INFLUENCE OF BINDER COMPOSITION AND MICROHARDNESS ON WEAR PROPERTIES OF LIGHT WEIGHT COMPOSITES

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Abstract
Self-Propagating High Temperature Synthesis (SHS) method was used to produce the light weight composites on boron carbide and boron nitride base by liquid condition of aluminum alloy binder. SHS method for recycling of composites with disintegration and attrition milling of powders was used too. Special wear tests in unlubricated sliding friction condition against steel, friction coefficient and hydroerosion in solution were performed. The wear mechanism, wear rate and the wear loss in solution during testing of different composites and phase’s microhardness of composites were investigated and results are compared.

Keywords: SHS, wear, friction, light weight composites, phase microhardness

INTRODUCTION
The light weight metal-matrix composites (MMCs) have been developed to provide engineering materials with the combined properties of cermets, ceramics and metals [1]. These composites usually manufactured on aluminum and boron carbide base. The composites typically have greater toughness than pure or monolithic ceramics and greater hardness than the metal of binder. The dry sliding wear rate and friction coefficient of these light weight composites [2], and tungsten and titanium carbides based cermets were comparable. The tribofilm formation on boron carbide in sliding wear depending on load, sliding speed and relative humidity were particularly investigated [3, 4]. The good abrasive resistance and high thermal shock resistance of low density ceramic matrix composites (CMC) for advanced friction system of emergency and cars brake [1, 5] were studied. The effects of temperature on wear resistance of metals reinforced with ceramic particles were studied [6].

The powder metallurgical industry is permanently expanding the application of its near net shape manufacturing technology into new and challenging fields. At present time a many compounds has been produced by SHS-process, for example, intermetallics, carbides, borides, nitrides, composites and other materials. The relation between microstructure and properties of materials has always been an important task for materials science.

The mechanism of combustion in simple and complex carbide systems in the presence of halogen-containing organic were studied [7]. They show that the halogenides of metals, which form during combustion strongly, intensify process of mass transfer and promote proceeding of carbidezation at rather low temperatures, including the low-exothermic and hard burning systems. In result the condition of chemical activation are summarized. Although many studies [2,5,7-10] have been dedicated to the investigation of SHS-process, its parameters and compounds forming are still not well understood.
The compositions phase formation as function at temperature was studied [8-10]. They show that the ignition seems to take place by a solid-solid reaction, but the eutectic proximity leads to solid-liquid interaction as soon as the explosion starts. For insulating properties evaluation the optimized materials were subjected to high temperature heat treatments.

The binder phase of composites [9] was reinforced with a variety of light weight ceramic particles such as Si$_3$N$_4$, B$_4$C, TiB$_2$, BN and 3Al$_2$O$_3$ · 2SiO$_2$. Because the boron carbide grains do not coming wet with binder metal (Mo, Ni, Co) during sintering by powder metallurgy technology the composites or cermets manufacturing is possible by self propagating high temperature synthesis (SHS) [11] and/or vacuum plasma spray (VPS) [12] processes use. Depending on processing parameters [13, 14] in these composites the properties increase when was formed boron aluminum carbide (Al$_3$BC), boron nitride (BN), and aluminum oxide (Al$_2$O$_3$) refractory compounds. When during processing aluminum content decrease down to zero and, for example, aluminum carbide (Al$_4$C$_3$) and aluminum boron carbide (Al$_3$B$_4$C$_7$) were formed in this composite, then during next months the properties were decreased and spontaneous disintegrating of material take place.

The present investigation aims is shown of binder composition and phases microhardness influence on the wear resistant properties and friction coefficient of light composites. In study the wear properties have correlation with hardness, formed from microhardness and elasticity of binder.

**EXPERIMENTAL**

In this study the elemental powders of boron carbide (B$_4$C), boron nitride (BN), aluminum (Al) and aluminum alloy (ADS-4), as basic components were used. Starting condition of boron carbide with grain size was at maximum 74.5 μm down to 10.8 μm by mean 17.2 μm. These starting powders on B$_4$C/Al-composite base were mechanically activated by disintegration and attrition mill use. The mean grain size after attrition milling was down to 40 nm. For B$_4$C-based cermets recycling processing study the heat treatment with following disintegrating milling was use. The attrition milling and mixing (with BN, C, Al and Cu) in different compositions (in wt. %) were used.

The mixtures (for SHS-processing) were compacted in the steel impervious containers and were pacing by vibration. The SHS-process ignition temperature was about at 800 ± 20°C and increase during SHS up to 1100 ± 20°C for this powders content. The ignition time and ignition temperature depends on grain size of boron carbide [10]. The heating rate was higher for nanomeasures powders after attrition mechanical activating. The energy values don’t calculate for these compositions in this study. After SHS-process immediately the capsules were heat densified under strength 150 MPa by liquid aluminum as binder phase. From these received materials the specimens with measures of 6x12x20 mm and 5x6x19 mm were worked out and polished for mechanical and different wear testing. These specimens were heat treated in zirconium oxide at 1080°C and then reheated in titanium carbide powders at 1460°C in vacuum. These surroundings were used as catalyst.

For comparison the tungsten (WC) and titanium (TiC) carbides base cermets are used.

The specimens for testing were manufactured by different regimes and divided in three groups. In first group were specimens manufactured on boron carbide (N1), tungsten carbide (N2), titanium carbide (N3) and boron nitride (N4) base. The specimens in second group the behavior of heat treatment (5 and 6) and third group (7 and 8) the behavior of grain size on wear properties and phase’s microhardness were used. The recycling
processing of composites depending on added binder components are investigated on base of specimens (N9-N12).

The microstructures were studied by optical (Nikon CX) and scanning (JOEL JSM-840 A) microscopes. The X-ray diffractometer (D5005, Bruker AXS) with the Win-Fit computing program was used to define the grain size. The microhardness of phases was measured on microhardness tester (Micromet-2001, Buehler) by test load 10-200 g. To determine the composites mechanical properties the universal hardness tester (Zwick Z2.5/TS1S) by test load 100 N was used. The tribological test of wear resistance and friction coefficient in dry sliding condition without any lubrication were measured according ASTM-B 611-85. The linear velocity was 2.3 m/s and normal load was 150 N accordingly for all specimens by maximal test distance 8 km.

In this work, on the experimental results base, we propose that the binder compositions and binder microhardness have the main influence on tribological properties of light weight composites.

RESULTS AND DISCUSSIONS

The microstructure of cermet (N1) is shown on photo (Fig.1a). The binder is aluminum alloy and the boron aluminum carbide (Al₃BC) content between of the large boron carbide grains is small, as nanomeasures grains were formed only. These boron aluminum content increases during vacuum heat treatment (N5 and N6) in estimated temperature value at 700°C up to 1100°C accordingly. With temperature increase up to 1150°C the boron aluminum content increase up to maximum. During vacuum heat treatment the free aluminum content decreases by temperature increase. By heat treatment at 1300-1500°C the free aluminum content of binder in composite decrease down to zero. The aluminum carbide (Al₄C₃) and aluminum boron carbide (Al₈B₄C₇) were formed. The compound microstructure was changed and the hard carcass with porous was formed. The attrition milled powders base formed microstructure (Fig.1b) have not large grains. Has was shown by the X-ray testing with Win-Fit computing program use the mean grain size decrease down to 40 nm.

But not only is the grain size and heat treatment temperature a prior by microstructure and properties of composites forming. The boron nitride (BN) grains and exactly, the boron aluminum carbide (Al₃BC) during heat treatment at 1080°C in zirconium
oxide (ZrO$_2$) during 3-5 hours, with low heating rate and low cooling rate after heating at this temperatures were formed. After this processing the mechanical properties were very high. The hardness increase up to HRA = 88 kgf/mm$^2$ without embrittlement of composite. For composite N4 manufacturing the boron nitride (BN) was in addition to boron carbide powder use. This composite phase’s microhardness (Fig.2, N4) is shown. The WC- and TiC-based cermets (Fig.2, N2 and N3) microhardness of binder is higher the B$_4$C-based cermets have. In result of this the wear rate (Fig.3a, N2 and N3) is lowest. The slurry testing results in solution during 144 h (Fig.3b, specimens N2 and N3) is different. The WC-based cermet weight loss is maximal and TiC-based cermets weight loss is minimal. The B$_4$C-based cermets hydroerosion weigh loss depends on binder composition. For specimens (N1, N4, N5 and N7) without heat treatment and, consequently, which contain the raw binder the weight loss are lower. The WC-hard phase (N2) have not resistant to spirit of salt.

![Microhardness chart](image)

**Fig.2.** The effect of chemical compositions and processing features on phase’s microhardness of different cermets, tested in this study.

1. SHS-processed B$_4$C cermet, a - boron carbide, b - Al binder with Al$_3$BC nanomeasures particles, c - Al with C. 2. VK15 - 35 hardmetal, a - WC hard phase, b - hardmetal. 3. TH30B - 9 cermet, a - TiC hard phase, b - TiC based cermet. 4. BN based composite, a - BN hard phase, b - binder I, c - binder II, d-binder AlB$_2$. 5. SHS-processed and vacuum heat treated at 700°C, a - B$_4$C hard phase, b, c - Al binder with Al$_3$BC and BN particles, d - Al with C. 6. SHS-processed and vacuum heat treated at 1150°C, a - B$_4$C hard phase, b, c, d - binder with Al$_3$BC, BN and Al$_4$C$_3$ particles, e - Al with C. 7. Fine grained B$_4$C with Al 60 wt.% cermet after heat treatment at 1080°C in ZrO$_2$ during 4h and 8. after double vacuum heat treatment at 1480°C, 0.5 h in TiC powder surrounding, a - B$_4$C fine grains in binder, b, c - binder, d-new hard phase Al$_3$BC. 9 and 10 - recycling processing, disintegration and attrition milled B$_4$C cermet powder with binder content of: Al - 41.4, C - 4.3, Cu - 3.6 and WC - NiCo (wt.%), 9 - after heat treatment at 1080°C in ZrO$_2$, 4h and 10 - after vacuum heat treatment in TiC powder at 1460°C, 0.5 h, a-hard phase, b, c - binder, d - new hard phase. 11 and 12 - recycling processing of spontaneous disintegrated and attrition milled B$_4$C cermet powder with Al - 50wt.%, 11 - heat treated at 1080°C and 12-double at 1470°C, a - hard phase, b, c - binder of aluminum borate base, d - new hard phase.

The recycling processing of boron carbide based composites were studied (specimens N9 – N12) accordingly. For spontaneous disintegration the heat treatment process at 1500°C temperature was use. As were shown [5] by heat treatment (in oxidizing surrounding) at 800°C and higher the resultant cermets contains boron carbide, aluminum, a
phase denominates as AlB$_2$, α-AlB$_{12}$, AlB$_{12}$C$_2$ and Al$_4$C$_3$ may be formed. These phases are believed to adversely affect the properties of the resultant cermets. Depending on the quality of these phase denominates of cermets the spontaneous disintegration process to take place [10]. By this the cermets spontaneous disintegration time maybe different. From this spontaneous disintegrated cermets powder with new binder metal the cermets N9-N12 were formed. After double SHS-processing these composites friction coefficient (Fig.3) by dry sliding and hardness of phases are lower by comparison of the other composites.

![Fig.3. Effect of composition chemical content and processing features on dry sliding wear rate and friction coefficient (a) and weight loss of hydroerosion wear of cermets (b).](image)

The fine- and nano- size composites hard phase microhardness measurement was complicated due to the small grain sizes. The microhardness of grains meeting points were measured only (Fig.1b). Depending on hard phase properties and receiving methods, the phase’s microhardness was different. After 30 minutes long vacuum heat treatment at 1080°C in a zirconium oxide powder in practical the composite microhardness was measured. After double vacuum heat treatment in titanium carbide powder the microhardness decrease. The lowest phase’s microhardness (Fig.2) has composites after recycling and vacuum heat treatment in titanium oxide powder.

These compounds of binder forming and their microhardness and mechanical properties were synergistically compared and as result the composites properties depends from these changes. The highest hardness and wear resistant properties have composites which contained the boron nitrides fine grains and has the vacuum heat treatment at 1080°C in zirconium oxide (or corundum) coarse grained powder as surrounding medium. After double vacuum heat treatment only in titanium carbide surrounding these composites has a lowest wear resistant properties (Fig.3a, b). These results of experiments showed that the mechanism of structure and properties forming are very different and depend on initial status of hard phase grains size, alloying elements compositions, heat treatment features and surrounding behavior.

Depending on phase composition and microhardness of binder of boron carbide based composites is possible to use these light weight materials, for example, in quality as friction material of advanced systems in aerospace industry, armor plates or tools for grinding etc.
CONCLUSIONS

• The given experimental results show that the binder composition and binder microhardness of composites formed on base of starting powders depending on processing features.
• The mechanical properties, phase’s microhardness and wear resistant during sliding and hydroerosion testing are maximal for composites with boron nitride hard phase. The friction coefficient was lower then other cermetes have.
• The attrition milled powder of spontaneous disintegration composites can be used as hard phase for recycling of new composites. The recycled composites wear rate do not lower then the wear rate of coarse grained composites after SHS and heat treatment. The friction coefficient of these composites is lowest.

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REFERENCES