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## Characterization of microstructure, local deformation and microchemistry in Alloy 690 heat-affected zone and stress corrosion cracking in high temperature water



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#### 1. Introduction

#### ABSTRACT

With increasing the distance from the weld fusion line in an Alloy 690 heat-affected zone, microhardness decreases, kernel average misorientation decreases and the fraction of  $\Sigma$ 3 boundaries increases. Chromium depletion at grain boundaries in the Alloy 690 heat-affected zone is less significant than that in an Alloy 600 heat-affected zone. Alloy 690 heat-affected zone exhibits much higher IGSCC resistance than Alloy 600 heat-affected zone in simulated pressurized water reactor primary water. Heavily cold worked Alloy 690 exhibits localized intergranular stress corrosion cracking. The effects of metallurgical and mechanical properties on stress corrosion cracking in Alloy 690 are discussed.

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Ni-based alloys and weld metals are used extensively for fabricating structural components in pressurized water reactors (PWRs). Stress corrosion cracking (SCC) in Ni-base Alloy 600 and its weld metals is one of the main issues of material degradation in PWR coolants [1–7]. Lots of crack growth rate (CGR) data for Alloy 600 base metals and weld metals in the as-received conditions or in the cold-worked conditions have been measured in simulated PWR primary water environments [1–6]. Many properties in a weld heataffected zone (HAZ) are different from those in the base metal and can contribute to the change of SCC resistance. The thermal cycles from welding process can result in residual stress and strain and the changes of microstructure, carbide distribution and local material chemistry and therefore affect the primary water stress corrosion cracking (PWSCC) behavior [8–10]. The inhomogeneity in metallurgical and mechanical properties in weld HAZs is schematically

http://dx.doi.org/10.1016/j.jnucmat.2015.06.025 0022-3115/© 2015 Elsevier B.V. All rights reserved. shown in Fig. 1, which is affected by welding methods, welding procedures and parameters. Young et al. [8] have investigated the SCC growth behavior of an Alloy 600 weld HAZ in high temperature pure water and the effects of microstructure, test temperature and electrochemical potential as function of dissolved hydrogen concentration. They reported that the CGRs in the Alloy 600 HAZ were about 30 times faster than those in the Alloy 600 base metal tested under the same conditions [8]. Lu et al. [9,10] have measured the grain boundary character distribution, grain boundary carbide distribution, grain boundary chemistry and local deformation in an Alloy 600 HAZ and correlated to its SCC behavior in simulated PWR primary water. The CGR in the Alloy 600 HAZ can be more than 20 times higher than that in its base metal. The effect of strainhardening in weld HAZs can be simulated partly by using cold worked Alloys. The grain boundary character distribution and local deformation in Alloy 600 cold worked to various degrees were measured. With increasing the degree of cold work, the hardness and kernel average misorientation increase while the fraction of  $\sum 3$ boundaries decreases. The PWSCC behavior in the Alloy 600 HAZ and cold worked Alloy 600 were investigated and compared [9,10].

Alloy 690 and its weld metals such as Alloys 52, 52M, 152 have been used in new PWR plants or used to replace previously used

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Fig. 1. Schematic of a weld exhibiting metallurgical and mechanical characteristics in HAZs.

Alloy 600 and its weld metals such as Alloys 82 and 182. It is important to evaluate the SCC resistance of Alloy 690 and its weld HAZ in PWR primary water environments and related influencing factors on SCC. The complexity related to bulk chemistry, grain boundary chemistry, grain boundary types and ratios, mechanical properties and other factors in Ni-base weld HAZs such as Alloy 600 HAZ and Alloy 690 HAZ has been pointed out [9–13]. Straining hardening has been identified to be one of the factors contributing to SCC in stainless steel and Ni-base alloy weld HAZs [9–16]. Andresen [12] reported the SCC growth rates under various loading modes for an Alloy 690/EN52M narrow gap weld HAZ in a simulated PWR primary water at 360 °C. The CGR in the HAZ was  $7 \times 10^{-12}$  m/s under the constant loading. The SCC growth was significantly enhanced by incorporating an unloading-reloading step in the trapezoidal wave loading mode, and the enhancement factor decreased with increasing the holding time during the trapezoidal wave loading. Alexandrean et al. [13] measured the CGRs under cyclic and constant loading modes for Alloy 690 HAZ in a PWR environment at 320 °C, and reported the measured CGRs to be in the range of 3.36  $\times$  10<sup>-12</sup> to 7.74  $\times$  10<sup>-12</sup> m/s under constant loading at K of about 30 MPa m<sup>0.5</sup>. It was found that different HAZs could exhibit different CGRs, considering the above reported CGR data and the effect of temperature on SCC growth. Different microstructures, bulk chemistries and microchemistries and local mechanical properties in weld HAZs as the results of different welding procedures and parameters could contribute to the observed differences in SCC growth behavior of HAZs. The effects of cold work and the details of cold work such as rolling methods and orientations on SCC of Alloy 690 were investigated. Cold work could significantly accelerate the PWSCC growth rate of Alloy 690 [12-16]. Scatted CGR data for asreceived Alloy 690 and cold worked Alloy 690 from different heats were observed. In the present work, the hardness distribution, microstructure and microchemistry at grain boundaries of an Alloy 690 weld HAZ are characterized. PWSCC growth behaviors of Alloy 690 HAZ and cold worked Alloy 690 in PWR primary water environments are investigated and compared to the results of Alloy 600 HAZ and cold worked Alloy 600. The effects of strain-hardening, microstructure and the bulk/grain boundary chemistry on SCC of Ni-base alloy HAZs are discussed.

#### 2. Experimental details

#### 2.1. Test materials

Alloy 690 base metal and weld heat-affected zone in an Alloy

$C_{1}$ $C_{2}$ $C_{2$	
Chemical compositions of Alloy 690 (wt. %).	

С	Si	Mn	S	Р	Cu	Ni	Cr	Fe
0.02	0.11	0.16	0.001	0.006	0.01	59.42	29.35	10.13

690/Alloy 52 weld were used in the experiments. The weld was prepared by Shielded Metal Arc Welding (SMAW) using thermally treated Alloy 690 base metal (called Alloy 690TT) and Alloy 52 weld metal. The Alloy 690 was obtained by mill annealing (MA) at 1100 °C for 1 h followed by air cooling. Thermal treatment (TT) was performed at 700 °C for 15 h. Alloy 690TT was used for preparing the weld block. The chemical compositions and mechanical properties of Alloy 690TT base metal are shown in Tables 1 and 2, respectively. The schematic of the Alloy 690TT weld block is shown in Fig. 2. Fusion line A1 was produced by the first welding. Fusion lines A and B were produced by the second welding after removal a part of the weld metal of the first welding. If not specially mentioned, the weld fusion line refers to fusion line A throughout the text. A 40% cross-rolled Alloy 690TT block, called as 40% CR Alloy 690, was prepared and used in the SCC tests for understanding the effect of strain-hardening on SCC. The CR procedure was similar to that has been used for Alloy 600 [9,10]. The parameters for the CR are listed in Table 3.

#### 2.2. Characterization of microstructure and local deformation

The values of Vickers hardness (HV) in the Alloy 690TT HAZ and in the 40% Cr Alloy 690TT were measured at a load of 9.8 N and a holding time of 15 s. The HV in the Alloy 690TT HAZ was measured as a function of the distance from the weld fusion line A. Microstructure and grain boundary properties were characterized by electron back scattering diffraction (EBSD). Local deformation was evaluated by HV and kernel average misorientation (KAM) measurement by EBSD technique with Hitachi S-4300 FE-SEM, TSL solutions camera control system VIT1000, image processing system DSP 2000, and interface controller MSC 2000. The EBSD pattern

Table 2					
Mechanical	properties	of Allov	690 base	metal at	RT.

- - - -

Yield strength, MPa	Tensile strength, MPa	Elongation ratio, %
256	619	59.9

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