Nano-structuring induced by shot peening and its effect on corrosion resistance of stainless steel

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Abstract - *The nanostructure in the surface of various stainless steels can be induced by high-energy shot peening. It has been characterized by means of X-ray diffraction and transmission electron microscopy. The effects of shot peening on corrosion resistance of the steel have been investigated by polarization curves and pit corrosion tests, and the surface morphologies of the corrosion samples have been characterized by scanning electron microscopy. The results have shown that a nanostructured microstructure with an average grain size in nanometer scale gets formed in the surface layer of the sample, and furthermore, it can also produce martensite of about 10-15% in volume fraction [1]. With increasing depth from the shot-peened surface, the grain size increases and fraction of martensite decreases. Comparing to the as-received bulk crystalline sample, the passive film on the surface of the shot- peened sample is easier to form and more stable. Shot-peening-induced surface nanostructuring can also have effect on corrosion resistance of stainless steels.*

Key Words: Iron alloy; Shot peening; Surface nanostructuring; Transmission electron microscopy; X-ray diffraction; Corrosion resistance

1. INTRODUCTION

Recently, attention is put on by researchers on to nanostructured materials since they can have enhanced mechanical, physical and chemical properties. In many cases, failures of a workpiece may initiate at the surface, such as fatigue, wear and corrosion. Surface microstructure and properties of structural materials can thus play an important role in failure control. So, surface nanostructuring will be able to improvee, in combination, mechanical properties and corrosion resistance to aggressive environment [2]. Fig.1 shows the basic set-up for ultrasonic shotpeening showing surface nanostructuring. It not only avoids the fabrication difficulties of large bulk nanostructured material but also combines the superior properties of nanomaterials to conventional engineering materials. This technology can be easily put in practice. So far, however, the corrosion behavior of nano-grained (NG) and ultrafine- grained (UFG) materials has received only limited attention. Vinogradov et

al. [3] have shown that there is no significant difference in polarization behaviour between UFG Cu produced by equi-channel angular extrusion and coarse-grained (CG) Cu.
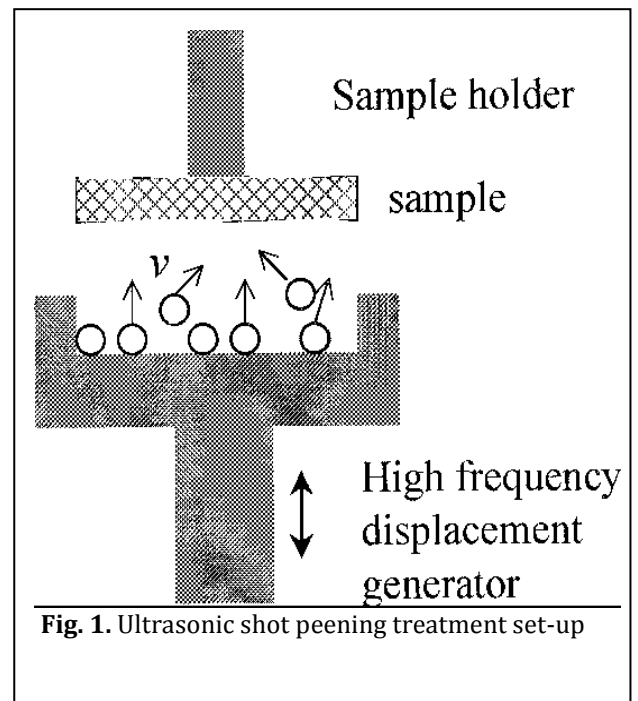

Fig. 1. Ultrasonic shot peening treatment set-up

The UFG Cu has a higher average dissolution rate than its CG counterpart. But the UFG Cu has a much lower localization of corrosion, which is known as a decisive factor in environmentally assisted fracture. Rofagha et al. [4] have shown the similar result for Ni, and they also reported that the corrosion resistance of a NG Ni – P alloy is lower than that for its CG counterpart [5]. Recent studies have shown that the corrosion resistance can be markedly improved not only for NG surfaces of 304 stainless steel [5] and commercial brass (70 – 30) [6] that were produced by sandblasting and annealing, but also for UFG Ti [8] produced by the equal channel angular pressing (ECAP) and surface nanocrystallized Ni – 22Cr – 13Mo – 4W alloy [8] by shot peening process followed by low-temperature annealing. The higher corrosion resistance of these UFG or NG alloys could be attributed to two factors. First, the segregation of

impurities to the grain boundaries in CG alloys could cause intergranular corrosion. Preferential corrosion at grain boundaries could be severe enough to drop grains out of surface, which significantly accelerate the corrosion rate. Second, the nanostructured surface could increase the density of diffusion paths available for alloying elements to migrate and rapidly form a protective passive film. These results reported suggest that the effect of nano-crystallization on corrosion resistance varies among alloy.

The aim of this paper is to study how microstructure changes in stainless steels by shot peening and its effect on corrosion resistance.

2. GENERAL PROCEDURE

A steel plate machined from a cylinder of stainless steel was used for the study. The chemical composition of the steel is calculated. After solid solution at a temperature for few hours, the surface of the plate was ground and polished with different grit paper, and then the plate was cut into two pieces with lengths of long and short. The longer one was used for shot peening to induce surface nanostructuring. The shorter one was used for comparison test. Shot peening was carried out by a flow of stainless steel ball with a diameter of ¨ 0.8 mm under 0.5 MPa for certain time. The shot-peened plate was cut into several pieces with size of 10 x 10 mm2, and then polished them to just remove the pits using 400 – 1000# waterproof grit paper and diamond grinding paste. These polished samples were used for X-ray diffraction (XRD) and transmission electron microscopy (TEM) analyses, and electrochemical behavior and corrosion resistance tests. Before electro- chemical test, samples were mounted using epoxy only allowing the shot-peened surface exposed to a corrosive medium.
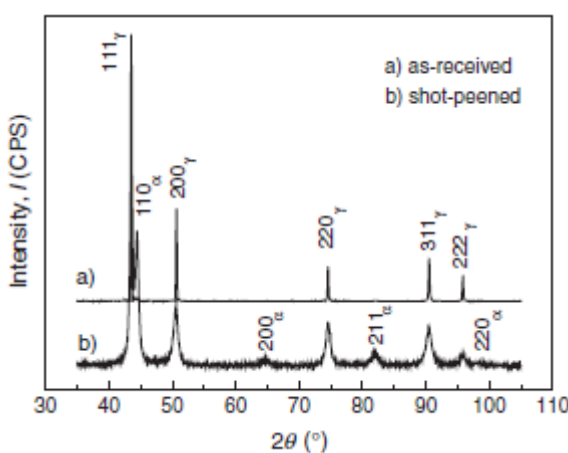

Fig.2. XRD patterns for shot-peened and as-received samples

XRD h – 2h scan of shot-peened surface was performed using a Rigaku D/max-2500/PC X-ray diffractometer with CuKa radiation and at a 2h scanning rate of 4-/min to determine the phase constituent and the average crystallite (or coherently domain) size in the surface layer. TEM

observation of the surface microstructure was carried out using a transmission electron microscope model of H-800 operating at 200 kV. The foil for TEM observation was prepared by one-sidedly thinning from the backside of the shot-peened surface into 30 Am in thickness using different grit papers, and then one-sidedly ion-milling it into perforation by using a 691 Precision Ion Polishing System.

Potentiodynamic polarization measurements for the surface of the sample with and without shot peening were done using an electrochemical workstation model of at room temperature, and a 3.5% NaCl solution was used as corrosive medium. A saturated calomel electrode was used as a reference electrode, and the platinum plate was used as an auxiliary electrode. A waterbath was used to keep a 6% $FeCl_3$ solution at 50 C. After the samples were immersed into the solution for 70 h, they were removed from the solution and immediately cleaned by using an ultrasonic cleaner, and then aired to dry. The eroded surface morphologies of samples were observed by a scanning electron microscope (SEM) in order to characterize the corrosion resistance.

3. RESULTS

Fig. 2 depicts the XRD patterns of the surface layer of as-received sample (Fig. 2a) and shot peened sample (Fig. 2b). It can be seen from Fig. 2b that besides the diffraction peaks of austenite (g) the peaks of a-martensite exist in the shot-peened sample. Comparing Fig. 2a to b, we can be clear that the full width at half maximum (FWHM) of diffraction peaks for the shot- peened sample is much bigger than that for as-received sample, and the average grain size in the surface layer of shot-peened sample can be determined to be ¨ 18 nm by using a XRD Pattern Processing and Quantification Program referred as Jade 5 developed by MDI Materials Data, Inc. These indicate that shot peening can produce surface nanostructuring of 1Cr18Ni9Ti stainless steel and form deformation-induced a-martensite in the surface layer. From Eq. (3) in literature [10], considering there are only two phases in a sample, the equation of volume fraction calculation of martensite (V_a) can be deduced to be

$$V_\alpha = \frac{\frac{1}{n}\sum_{j=1}^{n}\frac{p_\alpha^j}{R_\alpha^j}}{\frac{1}{n}\sum_{j=1}^{n}\frac{p_\gamma^j}{R_\gamma^j} + \frac{1}{n}\sum_{j=1}^{n}\frac{p_\alpha^j}{R_\alpha^j}} \qquad (1)$$

where n, I and R represent the number of peaks of the phase used in calculation, the integrated intensity for reflecting plane and the material scattering factor, respectively. R has been given in literature [10] by the equation

$$R = \frac{1}{v^2}\left[|F|^2 p\left(\frac{1+\cos^2 2\theta}{\sin\theta\sin 2\theta}\right)\right]e^{-2M} \qquad (2)$$

The TEM dark-field image and corresponding selected area diffraction (SAD) pattern of the microstructure in

surface of shot-peened sample contains ring shaped pattern. Equiaxed grains were observed of size ranges from 5 to 30 nm, and the average grain size is about 17 nm. It can also be seen that the SAD patterns present continuous, homogeneous and broadened concentric rings. This indicates further that the grain size is very tiny and the grains are random in crystallographic orientation. For the shot-peened sample, the grain size determined from TEM observation is a little less than that from the XRD calculation. This might be due to the fact that XRD results averaged the structure information of a surface layer of about 10 Am thick, while the TEM sample was a very thin film (less than 1 Am thick) at the sample surface. The TEM dark-field images were formed from diffraction beams in a section of arc in the first diffraction ring that can be indexed. From that we can measure the average grain size at the depth of about 10 A0 from the shot-peened surface to be 70 nm, which is much bigger than that at the shot-peened surface. Literatures [11] and [12] reported that the grain size evidently increases with an increase of the depth from the shot peened surface. Additionally, many have indicated nanostructured material was produced in the near surface layers by deformation under large sliding loads, and the grain size was coarsened with increasing depth from the surface. These results are similar to the study done on steels previously. Comparing before and after shot peening images, it is observed that the intensity of diffraction ring is much intense in case of shot peened samples. This suggests that the volume fraction of deformation-induced martensite decreases with the increase of the depth from shot-peened surface.

In order to measure the effect of shot peening on pit corrosion resistance of stainless steel, the un-shotpeened and shot-peened samples were immerged in 6% $FeCl_3$ solution at 50 C for 72 h. And then the eroded surface morphologies of both two samples were observed by means of SEM. The typical micrographs are observed. It can be seen that the number, size and depth of corrosion pitting marks on shot-peened surface are more than that on the surface of unshotpeened sample. Therefore, conclusion can be made that shot-peening-induces surface nanostructuring which can enhance the local corrosion resistance of stainless steel in the chlorine – ion-contained solution.

4. CONCLUSIONS

Nanostructuring of steel can be achieved by using high-energy shot peening. The nanograined microstructure with size of about few nanometers and about 17 vol.% deformation-induced a-martensite can be produced in the shot-peened surface. With increasing the depth from the shot-peened surface, the grain size increased, but the volume fraction of the deformation-induced a- martensite decreased. Shot-peening-induced surface nanostructuring can improve its potentiodynamic polarization behavior of stainless steel in 3.5% NaCl solution, markedly decrease the anodic current density and passivation-maintaining current density, induce a consider- ably enlarged passive region of the stainless steel

and raise the breakdown potential of passive film, and for as-received markedly enhance the overall and local corrosion resistance of the steel in the chlorine – ion-contained

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