Microwave Plasma Nitriding of Hollow Tube Inner Wall

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Microwave plasma nitriding at the internal wall surface of hollow tubes is described. Hollow tubes (1, 1.5, 2 and 3 mm inner diameter), with lengths of 5 and 12 mm were used for the investigation. Hardness values around 800 Hv were measured at both ends of the nitrided tubes for all diameters. The 3 mm inner diameter hollow tube had almost uniform internal wall hardness (above 700 Hv) along the entire tube length when subjected to microwave plasma nitriding at 773 K for 5 h in 50%N2-50%H2 mixing gas. A diffusion layer up to 0.6 mm was formed and compound layer of ε-Fe23N and γ-Fe4N was precipitated at the internal surface of the tube. The effective nitriding depth of the microwave plasma inside the 2 and 3 mm diameter tubes were 3 times the diameter of the hollow tube.

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1. Introduction

Internal surface modifications of industrial components such as dies, tubes and cylinders are widely used. These components are often required to have better corrosion and wear resistances and several investigations on inner coatings have been reported.1–5) Most of the reports were limited to the deposition of films by physical vapor deposition (PVD) techniques at internal wall surface. However, the line-of-sight character of PVD techniques makes the process difficult, because the depositing material must enter the tube almost perpendicular to the wall.6) Furthermore, PVD of films in hollow tubes with diameters smaller than 10 mm is difficult.7) This is because the transportation of sufficient energy for plasma generation inside the hollow tubes is very difficult. To provide a uniform surface modification along the internal surface of the tubes, it is necessary to use a uniformly distributed plasma source. This problem may be solved by the introduction of a microwave plasma source inside them. The high frequency of microwave plasma has advantages of giving a greater degree of ionization,8) and hence easier generation of the microwave plasma inside a small diameter tube. Nitriding of the internal wall of tubes by microwave plasma has not been reported.

In this article, we have investigated microwave plasma nitriding as a surface modification of the internal walls of steel tubes with inner diameter below 3 mm using nitrogen and hydrogen mixing gas.

2. Experimental Procedure

A schematic diagram of the microwave plasma nitriding apparatus is shown in Fig. 1. The microwave generator, working at the frequency of 2.45 GHz, could be operated at a power up to 1.2 kW. The plasma nitriding temperature was measured using a thermocouple embedded inside a steel plate and the temperature fluctuation was controlled within ±5 K during the nitriding process.

Commercial chromium molybdenum steel plate SCM420 was used as the hollow tube material for microwave plasma nitriding. The chemical composition of the material was 0.17 mass%C, 0.21 mass%Si, 0.74 mass%Mn, 1.03 mass%Cr, 0.14 mass%Mo, P and S below 0.03 mass%, and Fe as a balance. The as-received steel plate (17.5 mm × 12.5 mm × 5 mm thick) was machined with two or four channels (tubes). Channels had 1, 1.5, 2 and 3 mm inner diameter (i.d.) and length of 5 mm (both channel ends open) or 12 mm (one end open, one end closed).

The surface hardness of the internal wall was measured along the direction A and cross-sectional hardness was measured along the direction B with a Vicker’s microhardness indenter as shown in Fig. 1. Also, the nitrided channels were examined by X-ray diffraction and scanning electron microscopy.

3. Results and Discussion

Figures 2(a) and (b) show the effect of gas composition on the surface hardness profiles of the internal walls with 1, 1.5, 2 and 3 mm i.d.s. nitrided at 773 K for 5 h in N2-H2 mixing gas.
665 Pa (5 Torr). The average surface hardness of as-received steel was 260 Hv. The hardness values around 800 Hv were obtained at both edges of the hollow tube for all nitrogen compositions after nitriding. For 30%N₂-70%H₂ mixing gas, the internal wall hardness of tube with 3 mm i.d. slowly decreased with the distance from the both edges to the center and the hardness difference between the edge and the center was about 150 Hv. However, the internal wall hardness of hollow tubes with 1, 1.5 and 2 mm i.d.s. decreased rapidly with the distance when it exceeded 1 mm. Also, the center wall hardness decreased with reduction of the inner diameter of the tube.

When the nitrogen concentration was increased to 50%, the internal wall hardness profile of the hollow tube with 3 mm i.d. had almost uniform (above 700 Hv) throughout the tube as shown in Fig. 2(b). This result indicated that the microwave plasma was uniformly generated inside the 3 mm i.d. hollow tube. The center hardness decreased with the reduction of inner diameter. However, the center hardnesses of hollow tubes with 1, 1.5 and 2 mm i.d.s. were higher at 50%N₂ than those at 30%N₂.

The cross-sectional hardness distributions along direction B when hollow tubes were nitrided in 50%N₂-50%H₂ and 30%N₂-70%H₂ are illustrated in Figs. 3(a) and (b), respectively. This measurement was carried out at the depth of 1 mm from the edge. Figure 3(a) shows that the diffusion layer grew to about 0.3 mm at nitrogen concentration of 30% and Fig. 3(b) shows it was 0.6 mm at nitrogen concentration of 50% for all four diameter tubes. There was little variation between distributions at different diameters. No remarkable changes were observed.

Figure 4 shows an XRD pattern for the internal wall surface of the 3 mm i.d. hollow tube when it was nitrided in 50%N₂-50%H₂ at 773 K for 5 h. ε-Fe₂₃N and γ’-Fe₄N nitride compounds were precipitated at the internal surface of the hollow tube. Therefore, the high hardness at the internal wall surface of the hollow tubes was mainly caused by the precipitated nitride compounds. Cross-sectional SEM images of 3 mm i.d. nitrided hollow tube are shown in Fig. 5.
with 3 mm and 2 mm i.d. hollow tubes were approximately 9 mm and 6 mm, respectively. Therefore, the effective nitriding depth of microwave plasma inside the tube was 3 times the tube inner diameter.

4. Conclusions

Hardness values around 800 Hv were measured at both ends of the nitrided tubes for all diameters. The internal wall surface hardness profile of the 3 mm hollow tube had almost uniform hardness above 700 Hv all along the entire tube length by the formation of $\varepsilon$-$\text{Fe}_2\text{N}$ and $\gamma'$-$\text{Fe}_3\text{N}$ compound layers. Internal wall of hollow tube could be nitrided up to 3 times the inner diameter using microwave plasma.

REFERENCES