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Reaction-assisted shock consolidation of RSR Ti–Al alloys

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A new method for the shock consolidation of hard metallic powders has been successfully tested. This method extends the process developed by Sawaoka and Akashi for the processing of ceramics (U.S. Patent 4,655,830) to metallic powders. Shock-activated reactions between elemental mixtures of niobium and aluminum powders were used to chemically induce bonding between difficult-to-consolidate intermetallic TiAl compound powder particles. The highly exothermic reactions activated by the passage of shock waves form an intermetallic binder phase which assists in the consolidation of the very hard TiAl alloy powders. Shock impact experiments were carried out utilizing a twelve-capsule shock recovery system in which a plane wave generating lens is used for accelerating a flyer plate to velocities of 1.7 and 2.3 km/s. With these impact velocities, sufficient shock pressures are generated in the powders, contained in capsules, to result in shock-induced reactions between the elemental powders of the mix. Fully dense compacts were successfully recovered and were subsequently characterized by optical, transmission, and scanning electron microscopy, x-ray diffraction, and microhardness testing. Transmission electron microscopy revealed both microcrystalline and amorphous regions in the reaction zone. In one instance, the amorphous material crystallized under the heating effect of the electron beam. Shock induced reaction between elemental powders and with the TiAl powders, producing ternary compounds, was also observed.

I. INTRODUCTION

The intermetallic compound TiAl, possessing the ordered (gamma-fct) structure, has attractive and unique properties for extended high temperature applications.[1,2] Intermetallic compound powders made by rapid solidification processing are brittle and hard, making it difficult to consolidate the powders by conventional techniques. In this research program, shock-activated reactions[3] were used to chemically induce bonding between intermetallic compound powders.

Shock consolidation and shock synthesis of materials have been used to a considerable degree in research activity on materials development. Shock-wave consolidation of powders was used for the first time in the 1950s to produce high density parts from materials used in aerospace and atomic energy applications.[4] DeCarli and Jamieson[5,6] were the pioneers who synthesized diamond from graphite by dynamic pressure in 1961. Chemical reactions to aid in shock consolidation of hard ceramics were first used by Sawaoka and Akashi.[7] After encountering great difficulty in shock consolidation of cubic boron nitride, they added elemental powders of titanium, carbon, and aluminum to form a ceramic binder phase and enhance bonding between the boron nitride powders. The mechanism of chemically-induced shock consolidation of powders is shown in Fig. 1. The inert intermetallic compound powders A_xB are blended with elemental powders C and D. After the exothermic reaction, C and D form compound C_yD. The generation of heat and formation of C_yD promotes the bonding of otherwise difficult-to-bond A_xB powders.

The purpose of this investigation is to use and extend the method recently introduced by Sawaoka and Akashi[7] to consolidate intermetallic compound powders. Hard and brittle TiAl powders were used as the inert phase, and elemental niobium and aluminum powders mixed in different proportions were used as reactants. It was demonstrated that shock induced reactions can be used to help in the shock consolidation of hard and difficult-to-bond alloys.

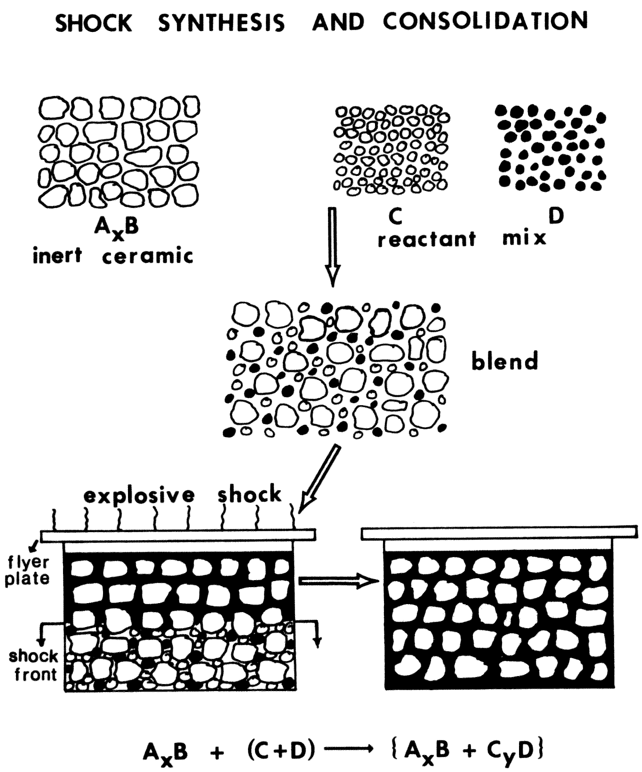


FIG. 1. Sequence of events in chemically-assisted shock consolidation of powders.

II. EXPERIMENTAL PROCEDURES

The cross section of the experimental setup is schematically shown in Fig. 2. The explosive system is used to drive the flyer plate at velocities ranging from 1000 to 2500 m/s. This system has been used by several investigators[8,9] and was introduced by Akashi and Sawaoka[10] for shock studies of powders.

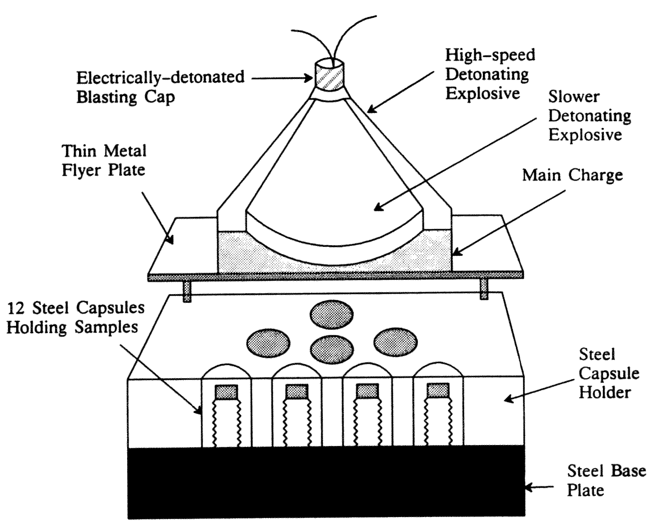


FIG. 2. Experimental setup to shock consolidate and shock synthesize powders.

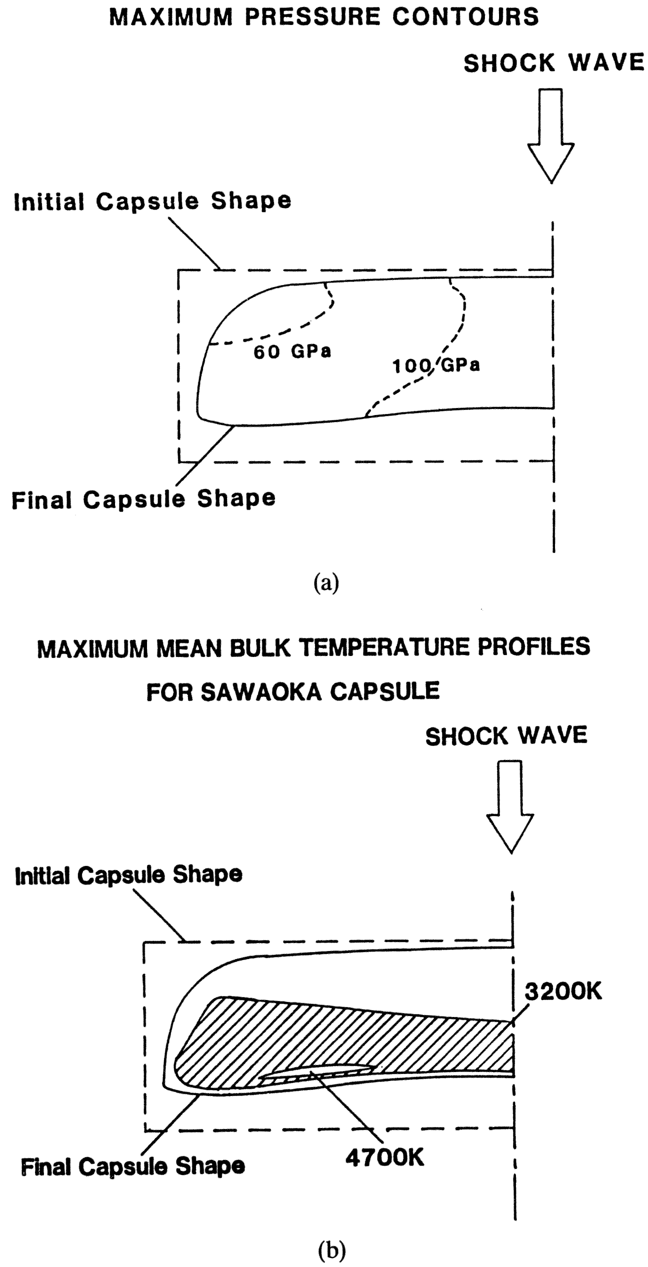


FIG. 3. Maximum mean bulk (a) pressure and (b) temperature profiles during passage of shock waves for Sawaoka capsule (adapted from Norwood *et al.*[11]).

The planar/parallel impact of the flyer plate on the system creates the high amplitude shock waves that transmit through the powder. The detonation is initiated from the detonator at the top of Fig. 2. The conical lens consists of explosives with two detonation velocities and generates a planar wave to the main charge. The flyer plate is accelerated by the main charge and impacts the capsules, inducing the desired shock waves into the powders. The momentum trap is used to trap the reflected tensile waves. Since the velocity of the shock wave in the powder is considerably lower than in the surrounding steel, the planar wave in the steel actually surrounds the powder capsule in a "pincer" action and penetrates along the lateral surface of the disk as well as on the top surface. Thus, the pressure and temperature distribution inside the capsule are nonuniform. Figure 3 shows the maximum pressure and mean bulk temperature contours in the powder for the Sawaoka capsules impacted at 2.5 km/s; these predictions were made by Norwood *et al.*[11] using a two-dimensional CSQ II computer hydrocode, for a hypothetical powder with 40% porosity. Although the experiments discussed herein used impact velocities, 1.7 and 2.3 km/s, the profiles provide good indicators of the variation of shock pressures and temperatures.

Figure 4 shows an optical micrograph of TiAl powder mixed with Nb and Al powders. The TiAl powders have a spherical shape (−80 mesh) and the elemental Nb and Al powders are irregular (−325 mesh). Two experiments were conducted at impact velocities of 1.7 km/s and 2.3 km/s. In these experiments two types of powder mixtures were used. In one case, only the elemental Nb and Al powders mixed in 50:50 and 65:35 (by wt) ratio were used and, in the other case, TiAl + Nb + Al powders were mixed in 70 + 15 + 15 ratio and 90 + 6.5 + 3.5 ratio (by weight). The powder mixtures were packed into capsules at approximately 65% of the theoretical density. Both the original powders and the recovered compacts were characterized by optical and scanning electron microscopy, x-ray diffraction, and microhardness testing.

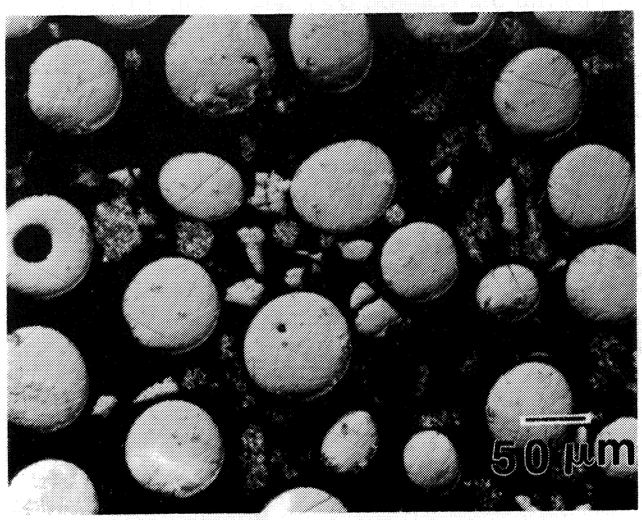


FIG. 4. Optical micrograph of powders after mixing.

III. RESULTS AND DISCUSSION

Most of the compacts prepared by this technique were successfully consolidated and/or synthesized. For all experiments, the extent of bonding or reaction was dependent on the position within the capsule. Both the shock pressure and mean bulk temperature are very dependent on the position of material within the capsule, as shown in Fig. 3. The bottom portion of the compact, opposite to the impact surface, experiences the highest mean bulk shock temperature. Figure 5 shows a typical cross section of a shocked compact containing elemental powder reactants. The darker region in the bottom is fully reacted, while the top portion is only compacted. The shape of the reacted-compacted interface follows the temperature contour of Norwood *et al.*[11] simulation, showing that the reaction requires a high mean bulk temperature. The discussion that follows will address the results of shock synthesis of one of the niobium-aluminum compounds and shock consolidation of intermetallic TiAl powders assisted by reaction synthesis.

A. Shock synthesis of niobium–aluminum compounds

Figure 6(a) shows the cross section of a part of the compact shock loaded at 2.3 km/s. This compact con-

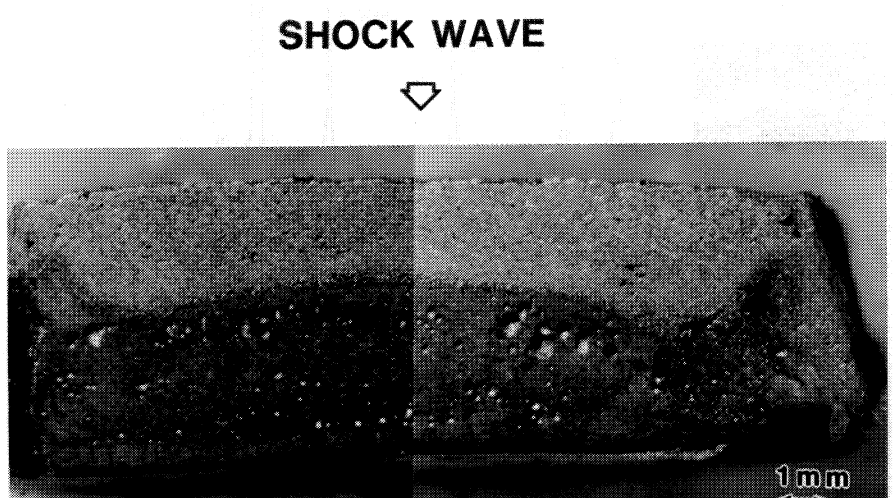
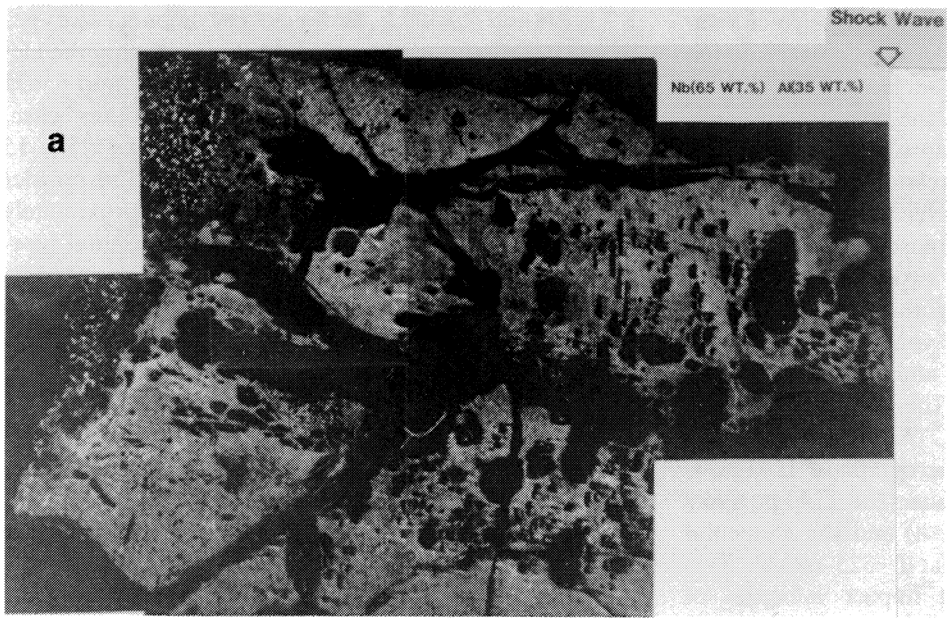


FIG. 5. Typical cross section of a shocked compact containing elemental powder reactants.



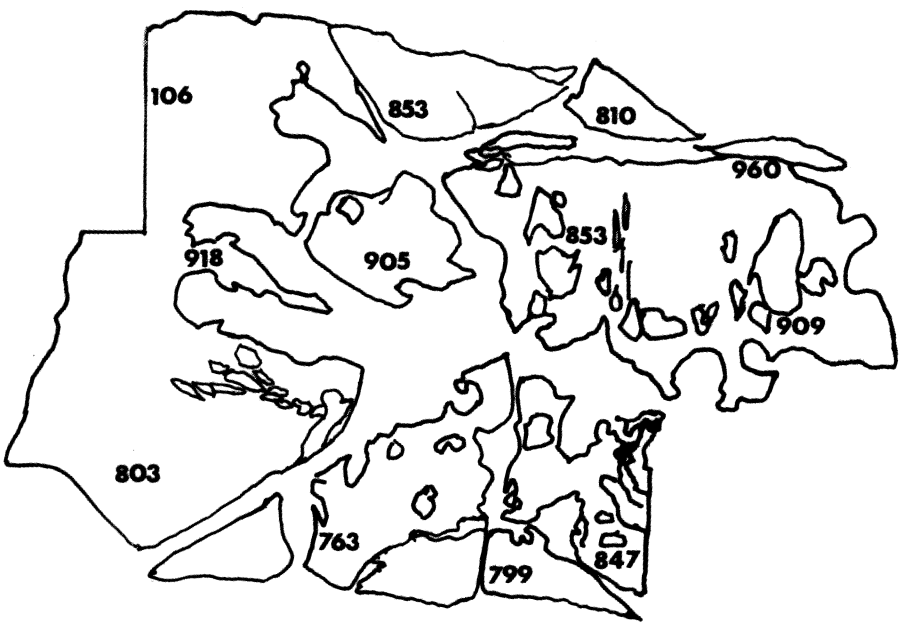
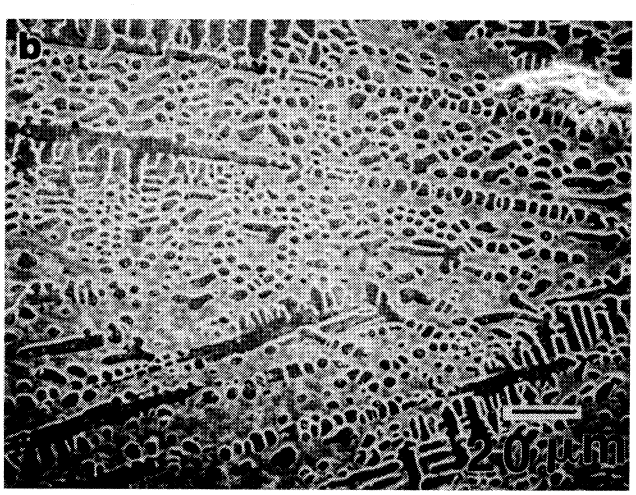


FIG. 6. (a) Micrograph of cross section of compact containing a mixture of 65 wt.% Nb and 35 wt.% Al powders after passage of shock wave (2.3 km/s impact velocity). Bottom figure shows microhardness measurements in various parts of the compact. (b) SEM image showing dendritic structure at higher magnification.



tained 65 wt.% Nb and 35 wt.% Al powder mixture. Most of the section had a microdendritic structure except the upper left-hand corner, where no reaction took place. The microhardness measurements (Vickers hardness number) are shown in Fig. 6(a). The reacted region had higher hardness numbers (800–900) than the unreacted region (around 100). Figure 6(b) shows the microdendritic structure at a high magnification. The x-ray diffractometer scans for this powder mixture and compact are shown in Fig. 7(a). Two intermetallic compounds, Al_3Nb and $AlNb_2$, could be identified. The powder mixture Nb–Al is shown in the upper trace, while the shock synthesized material is shown in the lower trace. From the Nb–Al phase diagram of Fig. 8, it is seen that the composition of the phases formed, $AlNb_2$ and Al_3Nb, brackets the initial composition of the powder mixture, 65 wt.% Nb and 35 wt.% Al.

B. Chemically-induced shock consolidation of TiAl

The hard and brittle TiAl powders are difficult to shock consolidate without additives. Thus, Nb and Al powders were added to TiAl powders with the intent of initiating a shock-induced reaction to generate heat and produce an intermetallic compound binder phase to assist in the consolidation of TiAl. Figure 9 shows half of the cross section of the sample containing 70% TiAl, 15% Nb, and 15% Al, shocked at 2.3 km/s impact velocity. It is clearly seen that the bottom portion of the capsule underwent better bonding. This corresponds in Fig. 3 to the region of higher mean bulk temperature. The microhardness readings in Fig. 9 show higher values in the highest mean bulk temperature regions.

Figure 10(a), at higher magnification, shows the optical micrograph of a region near the top of the cross section. It is clearly seen that Nb powders just surround TiAl powders; they did not react with Al powders. Figure 10(b) shows the quantitative analysis of Fig. 10(a) by SEM. There is no reaction between Nb and TiAl powders. At the central region of the cross section, Fig. 11(a), partial reaction took place with residual Nb particles remaining. The quantitative analysis by SEM is shown in Fig. 11(b). At the point marked H, there was reaction between TiAl, Al, and Nb. Figure 12(a) (bottom part of cross section) shows that Nb and Al powders reacted with each other and with TiAl powders. The reaction products bonded the TiAl powders together. Figure 12(b) shows that all points in this re-

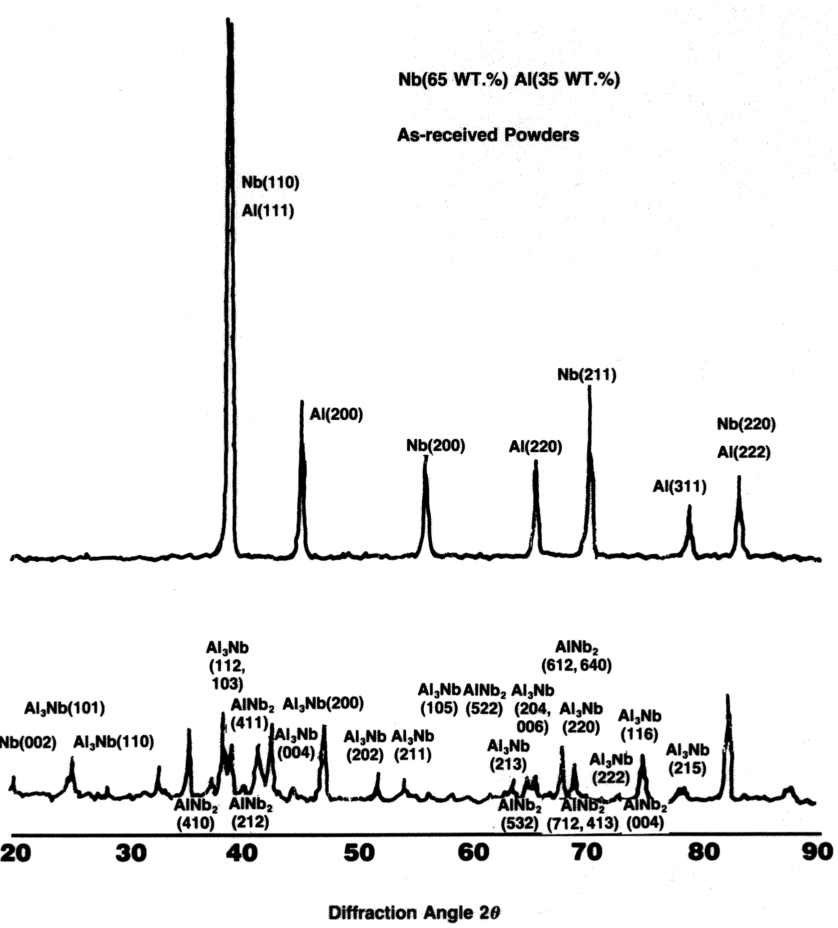


FIG. 7. X-ray diffractometer scans for 65 wt.% Nb and 35 wt.% Al powder mixture; top: prior to shock; bottom: after shock processing.

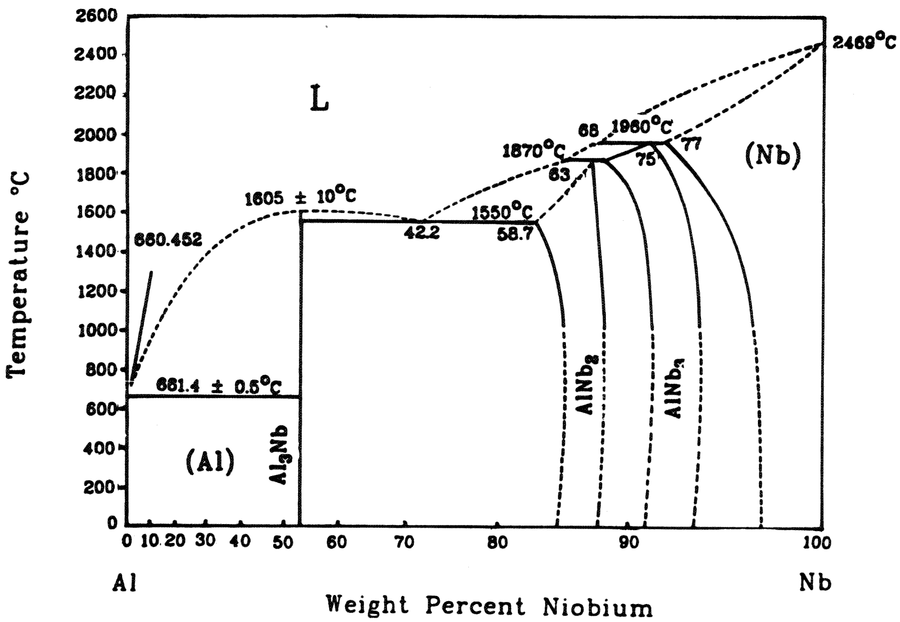


FIG. 8. Nb–Al phase diagram (from Elliot and Shunk[12]).

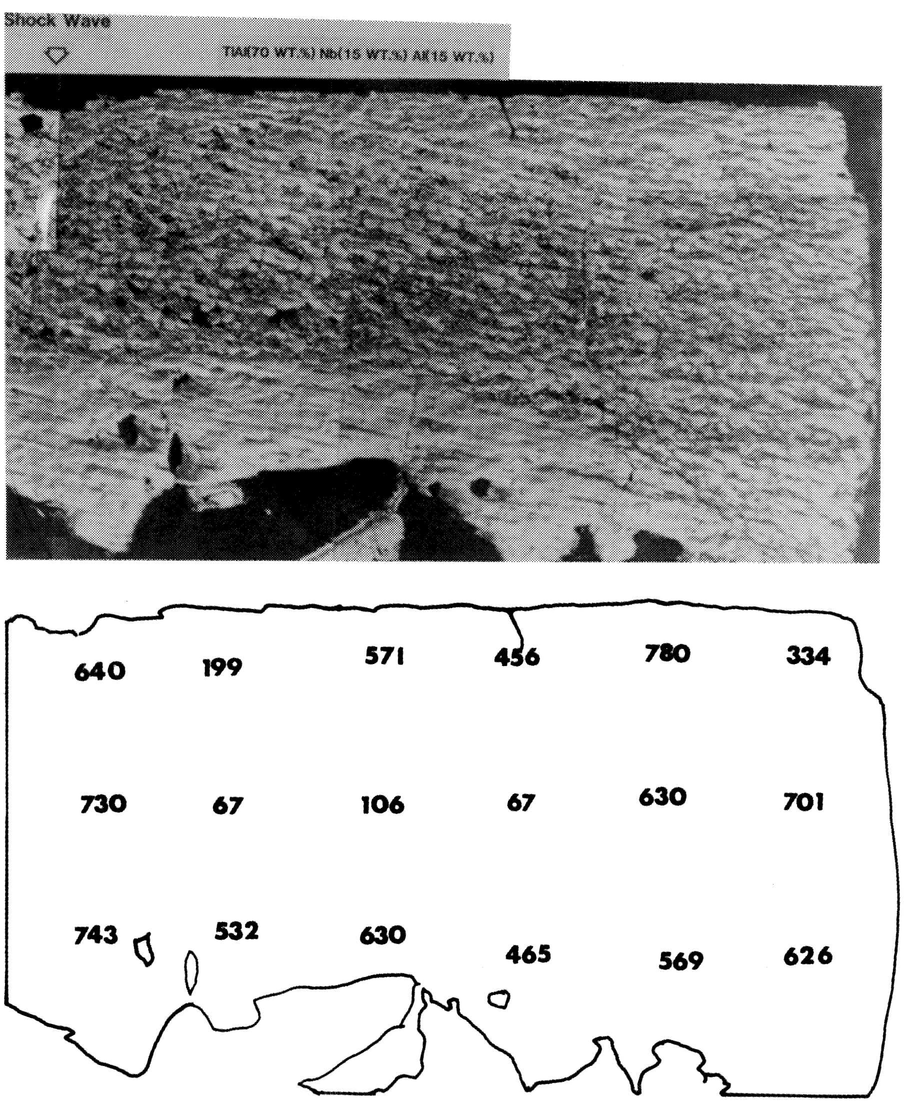
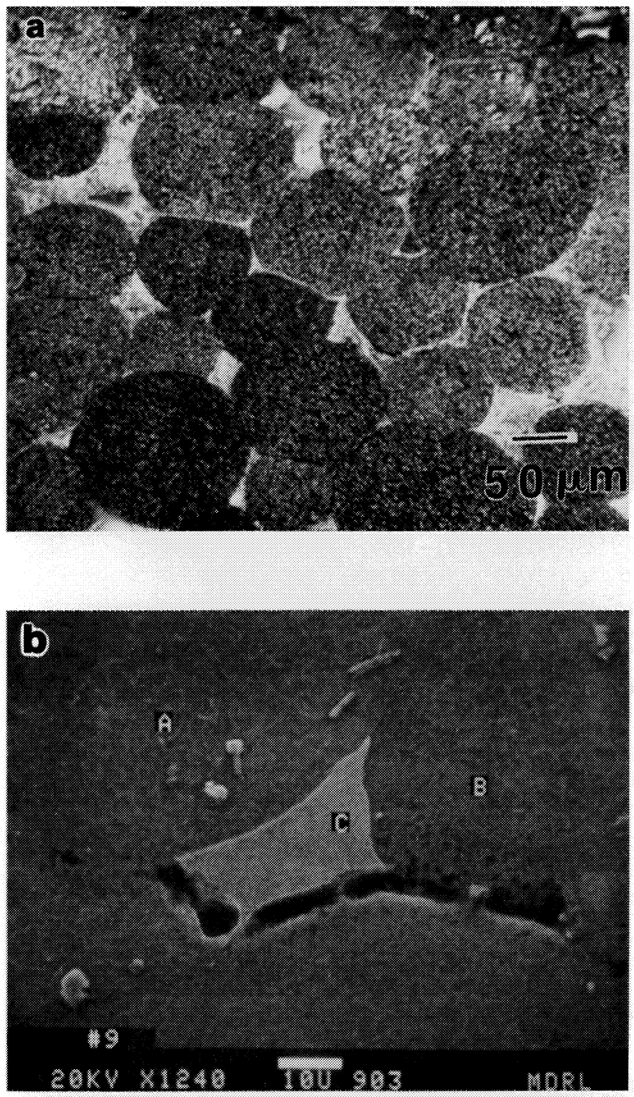
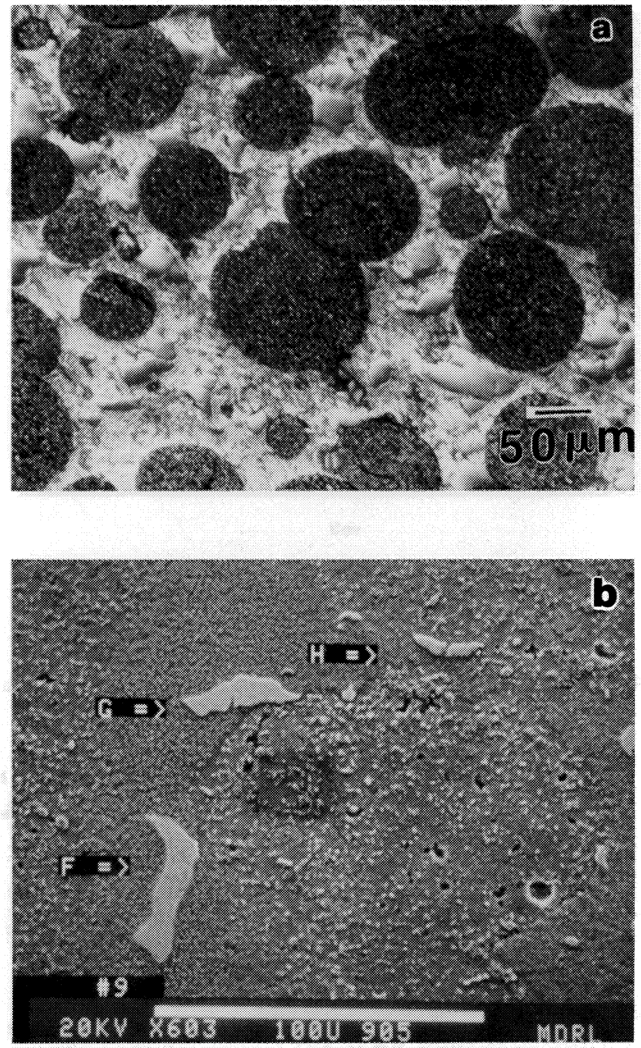


FIG. 9. Micrograph of cross section of compact containing a mixture of 70 wt.% TiAl, 15 wt.% Nb, and 15 wt.% Al powders after passage of shock wave (2.3 km/s impact velocity). Bottom figure shows microhardness measurements in various parts of the compact.



A: Al–43.2%; Ti–55.9%; Nb–0.9%
B: Al–33.9%; Ti–65.6%; Nb–0.2%
C: Nb–99.7%

FIG. 10. (a) Optical micrograph of the top region in Fig. 9. (b) Quantitative analysis of (a).



F: Al–1%; Ti–0.5%; Nb–98.5%
G: Nb–100%
H: Al–53.9%; Ti–39%; Nb–7%

FIG. 11. (a) Optical micrograph of center region in Fig. 9. (b) Quantitative analysis of (a).

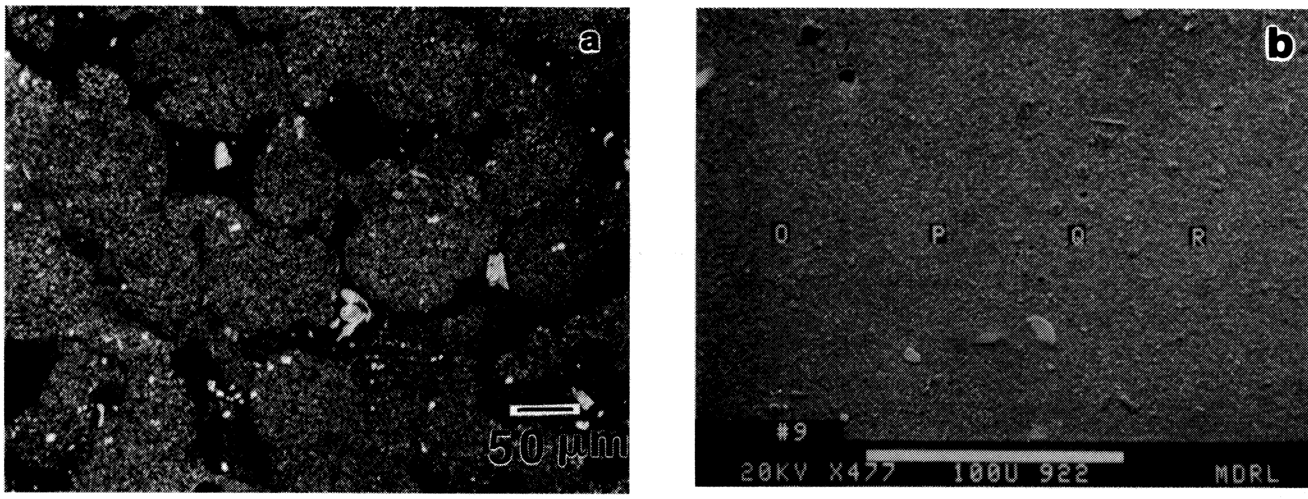
gion were reaction products which had almost the same composition.

It is interesting to notice that reaction does not take place as postulated in Fig. 1, but in a more complex mode, with involvement of the inert intermetallic compound and binders. Figure 13 shows the x-ray diffractometer traces of the original powder mixtures and of the shock consolidated compacts at two different velocities. While the TiAl and Ti_3Al peaks are retained (TiAl powder produced by rapid solidification processing actually contains some Ti_3Al), new peaks appeared after consolidation. These peaks (at two theta values of 22 and 29 degrees) could not be identified and are much more intense after 2.3 km/s impact. They are thought to be due to a Ti–Al–Nb ternary compound. These results clearly show that chemically-induced bonding is a concept that can be applied to assist in consolidation of metallic systems.

C. Transmission electron microscopy

Transmission electron microscopy was performed on TiAl powders bonded with niobium and aluminum. Three distinct areas could be observed: (i) amorphous and (ii) microcrystalline areas at interparticle regions, and (iii) monocrystalline material characteristic of the powder particle interiors.

(i) Amorphous areas within the interparticle regions are shown in Fig. 14. Two different areas are shown [Figs. 14(a) and 14(b)] in order to illustrate the



O: Al–54.3%; Ti–38%; Nb–7.6%
P: Al–53.5%; Ti–38.8%; Nb–7.7%
R: Al–53.6%; Ti–39.2%; Nb–7.2%
Q: Al–535; Ti–39.8%; Nb–7.2%

FIG. 12. (a) Optical micrograph of bottom region in Fig. 9. (b) Quantitative analysis of (a).

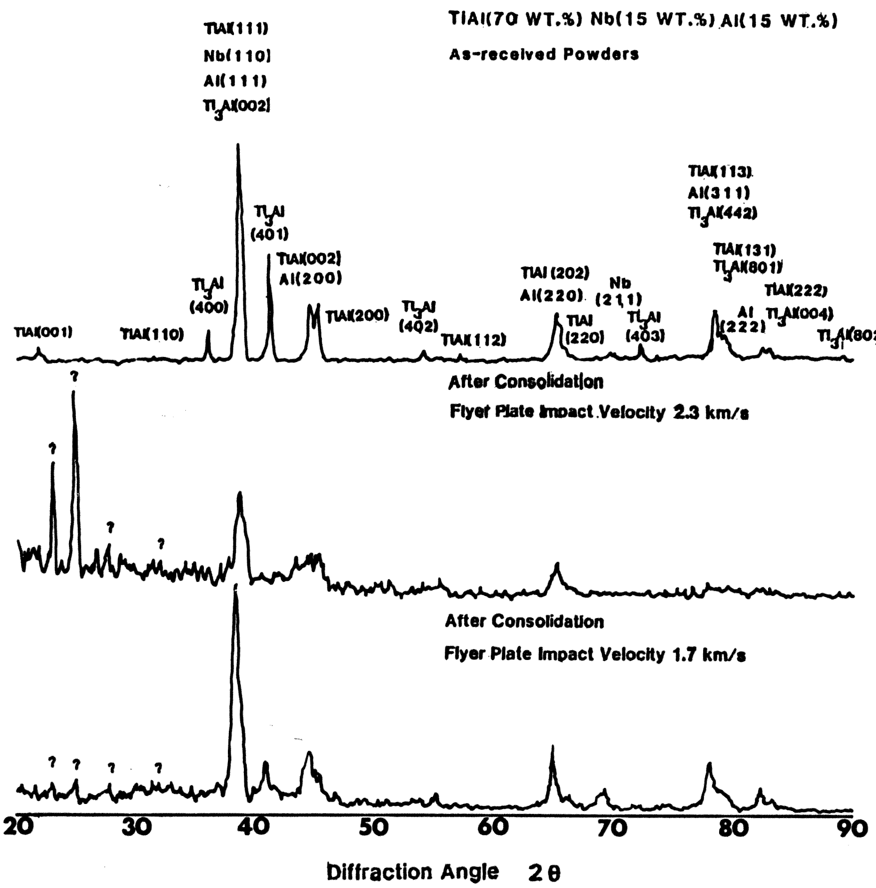
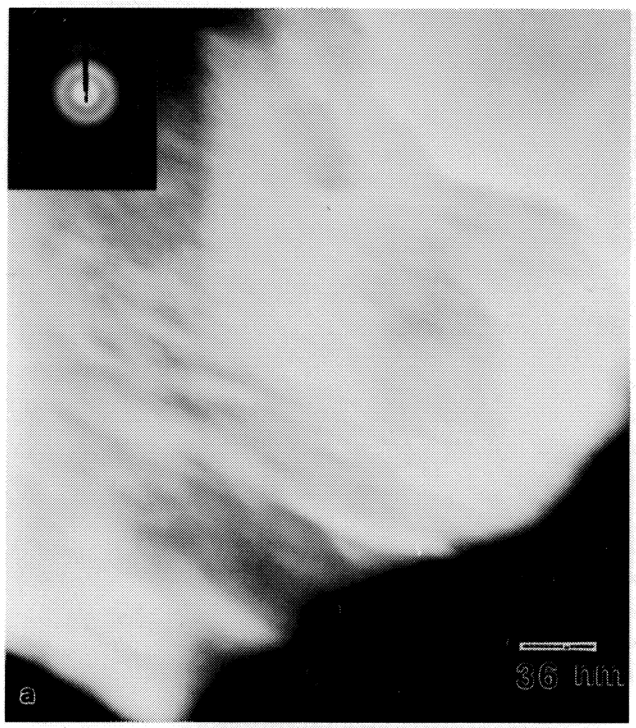


FIG. 13. X-ray diffractometer scans of TiAl + Nb + Al (a) in as-mixed condition; (b) after impact of 1.7 km/s; (c) after impact of 2.3 km/s.

fact that it is not an epiphenomenon. This material did not exhibit any clear features or boundaries. The selected area electron diffraction pattern shows diffuse rings that are characteristic of amorphous materials. The presence of amorphous phases in a shock consolidated glass-forming microcrystalline $Ni_{55.8}Mo_{25.7}Cr_{9.7}B_{8.8}$



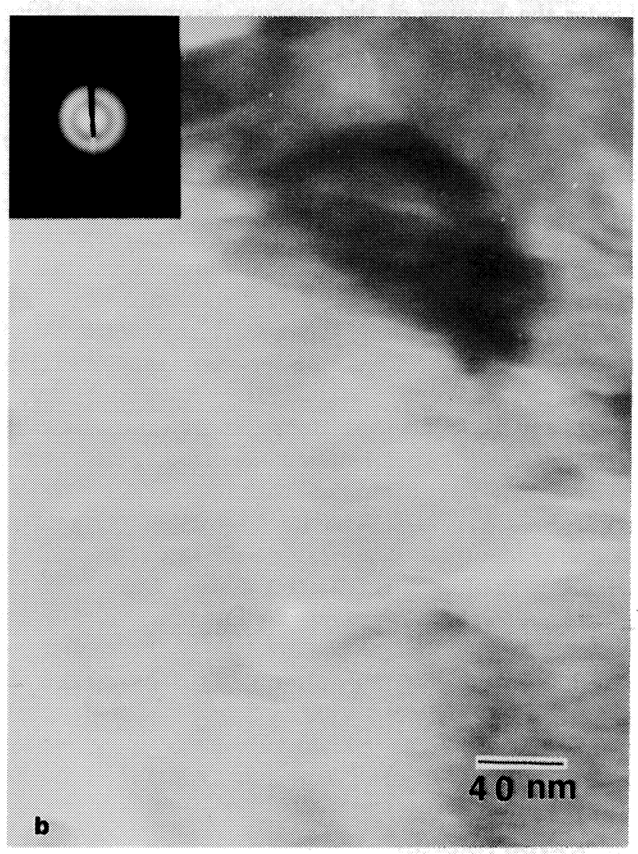
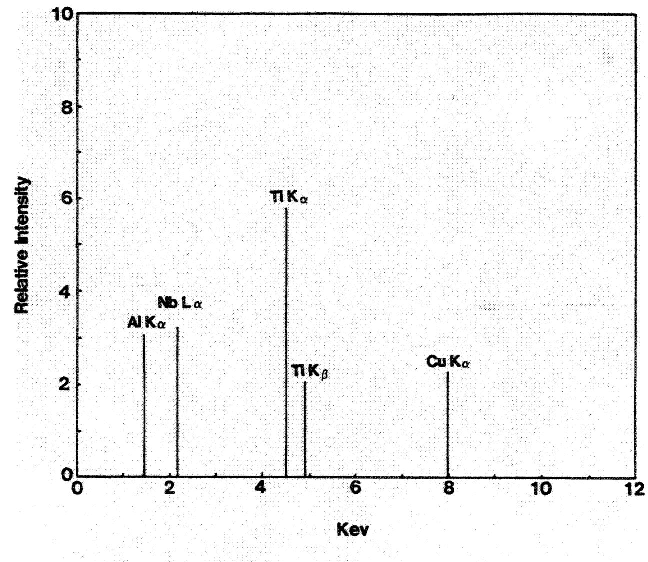
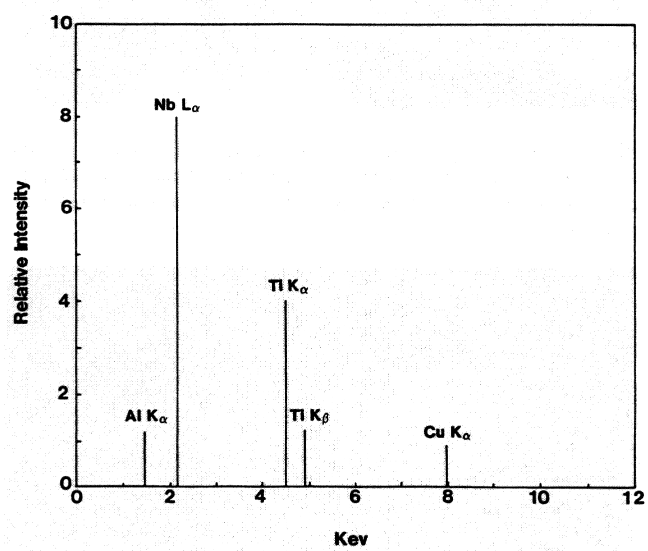


FIG. 14. (a) and (b) Transmission electron micrographs of two amorphous areas within the reaction zone in TiAl powder shock consolidated with Nb and Al.



(a)



(b)

FIG. 15. Schematic of the energy-dispersive x-ray analysis from amorphous (a) interparticle region and (b) reaction product region.

alloy has also been found by Vreeland *et al.*[13,14] The energy dispersive x rays used for identification of the atomic species indicated the three elements Nb, Al, and Ti, as shown in the schematic in Figs. 15(a) and 15(b). The x-ray scan shown in Fig. 15(a) corresponds to the composition of the TiAl particle. It represents the amorphous material formed due to melting and rapid resolidification of the near-surface regions of the TiAl particles. On the other hand, the x-ray scan in Fig. 15(b) shows a Nb-rich amorphous region but also contains Ti and Al. This is believed to correspond to the interparticle reaction region. The Cu peaks in both traces are due to the sputtering of the Cu from the slotted grid used for setting the 2.3 mm diameter TEM disk during ion milling.

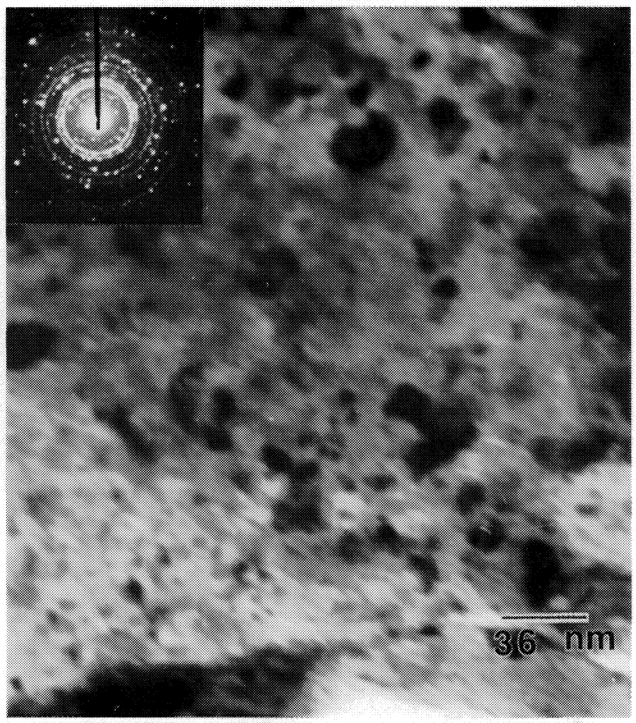


FIG. 16. Transmission electron micrograph of microcrystalline area (formed upon crystallization of amorphous material under the electron beam) within the reaction zone in TiAl powder shock consolidated with Nb and Al.

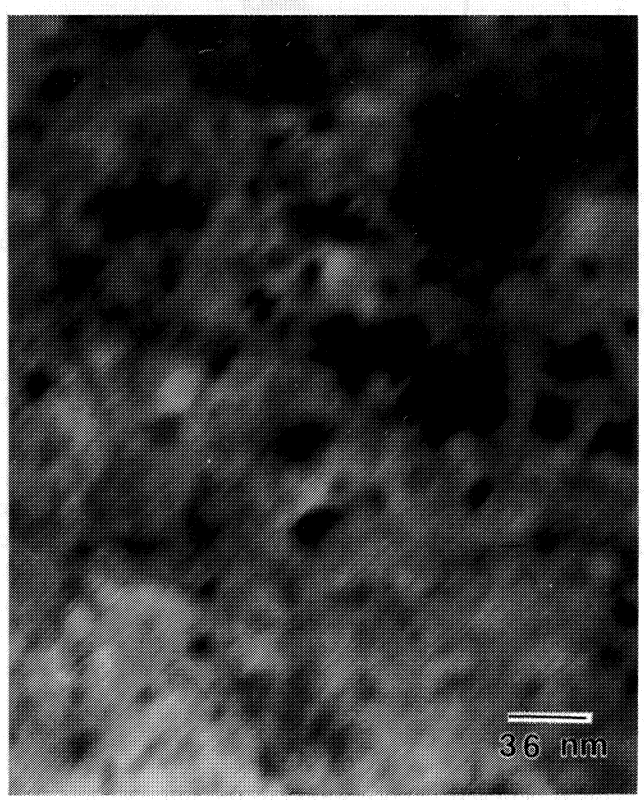


FIG. 17. Transmission electron micrograph of a microcrystalline area within the reaction zone in TiAl powder shock consolidated with Nb and Al.

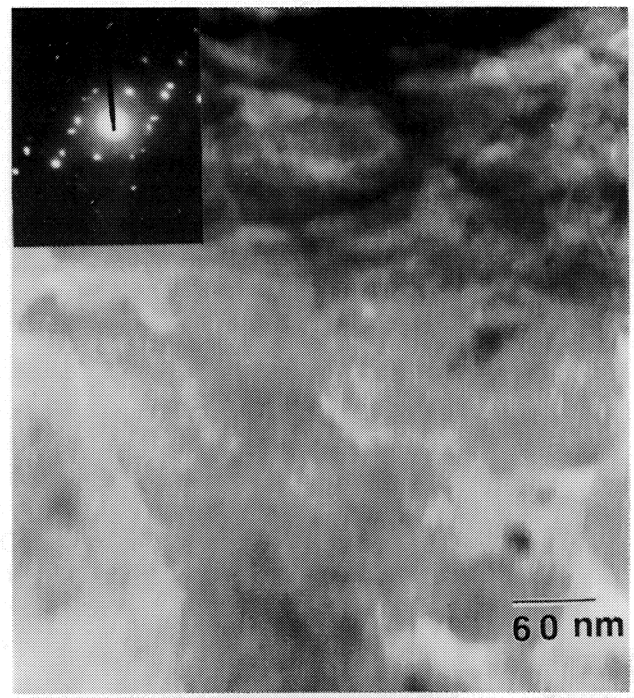


FIG. 18. Transmission electron micrograph of TiAl particle in TiAl powder shock consolidated with Nb and Al.

Under the heating of the electron beam one of these amorphous regions transformed into a microcrystalline structure while being observed. A bright-field TEM image of the transformed region is shown in Fig. 16.

(ii) Microcrystalline areas within the reaction zone at interparticle regions are shown in Fig. 17. These regions had the well-known microcrystalline structure found in many shock consolidated materials.[15,16] Qualitative x-ray analysis revealed that the microcrystalline material was a Nb-rich ternary compound.

(iii) Monocrystalline regions are representative of the interior of the TiAl powders; one such region is shown in Fig. 18.

It can be concluded from the transmission electron microscopy that reaction occurred, generating a Ti–Nb–Al ternary compound with microcrystalline and amorphous structure. The final structure (microcrystalline or amorphous) depends on the local composition as well as cooling rate. The fact that an amorphous region transformed into a crystalline one under beam heating shows that the cooling rate of the molten/reacted regions was sufficient to retain the amorphous region. Calculations conducted by Schwarz *et al.*[18] predict solidification of the melted region to occur within 200 ns, which is an order of magnitude less than the duration of the shocked state.

IV. CONCLUSIONS

It has been shown that chemically-induced shock consolidation is a process that can be applied to bond hard intermetallic compound powders. This concept has been applied to bond the intermetallic compound TiAl with the addition of niobium and aluminum elemental powders. Reaction occurs between the exothermically reacting Nb and Al powders and, also, between the inert TiAl and reactive (Nb + Al) powders. The reaction zone shows either a microcrystalline or an amorphous structure.

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