



# Surface microstructure and phase structure of zirconia ceramics under intense pulsed ion beam irradiation

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## Abstract

The surface microstructure and phase structure of zirconia ( $ZrO_2$ ) ceramics under intense pulsed ion beam (IPIB) irradiation were investigated in this work. Experiments were carried out on TEMP-4M accelerator with peak accelerating voltage, current density, and pulse duration of 220kV, 150 A/cm<sup>2</sup>, and 80ns. Micro-cracks with a height of up to 150nm above the surface were observed and were deduced to be formed by the tensile stress during the shrinking process. The cross-sectional observation indicates that columnar structure thin layer with thickness about 1.23 $\mu$ m was produced after IPIB irradiation. The GIXRD pattern indicates that obvious preferred orientation in (110) and (200) of t- $ZrO_2$  was occurred in near-surface region after IPIB irradiation. Opposite micro-stress in bulk and near-surface region was revealed by W-H plot after Rietveld refinement. The evolution of mass loss and surface roughness of samples with pulse number was also investigated.

## Introduction

Intense pulsed ion beam (IPIB) has been widely studied in past decades for various applications including surface treatment [1], [2], [3], [4], [5], [6], [7], thin-film deposition [8], [9], [10], nanopowder synthesis [11], [12], [13] and simulation of high thermal load in nuclear fusion reactors [14], [15]. With high-power density, IPIB induces a rapid rise and decline of temperature [16] in the near surface region of the target, leading to melting, evaporation, and re-solidification. The microstructure and phase structure of materials might be changed due to the intense thermal effect during IPIB irradiation. Meanwhile, shockwaves may be induced by IPIB, giving rise to the influence in a larger depth [17], [18], [19], [20].

Zirconia ceramics, with outstanding features such as low thermal conductivity, high hardness and chemical resistance, have been widely used in aerospace engineering and mechanical engineering [21], [22], [23], [24]. On the other hand, it is difficult to process ceramic materials by traditional treatment methods due to their high mechanical and chemical resistance [25]. The high-power density of IPIB makes it a candidate for ceramics processing. IPIB irradiation with different ion species, including,  $C^+$  [25], [26] and  $N^{2+}$  [21], were performed on  $ZrO_2$  ceramics by Ghyngazov et al. It was found that the electrical conductivity of  $ZrO_2$  had a significant improvement due to disturbance of the oxygen stoichiometric composition caused by IPIB irradiation. Similar effect on electrical conductivity of  $ZrO_2$  caused by pulsed electron beam was reported as well [25].

The changes in microstructure and phase structure of zirconia ceramics induced by IPIB were also reported. Micro-cracks and craters on the surface were the most common defects in microstructure caused by IPIB irradiation. Influence of IPIB on phase structure was analyzed using XRD technique which collects phase information from bulk region of samples with depth up to several tens  $\mu$ m. However, less effort was made to study the microstructure in 3D view which could provide more detailed information and there were few reports on analysis of the difference of phase structure in near surface and bulk region influenced by IPIB which introduces different effects over depth. The microstructure and phase composition have great influence on the properties of zirconia ceramics [24], [27], therefore, it is of great significance to make a deeper investigation on the influence of IPIB on the microstructure and phase structure to utilize it more effectively.

In present work, zirconia ceramics were irradiated by IPIB with different pulse numbers to investigate the irradiation effect of IPIB on the surface microstructure and phase structure. The surface and cross-sectional morphology of zirconia ceramics were analyzed in 2D and 3D view. The phase structure of zirconia ceramic in bulk and near-surface region was investigated using XRD and GIXRD with Rietveld refinement technique.

## Section snippets

### Experimental

The IPIB irradiation experiments were carried out on the TEMP-4M accelerator at Tomsk Polytechnic University. The main ion species of IPIB are about 85% carbon ions and 15% proton [28]. The working parameters of TEMP-4M for the experiments were pulse duration (FWHM), accelerating voltage, current density of 80ns, 220kV, 150 A/cm<sup>2</sup> corresponding to a dose of  $7.36 \times 10^{13}$  /cm<sup>2</sup> per pulse respectively. Polished  $ZrO_2$  ceramics with dimension of  $20 \times 20 \times 0.5$ mm<sup>3</sup> were placed vertically in the beam...

## Calculation results

The variation of ion concentration and irradiation-induced displacement damage (in units of dpa) with depth in ZrO<sub>2</sub> ceramic were calculated by SRIM in full-cascade mode. The threshold displacement energy was assumed to be 40eV for Zr and 50eV for oxygen in this ion–solid interaction simulation [30], [31]. According to the accelerating voltage and single energy simplification, when the SRIM program input, the ion energy is 220keV. The simulation results for 40 pulses are shown in Fig. 1. The...

## Conclusion

The surface microstructure and phase structure of ZrO<sub>2</sub> ceramic before and after IPIB irradiation were theoretically and experimentally analyzed. The simulation results of thermal effect shows that the temperature can reach boiling point of ZrO<sub>2</sub> under IPIB irradiation. The highest melting rate and re-solidification rate occur at the beginning of melting and ending of re-solidification respectively. Small particles and two types of cracks with zipper-like edge were observed on the surface after...

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper....

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