Research News

Phase Transition Seen at Alloy Grain Boundary

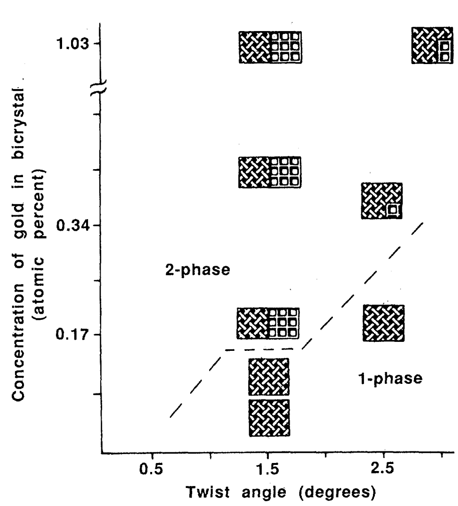
Cornell researchers have evidence for the proposition that gold in an iron alloy bicrystal preferentially collects at the grain boundary and triggers a change in its structure

A grain boundary is an interface between regions of different crystallographic orientation in a polycrystalline material. Materials scientists have speculated that a grain boundary of a given orientation might assume more than one structure; that is, it could undergo a phase transformation. Researchers from Cornell University now have evidence supporting this proposition for alloys consisting of iron and small amounts of gold. They have shown that increasing the gold concentration triggers a change in the grain boundary structure. Moreover, they have begun mapping out a phase diagram for the grain boundary by monitoring its structure as both gold concentration and boundary orientation are varied.

Apart from its academic interest as one of a growing number of two-dimensionally periodic systems exhibiting phase changes, grain boundaries in iron alloys have considerable technological importance. A problem of long standing, for example, results from the tendency of alloying elements, such as nickel and chromium, and impurities in certain types of steel to congregate at grain boundaries, which become weakened and make the alloy susceptible to fracture at low stress. In the metallurgical jargon this is called temper embrittlement of low-alloy steels.

Empirically developed sequences of high-temperature annealing have been developed that prevent segregation of alloying elements at grain boundaries, but materials scientists still do not understand the mechanism driving the embrittlement. One proposal, due to Edward Hart (now at Cornell), is that a two-dimensional phase transition at the grain boundary is responsible. Some materials scientists hope it may one day be possible to manipulate grain boundaries to minimize their deleterious effects while preserving their beneficial ones. At Cornell, Stephen Sass calls this practice interface science and engineering.

To study the influence of grain boundary segregation on boundary structure, Kurt Sickafus (now at the University of Cambridge) and Sass prepared so-called bicrystals that contained a single twist grain boundary between two crystalline regions. Twist means that the crystalline regions are rotated with respect to one another about an axis perpendicular to the boundary. Sickafus and Sass investigated boundaries that had twist angles from 1.5° to 3° and that were parallel to a cube face [(100) boundaries] in the body-centered cubic (BCC) crystal structure of iron. For these low angles, the structure of twist boundaries is described in terms of arrays of string-like (linear) defects called dislocations. Gold concentrations ranged from 0.092 to 1.03 atomic percent.



S. L. Sass, Cornell University

Grain boundary phase diagram

At low gold concentrations, the grain boundary structure consists of a network of <110> dislocations. At higher concentrations, either <110> alone or both <110> and <100> networks appear, depending on the orientation of the twist grain boundary. The area of the patterns indicates the relative amount of each network.

The Cornell researchers used conventional transmission electron microscopy to image the grain boundary structure. Although this technique cannot resolve individual atomic positions, it clearly shows the dislocation networks that make up the grain boundary. In pure iron with a 1.5° twist boundary, the network consists of a square array of dislocations that looks something like the squares in a chocolate bar. The dislocations lie around the edges of each square, which is about 100 angstroms wide, and they run in the directions of the diagonals of the BCC cube faces (<110> directions); that is the dislocation network is rotated 45° with respect to the cubic lattice.

When gold was incorporated into the iron with a concentration of 0.092 atomic percent, the grain boundary structure remained unchanged. But when the concentration reached 0.17 atomic percent, the investigators observed that some regions of the twist boundary retained the <110> network, but others exhibited a square network oriented so that the dislocations ran in the directions of BCC cube edges (<100> directions). Raising the gold concentration to 1.03 atomic percent resulted in the same mixture of <110> and <100> networks.

If the gold concentration was held at 0.17 atomic percent and the twist angle was increased to 2.5°, the grain boundary structure once again consisted of the <110> dislocation network. But, when the concentration was raised to 0.4 atomic percent, small patches of the <100> network appeared. The figure summarizes these and other results.

Sickafus and Sass interpret these findings in terms of a quasi-two-dimensional phase transition similar to the structural transformations that occur in multicomponent three-dimensional solids, with the <110> and <100> networks representing the two phases. (Since a grain boundary has a finite thickness, it is not strictly two dimensional.) The transformation is driven by the increasing concentration of gold at the grain boundary. Above a certain concentration, the two phases coexist. If it were possible to incorporate enough gold, presumably only the <100> network would persist. The twist angle plays the role of a thermodynamic variable that alters the concentration range over which the two networks can exist together.

For this interpretation to be correct, it is necessary to show that gold actually is associated with the phase transformation. If the gold is present in higher concentrations at the grain boundaries than in the crystalline regions and if the concentration in the <100> network were higher than that in the <110> network, that would suggest gold is at least related to the transition. The Cornell researchers verified that this was indeed the case by means of two techniques. Rutherford back scattering, which is an ion scattering technique that yields the concentration of gold with depth, showed that the gold was concentrated in the vicinity of the plane of the grain boundary. And energy-dispersive x-ray spectrometry in a high-resolution electron microscope demonstrated that the gold concentration was several times higher in the regions of the grain boundary described by the <100> network. ■

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ADDITIONAL READING

K. E. Sickafus and S. L. Sass, "Grain boundary structural transformations induced by solute segregation," *Acta Metall.*, in press.

Tokamak Sets Records In Temperature and Confinement

In an uncommonly successful series of experiments this year, researchers at the Princeton Plasma Physics Laboratory's Tokamak Fusion Test Reactor (TFTR) have set two new records for a magnetically confined plasma: a temperature of 200×10^6 K, and a confinement parameter of 1.5×10^{14} seconds per cubic centimeter. These milestones in turn put the TFTR scientists within striking distance of energy breakeven, in which the plasma produces as much fusion energy as is required to heat it up.

"What these experiments show is that physics is not the restriction," says Dale M. Meade, head of the laboratory's experimental division.

The breakeven experiments themselves, which were originally scheduled for this year and which will require the installation of special remote-handling equipment to deal with radioactive tritium fuel, have been delayed until late 1989 by funding constraints in the Department of Energy. In the meantime, as Meade suggests, the TFTR scientists have been exploring the basic plasma physics questions using nonradioactive deuterium.

In April, for example, the researchers achieved their record confinement parameter by first establishing a relatively low-density plasma in the tokamak, and then injecting high-velocity pellets of frozen deuterium using an injector developed at the Oak Ridge National Laboratory. As the pellets evaporated they deposited a high density of deuterium ions in the center of the plasma. Measurements of the resulting Lawson parameter—defined as the product of the particle density times the confinement time of the plasma—showed 1.5×10^{14} seconds per cubic centimeter, or nearly twice that of the previous record achieved by the Alcator C tokamak at the Massachusetts Institute of Technology in 1983. This value of the Lawson parameter is only about a factor of two below that needed for energy breakeven.

The Lawson number is a measure of the efficiency of plasma confinement, and is one of the two key parameters that determine whether or not a fusion reaction will produce net positive energy. The other is plasma temperature. This June, in another series of experiments, the TFTR scientists pushed toward their record temperature using a combination of powerful auxiliary heating and enhanced energy confinement.

The heating was accomplished by the well-established technique of neutral beam injection, in which the plasma is bombarded with intense beams of electrically neutral deuterium atoms. The TFTR has four such injectors, developed by the Lawrence Berkeley and Lawrence Livermore National Laboratories. Together they have a full-power rating of 27 megawatts, although for these experiments they were only used at half power.

The confinement scheme grew out of a recognition in the early 1980's that high-powered plasma heating can actually cause a deterioration in confinement. It turns out that good confinement at high temperatures requires a density profile that is sharply peaked in the center. However, when the plasma is heated to very high temperatures, ions tend to diffuse outward from the center and build up near the chamber walls, thus creating a relatively flat density distribution. The trick is to get rid of those ions.

In many fusion reactors, including all those being designed for the future, this is easy enough: magnetic "divertors" strip the ions away from the walls harmlessly. But TFTR was designed before the phenomenon was fully understood, and it has no divertors. Thus, the TFTR researchers have had to rely on ingenuity. It happens that the walls of the chamber are lined with graphite tiles. The researchers thus spent several weeks conditioning the graphite, hitting it with plasma pulse after plasma pulse so as to drive out all the absorbed gasses; their idea was that clean graphite would absorb the errant deuterium ions rather than letting them build up. And that is exactly what happened: in a series of test runs beginning 12 June, the scientists were able to reach temperatures of 200×10^6 K at a Lawson parameter of 10^{13}. This is well in excess of the minimum temperature needed for breakeven, although the Lawson parameter in this case is about a factor of 20 too low. The previous record of 80×10^6 K was set in 1980 at the Princeton Large Torus.

These same experiments also showed some preliminary evidence for the so-called bootstrap current. First predicted theoretically in 1971, this current is supposed to arise spontaneously in hot, high-density tokamak plasmas, and to flow in such a way as to sustain the confining magnetic field with a minimum of input from external transformers. If real, it will be a key to making fusion reactors that can produce a steady level of power instead of pulsing on and off as present-day tokamaks do.

Meanwhile, the obvious next step for the TFTR team is to combine pellet injection with high-intensity heating techniques so as to advance the Lawson parameter and the temperature simultaneously. In addition to heating the plasma by neutral beam injection, the researchers also plan to use a technique known as radio-frequency heating. In any case, by next year the current schedule calls for the demonstration of conditions in the deuterium plasma equivalent to breakeven in a tritium plasma. ■

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