Observation of Micropores by X-ray Computerized Microtomography: Fabrication of Porous Al with Deep Pores by Al-In Monotectic Solidification and Electrochemical Etching

Porous materials have potential for various practical applications because of their characteristic features. These include large surface area with respect to their volume, permeation of fluids, ability to hold fluids in their pores, and high strength to weight ratio. We have proposed a novel process for fabricating porous media in which deep pores are regularly aligned [1]. The process consists of two parts. The first part is the solidification process under a magnetic field to produce an aligned structure in which the minor phase with a rod shape is regularly aligned in the matrix phase. The second part is the electrochemical dissolution of the minor phase. A three-dimensional (3D) observation is strongly required to evaluate the aligned structure and the electrochemical dissolution process. X-ray computerized tomography with a high spatial resolution enables the observation of the aligned structure and the pores fabricated by the electrochemical dissolution process.

The X-ray computerized microtomography was performed at beamline **BL47XU** [2]. A monochromatized X-ray (15keV) was used to obtain a sufficient intensity and contrast for an X-ray transmitted through Al-In alloy specimens. The format of the transmission X-ray images was 1000 × 1018 pixels and the effective pixel size was 0.5 μm × 0.5 μm. The exposure time for every transmitted image was 1.5 s. Reconstructed images were obtained using 750 transmission images. The present configuration allowed a 3D observation with a spatial resolution of 2 μm.

The aligned structures have been produced by the unidirectional solidification of monotectic alloys such as Al-In, Al-Bi and Cu-Pb [3,4]. As compared to the eutectic alloys that are well known to form the aligned structure during the solidification, the growth condition to form the aligned structure is extremely limited in the monotectic alloys. However, the monotectic alloys have a significant advantage in fabricating the porous media. Since the minor phase precipitates as the liquid state during the monotectic solidification, any anisotropy due to the crystal structure does not distort the aligned structure. Thus, it is desired to extend the growth condition for the aligned structure of the monotectic alloys.

Al-10at% In alloys were unidirectionally solidified under a magnetic field of 10 T. The imposition of the static magnetic field during the unidirectional solidification successfully achieved the aligned rod-like structure even at the hypermonotectic composition (10at% In). Figure 1 shows the reconstructed image of the Al-In alloy solidified under a magnetic field of 10 T. The constituent phases (Al and In phases) were identified by the linear absorption coefficients of the reconstructed images. The X-ray computed microtomography shows that the continuous In rods with diameters of 10 - 20 μm are regularly aligned parallel to each other. Moreover, the In rods were reduced to be less than 1 μm in diameter by plastic deformation techniques because of the high ductility of the Al and In phases.

On the basis of the X-ray computed microtomography, a growth model for the monotectic alloys under a magnetic field has been proposed [1]. The In phase nucleates at the solidifying front during the solidification. The In liquid droplets can be pushed by the solidifying front [3,4]. When the diameter of a pushed droplet exceeds a certain value, the droplet is engulfed by the front. The sequence of nucleation, pushing and engulfment results in the distribution of the In droplet in the matrix during the conventional solidification. The imposition of the high magnetic field reduces the melt flow around the droplet and consequently

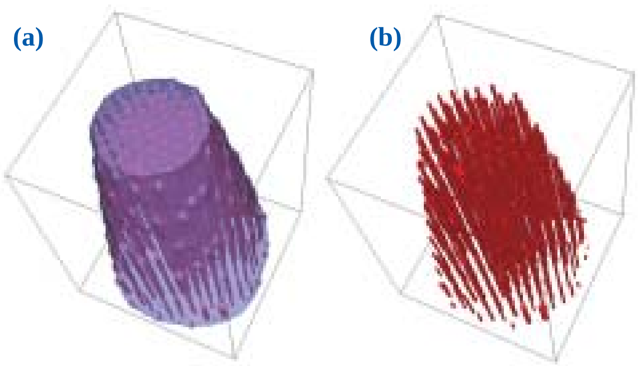

Fig. 1. 3D images of the Al-10at%In alloys which were unidirectionally solidified at a growth rate of 2.7 μm/s under a magnetic field of 10 T. **(a)** Image of the specimen used for the CT observation (Al matrix is semitransparent) and **(b)** In rods aligned in the Al matrix. The size of the 3D images is 380 × 380 × 500 μm^3. Blue: Al; Red: In.

enhances the engulfment of the In liquid phase. As a result, the cooperative growth of the Al and In phases produces the aligned structure.

A 10% HNO_3 aqueous solution was used for the electrochemical dissolution. As shown in Fig. 2, the electrochemical dissolution at a constant potential of -0.1 V for 12 h successfully removed the In rods from the matrix. However, the depth of the pores cannot be observed by SEM. Figure 3 shows the reconstructed 3D image of the Al-In alloys after the electrochemical dissolution for 24 h. Deep pores whose depth is more than 500 µm are produced by the electrochemical dissolution of the In rods. Deeper pores can be fabricated by the same procedures. Pores with a diameter less than 1 µm were also fabricated using the specimens reduced by the plastic deformation.

The computed microtomography using a hard X-ray and a high-resolution detector at SPring-8 significantly contributed to the fabrication of the porous media with deep pores. This technique will be widely applicable to evaluate microstructures of micro- and nanofabrications in various fields.

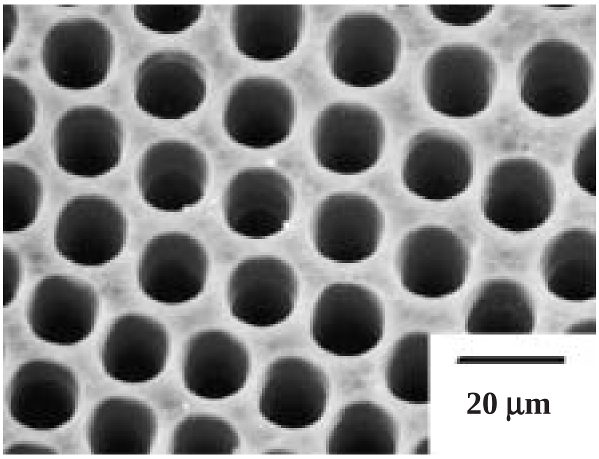

Fig. 2. SEM image of the porous Al produced by the electrochemical dissolution of the unidirectional solidified Al-10at%In alloy (growth rate: 2.7 µm/s; magnetic field: 10 T).

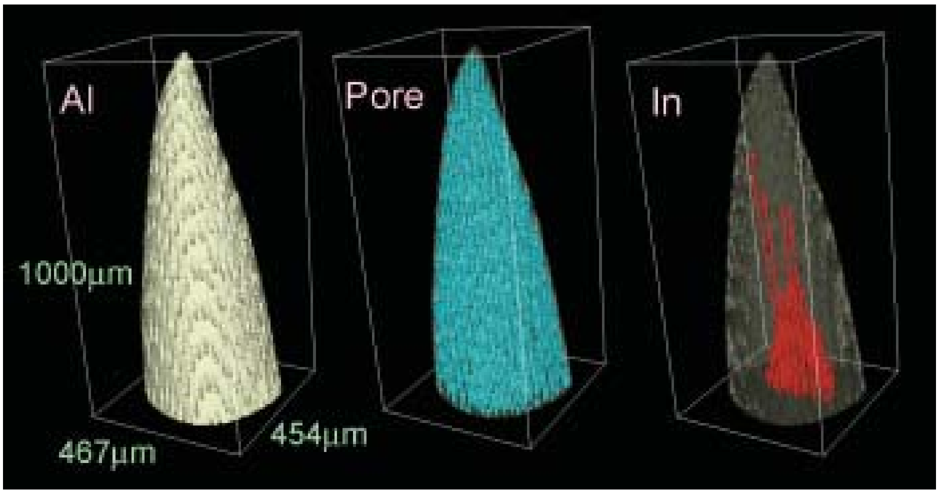

Fig. 3. 3D images of the porous Al produced by the electrochemical dissolution (24 h) of the unidirectional solidified Al-10at% In alloy. The size of the 3D images is $467 \times 454 \times 1000\ \mu m^3$.

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