

EFFECTS OF TEST PARAMETERS ON THE PRODUCTION OF HEXAGONAL BORON NITRIDE BY DCRN METHOD

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ABSTRACT

Hexagonal boron nitride (h-BN) is a versatile ceramic material due to its many unique physical and chemical properties, for instance low density, elevated temperature stability, high thermal conductivity, low dielectric constant, chemical inertness and lubricating property. Because of these properties, h-BN finds large application areas in industry. Carbothermal reduction and nitridation (CRN) ranked among the production methods of h-BN is an effective and economic technique. In this study, a novel method of ceramic powder production, so called dynamic carbothermal reduction-nitridation (DCRN) was performed and effects of the different test parameters were examined in producing h-BN. The granules were prepared from the mixture of boron oxide (B_2O_3), carbon and calcium carbonate ($CaCO_3$). These granules were placed in a graphite reactor, where they reacted with nitrogen gas in the atmosphere controlled tube furnace, which alumina tube was rotating with controlled speed. The effects of test conditions such as temperature, exposure time and rotation speed of the reactor on the formation of boron nitride were examined. The reaction products were characterized by X-ray diffraction (XRD) and Field Emission Scanning Electron Microscopy (FESEM). The optimum production conditions according to XRD analyses and FESEM examinations were determined as 1400 °C for 2 h reaction with rotational rate of 4 rpm.

The XRD patterns of reaction products obtained at different temperatures for 2 hours are shown in Figure 1(a). The intensity and width of the strongest h-BN diffraction line at $2\theta = 26.627^\circ$ increased with increasing reaction temperature. Both crystallite size and lattice strain increase the peak width and intensity and shift the 2θ peak position accordingly [Zak et al., 2011]. Therefore, the peak broadening was attributed to decreasing size of BN crystallites. The products synthesised at 1400 °C contained h-BN, C and some glassy phase.

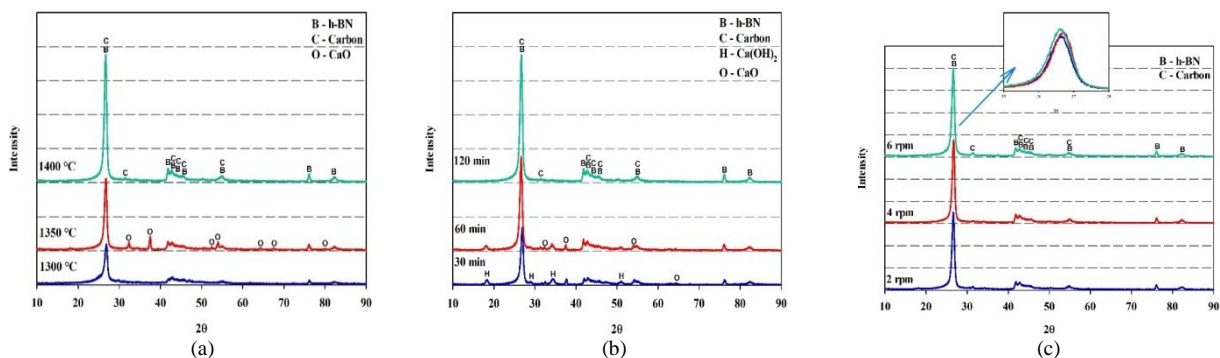


Figure 1: (a) The XRD patterns of reaction products obtained at different temperatures, (b) The XRD patterns of reaction products at various reaction times at 1400°C. (c) XRD patterns obtained at different rotating rates for 2 hours at 1400°C

The XRD patterns for different reaction time are shown in Figure 1 (b). The reaction products obtained at 1400 °C after 30 and 60 minutes contain C, h-BN, Ca and Ca(OH)₂ phases. Formation of the Ca(OH)₂ phase is attributed to hydration of free lime after the synthesis. The intensity and width of the main h-BN diffraction line increased with increased duration of the reaction.

The phases formed at all rotational speeds are h-BN, C and glassy phase and the intensity and width of the main h-BN diffraction maximum increase with increasing reactor rotation speed. However, agglomeration was observed in the powders rotated at 6 rpm.

To get rid of the glassy and undesirable other phases found in the reaction products, samples were subjected to chemical activation using 5 M HCl acid solution for 15 hours. After the chemical activation the samples were washed, filtrated and dried before exposure to oxidation at 800 °C for 15 h to remove the unreacted C. The XRD pattern which belongs to reaction products produced with DCRN method and the XRD pattern of reaction products obtained after carbon combustion are shown in Figure 2.1. As seen in the Figure 2.1(a), the reaction products prepared by the DCRN method contain both BN and C phases. After carbon combustion, all the peaks were indexed as hexagonal BN. The peak width and intensity changes at the XRD pattern of products obtained after carbon combustion. The prominent (002) plane indicate the existence of well-stacked layered structures in the h-BN powders [Yang et al., 2005].

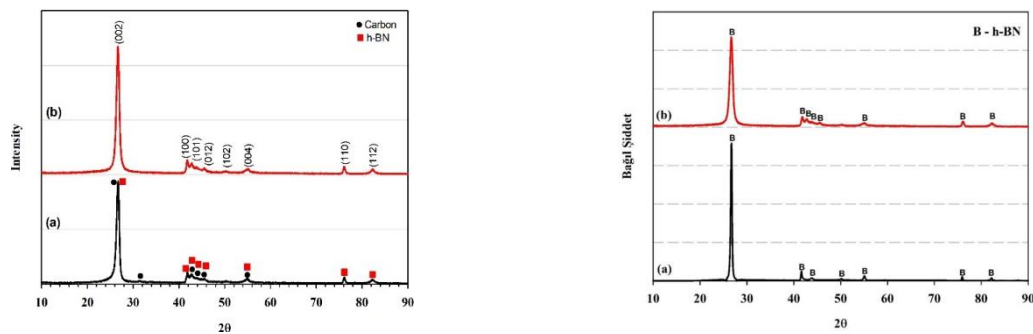


Figure 2.1: XRD patterns of (a) the reaction products produced with DCRN method and (b) products obtained after carbon combustion, Figure 2.2: XRD patterns of (a) commercial product of h-BN, final product obtained by DCRN

The FESEM micrographs of reaction products and final powder products are shown in Figure 3. After chemical activation and oxidation process, the flake morphology of h-BN particles is clearly visible in the micrograph. The flakes are restricted to nanoscale in thickness.

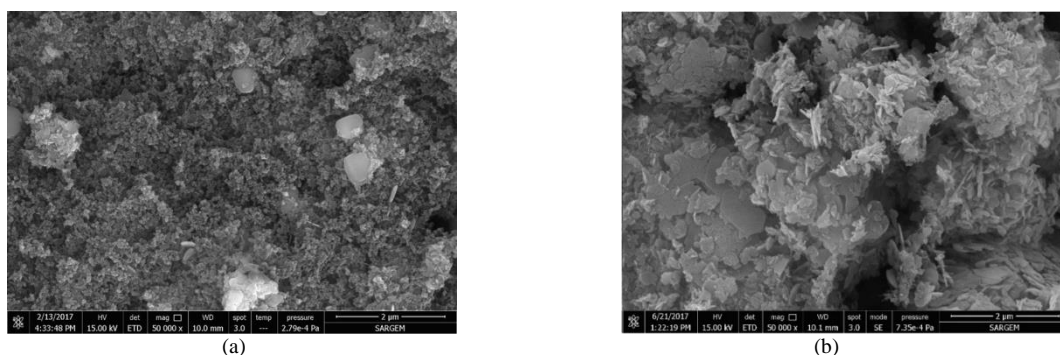


Figure 3: The FESEM micrograph of (a) reaction product obtained by DCRN, (b) final powder products. (Scale bar shows 2 μm.)

Keywords: DCRN, CRN, advanced ceramics, h-BN

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