TRANSMISSION ELECTRON MICROSCOPY OF Al_2O_3 – NbC COMPOSITE

R. M. R. Pasotti, J.C. Bressiani, and A. H. A. Bressiani.
Instituto de Pesquisas Energéticas e Nucleares
05422-970- C. P. 11.049 – Pinheiros – São Paulo, Brasil
e-mail: abressia@net.ipen.br

INTRODUCTION

The most common carbide added to alumina is titanium carbide at a level of 20-40% and all commercial alumina –TiC cutting tools are reportedly produced by hot pressing, as reactions between Al_2O_3 and TiC at higher temperature hamper pressureless sintering (1). In the current study alumina plus niobium carbide samples were pressureless sintered in order to obtain wear resistant materials potentially useful to be used in the manufacture of cutting tools, once NbC has many excellent physicomechanical properties, such as high melting point, good electric conductivity and high hardness as TiC does.

Composites based on Al_2O_3- NbC consist of finely dispersed niobium carbide grains in an alumina matrix. By adding NbC or another transition metal carbide to a high performance alumina ceramica a composite can be produced combining the good electrical conductivity of NbC with the high strength and wear resistance of the alumina (2). In that way the major advantage of adding NbC to Al_2O_3 is that the carbide limits the grain growth of the alumina matrix and results in a higher strength and hardness.

However one of the obstacles in densifying Al_2O_3 with a transition metal carbide is the reaction between Al_2O_3 and the carbide and any trace of free carbon during sintering process at elevated temperatures (3). Yttrium oxide has been effective in suppressing reactions between Al_2O_3 and TiC during pressureless sintering avoiding the gas generation (4,5). In our system an amount of 3% weight of Y_2O_3 was added to alumina in order to improve densification.

This paper presents microstructural investigation of compact-sintered Al_2O_3-NbC ceramics as obtained from MET images. In principle, the improved properties of polycrystalline ceramic materials are manifested in their microstructure. Especially, grain size distributions, grain and phase boundaries, segregation of doping elements or phase transformation phenomena play an important role and a proper control of all these features may lead to ceramics of unusual properties which can be designed for very special applications.

EXPERIMENTAL PROCEDURE

Conventional ceramic processing techniques were used to fabricate the Al_2O_3-NbC composites with commercially available Al_2O_3 and NbC powders. The composition analised in this work is Al_2O_3 10%wt NbC and 3%wt of Y_2O_3 was added to alumina with the goal of improving densification during sintering and avoiding possible reactions. The material was pressed in a die of 12mm in diameter to obtain pellets which were sintered in a graphite-resistance-heated furnace with flowing argon at 1650°C/30min and 1750 °C/15min. After sintering , relative densities were measured by using Archimeds method and crystalline phases was identify by X-ray diffraction.

Transmission electron microscopy was used to investigate the formed phases during sinterization which occur in small quantities and for a full microstructure characterization. The thinning to electron transparency was carried out by standard techniques. The samples were cut into slices of 200 μm thickness by diamond saw and abrasively drilled into discs of 3mm in diameter. The discs were dimple grinding to about 30 μm in the center of the disc and then argon ion milling to perforation (ion beam – Ar/ 6kV) getting a thin area of 0,5 μm thickness. Finally, the samples were coated with a layer of carbon to avoid charging during TEM observation. The characterization of crystalline phases has been done by using selected area diffraction patterns (SAD) performed on JEOL JEM 200-C Microscope operated at 100kV. The software DIFAT* was used to help in the identification of the diffraction patterns.

RESULTS AND DISCUSSION

The density of the samples sintered at different temperatures was higher than 98% of theoretical density. The X-ray diffraction patterns indicated the presence of mostly Al_2O_3 , NbC and $Y_{13}Al_5O_{12}$ (YAG) for both sintering temperatures. The formation of this last phase is due to an eutetic reaction between Al_2O_3 and Y_2O_3 (6).

By TEM the distribution of the phases and the Al_2O_3 grain size, which shows to be bigger in the sample sintered at 1750 °C/15 min, were observed. Figure 1 shows a comparison of the samples microstructure according with the temperature and gives an idea about the materials grain size of the composite.

Figure 2 shows the micrograph of the NbC grain among of the Al_2O_3 matrix in the sample sintered at 1750 °C. Using the bright field and dark field images technique the NbC grain was observed and its orientation was determined by selected area diffraction. Its structure is face centred cubic (fcc) with 0.45 nm lattice parameter. The dark field image and the diffraction spots of the NbC are shown in fugure 2(a) with the direction of the incident electron beam **B**= [0 1 –1] and the indexed pattern. Figure 2(b) shows the bright field image with a diffraction pattern of the α-alumina grain located beside the NbC grain (B=[0001]). The α-Al_2O_3 has a hexagonal structure with a=0.476 nm and c=1.299 nm.

The YAG phase, which was probably crystallised from a liquid phase during cooling, was located mainly within Al_2O_3 grain triple junction. Its structure is body centred cubic (bcc) with 1.201 nm lattice parameter. Figure 3 shows the YAG phase in both bright field and corresponding dark field images presented in the sample sintered at 1650 °C. The dark field was obtained from a spot of the electron diffraction pattern, which is shown in figure 3(a) with the [3 2 -4] direction of the incident electron beam. Figure 3(b) shows de bright field image and diffraction pattern of the alumina grain located around the YAG grain.

Structure details such as grain boundary nature determination (amorphous, crystalline) and the search for any other phase have not been done yet. However samples with higher NbC concentration and without Y_2O_3 addition have been prepared to continue the microstructure investigation of these particulate composites.

---

* Software developed by G. Carpenter and L. Benkins at the Metals Technology Laboratories, CANMET, Energy Mines and Resources – Ottawa, Canada.

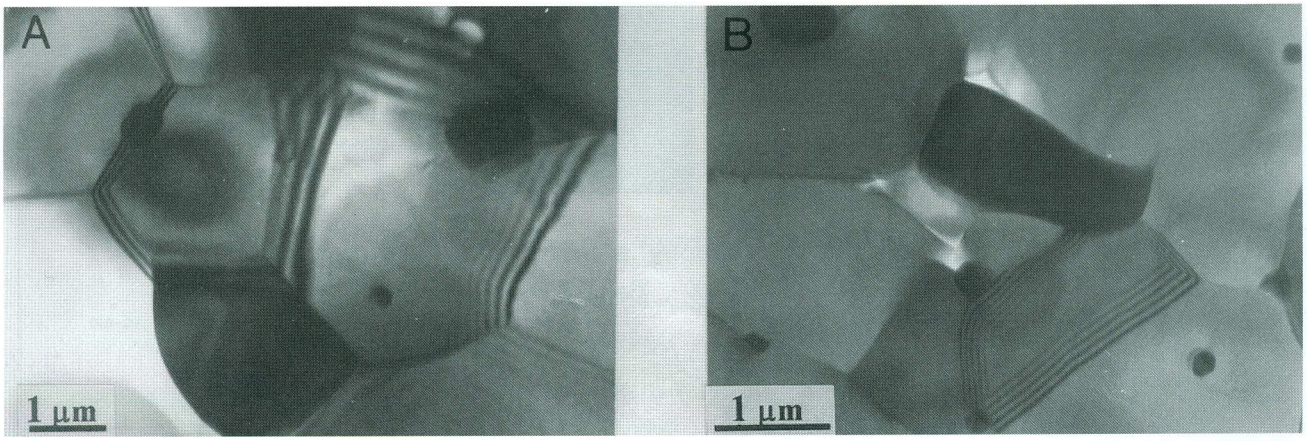

Fig. 1: TEM micrograph of the samples sintered at (a)1650°C/30min and (b) 1750 °C/15 min.

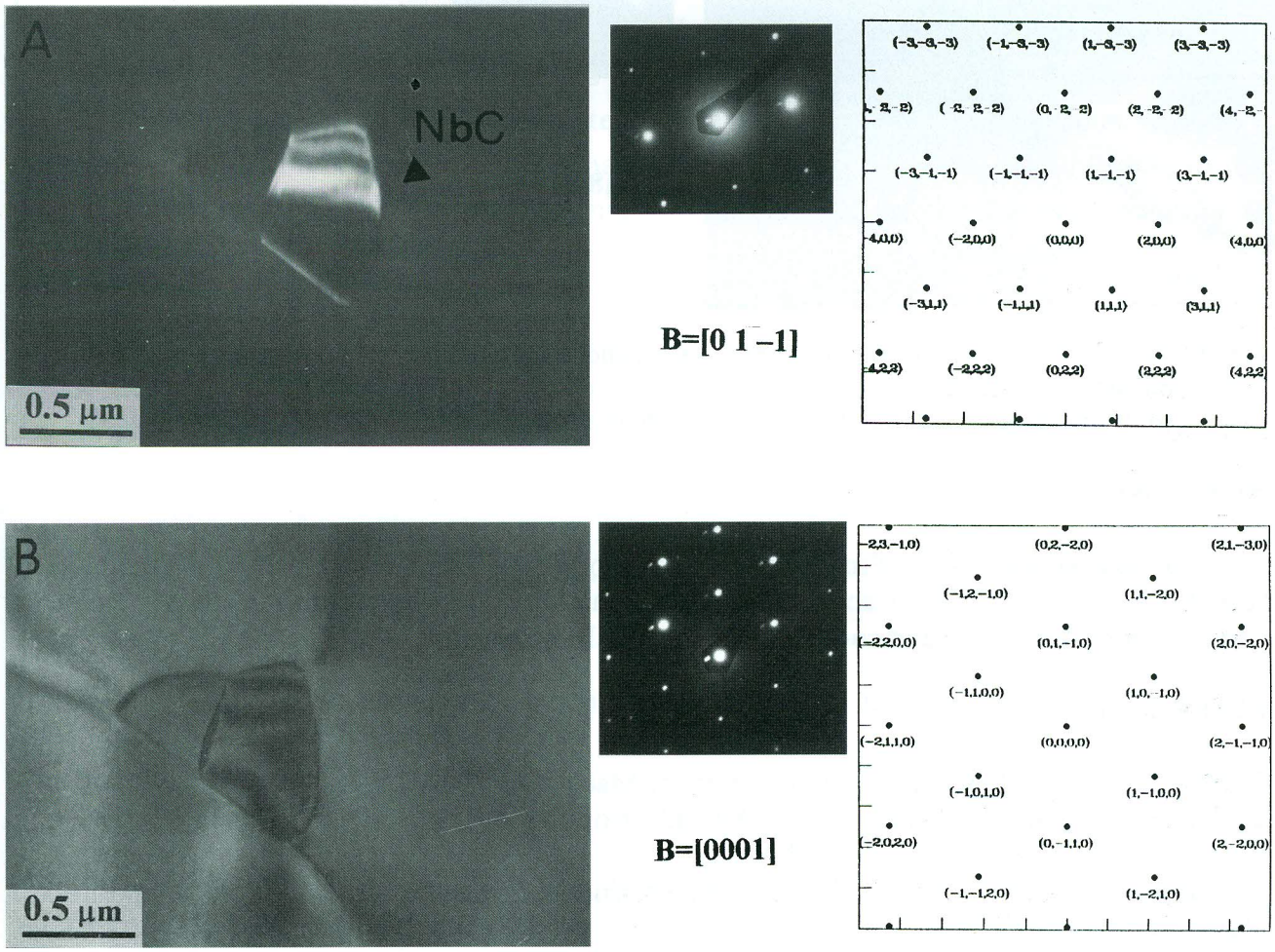

Fig. 2: (a) Dark field image of the NbC grain in the Al_2O_3 matrix. Diffraction pattern of the NbC by SAD and the indexed spots by DIFPAT software **B**=[0 1 -1].
(b) Bright field image with the diffraction pattern of Al_2O_3 . **B**=[0001].

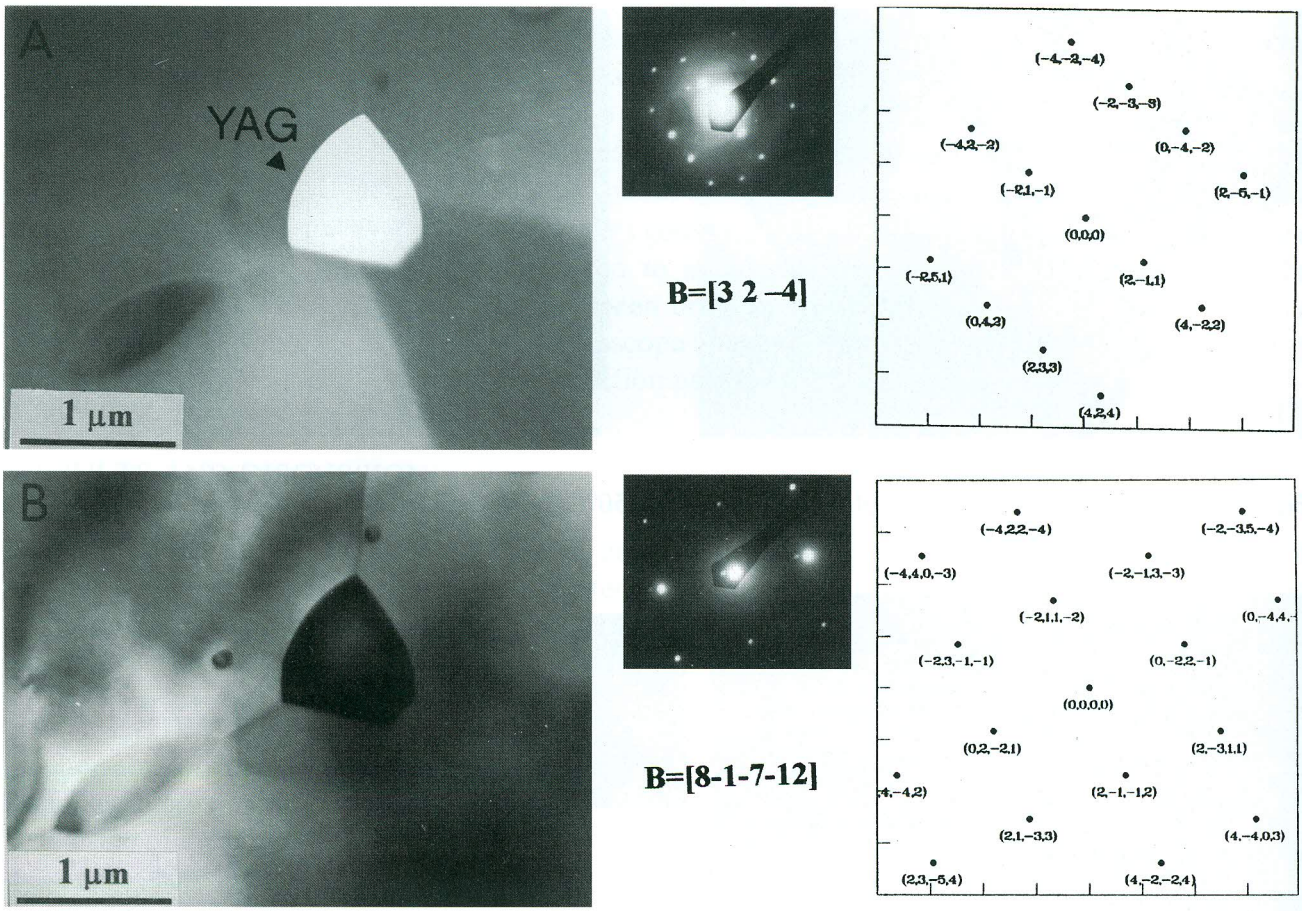

Fig. 3: (a) Dark field image of the YAG phase surrounded by alumina matrix with the correspondent diffraction pattern **B**=[3 2 -4].
(b) Bright field image with the diffraction pattern of Al_2O_3 . **B**=[8-1-7-12].

ACKNOWLEDGMENTS

Financial support of "Programa de Apoio ao Desenvolvimento Científico e Tecnlológico" (PADCT/Finep) is gratefully acknowledged. The authors are deeply indebted to Mr. Nildemar Messias for his assistance and co-operation in the TEM observations.

REFERENCES

1- Baldoni J.G., Buljan S.T. (1988) Ceramic for Machining. Am. Ceram. Soc. Bull., 67: 381-387.
2- Pippel, E., and Woltersdorf, J.(1989) Electron Microscopy of TiC Containing Alumina Ceramics. Phys. Stat. Sol. (a) 165:165-177.
3- Ishigaki T., Sato K., Moriyoshi Y.(1989) Pressureless Sintering of TiC-Al_2O_3 composites. J. Mater. Sci. Lett. 8: 678-680.
4- Chae K.W., Yeon D. (1993) Effect of Y_2O_3 Additios on the Densification of an Al_2O_3-TiC composite. J. Am. Ceram. Soc. 76:1857-60.
5- Chae K.W., Yeon D. (1995) Sintering of Al_2O_3-TiC Composite in the Presence of Liquid Phase. J. Am. Ceram. Soc. 78: 257-59.
6- Levin E.M., Robbins C. R., Mc Murdic H. F. (1964) Phase Diagrams for Ceramists. Edited by M. K. Reser. Americam Ceramic Society, Columbus, O. H.,: 122,Fig 311.