Sintering study of Al_2O_3/NbC/WC micro-nanocomposite

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Abstract

Ceramic materials based on alumina are considered excellent for produce cutting tools used to machining hard metals. However, low mechanical strength and toughness presented by these materials limit their application. Traditionally particles, such as TiC, TiN and ZrO_2, are added to the alumina matrix to improve their mechanical properties, increasing the range of applications. Recent studies have shown that the addition of particles of different sizes in alumina matrix can promote simultaneous increase in mechanical strength and tenacity. In this work sintering behavior of Al_2O_3 micro-nanocomposite containing nanometric particles of NbC and micrometric particles of WC, was studied by dilatometry using heating rate of 20°C/min up to 1800°C. The addition of carbides in alumina matrix is prejudicial to sintering causing an increase in temperature of shrinkage.

Introduction

Due to the increase in difficult-to-machine materials, and on the demand of improving efficiency during machining process and reducing costs, it is necessary to develop new ceramic cutting tools. In addition there are two factors that contribute for the research and development of ceramic cutting tools. First most of traditional hard alloy tools failed in processing new materials, and the most important thing is that the key composition, such as W, Co for high speed steel and hard alloy cutting tools, is declining worldwide so as prices rise. And second the main raw materials of ceramic cutting tools, such as Al, Si, are the most abundant resources in the earth's crust. Thus the application of advanced ceramic cutting tool in the field of high-speed machining has very broad prospects in the 21st century [1-2].

Al_2O_3-based ceramics are considered to be one of the most suitable tool materials for difficult-to-machine materials due to their high hardness, wear resistance, heat resistance and chemical stability [2].Whereas, the intrinsic drawbacks of Al_2O_3-based cutting tools, such as lower strength, lower fracture toughness and lower thermal shock resistance usually make them more susceptible to excessive chipping or fracture when machining hardened materials especially under an intermittent cutting condition, leading to a short tool life. Conventionally, Al_2O_3-based ceramic tool materials were strengthened and toughened by the addition of micro-sized particles like TiC, TiN, ZrO_2, (W, Ti)C, Ti(C, N), TiB_2, SiC or SiC whiskers [1-2]. Between several techniques for improve mechanical properties of composites addition of low percent of nanosized second phase particles within the matrix grains and on the grain boundaries promote significantly increase in flexural strength but slight increase in fracture toughness [3-8].

Researches [9] in recent years revealed that addition of two different particles with multi-size was an effective method for improving both the fracture toughness and flexural strength of Al_2O_3-

based ceramics simultaneously. By adding micro-sized WC and nano-sized TiC particles into Al_2O_3 matrix, Jun Zhao et al. [9-10] developed multi-scale Al_2O_3/WC/TiC micro- nanocomposite ceramic tool materials with significant improvement in flexural strength and fracture toughness relative to the Al_2O_3/TiC micro-composite.

In this paper will be presented results of an investigation carried out to evaluate the effect of the different amount of inclusions on sintering of Al_2O_3/WC/NbC micro-nanocomposite.

Experimental Procedure

To synthesis nanometric alumina-niobium carbide powders, a high energy ball mill and a mixture with the reactants Al-Nb_2O_5-C-Al_2O_3 were used. Commercial grade powders of aluminum (ALCOA), Nb_2O_5 (CBMM), carbon black and 0.8 mols of alumina (AKP-50 of Sumitomo - Japan) as diluent, were mixed and milled to form the composite as shown in equation (1).

$$Nb_2O_5 + 3.3\ Al + 2C + 0.8Al_2O_3 \rightarrow 2\ NbC + 2.5\ Al_2O_3 \quad (1)$$

The milling equipment consisted of a Shaker Mix type SPEX, with a vial and hardened steel milling balls. The milling conditions were those defined elsewhere [11-13]. To reduce the amount of aggregates formed during the reaction, the powders were milled further for 3 hours in high energy ball mill. The resulting powders were deagglomerated in a planetary mill for 1 hour, in an alcoholic suspension with 0.2% PABA (4-aminobenzoic acid) as deflocculant. After deagglomeration, iron contamination in the milled powders was removed by leaching with 20% HCl solution followed by repeated rinsing-decantation with water [14-15].

Based on previous works [9-10,16-17] composite mixtures with quantities of 6vol %NbC and 0,7vol%WC(ANW1) and 15vol%NbC and 2vol%WC (ANW2) were prepared using attritor mill, the suspension of the powders with ethanol, 0.2 wt% of PABA as defloculant and 0.5 wt% of oleic acid as lubricant and with ball/powder mass ratio of 2:1, was milled during 1h. The mixtures were then dried in roto-evaporator at 70ºC and 60rpm. Samples were prepared by uniaxial and isostatic pressing and sintering study was carried out in a dilatometer Netszch 402C in argon atmosphere with heating rate of 20ºC/min with holding time of 10min at 1800^{o}C. For study of microstructural evaluation of micro-nanocomposites ANW1 and ANW2 sample was sintered at 1350^{o}C/10min and 1500^{o}C/10min in a graphite resistance furnace Astro 1000, 4560,FP 20, Thermal Technology Inc. Final density was measured by Archimedes method. Microstructural examinations of fracture surfaces of sintered specimens were carried out in a Philips XL-30 and JEOL JSMG70/FEG scanning electron microscope.

Results and Discussion

Particles size, measured by Cilas and spherical equivalent diameters, obtained through the values of specific surface area, are presented in the Table 1. Comparing values of medium average particles size obtained by diffraction behavior of laser bean method and calculated from results of specific surface area it is possible to notice a considerable difference between the obtained results. This difference occurred probably due to the presence of dense agglomerates present in powder. Based in these results, the powders will be deagglomerated in the next step of processing.

Table 1. Mean Particles size measured by Cilas, Specific Surface Area and Particles size calculated using SSA of powder used in these researches.

Powder	Mean Particles size by Cilas [um]	Specific Surface Area[m^2/g]	Mean Particles size by SSA (um)
Al_2O_3 CT3000	0,4	7,4	0,27
NbC/Al_2O_3	0,075	16,7	0,047
WC	2,6	1,5	1,42

Fig. 1 present dilatometric study of alumina and micro-nanocomposites where is possible to observe that did not occurred an increase in the temperature of beginning of retraction for the micro-nanocompositos compared with pure alumina, however it is possible to observe in Fig. 1(b) that occurred an increase in temperature of maximum shrinkage rate with improvement in inclusions amount. It is also observed in Fig. 1(b) the presence of a dip in the curves of shrinkage rate for micro-nanocomposites, this phenomenon can be related to the rearrangement of particles due to the large difference in particles size of powders used in this study Table 1. It is also observed that all samples reached the end of the densification, but with a small difference in temperature, which can be confirmed with the measures of the percentage of theoretical density that was approximately 99% TD for all samples sintered at 1800°C/10min.

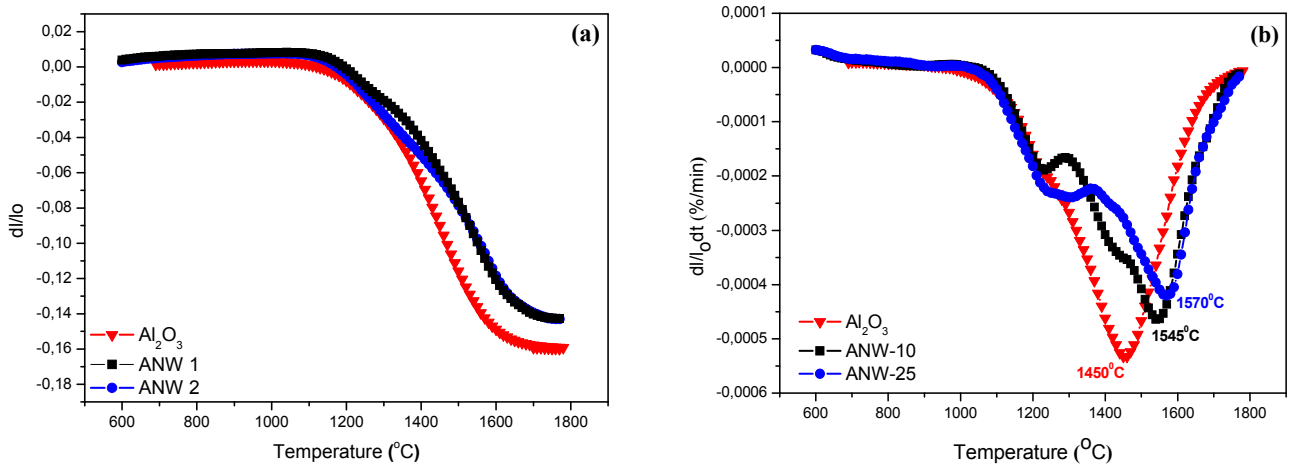

Figure 1. (a)Linear shrinkage in function of temperature and (b) Shrinkage rate in function of temperature for Alumina and micro-nanocomposites that was sintered at constant heating rate of 20ºC/min up to 1800°C.

Microstructural evolutions of ANW1 and ANW2 samples are presented in Fig. 2 and Fig. 3. In Fig 2(a), ANW1sample sintered at 1350°C/10min is possible observe the beginning of the neck formation, however in Fig. 2 (b), ANW2 sample sintered at 1350°C/10mon can be seen the presence of more round grains with smaller neck formation, in other words the increase of inclusions addition reduce diffusion and delays the sintering stages. Comparing ANW1 and ANW2 sample sintered at 1500°C it is already possible observe a increase in grain size Fig. 3(a), while for ANW2 sample Fig. 3 (b) the grain growth was inhibited by the increase of inclusions.

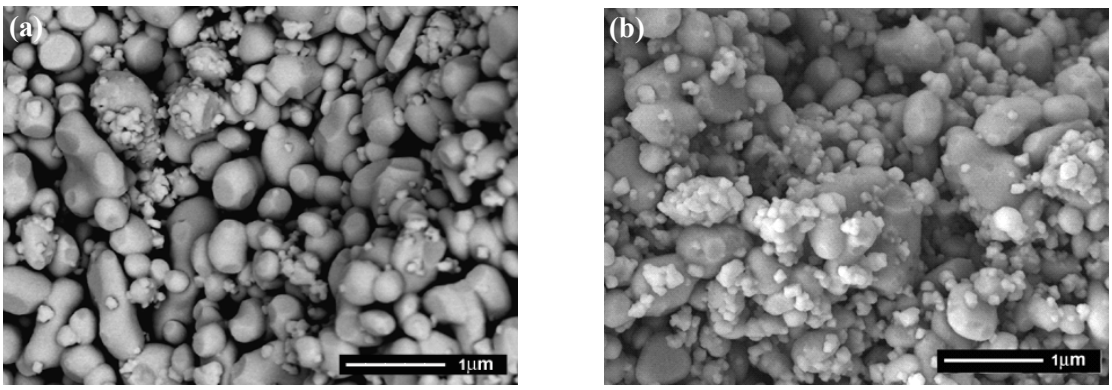

Figure 2. SEM of (a) ANW1 and (b) ANW2 sintered in a graphite furnace at constant heating rate of 20ºC/min up to 1350^{o}C/10min.

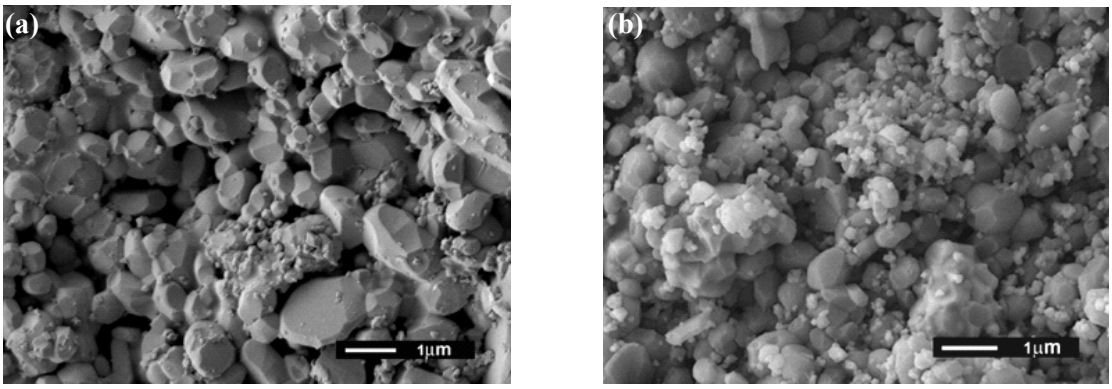

Figure 3. SEM of (a) ANW1 and (b) ANW2 sintered in a graphite furnace at constant heating rate of 20ºC/min up to 1500^{o}C/10min.

Fig. 4 (a) (b) and (c) present SEM micrographs of pure alumina, ANW1 and ANW2 samples, respectively, sintered up to 1800^{o}C/10min, where can be noted that the alumina sample without addition of inert inclusions presented a large distribution of grain size when compared with micro-nanocomposites samples, which showed more homogeneous grain sizes distribution. Can be also seen that ANW1 and ANW2 samples presented the same behavior observed in Fig. 3, sample ANW1 did not presented effective inhibition of grain growth compared with ANW2 sample. Traditionally the improvement of mechanical properties of composite materials with alumina matrix is attributed to a change in fracture type of intergranular to transgranular or by the mixture of these two types of fracture. In the case of micro-nanocomposite when comparing fracture micrographs of alumina without inclusions and micro-nanocomposites can be observed that the addition of WC and NbC particles in the alumina matrix promoted predominantly intergranular fracture, while the alumina without inclusion, due to the large distribution of particle sizes, showed a mixture of inter and transgranular fracture.

In the current study obtainment of micro-nanocomposites was made using condition defined in earlier work [16,17] that was the most efficient to obtain homogeneous mixtures of nanostructured inclusions in alumina matrix, for obtaining the micro-nanocomposites. Considering the difference between particle sizes of each phase presented in micro-nanocomposite showed in Table 1, it can be seen in Fig. 5 that the chosen method was also effective in obtaining samples with homogeneous dispersion of the different inclusions in alumina matrix.

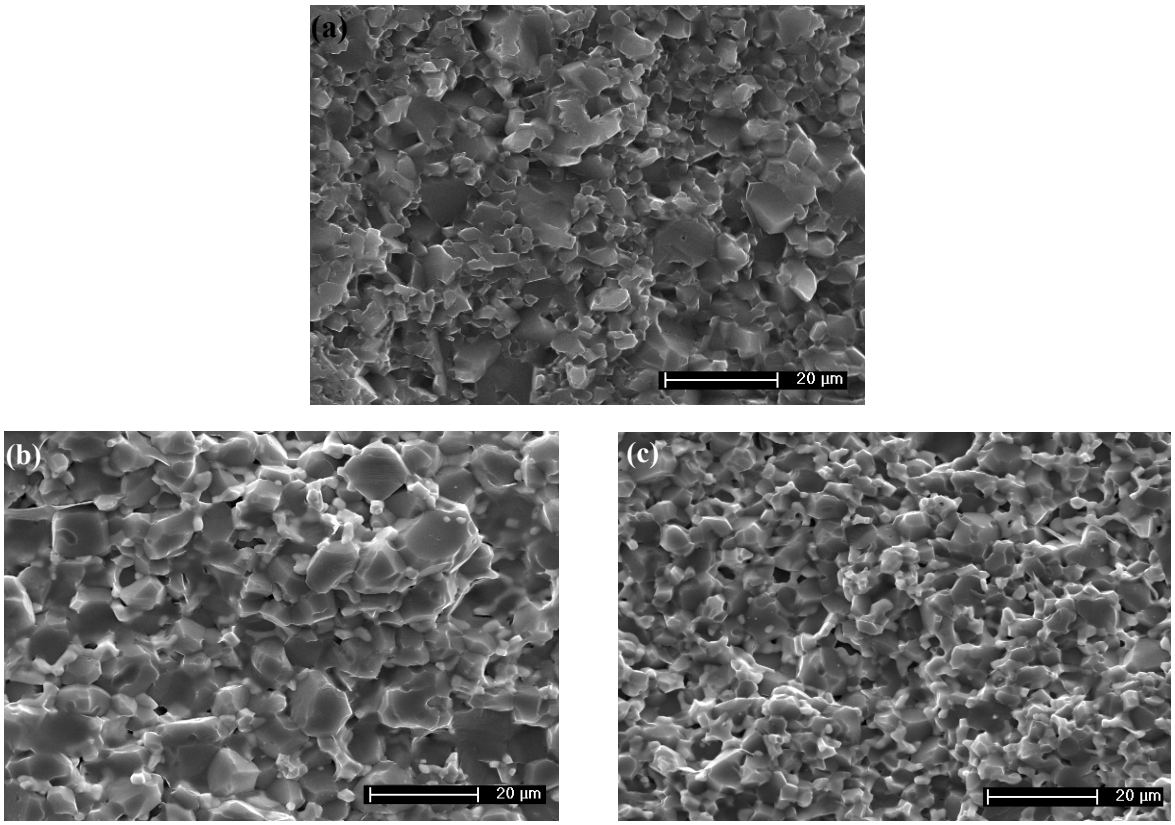

Figure 4. SEM of (a) alumina CT3000, (b) ANW1 and (c) ANW2 sintered in dilatometer at up to 1800°C/10min.

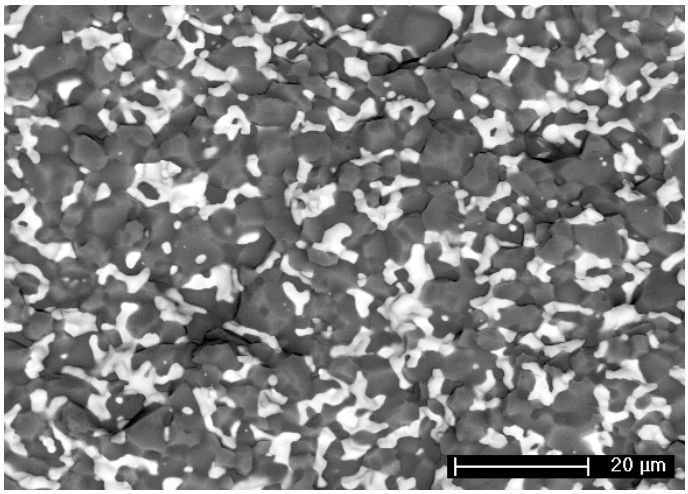

Figure 5. SEM of Alumina/NbC/WC micro-nanocomposite ANW2 sintered in dilatometer up to 1800°C/10min.

Conclusion

Sintering results indicated that the addition of even a small amount of nanometric NbC and WC inclusions in an alumina matrix decreased the sinterability of alumina, being necessary an increase in sintering temperature for obtaining dense materials.

The mixture procedure chosen for disperses, nanometric and micrometric inclusions in alumina matrix was an excellent method to produce homogeneous powder mixture.

Addition of carbide particles in alumina matrix was efficient in inhibition grain growth and this effect is improved with the increase of inclusions percentage. The presence of NbC and WC particles promote better homogeneity in alumina micro-nanocomposite microstructure compared to pure alumina. And promote a change in fracture type of alumina from of mix of intra and transgranular fracture to a predominantly intergranular fracture.

Acknowledgements

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