THERMALLY INDUCED CRACKING IN ALUMINUM/BORON CARBIDE COMPOSITE

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Abstract: *Thermal shock tests have been performed to determine the influence of rapid temperature change on crack nucleation and propagation in lightweight Al/B$_4$C composite. The composite was produced using self-propagating high-temperature synthesis (SHS) with subsequent heat treatment at 700°C in vacuum. Quenching in water was chosen as thermal loading. The specimens have been subjected to a specified number of thermal cycles and then analyzed. Post-shocked samples have been characterized by means of microstructural and fractographical investigations to analyze the crack paths.*
Key words: boron carbide composite, thermal cycling, quenching, crack path.

1. INTRODUCTION

Reaction sintered materials in the system aluminum/boron carbide were investigated. Development of composites of Al-B$_4$C system is of interest because the both constituents have low specific weight; additionally the system combines high hardness, high elastic modulus, outstanding wear resistance of ceramic and enhanced ductility, high strength/density ratio and corrosion resistance of metallic phase [1, 2]. The additions of ceramic exert influence on coefficient of thermal expansion and thermal conductivity [1]. B$_4$C as compare with other ceramic reinforcement (Al$_2$O$_3$, SiC) has lower density, resulting in composites with higher specific stiffness [2].

Thermal shock and thermal cycling are the life-limiting factors for components exploitable in rapidly changing thermal conditions and high-temperature materials. Monolithic ceramics, including B$_4$C, is known to be susceptible to thermal shock failure through their low coefficient of thermal expansion (CTE) [3]. Boron carbide with CTE (4.5-5.0)×10^{-6} °C^{-1} is one of the promising ceramic materials for the design of low CTE composites. Although the high-temperature application of Al/B$_4$C composites is limited because of a requirement that the matrix and reinforcement must be mutually chemically inert in the operating temperature range, the use of this class of materials is favorable in unlubricated sliding, i.e. in conditions where the friction temperatures may reach up to relatively high values [4]. Due to high neutron absorption of B$_4$C, the composites are expected to find applications in nuclear industry [2], where the thermal properties play an important role.

Composite materials processed and exploited at elevated temperatures exhibit thermal residual stresses when cooled to room temperature due to differences in CTE of the constituent phases. The CTE of Al is about five times larger than that of B$_4$C. The developed residual stresses have several undesirable effects such as diminished fatigue strength, accelerated stress corrosion, shape distortion [5].

From this point of view it is essential to study the Al/B$_4$C composites exposed to cyclically changing thermal loading. The understanding of thermal behavior and nature of thermal damage of B$_4$C-based composites can provide additional knowledge for their microstructural design.

2. EXPERIMENTAL

The materials were produced by reaction bonding of attrition mixed B_4C and Al powders. The mixed powders containing 52 vol.% B_4C were heated in a furnace up to temperature 850°C to initiate the exothermic reaction. When reaction became complete pressure was applied at liquid state of Al for better densification achievement. Synthesized material was then heat treated at 700°C in vacuum during 30 minutes for structural development. Low temperature was chosen in order to prevent the formation of brittle high-temperature phases [6]. Samples of parallelepipedic shape 20×12×7 (mm) were prepared by diamond polishing down to a diamond particle size of 1 μm.

For thermal loading samples were heated in a furnace and kept for 20 minutes at specified temperature in order to attain thermal equilibrium. The temperatures lie in the range 300-600°C. Heating was performed in air at atmospheric pressure. Heated samples were then dropped into quenching medium (water maintained at room temperature). The procedure described above represents one thermal cycle. Up to 20 cycles were done at each temperature.

The microstructural observations were done using Hitachi TM-1000 Scanning Electron Microscope (SEM) and optical microscopy (OM).

3. RESULTS AND DISCUSSION

3.1 Characterization of initial composite

The main microstructural character of Al/B_4C composites is the variety of microstructural types tailored [6-8]. Depending on processing conditions the microstructure can vary from the two-phase typical for traditional MMCs to the multiphase interpenetration one.

SEM-image of the microstructure of initial composite is presented in Fig. 1, where boron carbide particles are in dark-grey color and binder is in light-grey color.
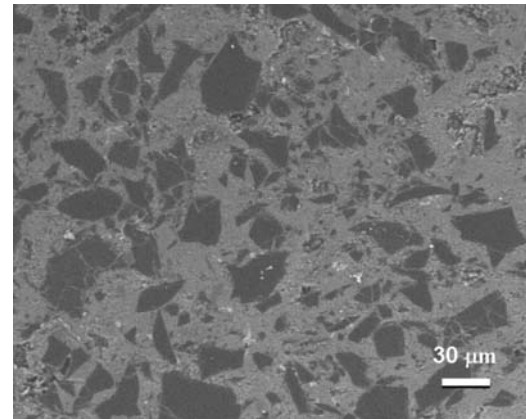

Fig. 1. SEM-image of initial composite.

The grains of B_4C are surrounded by a matrix composed of Al and reaction products. Some of the large grains are cracked due to polishing. Average size of carbide grains is 17.3 μm.

XRD-patterns [8] revealed that the main reaction product is ternary carbide Al_3BC; weaker lines correspond to reflections from aluminum carbide Al_4C_3 were observed also. The process of nucleation and growth of Al_3BC was described by Viala et al. [7]: Al_3BC phase is formed at the interface between B_4C and Al supersaturated in carbon and boron, and is aggregated in crystals surrounding the B_4C particles.
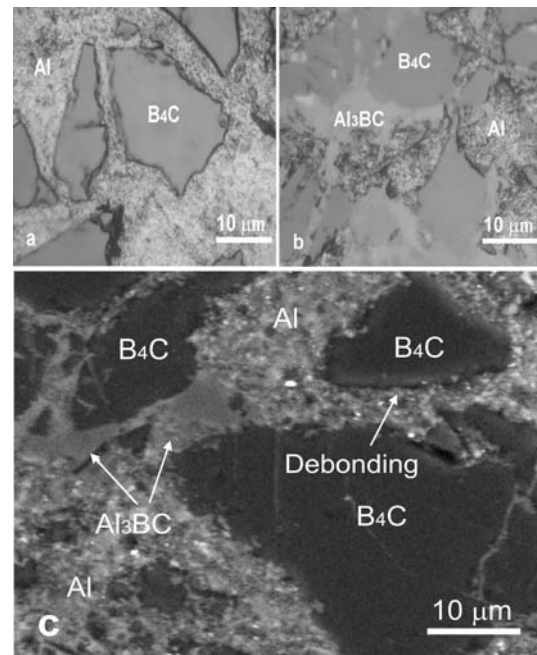

Fig. 2. OM- and SEM-images showing the absence and the presence of Al_3BC-phase around B_4C particles.

Microstructural investigations suggest that Al_3BC was formed only at some B_4C-grains in given processing conditions. As it is shown in Figs. 1 and 2, in some places Al_3BC crystals have already formed a quasi-continuous reaction zone, but mainly B_4C particles are still in direct contact with aluminum. Debonding at some Al-B_4C interfaces is also obvious (see Fig. 2).

The interfacial zone between reinforcement and matrix is an essential part of composites. The strength of the interface is dependent on the nature of reinforcement (added or formed in-situ), on adhesion, and extent of the interfacial chemical reaction. It should be noted also that adhesion work between Al and B_4C at 700°C is relatively low (about 320 MJ/m^2) [2].

3.2 Characterization of post-shocked composites

SEM micrographs of the samples cyclically (up to 20 cycles) quenched in water from 300°C and 400°C did not reveal of any large crack formation. It can be concluded that such thermal loading was not able to generate any catastrophic failure or to propagate the pre-existing flaws.

The formation of few large cracks in surface area was caused by quenching of samples from 500°C and 600°C. Some little cracks formed were stopped in the pools of aluminum or at B_4C-grains. Debonding of some individual particles was caused also.

In following are investigated the paths of large cracks formed as a result of thermal loading and possible mechanisms of their propagation are proposed. It must be noted that all large cracks were initiated from diamond pyramid indents or from surface macrodefects such as voids.

Fig. 3 shows three different response behaviors to indentation. There are three Vickers pyramid indents placed in different zones of microstructure: in a B_4C grain (Fig. 3, a), in a large pool of binder with no evidence of reaction products (Fig. 3, b), and in a zone with reaction products (Fig. 3, c).
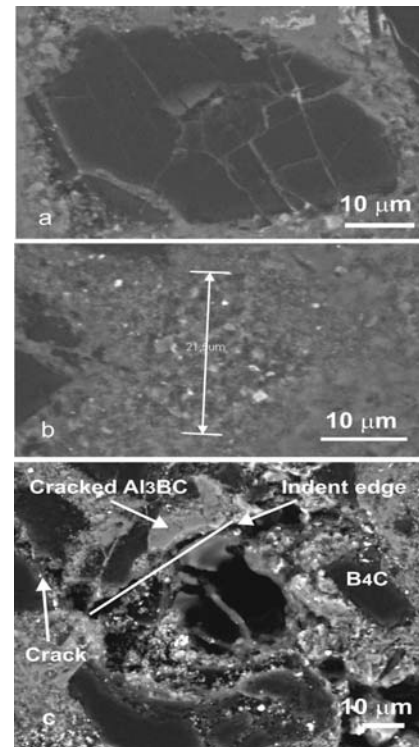

Fig. 3. Vickers indents in different zones of microstructure.

In the first two cases the crack nucleation and propagation into the matrix is suppressed by high ductility of Al. Otherwise, when the indent is placed in a region with reaction products, a crack is originated and propagated.
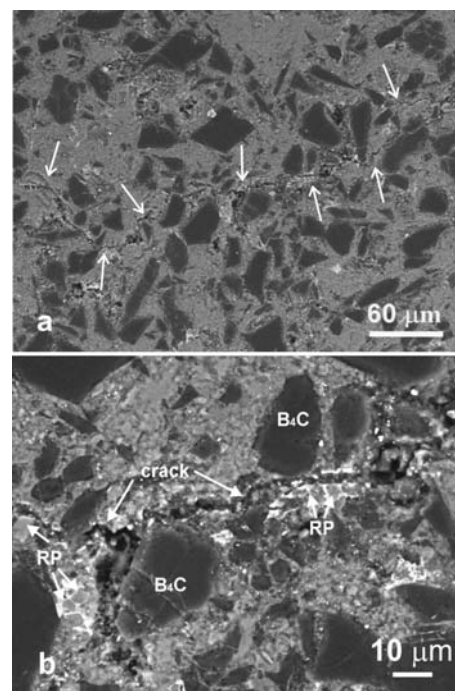

Fig. 4. Thermal crack path (600°C, 10 cycles); RP – reaction products.

In Fig. 4 is presented a typical path of a large crack in samples quenched from temperatures 500°C and 600°C.

Analyzing Fig. 4 it can be concluded that all fracture is going through the matrix near boron carbide grains, where the reaction product (Al$_3$BC) are formed and partly through the interface. The fraction of cracked B$_4$C particles along the crack paths is neglible. Voids formation at the Al-B$_4$C interface indicates the destruction of bonding.

No crack's widening was observed after 20 quenching cycles, as distinct from evident oxidation.

Based on microstructural observations, the toughening mechanism of crack deflection by boron carbide particles can be supposed, that was firstly proposed by Lange [11]. This approach means that when a crack intersects an obstacle, it is bent to some angle before it can move on. Tough boron carbide particles are expected act as obstacles.

An example of crack deflection by B$_4$C-particle is presented in Fig. 5. The stress needed to bypass the B$_4$C-particle must be greater than the fracture stress of the matrix [12].

The crack path near a particle will depend on the residual stress fields in this zone resulting from CTE mismatch and phase transformation during processing.
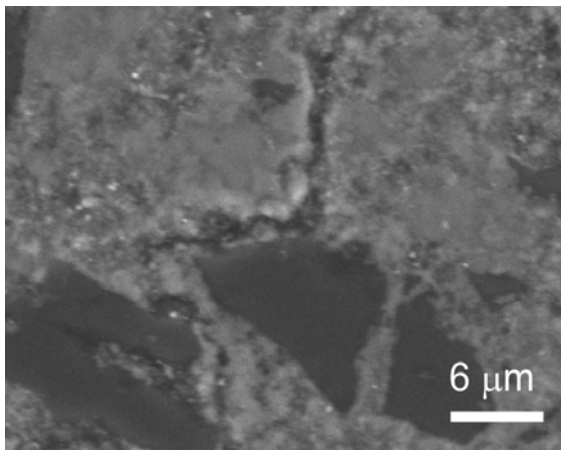

Fig. 5. Crack deflection by B$_4$C grain.

Table 1 gives an overview about the properties of composite's phases.

Table 1. Properties of phases; E – Young's modulus, G – shear modulus, B – bulk modulus, α – CTE, ν – Poisson's ratio.

	Al	B$_4$C	Al$_3$BC
E, GPa	69 [9]	460[2]	
G, GPa	27 [9]	180[2]	129 [10]
B, GPa	75 [9]	245[2]	175 [10]
α×10^{-6},K^{-1}	23.6[9]	5.0 [2]	
ν	0.35[9]	0.16[2]	

The spherical shell solution method based on Hashin's composite sphere assemblage [13, 14] was used to predict the thermal expansion stresses developed in composite after cooling from heat-treatment temperature. Under purely elastic conditions, after cooling from heat treatment temperature the radial stress at interface in particle and in matrix was found to be compressive (–445 MPa). At the same time the matrix undergoes to large tensile residual stress when cooled from heat-treatment temperature [14, 15].

While the spherical particle was concerned, the predicted equivalent stress distributes uniformly. Actually the particles are angular. Therefore there is a stress concentration in the pointed particle corner. With decreasing pointed corner degree of the particle, this concentration increases rapidly and becomes more and more intense [16].

The misfit strain induced by CTE difference can be accommodated in different ways: elastically, by localized plastic deformation of the matrix or interface rupture [17].

Surey et al. [18] have found that further reheating changes the sign of the radial stress at the interface, i.e. radial stress becomes tensile. This phenomenon is a matter of great importance since in the case of weak interface and/or large radial stress it may lead to decohesion. The absolute values of radial stresses caused by CTE

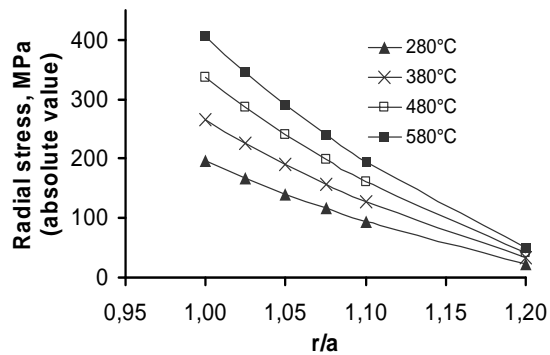mismatch in the case of purely elastic behavior are presented in Fig. 6.

Fig. 6. Predicted radial stress evolution in the matrix for different temperature drop; conditions are purely elastic. (a – radius of the particle, r – distance from the center of particle).

The results were calculated using spherical shell solution [13]. The values of stresses are expected to be high enough to cause the plastic deformation of the matrix. No cooling rate effect and influence of formation of reaction product were taken into account.

The effect of cooling rate on residual stress in composites with different volume fraction of ceramic was studied by Ho and Saigal [19]. They found that the composites subjected to a higher cooling rate experience higher residual stresses in the matrix.

Further heating of composite cause reducing of residual tensile stresses in matrix and may generate the compressive stresses, which magnitude is limited due to viscoplastic relaxation [15]. The following cooling cause the tensile stresses build up again. Due to high volume fraction and angular shape of boron carbide particles the accumulation of tensile stresses may reach high values exceeding the yield stress of Al [15]. The large stresses interacting with neighbouring defects of the microstructure are able to nucleate the crack.

SEM-micrograph (Fig. 7) of the fracture surface of a non-shocked sample subjected to three-point bending indicates that the crack front is propagated partly through the interface of B_4C-particles and partly through the matrix.
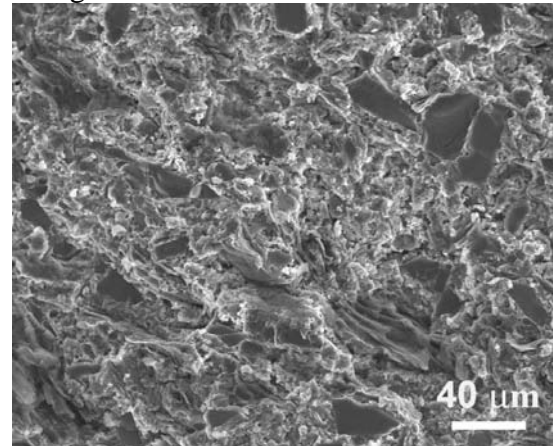

Fig. 7. Fracture surface of a non-shocked sample.

The overwhelming majority of B_4C-particles visible in fracture surface are located flatways. It can be proposed that there is a weak interface between large surfaces of B_4C and binder, caused by poor wettability of boron carbide by the liquid aluminum under present processing conditions [2]. There is also evidence of non-planar crack front that confirms above presupposed toughening mechanism of crack deflection by B_4C-particles.

4. CONCLUSION

Al/B_4C composites with low volume of reaction products are expected to be sustainable to low cycle thermal quenching up to 400°C.

The thermal shock failure is dominated by one or few major cracks. In a discontinuously reinforced matrix the rate of fracture extension is reduced by crack deflection. The crack initiation sites are mainly located in regions with higher reaction product volume. Thus, the reaction products are expected to act as embrittling species. The interface reaction is supposed to have harmful effects on the crack growth resistance of the composite.

Fractography shows the interface failures at large surfaces of B_4C-grains and evidence of non-planar crack, suggesting the poor adhesion at large surfaces of B_4C.

The uniformity, volume fraction, aspect ratio, shape and spacing of the B_4C particles are expected to have a strong effect on thermal stress distributions and thermal damage of particulate composite. Additional experimental approaches are needed to validate the predicted radial stresses, to clarify the actual stress distributions and to take into account the cooling rate effect and the influence of interface reaction.

5. REFERENCES

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