Development of a Density Measurement System for Specimens with Open Porosity in a Hot Cell

YoungJun Kim*, Heemoon Kim, Jae Ho Yang, Dong Seok Kim
KAERI, 111 Daedeok-daero 989beon-gil, Yuseong-gu, Daejeon, 34057, Korea
** Corresponding author: yjkim05@kaeri.re.kr*

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1. Introduction

Specimens with open porosity require impregnation of the pores for accurate density measurement using the Archimedes method [1,2]. In this study, a density measurement system applicable to specimens with open porosity was developed based on the Archimedes' principle. The existing Archimedes-based system in the hot cell was supplemented with a vacuum impregnation apparatus to establish a reliable procedure for measuring the density of specimens with open porosity. The system was developed to measure the density of sintered ceramic specimens in a hot cell, and its suitability was verified through density measurements of unirradiated specimens. The developed system was successfully applied to the density measurement of sintered ceramic specimens with open porosity. The results demonstrate the suitability of the developed system for density measurement of the specimens, and the system is expected to be applicable to other specimens with open porosity.

2. System Development and Configuration

As shown in Fig. 1, the existing density measurement equipment installed and operated in the hot cell is based on the Archimedes' principle. The system is equipped with a balance with a maximum readability of 0.01 mg and a remotely controlled vertical movement mechanism, which is designed to maintain a constant specimen immersion depth during measurement.

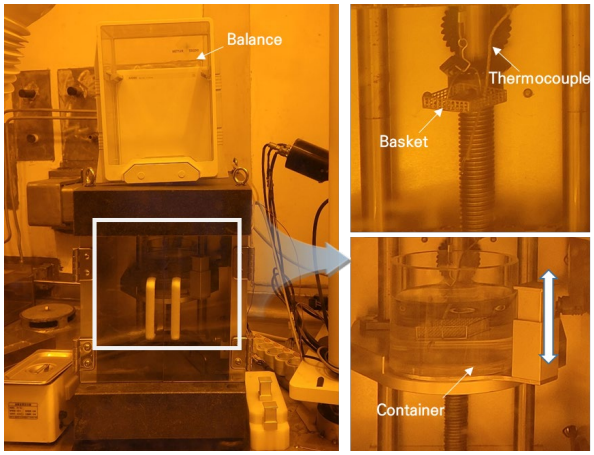


Fig. 1. Density Measurement System in Hot Cell

However, the existing system was not equipped for the determination of density of specimens with open porosity using vacuum impregnation. Therefore, dedicated equipment was developed to facilitate vacuum impregnation and handling of the specimens within the hot cell.

As shown in Fig. 2, the vacuum impregnation apparatus consists of an impregnation tray, a vacuum chamber, and a rotary pump. The apparatus was designed for remote handling within the hot cell and fabricated using transparent materials to allow clear observation of the specimens and impregnation process through the lead-glass window. The impregnation tray was designed to accommodate multiple specimens simultaneously, with separate compartments for each specimen to enable specimen identification. A thermocouple was also installed to monitor the temperature during the impregnation process. The vacuum chamber was designed to maintain the required vacuum level during the impregnation process and was equipped with a vacuum gauge to monitor the vacuum level. The developed apparatus was installed and tested in the hot cell under actual operating conditions. The apparatus was readily handled using the manipulator and successfully operated for vacuum impregnation, confirming its suitability for application within the hot cell.

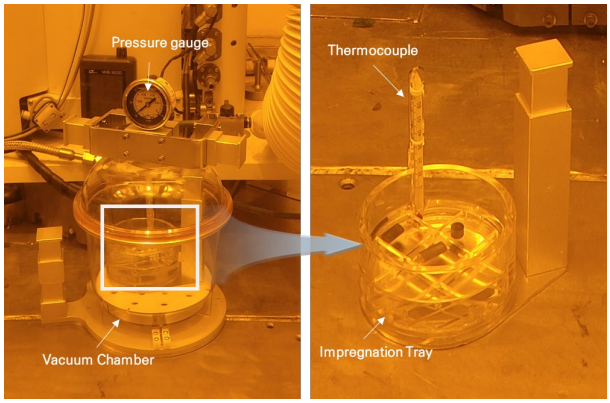


Fig. 2. Vacuum Impregnation Apparatus

3. Results

To verify the suitability of the developed vacuum impregnation apparatus, a commissioning test was performed using an unirradiated sintered ceramic specimen. The specimen was first weighed in air to

obtain its dry mass and then subjected to vacuum impregnation for 30 min using the developed apparatus. After releasing the vacuum, the specimen was maintained in water for at least 30 min to ensure complete saturation. The saturated specimen was then weighed in water, followed by removal of excess surface water and measurement of its saturated mass.

Prior to the measurements, the measurement equipment was verified. The balance was calibrated in-house using E2-class standard weights, and a KOLAS-calibrated thermocouple was used to measure the water temperature. The measured mass and temperature data were used to calculate the density according to Equation (1), and the results are presented in Table 1.

$$\rho = \frac{A}{B-C}\rho_{\omega} \qquad (1)$$

Where ρ_{ω} is the density of water (g/cm^3) at the measured temperature, A is the dry mass of the specimen in air (g), B is the mass of impregnated specimen in air (g), and C is the mass of impregnated specimen in water (g).

Table 1. Density measurement results from the commissioning test

Specimen	Reference Equipment (g/cm^3)	Hot Cell Equipment (g/cm^3)	Difference (g/cm^3)
Sample 1	5.31	5.34	0.03
Sample 2	6.53	6.55	0.02
Sample 3	1.90	1.92	0.02

The density measurement obtained using the developed measurement system showed good agreement with those obtained using the reference measurement system. The absolute differences between the two measurement systems were 0.02–0.03 g/cm³ among the three sintered ceramic specimens. These results demonstrate the suitability of the developed vacuum impregnation apparatus for measuring the density of sintered ceramic specimens.

4. Conclusions

In this study, a dedicated vacuum impregnation apparatus was developed for the density measurement of porous ceramic specimens within a hot cell. The existing Archimedes-based system was supplemented with this apparatus to establish a reliable procedure for measuring the density of specimens with open porosity. Commissioning tests showed good agreement with the reference equipment, with absolute density differences of 0.02–0.03 g/cm³. The developed system provides a practical method for density measurement of sintered ceramic specimens with open porosity during post-irradiation examination (PIE).

REFERENCES

[1] ASTM B962-23, Standard Test Methods for Density of Compacted or Sintered Powder Metallurgy (PM) Products Using Archimedes' Principle.

[2] ISO 18754:2020, Fine ceramics (advanced ceramics, advanced technical ceramics) – Determination of density and apparent porosity.