DEMONSTRATION OF NITRIDING BY DIELECTRIC BARRIER DISCHARGE AND INVESTIGATION OF TREATMENT RANGE CONTROLLABILITY

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ABSTRACT

Nitriding is a surface hardening process for metals to upgrade wear resistance and fatigue resistance. As a new trial, nitriding by dielectric barrier discharge was performed under atmospheric pressure. N_2, H_2 and Ar were used as the operating gases. As a result, the nitrided layer of about 1100 Hv was formed. Moreover, we found that the boundary of the treated and untreated area is distinct, that is, the hardness transition from base material to hard layer occurs within 100 μm.

1. INTRODUCTION

Nitriding is one of the surface hardening technique of metals. In nitriding, steel is heated up to 450-600°C and nitrogen atoms diffuse into steel surface to form hard layer. Nitriding for steels upgrades wear resistance and fatigue resistance [1]. Nitriding mechanism is that nitrogen is dissociated by kinetic energy of electrons in plasma, and active species (ion, radical etc.) is produced. Finally, nitrogen atoms thermally diffuse into metal and nitrogen compound is formed. A nitrided layer is separated into the compound layer and the diffusion layer. The compound layer is composed of Fe_4N and $Fe_{2\text{-}3}N$ produced by chemical reactions. The diffusion layer is solid solution of iron and nitrogen. In industry, plasm nitriding treatment is performed under low pressure so that vacuum equipment is required. On the other hand, we investigated the plasma nitriding treatment under atmospheric-pressure by pulsed-arc plasma jet. As a result, we have enabled simple nitriding without vacuum equipments [2]. This technique befits local treatment, but unsuits large-area treatment.

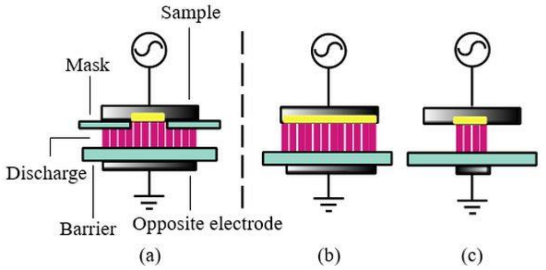

Fig. 1 Schematic of large area treatment and mask-free patterning by DBD. (a) Conventional patterning with masking. (b) Large area treatment. (c) Mask-free patterning.

As a new trial, nitriding by dielectric barrier discharge was performed under atmospheric pressure. One of the new possibilities of DBD is large-area treatment which may be achieved by expanding the area of the opposite electrode as shown in Fig. 1(b). As a previous study, the DBD nitriding with ammonia gas was researched by Yan *et al* [3]. However, because ammonia is toxic, measure for safety and exhaust gas treatment are needed. For this reason we started research on DBD nitriding treatment only using safe gases [2].

As another novelty, controllability of treatment area in DBD nitriding was also investigated in connection with patterning technology of nitriding. In conventional patterning of nitriding, a patterned mask is applied on the material to shield areas in which nitrogen diffusion is undesired as shown in Fig.1(a) [4]. In contrast, the use of a patterned opposite electrode will enable patterning of DBD, leading to patterning of nitriding without any masks as shown in Fig.1(c). Here, we would like to show some date implying the possibility of mask-free patterning of nitriding using DBD.

2. EXPERIMENTAL PROCEDURE

〈2・1〉 DBD treat furnace The schematic of DBD treat furnace is shown in Fig. 2(a) and the electrode system is detailed in Fig. 2(b). The barrier is made of an alumina plate of 2.5 mm in thickness and 50×50 mm^2 in area. A steel sample for nitriding treatment is used as an electrode, called "the sample electrode". The other electrode is called "the opposite electrode". The barrier is sandwiched between the two electrodes. Four spacers made of machinable ceramic (MACOR) is set below the four corners of the sample electrode. Four spacers made of machinable ceramic (MACOR) is set below the four corners of the sample electrode. The discharge gap is 1 mm. The opposite electrode is covered with ceramic bond to prevent spark. The AC voltage of 4.5 kV and 12.8 kHz is applied to the sample electrode by a high-voltage supply (LOGY ELECTRIC CO. LTD. LHV-13AC) as shown in Fig. 3. The electrode system is placed inside a quartz tube. The length of quartz tube is 500 mm and the inner diameter is 95 mm. The $N_2/H_2/Ar$ gas mixture is introduced into the quartz tube as the operating gas. Both ends of the quartz tube are sealed up for preventing invasion of O_2. The quartz tube is interpolated into an electric furnace to control the treatment temperature to ca. 530°C. A photograph of discharge is shown in Fig. 4.

〈2･2〉 Sample A hot work tool steel JIS SKD61 (Cr:5%, Mo:1%, Si:1%, C:0.4%) was used as the sample electrode in this experiment. A plane sample (area: 15×15 mm^2, thickness: 4 mm) was quenched by air blast cooling from 1025°C then was tempered to 550 Hv. The sample surface was polished to mirror finish and degreased ultrasonically in acetone bath. The nitriding treatment is performed at 530°C for 2 h. The micro-Vickers hardness tester (FUTURE-TECH, FM-300) was used for measuring the sample hardness.

3. RESULTS AND DISCUSSION

〈3･1〉 Formation of nitrided layer First, The demonstration experiment of DBD nitriding was performed. The gas flow rates are N_2: 1.8, H_2: 0.2 and Ar: 2.0 slm. The N_2 gas is used, of course, to produce nitrogen atoms. One of the purposes of H_2 gas addition is reduction of residual O_2. The other purpose is production of NH radicals because we regard NH as a key radical in our previous research on atmospheric-pressure plasma nitriding with the pulsed-arc plasma jet [2]. The purpose of Ar gas addition is promotion of discharge by Penning effect.

The hardness profile of cross-section is shown in Fig. 5 where the vertical axis is the depth from surface and the horizontal axis is the distance from edge of sample surface. Moreover, the hardness is indicated as grayscale. It is clearly seen that the hardness layer of ca. 1100 Hv is formed. That is, we achieved nitriding only with safe gases. Moreover, we can see that the treated range is limited sharply, approximately from 3 to 12 mm in the distance from the edge. This treated area corresponds to the shape of opposite electrode. This means that control of treatment area is possible by patterning the opposite electrode. The precision of area control is discussed in Sec. 3.3.

〈3･2〉 Comparison of gas composition Second, comparison experiment of gas composition was conducted. In addition to $N_2/H_2/Ar$ mixture mentioned

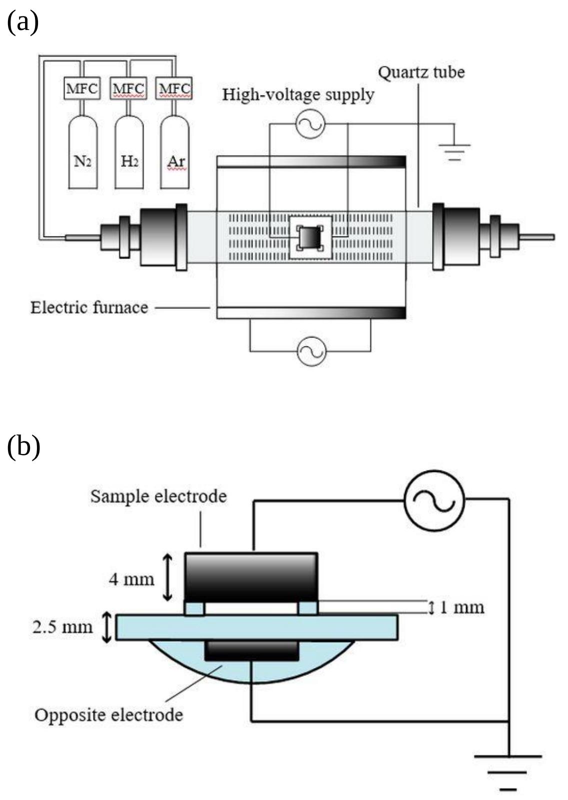

Fig. 2. Schematic of experimental setup.
(a) Treatment furnace. (b) Electrode system.

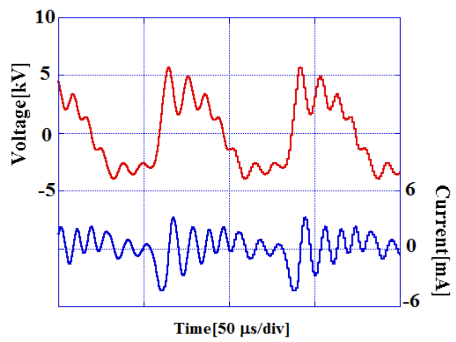

Fig. 3. Voltage and current wave forms.

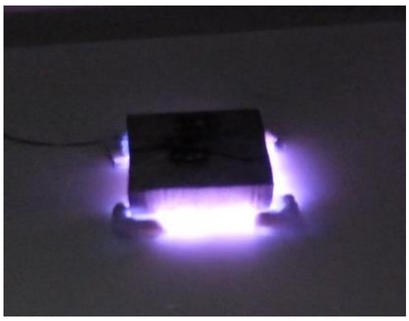

Fig. 4. Photograph of DBD.

above, we investigated N_2/H_2 mixture (N_2: 3.6, H_2: 0.4 slm) and pure N_2 gas (4 slm). The other experimental parameters were identical to the previous section.

The hardness gradient obtained for each operating gas is shown in Fig. 6. The vertical axis is the hardness and the horizontal axis is the depth from surface. As a result, similar nitrided layers with the maximum hardness of 1100-1200 Hv were formed for all gases. Note that the hard layer was formed only with N_2 gas. In contrast, we have seen that nitriding by pulsed-arc plasma jet was impossible without H_2 gas[2]. It was proved that the DBD nitriding does not require even H_2 gas as well as ammonia gas. This result indicates a possibility of safer and environment-friendly metal hardening technique.

〈3･3〉 Controllability of treatment area As show in Fig. 5, the hard layer is formed at only a limited area corresponding to the existence of opposite electrode, even though no masking is used. The boundary of hard layer and base material was investigated in detail to estimate controllability of treatment area. This result is shown Fig. 7, where the vertical axis is the hardness and the horizontal axis is the distance from edge of sample surface. The average hardness of hard layer is shown as a broken line, and that of base material is shown as a dotted line. The hardness transition from the base metal to the hard

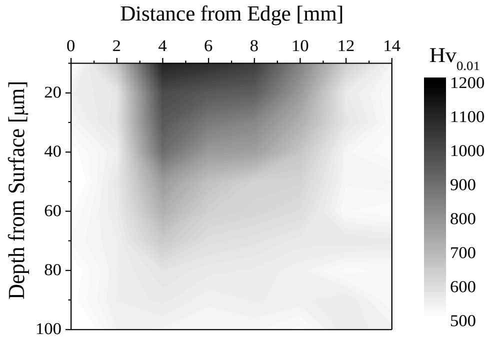

Fig. 5. Hardness profile of sample cross-section.

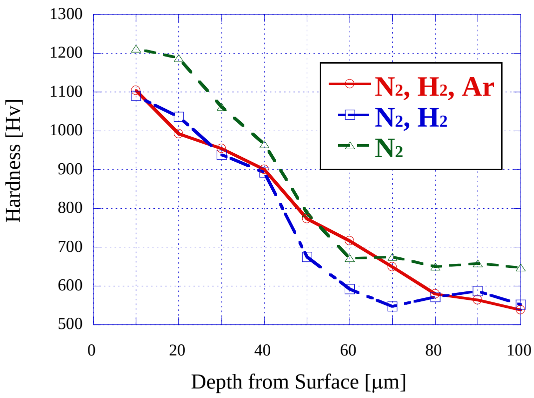

Fig. 6. Hardness gradient for several treatment gas.

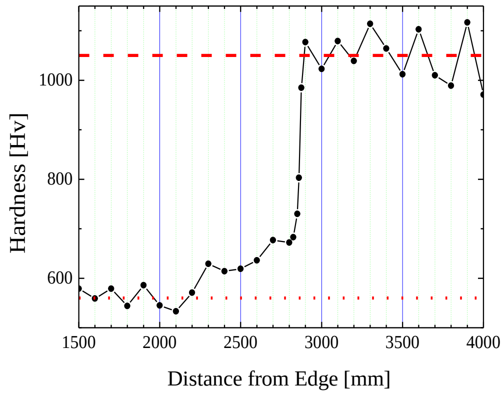

Fig. 7. Surface hardness profile in the vicinity of the boundary of treated and untreated area.

layer occurs within 2800-2900 μm. This means that treatment area control by DBD is possible with the spatial resolution less than 100 μm.

4. CONCLUSION

In this research, we demonstrated the DBD nitriding without ammonia for the first time. The hard layer is formed within only a limited area corresponding to the shape of opposite electrode. We consider that the large-area treatment becomes possible by enlargement of opposite electrode. Moreover, in the vicinity of the boundary of base material and hard layer, the hardness transition occurs within 100 μm. This fact indicates a new possibility of patterning of nitriding without masking, provided that the opposite electrode is patterned. As future subjects, we would like to realize large-area treatment and treatment area control with high accuracy.

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