

EBSD Characterisation of SPSed CeB₆ Thermionic Electron Emitter

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Cerium hexaborides (CeB₆) with simple cubic structure has attracted growing interests owing to its special electronic and magnetic performance. As an electron source, CeB₆ has low work function (ϕ) (2.5 eV), operation temperature and volatility which means a longer service life when used as a thermionic electron emitter [1]. Recently, many researchers have focused attention on the field emission properties of nanowires obtained by chemical vapor deposition (CVD) method. Compared to the nanowires and films, the polycrystalline bulk materials can provide large size, low cost, simple preparation and can be fabricated to various devices [1,2]. For this reason, in this study, polycrystalline CeB₆ was produced as a cathode material by using spark plasma sintering (SPS) technique. X-ray diffraction (XRD) result not given here showed that CeB₆ was detected as the only crystalline phase of any sintering temperature. However, backscatter electron images (BSE) indicated that three different phases might exist. At the same time, particle size and distributions could not be clearly identified from BSE images (Figure 1 (a)). Therefore, the aim of the study is to improve the results obtained from XRD and BSE imaging techniques by using energy dispersive x-ray spectroscopy (EDX) and electron backscatter diffraction (EBSD) techniques.

For this purpose, the samples having same composition were sintered in SPS furnace at 1650, 1750 and 1850 °C, respectively. EDX (Oxford Instruments, INCA Energy) and EBSD (Oxford Instruments, INCA HKL NordlysS) techniques were used to determine the chemical composition, amorphous/crystalline character and amount of existing phases. Furthermore, EBSD orientation maps of CeB₆ samples were used to determine the suitable sintering temperature to achieve lowest work function and highest emission. During EDX analysis accelerating voltage was selected depending on the size of the particles to reduce the spatial resolution.

Therefore, for B₄C and B₂O₃ phases 5 kV and for CeB₆ phase 15kV accelerating voltages were used. EDX analysis indicated that grains with white bright contrast represent CeB₆ particles, dark grey colored areas represents B₂O₃ amorphous phase and black colored phases represents the in-situ formed B₄C particles (Figure 1 (a-b)). B₄C particles were formed due to the reaction between graphite SPS mold and boron which was added to the starting composition.

In contrast to XRD analysis, EBSD analysis proved the EDX analysis that crystalline B₄C and amorphous B₂O₃ phase exist in the sample (Figure 2 (a-c)). In XRD analysis, B₄C could not be detected due to the low quantity. Particle sizes and their distribution were different in the samples sintered at different temperatures. In order to achieve highly emissive and low electrically resistive material, the sample must contain only a CeB₆ phase. EBSD phase analysis showed that best result were obtained at 1850 °C since the B₄C and B₂O₃ phases were eliminated at this temperature.

The previous investigations [1,3], the work functions of hexaborides are related to its surface: the order of for the different surfaces was $(\phi)(210) < (\phi)(100) < (\phi)(110) < (\phi)(111) < (\phi)(211)$. According to EBSD crystal orientation maps and inverse pole figures (Figure 2 (d-f)) obtained from sample sintered at 1850°C, (111) planes of CeB₆ crystals have higher density. EBSD results were supported by density and electrical resistance measurements. Therefore, 1850 °C was selected as the most suitable sintering temperature.

References:

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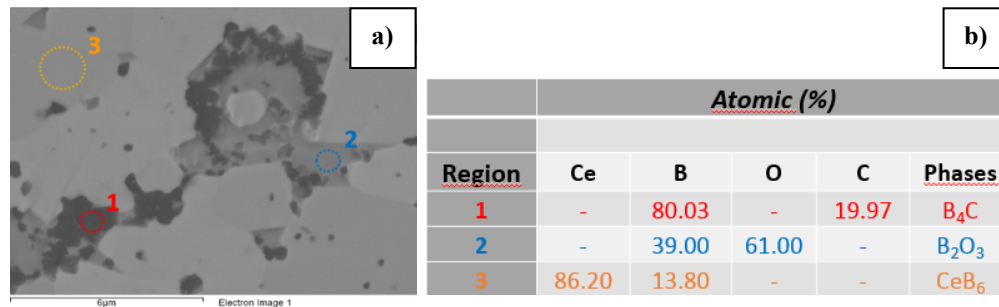


Figure 1. (a) BSE image of CeB₆ sample and (b) quantitative EDX results obtained from different existing phases.

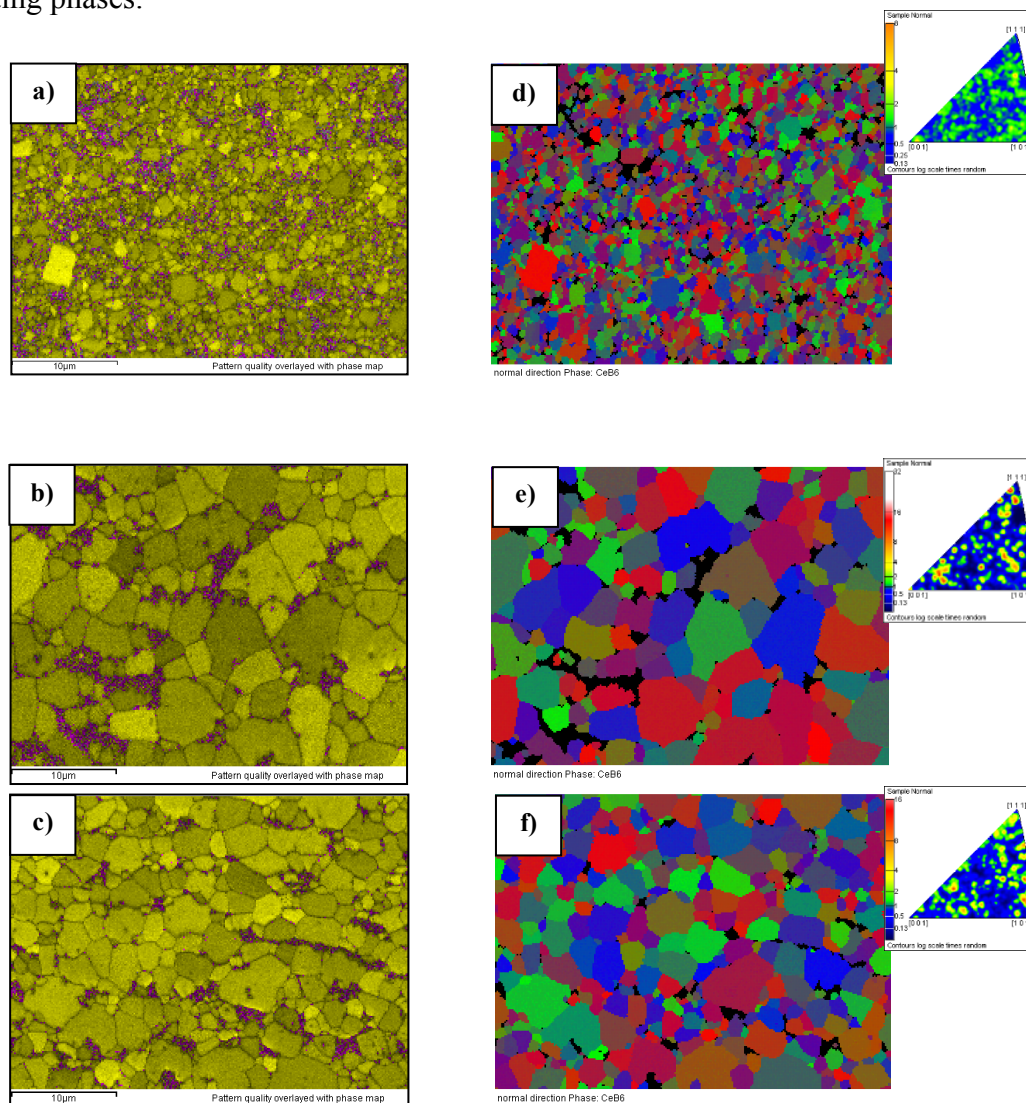


Figure 2. (a-c) EBSD phase maps and (d-f) crystal orientation maps-inverse pole figures obtained from sintered CeB₆ samples at (a-d) 1650°C, (b-e) 1750 °C and (c-f) 1850 °C.