Dual energy *K*-edge subtraction imaging of 3D inhomogeneous microstructure in highly alloyed aluminium foam

X-ray computed tomography (CT) gives unique chemical information within bulk materials in 3D/4D (i.e., 3D+time axis). In the case of pure binary systems in which two constituent atoms are relatively apart in atomic number, X-ray CT can provide chemical concentration distributions quantitatively. However, since the X-ray mass absorption coefficient is obtained simply from the sum of the absorption cross sections of constituent atoms, the quantitative measurement does not apply to ternary or more complex systems such as practical alloys.

K-edge subtraction imaging technique was originally developed for the diagnosis of neurovascular pathology with an iodine-containing contrast agent and later extended to lung imaging with an inhaled contrast agent containing xenon gas. After a couple of monochromatic images are acquired above and below the X-ray *K*-edge energy, logarithmically subtracting the two images enables the enhancement of the contrast of any structure containing a contrast agent. This technique was further extended by Ikeda *et al*. [1] to realize quantitative analysis in 3D Cs concentration demonstrations for partially molten granite.

In this work, dual energy *K*-edge subtraction imaging was performed to quantify the Zn distribution in Al-Zn-Mg foam. This foam, which is designed for ultra-lightweight energy absorption structures, is a newly developed metallic foam with a closed-cell structure. The fracture behavior of the cell wall has also been identified three-dimensionally, providing new insights into the damage evolution of metallic foams.

High-resolution X-ray tomography was performed at beamline **BL20XU**. A liquid nitrogen-cooled Si (111) double-crystal monochromator was used to produce monochromatic X-ray beams. Dual energies, 9.71 keV and 9.61 keV (above and below the *K* absorption edge of Zn, respectively), were chosen for subtraction imaging. The combination of the CCD detector (4000×2624 pixels, 2×2 binning mode, 5.9×5.9 μm^2 pixel size) and the optical lens (×20) provided an isotropic voxel with a 0.474 μm edge.

The 3D quantification of Zn concentration was obtained from the subtraction of the CT images corresponding to 9.71 keV and 9.61 keV. It is described as [2]:

$$g_{Zn} = \frac{\Delta\mu}{\Delta\mu_{Zn(above)} - \Delta\mu_{Zn(below)}} \times \frac{\rho_{Zn}}{\rho}$$

where g_{Zn} is the weight fraction of Zn, $\Delta\mu$ is the difference in linear absorption coefficient (LAC) between the two energies, ρ and ρ_{Zn} are the densities of bulk alloy and Zn, $\mu_{Zn(9.71)}$ and $\mu_{Zn(9.61)}$ are the LACs of Zn at 9.71 keV and 9.61 keV, respectively. The correction of LAC is necessary to ensure the accuracy of the LAC measurement [3]. It has been confirmed that when the same setup is used, the detection limit for Cu in Al alloys is similar to the EDS results in scanning electron microscopy (i.e., about 0.5 mass%), and a superior reproducibility of ±0.1 mass% is obtained [4].

Figures 1(a) and 1(b) show the reconstructed 3D image of a single cell wall and its 3D Zn distribution with a concentration map, as seen on the outside

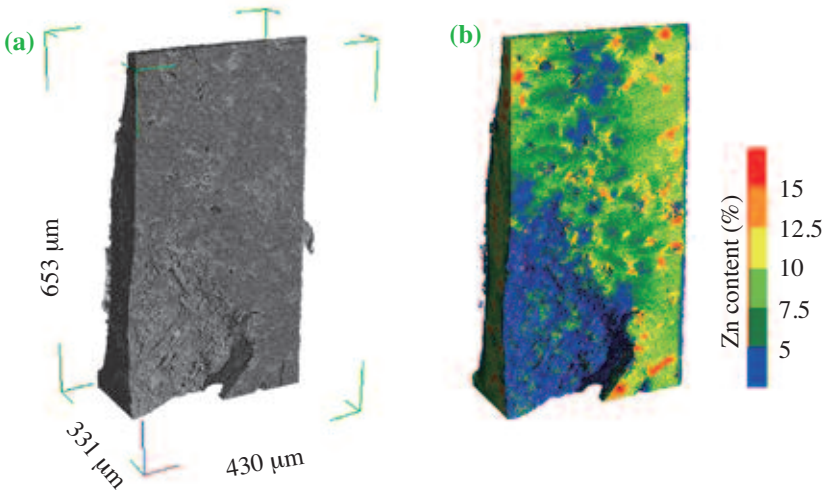

Fig. 1. 3D image of cell wall **(a)** and corresponding Zn distribution with concentration map on outside surface of cell wall **(b)**. [2]

surface of the cell wall. The quantitative visualization of Zn content is based on the $\Delta\mu$ information from two sets of CT image taken at 9.71 keV and 9.61 keV, respectively. A quite inhomogeneous distribution of Zn is found throughout the cell wall, which was cut from as-cast foam. The red regions indicate a higher Zn concentration, corresponding to Zn-bearing particles. The remaining aluminum matrix regions exhibit a wide variation in Zn concentration. The agglomeration of Zn-bearing particles would affect the fracture behavior of the cell wall.

In situ compressive tests of the cell walls were performed and 3D visualization was carried out to determine the fracture response and identify the interaction between fracture paths and Zn distributions or micropores. Figure 2 shows one representative crack path. In the 2D slice image in Fig. 2(a), some white areas and micropores are observed. In Fig. 2(b) showing the reconstructed Zn mapping by *K*-edge subtraction method, those white areas are proven to be highly concentrated Zn regions that are distributed non-uniformly in the foam. Particularly in the center part, the Zn-bearing particles agglomerate along a line. A crack is also observed in this region, and the fracture path is along the agglomerated Zn-bearing particles, which probably acted as sites for stress concentration, indicating an adverse effect of agglomerated particles on crack resistance. A 3D image of the crack's interaction with the Zn-bearing areas and micropores is shown in Fig. 2(c). The propagation of the crack along the particle agglomeration areas is visualized three-dimensionally. The crack approaches a coarse micropore at the crack tip, then the acceleration of crack propagation can be anticipated in this local area as a result of the anti-shielding effect. Our previous work [5] has shown that the brittle cracking of a cell wall is usually followed by the rapid collapse of the cell wall in the Al-Zn-Mg foam. The combination of local agglomerated particles and micropores is confirmed to exert an adverse effect on intrinsic crack propagation.

In summary, a highly heterogeneous distribution of Zn in the cell wall of Al-Zn-Mg foam was illustrated in 3D using dual energy *K*-edge subtraction technique. It has been clarified that the combination of local agglomerated Zn-bearing particles and micropores exerts an adverse effect on intrinsic crack propagation. The current element sensitive tomography has been proved to be an effective technique for determining the microstructure and property relationship of highly inhomogeneous foam materials in 3D.

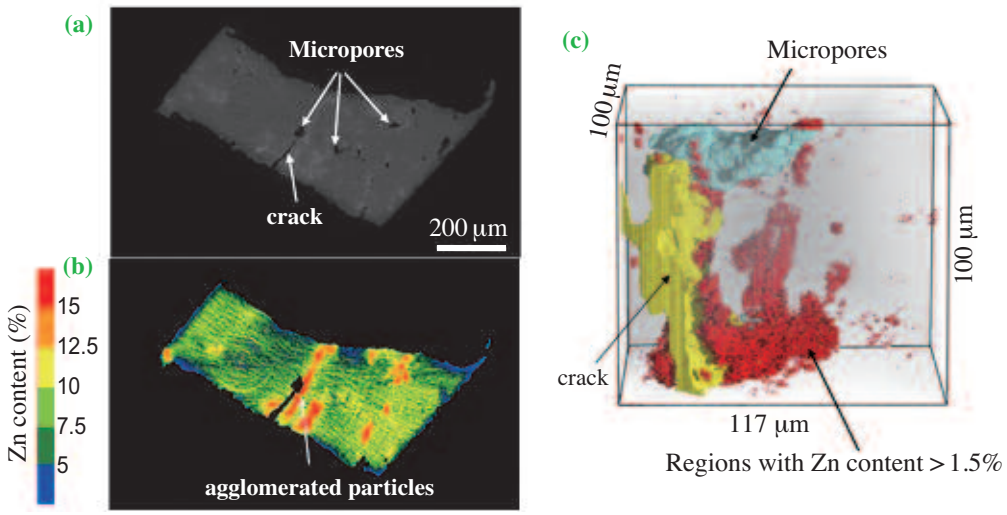

Fig. 2. Representative crack path through region of agglomeration of Zn-bearing particles and micropores: slice image taken at 9.61 keV **(a)**, Zn mapping **(b)** and 3D image **(c)**. [2]

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