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Short communication

Interaction of boron suboxide with compacted graphite cast iron

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Abstract

The chemical interaction of boron suboxide (B_6O) with compacted graphite cast iron (CGI) was investigated using static interaction diffusion couples between B_6O and CGI at 700 °C, 900 °C and 1100 °C for 1 h. This interaction offers the possibility to evaluate the potential of B_6O as a cutting tool. The microstructures and phase compositions of the interaction zones were investigated. At 700 °C and 900 °C the chemical interaction was minimal. However, at 1100 °C, Fe_2B and SiO_2 were formed at the interface. Hence, machining at 1100 °C is likely to result in chemical wear.

Keywords: E. Cutting tools; Boron suboxide; Microstructure; Machining

1. Introduction

A number of ferrous metals such as compacted graphite cast iron (CGI) and austempered ductile cast iron (ADI) possess very good properties in terms of strength and toughness and could find extensive use in the motor industry for the creation of high power diesel engines among other potential applications [1,2]. The drawback of such materials is the problem in machining these materials because of the high degree of wear (abrasive and chemical) [2,3].

Boron suboxide, being a superhard material comparable to cubic boron nitride, could be a potential candidate for the machining of difficult to machine ferrous metals. It has an average Vickers hardness of 45 GPa (100 g load) in single crystal form comparable to that of cubic boron nitride (H_V = 50–60 GPa) [4,5]. Recent work [3,6–8] has developed a method of producing dense B_6O materials without high pressure. The use of oxide or boride additives allows the densification by hot pressing [3,6,7] or FAST/SPS [8] and improves the fracture toughness (3.5–4 MPa $m^{1/2}$) in comparison to pure B_6O materials (≈2 MPa $m^{1/2}$). The aforementioned properties make the potential use of these materials as cutting tools a reasonable proposition.

Since the performance and characteristics of cutting tools depend on hardness, fracture toughness and chemical compatibility with work piece materials, an understanding of the thermochemical stability and behaviour of B_6O based materials both individually and in contact with the workpiece material is relevant to machining.

Static interaction diffusion couple experiments were used to investigate the interaction between various tools (cBN WC, PCD, SiAlON) and workpiece metals at expected machining temperatures by a number of authors [9,10]. The results obtained by this type of experiment, can yield useful insights into the machining process [9]. If diffusion occurs during machining, or an interaction layer is formed, these phenomena are most likely reproduced in a static interaction couple experiment. The much longer contact times permit a more accurate analysis and therefore a better understanding of the chemical wear process [9,10].

Therefore, this paper aims to investigate the interaction between B_6O and compacted graphite iron and to explore the potential of B_6O based materials as industrial cutting tools.

2. Experimental procedure

Pure B_6O samples, 18 mm in diameter and 3–4 mm in thickness were hot pressed (HP20 Thermal Technology) in

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Table 1
Detected phases by XRD on either side of the diffusion couple in order of abundance.

Temperature (°C)	Phases on B_6O side	Phases on Fe side	Thickness of the interaction zone (μm)
700	B_6O, C, FeO, SiO_2	Fe, SiO_2, Fe_3C	0
900	B_6O, Fe_2B, SiO_2,	Fe_2B, Fe, B_6O (pullout), SiO_2, C, Fe_3C	<10
1100	Fe_2B, SiO_2, B_6O, FeB	Fe_2B, C, Fe, FeB, B_4C	100

hBN-lined-graphite dies in argon at 1900 °C and a pressure of 50 MPa for 20 min. The samples were ground to remove adherent hBN lining and then polished using diamond slurry.

Compacted graphite cast iron (CGI) samples (composition (in wt%): (C: 3.75; Si: 2.19; Mn: 0.41; S: 0.014; Cr: 0.03; Cu: 0.86; Mg: 0.009; Sn: 0.11) of the same diameter (∼18 mm) and 3–4 mm in thickness were cut, ground and polished down to 0.05 μm diamond paste grit size.

The B_6O samples were placed between two CGI samples for the diffusion couple experiments which were performed in the hot press under an argon atmosphere at temperatures comparable to those expected during a turning operation namely 700 °C, 900 °C, 1100 °C and 1300 °C for 1 h under a load of 10 MPa in an hBN capsule.

Cross-sections of the reaction couples were polished using diamond slurry and were characterized using X-ray diffraction (PW1830; Philips; Cu Kα radiation, 2θ range: 10–80°, step size 0.02°). Microstructure observations were carried out using optical microscopy (Olympus BX41 M) and scanning electron microscopy (Philips, XL30 SERIES) with an attached EDX system.

3. Results

The XRD analysis of the hot pressed B_6O samples showed only the intended crystalline phase (B_6O). The SEM image of the polished CGI material is shown in Fig. 1. The expected ferrite and pearlite morphology along with characteristic vermicular graphite flakes are visible. The phase analysis of the CGI material shows the presence of iron, carbon and iron carbide (Fe_3C) phases. Additionally, EDS shows the presence of silicon.

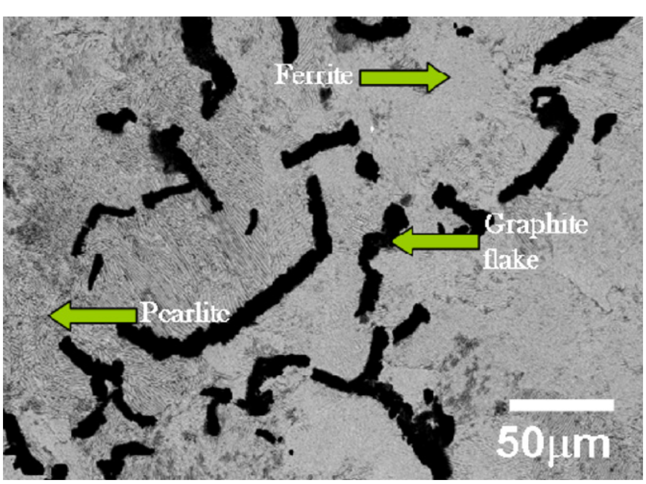

Fig. 1. Optical and SEM images of polished CGI material.

The phase analysis of the interface of the B_6O/CGI diffusion couple has been summarised in Table 1. Table 1 shows the detected phases, in order of abundance, on either side of the interface detected by XRD. An EDS line scan across the cross-section of the reaction zone showed no interaction between B_6O and CGI at 700 °C. This is in agreement with the fact that the couples fall apart after removal from hBN crucible. However, some small white spotted regions which were clearly visible on the B_6O surface were observed. These white spots were found to be rich in iron and carbon. The EDS analysis of the B_6O surface area (in general) shows that B, O, Si, Fe and C are present on the surface of the ceramic. The adherence of some

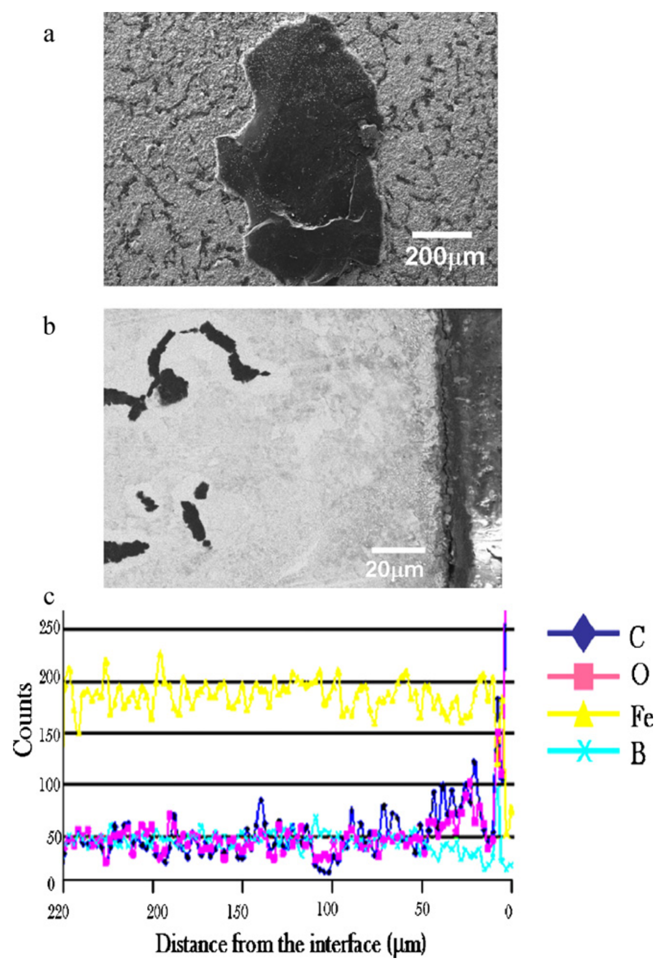

Fig. 2. SEM Micrographs of the CGI surface and the cross-section after the diffusion couple experiment at 900 °C. (a) Surface with a adherent piece B_6O, (b) cross-section and (c) corresponding line scan.

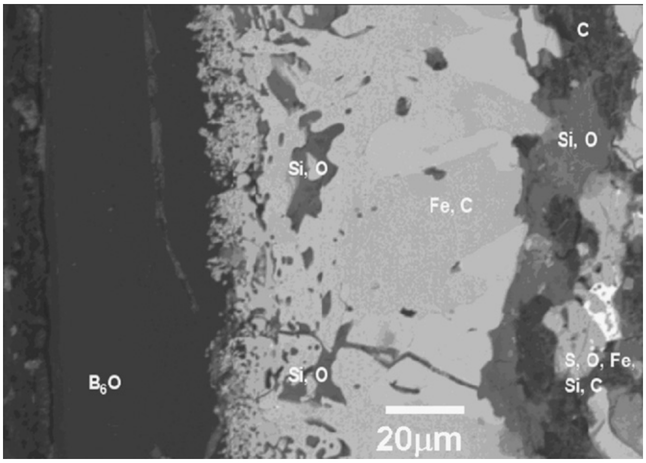

Fig. 3. SEM cross-section of the 1100 °C B_6O/CGI couple. Elemental abundance is indicated in regions scanned by EDS.

material from the CGI half of the diffusion couple indicates that some chemical interactions between the tool and workpiece materials start to occur at 700 °C. The appearance of the CGI surface was remarkably unchanged from the starting microstructure and composition.

The interaction at 900 °C resulted in some pullout of the B_6O material which adhered to the CGI pills (Fig. 2). The XRD scans of the separated diffusion couples reveal an increase in the amount of iron products (Fe, Fe_2B and Fe_3C) present on the B_6O surface (also these couples fall apart after removing the load) demonstrating that still limited reactions taking place at 900 °C. Fe_2B was by far the most significant product on the CGI side. Silicon dioxide was also detected, due to the very active silicon diffusion in the metal even at these temperatures. The surface of the CGI changed in that it appeared more rippled and featured than that of the annealed at 700 °C couple. The CGI side of the couple exhibited a SiO_2 rich region with a thickness of 10–20 μm (see Fig. 2). The B_6O side of the couple did not display any noticeable interface, aside from the pulled out B_6O that remained attached at the surface of the iron as shown in Fig. 2a.

The diffusion couple at 1100 °C displayed a marked change as a result of the heat treatment. The couple remained intact after removal from the furnace. It was difficult to separate the fused pills (B_6O and CGI) at the interface. Both surfaces after fracture appeared almost indistinguishable from one another under light microscopy. The graphite flakes of the CGI seemed to disappear almost completely in the near interfacial region. The separated CGI half of the couple showed borides (Fe_2B and FeB), with some carbon, iron and traces of B_4C. At the surface of the B_6O half of the couple, the dominant phase was clearly Fe_2B, with some carbon adhered to the B_6O.

Fig. 3 shows an SEM image of the cross-section of the interaction regions resulting from the diffusion couple

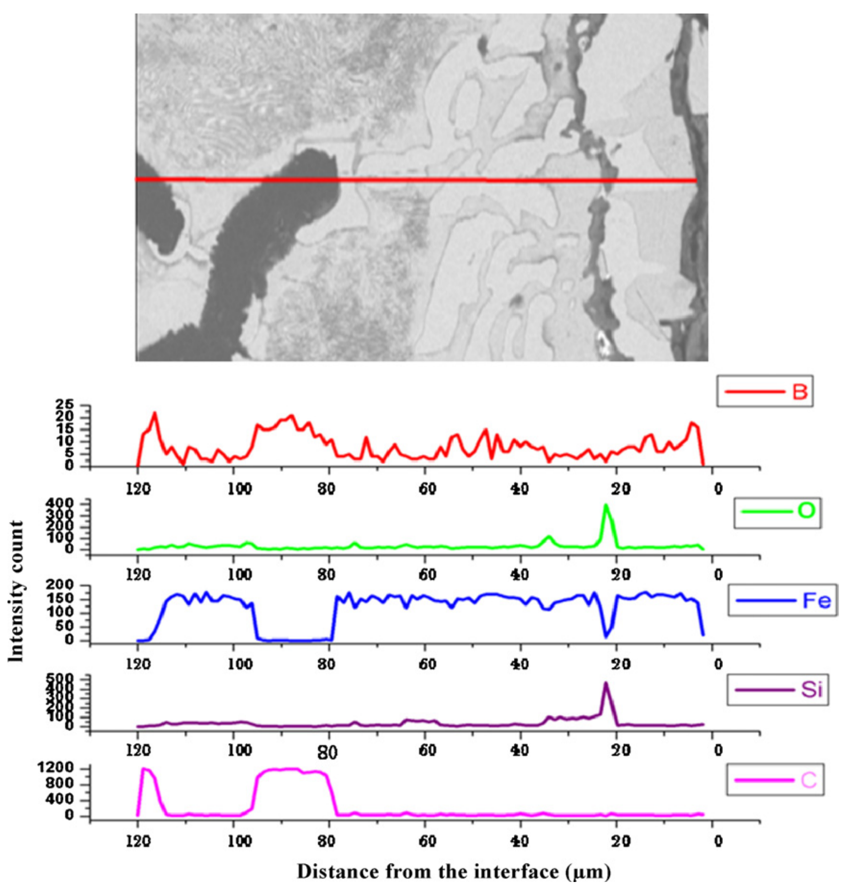

Fig. 4. Line scan of the 1100 °C diffusion couple on the CGI side showing regions and features of interest. The B_6O half of the couple was present on the right-hand side of this image.

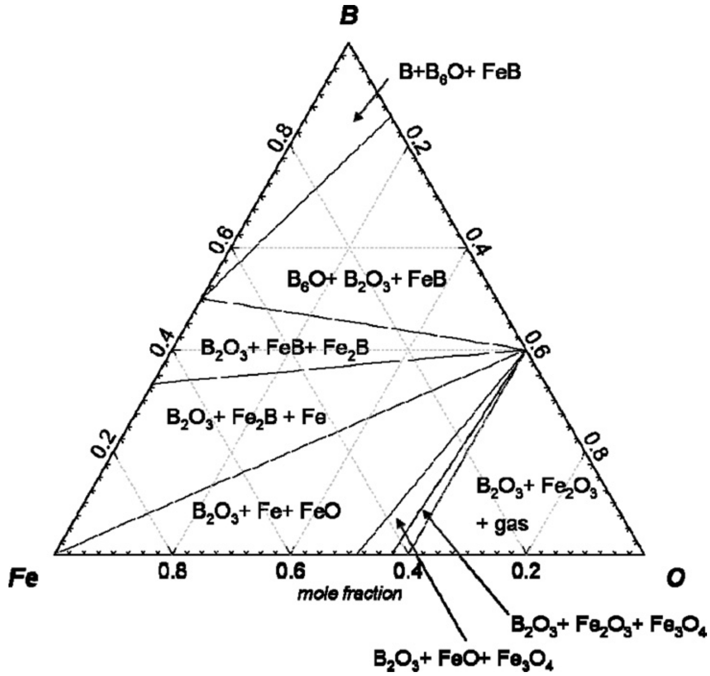

Fig. 5. Fe–O–B ternary phase diagram between 700 and 1100 °C.

performed at 1100 °C. The thickness of the interaction zone was about 100 μm. The B_6O appears to have reacted with the CGI material and distinct regions can be observed near the interface. The adherent piece of ceramic shows only boron and oxygen corresponding to a region of pure B_6O. The lighter grey region shows almost pure iron with a small amount of carbon. The dark regions at the interface correspond to SiO_2. Close to the CGI side, remaining carbon precipitations are also observed (Fig. 3). Fig. 4 shows the EDS line scans performed on either side of the interface. The most significant changes occur on the CGI side of the couple. The iron and carbon concentrations remain fairly constant (other than inside the graphite flakes). The boron content in the iron is at the detection limit (in areas with carbon some B is shown in the line scan. It is most likely caused by the non-exact separation of the EDX peaks of B and C during the measurement). The most significant observation is the formation of silicon dioxide layers around 20 μm away from the interface which was clearly visible as a band parallel to the interface (see also Fig. 3). The B_6O side of the couple showed only nearly constant amount of B and O.

4. Discussion

The B_6O/CGI diffusion couples showed very little interaction after 1 h at 700 °C and 900 °C under an argon atmosphere. The couples separated with very little pullout of each of the partners. It can therefore be concluded that the temperatures were too low to cause significant interaction other than the adherence of the CGI material around porous regions of the B_6O. This is also supported by the XRD data and EDS measurements.

The Fe–O–B ternary phase diagram generated using Factsage^{TM} 5.4.1 version and the B_6O data from [6] is shown in Fig. 5. The phase diagram does not change in the temperature range between 700 and 1100 °C. The only Fe containing phase that is in equilibrium with B_6O is FeB. Fe_2B and B_2O_3 are in equilibrium with iron. An interface consisting of boride layers would therefore be expected. In the experiment, only Fe_2B was formed at 900 °C, and aside from FeB_2, only small amounts of FeB were detected at 1100 °C. This indicates the formation of a protecting layer which strongly reduces the diffusion of B especially and as a consequence prevents the further interaction at 700 °C and 900 °C. In equilibrium B_2O_3 (liquid) formation is also expected in small amounts. If Si (dissolved in the CGI material) is taken into account, the formation of SiO_2 is more likely than B_2O_3 formation predicted by the B–Fe–O phase diagram. B_2O_3 formation would be more pronounced under, at least, partial oxidation conditions.

The adherence of some B_6O pieces on the Fe surface indicate that the local interaction with the workpiece that took place at 900 °C is slightly more intense than at 700 °C which is most likely caused by the higher mobility of the Si in the Fe and the initial formation of some glassy phases containing SiO_2 and B_2O_3. The EDS line scan shows a very thin surface layer of segregated Si and oxygen at the surface of the CGI (Fig. 2) supporting this conclusion. The XRD analysis of the separated couple showed Fe_2B at the interface thus also indicating the start of the chemical interaction.

Interaction at 1100 °C was clearly evident. The EDS line scan of the CGI side of the diffusion couple at 1100 °C showed some changes in composition in a layer of approximately 100 μm thickness at the interface. The presence of this layer indicates that the CGI materials reacted more intensely at this temperature. This interaction is strongly connected with the mobility of the alloying elements in the CGI. Substantial silicon and oxygen segregations occurred at the interface, showing that

SiO_2 forms close to the interface on the CGI side. A formation such as this would be also expected during machining operations. The XRD analysis of the separated couple showed Fe_2B and some FeB as new phases indicating the reduced kinetic hindrance of the diffusion process by the FeB_2 layer. In the interfacial area, the graphite rich areas on the CGI surface are dissolved indicating, that graphite also interacts with the B_6O probably forming B_4C and carbon monoxide. The B_4C phase was not detected, but the presence of small amounts of B_4C in B_6O is difficult to determine. With EDS, no C concentration was observed in the B_6O. EDS analysis reveals that no Fe is present (at least at a distance of 5 μm from the interface) in the B_6O either, indicating that iron cannot easily diffuse into the B_6O and thus iron diffusion into B_6O can be expected to be negligible during machining operations. The same is valid for silicon. Segregations of SiO_2 were found at the interface but no diffusion of SiO_2 into the B_6O material was found.

The results reveal that the interaction of B_6O with the CGI takes place only at the surface and no damage within the bulk of the B_6O occurs. The observed results are in agreement with the measurement of wetting angles of Fe on B_6O at 1200–1300 °C by Kharlamov et al.and Kirillova [11]. The Fe wets B_6O at 1300 °C ($\theta = 18^{\circ}$) but rapidly decreases if temperature decreases. At 1200 °C the wetting angle reduces to 50° indicating high interfacial energies at 700–1100 °C.

5. Conclusions

The chemical interaction between boron suboxide (B_6O) and compacted graphite iron (CGI) was studied using reaction couples, heat treated at 700 °C, 900 °C and 1100 °C for 1 h. The diffusion couple experiments showed the chemical interaction between B_6O and CGI to be minimal at 700 °C and 900 °C. At 1100 °C an interfacial zone of approximately 100 μm was observed where Fe_2B and SiO_2 were formed predominantly. The SiO_2 layer is the result of the high mobility of Si in iron. The observed dominant Fe_2B formation in the experiments is probably caused by the retardation of the diffusion of boron. This interlayer strongly reduces the interaction of B_6O and Fe at 700–900 °C. No diffusion of the components in the bulk materials was observed indicating that the interaction takes place only at the interface. The results indicate that the chemical interaction of B_6O with iron can determine the lifetime of B_6O cutting tools especially if the tip temperatures are higher than 1000 °C.

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