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Densification of commercial monoclinic zirconia through hot isostatic pressing

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Abstract: Monoclinic zirconia was densified using hot isostatic pressing (HIP) at 1000 ºC and 190 MPa for 1 h, using steel encapsulation and boron nitride as protective and release agent. The maximum of the average densities after HIP was 93.4% of theoretical density and the average final grain size was 220±16 nm. However, some regions exhibited nearly full density. X-ray Photoelectron Spectroscopy (XPS) revealed carbon contamination, no reduction of Zr^{4+} and no significant diffusion of boron or nitrogen.
Key words: CIP; HIP; XPS; Crystalline oxides.

Introduction and Experimental

Monoclinic zirconia polycrystal (MZP) was recently shown to exhibit superplasticity-like behavior [1, 2]. Densification of MZP leads to the fracture of the material if the temperature exceeds 1170 ºC. This necessitates the densification by using specialized ways such as hot isostatic pressing (HIP) [1, 2]. There are also other methods [3-9]. However, in these studies, aspects such as vacuum encapsulation, contamination and reduction of MZP during HIP have not been addressed. These will be addressed in the present study on HIP of commercial MZP.

MZP powder was purchased from Tosoh Co., Japan. X-ray diffraction (XRD, Siemens, Co-K_α radiation) and differential thermal analysis (DTA) run were carried out on the as-received powders. The powder was cold pressed at 29 MPa and then cold isostatically pressed (CIPed) at 500 MPa into compacts of 17 mm height. The compacts were encapsulated in austenitic steel (ASTM 303) capsules. A thin coating of boron nitride was given on the inner walls of steel and the outer surface of two MZP compacts and dried for 24 hours at 100 ºC for one compact and at 300 ºC for another compact. The capsules were evacuated, welded and HIPed with argon gas at 190 MPa and 1000 ºC for 1 h. The average bulk densities of the densified samples were measured by Archimedes method. Fractured surface of the samples were analyzed with a high resolution scanning electron microscope (HRSEM: Zeiss, DSM 982 Gemini) for their grain sizes. Possible contamination and reduction of MZP was analyzed with X-ray Photoelectron Spectroscopy (XPS). The experimental details and the methods of analysis of data can be found elsewhere [10].

Results and Discussion

XRD of the HIPed confirmed the existence of monoclinic phase. DTA runs confirmed the transformation temperature. The micrograph of a HIPed sample is shown in Fig. 1. The average bulk density of the sample was found to be 93.4% of theoretical density.

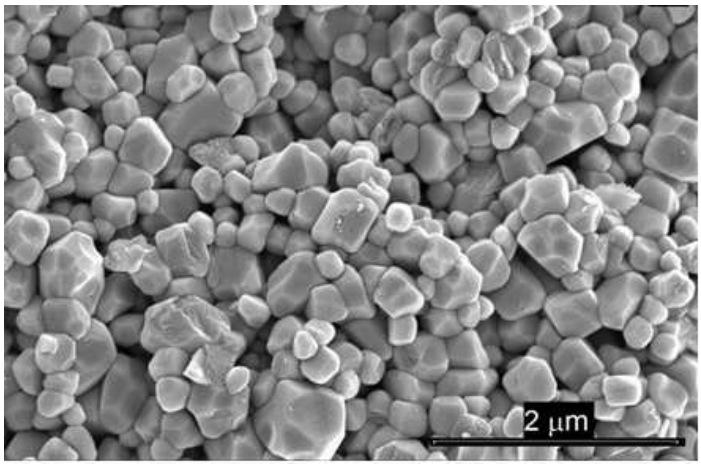

Fig.1: HRSEM of fractured surface of sample after HIP.

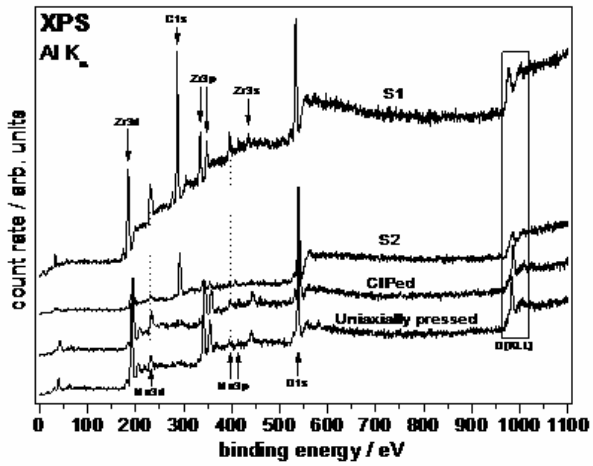

Fig. 2: XPS spectra of the uniaxially pressed, CIPed and HIPed samples (S1 and S2).

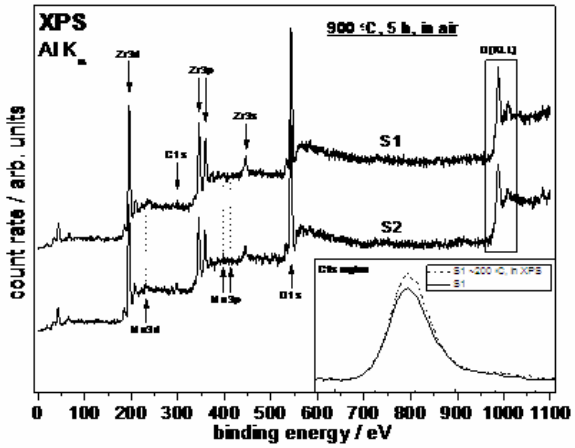

Fig. 3: XPS spectra of samples S1 and S2 after heating in air at 900 ºC (5 h). Inset: The C (1s) level of sample S1 heated up to ~ 200 ºC inside the XPS apparatus.

After HIP, the sample with BN dried at 100 ºC (labeled S1) turned dark gray. The one with BN coating dried at 300 ºC (labeled S2) retained its original color. XPS was done on all the HIPed samples together with one uniaxially pressed (at 397 MPa) pellet and one CIPed (at 500 MPa) pellet for comparison. These XPS results are shown in Fig. 2. The Mo peaks are due to the sample holder. Carbon was found at the surface in all the HIPed samples, in various quantities: about 65.5 at% for S1 and 54.7 at% for S2, while negligible carbon was detected for the uniaxially pressed (8.6 at%) and CIPed pellets (7.5 at%), thus confirming it to be coming from the HIP process. Further, carbon was found to increase to 77.6 at% for the sample S1 (Fig. 3, inset) when

the sample was heated to ~ 200 ºC in the XPS apparatus, in conjunction with de-gassing that could be observed with a significant increase of pressure. Similarly, for sample S2 the carbon content increased (however only slightly) to 55.4 at%. These results show that carbon (probably from BN) is not a surface contaminant, however comes from inside the sample to the surface. The two HIPed samples (S1 and S2) were later heated in air at 900 ºC for 5 h at the heating and cooling rate of 5 ºC min^{-1} and the XPS was performed again. The dark gray sample turned back to its original white color after this heat treatment. Carbon was found to have drastically reduced in both samples (≤ 2.8 at% for S1 and ≤ 8.6 at% for S2), as shown in Fig.3, further confirming that the carbon signals found previously in the samples were not artifacts.

In order to check the reduction of MZP, from Fig. 2, the Zr 3p peaks of HIPed samples were compared with the uniaxially pressed and CIPed samples. It was seen that these peaks could be approximated by a single Gaussian, showing no significant changes in the full width at half maximum. This suggests that there was no reduction of the sample. Therefore, the color changes should be attributed to increased C level (~ > 55 at% at the surface).

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