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Review Article

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Shock Wave Assited Chemical Reactions and Fabrication of Tantalum Aluminates

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ABSTRACT

The different composition Ta-Al based reactive blends of powders formed into cylindrical rods using two stage a hot explosive consolidation (HEC) method. The aims of investigation were to fabricate tantalum aluminates near to theoretical densities and to investigate its structure/property relationship. The processing temperature were changed up to 10000C. The intensity of loading was under the 10GPa. The investigation showed that the application of two stage HEC method is beneficial to the consolidation and synthesis of the Tantalum aluminates, resulting in near theoretical densities, good hardness values and the full formation of TaAl3 intermetallic compounds behind the shock wave front. The structure and property of the samples obtained, as well as the result of formation of tantalum aluminates depends on phase content of the precursors and shock loading conditions too.

Keywords: Tantalum Aluminates, Fabrication, Intermetallic, Temperature

Introduction

There still is a shortage of data concerning the study of explosive compacting of refractory and intermetallic powders. This is primarily caused by number of technological difficulties associated with shock wave processing [1-5]. Difficulties are related to the impossibility of direct consolidation and synthesis of powder blends. The problem is that during compaction of a mono-phase or composite material powder, the short period of the loading cycle is not enough for plastic flow to occur in and outside of the consolidating particles and, as a result, no new phases and common boundaries can form under and behind the shock wave front. Moreover, the stresses necessary to facilitate plastic flow of the particles and for the formation of common boundaries between them exceeds the ultimate strength of their steel containers. Typically, this happens where the consolidated powders are situated, therefore after compressive loading, the sample container is always destroyed.

Application of a low intensity shock wave, followed by sinter processing, to pure refractory and ceramic powders is not effective in most cases either [1]. The use of high sintering temperatures after consolidation leads to the annealing out of the as processed crystalline structure of the shock loaded particles. Lastly, the melting of the steel container may take also place which would interfere with obtaining an uncontaminated but well consolidated composite material.

The positive role of the use of high temperatures during the consolidation processes of refractory and hard alloy powders has been well documented [5-10]. Application of high temperatures in hot explosive consolidation (HEC) allows the consolidation mechanism to work by plastic flow and mutual collision of the compacting particles, resulting in the melting of their surfaces

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and formation of common interfacial boundaries and solid solutions at essentially, much lower loading intensities [5,6].

Among the neutron absorbing materials to be used in nuclear technology the isotope of Boron-10 (10B4C) is most effective and has the leading position. At the same time, it must be considered as well another (n, γ) absorbers. In this case the catching of neutrons is accompanying with exhaling of γ rays by transformation into the new isotope (daughter isotope). In spite of fact that 10B4C based composites seems are best as a neutron absorbing materials, the fabrication and treatment from mentioned composition are too expensive (expensive equipment and expensive final diamond treatment/polishing processes). Additionally, B4C ceramics are too brittle materials and any further application of rolling and welding technologies are impossible.

In contrast to B4C, the main attractive advantage of refractory tantalum (Ta) is that during the application no gas secretion occurs. In spite of fact that Ta has only half nuclear cost of B4C (on unit volume) the work period of it will be reducing more slower because of takes place transformation into Ta-182 and Tungsten(W) which have weight nuclear cost too. Advantages of Tantalum is maintaining of high absorption cross-section for fast neutrons (E>01-0.3 MEB) and low swelling after the neutron irradiation. The high melting temperature, low thermal expansion coefficient, low elasticity of steam, important acid resistance, not bed heat conductivity and good miscibility with structural materials makes Tantalum too attractive material for nuclear technique. Only too high elasticity and based on it a low strength and heat strength may limit application of Ta in nuclear industry. To prevent mentioned limitation in application of Tantalum may be solved by alloying by Aluminum (Al) or Carbon(C), Beryllium (Be). Application of Boron (B) seems twice interesting because of increasing absorption cross-section of neutrons. From the stand point of nuclear security most appropriate seems to be alloying by Aluminum (Al) because of their low absorption cross-section of neutrons. Application of mentioned alloying materials simultaneously essentially will improve the corrosion resistance of alloys. In addition of good elasticity and miscibility with different materials, Ta-Al (B, C) system belongs to reaction pair and self -propagating high temperature syntheses (SHS) with release of high temperature around 25000C there takes place. Depending on aluminum volume the intensity of exothermal reactions changes and correspondent released temperature too.

The objectives of this study were thus threefold. First, to demonstrate that using non-standard powder metallurgy technologies SHS and HEC the initial Ta-Al precursor powders could be consolidated to near theoretical densities and appropriate microstructures. Second, to show that, adding to starting reactive Ta-Al blends B powder and HEC at high temperatures allows to fabricate composites with increased absorption cross section. Third, to investigate the shock induced structural changes in both the Ta-A and Ta-Al-B reactive powder blends after HEC. The results of this effort are described.

Experimental Procedures and Materials

For this work we used a fairly, mostly spherical 5 micrometer Ta and Al powder, obtained from Dr. Daniel Brannagan while he was still at Idaho National Engineering and Environmental Laboratory, Idaho Falls, ID, USA. The powder is a one-component with purity 99.95%. The B and B4C powder were received from local High Technology National Center with grain size 5-10 μ.

In order to consolidate the Ta-Al & Ta-Al-B reactive powder mixtures to near theoretical densities in hot conditions two stage cylindrical set up of loading created in and presented on Figure 1 was used [11].

Step 1: cylindrical samples were consolidated at room temperature to increase its starting density and to activate before hot shock loading consolidating particles surfaces.

Step 2: preliminary consolidated cylindrical billets were reloaded second time above 9000C with intensity of compression under 10GPa.

Only Ammonite with a detonation velocity of 3.1 km/s, Ammonite diluted with NH4NO3, used as a passive were used as explosive materials. The corresponding intensity of loading on the wall of the steel container was under 10 GPa.

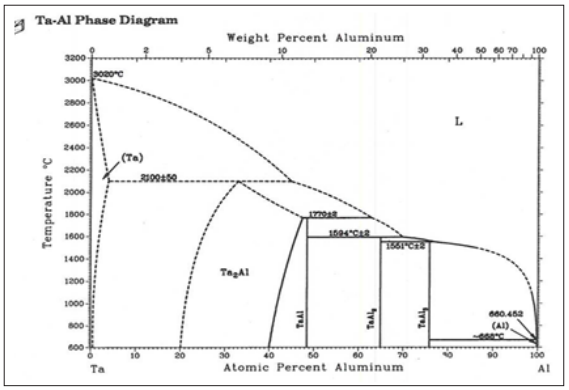


Figure 1: Ta-Al phase diagram

Only four intermetallic: Ta2Al- Tm= 2,100C; TaAl- Tm=1,770C; TaAl2-Tm=1,594C; TaAl3; Tm= 1,551C; Taking into account the data from the Ta-Al phase diagram, we can conclude that the selection of the phase composition and the use of various passive alloying elements to regulate the synthesis temperature are important technological parameters in its subsequent static or dynamic consolidation-synthesis processes. The figure #2 shows the experimental setup of two stage HEC of Ta-Al reactive blend.

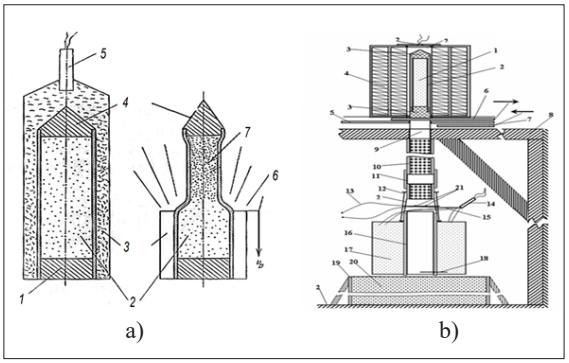


Figure 2: The experimental setup for two-stage shock wave compaction-synthesis of reactive Ta-Al blends a) densification by shock waves at room temperature: 1-Bottom plug of steel tube; 2. Precursor powders; 3. Explosive powder; 4. Upper plug

of steel tube; 5. Electric detonator; 6. Products of detonation; 7. Consolidated powders. b) HEC device: 1. consolidating powder material; 2. Cylindrical Steel container, 3. Plugs of steel container, 4. Heating wires of furnace, 5. Opening and closing movement of furnace, 6. Opening sheet of furnace 7. Closing sheet of furnace, 8. Basic construction of HEC device, 9. Feeding steel tube for samples. 10. Movement tube for heated container, 11. Connecting tube from rub, 12. Accessory for fixing explosive charge, 13. Circle fixing passing of steel container. 14. El. Detonator, 15. Detonating.

Results

During the consolidation of precursors at high temperatures essentially increases the residual temperatures as a sum of the preliminary temperature and resulting temperature behind of shock wave front. As a result, some negative effects may there take place: changes of geometry and shape of fabricated samples, diffusion of the atoms from steel container into the compacting powder with formation of the undesirable phases etc. In case of consolidation of the reactive blend powders there may considered 2 cases: first – hot consolidation below temperature of SHS reaction with followed initiating of SHS process behind of the shock wave front due to increased reminder temperature. And second- heating the reactive blends with initiating SHS reaction and followed shock wave loading. Taking into account that SHS reaction in reactive blends accompanied with essentially increased release of gas, the second approach for consolidation-synthesis of composites from reactive blends of powder becomes more problematic. In this case the problem can be solved by adding to starting powder blends some passive components such as carbides or borides depending of tasks and purpose of investigation.

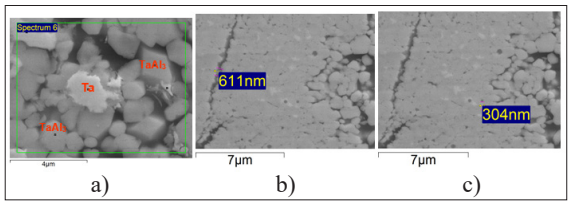
The figure #3 illustrates the above mentioned and shows the effect of SHS reaction on HEC billets upon the whole length of the samples.

a) b)

Figure 3: HEC billets at 940°C with intensity of loading under 10GPa

a) Heating of densified Ta-3Al sample above 950°C with initiating SHS reaction; b) HEC of preliminary densified Ta-3Al billet at 940°C with initiated and followed SHS reaction; As it's seen from picture (3a) the initiation of SHS reaction in cylindrical steel container at 950°C with followed reaction gas release resulting in swelling of container and further transportation of its towards of explosive charge inside of cylindrical pipe becomes impossible. The heating of containers with Ta-3Al reactive blends up to 940°C with post shock wave loading (before initiation of SHS process) allows HEC samples near to theoretical density. The effect of released gases may be observed on the surfaces of feeding steel tube and container's wall (Figure 3b) where the melted steel guide pipe and the container's wall are observed. The application of B4C passive additives in reactive blends reduces volume of released gases and processing temperature. As a result, the defect free billets (Figure 3c) are obtained.

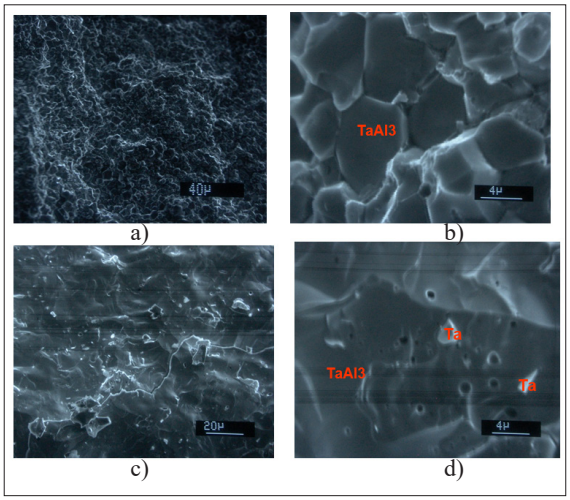
The figure 4 represents the SEM pictures of Ta-Al reactive blends after HEC at 900°C with intensity of loading under 10GPa



a) b) c)

Figure 4: The SEM pictures of HEC Ta-Al reactive blends consolidated and synthesized in one stage at 900°C with intensity of loading under 10GPa. after shock wave. a) The unreacted Ta phase and synthesized TaAl3 phases are observed; b & c) Some microcracks are observed. There is a wide distribution of the grain size: from about 5 μm to the submicron scale of 200 nm

The figure 5 represents the SEM fractures of different tantalum aluminates obtained after HEC at 9400C and post SHS processes.

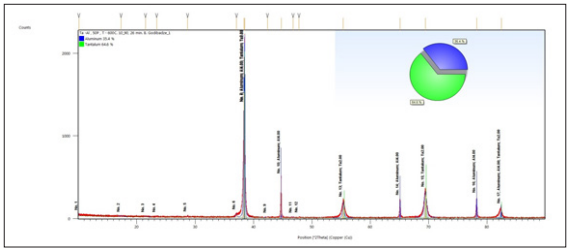


a) b) c) d)

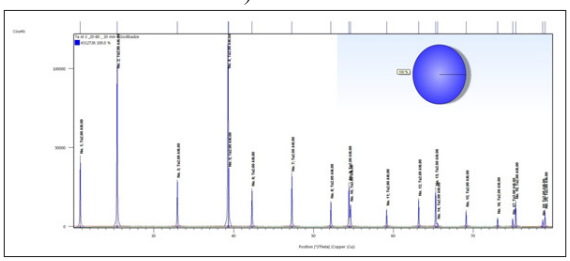
Figure 5: The SEM fractures of different composition Ta-Al intermetalic composites obtained after shock wave consolidation-sinthesis at 9400C with intensity of loading under 10GPa. a-b) The fractures of sinthesized TaAl3 intermetalics at different magnification; c-d) The fractures of sinthesized Ta2Al. The difference in sinthesized particle size is observed (b &d)

As investigation of fractures shows the initial stoichiometry of Ta-Al reacted blend may have influence on final phase formation and morphology of obtained tantalum aluminates after HEC. The comparison of fractures for Ta-3Al and Ta2-Al shows that the morphology of synthesized tantalum aluminates differs from each other. Personally, for aluminum reach composition the joint boundary between the synthesized tantalum aluminates is clearly observed when for aluminates with higher content of tantalum that was HEC in same conditions the joint boundaries between the grains too difficult to identify. This effect for aluminates which were HEC in same conditions may be only explained with higher strain-rate consistent in compositions as a result of increasing of tantalum content. As a meter of fact that during the shock wave loading, the created stresses in materials depends on its density (P=pDU) and straightforward increases with increasing of its

densities. Practically for Ta-2Al precursors during the HEC the intensity of loading was twice increased. From the other side the traces of unreacted Ta inclusion are observed (Figure 5d) that could be result of formation mainly TaAl3 phases behind of shock wave front. The reason of appearance of unreacted Ta particles in fully synthesized tantalum aluminates might be fact that the formation of TaAl3 takes place at Tm=1,551ºC and is energy beneficial in contrast to Ta2Al (Tm=2,100ºC).



a)



b)

Figure 6: The diffraction pictures of HEC Ta-3Al reactive blends obtained at different temperatures of processing with intensity of loading under 10GPa. a) HEC at 6000C; b) HEC at 9400C. As it's seen from diffraction pictures only HEC of Ta-3Al reactive blends above 9000C temperatures (at 9400C) provides full synthesis and formation of the single-phase tantalum aluminates in whole volume of fabricated samples (Figure 6b). The decreasing temperature below 9000C, partially chemical reactions and inclusions of unreacted Ta phase are observed (Figure 6a).

The results of SEM investigation are confirmed by diffraction picture of HEC Tantalum Aluminates. There was established that only at 940ºC temperature HEC may provide full synthesis in Ta-3Al reactive blends in whole volume. As for other tantalum aluminates (2Ta-Al, Ta-Al & Ta-2Al) fully synthesis was impossible and practically always traces of Ta reminder were observed in HEC samples. The reason seems to be essentially high value of its formation that exceeds Tm=1,600ºC (for TaAl2) and increases up to Tm=2,100ºC for T2Al aluminates. The Figure 6 confirms the above mentioned

The Data of hardness for different compositions tantalum aluminates after HEC presented in Table #1.

Table 1: Hardness values of tantalum aluminates after HEC

Precursor Composition	HEC temperature ˚C	Hardness Hv, kg/ mm2 Loading 100gr
Ta2-Al	950	126.64
Ta-Al	900	261.50
Ta-3Al	940	320.12

Discussion

In comparison with previous results connected with HEC Ni-Al and Ti-Al composites, the current application of HEC to Ta-Al composites gave better results. As it was shown in previous works the application of high temperatures and HEC composites of Ni(Ti)-Al did not provided fully transformation in intermetallic compounds. HEC of Ni-Al composites at 10000C in spite of good density and correct geometry provided only partial formation of nickel aluminates. Analyzing the situation connected with formation of aluminates it was concluded that the reason was low intensity of shock wave loading due to low preliminary density of precursors before HEC.

The use of Ta-Al precursors seems to be more promising, from the standpoint of features of HEC consolidation processes and formation of tantalum aluminate compounds. This is the high density of Ta powder (16.4 g/cm3) and the high reactivity of Ta-Al system. Application of HEC technology may be solved previously fixed problems connected with partial formation of aluminates, based on developed of high intensity of loading and post initiation of SHS process behind of shock wave front.

Results of the X-ray investigation show that only HEC at the 940ºC temperature allows to reach full phase formation after loading in whole volume of samples and to obtain single phase tantalum aluminates. The decreasing of temperature below the 900ºC and HEC of precursors with intensity of loading under 10GPa in all cases have no success.

The HEC of Ta-Al-20%B4C precursors and fabrication of the TaAl3- Ta2AlC -TaB high dense composites showed that there took place fully dissolution of B4C phase with further redistribution of the separate B and C towards of formation of new TaB and TaAlC phases.

The evaluation of hardness value for different Ta-Al composites including TaAl-B4C composites showed the influence of boron after HEC on the hardness value in tantalum aluminate composites. The Ta and Al proportion in precursors seems have influence on hardness value in HEC composites too.

Concluding Remarks

- Established that SHS reaction in Ta-Al preliminary densified composites starts at 9400C with further full transformation into the tantalum aluminates. It's established that formation of TaAl3 phase has priority and after HEC for any weight proportion of Al and Ta in the precursors there always formed TaAl3 compound due to its lowest temperature of synthesis.
- Established that by combination of HEC and SHS processes it's possible to consolidate bulk aluminates compounds near theoretical density with perfect structure and good hardness. In order to prevent cracking, it's recommended to HEC Ta-Al billets before SHS process around 9400C with post SHS reaction.

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