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Analysis of local dislocation densities in cold-rolled alloy 690 using transmission electron microscopy

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1. Introduction

Ni-based alloy 600 has been widely used in structural materials for nuclear power plants (NPP) owing to its high corrosion resistance. However, alloy 600 has been reported to degrade under the service conditions prevalent in nuclear power plants was reported. [1] Recently, alloy 600 was replaced by alloy 690, which has higher corrosion resistance. Service failure of alloy 690 in NPP has not been reported. However, some research groups reported that primary water stress corrosion cracking (PWSCC) occurred in severely cold-rolled alloy 690. [2] Transgranular craking was also reported in coll-rolled alloy 690 with a banded structure.[1]

In order to understand the effect of cold rolling on the cracking of alloy 690, many research groups have focused on the local strain and the cracked carbide induced by cold-rolling, by using electron backscatter diffraction (EBSD) [1] However, there are constraints in measuring the local strain using EBSD. Transmission electron microscopy (TEM) has been widely used to characterize structural materials because this technique has superior spatial resolution and allows for the analysis of crystallographic and chemical information. [3]

The aim of the present study is to understand the mechanism of the abnormally high crack growth rate (CGR) in cold-rolled alloy 690 with a banded structure. The local dislocation density was measured by TEM to confirm the effects of local strain on the stress corrosion cracking (SCC) of alloy 690 with a banded structure. The effects of intragranular carbides on the SCC were also evaluated in this study.

2. Experimental procedures

A thin foil specimen for TEM analysis was prepared by mechanical polishing, followed by jet polishing with 10% perchloric acid at −15 °C and a voltage of 25 V. TEM/STEM (JEOL JEM-2100F) was conducted at a voltage of 200 kV. In order to quantify the local strain in cold-rolled alloy 690, the dislocation density (ρ) was directly measured by the line intercept method, as shown in the following equation [4], [5]:

$$\rho = 2N/Lt \qquad (1)$$

where L is the total length of the randomly drawn lines, N is the number of intersections between the dislocations and lines, and t is the thickness of the thin foil specimen. The t value was measured by the electron energy loss spectroscopy (EELS) log ratio method as following equation. [6]

$$t/\lambda = \ln(I_t/I_0) \qquad (2)$$

Here, λ is mean free path for elastic scattering for the material, I_t is total number of electrons in the EELS spectrum, and I_0 is number of electrons having lost no energy.

3. Results and discussion

Intergranular carbide could increase the SCC resistance in alloy 600. However, intragranular carbide is known to reduce the SCC resistance of Ni-based alloys in a pressurized water reactor (PWR). While a number of studies have been carried out on intergranular carbides, there are very few reports on the effect of the intragranular carbides on the susceptibility of Ni-based alloys to SCC.

Energy dispersive spectroscopy (EDS) and selected-area diffraction patterns (SADP) were used to confirm the carbide structure in alloy 690 having a banded structure used in this study. Fig. 1 shows the bright-field STEM image and the elemental map of a 10% cold-rolled specimen. The size of the carbide was ~1μm and it had an ellipsoidal shape. Figs. 1(b) and (c) show the Cr and Ni maps, respectively. As shown in fig. 1(c), Ni was depleted of carbide because it contained significant amounts of Cr. The carbide structure was confirmed as $M_{23}C_6$ by SADP (not shown).

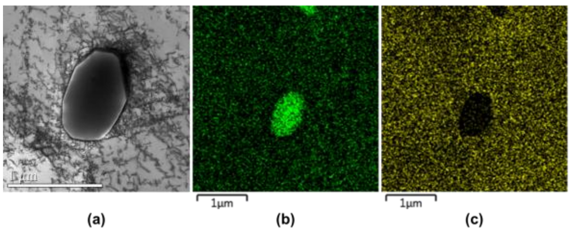

Fig. 1. Intragranular carbide image in banded region of alloy 690. (a) Bright field STEM image, EDS elemental mapping of (b) Cr , (c) Ni

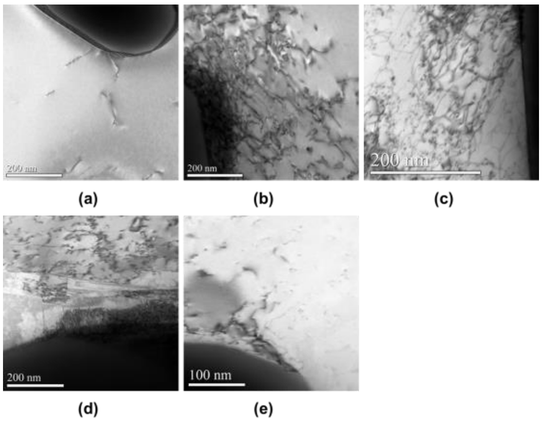

Fig. 2. Bright field STEM image at near the carbide in (a) as received and thickness reduction of (b) 10%, (c) 20%, (d) 30%, and (e) 40% specimen.

Figs. 2(a)–(e) shows a bright-field STEM image near the intragranular carbide. As shown in fig. 2(a), not many dislocations exist near the intragranular carbide in the as-received specimen. This indicates that the intragranular carbide could not induce dislocation densities by itself. However, as shown in Figs. 2(b)–(e), the number of dislocation densities sharply increased in cold-rolled alloy 690. The average dislocation densities near the carbide in the as-received specimen were $0.06*10^{11}/cm^2$. The average dislocation densities had increased to $1.87*10^{11}/cm^2$ in the 40% cold-rolled specimen. The average dislocation densities were 30 times higher than those for the as-received specimen.

Figs. 3(a)–(e) shows the bright-field STEM image in the matrix region away from the intragranular carbide. As shown in fig. 3(a), there were very few dislocations in the as-received specimen. However, the dislocation density drastically increased in cold-rolled alloy 690. The average dislocation densities of the 10%, 20%, and 40% cold rolled specimens were $0.39*10^{11}/cm^2$, $0.53*10^{11}/cm^2$ and $1.03*10^{11}/cm^2$, respectively. Dislocation densities in matrix of 40% cold-rolled specimen were similar to that of in intragranular carbide.

Fig. 4. shows the average dislocation densities versus thickens reduction ratio with error bar. The dislocation densities increased with increasing thickness reduction ratio. This trend occurred simultaneously in three different regions. However, the rate of increase of the dislocation densities was different. Dislocation densities near the intragranular carbide were consistently the highest among the three different regions for each thickness reduction ratio.

It has been reported that the intragranular carbide could act as a dislocation source in highly strained specimens. [7] The dislocation density in the interior of the grains could be directly affected by the intragranular carbide. Therefore, grains with intragranular carbide could easily be weakened compared to those without the carbide. Transgranular cracks can easily propagate through the weakened grains in cold-rolled alloy 690.

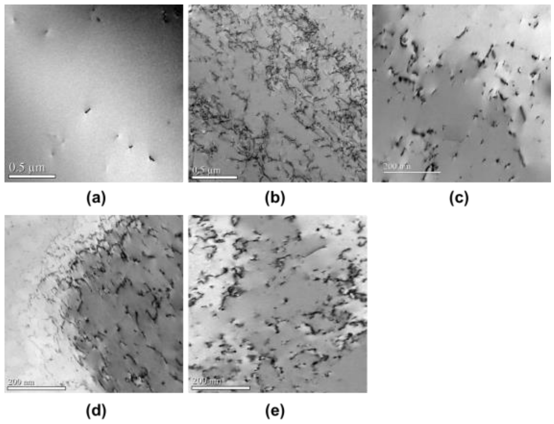

Fig. 3. Bright field STEM image in the matrix region of (a) as received and thickness reduction of (b) 10%, (c) 20%, (d) 30%, and (e) 40% specimen.

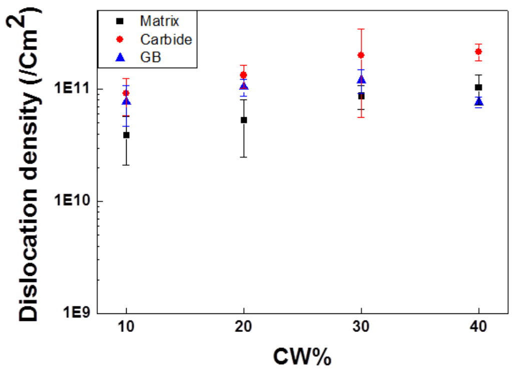

Fig. 4. The average dislocation densities versus thickness reduction ratio with error bar.

4. Conclusions

The local dislocation densities were directly measured using TEM to understand the effect of local strain on the SCC of Ni-based alloy 690 with a banded structure. The dislocation densities in the interior of the grains sharply increased in highly cold-rolled specimens due to intragranular carbide, which acted as a dislocation source. Therefore, transgranular cracks could easily propagate through the grain interior in the highly cold-rolled alloy 690.

5. Acknowledgement

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