The Influences of Ultrasonic Peening Treatment on Oxidation of Designed F/M Steels in Supercritical Water

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Summary

The influence of ultrasonic peening on oxidation of designed Ferritic/Martensitic (F/M) steels in supercritical water has been investigated with time range from 100 hours to 1000 hours. It was observed that in the tested time range, the weight gain of the samples with ultrasonic peening pre-treatment is much less than that of the ones without ultrasonic peening. Different oxide film formed on the surface after SCW exposure. These results indicate that designed F/M steels with ultrasonic peening have better oxidation resistant in 500°C, 25MPa supercritical water than the ones without ultrasonic peening.

1. Introduction

Supercritical water has been regarded as the ideal coolant for the Generation IV nuclear power plant concept. Even though it has been used in advanced fossil fuel power plant for many years, the corrosion is still a critical issue for the applying in nuclear power plant in which the thickness of the component is much thinner than in the fossil fuel power plant [1]. Ferritic-martensitic steel has been chosen as one of the candidate materials for the supercritical water reactor because of the low thermal expansion coefficient, good resistance to radiation-induced swelling, and high thermal conductivity [2].

Ultrasonic peening (UP) utilizes a loading scheme [5] consisting in use of steel pins positioned into impact head installed on the ultrasonic horn tip. Recently, the UP has been successfully used to improve the fatigue life of welded constructions [5, 6]. UP is similar to another surface treatment method, shot peening, which has been proved has positive effect on the oxidation resistance of F/M steels in supercritical water [7].

In the present study, the effect of ultrasonic peening on the oxidation of designed Ferritic/Martensitic steel in supercritical water was investigated.

2. Experiments

The Ferritic/Martensitic steels used in this study were designed by Dr. M. Li and fabricated by a factory in China. These steels have about 11% (wt%) Cr and also have very small amount of Si and Mn. The actual chemical composition tested by EDS was shown in Table 1. All the bulk materials have been cut into samples of dimension 20mm×10mm×2mm. The samples were ground with sand paper down to 1200# grit and finally polished to a 1 μm diamond finish. The samples were cleaned with acetone and then ultrasonically rinsed with acetone for 30 minutes. After completely drying, the samples have been divided into two groups. The first group was used to do the ultrasonic peening treatment. The following are the main parameters of UP treatment, frequency at 25 kHz; diameter is 5mm for 180s. Only the 20mm×10mm sides of each sample in group 1 have been peened. And the second group was used as the control group. After the peening, the peened samples were cleaned again using the same procedure mentioned above. Then all the samples were weighted with a digital balance and the dimensions were measured with a digital micrometer.

Elements	Cr	Si	Mn	Fe
Percentage	11.0	0.6	0.7	balance

Table 1 The actual composition of designed F/M steel.

The exposure experiments were conducted using tube capsules made of 316L stainless steels. A specified amount of demonized water and samples were sealed in the capsule and heated up to 500°C. Samples were kept at 500°C, 25MPa, with 8 ppm dissolved oxygen at the beginning of the test, for 100 hours, 250 hours, 500 hours and 1000 hours. After supercritical water exposure, samples were coated with Ni to protect the oxide film, and then mounted with epoxy resin and polished to investigate the cross section oxide scale by SEM.

The SEM and EDS facility used in this study is a Zeiss EVO MA 15 scanning electron microscope equipped with a Bruker Silicon Drift Detector for Energy Dispersive X-Ray analysis/ mapping with a peak resolution of 125 eV.

3. Results

3.1 Weight Change of Exposed Samples

The weight change (Δw) of the designed F/M steels as a function of exposure time (t) is shown in Figure 1 with the average of 3 samples each data point. The solid rectangles and open triangles denote the data of samples with and without peening treatment respectively.

The weight change data can be fitted in the following equations:

$$\Delta w = kt^n \qquad (1)$$

And

$$k = k_0 e^{\left(-\frac{Q}{RT}\right)} \qquad (2)$$

where the **Δw** is the weight change of the sample per unit area, **Q** is the activation energy for oxidation, **k** and $\mathbf{k_0}$ are the oxidation rate constant, **R** is ideal gas constant, **T** is the temperature in Kelvin, **t** is the exposure time in hour and n is time exponent which indicates the time dependence of the oxide growth. The fitted lines and corresponding fitting parameters are shown in Figure 1.

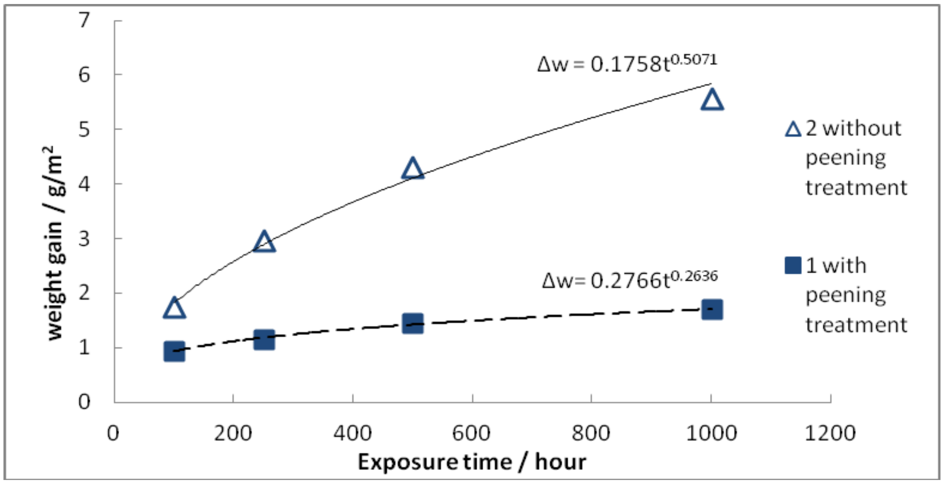
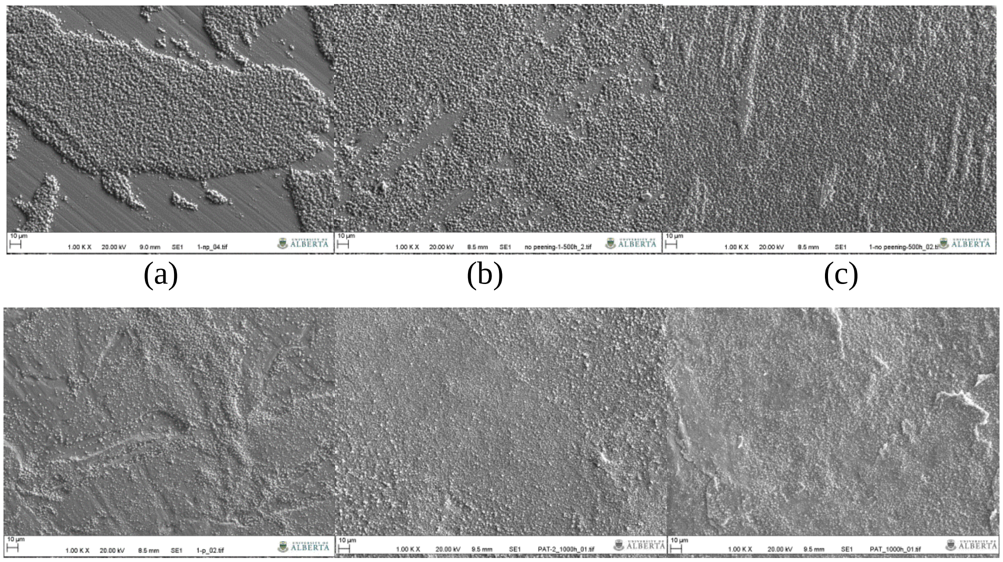


Figure 1 The weight change as a function of exposure time in supercritical water.

In the tested time range, it is observed that both the samples with peening treatment and the samples without peening treatment shows slight weight gain. And the samples in first group (with peening treatment) showed distinct less weight gain than the samples in second group (without peening treatment). The time exponents gained from experiments were 0.5071 for samples without peening treatment which is similar to the report in reference [2, 8] and 0.2636 for samples with peening treatment.

3.2 Surface Oxide Morphology

Surface morphologies (secondary electrons analysis images) of designed F/M steels with and without peening treatment after exposure for 250 hours, 500 hours, and 1000 hours are shown in Figure 3 respectively. Distinct difference has been observed between the samples with peening treatment and samples without it. By making a comparison between fig. 2 (a) and fig. 2 (d), it is obvious that for the first 250 hours exposure, regional oxide structure formed on the surface without peening treatment, but at the same time, much less oxide crystal could be found distributed on the peened surface. With exposure time increasing to 500 hours and 1000 hours, more oxide could be found on both of the two kinds of surfaces and the distribution became more uniform. The particle size of the oxide formed on the peened surface is relatively smaller than that of the oxide formed on the surface without peening. After 1000 hours exposure, the oxide structure formed on peened surface is dense and continuous while the oxide structure formed on the surface without peening is porous and with some small region uncovered. The difference between these two oxide films might lead to the difference of weight gain and oxidation resistance.


(a) (b) (c)

(d) (e) (f)

Figure 2 The surface morphology samples without peening treatment after SCW exposure for (a) 250 hours, (b) 500 hours, (c) 1000 hours, and peened samples after SCW exposure for (d) 250 hours, (e) 500 hours, (f) 1000 hours.

3.3 Cross-section Analysis

Figure 3 shows the cross-section micrographs (backscattered electrons analysis images) of the oxide film formed on the surface with and without peening after 500 hours supercritical water exposure. The cross-section micrographs and general chemical compositions at different locations of the oxides scales are shown in the corresponding X-Ray elemental mapping. A dual layer oxide film was found on the surface without peening. The thickness of this film is about 5 μm. From the corresponding elemental mapping in Figure 3 (a), the outer layer is enriched with Fe and O while the inner layer is enriched with Cr and O. It coincides with other results in reference [2, 3]. However, after 500 hours exposure, it is hard to find such dual layer oxide film on the surface with peening treatment. Instead a 1~2 μm oxide film is found. The much thinner layer explained why the weight gain of peened sample is much smaller. This thinner layer is formed with ion, chromium and oxygen. This thinner but more condensed layer prevents the oxygen from diffusing from the supercritical water into the metal substrate and makes samples more resistant to oxidation.

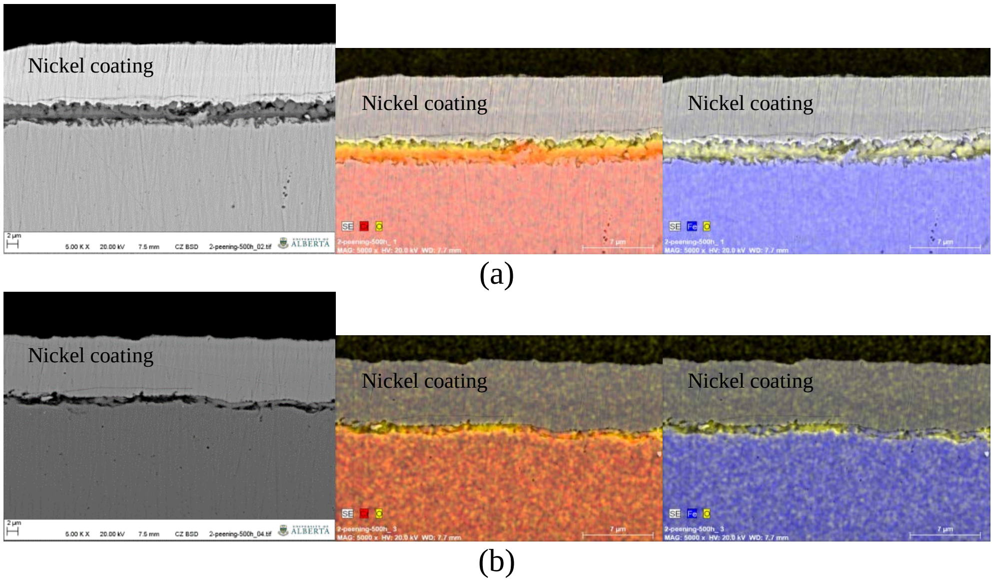


Figure 3 The cross-section micrographs and elements mappings of (a) sample without peening and (b) peened sample after 500 hours SCW exposure. (red: Cr, yellow: O, blue: Fe)

4. Discussion

According to some relative study about the grain refinement on corrosion behavior of F/M steels in supercritical water [7], this improvement of oxidation resistance found on the samples with ultrasonic peening treatment might result from the grain refinement achieved by introducing severe plastic deformation by ultrasonic peening and the recrystallization during supercritical water exposure. Grain refinement can lead to enhanced diffusion of elements from the bulk to the surface since the diffusion coefficient along GBs can be several orders of magnitude higher than that in the bulk lattice [9, 10]. This can also promote a quicker formation and self-healing of protective oxide scales [11].

5. Conclusion

The corrosion behaviour in 500°C, 25MPa supercritical water of designed F/M steels with and without ultrasonic peening treatment was investigated. Less weight gain and thinner oxide film were observed on samples with ultrasonic peening, which indicates that ultrasonic peening treatment has a positive impact on the corrosion resistance of designed F/M steels.

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