

Controlled High-Temperature Sintering of Boron Carbide

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Abstract—The paper discusses the boron carbide properties. Two methods are used to examine and control the sintering process of boron carbide. For achieving sintering is used a Tammann furnace. First, it is used an electric furnace with the thermal couple for determining transmission coefficient of ZnSe infrared window, observed with an infrared thermal camera. Analysis of the temperatures with and without infrared glass are discussed. After setting the transmission coefficient to the camera, the material is sintered to desired temperatures and for the temperature control is used an infrared camera. The result of the controlled temperature is achieved boron carbide material, obtained in strict temperature environment.

Keywords—Tammann furnace, control, temperature, thermography, zinc selenide, boron carbide, high-temperature

I. INTRODUCTION

The Boron carbide was synthesized more than a century ago in 1883 by Joly. The B_4C formula was identified in 1934 [1]. Today, the range of homogeneity of the compound ranged from $B_{4.3}C$ to $B_{10.4}C$. The composition of commercial boron carbide is typically close to the stoichiometry of boron: carbon of 4:1. Boron carbide is an extremely hard material with a lower hardness of only diamond and cubic boron nitride. In addition, it has a high melting point, approximately at 2490 °C (13,3 mol% C) and forms a eutectic with carbon at 2220-2260 °C. It has a high mechanical strength combined with low density and high cross-section neutron grip and it is a semiconductor [2]. It is difficult to sinter to full density, usually, boron carbide is produced with an addition of sintering aids such as fine carbon or silicon carbide [3].

Boron carbide is characterized by: Exceptional hardness; Low thermal conductivity; High elastic module; High compressive strength; Good nuclear properties; Low density. Boron carbide have a profile of properties that are appreciated in a wide range of applications such as mining industry, aerospace, military industry, abrasives requiring extreme hardness for use in polishing and bonding, diamond tools and water jet cutting, nozzles requiring wear and abrasion resistance to siphoning, sanding and water jet cutting machines, etc. The wide range of applications that this material provides deserve to be taken into account in new research and product design projects.

The aim of this work is to be examined the sintering temperature used for boron carbide. The control of the temperature is implemented by using an infrared thermal camera.

II. STATE OF THE ART

The most famous B_4C cold-sintering additive is carbon. Carbon additives are generally carried out by in-situ pyrolysis of a phenolic resin which serves as a compression agent and delivers ~ 30% by weight of carbon upon decomposition. Schwetz and Vogt [4] first use a carbon additive in the form of a phenolic resin of very fine, pure, stoichiometric B_4C with 10-22 m^2/g of surface area. They note that the addition of carbon facilitating up to 98% of the theoretical density at 2150 °C. At the same time, other independent studies have also demonstrated the importance of the carbon supplement [5, 6]. Suzuki [5] synthesizes B_4C with 25-30% by weight carbon supplements to 90-93% theoretical density at 2250 °C and monitors the formation of a liquid from the eutectic composition (30% C). Later Schwetz and Grellner [7] examined in detail the effect of carbon addition. They found that only the use of a B_4C high-throughput powder with a high surface area (over 15 m^2/g) and the addition of carbon could result in densified (over 95% of the theoretical density) finely-granulated microstructure articles.

III. USED DEVICES FOR ACHIEVING THE STUDY

The high-temperature melting point of boron carbide requires Tammann furnace (fig. 1). The maximum temperature work of the furnace is 2200-2300 °C, and the minimum is 1400 °C. The samples are placed in graphite tubes with a clearance of 125-130 mm and a thickness up to 10 mm. Their length is 1400 mm. The reflecting graphite tube is with a diameter of the lumen up to 140 mm and a length of 1500 mm. The isolation consists of a graphite powder. The power supply of the high-temperature Tammann furnace is based on IGBT technology. Also, a water-cooling system with high protections against incoming and outgoing low voltage, strong output current, and voltage, overheating. For achieving maximum performance of the furnace is mounted devices for automatic feeding and a device for receiving the finished products. Tammann furnace works in a controlled environment with protective gas, most commonly dissociated into hydrogen and nitrogen ammonia. The used technology can increase significantly the durability of the materials [8, 9, 10].

This working principle of the Tammann furnace requires the usage of service (measuring) infrared window. For using infrared thermography, the service window must be from a material, transmitting in the infrared wavelength. Such materials are germanium, zinc selenide, amir, quarts, etc. For this study is used a zinc selenide window.

The use of infrared window from zinc selenide glass preforms more accurate temperature measurement and control. The use of zinc selenide (ZnSe) glass in the Tammann furnace allows more accurate measurement of temperatures due to the linear characteristic of zinc selenide at temperatures above 700 °C, another important advantage is that also be a gas filter will be used at high temperatures of 2000 ÷ 2300 °C. Most often the transmittance of the window depends upon its thickness, used material, density, etc.

Today there is a wide market choice and different companies, which provides different thickness, material, etc. of infrared windows. This requires investigation and validation of the transmittance of the chosen infrared window. For that purpose is used an electrical furnace with a thermal couple working up to 1100 °C, shown on fig. 2.

The high-temperature control is observed and measured with infrared thermal camera FLIR P640 (Fig. 3) with a field of view 24° / 16°, a minimum range of measurement temperature with two regimes from -40 °C to 120 °C, from 0 °C to + 500 °C, and for this study was performed calibration and software update for achieving measuring temperatures up to 2100 °C. The camera temperature sensitivity is 0.06 °C at 30 °C, detector matrix 640/480 pixels, integrated color digital camera, image settings, metering mode, various lenses, and management software [9, 11, 12, 13]. The temperatures are validated with the build-in thermal couple of the electrical furnace.

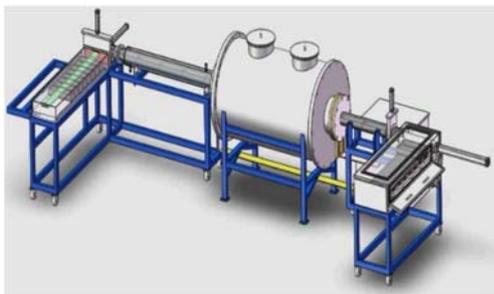


Fig. 1. Tamann furnace.



Fig. 2. Electric furnace.



Fig. 3. Thermal camera.

IV. EXPERIMENTAL RESULTS

The experiment is established in a laboratory with a controlled environment.

First is achieved the investigation for verifying the temperatures of the infrared thermal camera and the thermal couple from the electric furnace. The results from the thermal image, shown on fig. 4 matches to the furnace thermal couple. The emissivity coefficient is set to $\epsilon = 0,98$, and the measured temperature in the Bx1 (Box 1) is 828 °C. Next step is to calibrate the zinc selenide glass, shown on fig. 5. It is clear that without setting the transmission coefficient, the temperature is 471,4 °C. It is lower than the measured from the thermal couple of the furnace and from the thermal imaging camera.

Numerous experiments with different temperatures were made. Part of the measured temperatures are shown on fig. 6 is 915,6 °C. The measured temperature on fig. 7 is 1084 °C. For all measurements is chosen coefficient of transmissions, set to 0,41 and the temperatures are validated from the built-in thermal couple of the furnace.

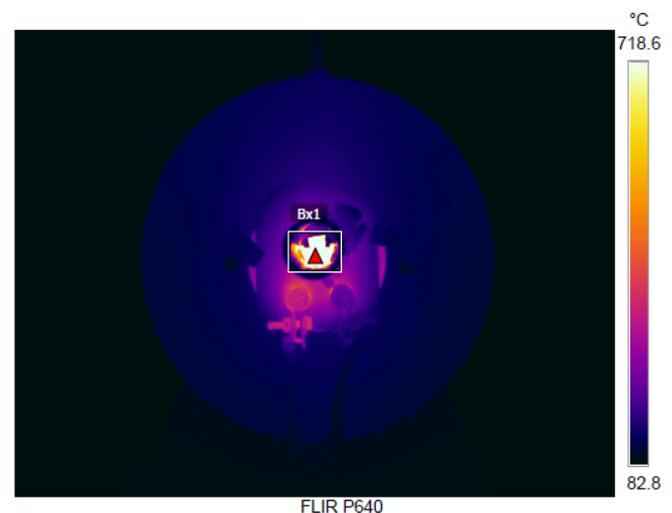


Fig. 4. Infrared image of examined detail.

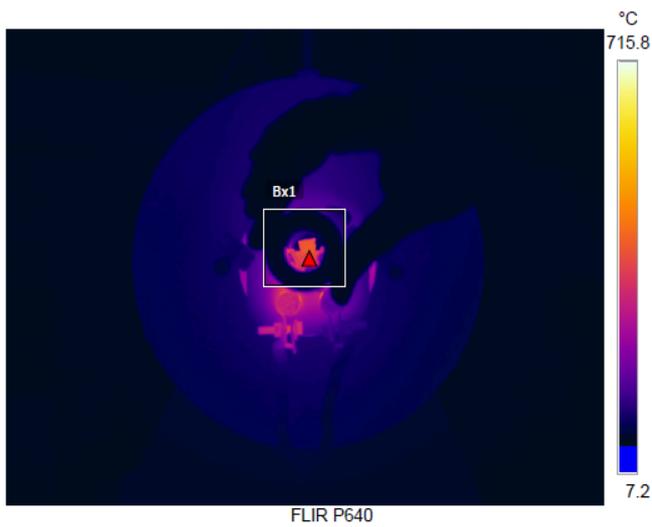


Fig. 5. Infrared image of examined detail with ZnSe glass.

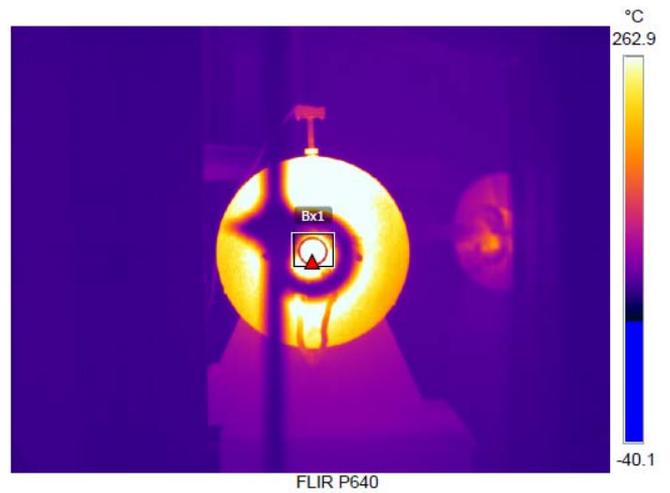
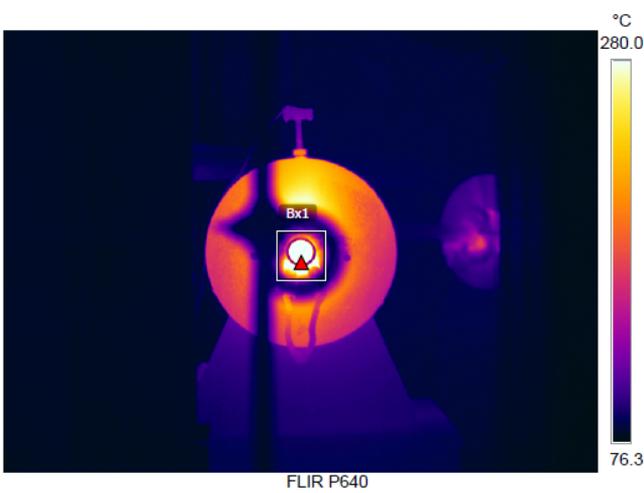


Fig. 7. Infrared image of examined detail with ZnSe glass at 1084 °C.



a) Infrared image



b) Digital image

Fig. 6. Testing with infrared ZnSe glass: a – infrared image of examined detail with ZnSe glass at 915 °C; b – digital image.

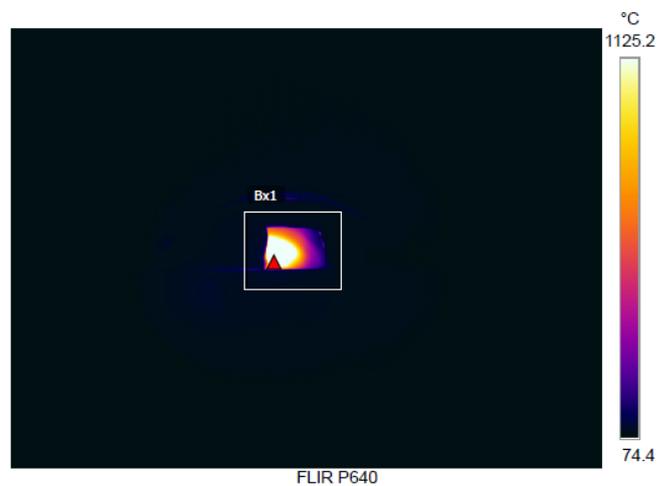


Fig. 8. Infrared image of examined detail with ZnSe glass at 1650,4 °C.

After defining the transmission coefficient next step is to make an attempts to sinter boron carbide at various temperatures 1950 ÷ 2000 °C, 2050 ÷ 2100 °C. The temperature is observed again with infrared camera trough service window using 3mm ZnSe glass. When the temperature rises above 2,100 °C, boron carbide products will melt, and at temperatures lower than 1950 °C there will be no sintering. The sintering temperatures for boron carbide are determined by the activating additives. The measured temperatures are shown on fig 8, with a detected temperature of 1650, 4 °C.

The controlled temperature prevented the boron carbide from melting and the sintered details, shown on fig. 11.

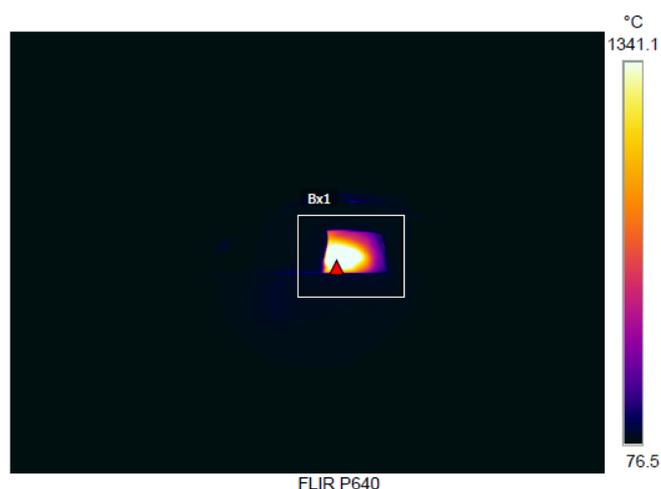


Fig. 9. Infrared image of examined detail with ZnSe glass at 1749,5 °C.

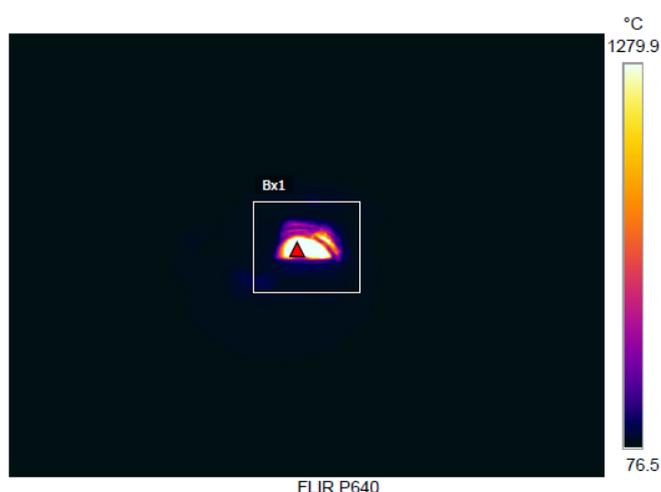


Fig. 10. Infrared image of examined detail with ZnSe glass at 2100 °C.

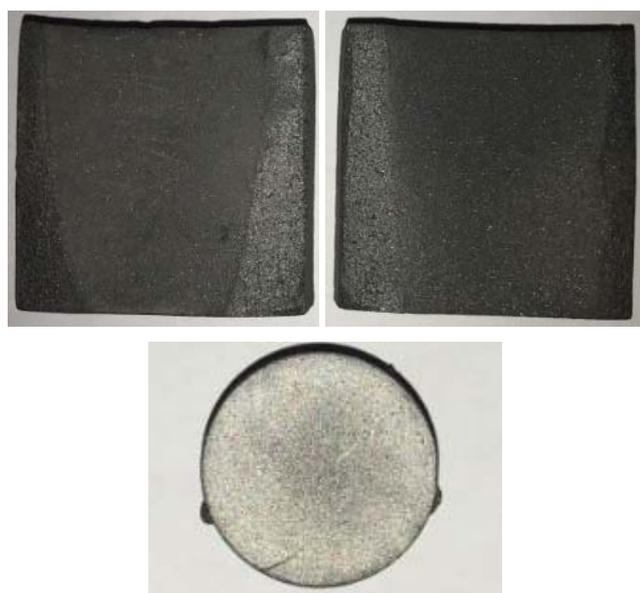


Fig. 11. Sintered Boron carbide details.

V. CONCLUSION AND FUTURE DEVELOPMENTS

Calibrating the ZnSe glass with an electric furnace and its built-in thermal couple gives the possibilities to adequate and

precise determination of the used ZnSe window transmission coefficient.

The strict control of the temperature during sintering the boron carbide gives the desired effect without melting the object.

High-temperature technology for the production of high strength and wear-resistant materials and alloys, using nanoelements. Example of high-wear resistant need is the mining industry and mills as well as the mill grinding media and environment. It is an innovation for supplying Bulgarian companies with materials without the need for import of expensive materials, equipment, and technology. Those materials can be included in the crystal lattice of the resulting alloys, as well as catalysts for the synthesis and sintering processes.

Next step is to be examined the internal structure of the boron carbide for cracks and other defects with 3D scanning computed tomography Nikon XT-H 225

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