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Technical Note

CONSIDERATIONS FOR METALLOGRAPHIC OBSERVATION OF INTERGRANULAR ATTACK IN ALLOY 600 STEAM GENERATOR TUBES

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ABSTRACT

This technical note provides some considerations for the metallographic observation of intergranular attack (IGA) in Alloy 600 steam generator tubes. The IGA region was crazed along the grain boundaries through a deformation by an applied stress. The direction and extent of the crazing depended on those of the applied stress. It was found that an IGA defect can be misevaluated as a stress corrosion crack. Therefore, special caution should be taken during the destructive examination of the pulled-out tubes from operating steam generators.

1. Introduction

Intergranular corrosion of nuclear steam generator tubes can be divided into at least three forms: intergranular stress corrosion cracking (IGSCC), intergranular attack (IGA), and intergranular penetration (IGP) [1]. In the case of IGSCC, the corrosion morphology consists of single or multiple major cracks with minor to moderate amounts of branching. The morphology of IGA is characterized by a relatively uniform attack of numerous grain boundaries to a uniform depth over the surface of the metallic materials. This is because corrosion is localized at and adjacent to grain boundaries with relatively little corrosion of the grains. Finally, IGP can be described as a mixture between the other two forms.

IGA has been one of the major corrosion degradation modes in steam generator tubes. It has been observed mainly on the outer diameter (OD) side of the tubes in the sludge piles on top of the tubesheet or in the deposits adjacent the tube support structures [2–5]. However, accidental ingress of thiosulfate into the primary water led to

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extensive IGA on the inner diameter (ID) side of the sensitized tubes [6].

We have found that steam generator tubes with IGA are easily crazed along the grain boundaries when under plastic deformation. Corroded grain boundaries would lose fracture toughness and become brittle. Therefore, a region with IGA/IGP defects is expected to be susceptible to cracking by an external stress. This article provides the metallographic characteristics of IGA in Alloy 600 steam generator tubes. In addition, the effect of the applied stress on the morphology change of the IGA region is discussed.

2. Materials and methods

The Alloy 600 tubing material used in this study was supplied by a commercial vendor. The tubes were mill-annealed in a temperature range of 1,024~1,070°C for 3 minutes and then cooled down to 500°C within 7 minutes. The nominal OD of the tube was 19.05 mm and the nominal wall thickness was 1.07 mm. The chemical composition is listed in Table 1. To accelerate intergranular corrosion, the tubes were additionally sensitized at 590°C for 10 hours in a vacuum furnace under about 5×10^{-6} torr.

Samples were prepared by cutting the tube circumferentially into 6-cm-long pieces. For manufacturing IGA on the inner side of the tubes, one end of each tube specimen was plugged with a Teflon cap so that the solution inside the tube did not leak out. Next, the tube specimen was filled with an oxidized solution of 0.1M sodium tetrathionate ($Na_2S_4O_6$). Solutions containing sulfur oxyanions has been known to accelerate the corrosion of nickel-based alloys and stainless steels along the grain boundaries [7,8]. By contrast, to produce IGA on the OD side of the tube, both ends of each tube specimen were plugged with Teflon caps. Next, the tube specimens were immersed in 0.1M $Na_2S_4O_6$ solution. In this way, IGA was grown on the ID or OD side of the tube at room temperature for 5 days.

The IGA tubes were deformed by applying several types of stress, such as hoop stress, three-axes stress, hard rolling, and indentation. If necessary, the tubes with IGA were cut into pieces of appropriate size. The subsequent morphology changes of the IGA area were observed using scanning electron microscopy. The detailed information about how stress or deformation was applied to specimens and where the morphology was observed in the specimen is described in the results and discussion section. Because this work is focused on the morphology change of the IGA specimen by an applied stress, the magnitude of the applied stress and the corresponding deformation extent are not quantified.

Table 1 – Chemical composition of Ally 600 tube (wt %).

C	Cr	Fe	Ni	Si	Mn	Ti	Al	S
0.025	15.52	9.30	Bal.	0.19	0.21	0.29	0.22	<0.001

Al, aluminum; C, carbon; Cr, chromium; Fe, iron; Mn, manganese; Ni, nickel, S, sulfur; Ti, titanium.

3. Results and Discussion

Fig. 1 shows the morphology change of the IGA tube surface by an applied stress. No defects were observed on the ID surface of the tube without any applied stress conditions, as shown in Fig. 1A. However, some crazing occurred along the tube axial direction when applying hoop stress by bending the specimen along the circumferential direction of the tube, as shown in Fig. 1B. The arrows indicate the same location before and after deformation. They just look like axial stress corrosion cracks (SCCs). Similarly, some crazing occurred along the circumferential direction of the tube by applying tensile stress. Therefore, they seem to be circumferential SCCs. When three-axes stress was applied to the IGA tube specimen, the surface was crazed into a radial crack-like morphology (Fig. 1C). Finally, the numerous attacked grains were apparently revealed through a distorted deformation (Fig. 1D). These results indicate that an IGA can be misunderstood as a SCC by a directional deformation. Similar behaviors were also observed on the tubes with IGA on the OD side. In the IGA region, the attacked grain boundaries become brittle, although they are extremely tight in nature. Therefore, the corroded grain boundaries are easily opened through a deformation by an externally applied stress.

Fig. 2A shows a circumferential cross section of the IGA tube. There was no evidence of IGA on the as-polished metallographic sample. However, when the same tube was expanded outward by hard rolling, IGA was clearly revealed by a crazing of the IGA region, as shown in Fig. 2B. The scratch and arrow indicate the same location before and after deformation.

Fig. 3A shows a circumferential cross-section of the IGA tube. There is no doubt that the feature of the defect type is a single SCC. In this case, it is reasonable to call this flaw a primary water stress corrosion crack (PWSCC) because it was initiated from the ID surface of the tube. However, when the same area was forced by a Vickers hardness indenter (Mitutoyo, model HM-122, Japan) at a load of 1 kg, abundant crazing along the grain boundaries occurred, as shown in Fig. 3B. The white arrows indicate the same location before and after indentation. Consequently, this result clearly indicates that this defect is IGA, not PWSCC.

Fig. 4 shows the fracture surface of the laboratory-grown IGA tube and PWSCC in a tube pulled from an operating plant. The fracture surface of the IGA tube specimen showed the same appearance as that of typical intergranular SCCs. Therefore, the intergranular nature of the fracture surface cannot be proof of SCCs. Among some mechanisms of PWSCC, the internal oxidation mechanism is related to oxygen penetration at the grain boundary, resulting in the formation of a brittle intergranular oxide [9]. The model predicts a strong dependence on the potential. When the potential is too low, oxidation is not possible; when it is too high, a compact oxide grows and prevents further oxygen diffusion and oxidation [9]. However, intergranular crazing observed in this work depends on the degree of intergranular corrosion before deformation.

The degree of IGA depends on not only the depth and width of the chromium depletion along the grain boundaries [10–12] but also the corrosive environmental factors [13,14]. Therefore,