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ABSTRACT
The changes in mechanical properties and thermal conductivities of fiber reinforced foam glass produced by adding 0 ~ 50 wt% basalt-fibers to 10 µm glass powder containing 6 wt% nano-carbon via low-temperature sintering at 750 °C were examined. The prepared basalt-fibers had an aspect ratio of 70.88. To study the microstructure of the foam glass, optical microscopy and scanning electron microscopy were conducted. To assess the mechanical properties and thermal conductivity of the sample, a compressive strength tester and a heat flowmeter were used, respectively. The sample was confirmed to have closed pores up to the addition of 15 wt% basalt-fiber, with open pores being observed from 20 wt% or more. Microstructure analysis showed that the sample was shown to have dense basalt-fiber clusters with an even distribution of pores at 13 wt% basalt-fibers, whereas irregular pores formed with more basalt-fiber addition. Compressive strength was increased by approximately 48 % compared to the sample without basalt-fiber addition while the sample with 13 wt% basalt-fibers showed gradual improvement up to a total of 2.96 MPa and then a gradual decrease with more addition. The thermal conductivity was seen to increase from 0.16 to 0.28 W/m·K with increasing basalt-fiber addition; nevertheless, the properties were suitable for using the samples as a thermal insulator.

1. Introduction
Foam glass is a form of glass with closed and open pores present within its structure. Additionally, foam glass is a heterophase system comprising a glassy solid and a gaseous phase, where gaseous pores are surrounded by solid walls [1]. Foam glass has advantages of greater corrosion resistance and thermal stability than those of existing porous polymers [2]. It exhibits low thermal conductivity and thermal expansion and excellent thermal stability and acoustic absorption properties, leading to its current applications for lining the interior of flue gas desulfurization (FGD) system smokestacks in thermal power plants and thermal insulation for buildings. However, problems such as destruction during construction or from operational thermal shock due to its poor durability because of its low strength and increasing manufacturing costs arising from expensive equipment and high-temperature processing have been reported [3]. To address these issues, studies are actively being conducted to improve foam glass strength via fiber addition and low-temperature foam glass manufacturing [4]. By introducing basalt-fiber layers to concrete, Sim et al. [5] observed an increase of 27 % in strength with the addition of three layers, while Song et al. [6] observed an approximate increase in strength of 12 %, from 85 MPa to 96 MPa, by adding 2 vol% steel fiber. For foam glass, H. W. Guo et al. [4] added 25 wt% glass fibers to improve the strength of foam glass and observed an increase of 13% in strength. By adding 15 wt% mullite fibers to foam glass, H. W. Guo et al. [7] reported an approximate increase of 53 %, from 5.3 MPa to 8.12 MPa, over standard foam glass. Basalt-fiber is produced by melting basalt rocks and extracting fibers; its tensile strength is higher than that of glass fiber by 24 % [8]. In addition, basalt-fibers have the advantage of better corrosion resistance than carbon fibers and do not generate toxic gases in high-temperature fires [5]. However, as of now, studies on the use of basalt-fibers in foam glass manufacture are lacking, while the aforementioned use of glass fibers in foam glass has the issue of potentially lowering the mechanical strength of the foam glass because of their similar melting temperatures, leading to pore deformation following the orientation of fibers. Furthermore, fibers such as mullite, sepiolite, ZrO2, and YSZ have high material costs, making their application difficult in the industrial field. To overcome these issues, basalt-fiber was selected for its more competitive price and ability to improve mechanical strength. By using basalt-fiber, we can expect excellent price competitiveness and improvement in strength in the future. Therefore, basalt-fibers were added to foam glass in this study, which has not been attempted previously, to address the strength shortcomings of foam glass, while observing changes in material properties such as microstructure and thermal conductivity.
2. Experimental procedure

To study the characteristics of foam glass manufactured from differing raw material particle sizes, 2 mm diameter bulk glass consisting of 82.64 wt% SiO$_2$, 9.43 wt% CaO, 4.91 wt% MgO, 2.18 wt% SO$_3$, and 0.84 wt% Al$_2$O$_3$ and 35 nm nano-carbons were prepared as raw materials. In case of glass powder having 626 °C of glass-transition temperature, the powder was purchased from the Korea Rose Association. Pulverization of the prepared bulk glass was achieved by adding Al$_2$O$_3$ balls in diameter to a jar of a planetary ball mill and dry grinding at 450 rpm for 6 h. Following pulverization, a sieve was used to obtain uniform size glass powder of 300 µm or smaller. We added 6 wt% nano-carbon to the pulverized glass powder, along with Al$_2$O$_3$ balls, and 50 ml of isopropyl alcohol (IPA) and then mixed by ball milling for 24 h at 250 rpm. This mixed sample was dried in an oven at 80°C for over 24 h to remove the solvent. The dried sample was turned back into a powder by ball milling at 250 rpm for 15 min. During this process, the basalt-fibers were inspected to ensure their aspect ratio and shape were maintained. The prepared sample was low-temperature sintered in a furnace at 750 °C to form pores in the mixed sample. The sintering process consisted of heating and sintering to 400 °C at a rate of 3 °C/min, holding at 400 °C for 60 min, heating to the maximum temperature of 750 °C at a rate of 10 °C/min and holding for 60 min, and then cooling at a rate of 1 °C/min. To check the pore shapes of the sintered samples, 50 µl of H$_2$O was dropped on the specimen surface using a micropipette. After waiting for 1 min, the shape and height of the droplet were observed using a macro camera to verify whether it is an open pore or closed pore. The produced foam glass was shaped into a block of 8, 8, and 6 mm in width, length, and height, respectively, to calculate its density by analyzing the total volume compared to the mass. At this point, the mass was measured using a microbalance with an accuracy of up to four decimal points. Afterward, the finished sample was polished with sandpapers to observe the microstructure and pore sizes. These observations were made using an optical microscope (AX-10, ZEISS) and a GIA microscope. In addition, a field emission scanning electron microscope (FE-SEM, S-4300, Hitachi) was used to confirm the surface microstructure of the fabricated specimen. Furthermore, to determine the aspect ratio (average length of the pore major axis/average width of the pore minor axis) of pores in the produced specimen, an image analysis program (iSolution DT x64 (IMS digital)) was used to analyze the 50x magnified cross-sectional image from an optical microscope. The mechanical properties of these samples were tested by assessing compressive strength in an Instron 3344 (INSTRON Co.). These tests were conducted at a strain rate of 50 mm/min with 4 × 4 × 8 (±0.2) mm sized samples. The results were analyzed by averaging across three or more specimens tested with identical methods. To study the insulation properties of the sintered sample, an HFM (Heat Flow Meter, NETZSCH Co.) was used to analyze thermal conductivity. Thermal conductivity was obtained by averaging three measurements made in an Ar atmosphere using a 260 voltage and 600 µs pulse width laser.

3. Results and discussion

The results of the H$_2$O droplet tests on the foam glass surface are shown in Figure 1(a). Closed pores were observed at 0 wt% basalt-fiber and open pores were observed above 20 wt%. Between 10 and 15 wt%, a mixture of closed and open pores was observed. This was probably caused by the increasing amount of basalt-fibers affecting pore connections and the aspect ratio of closed pores, a process gradually leading to open pores. Additionally, Figure 1(b) shows the density of the
produced specimens. The 50× magnified cross-sectional images of 0, 13, 20, 30, 40, and 50 wt% basalt-fiber-added samples are shown in Figure 2. The pore shapes changed with the increasing addition of basalt-fibers. Figure 2(a) shows the image of a sample with only 6 wt% carbon added and with no basalt-fiber, which showed spherical closed pores approximately 300-µm in size in the foam glass. Figure 2(b) shows a sample with 13 wt% basalt-fiber addition. Following the introduction of basalt-fibers, the shape of the pores was seen to change from spherical to elliptical. As indicated by the dotted circle, it was also confirmed that the basalt-fibers were evenly dispersed. Figure 2(c) shows the cross section of a sample with 20 wt% basalt-fiber added and confirms that the increasing of basalt-fiber content produces elliptical pores. Furthermore, the area indicated by the dotted circle shows areas concentrated with basalt-fibers, which was deemed the cause of open pores verified in the previous pore confirmation test. Figure 2(d-f) each show cross-sectional images of 30, 40, and 50 wt% basalt-fiber addition, respectively. Increasing amounts of basalt-fiber addition was confirmed to form elliptical pores as the major axis length increases; in addition, basalt-fibers present in high-density clusters in the area indicated by the dotted circle were also shown. This entangled cluster was seen to grow with the increasing addition of basalt-fiber. Therefore, it was deemed that a greater than 20 wt% addition of basalt-fibers contributes to pore shape change through coalescence and connection of pores with adjacent pores during the formation process, leading to closed pore foam glass becoming open-pore foam glass.

Figure 3 shows the microstructure of samples with 13 wt% and 50 wt% basalt-fiber addition shown in

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**Figure 2.** Optical microscopy images of samples with basalt-fiber addition of (a) 0 wt%, (b) 13 wt%, (c) 20 wt%, (d) 30 wt%, (e) 40 wt%, (f) 50 wt%.
Figure 2. Figure 3(a) on the left shows the microstructure of a specimen with 13 wt% basalt-fiber added, which verifies that mixed fibers are present in the pore walls between the foam glass pores. On the contrary, Figure 3(b) on the right shows the microstructure of a sample with 50 wt% basalt-fiber addition, which shows entangled areas due to excessive basalt-fiber addition. These fiber entanglements interfere with the formation of uniform pores, weakening pore walls and ultimately lowering strength. These results are consistent with those of Lang et al. [9] who discovered that strength decreases with excessive addition of Al$_2$O$_3$ fibers. Therefore, 13 wt% basalt-fiber addition produces uniform mixing within the pore walls and forms spherical closed pores, as previously verified in the microstructure study.

Figure 4 shows the aspect ratios of the major/minor pore axes and pore size according to different amounts of basalt-fiber shown in Figure 2. Figure 4(a) confirms that the aspect ratios of the pores in the produced specimens increase with increasing basalt-fiber addition. This increase in basalt-fiber was determined to be the reason behind the deformation of spherical pores as it interferes with pore formation. Figure 4 (b) confirms pore size according to the addition of basalt fibers. The pore size was between 450 ~ 550 µm regardless of the amount of basalt-fiber addition. These results were consistent with the microstructure results inconfrmed that aspect ratio increases while pore size is maintained with basalt-fiber addition.

Figure 5 shows compressive strength according to the additional amount of 0 ~ 50 wt% basalt fiber, and precise compressive strength in the range of 10 ~ 15 wt% was shown in a dotted circle on the upper-right. Compressive strength was seen to increase up to 13 wt% basalt-fiber addition, with greater than 13 wt% addition, resulting in a decrease in compressive strength. Up to 13 wt% basalt-fiber addition, the compressive strength is known to increase because of pull-out effects and crack deflection from adding fiber to the ceramic material. The pull-out effect is a phenomenon in which the ceramic strength is enhanced as stress from an applied external force is distributed by the large surface area of the fibers. Crack deflection is a form of crack blunting, whereby a crack tip in a matrix encounters a fiber surface, then stops propagating. Such improvements in strength because of the pull-out effect and crack deflection have been previously verified by Y. L. Zhang et al. [10], who observed a 25 % improvement in mechanical strength when a SiC and carbon fiber composite was added to Al$_2$O$_3$ and La$_2$O$_3$. It is also consistent with Logesh et al. [11] who found a 35 % improvement in compressive strength, up to 437 MPa, when 5 wt% carbon fiber was added to a silicon nitride...
matrix. When more than 20 wt% basalt-fiber was added, as shown in Figure 1, the pore volume increased while the amount of closed pores decreased, resulting in reduced compressive strength. In addition, as confirmed by the microstructures seen in Figure 2 and Figure 3, the increase in the amount of basalt-fiber-added results in excessively entangled clumps with non-uniform distribution and crossing fibers. These areas act as defects in the foam glass, which lower the compressive strength by becoming initiation points for microcracking. This is consistent with findings by Fang et al. [12] which determined that adding more than 3 vol% glass fiber to a cement matrix results in reduced compressive strength because of the uneven distribution of fibers causing concentration of stress and the comparatively lowered cement density acting as a defect source. Therefore, compressive strength increases when 13 wt% basalt-fiber was added as the external loads are dispersed by the fibers, whereas compressive strength decreases with more than 13 wt% basalt-fiber. Over the addition of basalt-fiber results in forming irregular pores, weakening pore walls, and eventually lowering compressive strength. We confirmed that a foam glass performs the role of lining materials at thermal power plants as the foam glass showed with 48% enhanced compression strength by the addition of 13 wt% basalt-fiber.

Figure 6 shows the thermal conductivity relative to the basalt-fiber addition amount. This verified that thermal conductivity increases with greater addition of basalt-fiber because the thermal conductivity of basalt-fiber, 0.8 W/m·K [13], is comparatively higher than that of foam glass, 0.115 W/m·K [14]. Thus, an increase in basalt-fiber addition results in an increase in thermal conductivity. This result is consistent with the increase in density due to the addition of basalt-fibers, as previously identified, and the increase in area where basalt-fibers in the microstructure were connected or concentrated. Furthermore, foam glass with 50 wt% basalt-fiber added showed the greatest thermal conductivity of 0.28 W/m-K, which is below the threshold value of 0.33 W/m-K for building thermal insulation material, as reported by Yun et al. [15], indicating that all basalt-fiber reinforced foam glass examples proposed in this study have suitable properties to be used as a fireproofing material. Therefore, by adding an appropriate amount of basalt-fiber to existing foam glass and performing low-temperature sintering at 750 °C, it was possible to produce a new fireproof compatible foam glass that has improved mechanical properties and whose thermal conductivity characteristics were not significantly changed.

4. Conclusions
Basalt-fiber between 0 and 50 wt% was added to 10 µm glass powder containing 6 wt% nano-carbon and was low-temperature sintered at 750 °C to produce reinforced foam glass. Addition of up to 13 wt% basalt-fiber in foam glass produced closed pores, while all foam glasses with greater than 20 wt% basalt-fiber addition showed open pores. The microstructure results showed uniform fiber distribution with 13 wt% basalt-fiber addition, while a greater amount of basalt-fibers addition showed entangled fibers. The aspect ratio was also seen to increase from 1.25 to 1.78 as basalt-fiber addition increased compared to samples without basalt-fiber, and the compressive strength increased by 48% to 2.96 MPa in samples with 13 wt% basalt-fiber addition. The thermal conductivity with increasing addition of basalt-fiber ranged between 0.16 and 0.28 W/m-K; all values are within the suitable range for

Figure 5. Compressive strength relative to basalt-fiber addition.
use of the foam glass as a thermal insulator. Therefore, we propose a foam glass fabrication process with appropriate basalt-fiber addition and low sintering temperature that shows improved compressive strength and reasonable thermal conductivity.

References