

## Microstructure and Hardness of ZrC Deposited on TRISO Coated Particles After Heat Treatment Above 1800°C

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### 1. Introduction

The TRistructural-ISotropic (TRISO) coated fuel particles are utilized as the fuel of the very high temperature gas-cooled reactors. TRISO coated fuel particles are embedded in the carbonaceous matrix, which is heat-treated above 1800°C for the graphitization of the matrix phase [1]. Therefore, a ZrC coating layer of the TRISO coated particles is subject to the high temperature and goes through the microstructural changes.

In this study, ZrC-TRISO coated particles were heat-treated above 1800°C and the changes in microstructure and hardness of the ZrC coating layer were investigated.

### 2. Experimental

Four layers consisting of a porous buffer layer, an inner PyC layer, a ZrC layer, and an outer PyC layer were deposited onto the ZrO<sub>2</sub> spherical particles by a fluidized-bed chemical vapor deposition (FBCVD) method. Especially, the ZrC layer was deposited from ZrCl<sub>4</sub>, CH<sub>4</sub>, H<sub>2</sub> and Ar at 1500°C. For the heat treatment, ZrC-TRISO coated particles were inserted into the graphite crucible located in an inert environment. Heat treatment was performed at 1800°C and 1900°C for 1h.

### 3. Results and Discussion

#### 3.1 Microstructure of ZrC coating layer

Fig. 1 shows TEM microstructure of a ZrC coating layer before/after heat treatment. No graphitic carbon is deposited in the near-stoichiometric ZrC phase. As-deposited ZrC has a relatively large grain size of approximately 400 nm with a granular shape, and ZrC grains are clearly distinguished by the grain boundaries. Significant grain growth occurred during heat treatment above 1800°C.

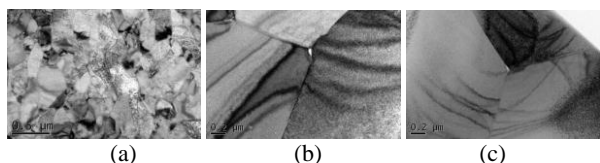


Fig. 1. TEM microstructure of the ZrC coating layer: (a) as-deposited, (b) after heat treatment for 1 h at 1800°C and (c) 1900°C.

Fig. 2 shows the inverse pole figure overlapped on image quality map of a ZrC layer deposited on the dense inner pyrolytic carbon. Significant grain growth only occurred in the inner region of the ZrC coating layer. ZrC grains in the outermost region were slightly grown from about 0.350 to 0.451 and 0.407  $\mu\text{m}$  after heat treatment at 1800°C and 1900°C, respectively.

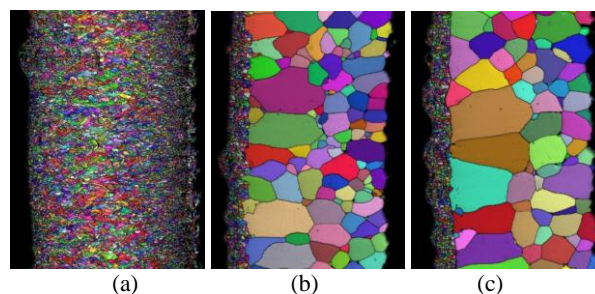


Fig. 2. Inverse pole figure overlapped on image quality map of ZrC: (a) the as-deposited and (b) after heat treatment at 1800°C and (c) 1900°C for 1 h.

It was revealed that the difference in the grain growth behaviors between the inner and the outermost region of ZrC coating was originated from the co-deposition of graphitic carbon [2]. As shown in Fig. 3, graphitic carbon was only observed in the outermost layer of ZrC coating which retarded the grain growth of ZrC during heat treatment.

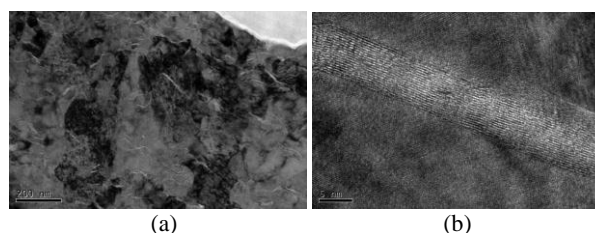


Fig. 3. TEM microstructure showing (a) the co-deposition of graphitic carbon in the outermost layer of ZrC coating and (b) graphitic carbon with the rough laminar structure.

The preferred orientation of chemically vapor-deposited ZrC at low pressure generally changes (111)  $\rightarrow$  (110)  $\rightarrow$  (100) with increasing the deposition temperature [3]. (100) is the energetically preferred direction for ZrC above 1400°C. As shown in Fig. 4, the ZrC coating deposited by FBCVD at 1500°C also has the (100) preferred direction which keeps its favored direction after significant grain growth during heat treatment.

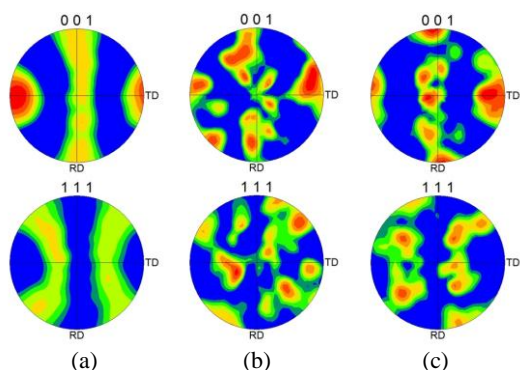


Fig. 4. Pole figure for the inner region of a ZrC layer before/after heat treatment

Fig. 5 shows the misorientation map of ZrC in the misorientation range of  $0 - 5^\circ$  before/after heat treatment. The misorientation information often indicates the residual stress developed in the coating layer [4]. For as-deposited ZrC, the residual stress was developed over the coating layer which may be caused by difference in CTE between ZrC and substrate PyC. The amount of residual stress was reduced by heat treatment.

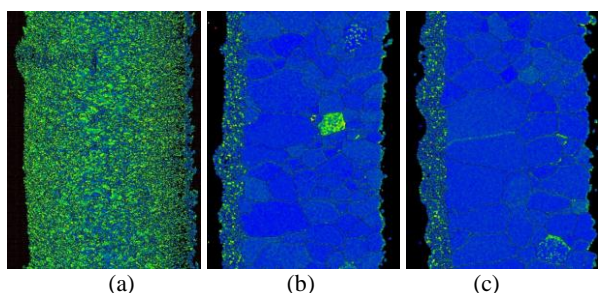


Fig. 5. Kernel misorientation map of the ZrC layers below 5 $^\circ$  of misorientation.

### 3.3 Hardness

It has known that hardness of ZrC depends on the grain size. The relationship between hardness,  $H$ , and grain size,  $d$ , can be expressed by Hall-Petch equation, as follows.

$$H = H_0 + K_H d^{-1/2}$$

where  $H_0$  and  $K_H$  are constants.

Average grain size of ZrC was changed from 0.404 to 2.17 and 3.07  $\mu\text{m}$  after heat treatment at 1800 $^\circ\text{C}$  and 1900 $^\circ\text{C}$ , respectively. As shown in Fig. 6, hardness decreased as the grain size increased according to Hall-Petch relation.

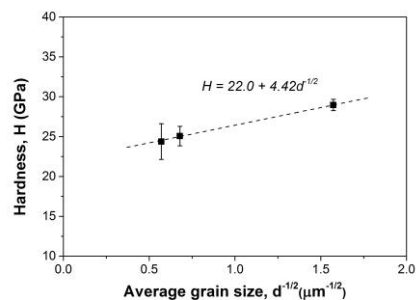


Fig. 6. Relationship between hardness and grain size for ZrC according to Hall-Petch relation.

## 3. Summary

TRISO coated particles with a ZrC barrier layer were fabricated by a FBCVD method using a chloride process. The near-stoichiometric ZrC was obtained but the graphitic carbon as a secondary phase was co-deposited in the outermost ZrC layer. For the near-stoichiometric ZrC, (100) was preferred direction which kept its favored direction after significant grain growth during heat treatment above 1800 $^\circ\text{C}$ . Hardness decreased with increasing the grain size according to Hall-Petch relation. On the other hand, the graphitic carbon co-deposited in the outermost ZrC layer retarded the grain growth of ZrC.

## Acknowledgement

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