JOURNAL DE PHYSIQUE
Colloque C3, Supplément au n°9, Tome 49, septembre 1988 C3-659

DYNAMIC POWDER CONSOLIDATION AND SYNTHESIS OF INTERMETALLIC COMPOUNDS

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Resume - Des reactions induites par une onde de choc ont étés employées pour aider à joindre des poudres métalliques inertes. Des mélanges de poudres de Ni et Al, Ti et Al, et Nb et Al ont étés ajoutés à des poudres de composition intermetallique (TiAl). Le passage de l'onde de choc a activé des reactions chimiques hautement exothermiques et a produit le soudage entre les poudres inertes. Les experiences d'impact ont étés realizés avec l'emploi d'un generateur d'ondes planes conique et d'une charge explosive de composition PBX9404. Le système contenant les capsules avec des poudres était protegé par une plaque metallique de protection, dans laquelle l'ecaillage se produit. L'onde de choc a été produite par l'impact d'une plaque d'acier doux accelerée à des vitesses de 1,7 et 2,3 km/s. Les vitesses d'impact ont étés suffisantes pour produire des pressions de choc qui ont complètement compacté les poudres. Les materiaux produits par cette méthode ont étés analisés par diffraction par rayons x, microscopie optique et de balayage, et par microdureté.

Abstract - Shock-activated reactions were used to chemically induce bonding between inert metallic powder particles. Elemental mixtures of Ni-Al, Ti-Al, and Nb-Al powders were added to intermetallic compound powders (TiAl). The highly exothermic reactions were activated by the passage of the shock waves and enhanced the bonding between the inert intermetallic powders. Shock impact experiments were carried out using a plane wave generating lens with PBX9404 as the high explosive, and a momentum trap recovery system. A mild steel flyer plate was accelerated to give impact velocities of 1.7 and 2.3 km/s. With these impact velocities, sufficient shock pressures were generated in the powders to result in fully dense compacts which were subsequently characterized by optical and scanning electron microscopy, X-ray diffraction and microhardness testing.

1 - INTRODUCTION

The intermetallic compound TiAl, possessing the ordered (alpha-2(HCP)) 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. It is 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.

The shock waves were produced by impact of an explosively accelerated flyer plate on an anvil containing capsules in which the powders were encased. Impact velocities of 1.7 and 2.3 km/s were used. The highly exothermic reactions of elemental powders were activated during the passage of the shock wave through the powders. The heat produced is dissipated on the surfaces of inert powders and the reacted elemental powders act as a cement to enhance bonding between the 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 50's to produce high density parts from materials used in aerospace and atomic energy applications /4/. DeCarli and Jamieson /5,6/ were the pioneers to synthesize diamond from graphite by dynamic pressure in 1961. Chemical reactions to help shock consolidation were first used by Sawaoka and Akashi /7/. After encountering great difficulty in shock consolidating cubic boron nitride, they added elemental powders of titanium, carbon and aluminum to enhance bonding between the boron nitride powders. The mechanism of chemically-induced shock consolidation of powders is shown in Figure 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. C_yD promotes the bonding of A_xB powders.

The purpose of this investigation is to use extend method of Sawaoka and Akashi /7/ to consolidate intermetallic compound powders. Hard and brittle TiAl powders were used as the inert phase and Nb-Al, Ti-Al, and Ni-Al powder mixtures were used as reactants.

2 - EXPERIMENTAL PROCEDURES

The cross-section of the experimental set-up is schematically shown in Fig. 2. The explosive system is used to drive the flyer plate at velocities ranging from 1,000 to 2,500 m/s. This system has been used by several investigators and is described by /8,9/. The planar/parallel impact of the flyer plate on the system creates the high amplitude shock waves that transmit through the powder. The detonation was 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

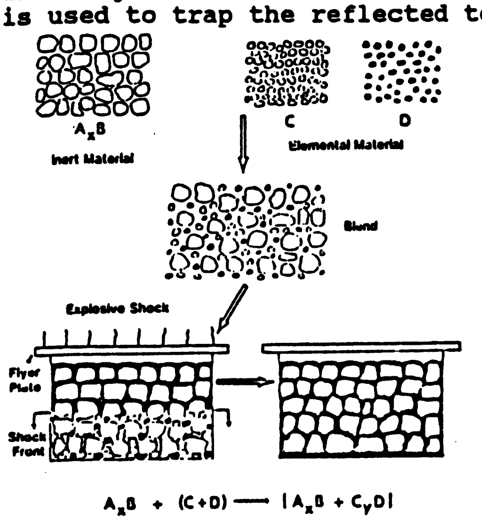


Fig. 1 - Sequence of events in chemically-induced shock consolidation of powders.

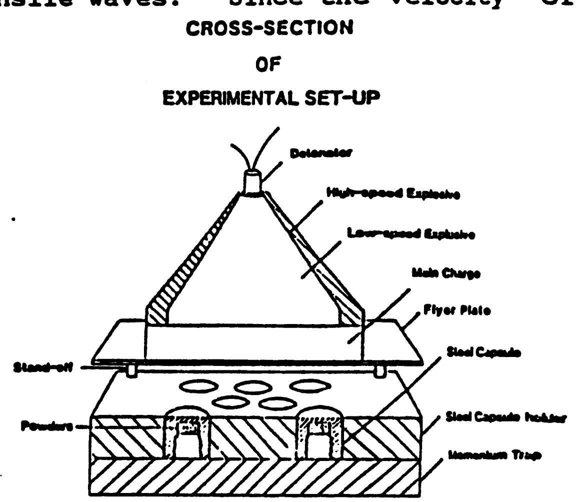


Fig. 2 - Experimental set-up to shock consolidate and shock synthesize powders.

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 fairly nonuniform.

The sizes, shapes, and purites of powders for the experiments are listed in Table 1. Fig. 3 shows an optical micrograph of the powder after mixing. The TiAl powders have a spherical shape and the elemental Nb and Al powders have irregular shapes.

Two experiments were done at impact velocities of 1.7 km/s and 2.3 km/s. Table 2 shows the compositions and packing densities of powders in these two experiments. Both the original powders and compacts were characterized by optical and scanning electron microscopy, X-ray diffracton, and microhardness testing.

3 - RESULTS AND DISCUSSION

Most of the compacts 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. Norwood et al. /10/ simulated the shock pressure and temperature distribution in the Sawaoka capsules with a two-dimensional CSQ code. Fig. 4 shows the mean bulk temperature distribution within the cross section of the capsule for a 2.5 km/s impact velocity. The bottom portion of the capsule, opposite to the impact surface, experiences the highest mean bulk shock temperature and pressure.

Fig. 5 shows the cross-section of Sample 5, Shot #2 (see Table 2). The capsule contains 65% Ti and 35% Al powder. 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.'s /10/ simulation, showing that the reaction requires a high mean bulk temperature. The higher magnification micrographs of top, center, and bottom portions are shown in Fig. 6. The TiAl structure changes considerably from the unreacted region in the top portion to the fully reacted region (microdendritic structure) in the bottom. The discussion that follows will address exclusively the system TiAl + Nb + Al and Nb + Al, although similar results were obtained with the Ti + Al and Ni + Al powder mixtures added to TiAl.

3.1 - Shock Synthesis of Niobium-Aluminum Compounds. Fig. 7(a) shows the cross-section of Sample 6, Shot #2. The composition of powders was 65 wt% Nb and 35 wt% Al. Most of the section had a microdendritic structure except the upper left-hand corner, where no reaction took place. The microhardness measurements (Vickers

TABLE 1. Powder Sizes, Shapes and Purities

Powder	Size (mesh)	Source and Shape	Purity
Ni	-325	CERAC irregular	99.9%
Nb	-325	CERAC irregular	99.8%
Ti	-400	CERAC irregular	99.5%
Al	-325	ASAR irregular	99.8%
TiAl	-80 (μ m)	spherical	

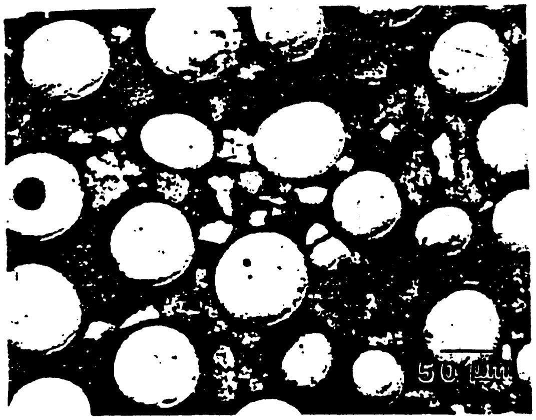


Fig. 3 - Optical micrograph of powders after mixing.

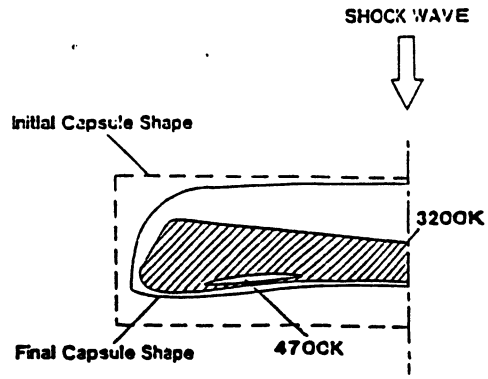


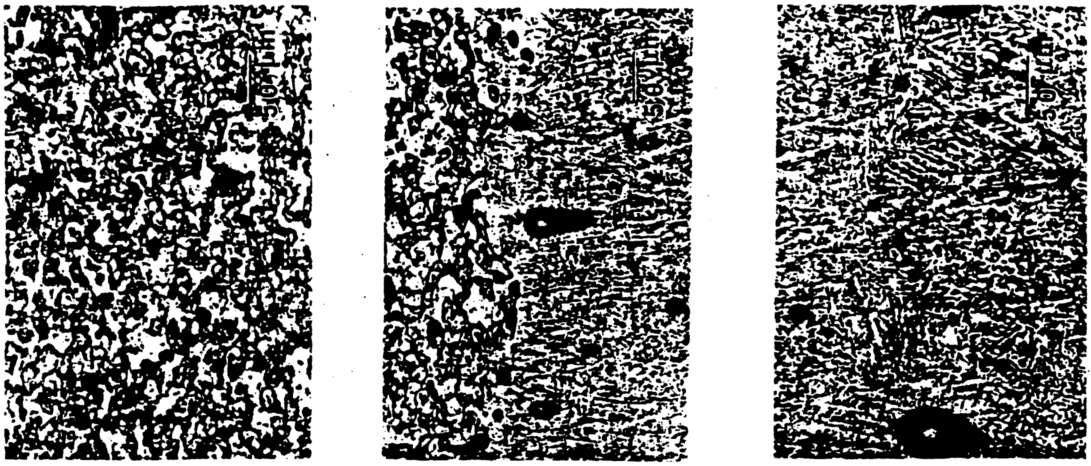
Fig. 4 - Maximum mean bulk temperature profiles during passage of shock waves for Sawaoka capsule (adapted from Norwood et al. /10/)

TABLE 2. Powder Mixtures and Loading Densities Used in Two Explosive Experiments.

Sample	Composition (wt%)	Loading Density (%) Shot #1 2.3 km/s	Shot #2 1.7 km/s
1	Ni(50)Al(50)	54.2	63.9
2	Ti(50)Al(50)	62.4	63.9
3	Nb(50)Al(50)	65.1	63.8
4	Ni(65)Al(35)	53.2	64.9
5	Ti(65)Al(35)	62.6	63.7
6	Nb(65)Al(35)	62.6	65.1
7	TiAl(70)Ni(15)Al(15)	65	64.8
8	TiAl(70)Ti(15)Al(15)	68	65
9	TiAl(70)Nb(15)Al(15)	67.5	64.8
10	TiAl(90)Ni(6.5)Al(3.5)	65	65.3
11	TiAl(90)Ti(6.5)Al(3.5)	72.4	64.4
12	TiAl(90)Nb(6.5)Al(3.5)	65	64.5

Fig. 5 - The cross-section of Sample 5, Shot #2.

hardness number) are shown in Fig 7(a). The reacted region had higher hardness number (800-900) than the unreacted region (around 100). Fig. 7(b) shows the microdentric structure at a high magnification. The x-ray diffractometer scans are shown in Figure 8(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 Figure 9, it is seen that the composition of the



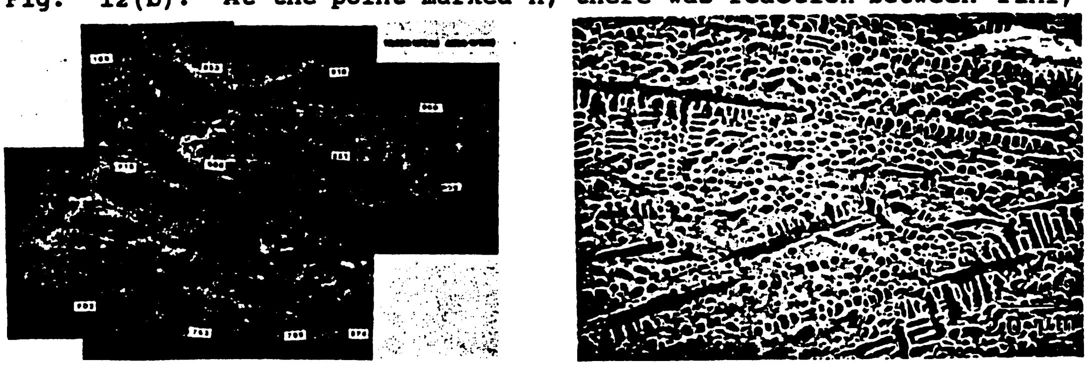
top center bottom

Fig. 6 - Optical micrographs in top, center, bottom portions of Fig 5.

phases formed, $AlNb_2$ and Al_3Nb, brackets the initial composition of the powder mixture, 65 wt%Nb and 35 wt%Al.

3.2 - <u>Chemically-induced shock consolidation of TiAl</u>. Nb-Al, Ti-Al, and Ni-Al powder mixtures were used to bond the TiAl powders. The hard and brittle TiAl powders are difficult to shock consolidate without additives. Fig. 10 shows half of the cross-section of sample 9, Shot #2 (70% TiAl, 15% Ni, 15% Al). It is clearly seen that the bottom portion of the capsule underwent better bonding. This corresponds in Fig. 4 to the region of higher mean bulk temperature. The microhardness readings in Fig. 10 show higher values in the highest mean bulk temperature regions in Fig. 4.

Fig. 11 (a), at higher magnification, shows the optical micrograph on the top of the cross-section. It is clearly seen that Nb powders just surround TiAl powders; they did not react with Al powders. Fig. 11(b) shows the quantitative analysis of Fig. 11(a) by SEM. There is no reaction between Nb and TiAl powders. At the center region of the cross-section, Fig 12(a), partial reaction took pace with residual Nb particles remaining. The quantitative analysis by SEM is shown in Fig. 12(b). At the point marked H, there was reaction between TiAl,



(a) (b)

Fig. 7 - (a) Cross-section of capsule containing a mixture of 65 wt% Nb and 35 wt% powders after passage of shock wave (Sample 6, Shot #1 and #2). (b) dendritic structure at higher magnification by SEM.

Al, and Nb. Fig. 13(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. Fig. 13(b) shows that all points in this region were reaction products which had almost the same composition.

It is interesting to notice that reaction does not take place as postulated in Figure 1, but in a more complex mode, with involvement of the inert intermetallic compound and binders. Figure 14 shows the X-ray diffractometer traces of the original powder mixtures and of samples 9 in Shot #1 and Shot #2. 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 2θ angles 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 compound. These results clearly show that chemically-induced bonding is a concept that can be applied to metallic systems.

4 - 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 Ti-Al, Nb-Al, and Ni-Al mixtures.

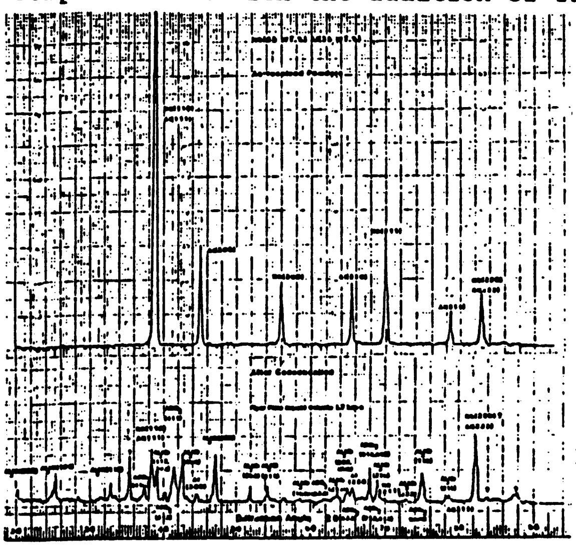


Fig. 8 - X-ray diffractometer scans from capsule in Figure 4; top - prior to shock; bottom - after shock processing.

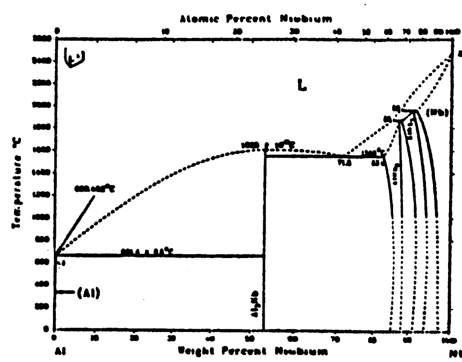


Fig. 9 - Nb-Al phase diagram (from Elliott and Shunk /11/)

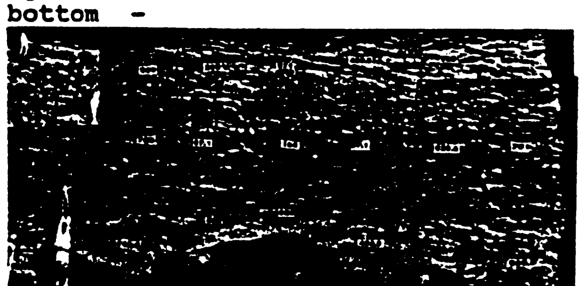
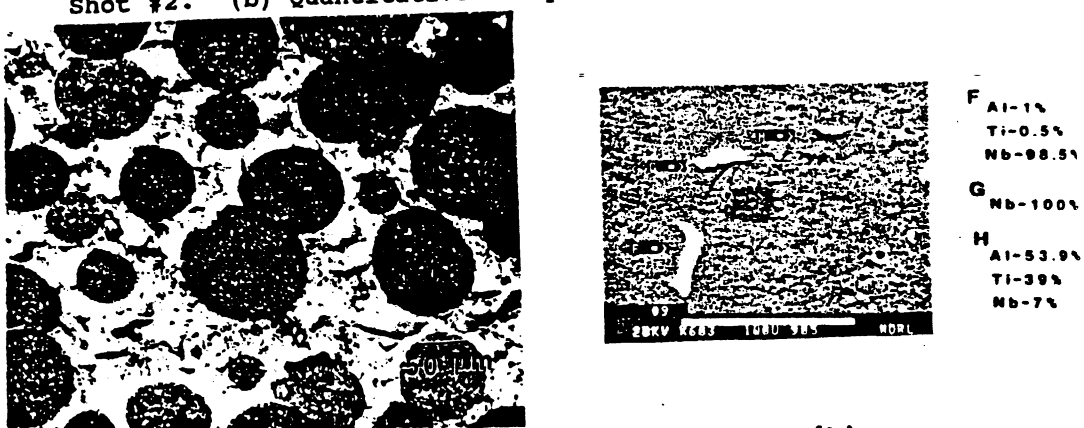


Fig 10 - Cross-section of capsule containing a mixture of 70 wt% TiAl, 15 wt% Nb, and 15 wt% Al powders after passage of shock wave (Sample 9, Shot #2).

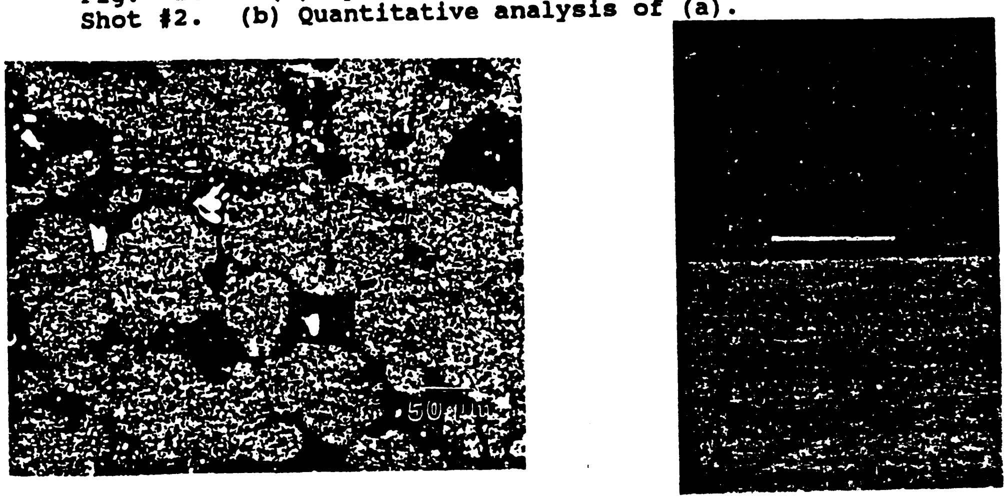
(a) (b)

Fig. 11 - (a) Optical micrograph of the top region of Sample 9, Shot #2. (b) Quantitative analysis of (a).



(a) (b)

Fig. 12 - (a) Optical micrograph of center region of Sample 9, Shot #2. (b) Quantitative analysis of (a).



(a) (b)

Fig. 13 - (a) Optical micrograph of bottom region of Sample 9, Shot #2. (b) Quantitative analysis of (a).

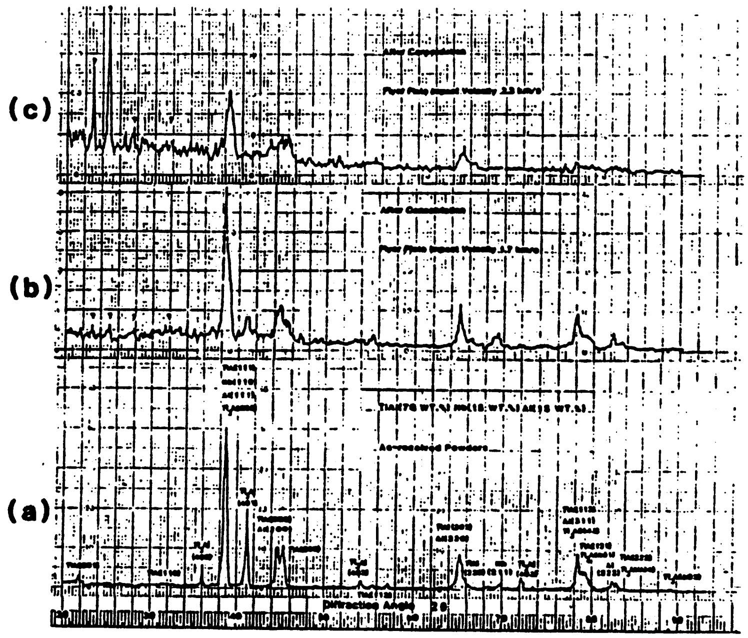


Fig. 14 - X-ray diffractometer scans of TiAl + Nb + Al (a) in as mixed condition; (b) after impact of 1.7 km/s (Sample 9, Shot #2); (c) after impact of 2.3 km/s (Sample 9, Shot #1).

5 - ACKNOWLEDGEMENTS

This research was sponsored by McDonnell Douglas Research Laboratories. The authors would like to thank Dr. P. J. Meschter for valuable discussions and experimental assistance. The help of Mr. T. Gould in setting up the experiments is greatly appreciated.

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