Preventing hydrogen embrittlement in stainless steel by means of compressive stress induced by cavitation peening

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Abstract: In this paper, it has been demonstrated that compressive residual stress induced by cavitation peening which is one of the mechanical surface modification techniques can reduce invasion of the surface of austenitic stainless steel by hydrogen. Cavitation peening was done with employing a cavitating jet in air. The specimens were prepared with different processing time of cavitation peening. Then, stress measurement was performed using an X-ray diffraction analysis. After that, the surface was charged with hydrogen employing a cathodic charging method. Hydrogen content was evaluated by a thermal desorption analysis using a gas chromatography. From the obtained results, hydrogen content was reduced along with increase in compressive residual stress at surface. In particular hydrogen content became to 15% at processing time of 2 s/mm introducing compressive residual stress of 378 MPa. In short, cavitation peening can drastically prevent invasion by hydrogen.

1 Introduction

Hydrogen has attractive potential to be clean, abundant and movable energy carrier in place of fossil fuels. Mechanical properties of metallic materials are affected by hydrogen. For instance, hydrogen concentrates around a grain boundary, crack tip [1] and inclusion and promotes intergranular cracking [2] and fatigue crack propagation [3]. This is well known as hydrogen embrittlement. Hydrogen concentration and stress gradient can be a driving force for hydrogen invasion and diffusion. So, there is a possibility that hydrogen invasion and diffusion can be controlled. Recently, it was reported that introduction of compressive residual stress may be an effective way to prevent hydrogen embrittlement cracking [4, 5]. In this report, cavitation peening was performed to introduce compressive residual stress.

Cavitation normally causes severe damage to hydraulic machinery such as pumps and propellers since cavitation bubble produces very large impact when it is collapsing, i.e., cavitation erosion. Soyama has succeeded in harnessing the impact for mechanical surface modification as same way as shot peening and laser peening by controlling the collapsing bubbles in cavitating jet [6]. The cavitating jet is totally different from water jet. The water jet utilises the impact produced by water droplet. So, high injection pressure is required over order of hundred MPa in order to modify the surface of metallic materials and treated area is relatively small. In contrast, the cavitating jet can be realised with low injection pressure around order of ten MPa [7] because the impact produced by cavitation bubble collapsing is very high. Cavitation cloud consist of cavitation bubbles in the jet. When those cavitation clouds hit the surface and spread out, the vortex cavitation grows along with the surface and then collapses with generating the impact. When the surface is exposed to the cavitating jet injected into water through a nozzle, the erosion ring is produced around a jet center. The plastic deformation pits are introduced at the exposed surface by the impacts. There are two types of cavitating jet: cavitating jet in water [8], cavitating jet in air [9]. The former can be generated when a high-speed water jet is injected into water, i.e., water filled chamber. The latter is a particular kind of the cavitating jet, which can be generated when the high-speed water jet into a low-speed water jet injected in air. This system makes it possible to treat using the cavitating jet such as cavitation peening without the water filled chamber. It means that the cavitating jet in air can treat large-scale components. The cavitating jet in water can introduce compressive residual stress deep area beyond 1mm from the surface [10] and the cavitating jet in air can introduce high compressive residual stress near surface [11]. In this paper, the cavitating jet in air was employed to prevent hydrogen invasion by hydrostatic pressure.

There are two hypotheses in which compressive residual stress prevent hydrogen embrittlement. One is to mitigate stress concentration around a crack tip due to crack closure effect. The mitigation of the stress field reduces hydrogen concentration around the crack tip [12]. The other is to prevent hydrogen content due to narrowing atomic spacing and it may make hydrogen harder to invade the surface. When peening technique is applied to prevent hydrogen embrittlement, the increase in surface roughness and phase transformation should be considered. Therefore, cavitation peening may be a very effective way to prevent it because there is no collision between solid bodies like shot peening.

In this paper, in order to investigate the effect of compressive residual stress induced by cavitation peening on the invasion of surface of austenitic stainless steel by hydrogen, cavitation peening was done with employing a cavitating jet in air, and then, stress measurement was performed using an X-ray diffraction analysis. After that, the surface was cathodically charged with hydrogen for 48 hours. Also, hydrogen content was evaluated by a thermal desorption analysis using a gas chromatography.

2 Experimental apparatus and procedures

The specimen under test was austenitic stainless steel Japanese Industrial Standards JIS SUS316L. The SUS316L has higher resistance to hydrogen embrittlement than ferritic and/or martensitic steel. The 0.2% proof stress and tensile strength were 304 MPa and 576 MPa, respectively. The size of the specimen was 25 mm square and 4 mm thick. The surface was polished using 1 μm diamond paste.

For cavitation peening, the cavitating jet in air was employed. Figure 1 shows the apparatus to generate the cavitating jet in air and Fig. 2 shows the nozzle geometry. The cavitating jet in air was generated by injecting high-speed water jet into low-speed water jet directly injected in air. The pressure of the high- and low-speed water jet was 20 MPa and 0.05 MPa, respectively. The inner diameter of the nozzle for high- and low-speed water jet was 1 mm and 20 mm, respectively. The outer diameter of the high-speed water jet was 12 mm. The standoff distance between the nozzle and the specimen surface was 30 mm. The nozzle was scanned and the scanning was performed five times with interval of 4 mm to treat whole surface uniformly [13]. The processing time per
unit length, $t_p$ was varied for $t_p = 0$ (Untreated), 0.25, 0.5, 1 and 2 s/mm.

The surface was charged with hydrogen by a cathodically electro-charging after cavitation peening. The solution used for hydrogen charging was 0.5 mol/l sulfuric acid at a temperature of 50 degree Celsius and charging was conducted under current control with a current density of 1.0 mA/mm². The cathodic and anodic electrodes were the specimen and a platinum wire, respectively. The area exposed to hydrogen was 20 mm in diameter and the charging time was 48 hours. After being charged with hydrogen, a thermal desorption analysis was conducted using a gas chromatography as shown in Fig. 3, in order to evaluate hydrogen content for each specimen. The tested sample was placed in a tube furnace which had an inner diameter of 44 mm, length of 500 mm and volumetric capacity of 1 L. The temperature was increased with the rate of temperature rising of 200 degree Celsius/h and kept at 800 degree Celsius for 1 h. In the furnace tube, Ar gas was flowed at 1 mL/s. The gas flowing out of furnace tube was trapped in a 50 mL syringe. Hydrogen has been included in the top 10 mL of gas trapped in the syringe. 5 mL of this gas was analyzed by using a gas chromatograph with thermal conductivity detector TCD. Ar gas of 99.9998% was used as a carrier gas, career pressure was 600 kPa, column temperature and TCD temperature was 130 and 180 degree Celsius, respectively, and TCD bridge current was 60 mA. On this chromatographic system, peak of hydrogen appears from the retention time of 1.5 min to 3 min. The integrated intensity from 1.5 min to 3 min was defined as the relative amount of hydrogen $Q$. Hydrogen content, $C_H$, had been corrected by a relationship between $Q$ and pure hydrogen gas content.

The residual stress, $\sigma_R$, was evaluated by a two-dimensional X-ray diffraction method employing a two dimensional position sensitive proportional counter (2D-PSPC). The stress measurements were conducted using Cr-Kα X-rays from a tube operated at 35 kV and 40 mA through a 0.8 mm diameter collimator and with an incident monochromator. The tested sample was placed in the diffractometer. The diffraction ring from the specimen was detected by 2D-PSPC. The stress tensors were calculated by the diffraction ring distortion. The measurement conditions were chosen by reference to the past report [14]. The detecting time was 120 s. The γ-Fe (2 2 0) plane was chosen as diffraction plane.

3 Experimental results

Figure 4 plots compressive residual stress, $\sigma_{CR}$, obtained from the X-ray stress measurement as a function of the processing time of cavitation peening, $t_p$. The $\sigma_{CR}$ is 8 MPa before cavitation peening ($t_p = 0$ s/mm). As shown in Fig. 4, compressive residual stress rapidly increases along with increase in the processing time. Then, it becomes saturated at $\sigma_{CR} = 378$ MPa at $t_p = 2$ s/mm.
Compressive stress with respect to depth

Decrease in hydrogen content along with increase in area of compressive residual stress, Fig. 6 plots the hydrogen content normalised by the hydrogen content for untreated specimen, \( C_H/C_{100} \), as a function of the processing time per unit length, \( t_p \). From the results, the \( C_H/C_{100} \) decreases as processing time, \( t_p \), increases. The \( C_H/C_{100} \) for \( t_p = 0.25 \) and 2 s/mm are 0.56 and 0.146, respectively. The hydrogen content for \( t_p = 2 \) s/mm becomes seventh part of the untreated specimen.

In order to reveal the suppressive effect of compressive residual stress on the hydrogen invasion, Fig. 6 plots the hydrogen content normalised by the hydrogen content for untreated specimen, \( C_H/C_{100} \), as a function of compressive residual stress, \( \sigma_{CR} \). The variation of \( C_H/C_{100} \) can be defined by a quadratic function given by equation (1):

\[
C_H/C_{100} = 1 - k_1 \sigma_{CR}^2
\]

where \( \sigma_{CR(0)} \) denotes compressive residual stress at surface and \( k_1 \) is constant. The relationship indicates compressive residual stress at surface is not sufficient to represent the suppressive effect on the hydrogen invasion. Therefore the depth profile of compressive residual stress should be considered. Figure 7 plots area of compressive residual stress with respect to depth, \( S_p \), respectively. The \( S_p \) has been defined in past literatures so as to represent suppressive effect on fatigue crack growth [15]. In cavitation peening, compressive residual stress is linearly introduced with respect to the depth due to its randomness [9]. As shown in Fig. 7, there is a strong linear correlation between \( C_H/C_{100} \) and \( S_p \) given by equation (2):

\[
C_H/C_{100} = 1 - k_2 S_p, \quad S_p = \int_0^{S_{pa}} \sigma_{CR}(z) dz
\]

where \( \sigma_{CR(z)} \) denotes the profile of compressive residual stress with respect to depth and \( k_2 \) is constant. The correlation coefficient is 0.997 and the significance level is 0.1%. From the result, the compressive residual stress plays a role to prevent the hydrogen absorption from the surface because of narrowing atomic spacing and increasing potential energy for hydrogen entry from external environment. The suppressive effect of cavitation peening increases as compressive residual stress increases. The compressive residual stress which nearly equals to or is beyond yield stress significantly reduces the invasion by hydrogen. Cavitation peening is one of the most effective ways to prevent hydrogen embrittlement.

4 Conclusions

In this paper, in order to reveal the effect of compressive residual stress on the invasion of surface of austenitic stainless steel by hydrogen, cavitation peening was done with employing a cavitating jet in air, and hydrogen content was evaluated by a thermal desorption analysis after being charged with hydrogen by a cathodically electro-charging method for 48 hours.

There is a strong linear correlation between hydrogen content and compressive residual stress considering the depth profile. The suppressive effect increases along with increase in the compressive residual stress. The hydrogen content for processing time of 2 s/mm becomes seventh part of the untreated specimen, which introduces compressive residual stress of 378 MPa.

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5 References


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