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Assessment by microtomography of 2D image analysis methods for the measurement of average grain coordination and size in an aggregate

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The microstructural evolution of Al–15.8 wt.% Cu slurry held at 555 °C was characterized in situ by X-ray microtomography. The average solid grain coordination and size measured on the reconstructed three-dimensional (3D) objects are compared with the values derived via image analysis carried out on 2D sections of the same objects. A new method is proposed for obtaining the average grain coordination by 2D image analysis. The method proves to be more accurate than the procedure commonly used in the literature.

Keywords: Synchrotron radiation X-ray microtomography; Quantitative image analysis; Metallography

Two-phase systems in which one of the phases consists of an aggregate of grains embedded into a matrix are common in materials processing. Typical examples are sintering bodies and semi-solid slurries [1]. The same parameters can be used for characterizing the microstructure of such systems independently of the nature of matrix phase, whether vapour, liquid or solid. This work relies on an experimental characterization of a semi-solid slurry by X-ray microtomography. Hence, for convenience, the matrix phase will be designated as being a liquid phase.

Basically, the morphology of an aggregate of solid grains embedded into a continuous liquid phase can be characterized by three non-dimensional parameters:

- the liquid phase volume fraction

$$u = \frac{V_{\text{liquid}}}{V_{\text{liquid}} + V_{\text{solid}}} \quad (1)$$

- the contiguity, defined as average fraction of the surface area of a grain that is shared with another grain [2]:

$$c = \frac{2A_{\text{ss}}}{A_{\text{sl}} + 2A_{\text{ss}}} \quad (2)$$

where A_{ss} and A_{sl} are the total solid/solid and solid/liquid interface areas in the system;

- the average coordination number, n_c (also sometimes called “connectivity”), defined as the average number of contacts of each solid grain with other grains.

According to stereology, exact values of the liquid fraction u and contiguity c can be derived from two-dimensional (2D) image analysis. In particular, contiguity is obtained exactly as

$$c = \frac{2N_{\text{ss}}}{2N_{\text{ss}} + N_{\text{sl}}} \quad (3)$$

where N_{ss} and N_{sl} are the number of solid/solid and solid/liquid interfaces intersected per unit length of test lines drawn on the 2D sections. In contrast, the exact value of the average coordination, n_c , is accessible only via 3D characterization. Like estimation of average grain size or average grain contact area, estimation of n_c from 2D images requires a model involving a hypothesis about grain shapes and size distribution. The most commonly used model for estimating n_c is due to Gurland [2], who assumes mono-sized spherical grains forming circular contacts with identical diameters.

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Three-dimensional reconstruction of a microstructure can be made by serial sectioning (e.g. [3]), but the choicest method for the characterization of a two-phase microstructure at the process temperature is X-ray microtomography [4,5]. Indeed, being non-destructive, this method allows in situ follow-up of the evolution of the local microstructure at high temperature. Computer-aided quantification of microstructural parameters from either 3D tomographic reconstructions or 2D images first requires the separation of individual grains by a transformation called “segmentation”. When a tomographic reconstruction of the object is available, the segmentation operation can be carried out either directly on the 3D reconstruction or on 2D sections of the reconstructed object. The latter procedure is thus identical to the common metallographic methods. The accuracy of n_c values derived from 2D images is affected by three factors: the procedure used for the segmentation, the procedure for measuring interface areas on the segmented objects and the pertinence of the hypotheses of the model underlying the data reduction scheme.

Tomography offers the possibility of characterizing an aggregate both directly on the 3D reconstructed object and indirectly via the analysis of 2D sections of the same object. In this paper, this possibility is exploited for assessing methods of measurement of grain coordination, average grain size and average grain contact area using 2D image analysis. A new method for estimating the average coordination n_c is proposed that assumes a random packing of spherical grains with a distribution of sizes. This method reveals more accurate than the method originally proposed by Gurland [2].

The analysis carried out in the present work is based on the results of an experimental characterization by X-ray microtomography of the microstructural evolution of an Al–15.8 wt.% Cu alloy held in the semi-solid state [5]. Only a summary of the experimental method is given here: the reader is referred to the original cited work for details.

The measurements were carried out on the ID15 beam line at ESRF, Grenoble. The specimen was a cylinder of 1.5 mm diameter and 3 mm height with a fine starting microstructure. It was placed on a rotating stage while being held in a small furnace at a temperature of 555 °C (the eutectic temperature of the alloy is 548.2 °C). The recording conditions were such that a spatial resolution of 2.8 μm was achieved with a scan time smaller than 15 s for a total of 400 projections. A scan was taken every minute from 1 to 80 min holding.

After reconstruction, the images were processed by carrying out successively (i) a 3D median filtering; (ii) a 3D thresholding of the solid and liquid phases on the greyscale images to obtain black and white images; and (iii) after smoothing of the generated surface, a segmentation of solid grains at each neck. Segmentation was performed on the 3D reconstructed volume using an Aphelion built-in routine called “ClusterSplitConvex”. This routine automatically computes a 3D distance function, i.e. the minimum distance from inside the solid phase to the liquid–solid interface, in order to create a basin centred on the solid grain. Then it applies a filtering, of which the strength is adjusted to limit the number of basins and avoid oversegmentation of the solid phase. Finally, it separates the grains using a 3D watershed method.

Alternatively, instead of performing segmentation on the 3D reconstructed volume, the segmentation could be done on 2D slices of this volume [6]. This second method will not be used in the present work as has been shown to be less reliable for the detection of the smallest grain contacts.

As already mentioned, measurement of the average grain size and neck size (i.e. grain contact area) requires a hypothesis about grain shapes. Fullman [7] established the relations for deriving, from parameters measured on 2D images, the average radius of a distribution of either spherical grains or disk-shaped platelets. The method involves the measurement of the distribution of the diameters, $D_{g2D,i}$, of the circles of intersection of the spherical grains with cross-sectional planes, or of the lengths, $D_{n2D,i}$, of the segments of intersection of the disk-shaped platelets with these planes. From these measured distributions, an average grain diameter \overline{D}_{g2D} or platelet diameter \overline{D}_{n2D} is derived as

$$\overline{D}_{2D} = \frac{N}{\sum_i \frac{1}{D_{2D,i}}} \quad (4)$$

where N is the number of measurements. Fullman [7] showed that, in the case of spherical grains or of circular necks, the average grain radius \overline{R}_g , or the average platelet radius \overline{R}_n , is obtained by the relation

$$\overline{R} = \frac{\pi}{4} \times \overline{D}_{2D} \quad (5)$$

That methodology was applied to the investigated Al–Cu slurry by assimilating the necks at grain contacts to thin circular disks randomly distributed in the body. As illustrated in Figure 1a and b, as grains are not perfectly spherical, their intersections with the image plane are not circular and the intersections of the necks are not perfectly straight segments. The distributions of the diameters $D_{g2D,i}$ of the grain sections and of the lengths $D_{n2D,i}$ of the neck sections were obtained from the 2D sections of the 3D-segmented object by measuring (using the Image J software) the primary and secondary axes of the ellipse that best approximates the grain sections (Fig. 1c) and the primary axis of the ellipse best fitting the neck sections (Fig. 1d). Grains and necks that touch the image border or that have an area less than 3 and 2 pixels², respectively, were neglected. The distributions were measured on the 250 slices available in the 3D-segmented object.

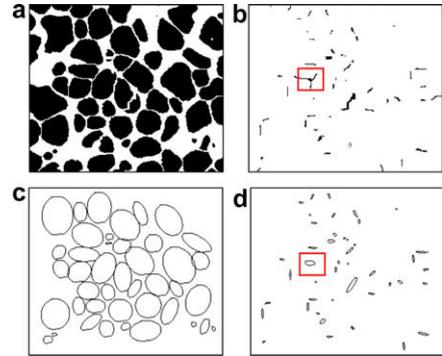


Figure 1. Two-dimensional section (263×228 pixels², i.e. $0.74 \times 0.64 \mu\text{m}^2$) extracted from the reconstructed volume of the specimen at 1500 s holding time, showing (a) the solid grains and (b) the necks, together with the best fitting ellipses for (c) grains and (d) necks.

The average coordination number n_c of the grains of a phase in a multiphase alloy can be expressed as

$$n_c = \frac{2N_{n3D}}{N_{g3D}} \quad (6)$$

where N_{n3D} is the number of necks per unit volume and N_{g3D} is the number of grains per unit volume. Making use of the expressions of N_{n3D} and N_{g3D} derived by Fullman [7] for the case of mono-sized spherical grains forming circular necks with identical diameters, Gurland [2] converted Eq. (6) into

$$n_c = \frac{16}{\pi^2} \left(\frac{N_{n2D}}{N_{g2D}} \right)^2 \frac{1}{c} \quad (7)$$

where N_{n2D} and N_{g2D} are, respectively, the numbers of necks and of grains observed per unit area on 2D sections and c is the contiguity, which can be derived from relation (3). In the present work, we propose an alternative method making use of the relationships demonstrated by Fullman [7] for populations of spherical grains and disk-shaped platelets with distributions of sizes:

$$N_{g3D} = \frac{2N_{g2D}}{\pi D_{g2D}} \quad (8)$$

and

$$N_{n3D} = \frac{8N_{n2D}}{\pi^2 D_{n2D}} \quad (9)$$

where $\overline{D_{g2D}}$ and $\overline{D_{n2D}}$ are given by relation (4). Eq. (6) then yields

$$n_c = \frac{8}{\pi} \left(\frac{N_{n2D}}{N_{g2D}} \right) \frac{\overline{R}_g}{\overline{R}_n} \quad (10)$$

While N_{n2D} and N_{g2D} must be measured on 2D sections, the fact that 3D reconstructions of the microstructure are available in the present work allows the use of either of two different values for the radii \overline{R}_g and \overline{R}_n in relation (10):

- the average equivalent radii calculated from the actual distributions of grain volumes and neck surface areas measured on the 3D reconstructed objects assuming spherical grains and circular necks;
- the average radii derived from analysis of the 2D sections using relations (4) and (5).

Let us first recall some of the results presented in Ref. [5]. The liquid volume fraction, u , was observed to progressively stabilize from an initial value $u = 0.34$ towards a plateau at $u = 0.32$ after more than about 1500 s holding: in order to focus only on the microstructural evolutions at constant u , we will thus consider in the following only the parameter evolutions from 1500 to 4800 s. Figure 2a presents the reconstruction of a few solid grains, illustrating the globular outlook of the grains and the size of the necks. It was shown in Ref. [5] that the globular grain shape remains fairly self-similar after 1500s. Figure 2b shows the evolution of the average volume of solid grains: the progressive coarsening of the microstructure at 555 °C amounts to a volume increase from about $4.87 \times 10^5 \mu\text{m}^3$ at $t = 1500$ s to about $6.14 \times 10^5 \mu\text{m}^3$ at

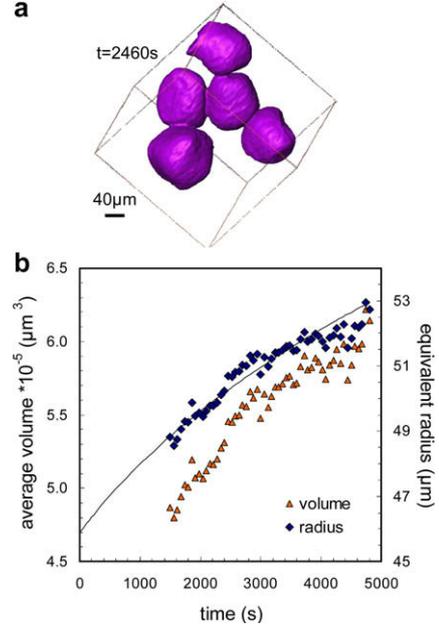


Figure 2. (a) Illustration of the grain and neck shapes, as visualized by tomography; (b) variation of the average size of grains with isothermal holding time (reproduced from Ref. [5]).

$t = 4800$ s, which translates into the equivalent sphere radius evolution shown in Figure 2b.

Figure 3 compares the evolutions of the average grain radius \overline{R}_g and average neck radius \overline{R}_n measured by two different methods:

- the points denoted “3D” are the averages calculated from the actual distributions of grain volumes and neck surface areas measured on the 3D reconstructed objects assuming spherical grains and circular necks [5];
- the points denoted “Fullman” are obtained from relations (4) and (5) using the distributions of grain and neck diameters D_{g2Di} and D_{n2Di} measured on 2D sections.

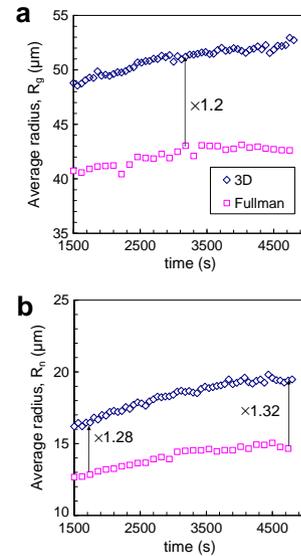


Figure 3. Evolution of (a) average grain radius, \overline{R}_g , and (b) average neck radius, \overline{R}_n . “3D” denotes points calculated from the distributions of grain volumes and neck surface in 3D reconstructed objects; “Fullman” denotes points obtained from 2D sections using relations (4) and (5).

Figure 3 shows that the use of Fullman’s relations brings about an underestimation of both \bar{R}_g and \bar{R}_n . This discrepancy results from the fact that the factor $\pi/4$ in Fullman’s relation (5) is strictly valid only for populations of spherical grains and circular platelets. This factor is likely to increase with increasing departure of grains and necks shapes from spheres and disks (obviously, the average aspect ratios of grains and necks are related). Empirically, Figure 3 suggests that, for the considered aggregate, the correct factor that should be used in Eq. (5) is similar in both cases: about $1.2 \times \pi/4$ for \bar{R}_g and $1.3 \times \pi/4$ for \bar{R}_n .

The average grain coordination number, n_c , was found to decrease continuously during isothermal holding, which means that microstructural coarsening also involves a change in the mutual arrangement of contacting grains. Figure 4 compares the evolutions of n_c , measured by four different methods:

- the points denoted “3D exact” are the n_c values directly measured on the reconstructed objects (these values can be considered as exact) [8];
- the points denoted “Gurland” are the n_c values derived from image analysis of 2D sections using Gurland’s relation (7);
- the points denoted “2D/3D” are the n_c values derived from relation (10) using the N_{n2D} and N_{g2D} data measured on 2D sections and the \bar{R}_g and \bar{R}_n values measured on the 3D objects;
- the points denoted “2D” are the n_c values derived from relation (10) using N_{n2D} , N_{g2D} , \bar{R}_g and \bar{R}_n , all measured on 2D sections (using relations (4) and (5) for the latter two).

Conspicuously, Gurland’s formula (7) is shown to bring about a large overestimation of n_c , whereas the n_c values computed from relation (10) are much closer to the “3D exact” values.

The origin of the large difference between the exact n_c values and the values derived from relation (7) can be ascribed to the fact that the Gurland’s model assumption of mono-sized particle and neck populations is not sufficiently verified in the investigated system. In particular, it is clear that necks present a wide size distribution.

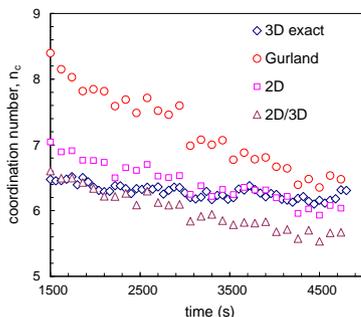


Figure 4. Evolution of the coordination number n_c measured either directly on the 3D reconstructed objects (“3D-exact”) or using Gurland’s relation (7), or using relation (10) with \bar{R}_g and \bar{R}_n calculated using relations (4) and (5) (“2D”), or using relation (10) with \bar{R}_g and \bar{R}_n measured on the 3D objects (“2D/3D”).

The present work reveals that n_c is more accurately estimated using Eq. (10), which accounts for the distribution of grain and neck sizes. In spite of the significant difference (Fig. 3) between the average diameters \bar{R}_g and \bar{R}_n measured either directly on the 3D reconstructed objects or on 2D sections using Fullman’s relations (4) and (5), Figure 4 shows that the “2D” predictions are only slightly larger than the “2D/3D” predictions and that, on average, the degree of agreement with the exact n_c values is similar. The use of relation (10) thus appears fairly “robust”. This is due to the fact that only the ratio \bar{R}_g/\bar{R}_n appears in Eq. (10): as the factor to be used in Eq. (5) in the case of non-spherical grains is similar for \bar{R}_g and \bar{R}_n (as shown in Fig. 3), the result is not much affected by the departure from Fullman’s hypothesis of populations of spherical grains and circular necks.

The evolution of the solid aggregate in an Al-15.8 wt.% Cu slurry maintained at a constant temperature has been characterized using in situ X-ray microtomography. Image analysis performed on 2D sections extracted from the 3D reconstructed objects provides measures of the number of solid–solid contacts and solid grains per unit area, and of the contiguity of the solid phase. The latter parameters allow estimation of n_c using the equation introduced by Gurland [2] for mono-sized spheres in contact via mono-sized necks. This n_c value is shown to overestimate the true n_c directly measured on the 3D objects. Comparison of measurements carried out on 3D objects and on 2D sections also allowed assessment of the equations derived by Fullman [7] for calculating, by 2D image analysis, average equivalent grain and neck radii in an aggregate of spherical grains with a distribution of sizes. Using these equations, a new equation is proposed for estimating n_c , which is much more accurate than Gurland’s equation in spite of the departure from Fullman’s hypothesis of spherical grains.

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