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EXPLOSIVE INDENTATION STUDY OF B_4C-$TiAl_x$ COMPOSITES FABRICATED BY THE DIPPING EXOTHERMIC REACTION PROCESS

JONG HO KIM, HARIS MASOOD ANSARI, HANEUL KIM, DO KYUNG KIM

Korea Advanced Institute of Science and Engineering, Kusong-dong, Yusong-gu, Daejeon 305-701, South Korea
truffle@kaist.ac.kr

SOON NAM CHANG

Agency for Defense and Development, P.O. Box 35-1, Yusong-gu, Daejeon 305-701, South Korea
snchang33@hanmail.com

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The aim of this study is to fabricate a high volume fraction B_4C-reinforced intermetallic matrix composite by the dipping exothermic reaction process and investigate the shock impact damage response of composites by explosive indentation experiment. It has been shown that the final microstructure of the dipping exothermic reaction process-fabricated composite can be tailored by treatment of the constituent powders and post heat treatment. The hardness and impact damage resistance of the fabricated composites were evaluated.

Keywords: Explosive indentation; composite; DERP; B_4C-$TiAl_x$.

1. Introduction

Ceramic reinforced composites have emerged as an important class of materials for structural, wear, thermal, transportation, and electrical applications, because of their excellent properties relative to those exhibited by the corresponding monolithic material.[1,2] Thus various fabrication methods have been developed, such as powder metallurgy, stir casting, and pressure infiltration method, etc. In conventional composites, which could be regarded as *ex situ*, the reinforcing phase is prepared separately prior to the composite fabrication.[3] In this case, the scale of the reinforcing phase is limited by the starting powder size, which is typically in the order of microns to tens of microns and rarely below 1 μm. The other main drawbacks that have to be overcome are the interfacial reactions and poor wettability between the reinforcements and the matrix due to surface contamination of reinforcements. Recently there has been a growing interest in the development of technologies for *in situ* production of composites, such as direct metal oxidation process, the exothermic dispersion process, self-propagating high temperature synthesis (SHS) and reactive gas injection.[3-5] Dipping exothermic reaction process is one

of the novel *in situ* process techniques for the fabrication of high volume fraction ceramic reinforced metallic-intermetallic matrix composites. Recently Song suggested that high volume TiC reinforced composites could be synthesized by using a dipping exothermic reaction process (DERP) as a new processing technique, utilizing the exothermic reaction between molten aluminum and preforms.[5]

B_4C is an important ceramic material with many useful physical and chemical properties. One of the outstanding physical and mechanical properties of boron carbide is its hardness, which is second to diamond and c-BN. This specific property comes along with other attractive properties such as high impact and wear resistance, low density, high melting point, and high capability for neutron adsorption.[6] However, its extreme sensitivity to brittle fracture and the difficulties involved in fabricating dense bulk materials have limited its use in industrial applications. The problem can be significantly reduced by the production of B_4C based composites. For applications at low temperature, aluminum or aluminum alloys is a material for choice.[6]

In this study, it is suggested that high volume fraction B_4C (>30 vol.%) reinforced intermetallic matrix ($TiAl_x$) composites can be fabricated by DERP. DERP is very quick (less than 3 minutes) and the infiltration temperature is also quite low (~900 °C) as compared to the one in conventional processes. It has been shown that the final microstructure of the DERP-fabricated B_4C-reinforced $TiAl_x$ composites can be tailored by treatment of the constituent powders prior to preform preparation and post processing. This involves sol-gel protective coating of B_4C with ZrO_2 prior to mixing with Al and Ti powders, and also post heat-treatment. Furthermore, the hardness and impact damage resistance of the fabricated composites can be tailored by B_4C powder coating and post heat-treatment.

2. Experimental Method

In this study preforms was prepared by mixing and compaction of starting powers which includes B_4C of 400 grit, Al of 10 μm average particle size and Ti of 45 μm average particle size. These powders weighed to the nominal composition as B_4C:Al:Ti = 50:20:30 and mixed using SPEX miller (SPEX CertiPrep Inc., NJ, USA) for 5 min in the condition of dry ball milling. The powder preforms were prepared with uniaxial pressure of 200 MPa. The prepared preforms were dried in a vacuum oven at 393 K for 4 hours to eliminate moisture. The prepared preforms were cylindrical shape, 25 mm diameter and about 40 mm height. To preserve the shape of the preform it was placed inside a graphite container. The DERP was performed with 99.9% pure liquid aluminum at 900 °C. The preforms were taken out after being held for 3 min from the end point of the reaction. The end point of the reaction can be determined by observing the evidence of dazzling light that originates from the reacted preform. Fig. 1 shows the schematic diagram of the experimental process and setup used for DERP.

Two types of preforms were prepared for DERP. One type consisted of uncoated B_4C with Ti and Al powders and the other type consisted of ZrO_2 coated B_4C with Ti and Al powders. $ZrOCl_2$ were used as precursor solutions for ZrO_2 sol-gel coating. The precursor

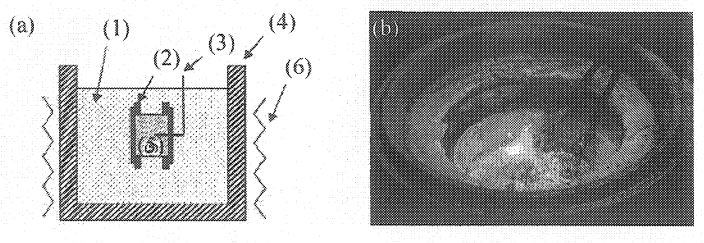


Fig. 1. (a) Schematic illustration of experimental setup for dipping exthothermic infiltration (1:Al melt; 2:graphite; 3:thermocouple; 4:crucible; 5:specimen; 6:furnace), and (b) dazzling light and fire emanating from specimen, indicating a strong exothermic reaction.

solution of 50 ml was added to 500 ml distilled water containing 25g B_4C powders along with NH_4OH at a rate of 1.6 ml/min to initiate heterogeneous precipitation on the surface of B_4C particles. The pH was monitored and the addition of NH_4OH discontinued when the pH reached 12. The powder was dried in the oven and placed in an alumina boat for heat-treatment at 1200 °C for 3 hours under flowing argon atmosphere.[7] After fabrication of composites, post heat-treatment was conducted as a route for the enhancement of mechanical properties and for microstructural development of the B_4C-$TiAl_x$ composites. Specimens were heat-treated in the tube furnace at 1000 °C under a flowing argon atmosphere for 5 hours.

Scanning electron microscopy, energy dispersive spectroscopy and X-ray diffraction were used to characterize the phase change and optical microscopy was used for microstructure characterization. Vickers hardness of the fabricated composites was measured to investigate the effect of sol-gel coating and post heat-treatment on the hardness of composites. Explosive indentation testing was conducted on the bonded-interface specimen.[8] The bonded-interface specimens were clamped with auxiliary fixture, and then explosive indentation was performed using an electric bridge wire (EBW) detonator. The EBW detonator has a cylindrical shape with stainless steel case and a bottom plate of 5 mm diameter. During explosions, the bottom stainless steel flyer generates impact shock into a specimen. Subsurface damage was characterized by merged optical mosaic images.

3. Results and Discussion

3.1. *As-infiltrated B_4C composites*

Fig. 2(a) shows the optical micrograph of the as-infiltrated composite. The microstructure mainly consists of dark B_4C particles in an Al matrix. The B_4C particles are surrounded by a phase which was characterized by XRD to be TiB_2. The XRD results reveal that apart from B_4C, Al and TiB_2 phases, a small amount of $TiAl_3$ and Al_3BC phase is also present. Al_3BC forms by the reaction of Al with B_4C in a temperature range of 680 to 1000 °C.[9] There was a preferential reaction of Ti and B_4C, due to which all the Ti source

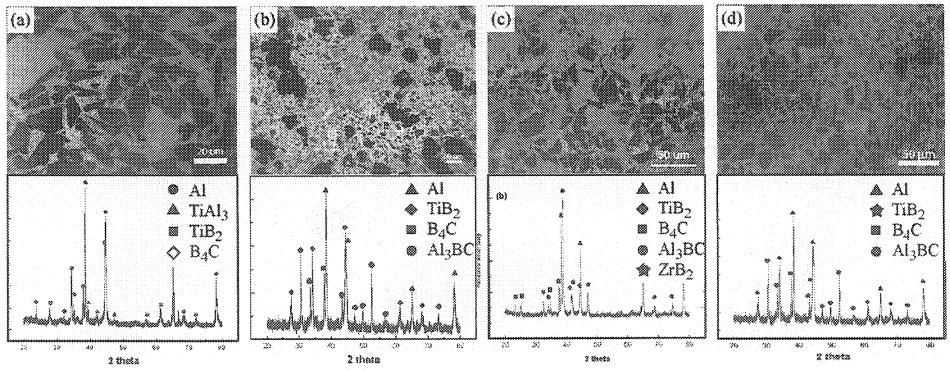


Fig. 2. Optical micrograph and XRD pattern of (a) as-infiltrated (b) as-infiltrated and post heat-treated (c) sol-gel driven ZrO2 coated and (d) sol-gel driven ZrO2 coated and post heat-treated B4C-TiAlx composites.

was consumed and $TiAl_x$ phase was rarely formed. The B_4C volume fraction is calculated to be 36 %, which looks reasonable figure considering the extent of Ti and B_4C reaction.

Fig. 2(b) shows the microstructure of the same composite after heat treatment at 1000 ^oC for 5 hours under a flowing argon atmosphere. The dark phase is B_4C, the gray phase is Al_3BC, and the bright phase is Al. XRD result reveals that there is no residual $TiAl_3$ phase after heat treatment. Wang et al. suggested that because Ti atoms mainly occupy the (001) planes in the $TiAl_3$ crystal structure, in the presence of B source, these planes will be attacked and TiB_2 will be formed because of the negative free energy change of TiB_2 formation.[10] Hence, $TiAl_3$ is expected to decompose to form TiB_2 and leave porosity in the places where it previously resided. The formation of Al_3BC was expected because of the large amount of Al present in contact with B_4C. The microstructure after heat treatment is more homogeneous, apart from the porosity, and if the porosity could be controlled by some means, the mechanical property is expected to increase from its current value.

3.2. *ZrO2 coated B_4C composite*

Sol-gel coating technique was used to coat B_4C powder with ZrO_2 and the coated powder was later heat treated and some of ZrO_2 phase was converted to ZrB_2. Fig. 2(c) shows the optical micrograph and XRD result of the fabricated composite. In case of ZrO_2 coating, the reaction is not as vigorous as in the previous case. Recovered sample shows good dimensional stability confirming the infiltration has taken place. XRD result reveals three major phase, B_4C, Al and $TiAl_3$, although there is still some TiB_2 present, but to a limited extent. In contrast to the previous results, the major peaks are $TiAl_3$. The micrograph reveals that surrounding the dark B_4C particles is a light TiB_2 phase, but the matrix now consists of an interconnected $TiAl_3$ phases, and pockets of a bright Al phase. The result shows that ZrO_2 coating completely suppresses the B_4C-Al reaction resulting in the enhancement of the Ti-Al reaction. The calculated volume fraction of B_4C phase is 36 %.

Fig. 2(d) shows the optical micrograph and XRD results of the ZrO_2 coated B_4C composite after heat-treatment at 1000 ^oC for 5 hours under Ar atmosphere. The

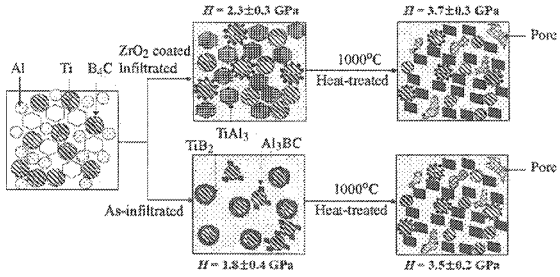


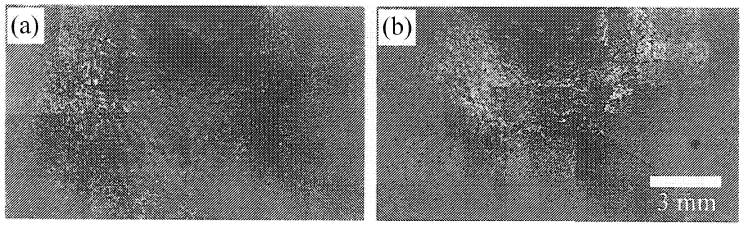
Fig. 3. Schematic diagram of the phase evolution during DERP and subsequent heat-treatment including the Vickers hardness values for each experimental condition and microstructure.

microstructure reveals that the $TiAl_3$ phase has disappeared totally and have give rise to Al_3BC phase, which is also confirmed by XRD and EDS. The Al_3BC formation has a good effect on the final microstructure of the composite. Most of the free Al in the matrix is consumed to form Al_3BC and the continuity of Al phase is disturbed. This has a drastic effect on the mechanical properties as discussed following section.

3.3. *Hardness and explosive indentation study*

Phase evolution during DEPR depends on the initial composition of the constituents and the heat treatment. Based on the phase identification data, the schematic diagram of microstructural development after fabrication of dipping exothermic reaction infiltrated and their subsequent heat-treated is given in Fig. 3. The Vickers hardness of these four samples was measured and those values are included in Fig. 3. There is a definite trend towards higher hardness as the Al phase depletes and more Al_3BC phase is formed. Due to Al depletion and decomposition of $TiAl_3$, some isolated porosity is generated, the generation of which is expected to saturate as Al is confined to small pockets, far away from boron source. The Al confined to the pockets cannot reach the remaining B_4C particles, and the hardness value is expected to saturate further heat-treatment.

Two kinds of samples, uncoated without heat-treatment and ZrO_2 coated with post heat-treatment were used for explosive indentation experiment. Due to the remarkable difference of the phase in the matrix and reinforcement particles, which results the difference of hardness, these two samples can be representatives of the ductile and brittle B_4C composites. Fig. 4 shows optical micrographs of subsurface damage of explosively indented samples. In case of uncoated sample, the damaged area is large and severe deformation is dispersed through entire deformed region. The damage pattern of uncoated specimen shows similar trends with that of explosively indented metals because most of matrix consists of Al phase. Furthermore, the shock impact is not effectively adsorbed by the reinforcement at all. In contrast to uncoated one, Fig 4(b) shows that apart from severe cracks, coated sample has relatively small deformed area after indentation. The damage of explosively indented sample reveals that brittle fracture mode prevails like ceramic materials. The major phases, B_4C, Al_3BC and TiB_2 act as a good reinforcement



to absorb the shock impact by generation of cracks. These severe cracking reveals that most of shock impact energy is consumed in the failure of the reinforcement phase. The damage response to the shock impact is primarily determined by the constituent of matrix phase. Al-based B_4C composite shows ductile deformation behavior and Al_3BC-based B_4C composites shows brittle fracture mode. This results implies that the mechanical properties of B_4C composites can be tailored by the coating of starting powder and post heat-treatment process to achieve desired properties.

4. Conclusion

The aim of this study is to fabricate a high volume fraction B_4C-reinforced intermetallic matrix composite by the dipping exthothermic reaction process and investigate the impact damage response by the explosive indentation. For optimum properties and homogeneous microstructure, sol-gel driven coating and post heat-treatment technique were applied. In case of applying ZrO_2 coating of B_4C particles, the matrix was predominantly $TiAl_x$, while in case of as-infiltrated sample, the matrix was Al. Post heat-treatment proved an effective processing step to get a more homogeneous microstructure. The major phases were B_4C, Al_3BC and TiB_2. Vickers hardness shows a remarkable improvement after heat treatment. Impact damage behavior of B_4C composite was determined by the matrix phase and the abundance of Al_3BC phase improves the impact damage resistance.

Acknowledgments

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