3D observation of high-aspect-ration pores In the micro- and nano-porous materials fabricated by crystal growth under magnetic field and electrochemical treatment

H. Yasuda 5439[1)], A. Tsuchiyama 4116[2)], T. Nakano 1544[3)], K. Uesugi 1544[4)], I. Ohnaka 5424[1)], N.Takezawa 9379[1)], T.Tamayama 13397[1)]

1) Department of Adaptive Machine Systems, Osaka University, Suita, Osaka 565-0871
2) Department of Earth & Space Science, Osaka University, Toyonaka, Osaka, 560-0043
3) Institute of Geoscience, National Institute of Advanced Industrial Science and Technology, Tsukuba, 305-8567
4) Japan Synchrotron Radiation Research Institute, Mikazuki-cho, Hyogo 679-5198

Porous materials have potential for various practical applications because of their characteristic features. For example, they have large surface area with respect to their volume, permeation of fluids, an ability to hold fluids in their pores, and high strength to weight ratio.

A novel process to fabricate porous media in which deep pores are regularly aligned, has been proposed by our group. The process consists of two parts. The first is the solidification process under a magnetic field to produce an aligned structure in which the minor phase with rod shape is regularly aligned in the matrix phase. The second is the electrochemical dissolution of the minor phase.

3D observation of the rods and the pores is required to evaluate the aligned structure and the electrochemical dissolution. X-ray computerized tomography with a high spatial resolution enables the observation of the aligned structure and the pores fabricated by the electrochemical dissolution. The X-ray computed microtomography was performed at a beam line BL47XU. The format of the transmission X-ray images was 1000× 1018 pixels and the effective pixel size was 0.5μm x 0.5 μm. X-ray energy was 15 keV and the exposure time for every transmitted image was 1.5s.

The aligned structures have been produced by the unidirectional solidification of the monotectic alloys such as Al-In, Al-Bi and Cu-Pb. The monotectic alloys have a significant advantage to fabricate the porous media. Since the minor phase precipitates as liquid state during the monotectic solidification, any anisotropy due to the crystal structure does not distort the aligned structure.

Al-10at%In alloys were unidirectionally solidified under a magnetic field of 10T. Imposition of the static magnetic field during the unidirectional solidification successfully achieved the aligned rod-like structure even at the hypermonotectic composition (10at%In).

A 10% HNO_3 aqua solution was used for the electrochemical dissolution. As shown in Figure 1, the electrochemical dissolution at a constant potential of –0.1V for 12h successfully removed the In rods from the Al matrix. Figure 2 shows the reconstructed 3D image of the Al-In alloys after the electrochemical dissolution for 24h. The In rods indicate that pores of which depth is more than 500 μm are produced by the electrochemical disillusion. Branching and termination of the pores were rarely observed in the Al matrix. Deeper pores were also fabricated by increasing time for the electrochemical etching.

By measuring the pore diameters in the disillusion direction, the dissolution rate of the Al matrix was estimated to be 1/50 smaller than that of the In rods. The dissolution rates indicates that pores of 20 μm in diameter and 1mm in depth can be fabricated.

The solidified structure was reduced by plastic deformation techniques because of high ductility of the Al and the In phases. The microstructure in which the In rods are less than 1μm in diameter is obtained by a conventional caliber rolling method. The control of pore size by the plastic deformation will be further beneficial for fabrication of the porous Al.

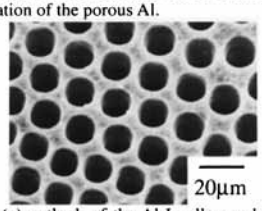

Figure 1. (a) outlook of the Al-In alloy and (b) In rods obtained by the micro X-ray CT.

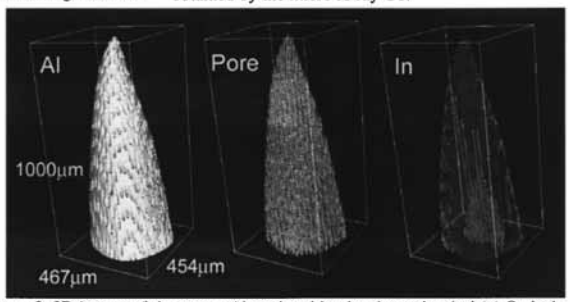

Figure 2. 3D images of the porous Al produced by the electrochemical (a) Outlook, (b) pores in the Al matrix.

X-ray Irradiation Effect on Nuclear Decay Process

K. Aoki(0001783), T. Mitamura(0005271), M.Terasawa(0003941), K.Hosono(0001601), T. Sekioka(0003939), Y.Ito[1)](0003629), H. Oohashi[1)], S. Sakakura[1)]0013258), T. Tochio[1)](0004007), K.Taniguti[2)](0005457), S. Maeo[2)](0008340), H.Yamaoka[3)] (0000084), T. Mukoyama[4)] (0005273) 、H. Kaji[5)] (0005451) and Y. Kawase[6)]

Himeji Institute of Technology, [1)] Kyoto University, [2)] Osaka Electro-Communication University, [3)]RIKEN Harima Institute, [4)]Kansai Gaidai University, [5)]Hungary Isotope Institute, [6)]Kyoto University Research Reactor Institute

According to nuclear decay theory, the atomic charge state can alter the lifetime of isomeric transition, because the internal conversion process, which competes with gamma transition, depends on number and orbit of bound electrons. The 388 keV isomeric state of ^{87}Sr have e/γ ratio of 0.211 (e_K/γ of 0.177). It is attempted to change the half-lives of the isomeric transition by stripping orbital electrons by means of synchrotron radiation (SR) at the BL47XU line of Spring-8.

Neutron irradiation using Kyoto University reactor, KUR, prepared the ^{87}Sr isomer. The radioactive specimens were sealed in thin Kapton film to form about 1 x 1 x 0.1 mm rectangular sheet. The energy of SR was 16.5 keV, which is enough to strip the K shell electrons. A large volume (50 cc) pure-Ge detector and a low energy photon detector (both ORTEC) were used to measure the 388 keV γ-rays and fluorescent X-ray, respectively. For both detectors, adequate metal absorbers were attached to prevent undesirable backgrounds.

Fig.1 shows a result obtained from the analysis of the sequences of the 388 keV photo-peak intensities from ^{87m}Sr decay. No remarkable change was observed in the lifetime of the isomeric state transition within the associated errors of nearly 99.8% confidence level. Precise analysis is now in progress.

The synchrotron radiation experiments were performed at the SPring-8 with the approval of the Japan Synchrotron Radiation Research Institute (JASRI) (Proposal No. 2003B0291-CM-np).