Life Enhancement of Hot-Forging Dies by Plasma-Nitriding

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Abstract

Hot-forging dies, made of AISI-H13 hot-work tool steel, were plasma-nitrided in order to improve the surface properties of the material. A diffusion layer without the white layer was formed on the material by controlling the nitriding parameters: vacuum pressure, temperature, N_2 / H_2 ratio and DC bias potential. The nitriding time was varied from 2 to 16 hours. Micro-hardness measurements were performed on the surface and through the diffusion layer for each nitrided material. Optical microscopy was performed on the as-received and nitrided materials. The surface hardness of the materials increased from 550 HV0.1 to 1300 HV0.1 after plasma-nitriding. Materials plasma-nitrided for 8-10 hours had the highest surface hardness. Surface hardness decreased after 10 hours of plasma-nitriding. Work pieces, made of AISI-1020 steel, were forged at 900°C by the plasma-nitrided dies and the lives of the dies were measured. The lives of plasma-nitrided hot-forging dies increased largely in comparison to those of the as-received dies. Highest life enhancement of the dies was achieved by 10 hours of plasma-nitriding.

Key Words: AISI-H13 hot-work tool steel, plasma-nitriding, hardness, hot-forging dies, life enhancement.

Plazma-Nitrürleme ile Sıcak-Dövme Kalıplarının Ömürlerinin Arttırılması

Özet

AISI-H13 sıcak-iş çeliğinden yapılan sıcak-dövme kalıplarının yüzey özelliklerin iyileştirmek için plazma-nitrürleme işlemi yapılmıştır. Nitrürleme parametrelerinden, vakum kabı basıncı, işlem sıcaklığı, N_2/H_2 oranı ve çalışma gerilimi kontrol edilerek, malzeme yüzeyinde, beyaz tabakasız bir difüzyon tabakası oluşturulmuştur. Nitrürleme işlemi 2 saatten 16 saate kadar çeşitli sürelerde gerçekleştirilmiş, nitrürlenen parçalardan alınan kesitlerde mikro-sertlik ölçümleri yapılmış ve optik mikroskopi ile içyapıları incelenmiştir. Plazma nitrürleme ile yüzey sertliğinin 550 HV0.1 değerinden 1300 HV0.1 kadar yükseldiği görülmüştür. Parçaların yüzey sertliğinin 8-10 saat nitrürlenen malzemelerde en fazla olduğu, 10 saatten fazla nitrürlenen malzemelerde yüzey sertliğinin azalmaya başladığı gözlenmiştir. Nitrürlenen kalıplarla 900°C de AISI-1020 çeliğinden parçalar dövülmüş ve kalıp ömürleri tesbit edilmiştir. Plazma nitrürlenmiş sıcak dövme kalıplarının ömürlerinin, nitrürleme yapılmamış kalıpların ömürlerine oranla önemli ölçüde arttırılabileceği gösterilmiştir. Ömür artışının en fazla 10 saat nitrürleme sonucu elde edildiği görülmüştür.

Anahtar Sözcükler: AISI-H13 sıcak-iş çeliği, plazma-nitrürleme, sertlik, sıcak-dövme kalıpları, ömür arttırma.

Introduction

In general, under high loads, temperatures and corrosive environments, the surfaces of machine parts are subjected to higher stresses than the interior regions. Unless there exists a material defect within the machine component, failure starts from the surface region. Contact surfaces of the machine components tend to wear much faster than the other regions due to contact loads and other stress inducing effects, e.g., heat and corrosion. By steel surface treatments such as nitriding and carburising, the surface hardness of the material is increased and this effect improves the wear properties of the materials.

Plasma-nitriding, also called ion-nitriding, or glow discharge nitriding is one of several advanced surface treatment processes for improving the surface properties of the materials (Kovacs and Russell, 1986; Spalvins, 1986). It is a thermo-chemical process, in which the surface chemistry of the material is changed under heat. Plasma-nitriding has advantages over traditional nitriding processes such as salt-bath-nitriding and gas-nitriding, including lower treatment times, lower temperatures and environment friendliness.

In the literature, plasma-nitriding surface treatments of various steels have been reported (Salik, 1985; Cohen et al., 1986; Özbaysal et al., 1986; Sun and Bell, 1991; Krauss, 1992). In the present study, the plasma-nitriding surface treatment process was applied to AISI-H13 hot-work tool steel. Plasma-nitriding time was varied from 2 to 16 hours in order to investigate the effects of time on the nitriding process. This material was used for making hot-forging dies. The effects of nitriding time on the hardness, diffusion layer depth and life of hot-forging dies were studied.
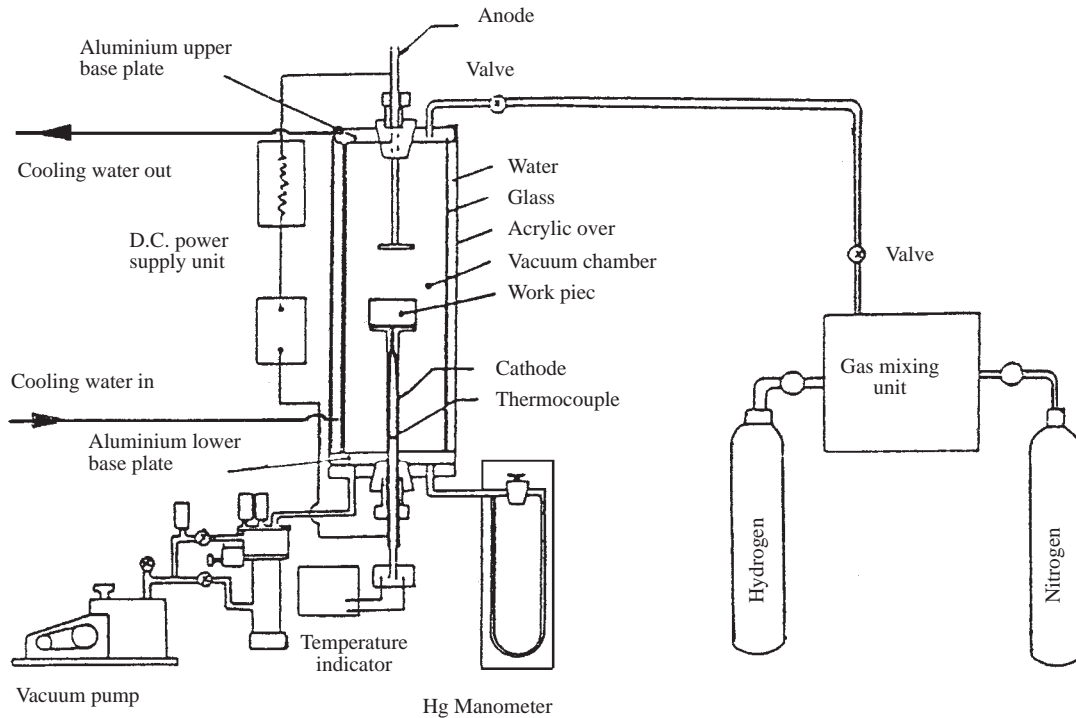

Figure 1. Schematic diagram of the plasma-nitriding system.

1. Experimental

1.1. Material

AISI-H13 hot-work tool steel was used. The chemical composition of the steel is given in Table 1. Dies of outer dimensions of 50 mm diameter and 20 mm thickness were made of this material. Dies were used to forge 4×4.2 mm^2 work pieces, which were used to operate the handles of double-glass plastic-frame windows.
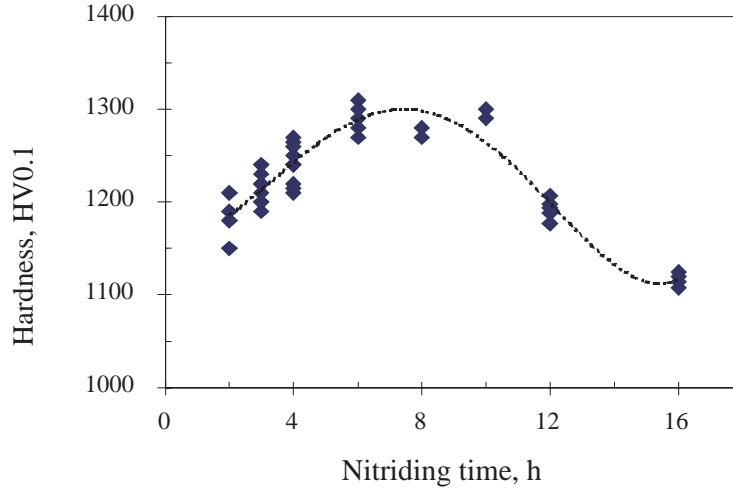
Figure 2. Surface hardness as a function of nitriding time.

Table 1. Chemical composition (wt.%) of AISI-H13 hot-work tool steel.

C	Mo	Cr	V	Fe
0.35	1.5	5.0	1.0	rest

Before the plasma-nitriding process, the following heat treatments were applied to the forging dies:

(i) 350°C-400°C, pre-annealing for 1 hour,

(ii) 800°C-820°C, annealing in an inert atmosphere for 1 hour,

(iii) 1040°C, austenization for 20 minutes,

(iv) 500°C-520°C, quenching in salt-bath, then cooling in air,

(v) 560°C, tempering 3 times, each for 2 hours.

After these heat treatments, the dies were machined to final shapes by numerical-control electro-discharge-machining. To prepare the dies for standard micro-hardness test measurements, the surfaces of the dies were polished with emery paper of grade 600. Finally, the dies were degreased before the plasma-nitriding process.

1.2. Plasma-nitriding

The plasma-nitriding apparatus was designed and built in our laboratory (Figure 1). Similar systems and their operations have been described by Kovacs and Russell (1986) and Spalvins (1986). Using this system, work pieces with up to $100 \times 100 \times 100$ mm^3 dimensions can be plasma-nitrided. The system is operated with a DC power supply. Under vacuum and with electric potential between anode and cathode, where the work piece is placed, ions develop in the H_2/N_2 gas mixture.

The plasma-nitriding of ferrous alloys, according to Spalvins (1986), can be explained by two different mechanisms:

i) under high-energy ion bombardment of the cathode, sputtered Fe atoms react with atomic nitrogen in the gas plasma and form unstable FeN. After condensation on the surface, FeN releases nitrogen to form lower order iron nitrides, i.e., Fe_2N, Fe_3N and Fe_4N.

ii) high energy ion bombardment of the cathode introduces vacancies and vacancy clusters on the metal surface. This increases the diffusion process. Diffused nitrogen and iron form the iron nitrides.

Depending on the experimental conditions, two different nitride layers may develop on the surface of the ferrous metals: a thin hard brittle white layer and/or a relatively thick diffusion layer strongly bonded to the core region, (Cohen et al., 1986; Sun and Bell, 1991). In the compound layer $Fe_{2-3}N$ and Fe_4N formation has been observed, and in the diffusion zone Fe_4N and $Fe_{16}N_2$ formation has been observed (Metin and Inal, 1987: Xu et al., 1996).

In this work, with the following plasma-nitriding experimental conditions, diffusion layer without the

white layer was obtained on the surface of the material. The plasma-nitriding experimental conditions were as follows:

    i) vacuum pressure: 9-11 torr,

    ii) temperature: $470\pm10°C$,

    iii) gas mixture: 20 vol. % N_2 and 80 vol. %H_2,

    iv) electric potential: 500 Volts.

In this work, plasma-nitriding time was the only experimental variable. By varying the time from 2 to 16 hours (plasma-nitriding was done at 2, 4, 6, 8, 10, 12 and 16 hours for each specimen), the effects of time on the diffusion layer thickness, surface hardness and total number of hot-forged work pieces under the specified dimensional tolerances were analyzed.

1.3. Micro-hardness Measurements

In order to analyze the effects of plasma-nitriding on the surface of the die materials, micro-hardness measurements were made on each plasma-nitrided die as well as on the as-received die. Vickers micro-hardness measurements were made with a Karl Frank Micro-hardness Test Machine (Mannheim, Germany). Applied load for the measurements was 100g. As-received (heat treated) material had a micro-hardness of 550 HV0.1. Micro-hardness measurements of the plasma-nitrided materials were made on the surfaces of the materials and also through the diffusion layer. From the diffusion layer micro-hardness measurements, the thickness of the diffusion layers was obtained.

1.4. Optical Microscopy

In order to see the effects of plasma-nitriding on the microstructure, optical microscopy (Olympus, Tokyo, Japan) was performed on the as-received die and for each plasma-nitrided die. For optical microscopy analysis, all the specimens were prepared from the dies and were etched in nital.

1.5. Hot-forging

The aim of this work was to use plasma-nitriding to improve surface properties of the hot-forging steel dies so that the total number of work pieces, which was initially 2000, could be increased. The material of the hot-forged work pieces was AISI-1020 steel.

The applied force for hot forging was generated by a 350 kN mechanical press. In order to find the number of hot-forged work pieces for each plasma-nitrided die, a counter was placed on the press. Before the forging operations, the work pieces were heated to 900°C in an oven near the press. A gage-master was used in order to check the dimensions of the forged work pieces. Initially the dimensions of the work pieces were 4×4.2 mm^2. Due to the wear occurring after a certain number of forging operations, the geometry of the dies changed, i.e., the dimensions of the forged work pieces increased. When the dimensions of the hot-forged work pieces became greater than 4.05×4.25 mm^2, i.e., $+0.05/2 = 0.025$ mm tolerance of the gage-master, the forging operation was stopped and the number of hot-forged work pieces, read on the counter, indicated the life of the dies.

2. Results and Discussion

Micro-hardness measurement results are shown in Figures 2 and 3, on the material surfaces and through the diffusion layer thickness, respectively. Accordingly, plasma-nitriding treatments were found to increase the surface hardness of the materials in comparison with the as-received material, which had a hardness of 550 HV0.1. The surface hardness varies with nitriding time. About 8-10 hours of ion-nitriding treatment increases surface hardness to a maximum of about 1300 HV0.1. The plasma-nitriding hardening mechanisms of low alloy steels are explained by Sun and Bell (1991). Nitrogen diffuses in the steel from the surface to the core. During the diffusion process, nitrogen partly replaces carbon in martensite, and forms metal nitrides, such as iron nitrides and chromium nitrides (Ozbaysal et al., 1986 Takada et al., 1986) in the matrix and also at the grain boundaries. Redistribution of carbon occurs in the diffusion zone, and carbon is pushed back towards the core region. Near the surface, carbon concentration decreases, and towards the core region a higher concentration of carbon is observed in comparison to the overall carbon concentration (Souchard et al., 1991; Sun and Bell, 1991). Diffusion of nitrogen and redistribution of carbon in the diffusion zone are functions of nitriding temperature and time. Increasing the time beyond 10 hours of ion-nitriding decreases the surface hardness of the materials. A similar decrease in surface hardness with increasing plasma-nitriding time has also been observed with plasma-source-nitrided stainless steel

material (Nurogaki et al., 1988) and plasma-nitrided material of AISI-H11 and H13 hot-work tool steel

(Ozbaysal et al., 1986; Souchard et al., 1991).

Figure 3. Change of hardness from surface to the core region.

In order to determine the diffusion layer thickness mechanically, micro-hardness measurements were conducted from the surface to the core region with each plasma-nitrided material, as shown in Figure 3. Accordingly, the hardness decreases from surface to the core region, where the hardness is assumed to be the same as the as-received material. To find the diffusion layer thickness using hardness versus distance from surface data, the following procedure was used. The thickness of the diffusion layer was taken from the hardness versus distance curve at a point where the curve values do not change more than 5% HV0.1. Using these data, diffusion layer thickness versus square root of nitriding time graph was plotted in (Fig. 4). A trend-line, which shows a linear dependence of diffusion layer thickness to the square root of time, was drawn through the data points. Thus, plasma-nitriding is controlled by a diffusion mechanism (Nunogaki et al., 1988; Sun and Bell, 1991).

The aim of this work was to improve the wear properties of the AISI-H13 hot-work tool steel, so that the life enhancement of hot-forging dies could be achieved. Using the method explained in section 1.5, the lives of hot-forging dies were obtained and their variation with plasma-nitriding time was plotted in Figure 5. For 10 hours of plasma-nitriding treatment, the number of hot-forged work pieces reached a maximum of about 17000 work pieces, which is

more than 8 times of 2,000 work pieces forged with the as-received dies. Data on the trend of number of hot-forged work pieces versus nitriding time shows similarity to the hardness versus nitriding time data given in Figure 2. This suggests that the life of hot-forging dies is controlled by their surface hardness. This is not surprising, since under repeated high impact loads and varying temperatures, which lead to mechanical and thermal fatigue, wear is controlled by the surface hardness of the materials.

Figure 6 shows the microstructure of the material ion-nitrided for 6 hours. In this figure, from left to right are shown the embedding plastic material (A), the diffusion zone (B) and the core region (C). Micro-hardness impressions through the diffusion layer and the core region are also shown in this figure. The approximate thickness of the diffusion layer with ion-nitriding time (Figure 4), which was obtained from hardness versus distance from surface data, Figure 3, can also be measured by using optical micrographs, such as that shown in Figure 6.

3. Conclusion

From the results, it can be concluded that plasma-nitriding of AISI-H13 steel improved the surface properties of the material. Surface hardness reaches a maximum of about 1300 HV0.1 after 8 to 10 hours of plasma-nitriding at 470°C. The hardness of the

as-received material was about 550 HV0.1. Plasma-nitriding this material under the described experimental conditions resulted in a diffusion layer only without the white nitride layer. Thus, the mechanism of plasma-nitriding in these conditions was a diffusion-controlled process. The maximum lives of plasma-nitrided hot-forging dies increased to more than eight times of that of the as-received die.

Figure 4. Diffusion layer thickness as a function of square root of nitriding time.

Figure 5. Number of hot-forged work pieces versus nitriding time.
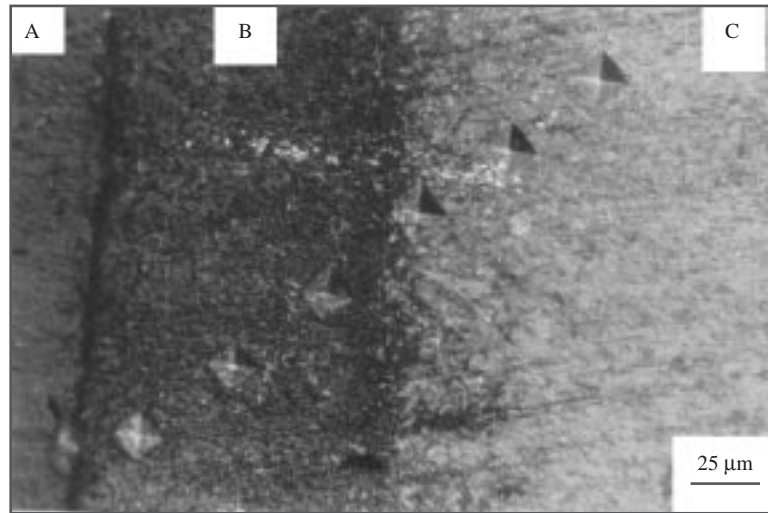
Figure 6. Optical micrograph of the 6 hours plasma-nitried die material. A: embedding plastic material, B: diffusion zone, C: core region.

References

Cohen, A., Boas, M. and Rosen, A., "The Influence of Ion Nitriding Parameters on the Hardness Layer of 15-5 PH Stainless Steel", Thin Solid Films, 141, 53-58, 1986.

Kovacs, W. and Russell, W., "An Introduction to Ion Nitriding, What is it? Why is it Used? Where is it Used?", "Ion Nitriding", Proceedings of the Conference, ASM International, Cleveland, Ohio, 9-17, 1986.

Krauss, G., "Advanced Surface Modification of Steels", J. Heat Treating 9 (2), 81-89, 1992.

Metin, E. and Inal, O.T., "Formation and Growth of Iron Nitrides During Ion-nitriding", J. Mater. Sci., 22, 2783-2788, 1987.

Nunogaki, M., Suezawa, H., Hayasi, K. and Miyazaki, K., "Plasma Source Nitriding", Appl. Surf. Sci. 33/34, 1135-1141, 1988.

Ozbaysal, K., Inal, O.T. and Romig, Jr., A.D., "Ion-nitriding Behavior of Several Tool Steels" Mater. Sci. Eng., 78, 179-191, 1986.

Salik, J., "Ion-beam Nitriding of Steels", J. Appl. Phys. 57 (4), 1328-1331, 1985.

Souchard, J.P., Jacquot, P. and Buvron, M., "Plasma Overcarburizing of Chromium Steels for Hot Working and Wear Applications", Mater. Sci. Eng., A140, 454-460, 1991.

Spalvins, T., "Plasma Assisted Surface Coating/Modification Processes: An Emerging Technology", "Ion Nitriding", Proceedings of the Conference, ASM International, Cleveland, Ohio, 1-8, 1986.

Sun, Y. and Bell, T., "Plasma Surface Engineering of Low Alloy Steel", Mater. Sci. Eng., A140, 419-434, 1991.

Takada, J., Ohizumi, Y., Miyamura, H., Kuwahara, H., Kikuchi, S. and Tamura, I., "Ion-nitriding of an Fe-19 wt % Cr Alloy", J. Mater. Sci., 21, 2493-2496, 1986.

Xu, X., Wang, L., Yu, Z. and Hei, Z., "Transmission Electron Microscopy Study on the Cross-Sectional Microstructure of an Ion-Nitriding Layer", Metal. and Mater. Trans. A, 27A, 1347-1352, 1996.