Explosive shock-compression processing of titanium aluminide/titanium diboride composites

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Explosive shock-compression processing is used to fabricate Ti_3Al and TiAl composites reinforced with TiB_2. The reinforcement ceramic phase is either added as TiB_2 particulates or as an elemental mixture of Ti + B or both TiB_2 + Ti + B. In the case of fine TiB_2 particulates added to TiAl and Ti_3Al powders, the shock energy is localized at the fine particles, which undergo extensive plastic deformation thereby assisting in bonding the coarse aluminide powders. With the addition of elemental titanium and boron powder mixtures, the passage of the shock wave triggers an exothermic combustion reaction between titanium and boron. The resulting ceramic-based reaction product provides a chemically compatible binder phase, and the heat generated assists in the consolidation process. In these composites the reinforcement phase has a microhardness value significantly greater than that of the intermetallic matrix. Furthermore, no obvious interface reaction is observed between the intermetallic matrix and the ceramic reinforcement.

1. Introduction

Intermetallic compounds are emerging as the high-temperature oxidation-resistant materials of the future [1-4]. In current research activities, considerable effort is concentrated on the use of Ti-Al intermetallic compounds because of their attractive characteristics of low density, high melting temperature, high strength, high corrosion and oxidation resistance [5-7]. In some cases, these materials exhibit improved strength with increasing temperature. Furthermore, recent research trends in processing technology have resulted in improved ductilities, thereby making intermetallics more useful than certain ceramics which have failed to keep up to their promise.

Conventional casting of large Ti-Al intermetallic compound ingots is difficult because of problems with brittleness and segregation. Considerable research within the materials community has also been devoted to the development of other processing techniques for making these alloys with improved mechanical properties. These new techniques are less well developed for Ti-Al alloys than for nickel-based alloys, because the primary liability of Ti-Al alloys is inadequate ductility at low temperatures. The ductility of these alloys can be significantly enhanced by grain-size refinement, which is possible [8] by techniques employing rapid solidification rate (RSR) processing, but creep resistance is adversely affected in RSR. If, however, a technique is developed to produce a dispersion of fine discrete particles of a refractory ceramic phase in Ti-Al alloys, then the creep resistance can be improved along with some enhancement in toughness.

At the same time, if the fine dispersion is thermodynamically stable, then an increase in elevated temperature strength can also be obtained.

Ceramic-reinforced intermetallic matrix composites offer significantly higher operating temperature capability and strength-to-weight ratios than those currently available. However, key problem areas which limit the successful development of these materials include reinforcement fibres or particulates that are currently available, matrix materials and composite processing methods. Particular issues to be resolved are mismatches in the coefficient of thermal expansion, chemical compatibility between fibre and matrix materials, and composite fabrication and joining processes.

The conventional method of fabricating composites involves sandwiching the reinforcement between sheets of matrix material, followed by hot pressing. Alternatively, rapidly solidified powders of the matrix material are mixed with the reinforcement and consolidated by hot pressing. Processing titanium aluminides in this manner is difficult because they have poor formability characteristics, they interact with the reinforcement at the temperatures and times needed for consolidation, and there is a thermal expansion mismatch between the titanium aluminide and reinforcement which can lead to extensive cracking during cooling from the consolidation temperature.

Among the alternative processing methods, techniques utilizing exothermic chemical reactions between elemental constituents, for example the combustion synthesis process [9, 10] and the Martin Marietta

XD Process [11], are unique because prolonged heating can be avoided and bulk solids with refined microstructures can be produced. In these processes, the high temperatures necessary for adequate metallurgical bonding are produced and cooling is attained in time durations too small to cause microstructural changes and significant atomic mobility. Thus, thermally-induced metallurgical and microstructural changes can be minimized.

Explosive shock-compression processing, utilizing the extremely rapid and intense deposition of energy in the powders by shock waves, can also be used. Shock compression loading places a material in an unusual combination of pressure and temperature states. In the case of a porous mass, the significant increases in pressures and temperatures can cause interparticle bonding between powders [12–19] as well as solid-state chemical reactions between powders [20, 21]. The consolidation of the powder occurs due to the shock energy being preferentially deposited at particle surfaces during the passage of shock waves. In the case of metallic systems, powder consolidation is achieved by deformation and high-velocity impact of particles, collapse of voids, and break down of surface oxides, leading to preferential heating and melting of particles near the surface and subsequent welding. The mechanism of consolidation in ceramics involves fracture and/or deformation of particles, filling of interstices, cleansing of surfaces, and preferential heating of particle surfaces leading to partial melting and welding or solid-state diffusional bonding.

Shock compression processing has several unique characteristics. Metastable, as well as fine, homogeneous structures of the starting powders can be preserved in the compacted material because the mean bulk temperatures generated during shock compression processing are considerably below the recrystallization temperature for most materials. Furthermore, the resulting compact often undergoes shock-induced strengthening which may result in improved mechanical properties. Finally, the technique can be used to synthesize novel phases and unique composites unattainable by conventional processing.

In this investigation explosively generated shock waves were used for processing titanium aluminide/titanium diboride composites. The experimental techniques and the characteristics of the shock-processed materials will be discussed.

2. Experimental procedure

Two types of RSP Ti–Al alloys were used in this study for the matrix material (Ti_3Al, α_2-b c t phase and TiAl, γ-h c p phase compounds). These were reinforced with TiB_2 powder particulates by the explosive shock-processing technique. A variety of geometries and fixtures are available for shock consolidation of powders and have been documented in the literature [15, 17]. These require a persistent high-pressure shock-wave loading over as wide an area as possible, thus ensuring a state of uniaxial strain over a large volume. Shock waves can be generated by (a) impact of a projectile which is accelerated in a gun either by compressed gas, gun powder, or even electromagnetically against the powder or a powder container, or (b) detonation of an explosive either in direct contact with the powder (or container) or explosively accelerated plate impact.

For the present work the detonation of explosives was used to generate shock waves for consolidating the powders. The geometry employed was a 12-capsule flyer plate impact system. The fixture configuration and the explosive loading assembly are shown in Fig. 1. A steel flyer plate is accelerated downwards by the detonation of an explosive charge, which is initiated by a plane-wave-generating conical lens assembly. In the present case the flyer plate was accelerated over a 10 mm stand-off distance, to impact the powders contained in steel recovery fixture at a velocity of 950 m sec^{-1}. The advantage of using this system is that one can use twelve different powder mixture combinations and subject them to identical shock conditions.

In the present work, eight different powder mixtures of varying matrix-to-reinforcement volume fractions were prepared (Table I) and loaded in the capsules at approximately 65% of the theoretical densities. The
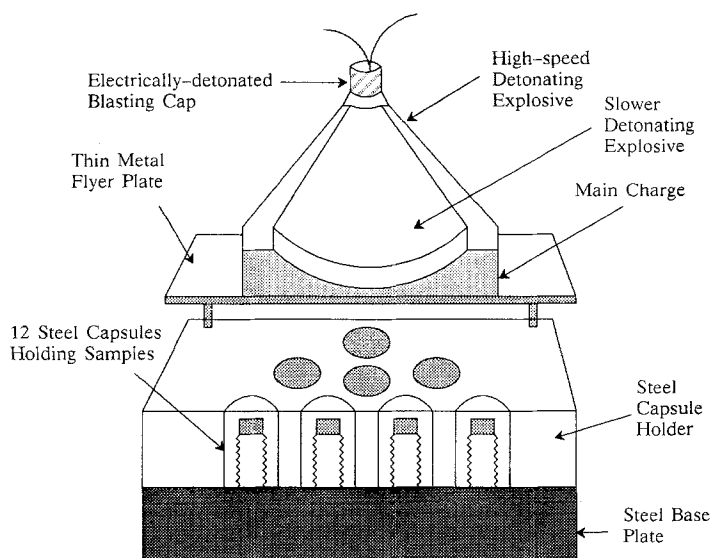

Figure 1 Schematic illustration of the 12-capsule shock recovery fixture and assembly for the high-pressure shock experiments.

TABLE I Matrix-reinforcement powder characteristics

Sample	Matrix	Reinforcement	Proportion (M:R)
1	Ti_3Al	—	100
2	Ti_3Al	TiB_2	70:30
3	Ti_3Al	Ti + B	70:21:9
4	Ti_3Al	TiB_2 + Ti + B	70:15:10.5:4.5
5	TiAl	—	100
6	TiAl	TiB_2	70:30
7	TiAl	Ti + B	70:21:9
8	TiAl	TiB_2 + Ti + B	70:15:10.5:4.5

Figure 2 Schematic illustration of the principle of shock-induced reaction-synthesis-assisted consolidation of very hard powders.

recovered shock-loaded compacts were characterized by optical microscopy and microhardness testing. It was not the objective of this study to determine the bulk properties of the shock-processed composites at ambient and elevated temperatures. Rather the emphasis was on characterization of the microstructure to determine the nature of bonding between the matrix and the reinforcements.

3. Results and discussion
In order to make titanium aluminide composites reinforced with titanium diboride, various mixture combinations were prepared. As listed in Table I, the Ti_3Al and TiAl alloy powders were mixed with either TiB_2, Ti + B, TiB_2 + Ti + B such that the titanium aluminide matrix always constituted 70% of the total composite volume. The matrix alloy powders were also consolidated by themselves in order to obtain a comparison between the properties of the composite and the intermetallic. The elemental titanium and boron powders were added with the intent of producing a shock-induced exothermic reaction between the two, such that the ceramic-based reaction product and the heat generated can assist in the consolidation of the titanium aluminide. This concept has also been the basis of a CETR patented technology [22] for consolidating very hard and difficult-to-bond ceramics. The principle of this concept is illustrated schematically in Fig. 2. In essence, the ceramic powder is mixed with an elemental powder mixture of titanium and boron, or titanium and carbon. The passage of a shock wave through this mixture initiates an exothermic combustion reaction resulting in the formation of a ceramic compound. The reaction product then provides a binder phase and the reaction heat assists in the consolidation of the ceramic.

The results of the Ti_3Al-based composites and the TiAl-based composites will be discussed next. The Ti_3Al- and TiAl-based composite compacts were recovered as fully dense and almost crack-free pellets (12 mm diameter × 5 mm thick). Optical micrographs of the Ti_3Al compact and TiAl compact containing no reinforcement are shown in Fig. 3a, b and c, d, respectively.

The white non-etching interparticle regions in Fig. 3a and the dark dendritic interparticle regions in Fig. 3b correspond to the near-particle surface material
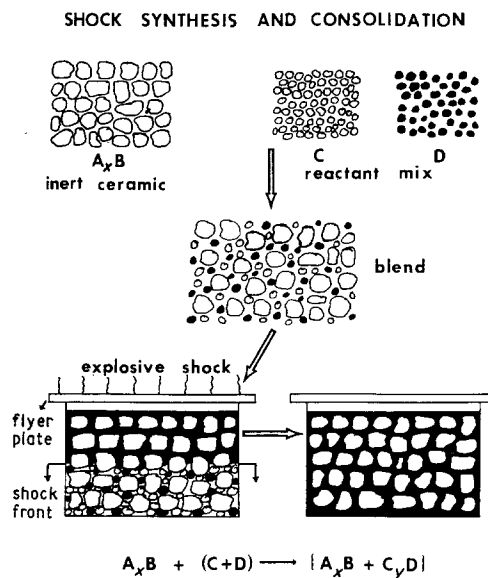
that melted and resolidified during the shock-consolidation process. The white contrast of the interparticle regions (in Fig. 3a) is characteristic of rapidly solidified microcrystalline material, while the dark contrast and the dendritic grains in Fig. 3b are also characteristic of the material that melted, but solidified relatively slowly. In fact, because of the large amount of melt, the solidification process resulted in the entrapment of a shrinkage void.

In contrast to Ti_3Al alloys, the TiAl compounds are much harder. Optical micrographs of the TiAl compacts shown in Fig. 3c and d, also reveal good bonding between the individual powder particles. However, in this case the amount of interparticle melting is limited. Also, due to the inherent brittle nature of this TiAl alloy, micro-cracking is observed as shown in Fig. 3d. It is important to note that the cracks, generated by tensile wave reflections, are transparticle in nature, which indicates strong bonding between the individual powder particles. These micrographs indicate that the structural characteristics of the starting Ti_3Al and TiAl powder particles are fully retained and that they are consolidated due to localization of shock energy at interparticle regions. This localization results in extensive plastic deformation and heating and melting at interparticle regions and subsequent welding.

The optical micrographs of the Ti_3Al + TiB_2 mixture compact is shown in Fig. 4a and b and that of the TiAl + TiB_2 mixture compact in Fig. 4c and d. Good bonding has been attained between the coarse (80 to 100 μm) Ti_3Al powders and fine (4 to 10 μm) TiB_2 powder particles (Fig. 4a and b). Although the Ti_3Al powder particles have been extensively deformed, its fine grain size and the structural homogeneity have been fully retained. When coarse powders are mixed with fine powders, much of the shock energy is generally localized at the fine powders. In spite of the fact that the TiB_2 powders are significantly harder than
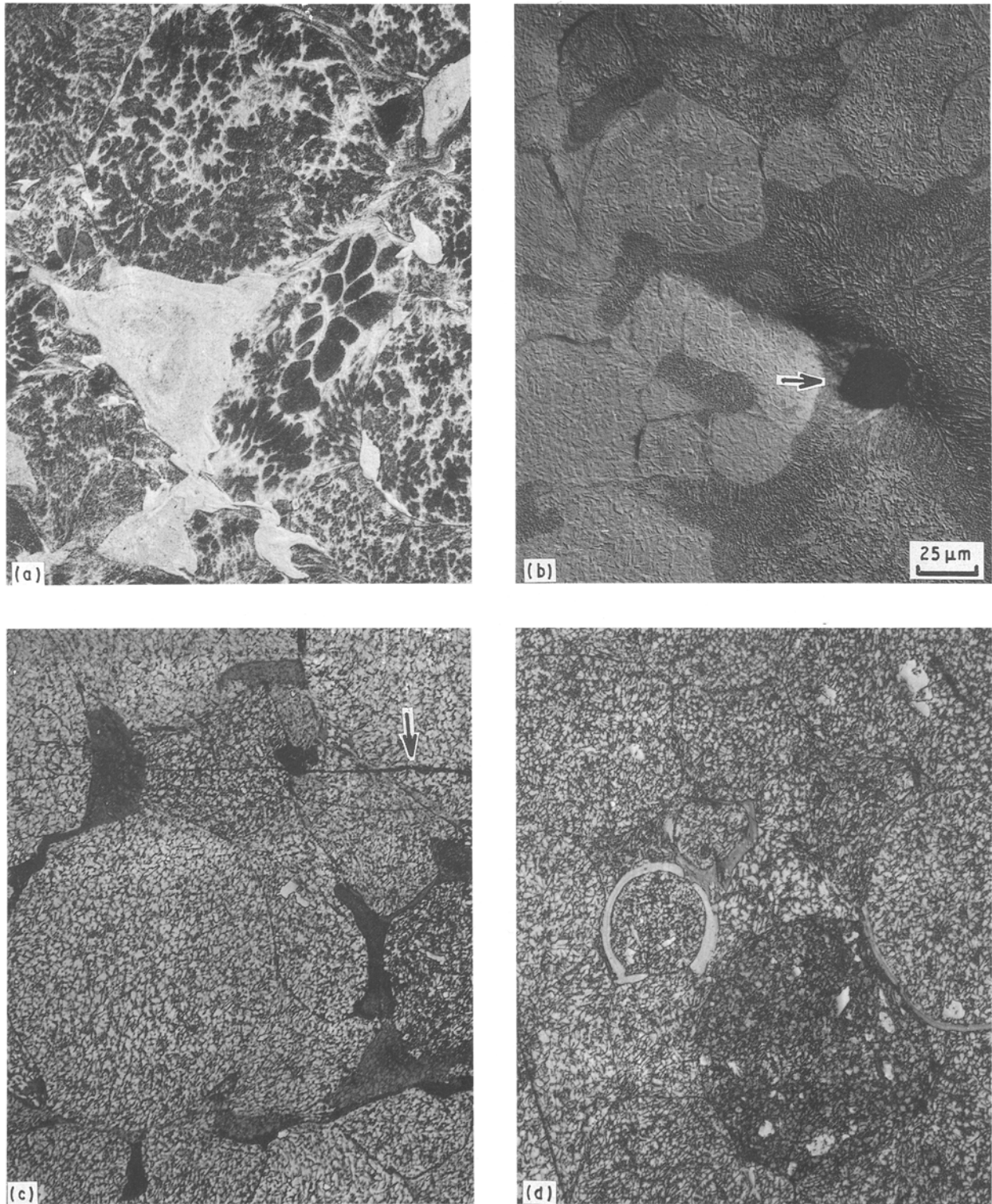
Figure 3 Optical micrographs of (a, b) Ti₃Al, and (c, d) TiAl compacts containing no reinforcement.

Ti$_3$Al, the localization of the shock energy on these fine TiB$_2$ particles appears to have resulted in a well-consolidated compact. Similar results are also seen for the coarse TiAl powders mixed with the fine TiB$_2$ particulates as shown in Fig. 4c and d.

The microstructure of the Ti$_3$Al and TiAl compacts mixed with elemental titanium and boron is shown in the optical micrographs in Fig. 5a, b and c, d, respectively. Although good bonding is observed in the various composite compacts, it is not clear if a shock-induced chemical reaction has occurred. The optical micrographs of these compacts show a fibrous structure at interparticle regions which may correspond to that of the melted and re-solidified reaction product.

A few fine unreacted titanium particles can also be seen (greyish particles in Fig. 5a and b and whitish in Fig. 5c and d, indicated by an arrow), but the bulk of the interparticle region has a fibrous structure. Thus, in the case of an elemental powder mixture, interparticle bonding appears to have occurred due to the
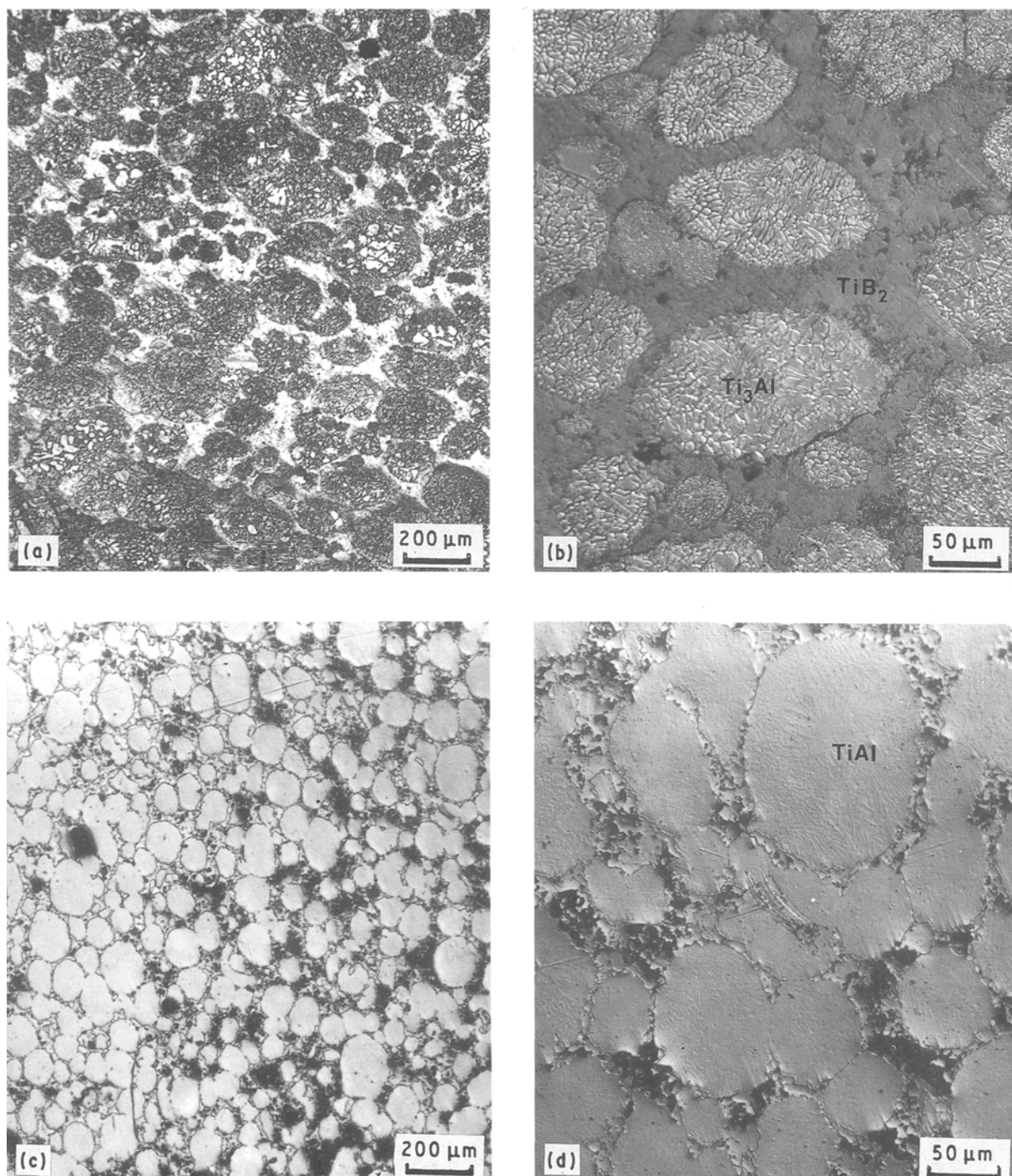
Figure 4 Optical micrographs of (a, b) Ti$_3$Al + TiB$_2$ mixture compact, and (c, d) TiAl + TiB$_2$ mixture compact.

binder phase provided by the shock-induced reaction product.

With the addition of fine TiB$_2$ + Ti + B powder mixture to the Ti$_3$Al and TiAl powders, the situation appears to be more complex. The optical micrographs of the Ti$_3$Al and TiAl powder compacts mixed with TiB$_2$ + Ti + B are shown in Fig. 6a, b and c, d, respectively. Whether the consolidation is aided by the fine TiB$_2$ particles or the shock-induced reaction between Ti + B is not clear. In the interparticle region marked by an arrow in the optical micrograph in Fig. 6b, it appears that fine TiB$_2$ particles are embedded in a possible reaction-product material. Although the adiabatic temperature resulting during the reaction

between titanium and boron is the same as the melting temperature of TiB$_2$, the contrast in this region reveals retention of the unmelted TiB$_2$ particles.

Microhardness measurements were conducted on these compacts of Ti$_3$Al and TiAl intermetallic compounds reinforced with TiB$_2$, Ti + B or Ti + B + TiB$_2$. The results of microhardness values (average of about 54 measurements on each sample) obtained at interparticle regions and within the particle interiors are listed in Table II. The average hardness of Ti$_3$Al particles ranges between 441 and 483 kg mm^{-2} and that of TiAl particles between 564 and 667 kg mm^{-2}. The microhardness of the interparticle region in the Ti$_3$Al compact (Sample 1) is 485 kg mm^{-2} which is
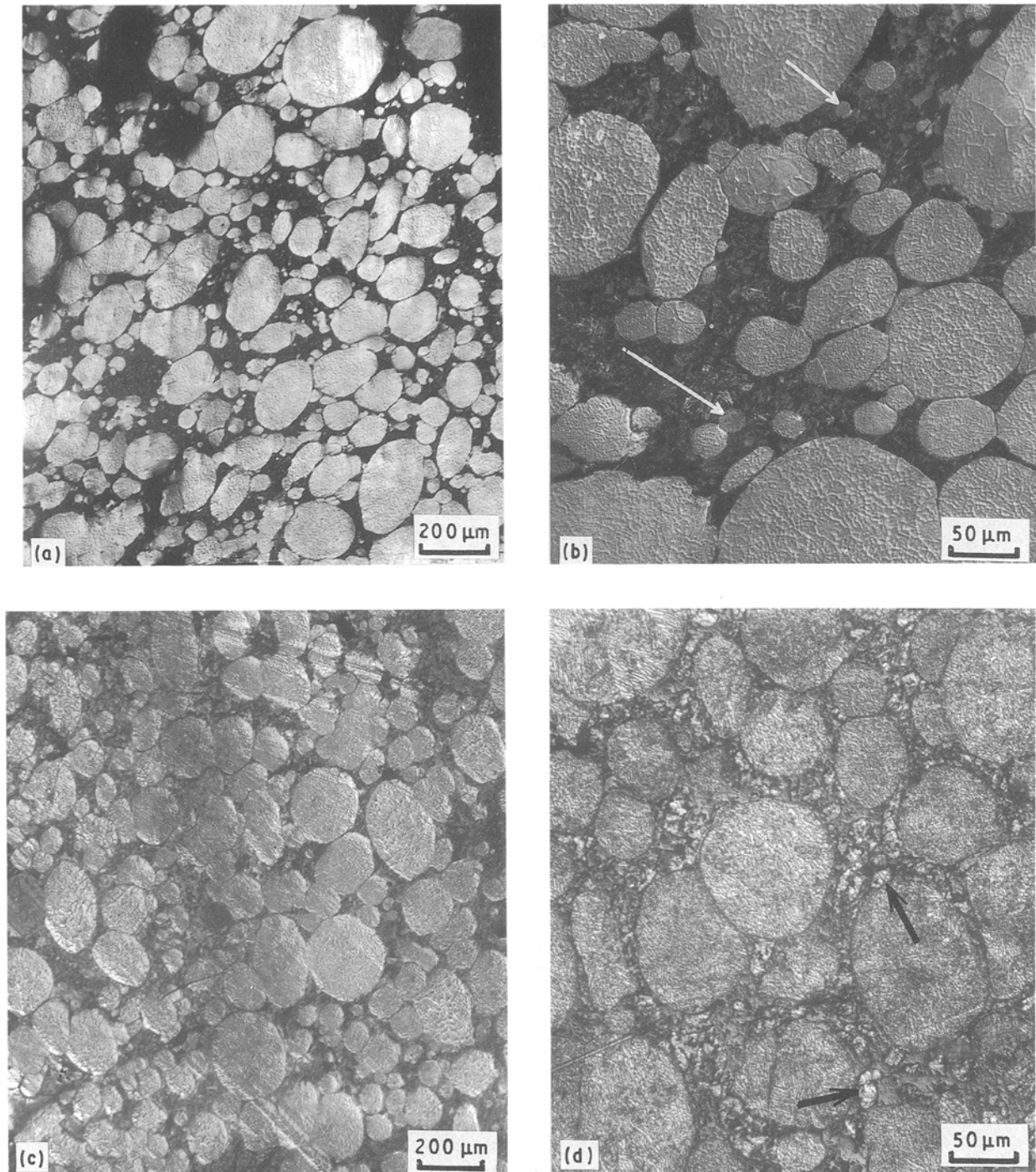
236

Figure 5 Optical micrographs of (a, b) Ti₃Al + Ti + B mixture compact, and (c, d) TiAl + Ti + B mixture compact.

somewhat higher than that in the particle interior. Because the interparticle regions in TiAl compact (Sample 2) were very small, accurate microhardness measurements were not possible.

In contrast, the microhardness at interparticle regions in those compacts containing TiB_2, Ti + B or TiB_2 + Ti + B, ranges between 634 and 976 kg mm^{-2} which is significantly greater than that in the particle interiors. These microhardness results indicate that except in the case of Sample 7 (containing TiAl + Ti + B) all of the compacts have a ceramic-based reinforcement, which was either added in the starting mixture or formed during the shock-compression process. The large standard deviation in microhardness

values at interparticle regions occur where the titanium and boron particles have remained unreacted. However, because the average hardness is greater than that of either constituents, it appears that the bulk of the elemental titanium and boron has reacted.

Limited SEM and EDX analysis was conducted to determine if the intermetallics reacted with either of the reinforcing TiB_2, Ti + B or TiB_2 + Ti + B powders. No evidence of a bulk interfacial reaction is observed within the limits of resolution with an SEM. Fig. 7a is a scanning electron micrograph of a compact containing Ti₃Al + TiB_2. The corresponding EDX analysis taken from a particle interior is shown
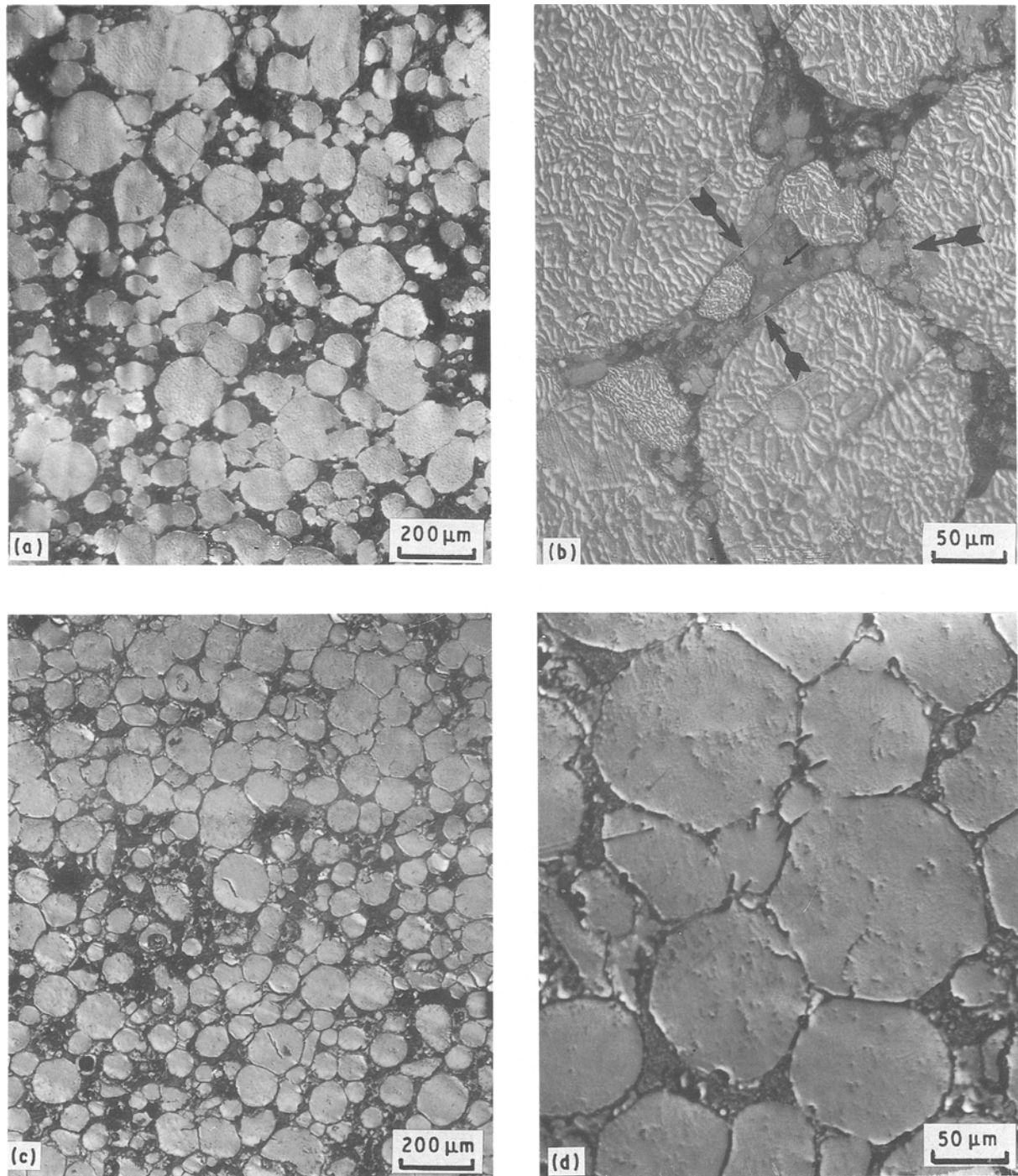
237

Figure 6 Optical micrographs of (a, b) Ti$_3$Al + TiB$_2$ + Ti + B mixture compact, and (c, d) TiAl + TiB$_2$ + Ti + B mixture compact.

TABLE II Microhardness values of shock-processed titanium aluminide/titanium diboride composite compacts

Sample	Constituents	Microhardness (kg mm^{-2})	
		Particle interior	Interparticle region
1	Ti$_3$Al	441 ± 39	485 ± 37
2	Ti$_3$Al + TiB$_2$	483 ± 28	634 ± 141
3	Ti$_3$Al + Ti + B	452 ± 60	795 ± 291
4	Ti$_3$Al + TiB$_2$ + Ti + B	477 ± 112	779 ± 248
5	TiAl	579 ± 92	—
6	TiAl + TiB$_2$	642 ± 52	976 ± 204
7	TiAl + Ti + B	564 ± 44	564 ± 231
8	TiAl + TiB$_2$ + Ti + B	667 ± 140	793 ± 291

in Fig. 7b and that from an interparticle region in Fig. 7c. Again the fine TiB$_2$ particles appear to have been well consolidated and assist in bonding the coarse Ti$_3$Al powders. The EDX analysis of the particle interior (Fig. 7b) shows titanium, aluminium and niobium peaks, whereas that of the interparticle region (Fig. 7c) shows predominantly titanium. Because a windowless EDX detector was not used, the presence of boron could not be detected. Although microprobe analysis or transmission electron microscopy may be more suited to determine matrix-reinforcement interfacial reactions, the SEM work provided a qualitative analysis that no bulk interfacial reactions had occurred.
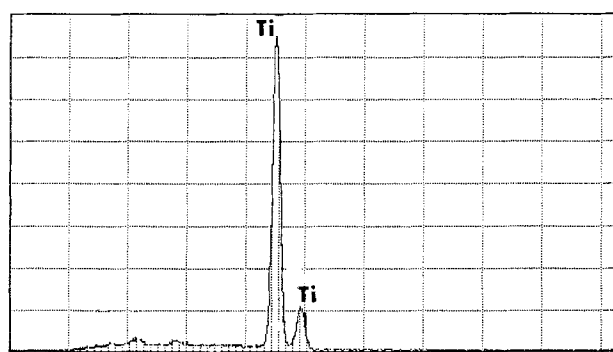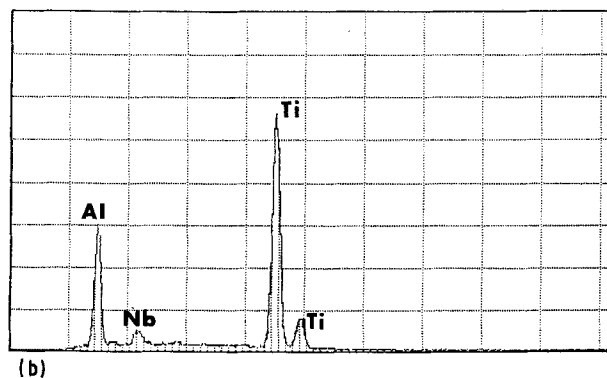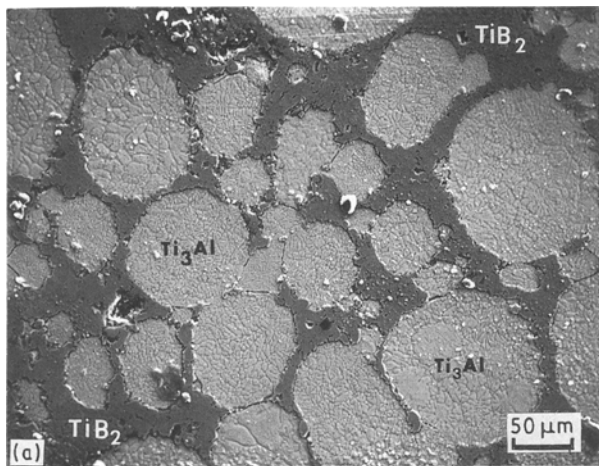
Figure 7 (a) Scanning electron micrograph of a compact containing Ti₃Al + TiB₂ mixture. (b, c) Corresponding EDX analysis taken from particle interior, and interparticle regions.

4. Conclusion

Titanium aluminide composites reinforced with titanium diboride compacts are fabricated using the shock-compression processing technique. The reinforcement is either added as TiB_2 particulates or an elemental mixture of titanium and boron, or both. In the case of the titanium aluminides Ti_3Al and TiAl, the consolidation occurs due to the localization of shock energy which results in plastic deformation and preferential heating and melting of the near-particle-surface material and subsequent welding. When fine TiB_2 particulates are added to the aluminides, the localization of the shock energy occurs at the small ceramic particles which undergo extensive plastic deformation and assist in consolidation of the coarse aluminide particles. With the addition of an elemental Ti + B powder mixture, the passage of the shock wave triggers an exothermic combustion reaction between titanium and boron. The resulting reaction product then provides a binder phase, and the heat generated assists in bonding the aluminide powders. Microhardness measurements indicate significantly higher hardness values at interparticle regions in contrast to those within particle interiors which further confirms the nature of the composite. Limited SEM and EDX analyses reveal no obvious reaction between the intermetallic matrix and the ceramic reinforcement.

References

1. J. SCHEFTER, *Popular Sci.* May (1986) 74.
2. S. W. KORTHALS-ALTES, *Technol. Rev.* January (1987) 43.
3. J. WADSWORTH, T. G. NIEH and J. J. STEPHENS, *Int. Mater. Rev.* 33(3) (1988) 131.
4. J. WADSWORTH, C. A. ROBERTS and E. H. RENNHACK, *J. Mater. Sci.* 17 (1982) 2539.
5. J. J. DE LUCCIA, R. E. TRABOCCO, J. WALDMAN and J. F. COLLINS, *Adv. Mater. Proc.* 5 (1989) 39.
6. T. M. F. RONALD, *ibid.* 5 (1989) 29.
7. J. D. DESTEFANI, *ibid.* 5 (1989) 37.
8. E. M. SCHULSON and D. M. BARKER, *Scripta Metall.* 17 (1983) 519.
9. W. L. FRANKHOUSER, K. W. BRENDLEY, M. C. KIESZEK and S. T. SULLIVAN, in "Gasless Combustion Synthesis of Refractory Compounds" (Noyes, Park Ridge, New Jersey, 1985).
10. Z. A. MUNIR, *Ceram. Bull.* 67 (1988) 342.
11. R. K. VISWANADHAM, S. K. MANNAN and B. SPRISSLER, "Nickel Aluminide/Titanium Diboride Composites", Martin Marietta Laboratories, TR-87-66C (1987).
12. "Dynamic Compaction of Metal and Ceramic Powders", National Materials Advisor Board, NMAB-394 (National Academy Press, Washington, D.C., 1983).
13. M. A. MEYERS and L. E. MURR (eds)., "Shock Waves and High-Strain-Rate Phenomena in Metals: Concepts and Applications." (Plenum Press, New York, 1981).
14. L. E. MURR, K. P. STAUDHAMMER and M. A. MEYERS (eds)., "Metallurgical Applications of Shock-Waves and High-Strain-Rate Phenomena" (Marcel Dekker, New York, 1986).

15. M. A. MEYERS, N. N. THADHANI and L. H. YU, "Explosive Shock Wave Consolidation of Metal and Ceramic Powders," "in Shock Waves for Industrial Applications," edited by L. E. Murr (Noyes, New Jersey, 1988) p. 265.
16. W. H. GOURDIN, *Prog. Mater. Sci.* **30** (1981) 51.
17. N. N. THADHANI, *Adv. Mater. Manuf. Proc.* **3** (1988) 493.
18. M. A. MEYERS and S. L. WANG, *Acta Metall.* **37** (1989) 925
19. N. N. THADHANI, A. H. MUTZ and T. VREELAND Jr, *ibid.* **37** (1988) 897.
20. "Shock Compression Chemistry in Materials Synthesis and Processing", NMAB Report no. 414 (National Academy Press, Washington, D.C., 1984).
21. R. A. GRAHAM, B. MOROSIN, E. L. VENTURINI and M. J. CARR, *Ann. Rev. Mater. Sci.* **16** (1986) 315.
22. T. AKASHI and A. B. SAWAOKA, US Pat. 4 655 830, 7 April (1987).