# Optimization of Sol-Gel Synthesized Preceramic Polymer Precursors for Fabrication of High Purity Boron Carbide ($B_4C$) Powders

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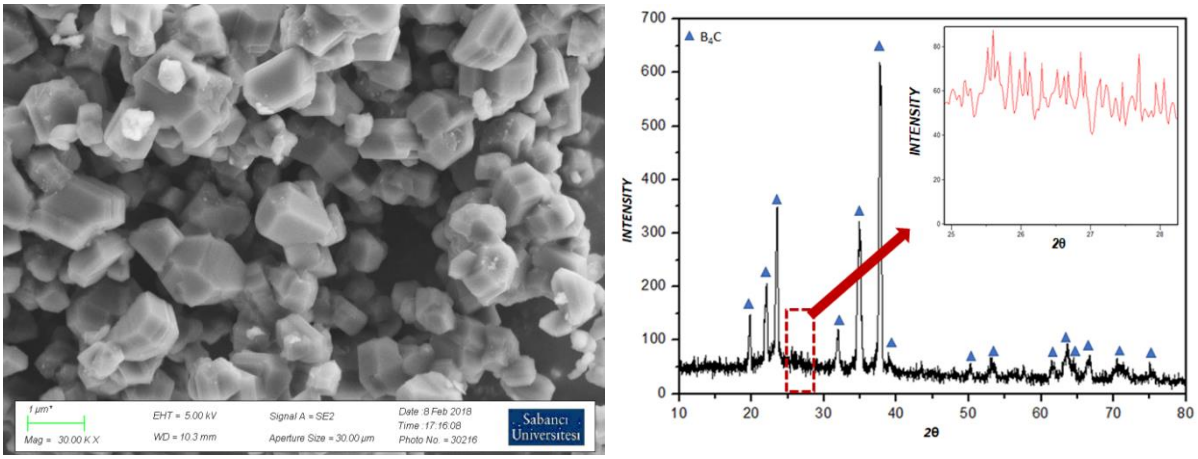
## Abstract

Boron Carbide ($B_4C$), due to its high hardness and elastic modulus, superior chemical stability, low density and high neutron absorption cross section, is well suited to a variety of industrial applications such as blasting nozzles, wire-drawing dies, powdered metal and ceramic forming dies, vehicle armour, bulletproof vest, nuclear reactor control rods and neutron absorbing shielding. The current fabrication techniques of boron carbide powders such as carbothermal reduction, direct synthesis from elements, vapour-phase reduction, and magnesiothermic reduction require expensive equipment and starting materials. For this reason, in recent years studies focused on the development of low-temperature synthesis techniques to reduce the production cost of boron carbide as well as to get better control over composition and particle morphology.

The production of non-oxide ceramics from polymeric precursors can be traced back to the 1960s. Preceramic polymers were used to synthesize ceramic powders, fibres, foams and to densify ceramic matrix composites by infiltration. A homogeneous mixture of starting materials at the molecular level in sol-gel synthesized preceramic polymers enables lower synthesis temperatures without the need for pressure, as compared with classical ceramic powder processing. Although improvements have been made in the development of new synthesis routes to preceramic polymers with controlled composition in the last decade, the ideal composition and production conditions to produce nuclear grade boron carbide powders have not been determined.

In this study, preceramic polymer precursors were synthesized by sol-gel technique. Condensed gel products were prepared by condensation and dehydration reaction of glycerol, ethylene glycol, tartaric acid, citric acid and boric acid in appropriate proportions. Thermal behaviour of condensed gel products was analysed by using DTA/TG. Fourier transform infrared (FT-IR) spectra of the gels were inspected in the 400–4000 $cm^{-1}$ wavenumber region. Nuclear magnetic resonance (NMR) measurements were also carried out to gain better understanding on gel network. After the calcination of condensed gels in atmospheric conditions, the preceramic polymer precursors were subjected to thermal treatment at temperatures ranging from 1300 to 1500 $^oC$. Microstructural and phase characterizations of

both precursor and final products were also carried out by using SEM and XRD techniques, respectively.




**(a)** **(b)**

**Figure 1.** (a) The SEM image and (b) XRD analysis result of $B_4C$ particles synthesized at 1500 °C for 5h.

The results indicate that borate ester bonds (B-O-C) were successfully achieved during condensation and dehydration reaction. The yield of the reaction strongly depends on the starting composition, thus the molecular structure of condensed gel product. It is also found that thermal decomposition behaviour and $C/B_2O_3$ ratio of preceramic polymer precursors strongly effect the final morphology and chemical composition of boron carbide powders. Figure 1 (a) shows the SEM image of the synthesized powders after thermal treatment at 1500 $^oC$ for 5h indicating that $B_4C$ particles with polyhedral morphologies are obtained. Figure 1 (b) shows the XRD pattern of the produced $B_4C$ powders does not contain any other residual phases such as boron sub-oxides or graphite.

As a conclusion, the influence of sol-gel synthesised preceramic polymer precursor characteristics on production of $B_4C$ powders were investigated in a wide range of starting composition. The relationship between the molecular structure of condensed gel product, thermal decomposition behaviour and $C/B_2O_3$ ratio of preceramic polymer precursor and the final morphology and purity of $B_4C$ powders will be discussed and addressed.

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