Density Measurement of Outer Pyrolytic Carbon Layers of Simulated Coated Particles by Using a Density Gradient Column

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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. The coating layers consist of a buffer PyC(pyrolytic carbon) layer, inner PyC(I-PyC) layer, SiC(silicon carbide) layer, and outer PyC(O-PyC) layer[1,2]. The O-PyC coating layer protects the SiC layer which is the primary barrier. The density of O-PyC layer is one of the most important material properties for evaluating the O-PyC layer. The O-PyC fragments are acquired by the broken coated particles. The O-PyC fragments are so small and irregular that it is not easy to measure the weight and volume of the O-PyC fragments. Density gradient column and standard floats can be used to measure such a small fragment as applied to measure the density of SiC layer[3-6]. In this study, the densities of the O-PyC specimens of simulated TRISO-coated particles with ZrO_2 kernel were measured by a density gradient column with a density gradient solution.

2. Density Gradient Column

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. The density of O-PyC layer is often less than 2.1 g/cc. The estimated density of the sample for this experiment was 1.4 g/cc. We tried to make a solution with density gradient ranging from 1.10 g/cc for the lower density to 1.60 g/cc for the higher density for the sample.

Fig.1. An O-PyC sample fragment to be measured by a density gradient column.

100 cc of the lower density solution was made by mixing 66 cc of ethanol and 34 cc of carbon tetrachloride. Here, the density of ethanol is 0.79 g/cc and the density of carbon tetrachloride is 1.60 g/cc. 100 cc of the lower density solution is poured into a flask connected to an outlet to the density gradient column. 100 cc of carbon tetrachloride is a higher density media, which is poured into the other flask connected to the flask with the lower density solution as shown in Fig. 2. Some of the higher density liquid flows into the flask with lower density solution by turning on the interconnecting valve between two flasks. Then density gradient solution is made by opening the outlet tap till the formation of a continuous unbroken flow of liquid through the filling tube in the density gradient column. The formed density gradient ranged from 1.20 g/cc of lower density to 1.50 g/cc of higher density.

Fig.2. A flask with the lower density solution in the right side and a flask with the higher density liquid in the left side to make a density gradient solution.

The density range of the density gradient solution can be evaluated by using standard floats. The densities of the used standard floats were 1.2299 g/cc, 1.3004 g/cc, 1.4006 g/cc and 1.4535 g/cc.

The density D_x of the sample is calculated by equation (1).

$$D_x = a + \frac{(x-y)(b-a)}{(z-y)} \qquad (1)$$

Where a and b are the densities of the two standard floats, which are at distance y and z in vertical position, respectively, and x is the distance of the sample located between y and z.

3. Density measurement of O-PyC layer

O-PyC fragments were prepared to measure the densities of them from the simulated TRISO-coated particles with ZrO_2 kernel instead of UO_2 kernel. The fragments of the broken coating layers usually contain parts of SiC layer and PyC layer. The O-PyC layer fragments have to be selected carefully among the fragments. The prepared test samples were put in the density gradient column. The positions of 5 specimens were measured after some time for the specimens to come to an equilibrium as shown in Fig. 3. The densities of specimens were 1.430 g/cc for sample 1 and 2, and 1.438 g/cc for sample 3, 4 and 5 as shown in Table 1. The average density of the samples was calculated to 1.435 g/cc.

1.4006 g/cc

(sample① sample②)
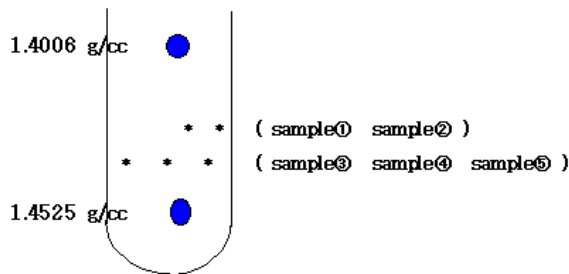(sample③ sample④ sample⑤)

1.4525 g/cc

Fig.3. Standard floats and O-PyC fragments floated in a density gradient column.

Table 1. Densities of O-PyC fragments floated in a density gradient column.

Sample Number	1, 2	3, 4, 5	Average density
g/cc	1.430	1.438	1.435

4. Conclusion

In this study, the density of O-PyC layers of coated particle was measured by a density gradient column with a density gradient solution. The experimental results are as follows.

- The density gradient solution ranging from 1.20 g/cc of lower density to 1.50 g/cc of higher density was made by mixing ethanol and carbon tetrachloride in a density gradient column.
- Test fragments were prepared from the simulated TRISO-coated particles with ZrO_2 kernel instead of UO_2 kernel.
- The positions of 5 specimens were measured after some time to come to equilibrium.
- The average density of the specimens was 1.435 g/cc.

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