MICROWAVES FOR SOL-GEL SYNTHESIS OF BORON CARBIDE (B_4C)

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The term "Advanced Ceramics" designates ceramics with especial characteristics that made them useful in different areas than traditional ceramics, such as bricks, sanitary articles and domestic porcelain. Ceramics could be divided in two great groups: functional and structural ceramics. Ceramics that perform electronic, electromechanical, magnetic and optical functions belong to the first group. The second group emphasizes the applications where better mechanical properties at high temperatures are required and offer great resistance to the abrasion.

Boron carbide (B_4C) exhibit high melting point (2447°C), as a semiconductor type-p show a very high Seebeck coefficient, and it changes to type-n depending of boron content. Other important property is its ability for absorbing neutrons and its thermal stability.

Boron carbide is obtained commonly from a reaction at high temperature of boric oxide and coal coke poured into an electrical arc furnace. Other technique consisted of loading the electrical arc furnace with mixtures of boron and metallic magnesium inside the center of a graphite bar, which works like an anode, producing a mixture of nanofibers and icosahedral carbide crystal (B_4C). Other techniques for obtaining this product involved radio frequency plasma, vapor electrochemical deposition and deposition with microwave plasma. Materials produced by these methods are of high purity and very good for thin film applications.

The Sol-Gel process is a technique employed for elaborating ceramics of great quality; the preparation of a material by this method is carried out by mixing the reactives in liquid phase for having a homogeneous dissolution at molecular level. This dissolution is put under a process of polymerization and gel formation. The resulting gel is transformed later into the desired compound by means of a controlled heat treatment during which the elimination of volatile components takes place, while density of the material is increased.

It is proposed in this research that the use of microwaves improves reaction of carbon and boron compounds, obtaining a boron carbide precursor that is more reactive than the obtained by conventional means. Then conduct the sol-gel process and later the thermal treatment with microwaves. Conventional processing was also considered for comparison purposes.

In this work, for the conventional or microwave experiments, the meaning of microwave processing is that all of the stages, but drying, were conducted with microwaves.

Carbon suppliers were black fume, calcined saccharose and saccharose; boron suppliers were boron metoxide, boron etoxide, and boron triisopropoxide and biglycerol borate. Reagents were poured into a reactor and temperature was maintained between 60°C and 100°C, conventional processing lasted between 9 and 15 hours, and microwave processing lasted between 2 and 3 hours. The formed gel was dried at 150°C for 24 hours under a nitrogen atmosphere. Then, under the same atmosphere a thermal treatment was conducted for 4 hours at 800°C

One important issue for microwave processing is that precursors must be microwave absorbent, otherwise they would not warm up. In cases where they were not good enough absorbents they were mixed with coal, up to 10%. Only those precursors that absorbed microwaves were chosen for the following stage, which consisted in having them encapsulated in quartz at high vacuum (10^{-4} torr) and placed into a multimode cavity for being exposed to 1400 Watts at 2.45 GHz. Power was applied for 800 minutes in on/off (15 min/10 min) cycles. Products were characterized right after this procedure, and after a lixiviation process.

Precursors and obtained materials were characterized by X-ray diffraction, Scanning electron microscopy, Thermogravimetrical analysis, and Infrared spectroscopy. The results showed a greater efficiency in the samples heated with microwaves based on the analysis performed on the mixtures with different alcohol for alcoxide generation, which allow to propose that microwaves could activate generation of ion alcoholates, which shows a 10:1 relation with normal heating. It was also found that microwave heating speeds up the reaction, and that morphologies are more defined and homogenous in the synthesis of precursors of boron carbide compared to those obtained conventionally. More over, the gels obtained by means of microwave are more stable than those obtained conventionally.

Regarding the kind of obtained product, it was found that more nanoparticles of boron carbide were obtained at temperatures of 1300°C in the microwave case, while it was verified that applying the microwave sol-gel technique, obtained particles of boron carbide are nanometric, either as nanofibers or amorphous. While, greater amounts of carbon nanotubes and less nanofibers are produced when the precursors were prepared by conventional means.