Quantitative Characterization of the Three-Dimensional Microstructure of Polycrystalline Al-Sn using X-Ray Microtomography

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Characterizing the three-dimensional topology of grain-boundary networks in polycrystalline materials is a crucial step in the modeling of properties that depend on the sample microstructure. Using absorption-contrast X-ray microtomography, we have carried out a large-scale microstructural characterization of polycrystalline Al doped with 2 at. pct Sn, which is immiscible in Al in the solid state. The segregation of Sn to the grain boundaries imparts a strong contrast in X-ray attenuation that can be reconstructed tomographically; however, the nonuniformity of the segregation process presents a formidable challenge to the automated segmentation of the reconstructions. By employing an iterative grain-finding algorithm followed by a novel grain-boundary-network optimization routine (based on a phase-field simulation of grain growth), we were able to extract reliable values for the size and topology of nearly 5000 individual grains in polycrystalline Al-Sn. The distributions and averages of these data deviate significantly from the corresponding microstructural parameters of the three-dimensional (3-D) Poisson–Voronoi tessellation often used to model polycrystalline samples. Much better agreement was observed with microstructures generated by the computer simulation of three-dimensional grain growth.

I. INTRODUCTION

The success of materials science as a scientific discipline rests to a great extent on the recognition that the properties of a material are determined largely by its microstructure. Essential to the study of structure-property relationships is the accurate and comprehensive characterization of microstructure[1]—an undertaking that has traditionally been carried out by optical or scanning electron microscopy applied to two-dimensional (2-D) sections of three-dimensional (3-D) samples or by transmission electron microscopy of very thin sections cut from bulk material. Although a great deal of information can be gleaned from such planar samples, there are a number of microstructural features that can be ascertained adequately only via a truly 3-D characterization procedure.[2,3] Examples of such features include the sizes, shapes, and spatial and orientation distributions of microstructural elements such as grains, precipitates, secondary phases, porosity, cracks, and grain boundaries.[4–7] Moreover, relationships between these elements, such as correlations and interconnectivities,[8,9] are inherently 3-D in nature and, therefore, difficult or even impossible to quantify on the basis of 2-D sections.

In the past, the three-dimensionality of materials microstructures has been investigated primarily by the technique of serial sectioning,[2,10–14] which, owing to its tedious nature, has been performed rather infrequently.[2] Recent advances in automation and data acquisition, however, have greatly improved the ease and speed of serial-sectioning studies.[8,15–17] In addition, promising results have been obtained from alternative methods for 3-D characterization, such as confocal microscopy,[18] X-ray tomography,[6,19–21] and X-ray microscopy.[22,23] All of these techniques provide the 3-D visualization of internal structure that is a prerequisite for a full characterization of the microstructure of a bulk material.

It is important to bear in mind, however, that visualization alone does not offer access to the quantitative microstructural information needed to elucidate the nature of structure-property dependencies. For this, it is imperative to develop efficient and reliable techniques for extracting the values of microstructural parameters from 3-D measurements[9,24–26]—a task known as image segmentation, and one that has proven to be just as challenging as obtaining the 3-D images to which the segmentation routines are applied.

This article focuses on the segmentation of 3-D images of cellular microstructures, such as those represented by the network of grain boundaries in a polycrystalline material or by the network of cell walls in a foam.[27] Among the many motivating factors for carrying out 3-D characterization of cellular systems is the strong influence exerted by the microstructure of such materials on the phenomenon of coarsening (in polycrystalline samples, usually referred to as grain growth), in which the average cell size increases monotonically with time, and the distribution of cell sizes evolves in a self-similar manner.[28,29] Owing to the space-filling nature of the ensemble of cells, this growth process is affected strongly by the local topology of the cells themselves.[30] In fact, for 2-D systems, the Mullins–von Neumann relation[31,32] indicates that topology is the dominant factor controlling microstructural evolution, as the rate of growth of each individual cell is strictly proportional to its number of sides. An analogous relation has often been speculated to hold in three dimensions, at least in a statistical sense.[33–36]
radiography. We have revisited the Al-Sn system using time-resolved mapping followed by optimization of the grain-boundary fraction of the grain boundaries. In order to quantify the microstructure of this material, it was therefore necessary to devise an image-segmentation scheme robust enough to identify the spatial extent of the individual Al crystallites, despite the absence in the tomographic reconstructions of a significant fraction of the grain boundaries.

To the extent that the missing grain-boundary information can be related to the noise and artefacts present in all 3-D visualizations of cellular materials, we propose that the image-segmentation method described subsequently may be applicable to a range of problems in 3-D microstructural characterization. In Section II, we describe the preparation of Al-Sn samples for tomographic study and the generation of reconstructed images from those measurements. In Section III, we present our image-segmentation procedure, which is a hybrid method involving an initial approximate grain mapping followed by optimization of the grain-boundary network. Finally, in Section IV, we compare the Al-Sn topological information obtained by the segmentation of tomographic reconstructions to the results of the previous study performed by Williams and Smith as well as to the properties of cellular microstructures generated analytically or via the computer simulation of grain growth in three dimensions.

II. EXPERIMENTAL

Ingots of Al-Sn with 2 at. pct Sn were prepared by annealing pieces of elemental Al (99.99 pct) and Sn (99.999+ pct) under vacuum at 750 °C in an alumina crucible and then allowing the melt to cool slowly (over ~2 hours) to room temperature. Pieces cut from the ingots were then remelted and cooled at a much faster rate (less than 1 minute) in order to suppress the extent of grain growth during solidification. Scanning electron microscopy images of slices through the ingots revealed that the Sn atoms segregated completely to the boundaries of the Al grains, regardless of the quenching rate. An average grain size of about 50 μm was observed after the second cooling step, and the grain size was found to be uniform throughout the entire volume of each ingot. In preparation for the tomographic measurements, samples of approximate dimension 0.5 × 0.5 × 5 mm³ were cut from the ingots with a wire saw and mounted on stainless steel posts 1 mm in diameter.

Microtomographic measurements were carried out at beamline ID22 of the European Synchrotron Radiation Facility, Grenoble, France, (ESRF) with monochromatic X-ray radiation at various energies between 10 and 25.6 keV, employing a combination of scintillator, magnification, and detector-channel rebin parameters chosen to yield an effective linear detector pixel dimension of 1.4 μm and a field of view exceeding 1.4 mm. The sample was rotated about its long axis in order to provide the set of distinct projections necessary for tomographic reconstruction. Typically, 625 projections were recorded with individual exposure times between 3 and 10 seconds, and then the sample was shifted along its long axis by 1 mm and the process repeated up to 4 times. In this manner, we were able to reconstruct contiguous regions in order to obtain a final volume much larger than that of a single tomographic measurement.

From each set of absorption-contrast microradiographs (Figure 1(a)), we computed the three-dimensional distribution of Al and Sn.