Mechanical Indicators for Initiation of Stress Corrosion Cracking in Ni Alloy Steam Generator Tubes

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Currently the majority of tubing used in new and replacement nuclear power reactor steam generators for CANDU reactors is manufactured out of Incoloy 800 (IN 800). This alloy is corrosion resistant and is less susceptible to primary water stress corrosion cracking and secondary side intergranular attack than its predecessor, Incoloy 600. The University of Western Ontario (UWO), McMaster University, the University of Toronto, and Queen's University have undertaken a collaborative program to try to understand the local stress conditions that can lead to both forms of SCC. The Candu Owner's Group (COG), Ontario Centres of Excellence and Emerging Materials Knowledge Ontario fund the project. One thrust of the project is to map stresses on a sub-grain scale using micro-Laue diffraction at the Advanced Photon Source (APS), Argonne National Laboratory. This unproven technique must be validated by comparison with proven techniques and modeling. To this end an experiment has been set up to compare the results of micro-Laue to neutron diffraction measurements extrapolated to the grain scale by modeling using both self consistent polycrystalline models and finite element polycrystalline models.

Three tensile test specimens were cut from 6 mm thick IN 600 plate and heat treated at 800°C for 1 hour. The samples were 5 mm x 5 mm x ~ 20 mm with the long axis parallel to the rolling direction (RD), which was the direction of the applied load. The specimens were then strained in tension to total strains of 0, 1 and 10% at UWO. EBSD orientation maps, micro-Laue orientation maps, and micro-Laue surface residual strain maps were made on a small area on each specimen. Bulk residual strain measurements were performed on the L3 beam line using a monochromatic beam and a 32-wire position sensitive detector. Measurements were made using four diffraction peaks ({111}, {200}, {220}, {311}). Five specimen directions were investigated: the three principal directions (RD, ND, TD), and two more directions in the RD-ND plane at 30° and 60° from RD.

For the neutron diffraction residual strain measurements, the zero-stress reference lattice parameter determined from a control sample was 0.35564 ± 0.00004 nm. This value is an average determined from all the measured d-spacings for the control sample (five values for four hkl planes). The quoted uncertainty is the sum in quadrature of the uncertainties in each value used in the average, which arise from the peak fitting and wavelength calibration. The neutron diffraction residual lattice strains for the specimen strained to1% are presented in Table 1. The table shows that the measured strains are small, of the order of magnitude of the uncertainty in many cases.

Table 1: Residual strains in specimen strained to 1% total strain

Direction	hkl	Strain (με)
RD	111	-134 ± 142
	200	-140 ± 132
	220	-63 ± 137
	311	-112 ± 132
ND	111	-112 ± 273
	200	466 ± 254
	220	128 ± 114
	311	-25 ± 136
TD	111	-34 ± 244
	200	135 ± 189
	220	-199 ± 148
	311	-53 ± 108
RD + 30	111	19 ± 165
	200	54 ± 180
	220	-118 ± 140
	311	98 ± 139
RD + 60	111	-190 ± 127
	200	-237 ± 66
	220	-105 ± 150
	311	-96 ± 149

Micro-Laue x-ray diffraction is a technique for studying mesoscale structure such as crystalline phase, local orientation, and local defect distribution including elastic and plastic strains [1, 2]. Micro-Laue x-ray diffraction lattice strain measurements at the APS were made in individual grains for the control (undeformed) and the 1% specimens.

For each crystal, the data consisted of a strain matrix for the crystal principal directions, a strain matrix for the sample principal directions, and crystal orientation. Multiple volume elements within each grain were analysed. i.e. The volume elements were smaller than the grains. The Laue data for a grain (that was compared to the ND data for a grain family) came from multiple measurements. Data from all of the volume elements were then aggregated and grouped into sets according

Table 2: Comparison of strains in selected grains in the 1% sample

Grain	Strain Direction	Strain (ND, με)	Strain (Laue, με)	Angle (ND to Laue)
1	ND (200) ND (111)	466 ± 254 -112 ± 273	ε_{zz} = 440 ± 1016	28.2 27.0
2	TD (200)	135 ± 189	ε_{yy} = 795 ± 1592	9.1
3	ND (200) ND (111)	466 ± 254 -112 ± 273	ε_{zz} = 486 ± 1104	33.5 35.6
4	TD (200) ND (111)	135 ± 189 -112 ± 273	ε_{yy} = 1261 ± 642 ε_{zz} = -582 ± 466	26.8 26.0

to crystal orientation. It should be recalled that micro-Laue diffraction yields deviatoric strains while neutron diffraction yields normal strains.

The orientation information from micro-Laue and neutron diffraction was used to match similar grain orientations studied using the two different techniques. This allowed the comparison of lattice strains measured using the two different methods. The results are shown in Table 2. The table shows that it is generally difficult to match orientations precisely (last column, Table 2). This is a consequence of the relatively small number of grains sampled by micro-Laue diffraction compared with neutron diffraction. The large grain population sampled in neutron diffraction means that, in a typical powder specimen, there are generally enough grains with a particular orientation with respect to the specimen axes to produce a good-quality signal. On the other hand, the small number of grains sampled in a typical micro-Laue diffraction experiment means that the probability of sampling a specific grain orientation is low.

It is difficult to draw conclusions from the data in Table 2 because of the large uncertainties in the micro-Laue diffraction data, and the very small strains, which are comparable to the uncertainties for both techniques.

The neutron measurements provide data for validation of the micro-Laue measurements. There is general agreement between the two sets of measurements, validating the development of the Laue technique. However, the comparison highlights the large uncertainties associated with the Laue measurements. The possibility of doing in-situ neutron diffraction measurements is being explored.

References

[1] G.E. Ice and B.C. Larson, Adv. Eng. Mater., 2, 643 (2000)
[2] B.C.Larson, W.G. Yang, G.E.Ice, and J.D. Budai, Nature, 415, 887 (2002)