# Effect of pre-strain on oxidation behavior of Ni-Cr alloys in air at 700 °C

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# 1. Introduction

Nickel-chromium alloys have been widely used as heat exchangers due to their superior oxidation resistance and good mechanical strength at high temperature. Many studies have shown that they have superior oxidation resistance at high temperature, for example, at 600, 700, and 800 °C [1-3]. For these materials to be applied as heat exchangers, they should undergo some process during fabrication, which in turn induces some strain. In addition, for nickel-chromium alloys to be utilized in a harsh environment, it is highly likely that their oxidation becomes one of the most important life limiting factors. Hence, it is necessary to study how pre-strain affects the oxidation behavior of nickel-chromium alloys, and detailed microstructural analysis needs to be performed to figure out the mechanism behind how pre-strain affects the oxidation behavior of nickel-chromium alloys.

#### 2. Methods and Results

As received alloys 263 and 740H were used in this study, and their compositions are shown in Table 1. The final heat treatment was conducted for alloys 263 and 740H at 800 °C for 8 and 5 h, respectively. Tensile tests were performed up to uniform strains of 10, 20, and 30%. Oxidation test specimens were then fabricated from the center of the tensile test specimens through electrical discharge machining (EDM). The width, length, and thickness of the oxidation test specimen with 0% pre-strain were 10, 11, and 1 mm, respectively. As the pre-strain increased, the length and thickness of the specimens decreased owing to the pre-strain. Before oxidation tests were performed, the specimens were ground using silicon carbide papers of ANSI 320, 600, and 800 grit; they were then polished using diamond suspensions (6, 3, 1, and 0.25 µm) and electropolished using ethanol (90%) and perchloric acid (10%) at a cell voltage of 20 V. Fig. 1 shows the XRD results of alloy 740H before and after the electropolishing. After the electropolishing, the XRD peaks became narrower,

indicating that surface defects, which were created mainly by grinding, were eliminated. In addition, the XRD peaks were shifted to higher  $2\theta$  values after the electropolishing, indicating that the surface area of specimens which experienced some extent of tension from the grinding was removed. These results indicate that electropolishing should be performed before performing oxidation tests since it is possible that the defects that formed during grinding and polishing affect oxidation behavior.

As mentioned above, oxidation tests were performed using the specimens that were subjected to pre-strain (0, 10, 20, and 30%) at 700 °C in a furnace which has air condition with atmospheric pressure. During the oxidation tests, the mass of the specimens was measured with a precision of 0.01 mg, and XRD analysis was performed on the top surface of the specimens after 1, 9, 27, 81, and 100 days. After 100 days, the specimens were cut as the angle of 45 degrees with the tensile test direction using a linear precision saw to observe the cross sections of the specimens, and the microstructures of the specimens were analyzed using SEM and EBSD. In addition, TEM specimens were extracted from the top surface by the focused ion beam (FIB) method.

Overall microstructures of the specimens were observed using SEM (FEI, Quanta 200 FEG) in backscattered electron mode at an accelerating voltage of 20 kV. Defects in the metal substrate and the phases of the oxide layers were analyzed by XRD (Malvern Panalytical, Empyrean; Cu K $\alpha$  radiation,  $\lambda = 1.54056$ Å) by the Gonio scan method. The measurement was conducted in a 2 $\theta$  range of 25–125° with a step size of 0.013° and a step time of 200 s. Defects in the metal substrate were also examined by EBSD (FEI, Quattro S) at an accelerating voltage of 20 kV. The oxide phases and very small-scale microstructures (<1 µm) such as the gamma-prime phase were analyzed using TEM. TEM specimens were prepared using the FIB (FEI, Helios NanoLab 450). The prepared TEM specimens were analyzed using an FEI Titan G2 instrument equipped with a probe spherical aberration corrector at an accelerating voltage of 200 kV

Table 1. Chemical composition (wt.%) of alloys 263 and 740H used in this study.

	Ni	Cr	Mo	Со	Ti	Al	С	Nb	Fe	Mn	Si	Cu
Alloy 263	Bal.	20.10	5.94	20.30	2.18	0.47	0.05	-	0.19	0.07	0.07	0.08
Alloy 740H	Bal.	24.00	0.60	19.62	1.30	1.18	0.04	1.00	2.19	0.23	0.21	0.01

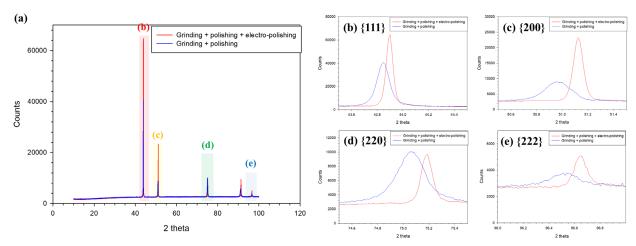


Fig. 1. XRD results of alloy 740H before and after performing electro-polishing [1].

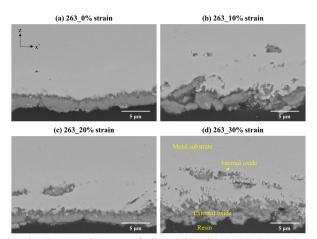


Fig. 2. SEM images of alloy 263 with 0, 10, 20, and 30% pre-strain after corrosion in air at 700 °C for 100 days [1].

Fig. 2 shows SEM images of alloy 263 with 0. 10, 20, and 30% pre-strain after corrosion in air at 700 °C for 100 days. It is apparent that the specimen with higher pre-strain contains more internal oxide. The defects that formed from the tensile tests apparently acted as the channels for oxygen diffusion from the oxide/metal interface to the inside of the metal substrate.

### 3. Conclusions

Effect of pre-strain on corrosion behavior of Ni-Cr alloys was analyzed. Tensile tests using alloys 263 and 740H were performed up to strains of 10, 20, and 30%; oxidation tests were then performed using the specimens with pre-strains of 0, 10, 20, and 30%. The results show that the specimen with higher pre-strain contains more internal oxide. It is obvious that the defects that formed from the tensile tests apparently acted as the channels for oxygen diffusion from the oxide/metal interface to the inside of the metal substrate.

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