

The Use of Thermal Energy in Shock-Wave Compaction of Ceramics and Intermetallics

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Abstract

Shock consolidation is a method that presents a bright potential but has been limited by inevitable cracking of compacts. In an effort of eliminate cracking while retaining the unique features of shock consolidation, three novel approaches have been implemented:

- 1- Use of local shock-induced reactions to increase temperatures of particle interfaces and enhance bonding.
- 2- Shock densification at a low pressure (just above threshold for pore collapse) followed by hot isostatic pressing or sintering.
- 3- Shock consolidation of pre-heated specimens.

These techniques were applied to rapidly solidified titanium aluminide powder and to non-oxide ceramics (silicon carbide, cubic boron nitride and diamond). Reduction of cracking was observed with interparticle melting and reactions. Microstructural results and mechanical properties are discussed.

Introduction

The increasing demand for hard ceramics and intermetallics represents the subject of current interest for scientists and engineers. These materials such as silicon carbide, cubic boron nitride, diamond and titanium aluminides are found to be very difficult to densify due to their very high strength. SiC has a demonstrated capability as wear parts including seals, valves, nozzles and wear pads; its advantages in advanced heat engines have been widely publicized. Diamond and cubic boron nitride are suitable materials for cutting tools. However, these ceramic powders are very hard and difficult-to-consolidate due to their strong covalent bonding nature. Production of dense compacts without additive has not yet been successful. Ti-Al-Nb alloys based on Ti₃Al intermetallic composition have shown a strong potential to meet the requirements of high specific strengths at elevated temperatures. In addition to that, considerable efforts are concentrated on the use of Ti-Al-Nb intermetallic compounds because of their attractive

characteristics of low density, high corrosion and oxidation resistance.

Dynamic consolidation has considerable potential for densifying these high strength materials which are very difficult to sinter by conventional techniques. Formation of dense compacts requires the collapse of the gaps between the particles as well as considerable amount of energy deposited at the particle surfaces for interparticle bonding. This ultra rapid deformation and energy deposition produces partial melting at the particle surfaces followed by a rapid solidification via heat conduction into the interior of the particles. A series of attempts have been made to consolidate these difficult-to-consolidate powders [1,2,3]. However, there exist two major problems. One is cracking of the compacts at both the microscopic and macroscopic levels. The other is a lack of uniformity in microstructure and mechanical properties within the resulting compacts. Three novel approaches have been implemented: (1) shock consolidation of pre-heated specimens (2) use of local shock-induced reactions to increase temperatures of particle interfaces and enhance bonding (3) shock densification at a low pressure (just above threshold for pore collapse) followed by hot isostatic pressing (hipping) [4,5]. Preheating, low shock pressures and post-shock hiping were used in order to eliminate or minimize cracks in the resulting compacts.

Experimental Setups and Procedure

The dynamic processing experiments were carried out in two types of fixtures: high temperature Sawaoka planar impact fixture (600-700 °C) and room temperature double tube cylindrical. A schematic illustration of these setups are shown in Figures 1 and 2, respectively. Stainless-steel flyer plate was driven by explosives to velocities from 1.2 to 1.9 km/s and impacted against capsules in the Sawaoka system; in the double tube system, the flyer tube impact velocity was approximately 800 m/s [6]. The impact of the flyer projectiles on the system creates high amplitude shock waves that transmit through the powders, ranging from a few GPa to more

than one hundred GPa. A detailed description of the system is presented elsewhere [7]. The high temperature planar impact system was used for the consolidation of α -SiC, c-BN and diamond. Room temperature double tube cylindrical setup was used for the consolidation of Ti_3Al-Nb .

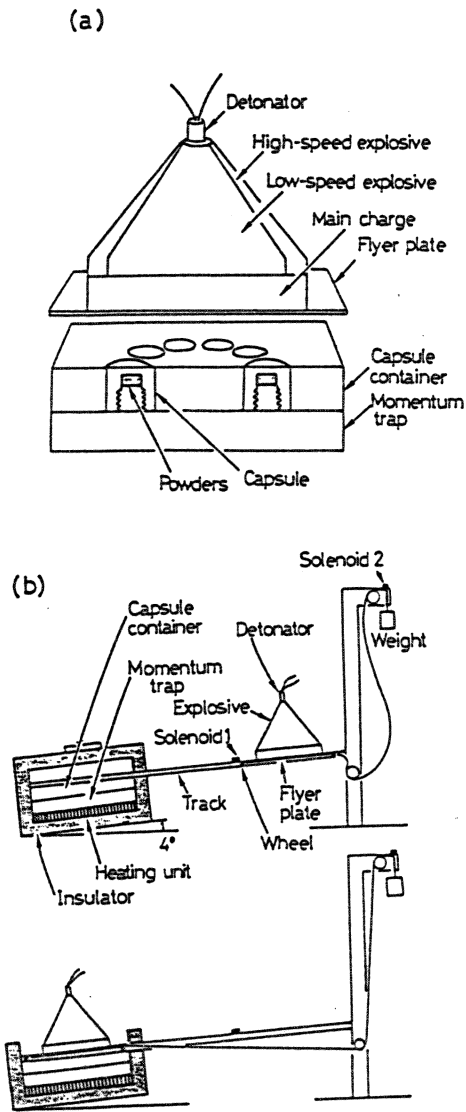


Fig. 1(a). Schematic illustration of planar impact system and (b) setup for hot consolidation.

Earlier experiments conducted by Wang et al. [8] on nickel-base superalloys indicated that preheating them to 500-700 °C had a very positive effect on the mechanical properties of shock consolidated superalloys. In general, the material yield strength decreases with increasing preheating temperature and the shock energy dissipated at the powder surfaces to produce melting layer is increased. The high temperature setup is described by Ferreira et al. [6]. For the second approach (addition of reactive elements), elemental powders were added to the ceramic powder and blended in a porcelain jar with a grinding medium until an apparent uniform color was achieved. In

this case, the elemental particles (because of their much lower strength) are deformed more intensely than the ceramic particles during the propagation of the shock wave, and they help to provide bonding among the ceramic particles by reacting exothermically and deformation heating. This technique was introduced by Akashi and Sawaoka for ceramics. Dynamic densification followed by hot isostatic pressing ("hipping") is the third approach used to improve the strength and ductility of the compacts. The powder was highly deformed and had a higher residual lattice strain after dynamic densification. Subsequent post-shock hipping is a good way to heal the residual strain and microcracks generated by passage of a rarefaction wave. Furthermore, the highly exothermic reactions of elemental powders (Ti and Al) added to the Ti_3Al will completely take place during the hipping stage and enhance the bonding between Ti_3Al particles.

Results and Discussion

A. Characterization of dynamically consolidated α -SiC powders

Two different sizes of pure SiC powders (7 and 44 μm) were used as starting materials. The high temperature planar impact system was used for the consolidation. Well-consolidated samples were obtained upon shocking preheated SiC powders with 65% of the theoretical density at an impact velocity of 1.9 km/s. The shock pressure was calculated to be 12 GPa by using the one dimensional impedance matching technique . However, cracking is not totally eliminated and circumferential cracks are noticeable. Scanning electron microscopic (SEM) analysis of recovered SiC compacts revealed that SiC particles underwent a considerable amount of deformation and the material was fully densified. Figure 3 showed microstructures of fracture surfaces of 7 μm SiC compacts. The fractograph indicates that cracks are transgranular and this is evidence for excellent bonding of the powders. The relative densities of

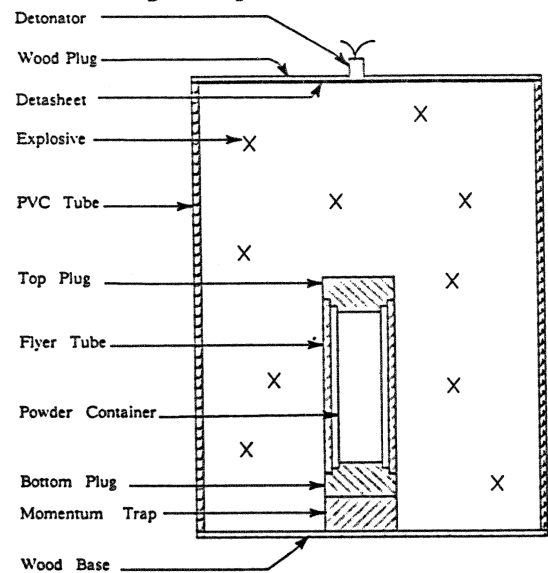


Fig. 2. Cylindrical axisymmetric double-tube system.

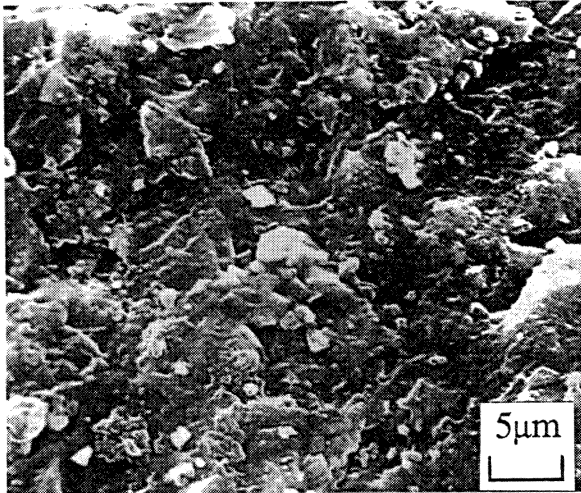


Fig. 3. Scanning electron micrograph of fracture surface of consolidated 7 μm SiC.

the 7 and 44 μm SiC compacts were 98.8% and 98.5% of theoretical, respectively. Both samples had a very close average microhardness value of 28 GPa. Transmission electron microscopic (TEM) analysis (Figure 4) revealed the substructural features of the 44 μm SiC compacts. We can observe lattice distortion in the interior of SiC particle, indicating that the particle was heavily deformed. The dark round regions (8-80 nm) in the micrograph are nanocrystalline SiC particles, while the white surrounding material corresponds to the amorphous phase. This interfacial layer was formed due to the very rapid collapse of the gaps between the particles as well as the rapid deposition of energy upon interparticle sliding. Furthermore, this thin interfacial layer (0.2-0.3 μm) and high thermal conductivity of SiC (13 W/m $^{\circ}\text{C}$) also promote the tendency for these nanocrystalline structure and amorphous phase. This energy deposition time is on the order of the wave transit time through the particle (a few nanoseconds). The melted or sublimated interparticle material resolidifies at cooling rates as high as 10^5 to 10^{10} $^{\circ}\text{C}/\text{s}$ [9]. It is believed that amorphous SiC phase forms at higher cooling rate than nanocrystalline structure and that the crystalline phase nucleates within the amorphous region while it is being cooled. However, another possibility is that melted or sublimated SiC reacted with oxygen to form $\text{Si}_x\text{C}_y\text{O}_z$ or SiO_2 which also has the tendency to form an amorphous phase.

B. Characterization of dynamically consolidated diamond powders

Three different sizes of natural diamond powders (4-8, 10-15 and 20-25 μm) were used as starting materials. The high temperature planar impact system was used for consolidation. The diamond samples were compacted at impact velocities of 1.2 and 1.9 km/s. The samples are well consolidated and the microhardness values of samples are in the range of 20-30 GPa. Diamond particles are very brittle and are subject to high bending moments due to their highly irregular shape. The number of cracks

generated in the consolidated diamond powders tends to increase with an increase in the particle size of the starting diamond powders [3]. Generation of small amounts of graphite was detected by X-ray diffraction in the compacted 10-15 and 20-25 μm diamond powders. The transformation to graphite usually occurs in larger particle size specimens consolidated at room temperature. For large particle size (10-25 μm), the interstitial void volume is larger than that of small particle size (4-8 μm). Relative movement of constrained particle, upon densification, is greater for large particle size, which causes larger deformation per interstitial site. Therefore, larger interstitial void volumes will result in larger shock energy deposited at the powder surfaces and cause phase transformation. Furthermore, this transformation is also enhanced with the help of preheating. Scanning electron micrograph from the interstitial melting areas of the compacted 20-25 μm diamond powders is shown in Figure 5. Small round diamond particles (0.5-1 μm) were observed in this sample. These small diamond particles are believed to be produced during the resolidification of the melting layer. Thicker surface layer (2-4 μm) were generated due to larger particle size.

The recovered samples of compacted 4-8 μm diamond/C(15wt%) and 4-8 μm diamond/Si(7.5wt%) mixture were also examined. Deformation of the softer component graphite decreases the pressure concentration and fills the pores between diamond particles. X-ray diffraction showed that part of the graphite powders was transformed to diamond. This is consistent with theoretical prediction [9] and results obtained by Potter and Ahrens [10]. These investigations showed, in room temperature experiments, that graphite transformed into diamond at pressures of 15-22 GPa. The consolidated diamond/C sample shows an extremely high microhardness value: > 55 GPa. However, the recovered diamond/Si sample is not very well consolidated. We expected the heat generated from reaction between silicon and carbon to enhance the bonding, but the reaction did not occur. Furthermore, the addition of silicon will reduce the effective pressure transmitted to contact areas between diamond powders due to poor constraint. This resulted in poor consolidation.

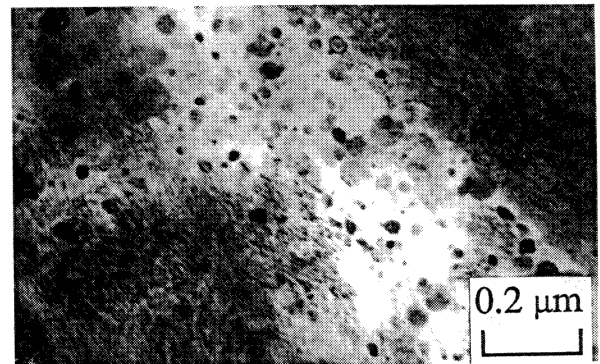


Fig. 4. Transmission electron micrograph of consolidated 44 μm SiC showing lattice distortion in the interior of SiC particle and the surface layer in the interparticle region.

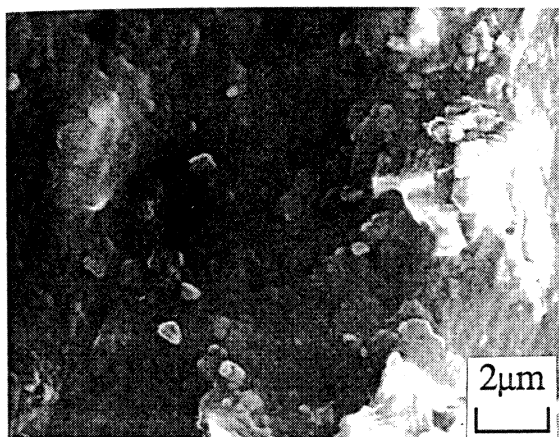


Fig. 5. Scanning electron micrograph of the melting layer after the resolidification from 20-25 μm diamond

C. Characterization of dynamically consolidated c-BN powders

The recovered c-BN (10-20 and 40-50 μm) samples are very well consolidated. Figure 6 is a fracture surface of 10-20 μm c-BN which shows the melting of particle surfaces. This transgranular fractograph suggests that the bonding of the particles is excellent. Small voids are observed, probably due to the gas generated by the decomposition of c-BN. The morphology of the c-BN is polyhedral. This particle geometry enhances the constraint between the particles during void collapse stage and results in larger energy deposition at the powder surfaces. Therefore, the microhardness value of the samples can reach 61 GPa. A highly angular particle shape would tend to decrease surface temperatures due to (1) poor constraint between particles and (2) generation of microcracks in the particle reducing the energy dissipation on the surface.

C-BN admixed with C (15wt%) and a mixture of Ti (11wt%) and C (9wt%) was also consolidated. The graphite powders are severely deformed and uniformly distributed on the surfaces of the c-BN powders after shock consolidation. However, the cooling rate was not sufficiently high for the transformation of the graphite into diamond. The remaining graphite causes the degradation of the consolidated c-BN sample. Recovered c-BN admixed with Ti and C composite showed low microhardness value. The exothermic reaction of Ti and C did not take place during the passage of the shock wave. The remaining Ti and C caused the degradation of the sample.

D. Dynamic densification and hot isostatic pressing of $\text{Ti}_3\text{Al}/\text{Ti}+\text{Al}$

A double tube setup with low explosive detonation velocity (2200 m/s) was chosen for $\text{Ti}_3\text{Al}+\text{Ti}+\text{Al}$ in order to minimize cracking. Successful densification was obtained using this explosive with low detonation velocity. The shock pressure was calculated to be 9.5 GPa. Scanning electron microscopic analysis of

recovered compacts after dynamic densification revealed that Ti and Al particles underwent a considerable amount of deformation and that the material was fully densified. The Ti_3Al particles seem to retain their initial sphericity. Well densified $\text{Ti}_3\text{Al}+\text{Ti}+\text{Al}$ compacts were obtained (~ 95-98 % of the crystal density) and machined for subsequent hipping. Hot isostatic pressing (hipping) was conducted at Degussa Electronics Inc. at 600 $^\circ\text{C}$ and 1000 $^\circ\text{C}$ for one hour. Ti and Al powders fully reacted with each other at both temperatures. Scanning electron microscopic analysis of the material recovered after hipping at 1000 $^\circ\text{C}$ (Figure 7) revealed that the reaction products bonded the Ti_3Al powders very well, as compared to the compacts produced using hipping at 600 $^\circ\text{C}$. Microvoids are not totally eliminated in the 600 $^\circ\text{C}$ -hipped samples. Compression tests also showed strong bonding between Ti_3Al powders after 1000 $^\circ\text{C}$ hipping. These 1000 $^\circ\text{C}$ -hipped specimens have an average ultimate compressive strength of 2 GPa and compressive fracture strain of 20%. In contrast, the average values of ultimate compressive strength and compressive fracture strain of dynamic-densified specimens are 1.4 GPa and 4.5%, respectively. Post-shock hipping can minimize the microcracks and tremendously increase the mechanical properties of the samples.

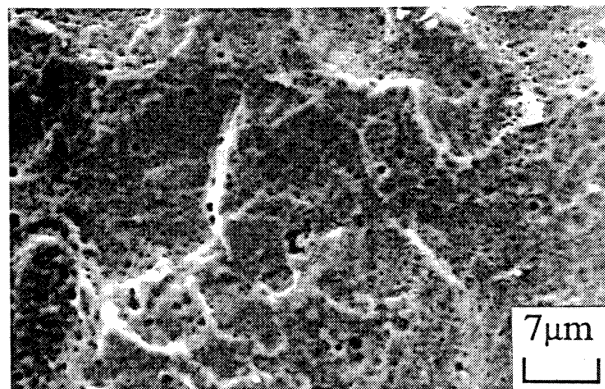


Fig. 6. Scanning electron micrograph of fracture surface from consolidated 10-20 μm c-BN.

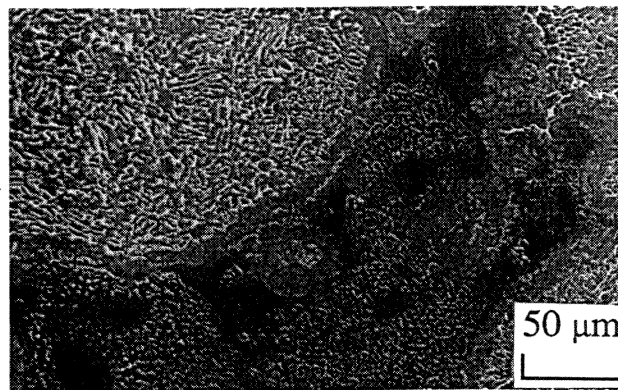


Fig. 7. Scanning electron micrograph from dynamically densified $\text{Ti}_3\text{Al}+\text{Ti}+\text{Al}$ alloy hipped at 1000 $^\circ\text{C}$.

Conclusion

In order to take advantage of the benefits of shock consolidation while minimizing its problems (cracking produced by reflected waves), three novel approaches have been investigated. Formation of a solid compact with good bonding requires generation of melting layers along the powder surfaces during shock consolidation. The size, shape and mechanical properties of adjacent powders are very important factors that can change the localized energy deposited at the powder surface. Interstitial void volumes between powders (relative movement of the particles upon densification) also play an important role in determining the final structure of the compacts. Preheating can reduce the yield strength of the powder and increase the shock energy deposited at the particle surfaces. Highly stressed and strained microstructures can occur in the sample during the shock consolidation. Post-shock HIPping can heal the residual stress and strain and enhance the bonding between particles.

Acknowledgments

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