Plasma Nitriding Performed under Atmospheric Pressure using Pulsed-Arc Plasma Jet

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Abstract: Plasma nitriding is achieved by spraying the nitrogen pulsed-arc plasma jet under atmospheric-pressure N_2/H_2 mixture. The quality of nitriding is found to depend on the H_2 flow rate, which has an optimal value. We propose a simple mechanism of this dependence. Moreover, the amount of H_2 necessary for best nitriding can be decreased to 1/20 by changing the way of H_2 addition.

Keywords: nitriding, surface hardening, atmospheric-pressure plasma, plasma jet

Plasma nitriding is one of the surface-hardening technologies utilized for a number of mechanical products such as automobile components, dies, and cutting tools [1-6]. As the treatment requires a vacuum system in conventional plasma-nitriding methods, the treatment is performed as a batch process and the capital cost becomes very high. To eliminate these shortcomings, we are developing a new plasma nitriding using atmospheric-pressure plasma technologies. As far as the authors know there are only a few literatures on successful surface hardening by atmospheric-pressure plasma nitriding, e.g., nitriding with a dielectric barrier discharge [4]. Of a wide variety of atmospheric-pressure plasmas, we have adopted the pulsed-arc (PA) plasma jet for the following two reasons [7]. First, the plume temperature of the PA discharge is suitable for nitriding. Second, the PA discharge can produce relatively high density reactive species.

Up to now, we have demonstrated that local nitriding is feasible with spraying the PA plasma jet onto a steel surface, provided that H_2 gas is added to the ambient N_2 atmosphere [8]. Here, we report the detail of H_2 addition influencing the quality of nitriding. Moreover, a new method of H_2 supply is described in which the H_2 amount necessary for nitriding can be decreased drastically.

The jet nozzle is composed of a coaxial cylindrical electrode system as shown in Fig. 1. The grounded external electrode is 35 mm in inner diameter and has an orifice of 4 mm in diameter at the tip. The tip of the internal electrode has a curvature

radius of 4 mm. The electrode gap, between the internal electrode tip and the inner wall of the external electrode near the orifice, is 18 mm. N_2 gas of 99.99% in purity is introduced into the gap at 20 L/min. The low-frequency voltage pulse (4-5 kV in height and 21 kHz in repetition as shown in Fig. 2) is applied to the inner electrode using a high voltage power supply. The maximum of the discharge current is ca. 1 A. The afterglow of the generated PA plasma is spewed out from the orifice, forming the jet plume.

Nitriding is performed in a closed cylindrical container. The jet nozzle is inserted into the container from the upper end. Prior to the generation of plasma jet, the N_2 gas flow from the nozzle purges residual O_2 inside the container. Note that it is necessary for performing nitriding to add H_2 gas. Our conventional way is to add H_2 to the N_2 atmosphere through a container port (the atmosphere H_2 mode) [8]. Here we newly tried an alternative H_2 supply, i.e., to add H_2 through the jet nozzle with the working N_2 gas (the nozzle H_2 mode). The N_2/H_2 gas mixture flows out through the exhaust ports mounted at the lower end. The container is not fitted with any pumping system so that the N_2 pressure inside the container is kept at 1 atm.

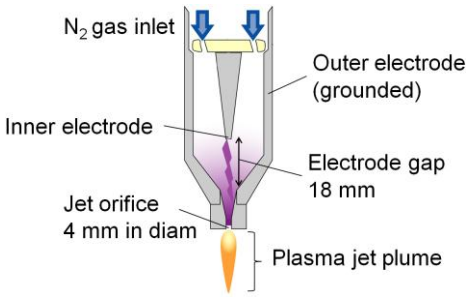


Fig. 1 Schematic of pulsed-arc plasma jet nozzle.

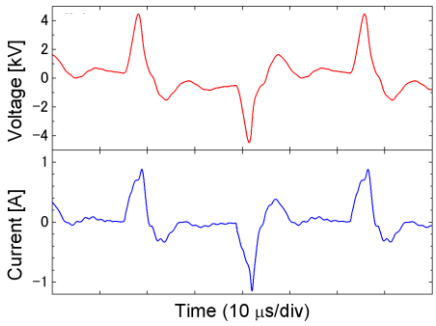


Fig. 2 Typical voltage and current waveforms.

The nitriding response is examined by spraying the jet plume onto the surface of disk-shaped samples (20 mm in diameter and 4 mm in thickness) of die steel JIS SKD61 which is frequently used to benchmark nitriding [3,6]. The sample is put on a ceramic heater to control the treatment temperature. The distance between the nozzle tip and the sample surface is 15 mm, The treatment temperature is 530°C. The treatment duration is 2 h. The visible jet plume covers the entire surface of the sample during the treatment.

Fig. 3 shows a typical metallographic structure of a treated sample cross-section. The white layer is

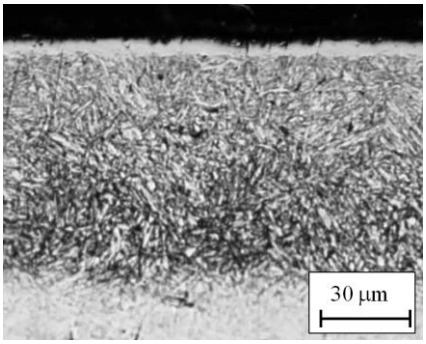


Fig. 3 Metallographic section of sample surface.

the compound layer, which is mainly composed of ε-$Fe_{2-3}N$ phase. The dark zone beneath the compound layer corresponds to the diffusion layer. The hardness profile is shown later. Fig. 4 shows the nitrogen distribution of the cross-section obtained with EPMA. We can see that the compound layer of less than 10 μm contains a dense nitrogen and the diffusion layer (down to 70 μm here) also has a considerable amount of nitrogen. This indicates that the hardening is truly attributed to N atoms diffusing into steel surface.

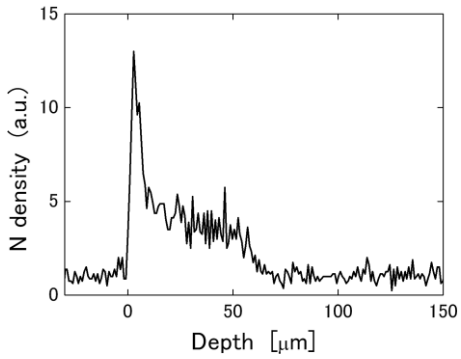
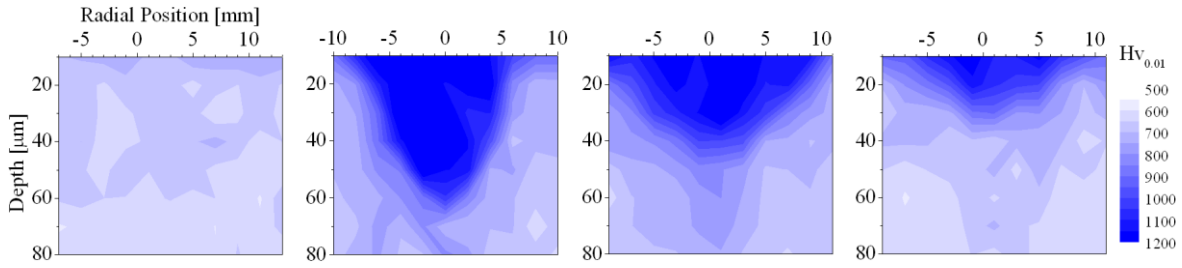


Fig. 4 Relative nitrogen density profile as a function of the depth from surface.

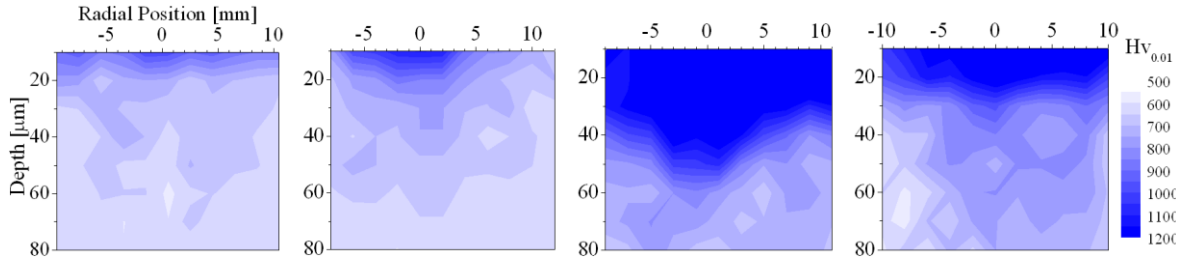
Fig. 5 shows the hardness profile of nitrided cross-section for several H_2 flow rates under the atmosphere H_2 mode. The results indicate that the quality of nitriding is low when the H_2 flow rate is too low, or too high. That is, there exists an optimal H_2 flow rate to provide the deepest hardened layer. In our experiments, the optimal value is 4 L/min. On the other hand, Fig. 6 shows the hardness profiles for the nozzle H_2 mode. One notices that this mode also gives a qualitatively same dependence on the H_2 flow rate. However, the flow rate necessary for nitriding is much lower than that for the atmosphere H_2 mode. The optimal H_2 flow rate, 200 mL/min, proves to be 1/20 of that for the atmosphere H_2 mode.

The optical emission spectroscopy revealed that the intensity of the NH radical is most prominent in the both H_2 modes. Fig. 7 shows the NH emission intensity as a



0.4 L/min 4 L/min 7 L/min 15 L/min

Fig. 5 Hardness profile of nitrided samples treated under the atmosphere H_2 mode.



20 mL/min 100 mL/min 200 mL/min 500 mL/min

Fig. 6 Hardness profile of nitrided samples treated under the nozzle H_2 mode.

function of the H_2 flow rate. It can be predicted from the result that a low H_2 flow rate provides a high-density NH radicals and the density decreases with increasing H_2 flow rate under the both H_2 mode. This dependence might be due to that a high H_2 flow rate leads to the production of NH_3 rather than NH. It should be emphasized here that a substantially analogous NH emission intensity is obtained under the nozzle H_2 mode although the horizontal scale for this mode is 1/40 of that for the other mode. The effective NH production under the nozzle H_2 mode is most likely caused by the direct supply of H_2 to the discharge region involving active chemical reactions.

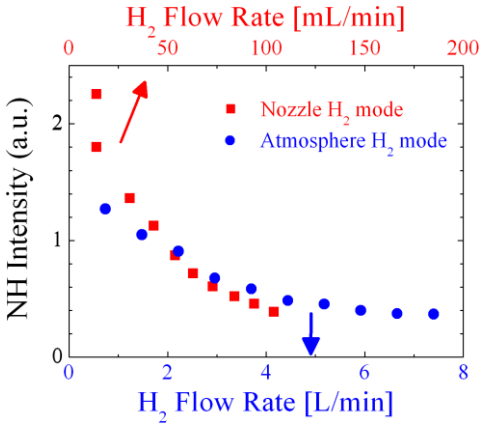


Fig. 7 NH emission intensity as a function of H_2 flow rate in the both H_2 mode.

By considering the presented results comprehensively, we construct a likely mechanism of the plasma-jet nitriding as follows:

(A) The HN radicals play a role of supplying N atoms to the sample surface.

(B) The low-quality nitriding for too high H_2 flow rate under the both H_2 mode is due to the lack of N supply to the surface.

(C) The low-quality nitriding for too low H_2 flow rate is caused by the surface oxidization by residual O_2 ramaining because of a low H_2 reduction ability.

(D) The efficient production of NH radicals under the nozzle H_2 mode provides the decrease in the H_2 amount necessary for nitriding to 1/20.

We hope that the PA plasma jet nitriding will offer an easy-to-use, economical hardening method to industrial and scientific fields.

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