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Hardness Curves for 31CrMoV9 Steel after Gas Nitriding

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This work is an experimental study of the hardness curve in the zone of diffusion for the gas nitriding process of 31CrMoV9 steel. 12 samples were subjected to gas nitriding at three different temperatures — 510 °C, 550 °C, and 590 °C — and for each temperature four different nitriding times were used. Hardness was measured with an automatic microhardness tester PCE from LECO with a load of HV0.1 (0.9807N), and the distance between the points was 50 μ m. It was found that the hardness of the diffusion layer changed from 602 HV to 787 HV. The maximum surface hardness was found at 510 °C after nitriding for 16 h. Depending on the process parameters, the depth of the diffusion layer ranged from 270 μ m to 700 μ m. From the results we can draw conclusions for the gradient curve of nitriding parameters, and from each curve we can find the depth of the diffusion of nitrogen in steel.

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1. Introduction

Gas nitriding is a thermochemical process of surface hardening on ferrous materials [1, 2]. This process is used to improve some surface properties such as wear, fatigue, corrosion, friction, and hardness [1–4]. This case involves atomic nitrogen from dissociating ammonia gas diffusing into the steel surface in the temperature range 500 °C– 590 °C [5, 6]. The nitriding of the steel can be visualized as bringing N₂ gas into contact with the surface of the workpiece under extremely high pressure, which becomes possible by using NH₃ mixed with N₂ or H₂ gas. This means that the fundamental reaction for this process is the catalytic decomposition of ammonia to form nascent (elemental) nitrogen [5].

The control parameters include time, temperature, and gas dissociation rate. In fact, the rate of dissociation expressed through nitriding potential is defined as $a_n = p_{\rm NH_3}/p_{\rm H_2}^{1.5}$, where $p_{\rm NH_3}$ and $p_{\rm H_2}$ are the partial pressures of the ammonia and hydrogen gases, respectively [5, 6].

During the gas nitriding process, the reaction occurs not only at the surface but also inside the material because of nitrogen atoms' diffusion from the surface towards the matrix. Nitrogen diffused into a steel surface is combined with alloying elements to form a fine dispersion of alloy nitrides [3]. As a result of the diffusion of nitrogen into the steel, a very hard, wear-resistant and corrosion-resistant surface develops over a tough matrix. The depth to which nitrogen has penetrated is called the nitriding layer or nitriding zone, which consists of the compound layer and diffusion zone [5]. The compound layer, also known as the "white layer", because it is not attacked by alcoholic nitric acid etching, consists predominantly of ε -Fe₂₋₃(C,N) and/or γ -Fe₄N phases and can greatly improve resistance to wear and corrosion. Under the compound layer is the diffusion layer, which is composed of an interstitial solid solution of nitrogen dissolved in the ferrite lattice, and nitride and/or carbonitride precipitation for the alloy steels containing the nitrides forming elements, and is responsible for a considerable enhancement of hardness and fatigue endurance [2, 5].

2. Experimental details

The chemical analysis of steel 31CrMoV9 measured with spark emission spectrometer model JY-132F, is given in Table I.

TABLE I

Chemical composition of 31CrMoV9 steel [wt%]

С	Cr	Mn	V	Mo	Si	S	Р	Al	Fe
0.29	2.17	0.70	0.15	0.27	0.26	0.004	0.016	0022	bal.

For our investigation we took 12 samples of dimensions $\varphi 35 \times 10 \text{ mm}^3$, then labeled them and processed their surface by mechanically grinding them with successive grades of SiC paper, starting with 320-grit and proceeding to 400-, and 600-grit papers, using water to keep the specimens cool [2]. Prior to gas nitriding, the steel specimens were normalized in an N₂ atmosphere at 860 °C for 2 h, followed by rapid cooling in oil, and then tempering at 630 °C for 2 h [2]. After thermal treatment, the specimens were ultrasonically cleaned in acetone for 5 min, dried in hot air, and then transferred to the nitriding environment. The gas nitriding was performed in air-doped ammonia atmosphere at $T = 510 \,^{\circ}\text{C}, 550 \,^{\circ}\text{C},$ and 590 °C for various nitriding times τ . The nitriding parameters and the respective labels of the specimens are given in Table II. After gas nitriding, the specimens were cut in cross-section, mounted in resin, and subsequently ground and polished with a final polishing step of 1 μ m. In order to avoid damaging and rounding off

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TABLE II

at the edges upon cross-sectional preparation, the specimens were protected by Ni-plating prior to mounting in resin.

The hardness was measured in the polished surfaces [7, 8] of samples with an automatic microhardness tester PCE from LECO. For this we used a small load of HV0.1 (0.9807N), in which the distance between the two measuring points was 50 μ m, while the load time was 10 s [9–11]. The total number of points in the sample was between 15 and 30 (depending on the nitriding parameters, i.e. on the depth of the nitrogen diffusion). We also measured the hardness at 5 points in the matrix zone at a depth of 3 mm. Through these points, we found the hardness curves for all samples, then for each curve we calculated the following parameters:

- surface hardness (SH),
- matrix hardness (MH) i.e. hardness of the nonnitrided zone,
- increase in hardness $(\Delta H = SH MH)$,
- depth of nitriding (D_{nt}) .

Gas nitriding parameters: temperature [°C], nitriding potential (NP) [atm⁻¹] and time [h], specimen labels SL, surface hardness SH, matrix hardness MH, increase in hardness ΔH , and depth of nitriding D_{nt} .

Т	NP	au	SL	SH	MH	ΔH	D_{nt}
510	3.4	16	31-1	787	298	482	305
		36	31 - 3	778	290	488	400
		64	31 - 5	776	287	489	500
		100	31 - 7	773	285	503	620
550	3.0	9	31 - 9	783	285	498	270
		16	31 - 11	758	287	471	425
		36	31 - 13	727	303	424	520
		64	31 - 15	709	302	407	650
590	2.82	4	31 - 17	744	290	454	280
		9	31 - 19	630	283	347	380
		16	31 - 21	640	262	378	525
		36	31 - 23	602	278	324	700

3. Results and discussions

Hardness curves were plotted for all samples, as a result of the measurement of the hardness as a function of the depth (Fig. 1). In order to interpret the results more precisely, the hardness values were calculated in the surface and matrixes, as well as the increase in hardness and depth of nitriding (D_{nt}) , see Table II. Determination of the nitriding hardness depth was defined in accordance with the DIN 50190-3 standard. This is ascertained from the hardness curve and lies at a hardness limit of 50 HV below the core hardness.

High values of hardness in the periphery are caused mainly by the iron nitrides in the compound layer, as well as by the formation of individual nitrides in the precipitation layer. It is known that chromium has a high affinity for forming bonds with nitrogen so precipitating as fine semi-coherent CrN nitrides with *B*1-type structure, directly responsible for observed hardening [8].

The curves of hardness differ from each other in absolute value and in depth. Figure 2 shows the influence of temperature in hardness curves for nitriding time of 36 h.



Fig. 1. The hardness curves at three temperatures: (a) 510 °C, (b) 550 °C, and (c) 590 °C.



Fig. 2. Effect of nitriding temperature on the hardness curves for the duration of 36 h.

4. Conclusion

• From Table II and Figs. 1 and 2, it can be seen that the depth of nitriding increased with increasing duration and temperature of nitriding. This can be explained because increased time provides more atomic nitrogen diffusion towards the matrix while an increased temperature raises the diffusion coefficient of nitrogen, providing the same effect [6];

- From Figs. 1 and 2 it can be seen that increasing duration and temperature decreases maximal hardness of the surface [12]. The reason for this is that discontinuous precipitation can occur by increase of the duration and the temperature, which may cause a reduction in hardness at the surface;
- As a result of the above-mentioned two effects, we can also derive a conclusion for the reduction in the gradient's curves with increasing duration and temperature.

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