Observation of Pore Size Change after Hot Isostatic Pressing of Vitrified Sludge Waste

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1. Introduction

The compression molding vitrification properties of sludge generated after decontamination for recycling radioactively contaminated tanks generated during the decommissioning of nuclear power plants were analyzed. The sludge accumulated in the radioactively contaminated tank was exposed to a relatively short vitrification temperature to vitrify it, and then Hot Isostatic Press (HIP) was used to perform compression molding. In general, when high temperature exposure occurs for a short time, it becomes a sintered state, which is the stage before vitrification. The sintered sludge through the Hot Isostatic Press process is compressed and molded for 2 hours, so the process time to make solidification is reduced and the volume of the sludge solidification is reduced compared to the existing vitrification process. Through visual observation, the volume of the vitrified sludge was reduced. In the future, the volume reduction will be evaluated using a 3D scanner. In addition, it is expected that the physical and chemical properties of vitrified sludge will change after porous removal. The size of vitrified sludge pores before and after Hot Isostatic Press was observed.

2. Methods and Results

When decommissioning a nuclear power plant, radioactively contaminated tanks of various material are generated, and sludge is generated according to the use and corrosion of the radioactively contaminated tank. For volume reduction of radioactively contaminated waste generated during decommissioning, metal recycling can be performed after removing corroded oxides and sludge from the contaminated tank. Inside the radioactively contaminated tank, there is a sludge deposited, which has various components depending on the use of the tank. The characteristics of wastewater flowing into the tank and the source of sludge that can be introduced along with the wastewater were analyzed to confirm the characteristics of the internal accumulated sludge of the radioactively contaminated tank used in the steam supply system. The weight percent of sludge is consisted of Fe_3O_4 (56 wt%) and NiO (44 wt%) [1]. In the case of sludge waste generated from radioactive contamination tanks, the contamination level is expected to be low level waste, but conditioning to remove mobilization is essential. Therefore, stabilization and solidification were performed with vitrification.

2.1. Vitrification composition and conditions

Vitrification is the binding of harmful substances in an amorphous structure. The composition of the vitrification and sludge is shown in Table I. The weight composition of the vitrified substance was determined by referring to the composition when vitrifying the metal oxide [2]. NiO and Fe_2O_3 have relatively high melting points. The vitrification temperature may vary depending on the melting point of the target material. B_2O_3 and Na_2O were added to lower the vitrification temperature. The ratio of vitrification substance to sludge waste is 2 to 1.

Table I: The composition of vitrified sludge

	Substance	Weight percent (wt %)
Vitrification substance	SiO_2	38.71
	Na_2O	9.68
	Al_2O_3	9.68
	B_2O_3	9.68
Sludge	Fe_3O_4	18.06
	NiO	14.19

In general, the vitrification time for producing glass is exposed to a vitrification temperature of 1000 ℃ or higher for about 8 hours to produce a glass. In this study, compression molding was performed with HIP after vitrification generation at 1150°C for 2 hours. Although it is a short vitrification generation time, it can satisfy the disposition criteria, and rather than making perfect glass, it focused on solidification that satisfies the disposition criteria. Additionally, pore removal was performed by HIP compression molding, which can result in volume reduction.

2.2 Compression molding using Hot Isostatic Press

The principle of hot isotropic press (HIP) is to fill in argon gas, which is an inert gas, and then perform isostatic pressing on all areas in contact with the gas. When isostatic pressing is performed, exposing the target material to a high temperature and applying force does not cause damage, but allows pore removal and volume reduction. Based on empirical experiments, vitrified waste was exposed at 700 ℃ and 4000 psi for two hours, including a temperature and pressure rising time.

2.3. Vitrified sludge pore image

After HIP compression molding, it was confirmed that the volume was reduced by visual observation. The pore size of the vitrified waste was observed through a SEM. The vitrification before HIP molding had a large number of pores about 1 mm in diameter could be seen with the visual observation. Before HIP compression molding, pores exceeding 400 μm were easily observed from small pores with 7 μm as shown in Figure 1. After HIP compression molding, as shown in Figure 2, there were relatively few pores found, mainly showing a smooth surface.

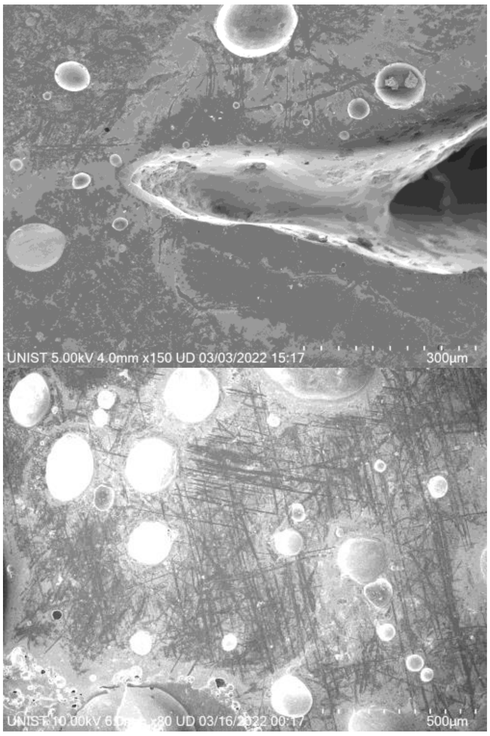

Fig. 1. Vitrified sludge pore SEM image before Hot Isostatic Press molding

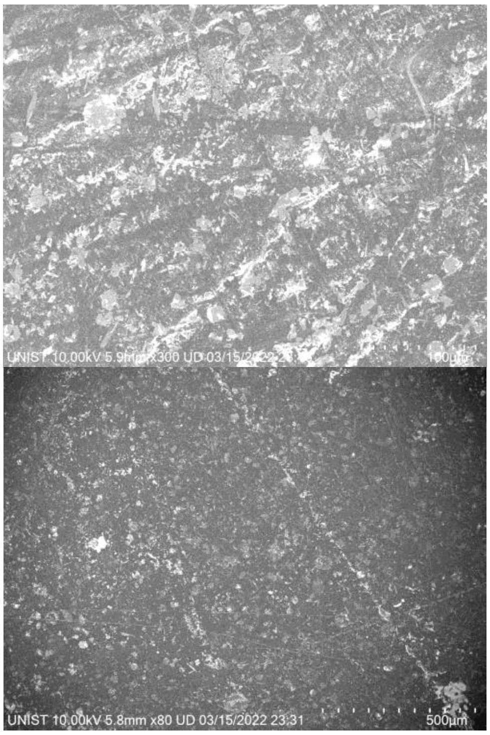

Fig. 2. Vitrified sludge pore SEM image after Hot Isostatic Press molding

3. Conclusion

Macroscopic volume reduction was confirmed through the HIP process by visual observation, and a decrease in the size and number of randomly distributed pores was confirmed by SEM images. Volume reduction through the HIP process is planned to confirm using a 3D scanner. It is difficult to prove porous removal of vitrified sludge with only partial SEM images. Therefore, it is planned to prove this by observing the change in pore distribution through Observation of pore distribution using Mercury Intrusion Porosimetry (MIP) analysis.

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REFERENCES

[1] Dong Youn Kim, Eun Hye Lee, Shang Woon Shin, “Analysis Report of Characteristics of Sludge from Dismantling of Radioactively Contaminated Tanks”, Nile Plant, TC-2007010, 2020. 8

[2] Vienna, John D. "Nuclear waste vitrification in the United States: recent developments and future options." International Journal of Applied Glass Science 1.3 (2010): 309-321.