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The Three-Dimensional Microstructure of Materials: Measurement and Analysis

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Recent advances in computational and experimental techniques now allows for the routine visualization of the three-dimensional microstructure of materials. This opens new routes to explore the relationship between materials processing and structure. Examples of three-dimensional reconstructions of microstructure in systems ranging from solid-oxide fuel cells to dendritic solid-liquid mixtures will be given. The reconstructions of the solid-liquid mixtures are produced using a custom designed micromiller and focused ion beam milling. The micromiller allows approximately 200 serial sections to be obtained in one day with little operator intervention and works best for structures with length scales on the order of 10-100 microns. Focused ion beam milling along with a scanning electron microscope was used for the solid oxide fuel cell reconstruction. This technique allows structures with smaller length scales to be imaged, with separations between sections on the order of 90 nm or smaller.

Equally important is the analysis of the data. Using this three-dimensional information it is possible to quantify the morphology of complex microstructures using measurements of the interfacial shape distribution, the probability of finding a patch of interface with a given pair of principle curvatures, the spatial anisotropy of the microstructure via measurements of the normals to the interfaces, and the genus of the microstructure. For example, shown in Figure 1 is the evolution of the microstructure of a solid-liquid mixture in the Pb-Sn system as a function of time during coarsening. It is clear from the reconstructions that at later coarsening times the microstructure is strongly anisotropic with much of the surface area having normals perpendicular to the z-direction. At t = 2880 min. cylinders are present that have approximately elliptical cross sections with large aspect ratios. By t = 5780 min. the aspect ratios of these elliptical cross sections have decreased towards circularity. To quantify the evolution of the anisotropy, shown in Figure 2 is the probability of finding a normal of a patch of interface pointing in a certain direction. The projections shown in Figure 2 are equal area projections from the unit sphere. The projection plane is located such that a cylinder oriented along the z-direction would yield a horizontal line of nonzero probability along the center of the circle. Initially the structure is quite isotropic. As coarsening proceeds, a clear preferential directionality can be observed, see Figure 2c. This preferred orientation reveals a strong two-fold symmetry with the majority of the interface having interfacial normals in the x−y plane with most pointed along the x axis. The cylinders with approximately elliptic cross sections are the dominant shape at this time and their normals are responsible for this peak. During coarsening these objects evolve towards cylinders with circular cross sections. Thus the peak at the origin decreases in magnitude and a nonzero probability is observed along a horizontal strip.

We will also discuss the analysis of three-dimensional reconstructions in a wide array of systems such as the solid-liquid systems discussed above and solid oxide fuel cells.

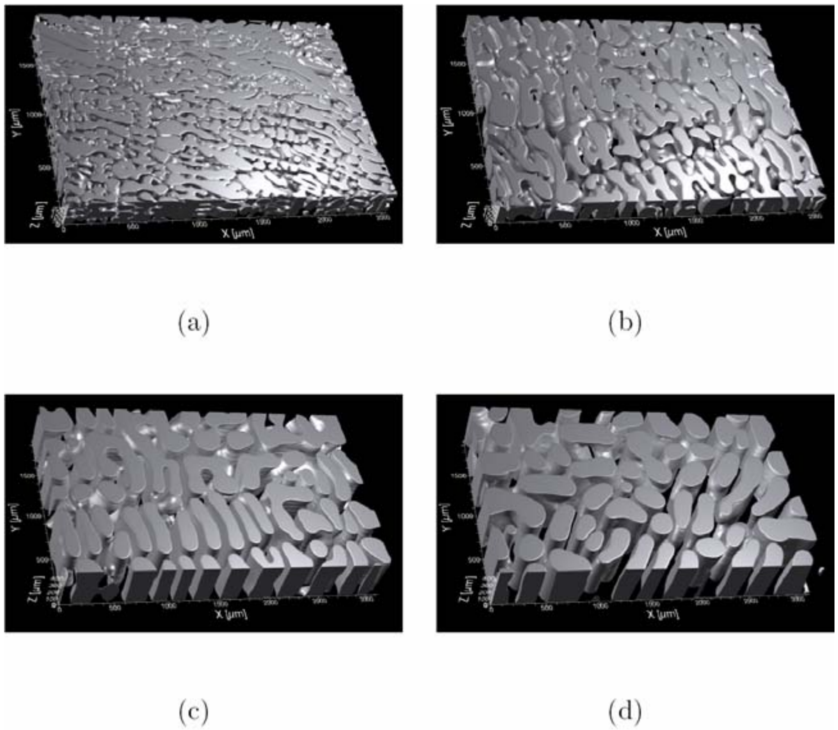

Figure 1. Three dimensional reconstruction of the microstructure of a Pb-Sn alloy coarsened for (a) 3 minutes, (b) 158 minutes (c) 2880 minutes and (d) 5760 minutes.

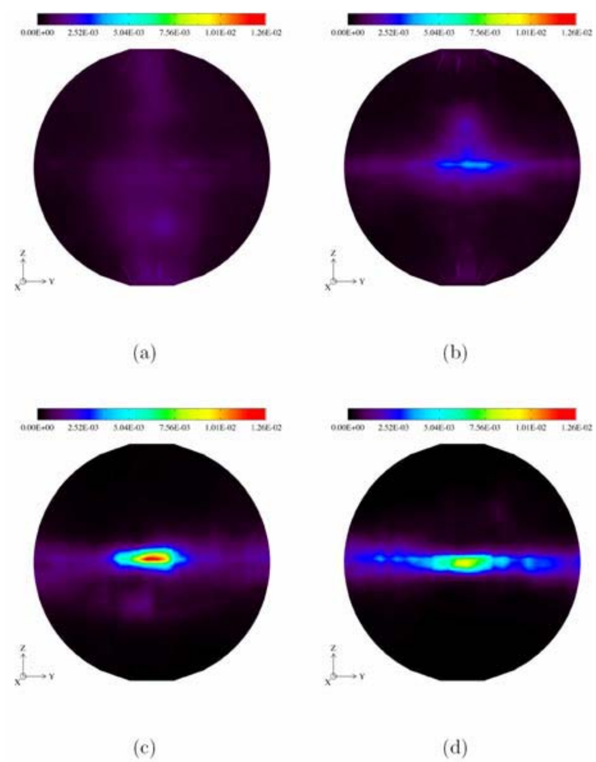

Figure 2. Interfacial normal distributions of the coarsened samples: (a) 3 minutes, (b) 158 minutes, (c) 2880 minutes (2 days), (d) 5760 minutes (4 days).

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