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# Study on surface nanocrystallization and resisting H<sub>2</sub>S stresscorrosion properties of pressure vessel steel welding joints

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Abstract: Many efforts were spent on the homogenization of microstructure and property of welding joints. A new surface nanocrystallization technique named Supersonic Particles Bombarding(SSPB) can be used for this purpose. Two kinds of pressure vessel steel welding joints, 16MnR and 0Cr18Ni9Ti, were chosen to be treated by SSPB. Transmission electron microscopy was introduced to examine the surface microstructure. And their ability to resist  $H_2S$  stress corrosion was enhanced significantly after the SSPB treatment. The mechanism for the results were analyzed as well.

Key words: supersonic particles bombarding; surface nanocrystallinzation; welding joints; resisting  $H_2S$  stress corrosion

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# 1 Introduction

The processing characteristics of welding are adding solders, heating and cooling quickly, which cause the inhomogenization of microstructure and properties of welding joints, and the inhomogenization is one of the main reasons that cause the failure of welding joints<sup>[1]</sup>. Many efforts have been spent on the homogenization of microstructure and property of welding joint. Supersonic Particles Bombarding(SSPB) is a new surface nanocrystallinzation technique<sup>[2]</sup>, and it can be used for this purpose. In this study, pressure vessel steels welding joints, 16MnR low alloy steel and 0Cr18Ni9Ti stainless steel, were chosen to investigate welding joints properties treated by SSPB.

# 2 Experimental methods

#### 2.1 Preparation of samples

The materials used are 16MnR low alloy steel and 0Cr18Ni9Ti stainless steel, Sample size : 16MnR: 290 mm $\times$ 240 mm $\times$ 20 mm;0Cr18Ni9Ti : 250 mm $\times$ 200 mm $\times$ 20 mm. Arc welding was used and E308-16 (A102) welding rods were chosen for welding.

### 2.2 Characterization of nanostructure

Microstructure observation were performed on a JEM-2000FX I TEM, and thin foils for TEM ware

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prepared by means of cutting, grinding and ion thinning on the untreated side.

### 2.3 Measurement of residual stress

Residual stress was analyzed by MSF-2903X X-ray stress diffractometer (with Cr radiation, voltage

30 kV, current 10 mA.). By using repeated electrochemical etching (etching solution : 10% nitric acid solution), the treated surface layer was removed layer by layer, so that residual stress along depth from the treated surface was measured and dctermined by  $\sin^2 \phi$ .



Fig. 1 Sample size of welding joints



Sustained load stress corrosion test equipment was used. According to sustained load tensile test method of metals for re-

sistance to sulfide stress corrosion cracking (SCC)(GB 4157-84), sample size was shown in Fig. 1.

# **3 Results and Discussion**

### 3.1 TEM observation

Fig. 2 and Fig. 3 are TEM micrographs of 16MnR and 0Cr18Ni9Ti welding joints and grain size distribution graph after SSPB treatment. It is concluded that the coarse grains of surface layer have been refined into nanometer regime and nanocrytallines are equiaxed.



Fig. 2 TEM micrograph of welding line (a), HAZ (b), basal metal (c) of 16MnR welding joints after SSPB treatment (1)-dark field; (2)-bright field; (3)-SAD; (4)-grain size distribution graph



Fig. 3 TEM micrograph of welding line (a), HAZ (b), basal metal (c) of 0Cr18Ni9Ti welding joints after SSPB treatment (1)-dark field; (2)-bright field; (3)-SAD; (4)-grain size distribution graph

#### 3.2 Measurement results of residual stress

The material properties are significantly affected by the distribution of surface residual stress. The surface residual stress distribution of welding joints after SSPB treatment are showed in Fig. 4 and Fig. 5. It is observed from Fig. 4 that the maximal compress stress of 16MnR welding line surface reaches its yield



strength and compress stress depth is 220  $\mu$ m. Fig. 5 shows that the maximal compress stress of 0Cr18Ni9Ti welding line surface exceeds its yield strength and compress stress depth is 360 $\mu$ m. As common shot peening is restricted by peening intensity, its maximal residual stress can only reach half of material yield strength, and its compress stress depth varies with the diameter of peening particles<sup>[3]</sup>.

### 3.3 H<sub>2</sub>S stress corrosion resistance results

Fig. 6 and Fig. 7 tell us that resisting  $H_2S$  stress corrosion property of 16MnR and 0Cr18Ni9Ti welding joints treated by SSPB is much better than untreated one. 16MnR fracture time increases 50% when load is lower than yield strength; 0Cr18Ni9Ti fracture time increases one time when load is lower than yield strength. The result is reversed while load is higher than yield strength for two steels.



After SSPB treatment, the microstructure of welding joints surface was refined into equiaxed nanocrystalline. Because the nanocrystalline size is fine and distributes equably, at the beginning of crack generation, the driving force for H induced crack is endured by much more fine nanocrystallines. Moreover, strain extents differ slightly between grain boundary and grain interior, so stress concentration slightly, and crack doesn't generate easily. At the stage of crack propagation, as the volume fraction of grain boundary in nanocrystalline is high, and the orientation of neighboring grains is different, the microcracks will be blocked in the grain boundary when it enter into one grain from another grain through grain boundary. Once the microcracks cross the grain boundary, propagation direction changes, which will consume much more energy, so the microcracks can't propagate and grow easily. Under the coordinate effect of nanocrystalline and compress stress formed by SSPB treatment on the surface of welding joints, its resisting  $H_2S$ stress corrosion property will be enhanced significantly, and this is consistent with the result of G. Echaniz. As for the comparable effects between grain refinement and compress stress, when the applied stress doesn't exceed material yield strength, the material surface doesn't deform, so the nanostructured surface layer will prevent H from penetrating into the material, and protect the material from stress corrosion. When the load exceeds yield strength, material surface deforms, however, material surface and matrix don 't deform conformably, and surface hardness exceeds the matrix hardness, so cracks generate on the surface, stress and H concentrate on the cracks, which is equivalent with the existence of defects. While untreated surface and matrix deform conformably, it results in the totally different experiment results when the load exceeds the yield strength.

Coarse grain, inhomogeneous microstructure in the HAZ of welding joints can increase the SCC sensitivity. It is investigated that different welding joints zones have different resisting SCC properties. For single pass welding, the coarse grain zone of HAZ is most sensitive to SCC. In our experiment, the untreated samples fractured in the HAZ, while treated samples fractured mostly in the welding line, and this indicates that HAZ is sensitive to SCC. There are many defects on the welding joints surface, such as microcracks, which can accelerate the formation of crossion induced cracks and increase SCC sensitivity.

When the welding joints were treated by SSPB, a nanoctructured surface layer with novel property was formed, and this eliminates the inhomogeneity of surface microstructure; At the same time, many surface defects were removed too, so its resistance to SCC was enhanced. Nanostructured surface layer can increase the resistance to dislocation mobility and surface deformation, and block the formation and development of dislocation slip step. This indicates that SNC treatment can enhance the sensitivity of resistance to SCC of welding joints.

## 4 Conclusion

(1) A nanostructured surface layer and compress stress layer with certain depth were formed on 16MnR and 0Cr18Ni9Ti welding joints by SSPB treatment.

(2) Homogeneity of microstruture by forming a nanostrutured surface layer on welding joints were achieved.

(3) Welding joints property of resistance to SCC was enhanced significantly by SSPB treatment.

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