metall. Mater.* (1993).
- [8] Rhodes D. *et al.*, *Metall. Trans.* **21A** (1990) 1589-1594.
- [9] Gourdin W.H., *Prog. Mater. Sci.* **30** (1986) 39-80.
- [10] Schwartz R.B. *et al.*, *Acta Metall.* **32** (1984) 1243-1255.
- [11] ASM Metals Handbook (American Society for Metals, Metals Park, Ohio, 1980, 3) p.374.
- [12] Chiba A. *et al.*, *Scripta Metall.* **22** (1988) 213-217.
- [13] Wright R.N. *et al.*, *Metall. Trans.* **20A** (1989) 2449-2457.
- [14] Nakajima H. and Koiwa M., *ISIJ-International* **31** (1991) 757-766.
- [15] Takahashi Y. *et al.*, *J. Mat. Sci.* **27** (1992) 485-498.
- [16] Okazaki K. and Conrad H., *Acta Metall.* **21** (1973) 1117-1138.
- [17] Snide J.A., AFML Report No. AFML-TR-67-354 (1968).
- [18] Klein M.J. *et al.*, AFML Report No. AFML-TR-69-242 (1969).
- [19] Chamberlain M.B., *Thin Solid Films* **72** (1980) 305-309.
- [20] Martineau M. *et al.*, *J. Mat. Sci.* **19** (1984) 2749-2761.