

# 3D Characterisation of the Fe-rich intermetallic phases in Al-5%Cu alloys by synchrotron X-ray microtomography and skeletonisation

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## Abstract

Synchrotron X-ray microtomography and skeletonisation method were used to study the true 3D network structures and morphologies of the Fe-rich intermetallic phases in Al-5.0%Cu-0.6%Mn alloys with 0.5% and 1.0% Fe. It was found that, the Fe-phases in the 1.0%Fe alloy have node lengths of 5-25 $\mu$ m; while those in the 0.5%Fe alloy are of 3-17  $\mu$ m. The Fe-phases in the 1.0%Fe alloy also developed sharper mean curvature with wider distribution than those in the 0.5%Fe alloy. Combining SEM studies of the deeply-etched samples, the true 3D structures of 4 different type Fe phases in both alloys are also revealed and demonstrated.

**Keywords:** Synchrotron X-ray tomography; skeletonisation analysis; Fe-rich intermetallic phases; Aluminium alloys; Solidification

Aluminium (Al) alloys are widely used in the transportation, building and packaging industry because of their lightweight, high specific strength, high corrosion resistance, and excellent recyclability [1]. In modern vehicles, Al alloys are playing increasingly important roles in reducing the weight of vehicles and, hence, fuel consumption and CO<sub>2</sub> emissions in transportation [2]. Approximately 90% of the Al alloys used in land vehicles are from recycled sources for cost reduction and sustainability [3]. In Al alloys, especially recycled Al alloys where Fe concentration is often higher than 0.5% (weight percentage), Fe is the most common impurity element, and it can be easily picked up in sorting and remelting processes during Al recycling [4]. Normally, when the Fe in an Al alloy is >0.05% [4], brittle Fe-rich intermetallic phases (named Fe-phases hereafter) form and their size, morphology and distribution have profound effects on the castability and mechanical properties of the final parts. In most cases, these Fe-phases, especially when the needle-like or plate-like phases, such as  $\beta$ -Al<sub>7</sub>Cu<sub>2</sub>Fe phase, are detrimental to the alloys [5]. In some alloy systems, neutralisation elements, *e.g.*, Mn and Si, can be used to alter the morphology of the Fe-phases to a less harmful type [6- 8].

Quantitatively understanding of the size, morphology and distribution of the Fe-phases are of paramount importance in the physical metallurgy of recycling Al alloys, and in manufacture high-quality components for the transportation industry. In the past, majority of the research on Fe-phases was conducted using 2-dimensional (2D) imaging methods [6-8], *i.e.* optical and/or electron microscopy, which gives very limited information about 3-dimensional (3D) structures/morphologies, and the spatial interconnections and correlations between the different phases. Recently, a number of investigations [9, 10] have been made by

1 using scanning electron microscopy (SEM) and focused ion-beam (FIB) tomography to  
2 reveal the well connected and branched 3D network structure in Chinese script type  $\alpha$ -Fe-  
3 phases in Al-Si alloys (the typical composition is  $\text{Al}_{14}\text{Fe}_{2.8}\text{Si}_2$ ). 3D morphology of Fe-phases  
4 has also been characterised using serial sectioning plus optical [11] or electron microscopy  
5 [12]. However, Focused Ion Beam (FIB) is normally used for sectioning sub-micrometre  
6 features [12], not for those of length scale in many hundreds, even thousands of micrometres,  
7 such as the Fe-phases in present study, and serial sectioning is often very time-consuming.  
8 Recently, synchrotron X-ray tomography has been used to study the 3D microstructures of a  
9 wide range of multiphase alloys [13-16]. For example, the nucleation and growth of the Fe-  
10 phases in 3D in Al-Si alloys were reported in [4, 17-20]; and the snapshots of the 3D Fe-  
11 phases in Al-Cu alloys were given by Gutiérrez, *et al* [21]. However, Gutiérrez, *et al* did not  
12 segment the individual Fe-phases [21]. Hence, the detailed 3D structures of the Fe-phases,  
13 and their spatial interconnection with other phases such as  $\text{Al}_2\text{Cu}$  have not been revealed.  
14 Normally, 4 different types Fe-phases exist in the Al-Cu alloys with Fe concentration of 0.5 -  
15 1.0% [22, 23]. They are plate-shaped phases  $\beta$ - $\text{Al}_7\text{Cu}_2\text{Fe}$  and  $\text{Al}_3(\text{FeMn})$ ; and Chinese script-  
16 type phases,  $\alpha$ - $\text{Al}_{15}(\text{FeMn})_3\text{Cu}_2$ , and  $\text{Al}_6(\text{FeMn})$ . These Fe-phases are very different to the  
17 plate-shaped Fe-phases found in the Al-Si alloys [4, 17-20]. So far, no reports have been  
18 found that describe the true 3D structures of the 4 typical Fe-phases present in the Al-Cu  
19 alloys [22, 23] and their spatial interconnections and correlations.  
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46 In this paper, we used synchrotron X-ray microtomography and skeletonisation method to  
47 study the 3D network structures and morphologies of the Fe-phases and the associated  $\text{Al}_2\text{Cu}$   
48 phases in two alloys: Al-5%Cu-0.6%Mn with 0.5% and 1.0% Fe (named 0.5Fe alloy and  
49 1.0Fe alloy, respectively, hereafter). Higher Fe content was deliberately added into the two  
50 alloys to mimic those often found in the recycled Al alloys. The complex 3D network  
51 structures of the Fe-phases and the  $\text{Al}_2\text{Cu}$  phases, their mean curvature distributions and the  
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1 inter-dependence between the Fe-phases and the Al<sub>2</sub>Cu phases were reported for the first  
2 time. Furthermore, the true 3D morphologies of the 4 different types Fe-phases in Al-5%Cu  
3 alloys are also revealed, providing more quantitative 3D information for understanding the  
4 structures of the Fe-phases.  
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11 Both alloys were made by using pure Al ingot (99.9%), Al-20% Cu, Al-10%Mn and Al-  
12 10%Fe master alloy with the correct charge weight. The feedstock materials were held inside  
13 a clay-graphite crucible and melted at 780 °C in an electric furnace. After temperature  
14 homogenisation, the melt was then cooled to 710 °C, and poured into a steel permanent  
15 mould (Ø 65 mm × 70 mm) preheated to 200 °C to form an ingot. Cylindrical samples (~Ø10  
16 mm × 20 mm) were cut from the edge of the ingot and then machined into Ø 2 mm × 5 mm  
17 for tomography scans. The solidification time at the location where the samples were taken  
18 was ~ 42.5 s with an average cooling rate of ~ 2.5 K/s [24]. Routine 2D microstructure  
19 characterisation was made using a FEI Quanta 200 Field Emission Gun scanning electron  
20 microscope (SEM) equipped with an energy-dispersive X-ray analyzer. For the SEM  
21 samples, 10% NaOH aqueous solution was used to dissolve the Al matrix (20 min) in order to  
22 expose more of the Fe-phases embedded inside the Al matrix. Synchrotron X-ray tomography  
23 experiments were performed at the TOMCAT beamline X02DA of the Swiss Light Source  
24 (SLS), Paul Scherrer Institute, Switzerland. The experimental parameters used are given in  
25 Table 1 [25]. A white beam from a superconducting bending magnet source was used with a  
26 400 µm Al filter to remove the low energy tail of the incident beam and to reduce heat load  
27 on the sample and detector. The imaging system consists of a 100 µm LuAG: Ce scintillator  
28 (Crytur) coupled to a white-beam compatible microscope with a 6.8 × magnification (Optique  
29 Peter). For each scan, 2000 projections were acquired over 180° of sample rotation.  
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1 Tomographic reconstructions were performed on the TOMCAT cluster [26] using the  
2 GridRec algorithm [27] coupled with the Parzen filter [28].  
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4 Fig. 1a shows the typical cross-sectional slice obtained from the synchrotron X-ray  
5 tomoscan, and an area of interest was extracted and showed in Fig. 1b. The corresponding  
6 SEM images and the deeply-etched Fe-phases are shown in Fig. 1c and d respectively. Both  
7 X-ray images and SEM images show that the pore is in dark, the Al matrix is in dark grey,  
8 the Fe-phases are in light grey, and the Al<sub>2</sub>Cu phases are in white colour. Open source image  
9 processing software, Image J [30] was used to adjust the contrast between the different  
10 phases. Then, the 3D bilateral filter was applied to the tomography datasets to increase the  
11 contrast and reduce noise. Finally, the pores, Al dendrites, Fe-phases, Al<sub>2</sub>Cu phases were  
12 segmented by using different global threshold values (Pore: 0 ~ 10688,  $\alpha$ -Al: 10689 ~ 26438,  
13 Fe-phases: 26439 ~ 38814; Al<sub>2</sub>Cu: 38815 ~ 65535). 3D segmentation and feature rendering  
14 were performed using Avizo Lite v9.0.1 (VSG, France) and Viper, the University of Hull's  
15 High Performance Computer (HPC) cluster. The reconstruction data were in 16-bit format.  
16 Normally, a sub-volume of 500<sup>3</sup> voxels with a voxel size of (1.62 micron)<sup>3</sup> was chosen for  
17 further analyses. However, from the X-ray absorption contrast only, it is not possible to  
18 distinguish and segment the 4 different types of Fe-phases in the two alloys. Hence, the  
19 segmented Fe-phases from the X-ray tomoscans contain all 4-type Fe-phases. By comparing  
20 the SEM images of the Fe-phases revealed by the deeply-etched samples with those showed  
21 in the X-ray tomography, we are able to identify the 4 different type Fe-phases as discussed  
22 later in the paper.  
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24 Fig. 2 shows the 3D colour rendering of the Fe-phases, Al<sub>2</sub>Cu phases and  $\alpha$ -Al matrix and  
25 their mean curvature distributions for the 0.5Fe and 1.0Fe alloys, respectively. The mean  
26 curvature  $H$  [31] is defined as:  
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$$28 \quad H = 0.5 * \left( \frac{1}{R_1} + \frac{1}{R_2} \right) \quad (1)$$

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1 where  $R_1$  and  $R_2$  are the two principal radii of curves respectively. Local curvature is an  
2 important geometrical parameter for the interface between two phases (dendrites or  
3 intermetallics) formed during the solidification processes, influencing the diffusion of solutes  
4 and therefore the final morphology of the phases.  
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10 Fig. 2a and b show the complex network and intricate morphology of the interconnected  
11 Fe-phases and  $Al_2Cu$  phases. Red shows the Fe-phases, green for the  $Al_2Cu$  phases, blue for  
12 the Al matrix. These phases conglomerate together in the  $\alpha$ -Al inter-dendritic region in the  
13 chosen volume of  $162\mu m \times 162\mu m \times 162\mu m$ . Fig. 2c and d show the 3D network of the Fe-  
14 phases with their mean curvatures for the 0.5Fe and 1.0Fe alloy, respectively. The Fe-phases  
15 form a spatially interconnected complex 3D network. The distributions of their mean  
16 curvatures follow the Gaussian distribution (Fig. 2g). The distribution peak position ( $\mu$ ) of the  
17 Fe-phases increase from 0.64 to 1.10 and the standard deviation ( $\sigma$ ) increases from 1.33 to  
18 2.71 as the Fe increases from 0.5% to 1.0%. The Fe-phases in the 1.0Fe alloy have more  
19 positive and negative mean curvatures. Similarly, Fig. 2e and f show that the structures of the  
20  $Al_2Cu$  phases are also interconnected 3D network. Fig. 2h shows that the mean curvatures of  
21 the  $Al_2Cu$  phases also follow Gaussian distribution. The distribution peak position ( $\mu$ )  
22 increases from 0.34 to 0.75 as the Fe increases from 0.5% to 1.0%, and the standard deviation  
23 ( $\sigma$ ) increases from 0.71 to 2.52, indicating that  $Al_2Cu$  phases of more positive mean  
24 curvatures also exist in the 1.0Fe alloy. *Much richer and clearer 3D information in different*  
25 *view angles for the Fe-phases and  $Al_2Cu$  phases are illustrated in the four accompanying*  
26 *videos.* The increase of the mean curvature is mostly due to the different solidification  
27 reactions in the 0.5Fe alloy and 1.0Fe alloy. In the 1.0Fe alloy, the solidification reactions in  
28 the range of 649-653 °C are:  $L \rightarrow \alpha-Al$ ;  $L \rightarrow \alpha-Al + Al_3(FeMn)$ ;  $L \rightarrow \alpha-Al + Al_6(FeMn)$   
29 [22]; then at 542 °C:  $L \rightarrow \alpha-Al + \beta-Al_7Cu_2Fe + Al_3(FeMn) + Al_6(FeMn) + Al_2Cu$  [22, 32].  
30 The  $Al_3(FeMn)$  and  $Al_6(FeMn)$  phases are more rod-like phases with sharp edges as  
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1 discussed later, resulting in a high proportion of positive and negative mean curvature. While  
2 in 0.5Fe alloy, the solidification reactions in the range of 589-597 °C are:  $L + Al_6(FeMn) \rightarrow$   
3  $\alpha-Al_{15}(FeMn)_3Cu_2$ ;  $L + \alpha-Al_{15}(FeMn)_3Cu_2 \rightarrow \beta-Al_7Cu_2Fe$  [22], and then in 542-537 °C:  $L \rightarrow$   
4  $\alpha-Al + \alpha-Al_{15}(FeMn)_3Cu_2 + \beta-Al_7Cu_2Fe + Al_2Cu$  [23, 32].  $\alpha-Al_{15}(FeMn)_3Cu_2$  have typical  
5 Chinese script morphology, leading to a relatively low proportion of positive curvatures. In  
6 both alloys, the  $Al_2Cu$  phases formed through the multiple-phase eutectic reaction and in  
7 close contact with the Fe-phases and the  $\alpha-Al$  formed prior to the eutectic reaction. Hence,  
8 the  $Al_2Cu$  phases somehow “inherit” the characteristics of the Fe-phases, i.e. more “flat”  
9 (near zero mean curvatures) Fe-phases resulted in more “flat”  $Al_2Cu$  phases as in the 0.5Fe  
10 alloy case; while more “sharp” (higher positive or higher negative curvatures) Fe-phases led  
11 to more “sharp”  $Al_2Cu$  phases as in the 1.0Fe alloy case.  
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27 We used the skeletonisation function available in Avizo® to peel off the 3D network of  
28 the Fe and the  $Al_2Cu$  phases down to a skeleton (1-voxel thickness) with connecting nodes.  
29 The length of the curve between each node and the number of the connecting nodes can be  
30 calculated, and therefore the 3D characteristics of the skeleton (the 3D characteristic of the  
31 phase branches) can be quantified [31]. *It should note that, for the phases of relatively flat*  
32 *shape, the skeletonisation can only pick up their edges as demonstrated more clearly in the*  
33 *companying videos.* Fig. 3a and b show the skeletons of the Fe-phases in the 0.5Fe and 1.0Fe  
34 alloys, respectively. The Fe-phases in the 1.0Fe alloy have more branches. The node length  
35 distributions (Fig. 3e) show that the length of Fe-phases in the 0.5Fe alloy is shorter (in the  
36 range of 3-17  $\mu m$ ) than those (5-25 $\mu m$ ) in the 1.0Fe alloy, indicating that Fe-phases of the  
37 0.5Fe alloy are much compacted than those in the 1.0Fe alloy. This is consistent with the  
38 findings reported in [6] that the Fe-phases become less compact with increasing Fe  
39 concentration. Fig. 3c and d show the skeletons of the  $Al_2Cu$  phases in the 0.5Fe and 1.0Fe  
40 alloys, and the distributions of the node lengths are shown in Fig. 3f. The skeleton structures  
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1 of Al<sub>2</sub>Cu become more complex with the increase of Fe. The node length of Al<sub>2</sub>Cu in the  
2 0.5Fe alloy is in the range of 5-20 μm, while the length for 1.0Fe alloys is in the range of 3-  
3 15 μm, indicating that the node length of Al<sub>2</sub>Cu in the 1.0Fe alloy is relatively shorter than  
4 those in the 0.5Fe alloy. This is related to the increase of the volume fraction of Fe-phases  
5 with increasing the Fe content [22], which occupy more room and consumed more Cu in the  
6 residual liquid before the melt approaches the eutectic reaction at ~546 °C, and then the  
7 Al<sub>2</sub>Cu phases form through the eutectic reaction. Hence the growths of Al<sub>2</sub>Cu phases are  
8 restricted within the space enclosed by the Fe-phases and the Al dendrites, resulted in an  
9 overall shorter length.  
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22 SEM-EDX analysis shows that composition of the 4 different type Fe-phases are: α-  
23 Al<sub>15</sub>(FeMn)<sub>3</sub>Cu<sub>2</sub> (Al: 77.43±2.62%, Fe: 12.09±2.73%, Mn: 3.74±0.87%, Cu: 6.73±2.32%,  
24 at.%); β-Al<sub>7</sub>Cu<sub>2</sub>Fe (Al: 72.96±2.44%, Cu: 20.92±0.51%, Mn: 1.87±0.49%, Fe: 4.24±0.10%),  
25 Al<sub>3</sub>(FeMn) (Al: 81.58±0.87%, Cu: 5.52±1.40%, Mn: 2.32±0.38%, Fe: 10.56±1.01%) and  
26 Al<sub>6</sub>(FeMn) (Al: 83.67±0.86%, Cu: 3.30±0.47%, Mn: 3.17±0.01%, Fe: 9.86±0.09 %). Fig. 4  
27 shows the volume rendering of 3D morphologies of the 4 type Fe-phases extracted from the  
28 tomographic images by carefully comparing with the deeply-etched phases obtained from the  
29 SEM observation. Fig. 4a shows the morphology of the Al<sub>3</sub>(FeMn) phases. In 3D, it is more  
30 or less like a long rod-like structure with a few small branches. While in 2D characterisation,  
31 they were often recognized as “needle-shaped phases” in the longitudinal direction. It is  
32 formed during the reaction: L → α-Al + Al<sub>3</sub>(FeMn). Fig. 4b shows that the Al<sub>6</sub>(MnFe) phase  
33 develops more branches, and become more complex in 3D. While the deeply-etched image  
34 further confirms such features. Fig. 4c reveals that the α-Fe is 3D complex network and  
35 apparently a typical 2D cross-sectional through the network could render a typical “Chinese  
36 script” in a 2D view field. This complex morphology is because Al<sub>6</sub>(FeMn) transforms to α-  
37 Fe through the peritectic reaction: L + Al<sub>6</sub>(FeMn) → α-Al + α-Al<sub>15</sub>(FeMn)<sub>3</sub>Cu<sub>2</sub> at 589-597 °C  
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1 [22]. Thus,  $\text{Al}_6(\text{FeMn})$  is the nucleation site for the  $\alpha$ -Fe phase. The  $\alpha$ -Fe morphology  
2 presented in this study is significantly different from previous studies [6, 21, 22] reported in  
3 Al-Cu alloys, perhaps because those previous studies only observed a small volume of the  
4 deeply-etched samples [22] or FIB cut samples [10]. The  $\beta$ - $\text{Al}_7\text{Cu}_2\text{Fe}$  phase in both deeply-  
5 etched SEM samples and tomography samples are plate-like (Fig. 4d in a volume of  $20 \times 10$   
6  $\times 4 \mu\text{m}^3$ ). The plate-like morphology is similar to the  $\beta$ - $\text{Al}_5\text{FeSi}$  in Al-Si alloys reported in  
7 previous studies [4, 14]. Such a large faceted structure promotes the formation of porosity  
8 defects and act as the sites for crack initiation during mechanical loading [17].  
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20 In this paper, the true 3D network structures and morphologies of the Fe-rich intermetallic  
21 phases in Al-5.0Cu-0.6Mn alloys with Fe concentrations of 0.5% and 1.0% were studied and  
22 quantified for the first time. The Fe phases in the 1.0Fe alloy are complex 3D networks with  
23 well-developed branches of the node lengths of 5-25 $\mu\text{m}$ . While the Fe-phases in the 0.5Fe  
24 alloy have the similar 3D structures, but more compact and shorter branch node lengths (3-17  
25  $\mu\text{m}$ ). The Fe phases in the 1.0Fe alloy also developed sharper curvatures than those in the  
26 0.5Fe alloy with the standard deviation of the mean curvature distribution decreased from  
27 2.71 to 1.33. Furthermore, the true 3D structures of the 4 different type Fe phases,  
28  $\text{Al}_3(\text{FeMn})$ ,  $\text{Al}_6(\text{MnFe})$ ,  $\alpha$ - $\text{Al}_{15}(\text{FeMn})_3\text{Cu}_2$ ,  $\beta$ - $\text{Al}_7\text{Cu}_2\text{Fe}$  in the two alloys are revealed and  
29 demonstrated.  
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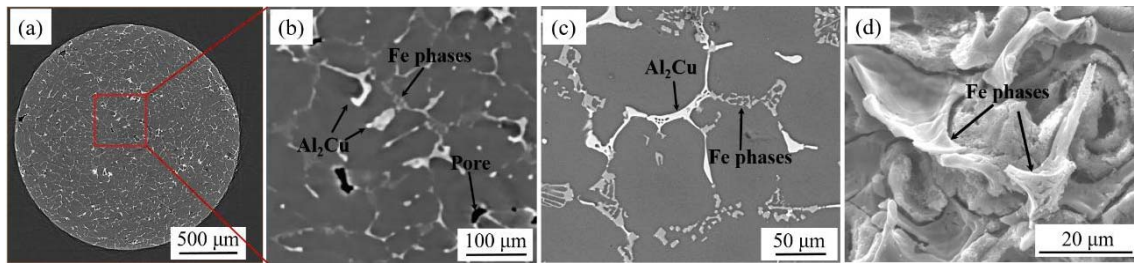
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**Table 1** The parameters used for the tomography acquisition at TOMCAT, SLS.

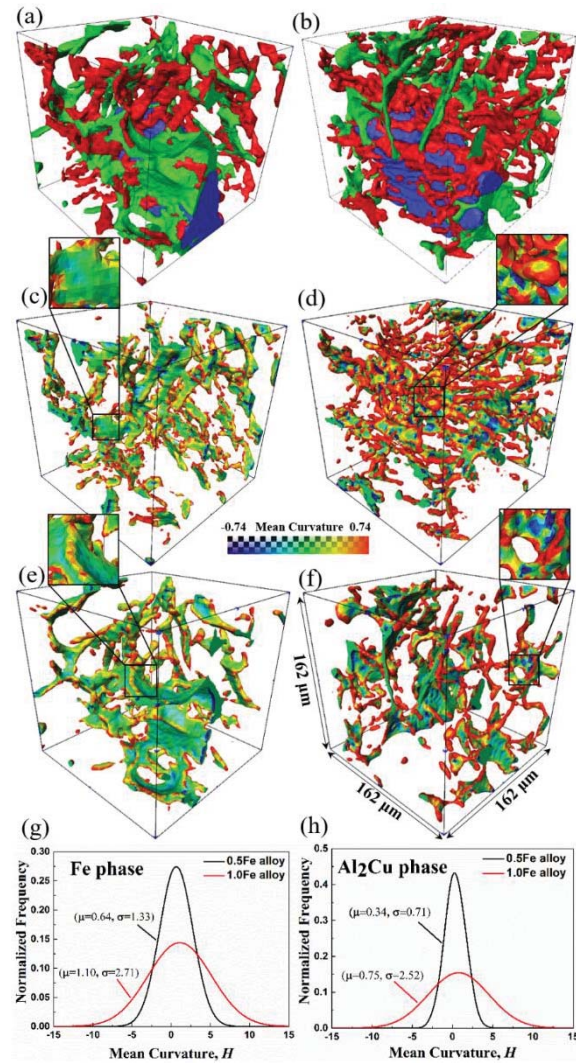
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X-ray Beam	Polychromatic radiation
Scintillator	LuAG: Ce 100 $\mu\text{m}$
Detector	GigaFRoST [29]
Effective pixel size	1.62 $\mu\text{m}$
Detector area	1392 $\times$ 1392 pixels
Exposure Time	7.0 ms
Magnification	6.8 $\times$
No. of Projections	2000
Sample-to-Scintillator distance	180 mm

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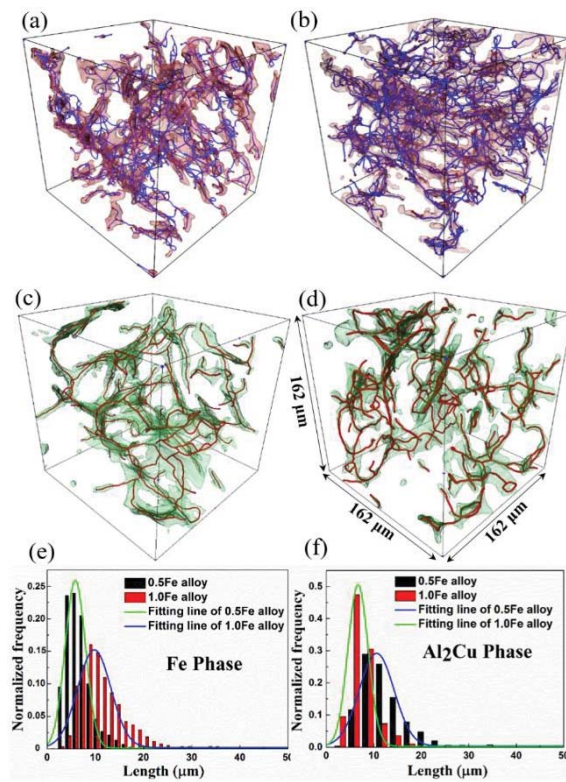
**Fig. 1.** (a) a typical 2D slice from the tomography scan of the 0.5Fe alloy; (b) the enlarged image of the framed area in (a) and processed using a 3D bilateral filter; (c) a typical SEM image of the 0.5Fe alloy; (d) a SEM image, showing the deeply-etched Fe phases.



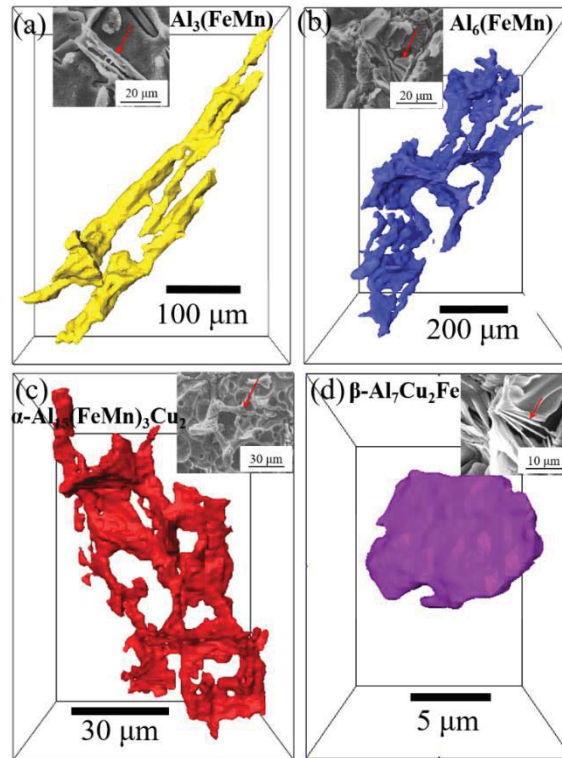
**Fig. 2** Typical 3D volume rendering of the Fe phases and Al<sub>2</sub>Cu phases and their mean curvatures in (a) the 0.5Fe alloy and (b) the 1.0Fe alloy (red: Fe phases, green: Al<sub>2</sub>Cu phases, blue:  $\alpha$ -Al matrix). 3D morphology coloured with its mean curvature: (c) Fe phases in the 0.5Fe alloy; (d) Fe phases in the 1.0Fe alloy; (e) Al<sub>2</sub>Cu phases in the 0.5Fe alloy; (f) Al<sub>2</sub>Cu phases in the 1.0Fe alloy. The distributions of the mean curvatures of: (g) the Fe phases, and (h) the Al<sub>2</sub>Cu phases, respectively.



Figure 3



**Fig. 3** Skeletons of the Fe phases in: (a) the 0.5Fe alloy and (b) the 1.0Fe alloy; skeletons of the Al<sub>2</sub>Cu phases in (c) the 0.5Fe alloy and (d) the 1.0Fe alloy; (e) and (f) the distributions of the node lengths of the Fe phases and Al<sub>2</sub>Cu phases, respectively.



**Fig. 4.** 3D structures of 4 different Fe phases and their SEM images (insets) of deeply-etched morphologies: (a) rod-like  $\text{Al}_3(\text{MnFe})$ ; (b) Chinese script type  $\text{Al}_6(\text{MnFe})$ ; (c) Chinese script type  $\alpha\text{-Al}_{15}(\text{FeMn})_3\text{Cu}_2$ ; (d) plate-like  $\beta\text{-Al}_7\text{Cu}_2\text{Fe}$ .