11th International Conference on Advanced Materials
Rio de Janeiro Brazil
September 20 - 25
ICAM 2009

High pressure-high temperature sintering of nanostructured superhard material

A. L. D. Skury[(1)*], M. G. Azevedo[(1)], G. S. Bobrovnitchii[(1)], S.N. Monteiro[(1)]

(1) LAMAV, Universidade Estadual do Norte Fluminense (UENF), e-mail: luciaskury@pq.cnpq.br
* Corresponding author.

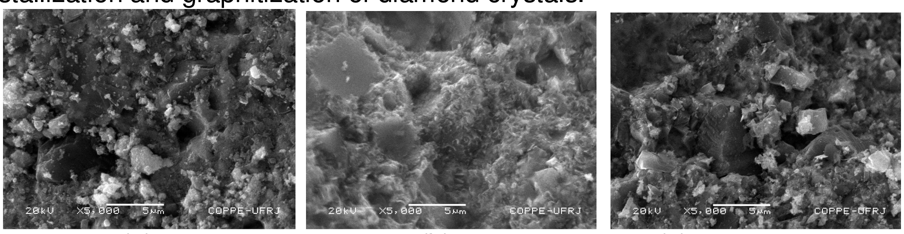
Abstract – This study aimed at assessing the achievement of superhard nanostructured composite in the Nb_2O_5-Diamond system by application of high pressures and high temperatures. The nanostructured powder used in the sintering was obtained by high-energy ball milling. At the end of the milling / mixing, the mass obtained was sintered at different temperatures (1100℃ to 1400ºC) under a pressure of 6GPa. The results obtained after the analysis of fracture of the compact region indicates that the increase in temperature is affecting the morphology of the particles constituting the matrix and the amorphous carbon added to the system functioned as an inhibitor of the process of graphitization.

.

Nanostructured superhard materials has been syntherized under high pressure and high temperature. Each sample is composed of 70% of diamonds (by weight) and 30% binder (80% Nb_2O_5 and 20% amorphous carbon). The milling of the components was made in high-energy mill. The sintering process of the samples was conducted under pressure of 6 GPa and temperature ranging from 1100 to 1400°C, and these conditions maintained for approximately 30 seconds.After the process of sintering the samples were broken and the region of fracture was analyzed by SEM and X-ray diffraction.

As seen in Figure 1(a), considering the scale of the micrographs and particle size of powders used for the preparation of sintered nanopó to note that the process of milling was efficient since crystals can be seen, Figure 1. There is also that the matrix consists of clusters of particles with sizes less than 1μm. But it also noted the presence of pores. The analysis of the micrographs of Figure 1(b) shows that, apparently, the increase in temperature is causing changes in the morphology of the matrix, although there is the presence of porosity. Looking to Figure 1(c) note that the morphology of the particles constituting the matrix metal is present as separate from those observed for samples obtained at lower temperatures. The increase in temperature caused the formation of clusters of particles with a "rectangular" form.

In Figure 4 shows the difractogram characteristic for the samples sintered at 1200 and 1400°C. According to the XRD analysis, it is clear that contamination of samples of iron, which comes from the jar of steel used in high-energy milling. It identified the presence of oxides and iron carbide. Another fact to be highlighted concerns the amorphous carbon present in the mixture. As can be seen in diffractograms, the amorphous carbon was converted to graphite. Therefore, taking into account the micrographs of the samples, it can be assumed that the amorphous carbon in the system is acting as an inhibitor of the process of recrystallization and graphitization of diamond crystals, as no marks were observed on the surface of these processes of crystals. According to the analysis of XRD is based on results of the research described in this work, we can conclude that the high-energy milling promotes the formation of compounds that act as binders in the process of sintering of the diamond. The increase in temperature, apparently, is influencing the morphology of the particles constituting the matrix. The increase in temperature caused the formation of clusters of particles with a "rectangular". The amorphous carbon in the system is acting as an inhibitor of the process of recrystallization and graphitization of diamond crystals.



(a) (b) (c)

Figure 1: SEM image of a fracture region, **a)** 1100 °C **b)** 1200 °C and (c) 1300 °C

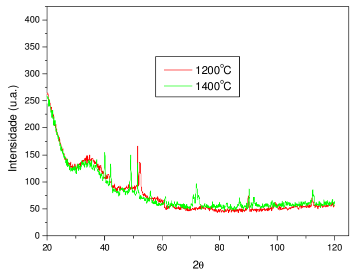


Figure 2: X-ray diffractogram of samples.