3D X-rays application for precision measurement of the cell structure of extruded polystyrene

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Abstract. While the thermal performance of existing insulation materials have been determined by blister gases, the thermal performance of future insulation materials will be dependent on the cell size and independent foam content as we use eco-friendly blister gases with a higher thermal conductivity. However, with the current technology we are only able to guess the whole cell size and independent foam content through SEM applied 2D fragmentary scanning but are still far from the level of accurate cell structure data extraction. Under this situation, we utilized X-ray CT scanned 3D images to identify and shape the cell structure and proposed a method of inferring the whole distribution and independent foam content as accurately as possible. According to X-ray CT scanning images and SEM images, the shape was similar but according to tracer applied CT scanning images, the cell size distribution was 380–400 μm within the range of the general insulation diameter distribution which had the highest reliability. As for extrusion foaming polystyrene, we need additional image processing to identify the independent foam content as its density is too low. So, it is recommended to raise the 3D cell structure completeness of XPS by improving the scanning accuracy.

1. Introduction

In 1992, Korea officially signed the Montreal Protocol as a developing country and the ozone layer depleting substance reduction schedule was fixed separately for advanced countries and developing countries, starting from the substances having the ODP index of 1.0. In case of CFC compound, according to the total abolition schedule by the end of 2009, the extruded polystyrene industry of Korea began CFC-12 reduction and total abolition from 1996 for the first time in the domestic industry but started to use HCFC foaming agents, a potential substitute under the Protocol [1].

As for HCFC foaming agents, the average amount of production and consumption in Korea were fixed in 2009 and in 2010 respectively, and actual reduction of them is scheduled to commence from the beginning of 2015 [2]. Previously, Korea adopted the quota system in 2014 and has executed the reduction schedule according to the agreement of staged HCFC compound reduction, by 35% until 2020, by 67.5% until 2025, and total abolition until 2030.

Under the Montreal Protocol reduction schedule, Korea is obliged to complete total abolition by 2030, starting from 10% new of XPS insulation
development based on eco-friendly foaming agent while we are maintaining the existing thermal conductivity of XPS or securing better thermal performance [3].

To use blister gases having a higher thermal conductivity as an alternative, we must not only have a discussion on the method of upgrading the insulation performance to enter the existing insulation market but also study the cell size and independent foam (Closed Cell) whose thermal conductivity is calculated by the Knudsen Effect theory. Accordingly, in this paper we observed the insulation cell structure using X-ray applied 3D and compared the result with the existing 2D scanning images in order to study the insulation X-ray scanning method which is used for the observation of cell size and independent foam.

2. Relation between cell structure and thermal conductivity

Knudsen Effect, the basic foundation concept of Aerogel which is now taking the center stage as the next generation insulation says the gas based thermal conductivity will significantly decrease due to lower adiabatic gas liquidity in a foam if the foam size is below $70 \mu m$ [4]. In other words, if we reduce the foam size to the nanometer level, we are able to reduce the thermal conductivity innovatively thanks to radiant heat, gas convection, and conduction. As a result, total thermal conductivity of the polymeric foam ($\lambda_{\text{total}}$) can be expressed in equation (1) as the sum of the three contributions: thermal conductivity through gas phase ($\lambda_{\text{gas}}$) and solid phase ($\lambda_{\text{solid}}$), and radiation ($\lambda_{\text{rad}}$):

$$\lambda_{\text{total}} = \lambda_{\text{gas}} + \lambda_{\text{solid}} + \lambda_{\text{rad}}$$

The plastic foam insulation has a thermal conductivity of about 0.03 W/m·K. According to the Almanza study, the thermal conductivity of low-density Polyolefin foam whose cell size exceeds 100μm is known to be determined by heat radiation (20–40%), the thermal conductivity of plastic (10–15%), and the adiabaticity of blister gases injected in the foamed plastic (40–65%) [5, 6]. However, as shown on Fig 1, we can see its insulation performance is lower than that of R134a in HCFC series, the existing foaming agent because it has a higher temperature based thermal conductivity of CO$_2$ [3].

![Figure 1. Gaseous thermal conductivity](image)

As thermo-plastic resin foaming is greatly dependent on the thermal conductivity of foaming agent (blister gas), we need the technology of lowering the ultimate thermal conductivity by reducing the cell size for commercialization because the thermal conductivity tends to increase when we replace the existing HCFC blister gas with blister gases having a higher thermal conductivity, for example, CO$_2$ gas.
3. 3D X-ray application for organic insulation cell structure measurement

While the thermal performance of existing insulations have been determined by blister gases, the thermal performance of future insulations will be dependent on the cell size and independent foam content as we use eco-friendly blister gases with a higher thermal conductivity. However, with the current technology we are only able to guess the whole cell size and independent foam content through SEM applied 2D fragmentary scanning as showing Fig 2 but still distant from the level of accurate cell structure data extraction. To keep up with the tendency of NIMs development for insulation, we utilized X-ray scanned 3D images to identify and shape the cell structure to propose the method of calculating the whole cell size distribution and independent foam content as accurately as possible.

![Figure 2. Foam morphology of PS/CNT 0.25 wt%](image)

The X-ray CT equipment located at Korea Institute of Building & Civil Engineering Institute was implemented to analyse the cell structure of samples in terms of cell size distribution. It is capable of attaining high resolution up to 3.5 μm and the maximum voltage and current are 225 kV and 3.0 mA, respectively. Although the cell size of sample is expected to be large enough for the CT system can identify, it is very restricted to separate each cell structure from the CT image due to relatively thin walled structure of polystyrene which is hard to be identifiable in CT image. We contrived new method to shape and size the cell structure by infiltrating a tracer, i.e., potassium iodide, into the cell structure, because the tracer can be well attenuated as the X-rays pass through it.

Each 2D sliced image was binarized to separate the cells filled with the tracer followed by consequential stacking along the height. However, it does not necessarily ensure the identification of individual cells because the boundaries between cells were relatively fuzzy. Therefore, we deployed the relevant image processing techniques to separate them. The reconstructed cells image is illustrated in Fig 3 and Fig 4 shows 3D patch and isosurface of cell structure extracted from CT image analysis.

![Figure 3. 3D X-ray CT images acquired from tracer infiltrated specimen.](image)
The volume of individual cell fragments is calculated by voxel counting and the cell size could be obtained by converting it into a diameter of equivalent volume of sphere (Fig 5). The average cell size is estimated to be approximately 384 μm which corresponds to the range (380 ~ 400 μm) of the cell size calculated from SEM image.

Figure 5. Cell size distribution calculated as a diameter of equivalent volume of sphere.

4. Conclusion
This study investigated the acquisition of size for 3D cell fragments in extruded polystyrene using X-ray computed tomography combined with a newly contrived method of using a tracer and image processing techniques.

The size distribution of cell fragments follows a log-normal distribution and corresponds well to measured SEM data. According to X-ray CT scanning images and SEM images, the shape was similar but according to tracer applied CT scanning images, the cell size distribution was 380~400 μm within the range of the general insulation diameter distribution which had the highest reliability. As for extrusion foaming polystyrene, we need additional image processing to identify the independent foam content as its density is too low. So, it is recommended to raise the 3D cell structure completeness of XPS by improving the scanning accuracy. This stereological analysis of particulate objects has substantial potentials in estimating size or shape dependent parameters for building materials.

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