

Diffusion studies involving nanometric and submicrometric based alumina composites with gray cast iron

Karolina Pereira dos Santos Tonello ^a; Vânia Trombini ^b; Ana Helena de Almeida Bressiani ^c and José Carlos Bressiani ^d

IPEN Avenida Professor Lineu Prestes, 2242 – Cidade Universitária- São Paulo, SP, 05508-000, Brazil

^a kptonello@ipen.br, ^b vthernandes@ipen.br, ^c abressia@ipen.br, ^d jbressia@ipen.br

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Machining processes require tool materials with properties such as high hardness at elevated temperature, high fracture toughness and chemical stability with the workpiece. Advances in science and industry, as well as the development of harder materials have permitted cutting tool technology to evolve. In cutting processes, the contribution of different wear mechanisms to total wear is related to the mechanical and chemical properties of the two materials in contact. The high temperatures at tool-workpiece contact zones often result in diffusion of material from the workpiece to the cutting tool. Diffusion experiments were carried out to understand wear mechanisms involved at cutting edges of ceramic tools and the influence of microstructure on diffusion without the interference of mechanical wear processes. The chemical stability was analyzed from static interaction couple experiments at 1100°C with ceramic composite materials and gray cast iron. To investigate the influence of grain size on diffusion, sub-micrometric and nanometric alumina based composites with NbC as the second phase were used. These experiments showed that the influence of grain size on diffusion and the relative inertness of the composites in the presence of gray cast iron.

1. Introduction

During machining process the cutting tool is subjected to mechanical, thermal and chemical loads. The decrease of cutting tool life is a consequence of simultaneous wear mechanisms, such as abrasion, adhesion, flank wear and diffusion wear loading in tool – workpiece interface [1,2]. The increase temperature in the cutting zone may lead to an increase chemical interactions between the cutting tool and the workpiece [3,4,5]. These tribological relationships can also cause changes in resulting surface [6,7]. In these wear processes described adhesion and diffusion are particularly affected by the chemical stability of cutting tool [8,9].

Ceramic composites are used for cutting tools due to their thermal and mechanical properties. Microstructure is an important parameter in wear studies involving ceramics applications because of its influence on mechanical and chemical properties [10,11,12]. The presence of a second phase also can change the mechanical properties and chemical stability of cutting tool [13,14].

In 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 [15,16]. 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 [17, 18]. 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 [19,20]

In this investigation the chemical compatibility between composites and gray cast iron was studied by means of static diffusion tests, without the influence of dynamic parameters. The influence of grain size caused by the second phase and specific starting characteristics on chemical stability was also studied.

2. Experimental Procedure

The static diffusion tests were carried out between two types of ceramic composites and gray cast iron in pieces of 12x12x4. In this work were used composites with sub-micrometric microstructure and nanometric microstructures. These samples had densities above 96% of theoretical density. 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. Interaction couples were prepared as a sandwich type mounting, with the gray cast iron (GCI) chip between two ceramic samples. The sets were mounted in a graphite matrix and placed in a hot pressing furnace with graphite heating element. Samples were pressed together under 8 MPa. The samples were heated up to 1100°C at 15°C/minute and the holding time was 5 hours.

Alumina microcomposites and nanocomposites with NbC as second phase were chosen for diffusion testes. The metal used in this work was gray cast iron (GCI) class FC-300 with a pearlite matrix (Tupy-BR). The chemical composition of gray cast iron was provided by the manufacturer and is given in table 1.

Table 1: Nominal composition of gray cast iron (wt%)

| | Si | Cu | Sn | Mn | Cr | Ti | S | P |
|---------------------|---------|------|-------|------|------|------|-------|------|
| GCI - FC 300 | 2.2-2.6 | 0.86 | 0.094 | 0.44 | 0.27 | 0.01 | 0.093 | 0.06 |

The ceramic samples were prepared by two routes resulting in different composites:

2.1 Microcomposites

Sub-micrometric composites were prepared by adding 1.5 volume% of NbC (H.C.Starck) to a commercial alumina A16 (Alcoa). The alumina was ball milled in water for 15 hours and dried in an oven for 24h at 120 ° C. The amount of impurities was measured by x-ray fluorescence analysis of the alumina powders, given in Table 2.

Table 2: Chemical composition of the two commercial alumina powders (wt %)

| Compounds | Al ₂ O ₃ | SiO ₂ | MgO | Na ₂ O | CaO | SO ₃ | Fe ₂ O ₃ |
|----------------|--------------------------------|------------------|-----------|-------------------|-----------|-----------------|--------------------------------|
| 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 |

The composites were prepared in attritor for 6h with rotation of 400rpm and isopropyl alcohol as a liquid. For alcohol removal powders were passed in rotoevaporador to 90°C and 60 rpm, and then placed in an oven at 120 ° for 24 hours. Samples were prepared by uniaxial and isostatic pressing and sintering was carried out in a graphite resistance furnace at 1650°C under argon atmosphere and holding time of thirty minutes.

2.2 Nanocomposites

For alumina-niobium carbide powders synthesis was used a mixture of the reactants Al, Nb₂O₅ and C (carbon black), 0.8 mols of alumina (AKP-30 of Sumitomo - Japan) as diluents.



The reactive milling was performed in a SPEX 8000 shaker/mill apparatus with hardened steel vial and balls (10mm in diameter) and charge ratio of 4:1. The final compositions of alumina matrix composites samples with 5-vol% of inclusions were obtained by adding the dispersed RM products to a commercial ultra-fine alumina powder (Sumitomo, AKP53). The powder were mixed

in alcohol suspension with 0.2 w% of PABA and 0.5 w% of oleic acid in a conventional ball mill. After drying the mixtures at room temperature under flowing air, cylindrical specimens were formed by uniaxial pressing at 60 MPa and isostatic pressing at 200 MPa. The sintering was performed in a furnace with tungsten heating element at 1550°C for 120 min with heating rate of 10°C/min, under high vacuum.

After the diffusion tests the samples were mounted for metallographic studies in a slow drying resin, sliced and polished in diamond paste. The samples were examined in a scanning electron microscope. Semi quantitative EDS analysis was carried out to determine the composition and distribution of elements in ceramics and gray cast iron. Mapping was done to determine the distribution of elements along the materials.

3. Results and discussion

Aluminas used in this study had different characteristics in relation to the initial particle size and purity of raw materials. Nanocomposites were produced with high purity alumina (>99,9 %) and micro composites were produced with commercial alumina, with composition presented in Table 2. The composites had different properties, the $\text{Al}_2\text{O}_3+5\text{vol.}\% \text{ NbC}$ nanocomposite had a density of 99.1% and hardness of 17.7 GPa, while the $\text{Al}_2\text{O}_3+1.5\text{vol.}\% \text{ NbC}$ microcomposite had density of 96.8% and hardness of 15.9 GPa. The presence of SiO_2 and CaO in A16 could change the diffusion, resulting in the formation of phases during tests [21]. Morphology across the interaction layer after 5h at 1100°C in argon atmosphere is shown in Fig. 1. Micrographs presented in Fig 1 show that there was no interlayer phase between the ceramic and the gray cast iron. The difference in coefficient of thermal expansion of materials caused the separation of pair during cooling. The absence of cracks resulting from this separation in surfaces is another indication that there was no phase transformation. This absence of new phases at the interface does not indicate lack of diffusion, but indicates low chemical interaction between the materials of the couple.

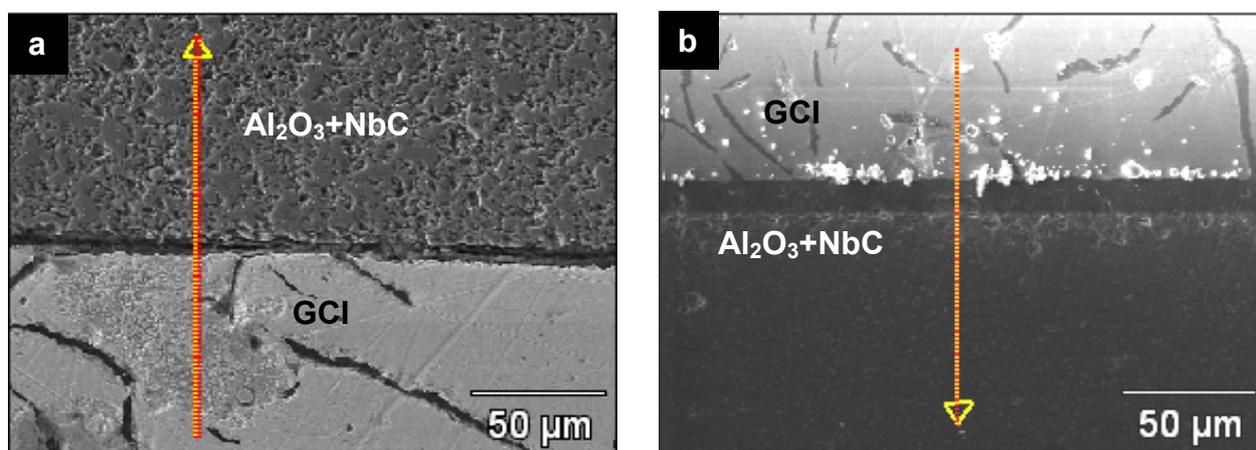


Fig. 1: SEM micrographs of cross sections of interaction couple gray cast iron with (a) micrometric composite and (b) nanocomposite

In this work the changes in chemical stability was studied as a function of changes in microstructure of materials. The study was realized by measurement of peak detection of the elements of the surface to the interior of the samples, Fig. 2. Arrows in Fig. 1 indicate the direction in which the measurements were made. While alumina with large grains increases mechanical wear, samples with refined microstructures provide greater area for diffusion wear, thus leading to higher chemical wear. The diffusion studies in ceramic samples show that no differences were observed in the amount of the element Fe in samples prepared with the two types of alumina. In it is noted that there was no significant change in concentration of these elements for any of the ceramic matrix used.

Change in the concentration of silicon and aluminum in iron was also not identified. These results indicate low chemical reactivity Al_2O_3 and NbC with iron.

Despite the reduction in grain size of the matrix, this did not cause any significant increase in the amount diffusion materials. In this case the detection of elements such as iron in the matrix of alumina and aluminum in gray cast iron can be explained by the transfer of the material during the cutting and polishing observed by the distribution pattern along the surface of the samples, details of Fig 1.

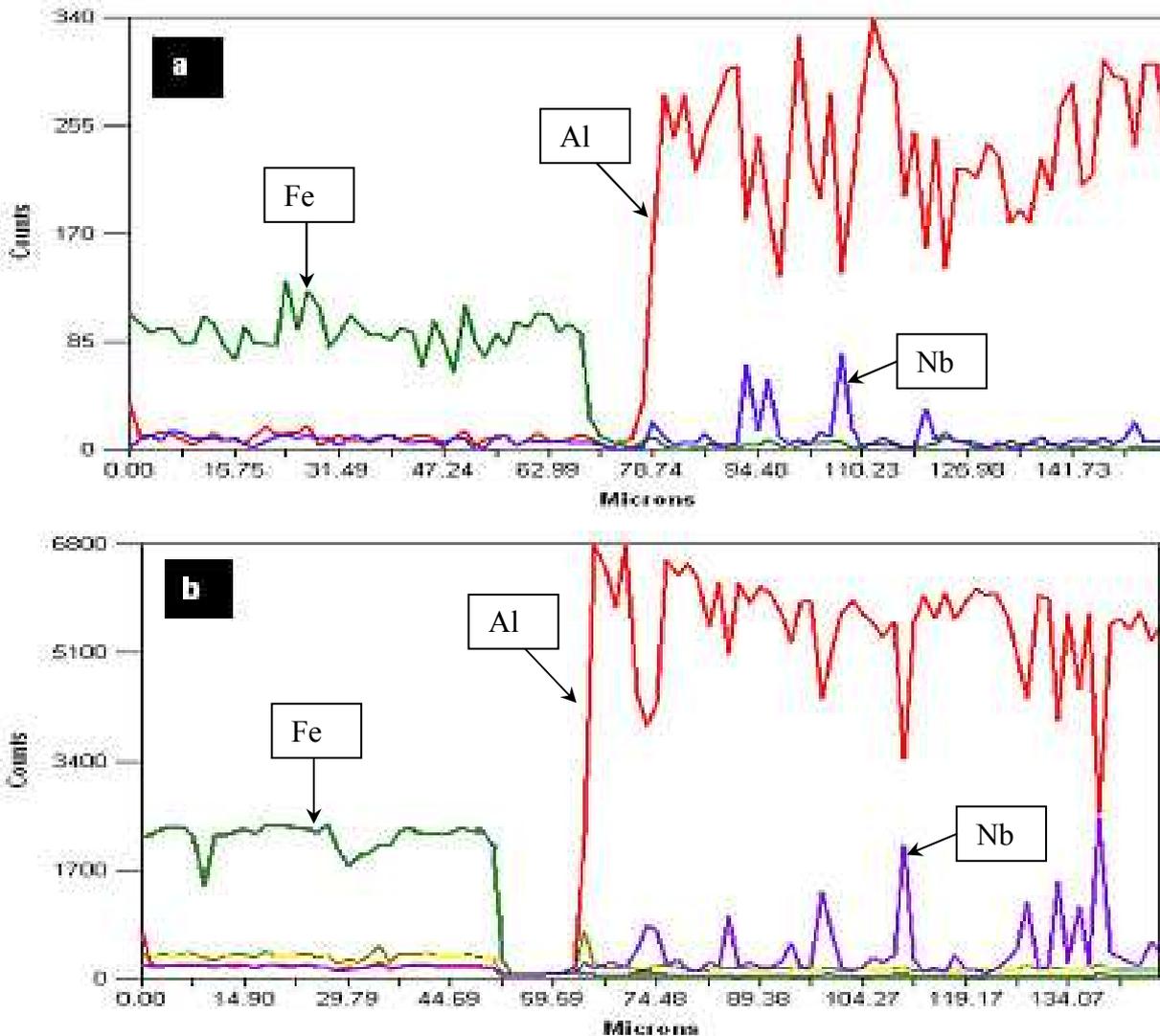


Fig. 2: detection of Fe, Al, and Nb in (a) micrometric composite (b) nanometric composite

The composites used in this study presenting differences in microstructure such as grain size and distribution that could alter diffusion due to modifications in transport phenomenon. Theoretical studies show that polycrystalline diffusion could rise with the decrease in grain size due to differences in grain boundary, surface and volume diffusion. 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 $D_{\text{surface}} > D_{\text{grain boundary}} > D_{\text{bulk}}$. In terms of activation energies, the relation is: $E_{a_{\text{surface}}} < E_{a_{\text{grain boundary}}} < E_{a_{\text{bulk}}}$. Materials with smaller grain size and therefore a larger relationship between grain boundary and volume tend to present elevated diffusion rate, while those with larger grains have lower diffusion rates. This leads to better results for samples produced with coarser alumina. The known high chemical stability of alumina did not allow the formation of phases during the diffusion studies. Another hypothesis of this study was that the presence of carbide in the alumina matrix could cause changes in chemical stability of

composites due to chemical similarity of NbC studied with other types of carbides, such as SiC and TiC, known for their higher solubility in pure iron [5, 19-21]. But in this work there was not detected phase formation or chemical interaction between NbC and GCI.

With decreasing grain size the amount of boundaries in the samples increased, thus providing more area for atomic diffusion. In this work, it has been possible to verify that there was not relationship between microstructure and chemical wear for different alumina studied. However the chemical stability in static studies can be altered by mechanical loads during machining, making necessary a complete study of the influence of microstructure in tool wear phenomena.

4. Conclusions

This study has shown the diffusion behavior of the studied composites when in contact with gray cast iron under certain conditions. Based on the degree of adherence and the concentration of materials transferred was possible to determine the relative chemical stability between ceramics and gray cast iron the materials studied when in contact at 1100 °C. These tests show that there was no phase formation between the ceramics and the CGI. The decrease in grain size or the presence of second phase in matrix did not cause an increase in the diffusion of materials for ceramics or for iron. These results indicate that materials used in this work can be used as cutting tools.

5. References

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