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TEM Study of a Hot-Pressed Al_2O_3-NbC Composite Material

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Alumina-based composites have been developed in order to improve the mechanical properties of the monolithic matrix and to replace the WC-Co material for cutting tool applications. Al_2O_3 reinforced with refractory carbides improves hardness, fracture toughness and wear resistance to values suitable for metalworking applications. Al_2O_3-NbC composites were uniaxially hot-pressed at 1650 °C in an inert atmosphere and their mechanical properties and microstructures were analyzed. Sintered density, average grain size, microhardness and fracture toughness measurements and microstructural features were evaluated. Results have shown that the mechanical properties of alumina-NbC are comparable to other carbide systems. Microstructural analysis has shown that the niobium carbide particles are mainly located at the grain boundaries of alumina grains, which is an evidence of the "pinning effect", produced by NbC particles.

Keywords: *alumina, niobium carbide, mechanical properties, microstructure*

1. Introduction

Composite ceramic materials based on alumina have been developed as a technological alternative to tungsten carbide[1-4]. Nowadays, WC-Co materials still dominate the cutting tool market, because of their superior mechanical properties[5]. Titanium nitride, tungsten carbide, mixed carbides of tungsten and titanium and specially titanium carbide have been intensively investigated as reinforcement for alumina[2,4,6-8]. Alumina-TiC has been already used in finishing operation of gray cast iron and hardened steel[9]. Moreover, the presence of these hard particles reduces the grain growth of alumina[10,11], which contributes to the mechanical performance of the composite. Reports have shown that the properties of composites depend basically on the density of the sintered materials. The densification of alumina with hard particles can be realized only in pressure-assisted sintering, by using some sintering additives as Y_2O_3, TiO_2-MnO and high sintering temperatures[6-8,12,13].

Niobium carbide can also be used to reinforce alumina, due to its high hardness (> 20 GPa), high stiffness (340 GPa) and high melting temperature (3600 °C). On the other hand, Brazil holds the main world niobium reserves, making the study of this metal strategic. Recent studies published in the literature have indicated that niobium carbide presents a good potential to be used as a reinforcing element for alumina[10,12-14]. The results have shown that the maximal reinforcing effect was obtained in alumina composites with 30 wt. (%) of niobium carbide[14]. The objective of the present work is to investigate the mechanical properties and the microstructure of a hot-pressed alumina- 30 wt. (%) NbC.

2. Experimental Procedure

The starting powder consisted of alumina APC-2011 SG (Alcoa, Brazil) and niobium carbide (Herman Starck Berlin, Germany). The raw materials present an average grain size of 2.3 µm and 1.5 µm, respectively. Alumina composites reinforced with 30 wt. (%) of NbC were mixed in a planetary ball mill during five hours. Subsequently, the homogenized mixture was uniaxial hot-pressed at 1650 °C under 80 MPa in flowing argon. The densities of hot-pressed specimens were determined using the Archimedes method. Microhardness (H_V) and fracture toughness (K_{IC}) were calculated by measuring the lengths of the cracks and the diagonal produced by the indentation method on the polished surface. Each test was repeated at least six times. X-ray diffraction analysis was carried out in order to identify the crystalline phases. Grain size distributions of alumina grains were estimated by scanning electron microscopy and image analysis using the IMAGE-C computer program (INTRONIC, Germany).

Transmission electron microscopy (TEM) was used to investigate some microstructural aspects of the sintered specimens. Samples for TEM analysis were first machined, ground and hand polished to a thickness of approximately 100 µm and then ion lilled (Gatan DuoMill 600, 6 kV, 1 mA) to electron-transparency).

3. Results and Discussion

Figure 1 shows the XRD pattern of alumina reinforced with 30 wt. (%) of NbC. X-ray analysis of the sintered composite revealed only the presence of Al_2O_3 and NbC. No evidence of niobium oxides (NbO, NbO_2, etc) or other new crystalline phases were found. Similar results have been reported for pressureless sintering of alumina-NbC composites[10,12-13]. These works have also shown that the addition of sintering additives as Y_2O_3 and TiO_2 + MnO causes a formation of new crystalline phases as $Y_3Al_5O_{12}$, Al_2TiO_5 and $AlTi_4C_2$, respectively.

Specimens sintered at 1650 °C depicted densities values from 98 to 99.5% TD and average grain size between 2.6 and 3.5 µm (Table 1). The presence of niobium carbide has reduced the density of the composite material, which can be caused probably by a pinning effect. The pinning effect was also observed in the literature for alumina reinforced with NbC[14] and SiC[11,15]. The addition of hard particles decreases the mobility of the grain boundaries during the sintering process, causing a decrease of the density and of the alumina

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average grain size. Similar observations have been also reported for other composite systems[4,6,11,15-17].

Figures 2 and 3 show the mechanical properties of the Al_2O_3-NbC composite material investigated in this work. For comparison, hardness and fracture toughness of others composite materials and tungsten carbide are also presented. The fracture toughness value of Al_2O_3-NbC composite material investigated in this work is in good agreement with those obtained for alumina reinforced with TiN, Ti(C,N)[3,9] and TiC (4.5 – 5.0 MPa.m$^{1/2}$)[2,4,6-8] and is slightly higher than Al_2O_3+(W,Ti)C[16] and Al_2O_3+Y+NbC (3.5 - 4.0 MPa.m$^{1/2}$)[12]. This result shows that the presence of niobium carbide and titanium carbide did not produce a strong barrier for the crack movement in an alumina matrix as observed in the presence of tungsten carbide

Although cemented carbides (11 MPa.m$^{1/2}$) and cemented carbide ceramic composites (7.5 – 9.0 MPa.m$^{1/2}$)[18,19] have shown a significant improvement in fracture toughness, these materials lack intrinsic advantages with respect to fast cutting speeds and thermal stability[5].

Contrary to what was observed for fracture toughness, the hardness values did not change significantly, regardless of the type of refractory particles. Alumina reinforced with 30 wt. (%) of NbC has shown hardness comparable to Al_2O_3-TiC and WC-Co, which is an evidence that TiC can be replaced by NbC without degrading the mechanical properties, indicating that this composite material has a good potential to be developed.

Further investigation is under way to study the influence of mixed carbides as NbC + WC and NbC + TiC in an alumina matrix.

TEM micrographs were used to observe the morphological features of the microstructure of the Al_2O_3-NbC composite (Figures 4 and 6). The microstructure of the composite material consisted of a

Table 1. Properties of the alumina-NbC composite material obtained in this work.

Composition	Theoretical Density (%)	Average grain size D50-(μm)	Hardness (GPa)	Fracture Toughness (MPa.m$^{1/2}$)
Alumina	99.5	3.5	16.8	2.9
Al_2O_3 + NbC	98.0	2.6	20.0	4.4

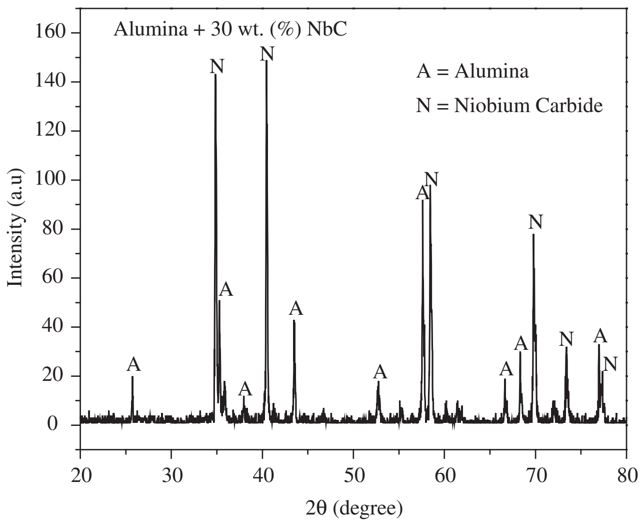


Figure 1. X-ray pattern of alumina-30 wt. (%) NbC sintered at 1650 °C.

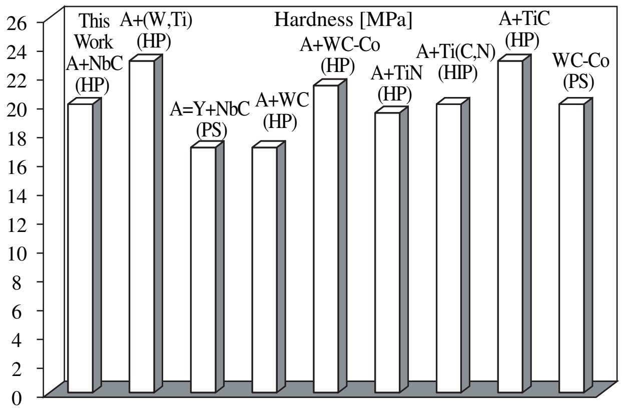


Figure 3. Comparison of hardness values from different composites materials (1-4,6,9,12,16-19). HP=Hot-pressed, PS=Pressureless sintering, HIP=hot-isostatically pressed.

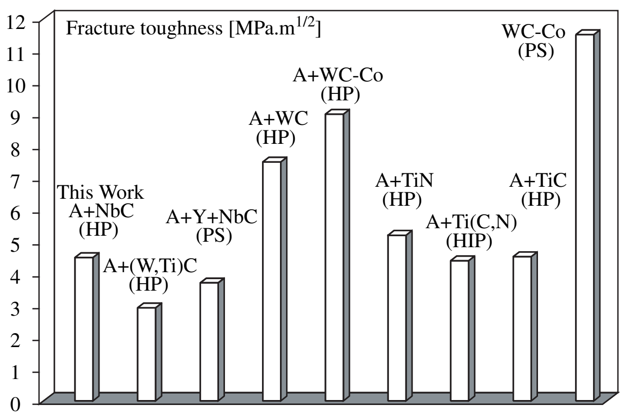


Figure 2. Comparison of the fracture toughness values from different composite materials (1-4,6,9,12,16-19). HP=Hot-pressed, PS=Pressureless sintering, HIP=hot-isostatically pressed.

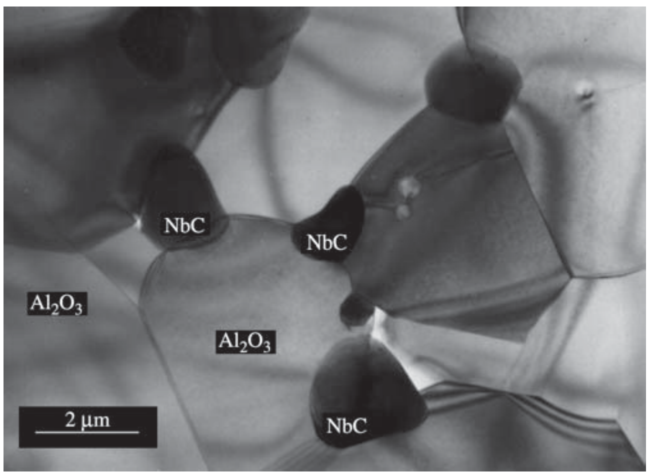
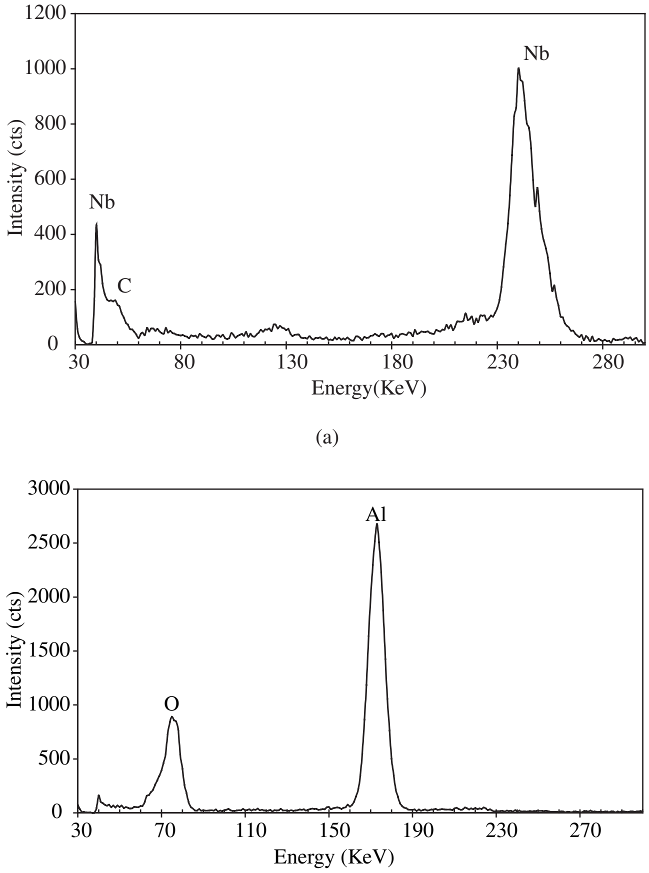


Figure 4. TEM micrographs of alumina reinforced with 30 wt. (%) of NbC.

large-grained matrix of alumina with fine-grained carbides. The material exhibited a homogeneous microstructure and the NbC grains are located along the alumina grain boundaries (Figure 4). No presence of NbC clusters was observed. The bright and dark particles were analyzed by EDS technique (Figures 5a and 5b). The data obtained by EDS confirms that the dark and bright regions consisted mainly of niobium and aluminum oxide, respectively. The presence of niobium carbide particles at grain boundaries is an evidence of the "pinning effect" produced by the addition of hard particles. Figure 6 shows the interface between niobium carbide and alumina grains. TEM analysis of these boundaries did not identify an intergranular phase. The presence of an amorphous or a new crystalline phase was not detected.



(b)

Figure 5. a) EDS analysis of the dark particles; b) EDS analysis of the bright particles.

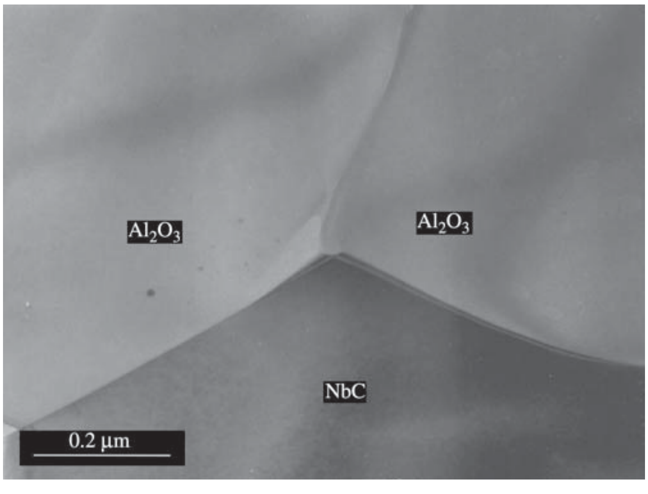


Figure 6. TEM micrograph of the alumina-NbC interface.

4. Conclusions

The results obtained in this work revealed that:

- Dense specimens of Al_2O_3- 30 wt. % NbC were obtained by hot pressing;
- Incorporation of NbC into alumina has caused a pinning effect, reducing the average grain size and the density of the monolithic matrix;
- X-ray diffraction has showed only the presence of Al_2O_3 and NbC. No other crystalline phases were found;
- Alumina-NbC composite has showed mechanical properties comparable to other carbide systems;
- TEM micrographs showed that NbC particles are preferentially located at alumina grain boundaries.

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