Diffusion Study of Alumina Gray Cast Iron Pair

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Key words: alumina, ceramics, diffusion

Specific properties of structural ceramics such as their mechanical and thermal stability permit their use in high-speed cutting processes that involve deep cuts and low wear rates. The process by which wear occurs in tribological contacts is complex and cannot be explained by one simple wear mechanism. The contribution of different mechanisms to the total wear of a component is related to the mechanical and chemical properties of both the materials and the contact interface. In this investigation, diffusion tests were carried out to determine the influence of chemical interactions between alumina composite and gray cast iron, without the interference of mechanical wear mechanisms. These tests were carried out at 1100°C using static couples. The tests did not generate a strong metal–ceramic interface, indicating that the alumina composites were chemically stable under the test conditions.

Introduction

Advances in science and industry as well as the development of harder materials have contributed towards the evolution of cutting tool technology ^[1,2]. Cutting applications such as dry cutting and high speed machining lead to temperature increases in the cutting zone, and this in turn could increase chemical interactions between the cutting tool and the workpiece ^[3]. Ceramic tools are generally used for machining under dry conditions ^[4,5].

Wear mechanisms of cutting tools can be classified as mechanical wear or chemical wear. Microstructure is an important parameter in wear studies involving ceramics because of its influence on hardness and mechanical properties ^[6]. The effects of grain size on wear behavior of ceramic cutting tools were studied by Goh et al ^[7]. They found that volume flank wear rate in the initial stages of cutting increased with increase in grain size of the insert and decrease of grain size improved the stability of the cutting edge. In the case of alumina cutting tools, the grain size affects abrasive wear. Small grains favor abrasive wear, whereas large grains favor brittle loss of the edge mass (chipping). This effect is linked to lower hardness and higher residual stress-intensity, thereby extending the cutting path ^[8]. Another important wear mechanism affected by microstructure is diffusion wear. During machining operations, the increase in temperature at the contact zone causes migration of material from the workpiece to the cutting tool, leading to a weakened cutting edge and eventual breakage of the tool ^[9]. Diffusion studies between ceramics and metals using a static couple is a method to understand diffusion wear, due to complexities of wear in machining process ^[10]. Kahlin et al ^[11] used the static couple approach, without the influence of dynamic parameters to study diffusion processes. They predicted that Si₃N₄ was not chemically stable in contact with FeO at temperatures above 1000°C.

In this investigation, static diffusion tests, without the influence of dynamic parameters, were performed using ceramic composites manufactured with two different types of commercial alumina and gray cast iron, to investigate tool - workpiece chemical compatibility. The influence of grain size caused by the second phase and specific starting characteristics on chemical stability was also studied.



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Experimental Procedure

Materials

The materials used in this study were Al_2O_3 : NbC composites. Samples of this composite were prepared by adding 1.5 volume% of NbC (H.C.Starck) to two types of commercial alumina, namely, A16 and A1000 (Alcoa). To this, 500ppm of MgO (H.C. Starck) was added as a sintering aid. The composition of the composites is shown in Table 1. The density and microstructure of these composites have been reported elsewhere ^[12].

ruble 1. Composition of the composites									
Codes	A1M	A1M5N	A6	A65N	A6M	A6M5N			
Al_2O_3	100	98.5	100	98.5	100	98.5			
NbC (%	-	1.5	-	1.5	-	1.5			
MgO	500	500	-	-	500	500			

Table 1: Composition of the composites

For the diffusion tests, samples with densities higher than 96% and with a specific composition and microstructure were chosen. The gray cast iron samples were CGI class FC-300 with a pearlite matrix, from Tupy.

Diffusion tests

To avoid the influence of surface roughness on chemical reactivity of ceramic-metal couple, the samples were ground and polished to a mirror finish. The samples were ultrasonically cleaned in ethanol for five minutes. The interaction couples were prepared as a sandwich, with the gray cast iron (GCI) chip between the two ceramic samples as shown in Fig. 1.



Fig. 1: Sandwich type interaction couple

Identification codes for the couples and the sintering temperature of the high density ceramic samples are given in table 2. Composites containing 1.5 vol% of NbC were used in all cases. The sets were first placed in a graphite matrix and then in a hot pressing furnace with graphite heating elements. The samples were pressed together at 8 MPa and heated to 1100°C at 15°C/ minute. The samples were held at this temperature for 5 hours.

Table 2: Identification of the couples and sintering temperatures.

Couple	Samples	Sintering
Α	A6 - A65N	1600°C
В	A6 - A65N	1650°C
С	A6M - A6M5N	1750°C
D	A1M - A1M5N	1650°C

After the diffusion test the samples were mounted for metallographic studies in a slow drying resin. The sandwich type couple was polished across its section to a mirror finish using a diamond suspension. The samples were examined in a scanning electron microscope. Semi-quantitative EDS analysis was carried out to determine the elemental composition. EDS measurement was made each 2 μ m until 10 μ m, and then from 10 μ m until 60 μ m in the middle of



samples. Comparison standards were made on each material used in this work, in regions $2000\mu m$ distant from interface.

Results and discussion

The ceramic samples used in this study were characterized and the results have been reported elsewhere ^[1212]. The densities of the samples used in this diffusion study were quite close and above 96%. The best sintering results were reached at 1600°C and 1650°C for the two types of commercial alumina. However, these alumina samples presented differences in their microstructure due to differences in aspects such as particle size, distribution and purity. Figure 2 shows the microstructures of the two types of alumina sintered at 1750 C. It can be seen that the grain size of A6 is smaller than that of A1, due to the characteristics of the starting materials. In the case of A1, the presence of MgO was necessary due to the high impurity content ^[12]. Other microstructure modifiers are sintering temperature and the presence of NbC. An example of the effect of these is shown in Fig. 2. With an overview of the microstructures the distinct grain size for two alumina samples sintered at the same conditions can be observed. Sample A6M5N, Fig 1(b) shows smaller grains when compared to alumina without niobium carbide, Fig. 1(a). Comparing micrographs the presence of NbC caused refinement in the microstructure because of pinning effect.



Fig. 2: SEM of alumina samples sintered at 1750°C. a) A6M, b) A6M5N

Diffusion results

The results of x-ray fluorescence analysis of the alumina powders are presented in table 3. Differences in composition between the two types of commercial alumina are evident. The higher SiO₂ and CaO content in A1000 can result in the formation of phases at the grain boundaries, affecting grain growth and diffusion ^[12,13].

Compounds	Al_2O_3	SiO ₂	MgO	Na ₂ O	CaO	SO ₃	Fe ₂ O ₃
-	_		0			-	-
A1000 [%]	99.5±0.3	0.21±0.05	0.06±0.01	0.07±0.01	0.06±0.01	0.020±0.005	0.04±0.01
A16 [%]	99.7±0.3	0.10±0.05	0.05±0.01	0.05±0.01	0.04±0.01	0.02±0.01	0.020±0.005

Table 3: Chemical composition of the two commercial alumina powders (wt%)

The chemical composition of gray cast iron was provided by the manufacturer and is given in table 4. Among the alloying elements, the Si content is of interest as this element is also present in alumina.

Table 4: Chemical composition of gray cast iron (wt%)

Element	Si	Cu	Sn	Mn	Cr	Ti	S	Р
Composition [%]	2.2-2.6	0.86	0.094	0.44	0.27	0.01	0.093	0.06



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Analysis before the diffusion testes showed that the concentration of iron and silicon in the ceramics was nearly zero. In the grey cast iron (CGI) the Si concentration was ~ 2 wt% and Al, zero. The elements that were measured were Al, Fe and Si. The other elements present in the ceramic sample and GCI were not or measured by EDS in this study. The diffusion study was conducted in three stages. In the first stage, the Fe concentration in the ceramic samples was analyzed. In the next stage the Al concentration in the GCI sample was measured. In the third stage, the Si concentration in the GCI sample was determined. The morphology across the interaction layer after 5h at 1100°C in an argon atmosphere is shown in Fig. 3. The micrographs show that there was no interlayer phase between the ceramic and the GCI, with separation of pair and presence of impregnation resin on the interface. The absence of new phases at the interface does not indicate lack of diffusion, but indicates a chemical interaction between the two materials of the couple.



Fig. 3: SEM micrographs of cross sections of interaction couple (a) -(b) couple C: A6M-A6M5N

Overall, the diffusion studies in ceramic samples show that the concentration of iron decreases with distance from the interface (Fig. 4). Differences were observed in the amount of iron in samples prepared with the two types of commercial alumina. The amount of iron in A1M and A1M5N was less than that in the samples prepared with alumina A16. In the presence of NbC in the matrix, there was no significant change in the concentration of iron. This indicated low chemical reactivity of NbC with iron. Despite the reduction in grain size of the matrix, observed in the presence of the inert additive, (Fig. 3) this did not cause any significant increase in the amount of iron in the matrix.



Fig. 4: Weight percent of iron in (a) monolithic alumina and (b) alumina composite



Determination of the aluminum concentration in iron was done depending on the ceramic sample in contact (Fig. 5). In this case also, less aluminum (< 2 wt%) was observed in iron samples in contact with samples of A1M and A1M5N, compared to that in A16 alumina based ceramics. Distinct microstructures caused differences in the extent of diffusion. All iron samples interacted with composites based on A16 alumina. There was no significant decrease in the amount of aluminum with distance from the interface, up to 60 μ m. In these samples the decrease in aluminum concentration was less for pure alumina in all cases. These samples had decrease behavior at distances closer to the interface. In composites the presence of Al in gray cast iron was slightly higher than that in the two types of pure alumina. The low sintering temperature promoted small differences in diffusion. The grain growth effect caused by increase in temperature was marked in sample A6 sintered at 1600°C and 1650°C. In composite samples A65N sintered at 1600°C and 1650°C, the temperature increase caused no major impact on diffusion due to the pinning effect caused by inert carbide particles.



Fig. 5: Weight percent of aluminum in gray cast iron in contact with (a) monolithic alumina and (b) alumina composite

The amount of silicon in iron was studied, (Fig. 5). In general, the concentration of Si did not decrease with distance from the interface. The differences in microstructure and the presence of niobium carbide did not cause significant changes in the amount of silicon. Exception to this was when iron interacted with sample A6M5N, and this probably indicates the presence of silicon at the interface. Another difference was noted in the presence of NbC. The pinning effect promoted by the carbide inclusions caused decrease in grain size. With decreasing grain size the amount of boundaries in the samples increased, thus providing more area for atomic diffusion. Interaction of gray cast iron with monolithic alumina was slightly lower than in samples with NbC. However the presence of NbC did not cause significant changes in diffusion. Further studies are needed to understand the effect of second phases upon diffusion.

The diffusion of Fe in the ceramics can be explained by microstructure effect and differences in grain boundary, surface and volume diffusion. Diffusion is a transport phenomenon where the concentration gradient is generally the driving force. In polycrystalline materials, the grain boundary diffusion rate is much higher than volume diffusion and surface diffusion is even higher. Thus, the diffusion rate is given by relation $D_{surface} > D_{grain boundary} > D_{bulk}$. In terms of activation energies, the relation is: $Ea_{surface} < Ea_{garin boundary} < Ea_{bulk}$. Materials with smaller grain size and therefore a larger relationship between grain boundary and volume tend to favor diffusion, while those with larger grains have lower rates of diffusion. This leads to better results for samples produced with coarser alumina^[14]. Different microstructures favor different wear mechanisms. While alumina with large grains favors mechanical wear, samples with refined microstructures provide greater area for diffusion wear, thus leading to higher chemical wear. In this study, it has

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been possible to verify this relationship between microstructure and chemical wear. However, a complete study of tool wear phenomena is very complex and the influence of grain size can also be affected by mechanical forces during turning.



Fig. 6:Weight percent of silicon in gray cast iron in contact with (a) monolithic alumina and (b) alumina composite

Conclusions

Static diffusion tests between alumina and gray cast iron were carried out. Differences in grain size of alumina samples caused differences in the amount of material that diffused in the ceramic and in gray cast iron. Samples with larger grain size, A1M and A1M5N, had diffusion to a lesser extent compared to all the samples produced with A16 alumina. It was possible to predict the close relationship between microstructure and diffusion as well as the relative inertness of the composite in the presence of gray cast iron. These preliminary results, based on high temperature chemical stability approach, still does not allow specification of an appropriate sample for turning gray cast iron, due to other wear mechanisms involved in metal cutting.

Acknowledgements

The authors would like to thank the financial support received from CNPq and FAPESP.

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Advanced Powder Technology VII

doi:10.4028/www.scientific.net/MSF.660-661

Diffusion Study of Alumina Gray Cast Iron Pair

doi:10.4028/www.scientific.net/MSF.660-661.739

