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Shock compression of Ti–B–Cu powder mixtures: Microstructural aspects

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ABSTRACT

Shock compression of Ti–B–Cu powder mixtures at a pressure of 3 GPa was investigated. The reaction between titanium and boron was initiated by the shock wave; however, it was not completed. Short high-energy mechanical milling of the powder mixtures before the shock compression ensured full conversion of titanium and boron to titanium diboride. Due to the geometrical issue of the interaction of the shock wave with the powder sample in a cylindrical ampoule, the outer part of the sample was denser but less uniform in the microstructure than the central part. The central part of the sample showed a nanocomposite structure with even distribution of 100–300 nm grains. The size of grain agglomerates of each of the two phases was not larger than 1 μm. In the outer part Cu and TiB_2-enriched areas up to 5–10 μm in size could be found. The observed difference is explained by intensive turbulization and mixing in the central part of the ampoule during shock compression.

1. Introduction

Shock compression of powders is a powerful tool of synthesis and consolidation of materials [1–3]. Being an extremely fast and highly energetic method, shock compression can be promising for the synthesis of fine-grained materials. A sample subjected to shock compression is usually confined within a holder or an ampoule. Each certain geometry of the ampoule and the sample will feature particular energy distribution during compression. As a result different zones of as-compacted materials may develop different phase composition and microstructures [4,5]. In the recent literature, there has been a discussion of the "positive" and "negative" effects of the formation of the so-called "central" zone featuring highly energetic turbulent flows during shock loading [3,4]. The negative effects are mentioned when it is difficult to obtain the same microstructure of a material along its cross-section. On the other hand, the turbulent phenomena can increase reactivity of the components of a mixture, which is in many cases, a positive result. It may also be possible to mechanically refine the microstructure of a material by grinding and pulverizing the particles or create conditions for chemical formation of small particles by decreasing the size of reactants.

In the present work, these issues were addressed in the investigation of shock compression of the Ti–B–Cu powder mixtures. This system is interesting due to a possibility of fabrication of a titanium diboride–copper composite, which is a promising material for electrical contacts, switches and erosion resistant parts. An ampoule of cylindrical geometry, which is commonly used in shock consolidation, was employed in this study. In cylindrical containers, no reactions in powder mixtures are usually observed in the part located at about 1/4 ampoule length from its top (the side adjacent to the igniter) [6]. In the rest of the sample, the central and peripheral zones can be distinguished. The central zone (Mach zone) in cylindrical ampoules has a conic shape and widens in the direction to the bottom of the ampoule. The peripheral zone is the part of the sample adjacent to the ampoule wall. Since the conic shape of the central zone in cylindrical ampoules has been previously well established [6], in the present paper we basically focus on the microstructural features of the Ti–B–Cu material. The samples for this study were taken from the middle part of the ampoule (at about 1/2 ampoule length from its top) to investigate the difference in the microstructure of the central and peripheral zones.

2. Experimental

Copper (99.7% purity, 40 μm average particle size), titanium (99.5% purity, 10 μm average particle size) and amorphous boron (94% purity, B94A, submicron) powders were used as raw materials. The composition of the mixtures was (Ti–2.1B) + 60 wt.% Cu. Mechanical milling of the mixtures was carried out in the high-energy planetary ball mill AGO-2 of the friction type with ball acceleration of 600 m s^{-2} [7]. The milling vials were vacuum pumped and filled with argon.

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Shock compression of the powders was performed in air in a steel ampoule 80 mm long with 15 mm external and 10 mm internal diameters. The powders were loaded in the ampoule, which was then treated on the vibro-table to increase the packing density of the powder particles. For the mechanically milled sample, the relative density of the powder charge before the shock compression was about 45% calculated relative to the fully dense and fully reacted product TiB_2–Cu. For the unmilled powders, the density was lower (30–35%). An axisymmetrical set-up described in [8] was used. The ampoule was placed along the axis of the cylindrical charge of an explosive ignited from one of the ends. No central rod was placed in the ampoule creating conditions for the formation of a conic central zone (Mach zone). The ampoule was plastically compressed during the explosion and, in turn, it compressed the powder sample to 8 mm in diameter.

The products of shock compression were examined by X-ray diffraction (XRD) phase analysis. Detailed characterization of the microstructure of the shock-consolidated materials was made by means of scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The microstructure of the shock-compressed samples was shown by etching the polished surface of the cross-section of the sample. To reveal different aspects of the microstructure, the sample was etched with either of the two solutions. They were ammonium persulphate $(NH_4)_2S_2O_8$ aq. solution (1:5) or iron chloride $FeCl_3$ solution (10 g $FeCl_3{\cdot}6H_2O$, 3 ml of concentrated HCl, 100 ml H_2O).

3. Results and discussion

During shock compression the initial structure of a powder mixture is very important because it strongly influences energy flow and distribution in the sample during shock loading. In this work two powder batches were shock-compressed. One of them was prepared by mixing the raw powders in the mortar; the other was mechanically milled in the AGO-2 mill for 2 min. In our previous work [9,10], the mechanically milled mixture prepared under the same conditions was studied by transmission electron microscopy and XRD. Though milled for a short time, the powder mixture acquires a composite structure and experiences grain refinement due to high-energy milling regime used; however, no new phases are formed. Thorough intermixing between titanium, copper and boron grains occurs along with an increase in the interface area in the system, though some agglomerates of titanium and copper grains may still be present in the mechanically milled mixture.

The XRD patterns of the shock-compressed material were taken from the cross-section of the samples. In the unmilled mixture, only partial interaction between titanium and boron occurred (Fig. 1a). Unreacted titanium can be detected by the XRD along with the product of the reaction—titanium diboride. In the mechanically milled mixture, the reaction was complete after the shock compression resulting in the formation of a two-phase TiB_2–Cu structure (Fig. 1b).

The calculated dynamic pressure in the shock compression experiments performed in this work was 3 GPa. Since low shock pressures cause low average temperatures in the sample, chemical reactions at such conditions may not occur or may be incomplete. Full conversion of reactants into a product under low shock pressures is a challenge discussed in a number of recent publications [11,12]. Thus, Mali et al. showed that the yield of the reaction between magnesium and boron to form magnesium diboride increased with an increase of dynamic pressure during shock wave synthesis [12]. The average temperature in the Ti–B–Cu sample generated by shock compression itself was estimated to be about 100 °C. This temperature is very low to thermally initiate the reaction between titanium and boron in the bulk of the sample. But the interaction does occur and one of the possible explanations is a local rise in temperature in the vicinity of the contacting reactants. According to Nesterenko [13], the temperature of the hot spots in the powder charge of micron-sized particles subjected to relatively low shock pressures reaches 800 °C. The hot spots are believed to be located in the vicinity of collapsing pores in the powder charge. When the exothermic reaction is triggered, the temperature increases in the hot spots and in the rest of the sample. In order to roughly estimate the temperatures developed, the combustion temperatures measured on the pre-compacted samples of Ti–B–Cu powders reacting in a self-propagating mode can be used. As was reported in [14], in the Ti–B–Cu mixtures of the same composition mechanically milled in the conditions close to those used in the present work, the combustion temperature was about 1350 °C. So, we can expect this temperature to develop in the shock-compressed reacting Ti–B–Cu mechanically milled powders.

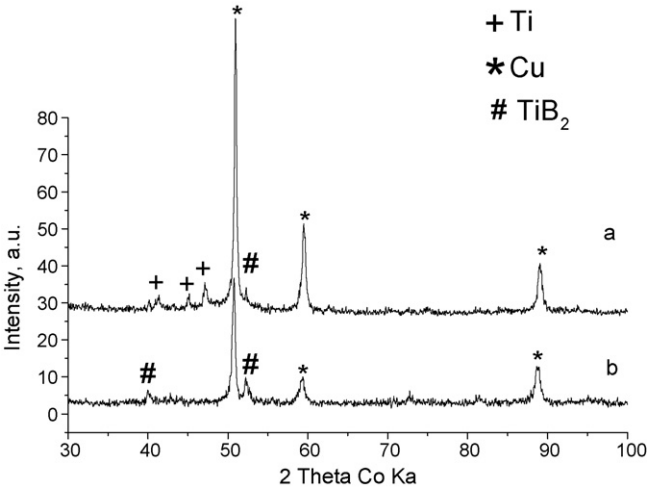

Fig. 1. XRD patterns of the shock-compressed Ti–B–Cu samples: (a) obtained from the unmilled powder mixture and (b) obtained from the mechanically milled powder mixture.

The peripheral zone of the compacted material was approximately 1.5 mm thick at half-length distance from the top of the ampoule as can be seen from the optical micrograph of the cross-section of the sample (Fig. 2, zone A). The detailed investigation

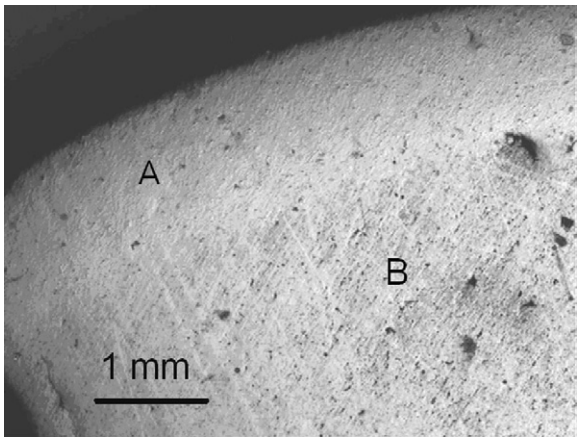

Fig. 2. Optical image of the cross-section of the shock-compressed sample obtained from the mechanically milled Ti–B–Cu powder mixture ((A) peripheral part and (B) central part).