Density Measurement of Outer Pyrolytic Carbon layer of Simulated TRISO-Coated Particles with HfO_2 Kernels

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1. Introduction

The TRISO-coated fuel particle for a HTGR(high temperature gas-cooled reactor) is composed of a nuclear fuel kernel and outer coating layers[1,2]. The O-PyC coating layer protects the primary barrier, SiC layer. The density of O-PyC (outer pyrolytic carbon) layer is measured to evaluate the coating layers. The O-PyC fragments are acquired from the broken coated particles. The fragments are so small and irregular that it is not easy to measure the weight and volume of the O-PyC fragments. The density gradient column and standard floats are usually used to measure such a small fragment[3-6]. The density of it can be also measured by micro balance and pycnometer. The other method is to use immersion density of an object with micro balance and cradle. In this experiment, the O-PyC specimens of 3 kinds of simulated TRISO-coated particles with variations of O-PyC layer density according to the process temperature for O-PyC layer were measured by these 3 methods. Simulated TRISO-coated particles contain HfO_2 kernels with similar density to UO_2 kernels.

2. Coating Process Parameters

The process temperature in the coating process of O-PyC layer was adjusted to control the density of O-PyC layer. The temperature was 1400 °C for the coated particles of TR217 batch. The temperature was 1300 °C for the coated particles of TR225 batch to increase the O-PyC layer density. It was 1350 °C for the coated particles of TR226 batch to optimize the density.

3. Density measurement method for O-PyC layer

The model of the density gradient column used for this study is DC/02 made by LLOYD Instruments Ltd.[6] The density gradient solution should be made in consideration of the density of the sample to be measured. We tried to make a solution with density gradient ranging from 1.28 g/cc of lower density to 2.18 g/cc of higher density. Table 1 shows the density of each liquid. 200 cc of gradient solution was made as shown in Table 2. The density of the sample is calculated by the position of the sample in a density gradient column [6].

Table 1. Density of liquid.

Liquid	Density, g/cc
Ethanol	0.79
Dibromoethane	2.18

Table 2. A solution to measure the density of O-PyC layer in a density gradient column.

Lower density, g/cc	1.28
Upper density, g/cc	2.18
Lower density liquid	Ethanol (65 cc) + Dibromoethane (35 cc)
Upper density liquid	Dibromoethane (100 cc)

The O-PyC layer density can be calculated by measuring the weight and the volume of O-PyC layer. The density, d_{opyc}, of O-PyC layer was calculated by measuring the weight, w_{cp}, and the volume, V_{cp}, of a TRISO-coated particle, and the weight, w_c, and the volume, V_c, of the particle without the O-PyC layer removed by the oxidation process at 750 °C for 4 hours by equation (1). Here, the volume of some particles was measured by pycnometer.

$$d_{opyc} = \frac{w_{cp} - w_c}{V_{cp} - V_c} . \quad (1)$$

The weight and the volume of TRISO-coated particles as well as the weight and the volume of coated particles without O-PyC layer can be also measured by an immersion method with micro-balance and cradle. The density of O-pyC layer was also calculated by the equation (1).

4. Density measurement of O-PyC layer

O-PyC fragments were prepared to measure the density of TRISO-coated particles in a density gradient column as shown in Fig. 1. The prepared test samples were put into the density gradient solution of the column. The positions of specimens were measured after some time to come to equilibrium as shown in Fig. 2. The densities of specimens were 1.689 g/cc for TR217, 1.915 g/cc for TR225 and 1.979 g/cc for TR226, respectively as shown in Table 3.

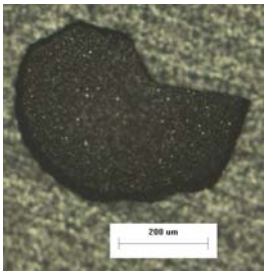

Fig.1. An O-PyC specimen of TR225.

Fig.2. Standard float and O-PyC fragments floated in a density gradient column for TR225 sample batch.

The O-PyC layer was removed by the oxidation process at 750 °C for 4 hours after the weight and the volume of TRISO-coated particles were measured. Then, the weight and the volume of the particles without the O-PyC layer were measured. Here, the volume of some particles was measured by helium gas pycnometer. The weight and the volume of TRISO-coated particles as well as the weight and the volume of coated particles without O-PyC layer can be also measured by an immersion method with ethanol as a solution. Table 4. and Fig. 2 show the results of the measurments.

Table 3. Densities of O-PyC layers.

Method / Coating temperature	Pycnometer (g/cc)	Immersion (g/cc)	Density gradient column (g/cc)
1300 °C (TR225)	1.983	2.118	1.979
1350 °C (TR226)	1.917	1.865	1.915
1400 °C (TR217)	1.623	1.707	1.689

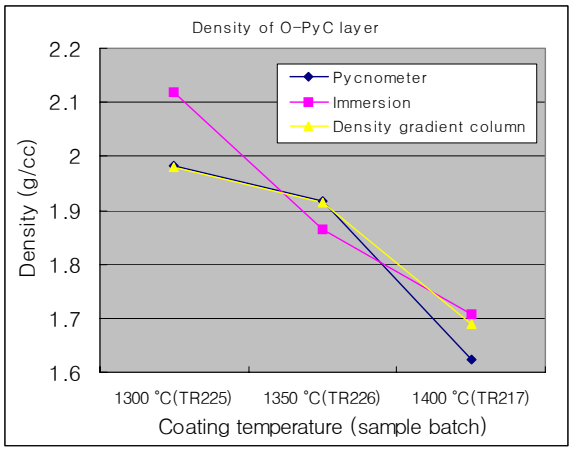

Fig.2. Standard float and O-PyC fragments floated in a density gradient column for TR225 sample batch.

5. Conclusion

In this experiment, the densities of O-PyC layer of TRISO-coated particles were measured by a density gradient column, pycnometer and immersion method. The experimental results are as follows.

- TRISO-coated particles were fabricated according to the coating process temperature.
- Density gradient solution were prepared in the density gradient column for O-PyC sample.
- The volume of particles was measured by helium gas pycnometer or immersion method.
- The density of O-PyC layer increased by decreasing the coating temperature.
- The density of O-PyC layer was 1.92 g/cc for TR226 batch with coating temperature of 1350 °C.
- The density of O-PyC layer will be effectively measured by pycnometer instead of density gradient column which uses toxic liquid and requires O-PyC specimens acquired by careful treatment.
- The coating process for O-PyC layer was established by optimizing the density of O-PyC layer.

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