Experimental Study of the Nitriding Layer by Steel 17CrMoV10

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Abstract: The aim of this research, is the experimental study of the nitriding layer during the process of nitriding in gases in Steel 17CrMoV10. It is well known that the process of nitriding is a thermal-chemical process that occurs at temperatures 500-600°C, where the enrichment of the peripheral surface with nitrogen is achieved. This surface is also, called a nitriding layer or diffusions layer. The aim of the process of gas-nitriding is the improvement of the properties of the material, such as, increase of the mechanical hardness and corrosion resistance, increase of resistance under thermal activities and resistance against consumption, etc. Final results of the process depend on the parameters that define the process: Temperature, duration of the process, concentration of the alloy elements in steel, etc. Depending on these parameters, by experimental methods-metallographic, microhardness measurements and microsond-we have studied the diffusions layer.

Key words: Nitriding layer, mechanical hardness, concentration of the alloy elements, depth of nitriding concentration of nitrogen, diffusions, depth redistribution of carbon

INTRODUCTION

By nitriding process, the change of spare parts (details) of various machineries is caused. Change of the properties of the details can be explained as change of the state of the matrix and of the peripheral layer, as well as a change of the macro and microgeometry of the surface (Spies *et al.*, 2004; Syla *et al.*, 2003). The surface is mainly composed from the layer of the iron nitrides, called the Compound Layer (CL). Following the compound layer, the Precipitation Layer (PL) from the particular nitrides (CrN, AlN, TiV, VN) of alloy elements, is formed. Compound Layer (CL) is discernible clearly from other parts of the nitrided layer, as well as from the matrix of the material. It consists mainly of 2 iron nitrides, which have different crystallographic structures. Both nitrides $\varepsilon\text{-Fe}_2 N_{(1-x)}$ and $\gamma\text{-Fe}_4 N$, extend in the interior of the compound layer, side by side, depending on the gradient of the concentration of nitrogen. This layer, during the observation by optical microscope, is clearly discernible as a bright layer and almost without a structure, in spite of the precipitation layer. Precipitation layer continues behind the compound layer and is developed further in depth of the surface. It (PL) consists mainly of the compound of the diffusing element (nitrogen) and the alloy elements of steel (Spies *et al.*, 2004). In Fig. 1, the
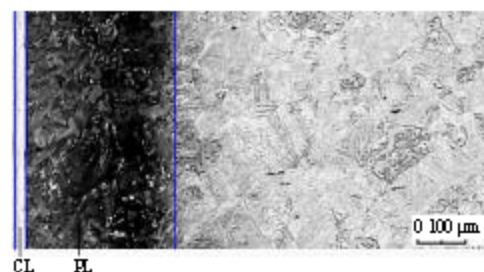

Fig. 1: CL and PL for the sample 17-19

photos of schliff are shown, where the Compound Layer (CL) can be seen clearly, followed by the Precipitation Layer (PL).

MATERIALS AND METHODS

State of samples: Chemical analysis of steel 17CrMoV10 is determined by the Funkenemision JY-132 F spectrometer, Table 1. Microstructure of the original material is re-established by thermal treatment. In the Table 2, the conditions of this treatment are given.

Afterwards, 12 samples of the width 10 mm are cut, labelled and their surfaces processed by schliffing with granular size of 320. In Table 3, the parameters of nitriding given: temperature/time and respective labels of samples.

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Table 1: Chemical composition of steel

Participation of elements (%)

C	Cr	Mn	V	Mo	Si	S	P	Al
0.16	2.41	0.40	0.14	0.26	0.34	0.009	0.024	0.023

Table 2: Thermal treatment/improvement

Size of the sample	Thermal treatment
Cylindrical form Ø 70 mm, length 23 cm	Tempering: Austenization 20 min, 960°C in N_2; Fast cooling in oil; Restoration: 2 h/630°C

Table 3: Sample pattern

Temperature (°C)	Time (h)	Sample label
510	16	17-1
	36	17-3
	64	17-5
	100	17-7
550	9	17-9
	16	17-11
	36	17-13
	64	17-15
590	4	17-17
	9	17-19
	16	17-21
	36	17-23

The time of nitriding is chosen so that for different temperatures, comparable layers could be seen (Syla *et al.*, 2003). For each temperature, 4 different times are chosen. In fact, with increasing temperature, the duration of the process is decreased, providing a comparable formation of layers. Nitriding process is undertaken in an ammoniac atmosphere doped with air.

Methods of investigation

Metallographic: For metallographic investigation, from nitrided samples the cubes with side length about 10 mm, are taken. The samples are first nickelled galvanically. Then, they are lodged in a plastic mass. After the schliffing and polishing, the corrodation in nitric acid 2% is carried out. For an optimal contrast, time of corrodation varies (Schumann and Oettel, 2005). Microstructure is analyzed with optical microscope Neophot 30 from Carl Zeiss Jena. With a CCD camera the photos of the peripheral layer are taken and the width of the compound layer and precipitation layer is determined.

Hardness measurement: With an automatic micro-hardnessmeter PCE from LECO, measurement for all samples is carried out. A small load of HV0, 1 on the surface of polished schliff is used, in a certain distance between the points where a pressure of 50 μm is exerted. Number of points is between 15 and 30 (depending on the depth of diffusions of nitrogen, which tells about the conditions under, which the process of nitriding is carried out). Then, measurement of the hardness for 5 points in the matrix zone in the depth 3 mm in nonnitrided zone, is carried out. Each curve is determined from these quantities:

- Peripheral hardness RH
- Matrix hardness KH, i.e. hardness of the nonnitrided zone of the sample
- Increase of hardness ΔH = RH-KH
- Depth of nitriding N_{ht} following the procedure of the hardnessing

Microanalysis with electron rays-microsond: For the investigation with microsond the samples must be lodged in tin and eventually schliffed and polished. The profile of the concentration of nitrogen and carbon is found with microsond JXA 8900 RL (Jeol). The distance between 2 measuring points is 5 μm.

RESULTS AND DISCUSSION

Metallography-optical microscopy: An example of the microscopic investigation is shown in Fig. 1. The peripheral zone is shown magnified, which gives a clear view of microstructure. On the left the compound layer can be seen and on its left the zone of pore is observed clearly, which is caused due to the recombination of nitrogen's atoms. The width of the zone of pores, Fig. 2, is roughly 30-50% of the width of the compound layer (Syla *et al.*, 2003; Langenhan and Spies, 1992). The precipitation layer follows the compound layer. With the aid of optical microscope, the width of both layers has been determined, Table 4. From the results, it is clearly seen that dependence exists between the parameters of nitriding and the width of layers.

Curves of hardness: For all the samples, the curves of hardness are drawn, as a result of the measurement of the hardness as a function of the depth. Figure 3 represents an example. In order to interpret the results more precisely, the values of hardness are calculated in the periphery and matrix, increase of hardness, as well as the depth of nitriding N_{ht} according to the procedure of hardnessing, Table 5. 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. Curves of hardness differ from each other in absolute value and in depth. It is proved that by increasing the temperature for a given time, the hardness of the periphery decreases, Fig. 4. On the other hand, for a given temperature the depth of nitriding is increased with increasing the duration of the nitriding process. This can be explained by the quadratic dependence of the depth on time (diffusions law). For all the curves of hardness, intensity eventually decreases up to the value of the hardness of the matrix.
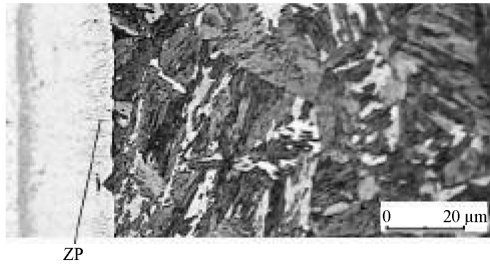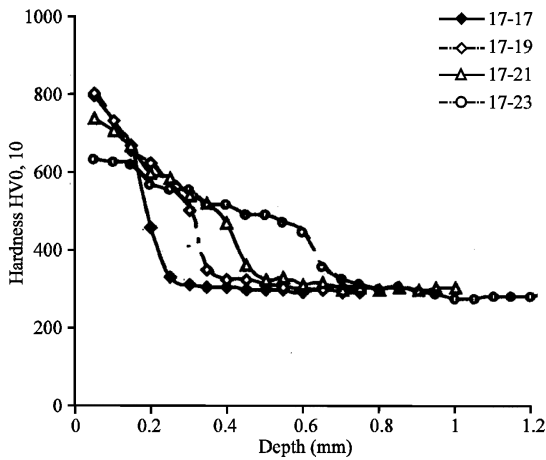
Fig. 2: Zone of Pores (ZP) for the sample 17-23

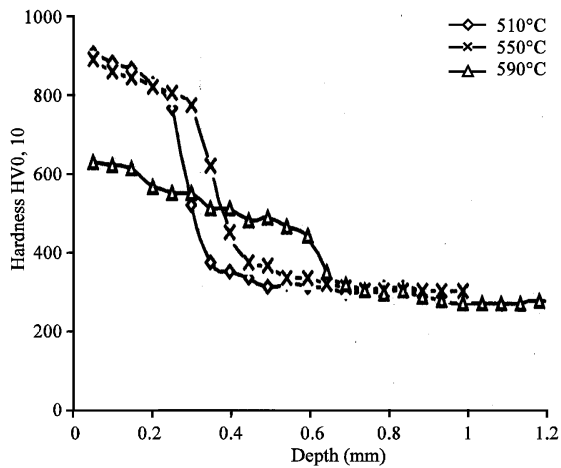Fig. 3: The curves of hardness for 590°C

Fig. 4: Effect of the temperature on the curves of the hardness for the duration 36 h

Profile of the concentration of nitrogen: In Fig. 5, the profile of concentration of nitrogen and carbon for the sample 17-7 is shown and in Table 6 maximal concentrations of nitrogen in CL and PL are given, as well as the depth of diffusions of the nitrogen x_{Nn} in μm for all conditions of nitriding (all the samples).
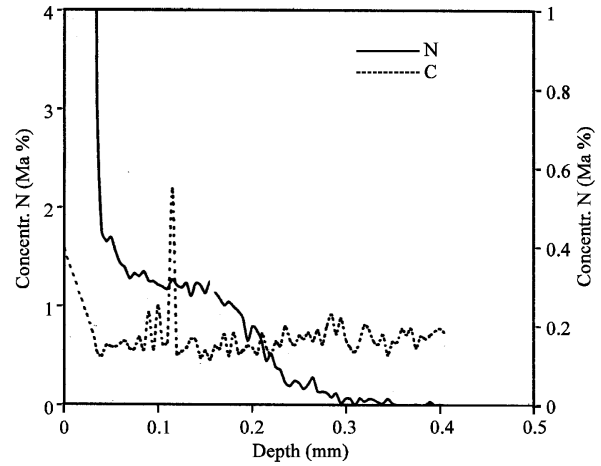

Fig. 5: Profile of concentration of N and C for the sample 17-7

Table 4: The width of CL and PL for steel 17CrMoV10

Sample	Width (μm)	
	CL	PL
17-1	9.80	250
17-3	12.9	288
17-5	16.0	375
17-7	21.3	480
17-9	14.0	193
17-11	17.2	273
17-13	22.1	448
17-15	22.8	580
17-17	17.3	213
17-19	23.6	319
17-21	28.0	419
17-23	31.1	600

Table 5: Results of microhardness

Sample	Measured quantities			
	RH	KH	ΔH	N_{ht} (μm)
17-1	893	322	571	240
17-3	906	329	577	340
17-5	890	316	574	460
17-7	869	327	542	550
17-9	899	300	599	260
17-11	840	300	540	360
17-13	823	304	519	540
17-15	775	300	475	640
17-17	795	311	483	235
17-19	801	305	496	350
17-21	741	302	439	460
17-23	629	279	350	680

Concentration of nitrogen in the precipitation layer extends in the zone from 7.92-12.72% in weight. Maximal amount of nitrogen in the precipitation layer, in the boundary of the compound layer, depends mainly on the composition of the building elements of nitrides. Distance from the periphery of the layer to the location where the nitrogen is spread, is defined as the penetration depth of the nitrogen x_N. From results given in Table 6, it is seen that dependence exists between the duration time of nitriding and the diffusions depth of nitrogen. From the

Table 6: Results of microsond

| Sample | N (Ma %) | | x_N (μm) |
	PL	CL	
17-1	8.66	1.62	280
17-3	9.94	1.36	390
17-5	8.68	1.58	500
17-7	9.46	1.58	590
17-9	12.72	1.46	290
17-11	11.47	1.79	380
17-13	10.59	1.68	520
17-15	10.10	1.65	630
17-17	8.22	1.46	280
17-19	7.97	1.57	370
17-21	8.18	1.58	480
17-23	7.92	1.71	690

distribution of carbon in the precipitation layer 2 very important conclusions can be drawn. During this process, not only within the precipitation layer, but also outside it (on both sides), we will have redistribution of carbon. Within PL we have greater oscillations of the concentrations of nitrogen rather than outside it. This effect is obvious for all samples and it proves that after the nitriding process a nonhomogeneous redistribution of carbon occurs. Besides diffusions in the interior of the sample, carbon will diffuse also in the direction of surface of the sample (decarbonization process) and as a result we have an increase of concentration at the boundary with the compound layer (Spies and Bergner, 1992).

CONCLUSION

According to the experimental results, we can conclude that: The development of CL and PL is strongly influenced on temperature of nitriding for other same conditions. These developments are controlled by the diffusions of nitrogen in depth of the surface of steel. Precipitation layer develops in depth of about (12.3-25.2) times greater than that of compound layer. With increasing temperature or duration (Table 5), maximal hardness of the periphery is decreased. This occurs for 2 reasons: The first reason is that by increasing the temperature or duration, the porosity is increased (Syla *et al.*, 2003), whereas by increasing the porosity the hardness is decreased. The second reason is that by increasing the duration interval of the gasnitriding, the precipitates of the chrome nitrides are increased, which influences the decrease of hardness. Analysis by microsond, based on the measurements of hardness, determinations with optical microscopy and theoretical calculations, has shown the possibility of the linear approximation of the depth of nitriding as a function of the time duration of the process in power (1/2) that is: $N_{ht} \sim t^{1/2}$.

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