Synthesis of Novel Aluminide-Based Materials

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Because of their unique properties, new materials must often be synthesized using unusual and innovative methods. For example, explosively generated shock waves have been used to assist in the consolidation of aluminide powders, resulting in full densification. Reaction synthesis, making use of a solid / liquid reaction to form an alloy, has also shown promise as a means of fabricating aluminides. Using reaction synthesis to form ternary intermetallic compounds may result in more favorable crystal structures and better ductility, but more investigation is necessary.

INTRODUCTION

Aluminide intermetallics have the desirable property combination of low density, high strength and general oxidation resistance. However, these materials are limited in their application since they typically exhibit poor ductility and fracture toughness. To improve the mechanical properties of aluminide intermetallics, it is necessary to determine how alloy chemistry and processing conditions can influence crystal structure and deformation behavior. In addition, innovative processing methods are required to produce these new materials. In experiments at the McDonnell Douglas Research Laboratories and the Center for Explosives Technology Research at the New Mexico Institute of Mining and Technology, novel methods are being used to produce aluminide-based alloys in experimental quantities.

The use of explosively generated shock waves in the synthesis of new materials is a relatively recent concept. DeCarli and Jamieson^{1,2} were the first to synthesize diamond from graphite by using dynamic pressure, and today this is a successful industrial process.³ Shock-induced chemical reactions were first used by Sawaoka and Akashi to aid in shock consolidation of hard boron nitride powders.⁴ Additions of elemental titanium, aluminum and carbon powders to boron nitride were found to enhance interparticle bonding and to allow full densification of the compact. There has been little investigation, however, of the potential of using shock synthesis to produce novel intermetallic compounds with improved properties. In our experiments, an explosively generated shock wave is used to simultaneously synthesize niobium-, nickel- and titanium-aluminide phases and to consolidate titanium aluminide powders. The goals of this work are to apply shock synthesis to as-yet-unexplored alloy systems, and to establish the underlying mechanisms of the synthesis-assisted consolidation process.

Another method of synthesizing an aluminide phase involves the use of a solid/liquid reaction to form the alloy. This synthesis approach makes use of the fact that refractory metals such as niobium rapidly react with molten aluminum to form refractory metal aluminide phases. These reactions are highly exothermic and proceed rapidly until one or all of the reactants are completely consumed. The use of a solid/liquid reaction to produce an intermetallic compound is not a new concept, especially when one considers the large number of intermetallic compounds formed naturally by peritectic reactions. In addition, processes such as transient liquid-phase sintering have been used to produce a variety of materials from blended elemental powders. The distinctions to be made between these processes and reaction synthesis are that the solid/liquid reaction used here is applied to a congruently melting system rather than to a peritectic system, and that the resultant product is a single-phase material, rather than the typically multiphase product of liquid-phase sintering.

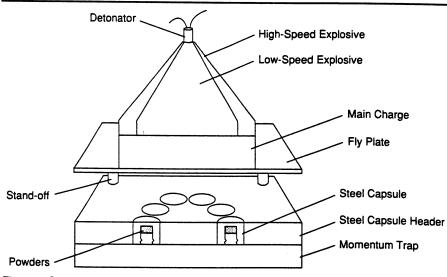


Figure 1. Shock-wave synthesis experimental apparatus.

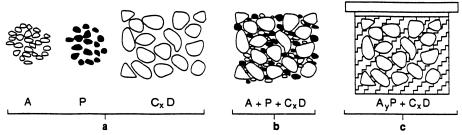


Figure 2. Shock-wave synthesis-assisted consolidation. (a) Elemental and intermetallic powders. (b) Powder mixture. (c) Shock-wave consolidated. A, P = elemental powders; $A_yP =$ "binder" intermetallic compound; and $C_yD =$ compound to be consolidated.

EXPERIMENTAL PROCEDURES

In the shock-wave synthesis-assisted consolidation process, TiAl powders are blended with elemental powders, which will form the shock-synthesized binder phase, and are packed into a stainless-steel capsule held in a steel plate. The powder mixture is simultaneously reacted and consolidated by a plane-wave shock front produced by a cylinder of explosive material and conducted to the sample by means of a flyer plate. The experimental setup is shown schematically in Figure 1.

For reaction synthesis, elemental aluminum and niobium powders are blended together in the appropriate stoichiometric ratio, canned and degassed, and sealed in vacuum. The powder pack is then placed in a hot isostatic press (HIP) unit and heated to a temperature sufficiently high to melt the aluminum and initiate reaction with the solid refractory metal powder. Upon completion of the reaction, the compact is heated to the consolidation temperature and argon gas pressure is applied to fully densify the reaction product.

Materials produced by these synthesis methods have been characterized by optical metallography, scanning and transmission electron microscopy, and x-ray analysis.

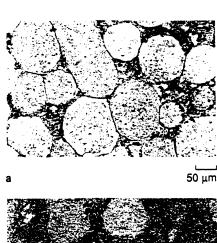
SHOCK-WAVE SYNTHESIS-ASSISTED CONSOLIDATION

The mechanism of chemically induced shock consolidation of powders is shown schematically in Figure 2. The inert intermetallic compound powder C_xD is blended with elemental powders A and P, which will react during shock processing to form the binder phase A_xP . The generation of heat, local melting at particle interfaces, and formation of the A_xP phase all promote bonding of the C_xD particles. In this study, hard and brittle TiAl powders, which are difficult to consolidate using conventional methods, were used as the inert phase, and Nb-Al, Ti-Al and Ni-Al elemental powder blends were used as the reactants. Optical micrographs of shock-processed compacts are shown in Figure 3. In all cases, full densification was achieved by the reaction-assisted consolidation process, with TiAl powder particles surrounded by reacted regions.

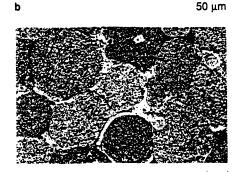
Characterization of the compact containing niobium and aluminum powders showed that reaction occurred, resulting in the formation of a Ti-Nb-Al ternary compound having both microcrystalline and amorphous structures. The final structure (amorphous or crystalline) depends on the local composition as well as the cooling rate experienced after passage of the shock front. The fact that amorphous regions were observed to transform to microcrystalline regions from heating by the electron beam during analysis indicates that the cooling rate experienced by the molten/reacted regions was sufficiently rapid to allow formation of the amorphous phase. Calculations performed by Schwarz⁵ predict that solidification of the interparticle melted regions occurs within 200 ns, which is an order of magnitude less than the duration of the shocked state. Shock-wave synthesis-assisted consolidation of TiAl powders appears to be a viable approach to the production of TiAl alloy compacts.

REACTION SYNTHESIS

The intermetallic phase which forms during reaction synthesis is that phase which is in equilibrium with molten aluminum at the synthesis temperature. For the binary niobium-aluminum system, this aluminide phase is Al_3Nb at synthesis temperatures between 800 and $1,000^{\circ}C$. Since the Al_3Nb phase is a line compound with no detectable homogeneity range, proper control of alloy stoichiometry is critical. Use of a low-temperature synthesis reaction allows precise control of alloy stoichiometry by avoiding volatilization of the low-melting-point component aluminum which would tend to occur during conventional melting operations. Uniformity and time of reaction are controlled by the powder characteristics. Large niobium powder particles result in incomplete reaction, as shown in the optical micrograph of Figure 4a of a compact produced using spherical niobium powders. By using smaller, irregularly shaped niobium powder, more complete reaction and



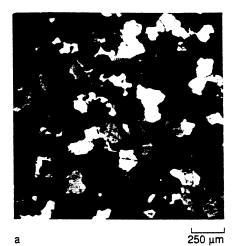




c 50 μm Figure 3. TiAl alloy compacts produced by synthesis-assisted shock-wave consolidation. (a) 70TiAl-15Ni-15Al. (b) 70TiAl-15Ti-15Al. (c) 70 TiAl-15Nb-15Al.

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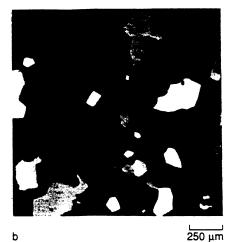


Figure 4. Optical micrographs of reaction-synthesized Al_aNb compacts showing: (a) incomplete reaction and (b) complete reaction and uniform microstructure.

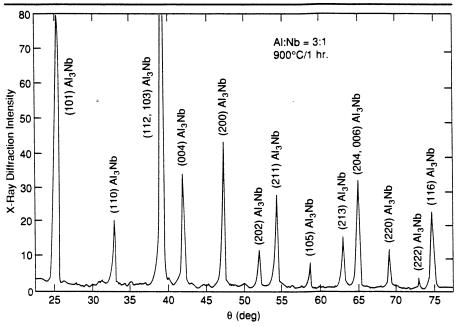


Figure 5. X-ray diffraction pattern of reaction-synthesized Al₂Nb.

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uniform microstructure are obtained as shown in Figure 4b. The x-ray diffraction pattern of this material (Figure 5) indicates that the compact is pure Al₂Nb phase.

One of the problems inherent in intermetallic compounds such as Al, Nb is a lack of ductility provided by the crystal structure. This phase has the DO22 crystal structure, and deformation modes commonly observed are difficult to activate. 6 It is possible, however, that more complex ternary aluminides based on Al₂Nb, such as $NbTiAl_3$, $Nb_2Zr_3Al_5$, and Nb_2VAl_2 , could be produced to obtain a more favorable crystal structure with greater ductility. These ternary phases may also have higher melting temperatures, wider ranges of homogeneity such that stoichiometric variation does not lead to melting, and improved oxidation resistance relative to the binary aluminide phase. The characteristics of some of these ternary phases produced by reaction synthesis are currently under investigation.

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