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Chemical & Materials Science

Polychromatic X-ray Microdiffraction (PXM) of Local Stress Corrosion Cracking in Alloy 600 using the VESPERS Beamline

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Introduction

Nickel-based alloy tubes, such as Alloy 600 (A600), used in most of the older pressurized water reactor (PWR) power stations are susceptible to stress corrosion cracking (SCC). In fact, SCC of A600 tubing is the single most important reason that nuclear steam generators are replaced. Local mechanical stresses from scratches and dents can result in strains at critical grain intersections leading to enhanced initiation of chemical attack.

Such local mechanical stresses can be detected non-destructively by a Laue X-ray diffraction (XRD) technique that is now available at the VESPERS beamline at the CLS. The technique uses a finely focussed polychromatic (white) beam to create diffraction patterns from a volume under a spot of 1-2 µm diameter on the sample surface. All allowable planes in the volume, satisfying the Bragg condition, give reflections and these are detected on a charge-coupled device (CCD) screen above the sample. This Laue based technique eliminates the need for sample rotation thus simplifying spatial mapping compared to Bragg diffraction.

Indexing of the diffraction patterns with respect to a reference strain-free model of the same crystal provides the crystallographic orientation as well as the deviatoric elastic strain tensors for each direction in the stressed sample. As well, the shape of the diffraction spots gives information on the plastic strain sustained by the crystal as expressed by the direction and density of the dislocations. By a raster scan of the sample, maps of the crystallographic orientation, strain tensors and von Mises strains are obtained [1, 2]. During the scan, X-ray fluorescence spectra are also obtained for each point, thus providing an elemental composition map of the same area.

The strain(stress) fields have been studied in samples of Alloy 600 tubing that were exposed to SCC conditions that led to the formation of microscopic cracks in the tubing exterior surface. Polished cross sections of the tubing were prepared and areas with cracks were identified. A series of Laue XRD and X-ray Fluorescence (XRF) maps were collected on the VESPERS beam using a step size of 3 micrometres. The polychromatic photon beam flux is so intense that the diffraction patterns appeared on the CCD screen within a fraction of a second. However, at present, a delay in the data transfer from the CCD requires a wait time of 5 seconds between image acquisitions at each step across the sample. At present, the Laue diffraction data is indexed after conclusion of the experiment using software kindly provided by Dr. B. Larson of Oak Ridge National Laboratory. However, on-line processing is under active development.

Science

The cross-section can be seen in the scanning electron microscope image (Figure 1a) showing a crack. The microscopic Laue XRD patterns are indexed to provide a map of the local crystallographic orientations (Figure 1b) showing each grain in the nickel alloy, and the relationships of the crystallographic directions to each other. The grains indexed do not exactly correspond to those seen in Figure 1a: information in the latter case comes from the near surface, while in the case of Laue maps, the information is often produced tens of micrometres below the surface. None the less, there are sufficient common features in Figures 1a and 1b that the locus of the (visible) crack has been identified in the Laue map and it is outlined in green. The dark regions in the map are those that were not able to be imaged often because of local major plastic deformation. Many of these are seen to occur along the crack locus as well as ahead of the apparent crack tip. The indexed patterns can also be analyzed to determine the (residual) local elastic strains. A map of the von Mises strain (Figure 1c) shows localized high elastic strain near the position where the tip of the visible crack is observed in the Laue XRD data.

Discussion

Polychromatic X-ray microdiffraction for the measurement of microscopic stresses in materials will see increased use at VESPERS in the next couple of years because of a worldwide demand for access to such facilities. However, PXM has a much larger potential for use in the identification of unknown crystalline phases in geochemistry and materials science. While PXM does not provide a cell parameter as does Bragg diffraction, its pattern could be indexed with cell parameters of candidate materials until a match is found. Such a process is made more possible by the availability of high speed indexing operations using cell processors.

References

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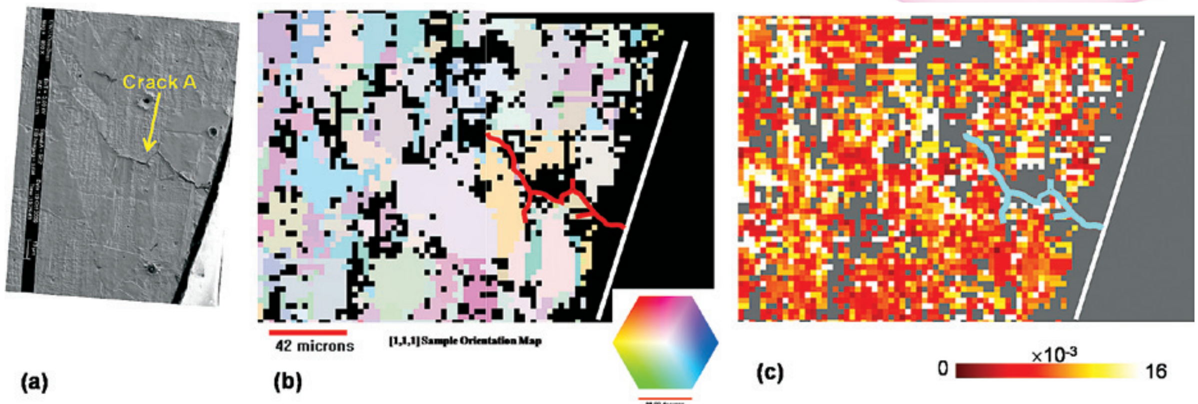

Figure 1: (a) Scanning elecron microscope image of a cross section of Alloy 600 that had been exposed to reactor chemistry conditions known to cause stress corrosion cracking. (b) Laue XRD map of the section showing the crystallographic direction of each grain with repect to the 111 pole. Near the outer surface (see white line), no diffraction patterns can be indexed and therefore no map is produced; this is believed to be caused by high plastic deformation near the outer surface as a result of sample preparation processes(1). Figure 1(c) shows a plot of von Mises (VM) elastic strain that is calculated from the geometric mean of the six deviatoric strain components; regions of low VM strain are characterised by red while the highest strain regions are white.