Fabrication of HTGR Fuel Compacts by Graphite Powder Compaction

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Keywords : HTGR, Fuel Compact, TRISO

1. Introduction

In this work, graphite matrix powder for block-type HTGR fuel applications was produced using a jet-milling-based process derived from the A3-27 methodology. Novolac resin combined with an externally supplied curing agent was employed to ensure improved storage stability and reduced contamination during powder preparation. The jet-milling process enabled effective particle size refinement through inter-particle collisions, providing a homogeneous powder suitable for compaction studies.

This study focused solely on the consolidation behavior of the graphite matrix powder itself. Warm pressing was conducted under various temperature and pressure conditions to examine their influence on densification characteristics. The green compacts were subsequently subjected to thermal treatments, including carbonization and high-temperature purification, to simulate the matrix processing sequence used in HTGR fuel fabrication.

Bulk density measurements were performed after purification to evaluate the effects of compaction parameters and heat treatment on final matrix density. Through systematic variation of pressing temperature and pressure, the relationship between processing conditions and densification behavior was analyzed to establish optimal compaction parameters for future HTGR fuel compact manufacturing.

2. Methods and Results

2.1 Matrix Graphite Powder Production with Jet-Milling

Natural graphite, artificial graphite, novolac resin, and hexamethylenetetramine (HMTA) were combined according to the weight fractions listed in Table 1. The blended powders were mixed in a three-dimensional mixer for 5 hours to achieve uniform distribution of the resin and curing agent throughout the graphite matrix.

Pulverization was carried out using a 2-inch jet milling system (Jet Pulverizer Co.), as shown in Figure 1. To obtain fine and contamination-free graphite powders suitable for matrix fabrication, the milling process was performed under a fixed set of operating conditions. The powder was supplied to the milling chamber at a constant feed rate of 1 g/min, while the grinding pressure was maintained at 8 bar. These processing conditions were selected to produce a uniformly refined powder with controlled particle size characteristics.

Figure 2 presents the SEM image of the jet-milled graphite powder. The micrograph shows uniformly refined particles with irregular morphology typical of jet-milled graphite. Particle size distribution analysis indicated an average particle size of 2.9 μm, while the D90 value was measured to be 5.1 μm, confirming that the majority of the particles were within the fine size range suitable for compaction.

Natural graphite	Artificial graphite	Novolac Resin	HMTA
64 wt%	16 wt%	19 wt%	1 wt%

Table 1 Mixing ratio of law materials powder for jet-milling

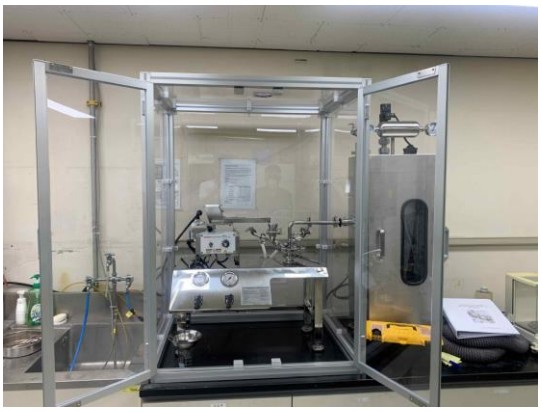

Figure 1. 2-inch jet-milling equipment

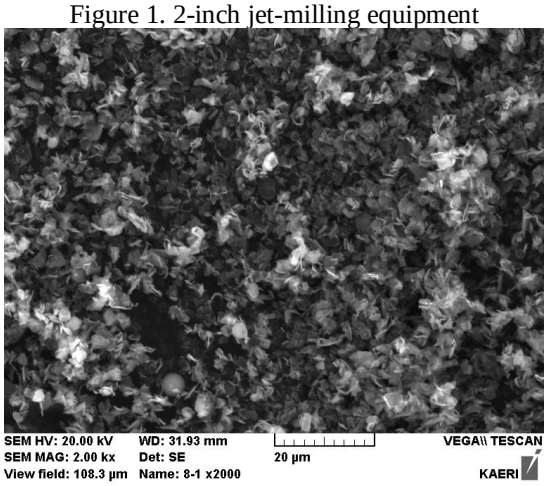

Figure 2. Jet milled matrix graphite powder

2.2 Fuel Compaction Fabrication

Compaction was performed at three temperature conditions: room temperature, 100 °C, and 170 °C. For each temperature, pressures of 18.8 MPa, 37.7 MPa, 56.5 MPa, and 75.4 MPa were applied. During warm pressing, a thermocouple was inserted at the contact interface between the mold and the specimen to accurately monitor the processing temperature.

Following compaction, a multi-step heat treatment was conducted. The temperature was increased from room temperature to 800 °C at a slow heating rate of 0.71 °C/min and held for 2 hours to promote carbonization of the novolac resin. The deliberately low heating rate was adopted to mitigate crack formation caused by gas release during resin curing and decomposition. After carbonization, the temperature was further elevated to 1800 °C at 5.92 °C/min and maintained for 2 hours specifically to achieve high-temperature purification of the graphite matrix by removing residual volatiles and impurities. Finally, the specimens were cooled to room temperature at a rate of 7.4 °C/min.

The densities of the specimens after compaction and subsequent heat treatment are summarized in Figure 3 and Table 2. In general, higher compaction pressure resulted in increased bulk density. However, a reduction in density was observed after heat treatment due to mass loss associated with resin decomposition and purification. Specimens pressed at elevated temperatures exhibited relatively smaller density decreases after heat treatment. This behavior is attributed to partial curing of the novolac resin during warm pressing, which allowed volatile gases to be released prior to the high-temperature carbonization step, thereby reducing additional mass loss during subsequent heat treatment. According to the AGR-5/6/7 fuel specification, the required minimum fuel compact density is 1.65 g/cm³. [2] Among the tested conditions, only the specimen compacted at 170 °C under 75.4 MPa satisfied this density criterion after heat treatment.

After sectioning, the specimens were mechanically polished, followed by ultrasonic cleaning to remove residual debris generated during the polishing process. This procedure was conducted to prevent polishing dust from being trapped within surface pores, which could obscure the true microstructural features and lead to underestimation of porosity. Figures 5 and 6 show optical microscopy (OM) images at 200× magnification of the specimens compacted at 170 °C and subsequently heat-treated. The horizontal surface, corresponding to the punch-contacted face, and the vertical surface, representing the cylindrical sidewall, are presented for comparison. The specimens pressed at higher pressures, particularly 56.5 MPa and 75.4 MPa, exhibit noticeably reduced porosity compared to those compacted at lower pressures. This observation is consistent with the measured density results, confirming that increased compaction pressure enhances densification even after heat treatment. No distinct anisotropy associated with the pressing direction was observed between the punch-loading direction and the radial direction. The pore distribution and microstructural characteristics were comparable on both the punch-contacted surface and the cylindrical side surface, indicating that directional effects induced by the applied punch pressure were minimal.

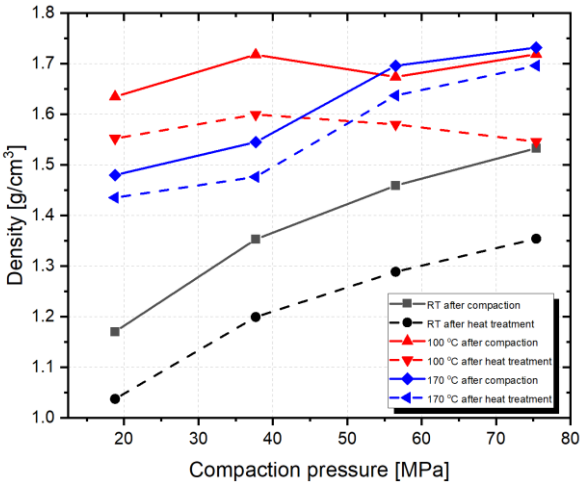

Figure 3. Density of fuel compacts after compaction and heat treatment

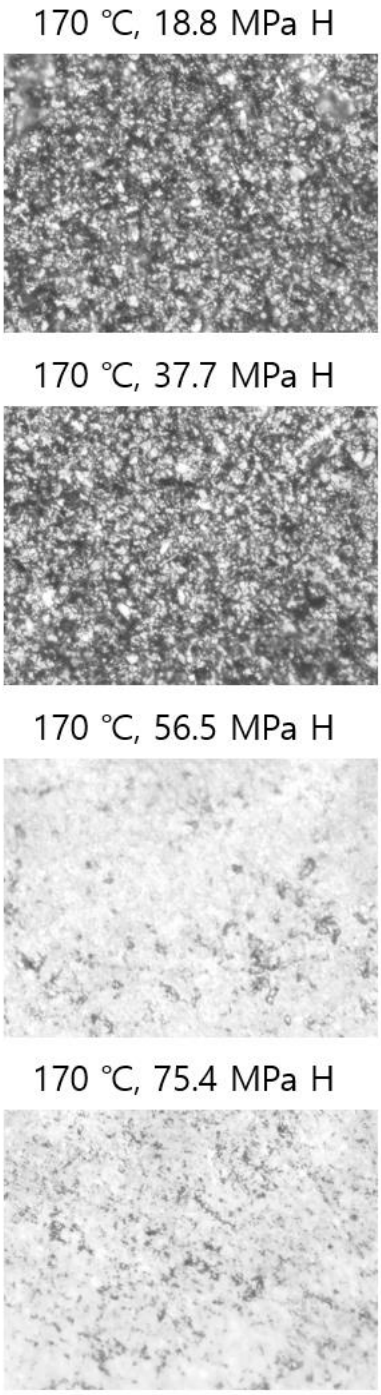

Figure 4. Horizontal surface of the compact pressed at 170 °C and subsequently heat-treated

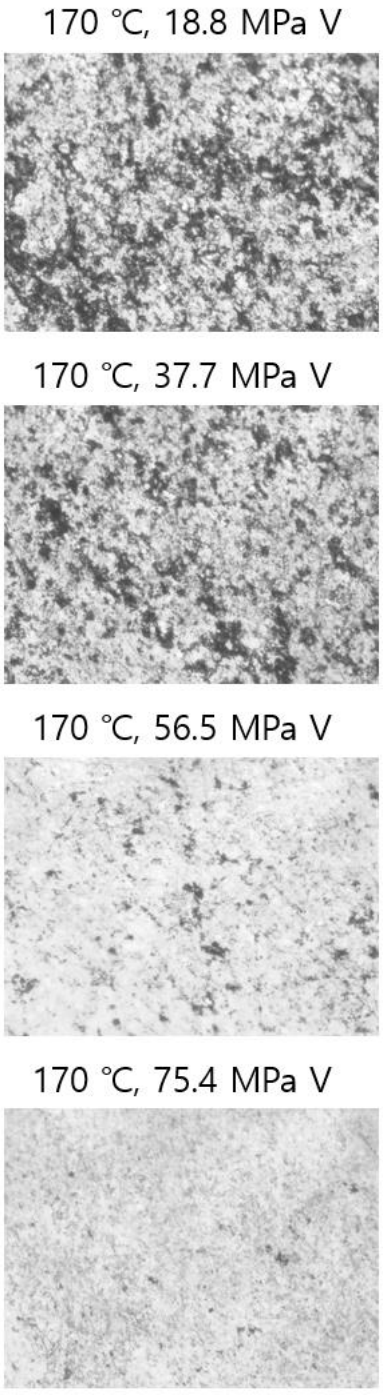

Figure 5 Vertical surface of the compact pressed at 170 °C and subsequently heat-treated

3. Conclusions

In this study, graphite matrix powder for HTGR fuel compact applications was successfully prepared using a jet-milling process based on the A3-27 methodology. The refined powder exhibited a fine particle size distribution, with an average particle size of 2.9 μm and a D90 of 5.1 μm, demonstrating its suitability for compaction.

Warm pressing under various temperature and pressure conditions revealed that compaction pressure is the dominant factor governing densification. Higher applied pressures resulted in increased bulk density and reduced porosity. Although density decreased after heat treatment due to resin carbonization and purification, specimens pressed at elevated temperatures showed relatively smaller density reductions, attributed to partial curing and pre-release of volatiles during warm pressing.

Microstructural observations confirmed that increased compaction pressure led to a significant reduction in pore fraction, while no noticeable anisotropy associated with the pressing direction was observed. Among the investigated conditions, only the specimen compacted at 170 °C under 75.4 MPa satisfied the AGR-5/6/7 fuel specification requirement of a minimum compact density of 1.65 g/cm³ after heat treatment.

These results indicate that optimized warm pressing conditions are essential to achieve the required densification of graphite matrix compacts for future HTGR fuel fabrication.

Acknowledgement
This work was supported by the Korea Atomic Energy Research Institute R&D program (Contract No. 521410-26)

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