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Research Article

Metallographic investigation of the diffusion zone of 31CrMoV9 steel after gas nitriding

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Abstract:

Gas nitriding is a process of thermal treatment of steels, which is carried out at temperatures higher than 500^0C. During this process, the peripheral layer of the steel is enriched with nitrogen and thus changes its physical properties. The layer enriched with nitrogen consists of two zones: compound and diffusions or precipitates zone. The thicknesses of the zones depend on nitriding parameters: temperature, duration of nitriding and nitriding parameter. For research we had the chromium-enriched steel 31CrMoV9. This steel is taken because, based on experience and investigation of industry experts, 31CrMoV9 alloy steel stands out for its exceptional combination of strength, fatigue resistance, and machinability. This fact is also mentioned by some of the authors [1]. The nitriding of this steel was done at three different temperatures 510^0C, 550^0C and 590^0C. For each temperature, there were four different durations of stay of the samples in the furnace where nitriding was performed. Then, the samples were investigated by various experimental methods. In this contribution, we will present the results of metallographic research, namely optical microscopy. With the help of the optical microscope, we found the thicknesses of the diffusions zones from which we calculated the growth kinetics of these zones.

1. Introduction

Gas nitriding is a thermo-chemical surface treatment process during which nitrogen is transferred from an ammonia atmosphere into the surface of steels at temperatures above 500℃ [1,2]. In this case, the surface of the steel is enriched with nitrogen, and as a result, some physical properties of the steel are improved. Mainly increases resistance to wear, corrosion and mechanical resistance. There are also two different nitriding methods: plasma nitriding and bath nitriding. The purpose of all three nitriding methods is to cause nitrogen diffusion into the steel surface. The nitrogen-enriched surface forms the nitriding layer which consists of two zones: of compounds layer and of diffusions zone (Fig.1) [2-8]. The thickness of these zones depends on the nitriding conditions: duration, temperature and number of nitriding as well as the type of steel.

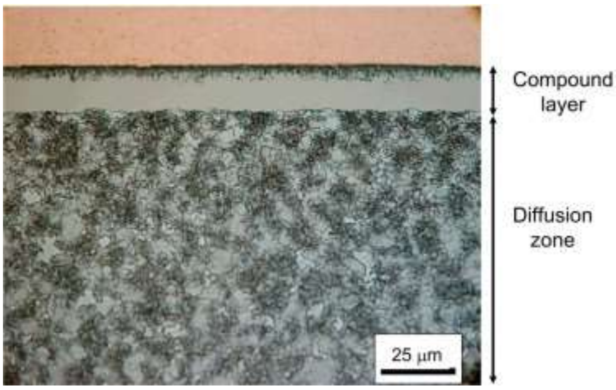


Figure 1. *Structure of nitriding layer.*

In this paper, we have shown the results of metallographic investigation of the diffusion zone depending on the nitriding parameters for steel 31CrMoV9.

2. Experimental part of the investigation

Initially, heat treatment/improvement of the microstructure of the samples was performed [2,9]. In Table 1 the heat treatment conditions are given.

Table 1. *Heat treatment / tempered.*

Specimen size	**Heat treatment**
Cylindrical shape Ø 35 mm; length 20 cm	Tempering: Austenitization 20 min, 860 °C in N_2. Rapid cooling in oil, tempered 2h in 630 °C

Then, 12 samples with a thickness of 10mm were cut, which were marked and then their surface was processed by grinding with a grain size of 320 *μm*. Table 2 gives the nitriding parameters temperature / time and the corresponding sample numbers or sample descriptions.

Table 2. *Sample overview.*

t (°C)	**Nitriding time (h)**	**Sample marking**
510	16	31-1
	36	31-3
	64	31-5
	100	31-7
550	9	31-9
	16	31-11
	36	31-13
	64	31-15
590	4	31-17
	9	31-19
	16	31-21
	36	31-23

The nitriding time is chosen in such a way that comparable layers are obtained at different temperatures. Nitriding is carried out in an ammonia atmosphere with air addition. The nitriding potential (nitriding number) is such that it guarantees the formation of an ε-layer of nitriding (variant ε). The process parameters are shown in Table 3, [10].

Table 3. *Process parameters.*

t (°C)	**The nitriding potential**
510	3,4
550	3
590	2,82

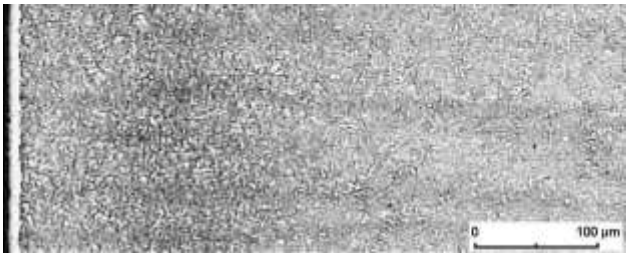
For metallographic investigation, cubes with edge lengths of approximately 10 mm will be taken from the nitride samples. The samples will first be galvanically nickel-plated. Then they will be housed in a plastic mass. Grinding and polishing are carried out in several steps, table 4.

Table 4. *Metallographic parameters.*

Preparatory steps	Parameters			
	Grain size (μm)	Time (min)	Force (N)	Speed (rot./min)
Grinding	220	2	150	300
	500	2	150	300
	800	2	150	300
Polishing	6	5	150	150
	3	3	150	150
	1	1,5	150	150

The polishing cloth for grain size 6 μm was Loch, for 3 μm it was LanW and for 1 μm it was PAN.
After polishing, the samples were etched in 2% nitric acid. For optimal contrast, the etching time was varied.

2. Results and discussions



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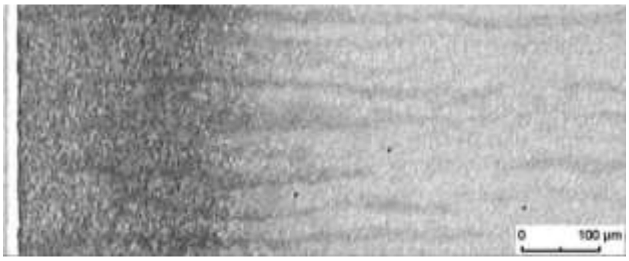

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Figure 2*. Samples with the same nitriding time, t=16h, but with different temperatures (see table 2).*

The microstructure was analyzed with a Neophot 30 optical microscope from Carl Zeiss Jena. With the help of a CCD camera, photographs of the peripheral layer were taken, and the thicknesses of the compound and the precipitate layer were determined [11,12].

For 12 samples, photographs of the polished surface were taken and, at the same time, with the help of additional microscope equipment, the nitride layers were determined. In the following figure we have presented three examples of photographs of different samples (three samples with the same nitriding time, t=16h, but with different temperatures)

Based on the results of the microscopic investigation, we found the thickness of the diffusion layers for all samples [10].

Table 5. *Thickness of diffusion zone for all samples.*

Temperature (°C)	Nitriding time (h)	Thickness of the diffusion zone (μm)
510	16	245
	36	351
	64	405
	100	619
550	9	245
	16	319
	36	504
	64	693
590	4	239
	9	340
	16	465
	36	654

The enrichment of the steel surface with nitrogen is a diffusion process, which is described by the Fick's differential equation [12,13]

$$\frac{\partial c}{\partial t} = D\frac{\partial^2 c}{\partial^2 t} \qquad (1)$$

from which for the diffusion depth of nitrogen atoms we get,

$$x^2 = 2kDt \qquad (2)$$

Or

$$x = \sqrt{2kDt} = C \cdot \sqrt{t} \qquad (3)$$

Based on expression 3, the results obtained by metallography (Table 5) are presented in the following graph (Figure 3.

The dependence of the diffusion coefficient on temperature is known,

$$D = D_0 e^{-\frac{Q}{RT}} \qquad (4)$$

From where for the depth of the division we can get in the following form,

$$x = \sqrt{2kD_0 e^{-\frac{Q}{RT}} t} = C(T)\sqrt{t} \qquad (5)$$

where

$$C = \sqrt{2kD_0 e^{-\frac{Q}{RT}}} = K^{`} e^{-\frac{Q}{2RT}} \qquad (6)$$

Or

$$\ln C = \ln K^{'} + \left(-\frac{Q}{2RT}\right) \qquad (7)$$

from this equation where do we build Arrhenius plot, which expresses the dependence of lnC on 1/T (Figure 4), [3].

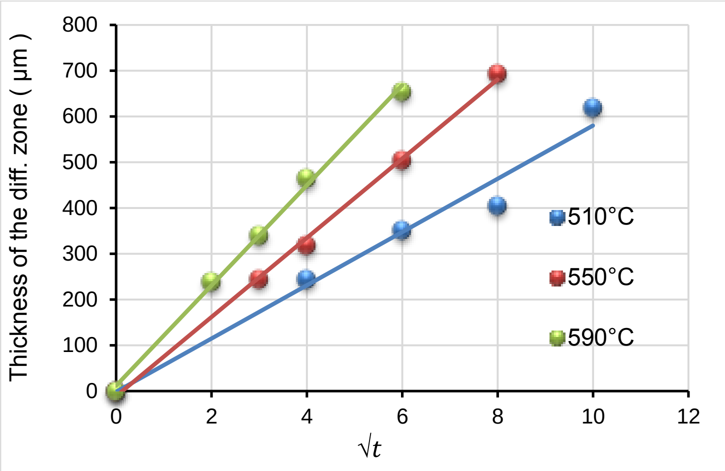

Figure 3. *Graph* $x = C \cdot \sqrt{t}$ *for all samples.*

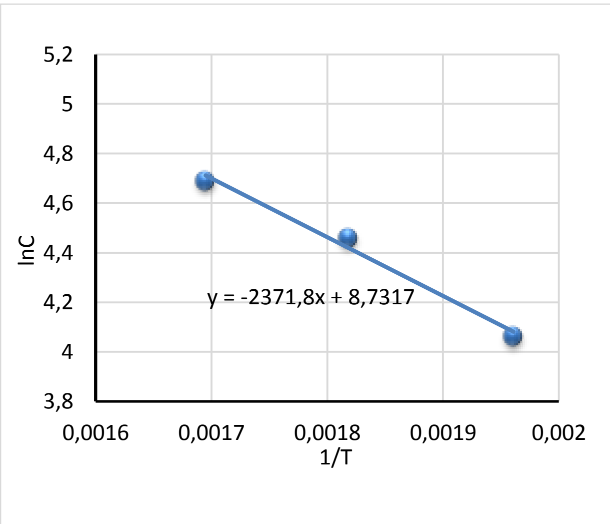

Figure 4. *Graph* $lnC = f\left(\frac{1}{T}\right)$.

With simple calculations, from graph of Figure 4 we can find the diffusion depth (expression 5),

$$x_N^T = 39371{,}88 \cdot e^{-\frac{2371{,}8}{T}} \sqrt{t} \qquad (8)$$

3. Conclusions

- The nitriding process is the diffusion process
- The layer enriched with nitrogen is called the
- diffusion layer
- The diffusion layer can be precisely examined with an optical microscope
- From the results of optical microscopy, the growth equation of the diffusion layer can be found

Author Statements:

- **Ethical approval:** The conducted research is not related to either human or animal use.
- **Conflict of interest:** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper
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- **Data availability statement:** The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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