Nanostructures in 3D

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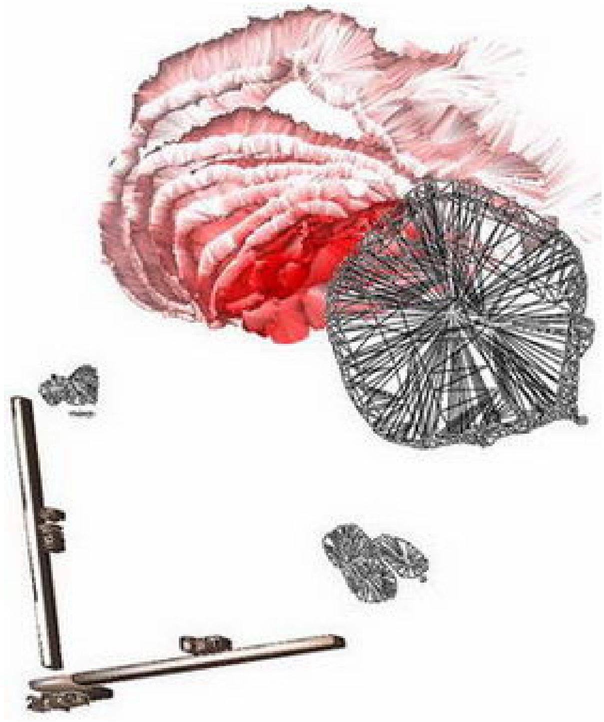

The 3D gradient of the crystallographic orientations in an intermetallic iron-aluminium specimen (lattice curvature) close to a very hard Laves phase (appears as a transparent net). The various layers of colour indicate increasing changes in the crystal orientation from the reference point directly at the interface between matrix and Laves phase in two degree misorientation intervals. Image: Max Planck Institute for Iron Research

Max Planck researchers from Düsseldorf unveil the first three-dimensional electron microscope for examining nanomaterials structure. It is the world's first electron microscope for simultaneously and automatically investigating in three-dimensions the phase content, crystallographic texture, and crystal interfaces of materials - co-designed and put into service at the Department of Microstructure Physics and Metal Forming at the Max Planck Institute for Iron Research in Düsseldorf, Germany. The device contains a high-resolution scanning electron microscope and an -ion-beam microscope.

In the past, these two types of microscopes have been used separately; now, they have been integrated into a single instrument together with an arsenal of detectors which can measure electron diffraction patterns and orientations, as well as perform chemical analyses. This allows scientists to see the inner structure of nanomaterials, biological matter, and high-performance steels, in ways that other microscopic procedures cannot - and in full 3D.

The chance to investigate microstructures three-dimensionally is of great use to materials scientists. Until now, there have been two ways to make such observations. The first is employing x-rays or electron or neutron beams, respectively. The advantages of these methods are that it they are not destructive and let scientists reconstruct the three-dimensional structure. But this process is also time-consuming and provides only limited information - particularly for crystalline materials. Also, the lateral resolution is about two orders of magnitude below that of the new 3D-approach the Max Planck scientists have now unveiled, which reaches at least 40 cubic nanometres.

The second option has been to take an image of the material in slices, and then reconstruct it tomographically in three-dimensions. The new microscope makes three-dimensional images by first taking a two-dimensional picture using the desired crystallographic or chemical method. Then, with nanoscopic precision, an ion beam slices off a piece of the material - and the layer underneath can be analyzed. The researcher can then investigate the material slice-for-slice, and in the end, the device produces a digital three-dimensional image.

The microscope functions fully automatically. This allows relatively large areas to be investigated - for example, volumes of 70 x 70 x 70 micrometres. The powerful combination of automatically slicing off material and looking at it in high resolution produces a range of crystallographic information much broader than most other microscopy techniques. That information includes the exact form of the embedded crystals, the position and crystallographic characteristics of internal interfaces, the density of defects in grains, and very fine textural details. All these characteristics can be measured at a lateral resolution of about 40 cubic nanometres - and depending on the material, even more finely.

Max Planck researchers working with Prof. Dierk Raabe and Dr. Stefan Zaefferer have already looked at steel-related iron-aluminium intermetallic alloys. These alloys are characteristically highly resistant to oxidation and sulphidation at high temperatures. On the other hand, they are not yet completely hard and homogenous. For this reason, the scientists are adding tiny particles and chromium, in an attempt to refine the materials for technical application. Such alloys could be used in newly-designed high-temperature gas turbines for conventional power plants, allowing plants to operate more efficiently and ecologically, and bringing down energy costs.

In the case of the iron-aluminium intermetallic alloy, Raabe and his colleagues are using the instrument to look at the effect of embedded particles on the microstructure and macroscopic characteristics of the material. In particular, the scientists investigated how tiny inclusions in the intermetallic matrix influence the orientation of the alloy’s crystal lattice.

In hot-formed samples the scientists were able to observe for the first time in 3D how soft crystal orientations of the matrix developed substantial orientation gradients around hard particles. The crystallographic orientations took on specific patterns, characterised by the building up of systematic orientation gradients in a number of successive layers which had increasing orientation distance from the interface with the hard inclusion. Because of these strong crystallographic gradients, new seed crystals developed, homogenising the material and improving its mechanical characteristics in view of high-temperature application in modern power plants.

Original work:

J. Konrad, S. Zaefferer, D. Raabe
Investigation of orientation gradients around a hard Laves particle in a warm rolled Fe3Al-based alloy by a 3D EBSD-FIB technique
Acta Materialia 54 (2006) 1369

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