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ENERGY-FILTERED IMAGING OF PRECIPITATES IN FeCuN ALLOYS

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The microstructures of a series of model ferritic alloys are being characterized by transmission electron microscopy (TEM) techniques. The alloys have varying concentrations of Cu (0 to 0.9 wt%), N (5 to 120 appm), and other solutes to allow a systematic study of the effects of various elements on embrittlement. The first step in this process is the characterization of the alloys in their as-received state: solution treated at 775°C for 17 h, quenched in a salt bath at 450°C for 180 s, and subsequently air cooled.

The imaging portion of this initial study is complete; however, several questions about the chemical content of many of the precipitates remain unanswered. The heat treatment described above resulted in extensive precipitation, especially in the high N alloys. It is difficult to distinguish between α'' nitride platelets and the ϵ -carbide platelets which formed in many of the alloys. High-angle tilting is difficult with these ferromagnetic specimens, and the $g \cdot R = 0$ invisibility criterion previously used for distinguishing the α'' nitrides¹ has given ambiguous results. It is necessary to use analytical methods to determine the chemical content of the platelets. To this end, energy-filtered imaging is being used to identify these and other precipitates in these alloys. A Gatan Imaging Filter (GIF), installed on a Philips CM30 operated at 300 kV, was used. Figure 1(a) shows a conventional TEM image of a 0.17 wt% carbon alloy with $g = 011$ and beam direction near $[100]_{\alpha}$; a large edge-on (010) platelet runs diagonally across the image. Figure 1(b) shows the C elemental map of the same platelet acquired with a 30eV window centered at 298 eV and with the background calculated from two pre-edge, 30eV windows centered at 217 eV and 258 eV. The image clearly shows that the platelet contains C, identifying it as an ϵ -carbide, as well as the fact that the platelet apparently formed on or near a spherical carbon-containing precipitate.

To study the affect of Cu precipitation on the hardening of the alloys, a 0.9 wt% Cu alloy was annealed at 550°C for times up to 15 h. The Cu precipitates that form undergo a phase change from bcc to a faulted fcc (9R) crystal structure when they are about 6 nm in diameter.² These fcc precipitates have been well characterized by conventional TEM techniques for these alloys. However, the smaller bcc Cu precipitates are difficult to image in these strongly oxidation-prone specimens. Energy-filtered imaging is being evaluated as a possible technique to image and measure the size distribution of the small Cu precipitates. Figure 2 shows examples of energy-filtered images from an annealed .9 wt% Cu alloy. Figure 2(a) shows the Fe jump-ratio image calculated by dividing an image acquired with a 30eV post-edge window centered at 725 eV by an image acquired with a 30eV pre-edge window centered at 685 eV. Figure 2(b) shows the Cu jump-ratio image for the same region with images acquired at 948 eV and 908 eV. Black arrows mark the same precipitates in (a) and (b) clearly showing that the Cu precipitates appear as a lack of signal in the Fe image. These preliminary results are very promising, and the search for smaller Cu precipitates is in progress.³

References

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3. This research is sponsored by the Division of Materials Sciences, U.S. Dept. of Energy and the Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission under interagency agreement DOE 1886-8109-8L with the U.S. Dept. of Energy under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems Inc. and by an appointment of P. M. Rice to the ORNL Postdoctoral Research Associates Program administered jointly by ORNL and Oak Ridge Institute for Science and Education.

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Fig. 1.- Edge-on platelet which precipitated in a high-C (0.17 wt%) alloy.
 (a) Conventional TEM image with $g = 011$ (white arrow) and beam direction near $[100]_{\alpha}$.
 (b) Carbon elemental map calculated from energy-filtered images recorded with 30eV pre-edge windows centered at 217 eV and 258 eV, and a 30eV post-edge window centered at 298 eV.

Fig. 2.- Fcc Cu precipitates in an annealed high-Cu (0.9 wt%) alloy. The black arrows point the same precipitates in both (a) and (b).
 (a) Fe jump-ratio image calculated by dividing an energy-filtered image acquired with a 30eV post-edge window centered at 725 eV by a pre-edge image centered at 685 eV.
 (b) Cu jump-ratio image of the same region calculated by ratioing images acquired with 30eV post-edge and 30eV pre-edge windows centered at 948 eV and 908 eV, respectively.

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