Overheating of Plasma-Nitrided Workpieces with Cylindrical Blind Holes

R.S. De Sousa and C. Alves, Jr.

In the plasma nitriding process, the workpiece is uniformly covered by glow discharge and is heated by ionic and neutral species bombardment. In this study, AISI 304 stainless steel workpieces with cylindrical blind holes were plasma nitrided in an atmosphere of H_2-$20N_2$ at 1000 Pa, 773 K, for 2 h. The influence of the hole dimensions on the temperature uniformity was investigated. This work is important for the plasma nitriding of workpieces with complex geometrical shapes, such as gearing, crankshafts, dies, and injection nozzles.

Keywords
heat treatment, nitriding, plasma nitriding

1. Introduction

PLASMA nitriding is an advanced surface modification technology that has experienced much industrial development in the past 20 years (Ref 1). In this technique, an electrical discharge in a gas having nitrogen under low pressure is used to promote bombardment of ions and neutral species on steels and cast irons.

In the plasma nitriding process, the pressure used is between 100 and 1000 Pa, electron densities range from 1×10^9 to 1×10^{11}/cm^3, and the ionization fraction is as low as 10^{-6} to 10^{-3} (Ref 2). The difference in mobility between electrons and positive ions causes surfaces in contact with a discharge to assume a negative potential with respect to the plasma. The plasma potential is nearly uniform throughout the glow region. Between the glow and the electrode is a narrow region called the sheath cathode. In these conditions, the sheath thickness is about 1.0 mm (Ref 3).

The workpiece is heated by the bombardment of the discharge. In this way, each point in the workpiece behaves the same to the thermal input heating element, whose power input is given as V_dJ_d, where V_d is the discharge potential and J_d is the current density on the workpiece. One portion of this power is used to heat the workpieces, and the rest is dissipated by conduction, convection, and mainly in the form of radiation (Ref 4). This balance of energy defines, under stable operating conditions, the temperature at each element of the surface area of the workpiece.

It is very important to have good temperature uniformity on the workpieces, and this is affected significantly by workpiece geometry. Surfaces with different shapes lose heat at different rates, and parts of workpieces with a high ratio between the surface area and the volume can become overheated during treatment (Ref 5).

The objective of this paper is to investigate this situation in the plasma nitriding of parts with cylindrical blind holes.

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2. Experimental Work

The plasma nitriding unit used in this work (Fig. 1) consists of a reactor developed in the Department of Materials Engineering at the University of São Carlos-Brazil. Basically it has the following parts: nitriding chamber, power supply, vacuum system, and a microprocessor for collecting and controlling the data.

Stainless steel rods (type 304), 12.7 mm in diameter and 20 mm long, were drilled longitudinally with 3, 6, and 10 mm diam holes with depths of 2, 5, 10, 15, and 17 mm. The internal surfaces of the holes were polished using silicon carbide papers with grit sizes of 220, 340, 400, and 600. All the samples were heated at 1323 K for 20 min and cooled in water to recrystallize the grains damaged during machining. Final cleaning of the samples was carried out using acetone and ultrasonic bath.

The samples were nitrided at 773 K for 2 h with a working pressure of 1000 Pa. This pressure value was selected in order to increase the temperature difference between the two points of the samples. Previous tests were performed with different pressures: 100, 600, and 1000 Pa. After the plasma nitriding treatment, the samples were partially cut parallel to the axis of the cut cylinder and embedded in acrylic resin, then polished using 15 µm chromium oxide and 0.3 and 0.5 µm alumina.

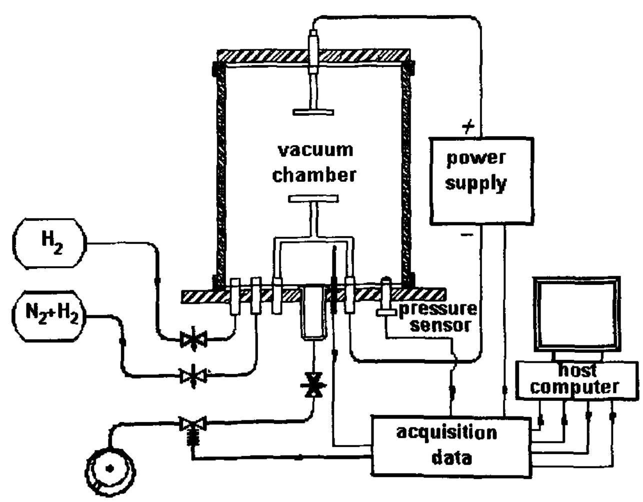

Fig. 1 Schematic of the plasma nitriding unit constructed for this work

Chemical etching was carried out with 50% HF plus 50% H_2O under immersion for 4 s. The samples were observed in an optical microscope of Carl Zeiss (type Neophot) to verify the microstructural aspects and the uniformity of the nitrided layers. A Vickers microhardness apparatus, model KL-2 (Stiefelmeyer) with a 50 g load was used for microhardness measurements.

3. Results and Discussion

The power imparted to the workpieces by the ion and neutral bombardment is partly used to heat the substrate being nitrided. The thermal balance at steady state is:

$$V_d J_d = \rho \frac{v}{A} c_p \frac{\Delta T}{\Delta t} + \sigma\varepsilon\,(T_p^4 - T_w^4)$$

or

$$V_d J_d = \frac{\rho c_p(\Delta T/\Delta t)}{A/v} + \sigma\varepsilon\,(T_p^4 - T_w^4) \quad \text{(Eq 1)}$$

where:

- V_d = potential difference between the workpiece and the plasma
- J_d = current density on the workpiece
- ρ = substrate material density
- c_p = specific heat of the substrate material
- $\Delta T/\Delta t$ = rate of the temperature rise of the substrate
- A/v = surface area to volume ratio
- σ = Stefan-Boltzmann constant, 5.67×10^{-8} W/m$^2 \cdot$ K^4
- ε = surface emissivity coefficient
- T_p = workpiece temperature
- T_w = wall temperature

That is, a part of the power imparted by the plasma, $V_d J_d$, is used to heat the workpiece, $\rho c_p(\Delta T/\Delta t)$, and another is dissipated by radiation to the chamber wall, $\sigma\varepsilon\,(T_p^4 - T_w^4)$. At the condition pressure and temperature used, conduction and convection losses are negligible. Equation 1 shows that if there are two workpieces with different values of A/v, the workpiece with the higher A/v will be heated faster. The reason for this is that the energy input per surface area is the same for two workpieces, and in order for the first term of Eq 1, $\rho c_p(\Delta T/\Delta t)$, to remain constant, an increment in $\Delta T/\Delta t$ is necessary to compensate for the increase in A/v (Ref 3). This leads to an increase in the temperature T_p, so that the sum $[\rho c_p(\Delta T/\Delta t)]/(A/v) + \sigma\varepsilon\,(T_p^4 - T_w^4)$ remains constant.

Table 1 Temperature difference, °C, for various hole depths

Hole diameter, mm	2 mm	5 mm	10 mm	15 mm	17 mm
3	0	4	8	25	...
6	0	7	14	20	...
10	0	8	14	20	45

Rather than two workpieces with different A/v ratios, only one workpiece having regions with different A/v ratios was used in this work. As shown in Fig. 2, the region next to the surface of the hole has an A/v ratio greater than the region next to the bottom of the hole. The temperatures were measured at two different points in the samples, one on the surface (T_s) and another at the bottom (T_b), using thermocouples (Fig. 2). The sur-

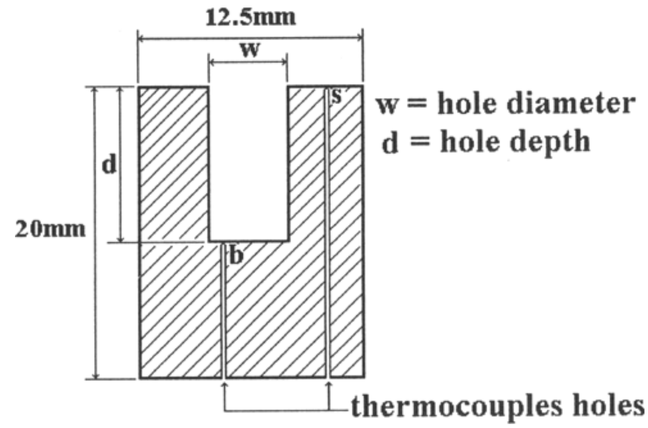

Fig. 2 Schematic of the sample used in this experiment

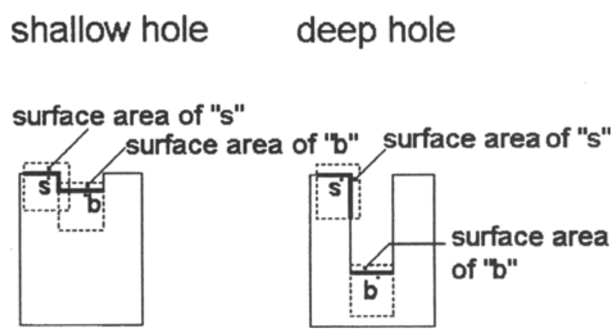

Fig. 3 Effect of A/v ratio in the plasma nitriding of holes with different depths

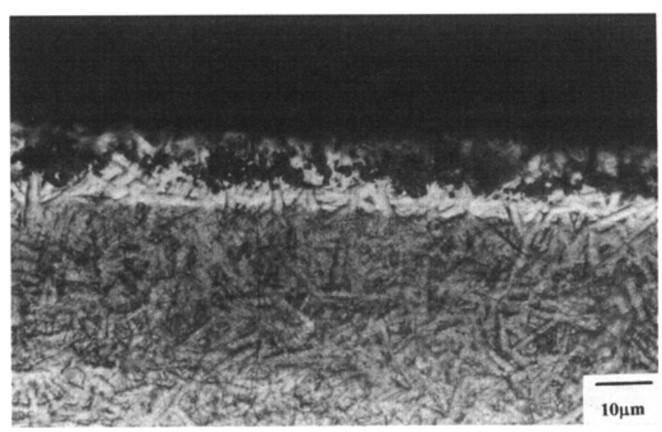

Fig. 4 Nitrided region (internal surface of the hole) of AISI 304 stainless steel workpiece showing a fibrous microstructure with inferior hardness

face temperature (T_s) was maintained constant at 773 K in order to control the discharge potential. The results showed an increase in temperature difference ($T_s - T_b$) between the surface and the bottom of the holes in relation to the increase in depth of the holes for all diameters (Table 1).

Figure 3 explains this effect. Consider two workpieces, one having a shallow hole and another having a deep hole. Imagine two points, one in the surface, *s*, and another in the bottom, *b*; around each point is delimited a region of influence (dashed lines). This region defines in each part the effective surface areas (thick solid lines) that contribute to the heating of the point due to the bombardment by plasma. That is, the greater this surface area, the greater the heating of this point. This way, when the depth of the hole is increased there is an increase in the effective surface area of point *s*, while for point *b* this area remains constant. In relation to volume, Fig. 3 shows that an increase in the depth causes removal of mass from the part, reducing the volume of the material near point *s*, but the same does not happen to point *b*. Consequently, in general, there is an increase in the effective surface area and a decrease in volume for point *s* in relation to point *b*. Both effects cause an increase in the A/v ratio of the surface in relation to the bottom, resulting in an increase in temperature on the surface.

The nonuniformity of temperature on the surface of the parts leads to the dispersion of hardness values and layer thickness and at times can cause faults due to overheating. Figure 4 shows a nitrided region with lower than average hardness. A fibrous microstructure is observed in the substrate and nitrided layer. This microstructure is perhaps associated with overheating in this region, because the microstructure appears as acicular grains in regions with higher superficial hardness. Ruset (Ref 6) found differences in temperatures of up to 80 °C in the treatment of crankshafts and relates this to the complicated shape of the component. In this work the maximum temperature difference was found to be 45 °C for a hole 10 mm in diameter and 17 mm deep (Table 1).

4. Conclusions

Workpieces with cylindrical blind holes were plasma nitrided in an atmosphere of H_2-20N_2 at 1000 Pa, 773 K, for 2 h. The influence of hole dimension on temperature uniformity was investigated.

A temperature difference of up to 45 °C between the surface and the bottom of the cylindrical holes was observed. This could be attributed to the effect of the ratio between the surface area and the volume at different points in the samples.

The effect of overheating was observed at higher pressure levels. The nonuniformity of temperature on the part surfaces leads to the dispersion of hardness values and layer thickness and at times can cause faults due to overheating.

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