

Influence of alloying elements on gas nitriding process of high-stressed machine parts of weapons

Z. Pokorný*, D. Dobrocký, J. Kadlec, Z. Studený

¹*Department of Mechanical Engineering, University of Defence in Brno, Brno, Czech Republic*

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Abstract

The chemical composition of the steel is one of the primary characteristics that influence the mechanical properties of high-stressed machine parts such as barrels of small arms weapon. Heat treatment is the most important technology to reach requested mechanical properties. By a suitable combination of mechanical and tribological properties, it is possible to improve the surface of highly stressed parts as to improve the wear resistance of barrel surface. Thus, surface technology is next step for obtaining new properties as tribological properties or preferable mechanical properties. Due to obtain suitable microstructure and wear resistance the samples of C35, 34Cr4, 37Cr4 and 42CrMo4 were heat-treated with following chemical heat treatment by gas nitriding process, for 6 hours. This paper deals with the influence of alloying elements of structural steels on surface microhardness and depth of diffusion layer. The gas nitriding process caused the creation of a compound layer on the surface of the steel. This layer leads to significant improvement in wear resistance. The concentration of alloying elements was analysed by OES methods. The technology of nitriding was applied to annealed, tempered and quenched steels. After chemical heat treatment, new surface morphology was created. The surface layers of the samples were analysed by microhardness method; surface morphology was evaluated by SEM method. Experimental part concerns structural steels with a concentration of alloying elements to 1 wt.%. In experimental part, the influence of alloying elements on the diffusion process in Fe-C system and the microhardness of the surface was proved.

Key words: gas nitriding, microhardness, chemical composition, diffusion, barrel

1. Introduction

High-stressed machine parts, such as barrels, are usually made of steels, while wear and erosion have been a limiting factor in barrels performance since their invention [1, 2]. The most frequently used materials for high-stressed parts as barrels or gears are CrMoNi or CrMoV steels [1, 3–5]. Currently, modified chemical heat treatment processes, such as gas nitriding, are widely used to enhance the properties of barrels and gears. Generally, the term gas or plasma nitriding also means a surface treatment of the material, when the use of the gas or plasma process cause gradual saturation of the surface of components by nitrogen comprised in the gas under predetermined temperatures and pressures [6, 7]. Nitrides occur inside the material as a result of the nitriding process. How-

ever, there are some exceptions regarding Cr steels. Due to the diffusion process during nitriding, there is an absence of chromium nitrides and carbides in the surface of the nitride layer. The absence of chromium in solid solution by precipitation of chromium nitrides and carbides leads to significant decrease of corrosion resistance [8–10]. The process aims to achieve an enhanced surface hardness, better wear resistance, reduced friction coefficient, increased fatigue limit and corrosion resistance. The nitrides of iron in the diffusion layer caused a low increase in surface layer microhardness. The alloying elements as molybdenum, vanadium, aluminium or chromium caused improvement of mechanical properties. During the gas nitriding process, mostly two different nitride layers are formed. On the surface of the material, the compound layer consists of ϵ -Fe₂₋₃N, and γ '-Fe₄N phase should

*Corresponding author: tel.: +420 973 442 604; e-mail address: zdenek.pokorny@unob.cz

Table 1. The matrix of the chemical composition of steels

Steel	Sample	Chemical composition (wt.%)						
		C	Mn	Si	Cr	Ni	Mo	Al
C35	A1	0.36	0.68	0.34	0.05	0.04	0.01	0.003
34Cr4	A2	0.35	0.70	0.35	1.06	0.05	0.01	0.035
37Cr4	A3	0.39	0.66	0.31	1.00	0.19	0.01	0.043
42CrMo4	A4	0.39	0.81	0.31	1.09	0.06	0.19	0.029

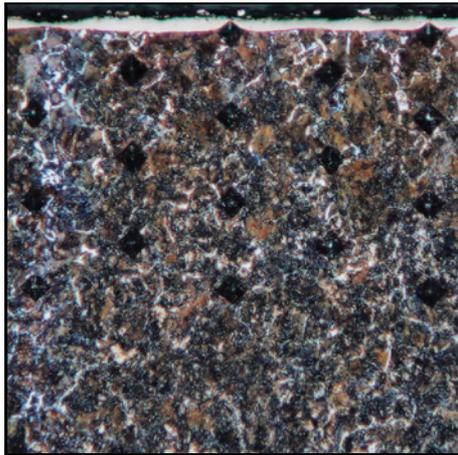


Fig. 1. Annealed structure characterization of A1 sample by optical microscopy, nitrided layer after gas nitriding with visible indentation test, magnification 1000 \times .

be formed [11, 12]. This type of layer is very hard and brittle with good friction and anticorrosion properties [13, 14]. Thickness and microhardness of γ' -Fe₄N often depend on quantity and quality of nitride alloying elements [15, 16]. The composition of diffusion nitride layers can be effectively influenced by the chemical composition of the nitriding atmosphere, esp. ratio of nitrogen, hydrogen, and pressure in case of plasma environments [17, 18].

The presented paper aims to study the effect of chemical composition and microstructure on the mechanical properties of steel after gas nitriding process. The concentration of nitrogen and carbon in crystal lattice influences the strength of steels due to nitrides and carbides formation. The concentration of alloying elements influences the diffusion process during chemical heat treatment (diffusion procedures). The optimal concentration of alloying elements influences not only mechanical properties, such as strength, hardness, ductility or microhardness, but it also leads to reduced establishing of limit states. By applications of available gas nitriding technology, it is possible to reach significant enhancement of surface microhardness of raw material (core) in annealed, tempered or quenched structural state. The increase of surface microhardness significantly depends not only on initial microhardness of core but also on chemical compo-

sition, especially on the concentration of alloying elements as chromium, molybdenum, nickel or vanadium [16, 19].

The influence of the concentration of alloying elements on diffusion parameters during gas nitriding process was studied experimentally.

The results of the comparison of microstructure and properties of samples with different types of heat treatment are discussed in this paper.

2. Experimental work

The influence of alloying elements on diffusion process was experimentally studied regarding chemical composition, microstructure, microhardness and surface morphology.

2.1. Materials

The materials used for this paper are steels C35, 34Cr4, 37Cr4, 42CrMo4. The raw material was transformed by vacuum melting and forging into the bars. Three series of the sample were prepared from forged steels. Each series takes 20 samples (five from each steel). The samples of C35, 34Cr4, 37Cr4, 42CrMo4 steels, marked from A1 to A4 were made from cylindrical bars in untreated ferrite-pearlite structure state (Fig. 1).

2.2. Heat treatment

The first series of samples was annealed for 1 h at 850 $^{\circ}$ C, the second series was quenched for 1 h at 650 $^{\circ}$ C, and the third series was tempered for 1 h at 650 $^{\circ}$ C.

The matrix with chronologically ordered steels on the base of chemical composition was prepared (Table 1). The chemical composition of all samples was performed by the OES method on advanced optical emission spectrometer for metal analysis Q4 Tasman. The results of analysis obtained by the Fe130 method are displayed in Table 1. Table 1 shows the details of the chemical composition of all samples. Each sample in Table 1 contents presents an alloy element with defined concentration.

Table 2. Surface hardness and microhardness after different types of heat and chemical heat treatment processes

Heat treatment	Quenching	Tempering	Quenching + nitriding	Tempering + nitriding
Microhardness	HV 0.5		HV 0.05	
A1	338.6 ± 17.7	226.6 ± 5.9	450.7 ± 13.1	380.4 ± 12.7
A2	660.6 ± 29.4	284.7 ± 10.8	820.8 ± 16.7	742.3 ± 20.2
A3	675.6 ± 22.8	280.7 ± 12.5	770.3 ± 21.1	721.5 ± 18.9
A4	687.4 ± 17.8	303.7 ± 12.5	870.8 ± 23.2	780.7 ± 21.5

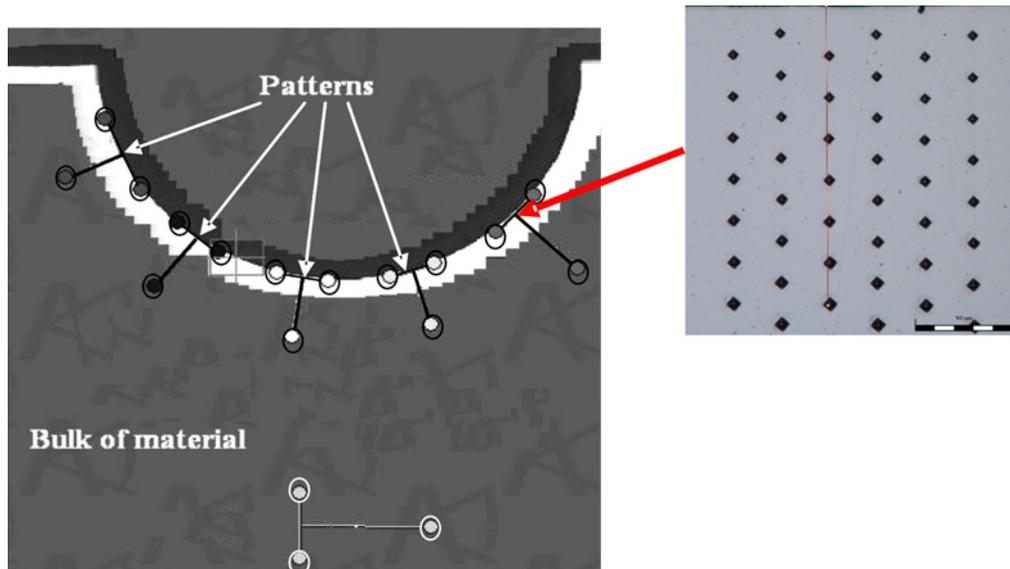


Fig. 2. Image of the measured sample; location (position) of measurement patterns on the top of the barrel; the pattern of the bulk of material below.

The different types of heat treatment caused a change of the redistribution of alloying elements in the crystal lattice, change of mechanical properties and the diffusivity. Alloying elements have a remarkable influence on the diffusivity of interstitial elements of nitrogen and carbon during the chemical heat treatment process.

2.3. Chemical heat treatment

After heat treatment, the Vickers microhardness of samples was determined, and the results are given in Table 2.

The surfaces of samples were wet ground using SiC grinding papers to achieve a smooth surface. Before gas nitriding process, all samples were cleaned in ethanol using an ultrasonic cleaner for 20 min.

Heat treatment was followed by chemical heat treatment in gas. Gas nitriding was provided by using NITREX appliance for 6 h at 490 °C. In this experiment, a two-stage process was used to reach requested depth of layer.

The compound layer and diffusion layer were created in all samples (Fig. 1). Surface structural modifications were investigated on the cross-section using

3D optical microscope Olympus DSX 500i with the magnification 1000×.

Surface microhardness after gas nitriding was measured by using automatic microhardness tester LM 247AT Leco equipped with AMH43 software with the 0.05 kgf test load and 15 s holding duration. The indentation tests were performed in cross-section (see Fig. 2). Each microhardness value was determined as an average value at least from three measurements. The depth of the hardened case was typically defined as the depth where the hardness reaches 50 HV above core hardness by DIN 50190.

The diffusion layer was created in all samples what is visible in Figs. 3–6.

The morphology of nitrided layers after gas nitriding was observed on the cross-section by SEM method on scanning electron microscope HITACHI TABLETOP 3300. The porosity of surface compound layer was analysed from the surface and cross-section (Figs. 7a–d).

3. Results and discussion

Samples from A1 to A4 were nitrided in the gas

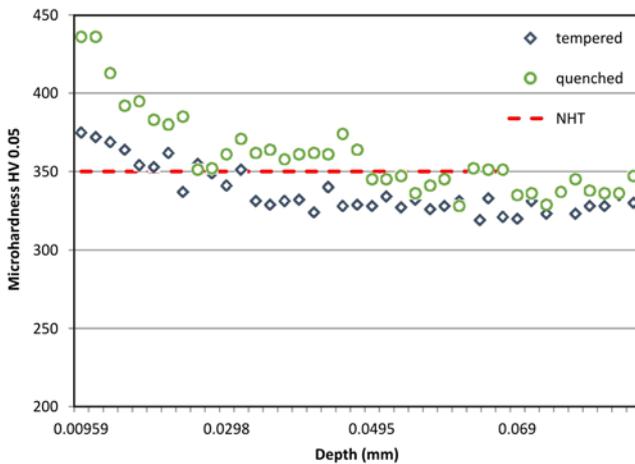


Fig. 3. Microhardness vs. depth of diffusion layer for a cross-section of sample A1.

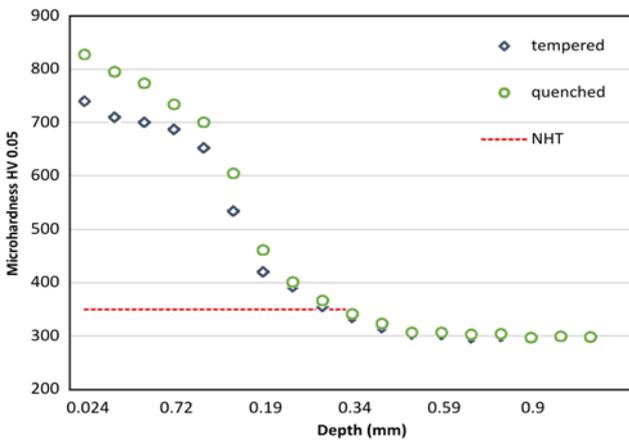


Fig. 4. Microhardness vs. depth of diffusion layer for a cross-section of sample A2.

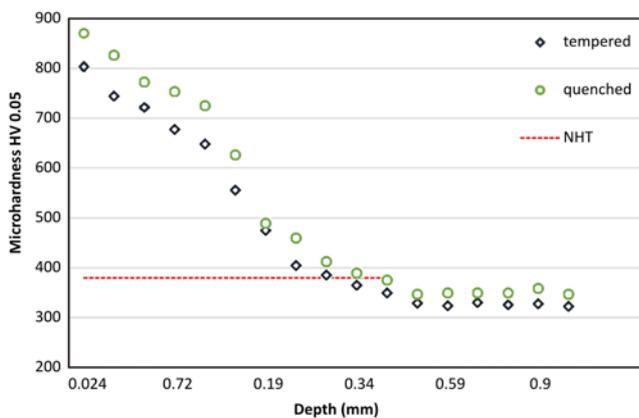


Fig. 5. Microhardness vs. depth of diffusion layer for a cross-section of sample A4.

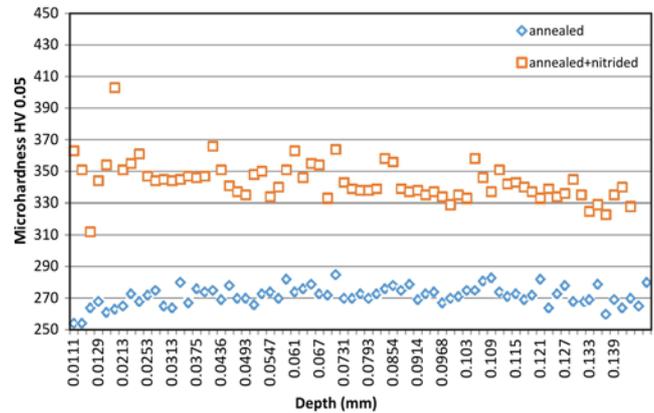


Fig. 6. Comparison of microhardness vs. depth of diffusion layer for a cross-section of sample A1 after annealing.

atmosphere for 6 h at 490 °C, the heat treatment temperature of samples was 650 °C. The surface properties were changed. The results and discussion are as follows:

The diffusion layer formed in sample A1 after gas nitriding process demonstrated a very low increase of surface microhardness by comparison with an initial microhardness of core after heat treatment (Figs. 3, 6).

Experiments showed a very low influence of the microstructures after annealing, quenching and tempering of A1 samples on increase of surface microhardness and depth of diffusion layer after nitriding (see Figs. 3–6). The depths of the diffusion layer in ferrite-pearlite, martensite and sorbite structures state were measured to 0.05 mm. This phenomenon is caused by the low content of nitride alloying elements in case of sample A1. The higher increase of microhardness values was identified by microhardness method in quenched A1 samples.

Generally, the highest values of surface microhardness of a diffusion layer after gas nitriding were measured in all samples after the quenching process. This phenomenon is caused by redistribution of interstitial and alloying elements in the crystal lattice. It is evident that it is more suitable to provide gas nitriding after quenching than after tempering treatment. It allows obtaining harder surface layer and deeper case depth of nitriding. This phenomenon was confirmed and verified for all tested samples (Figs. 3–5).

Experiments proved the considerable influence of chrome concentration on the surface microhardness after gas nitriding process. This phenomenon was confirmed by the measurement of surface microhardness of quenched sample A2 (Table 1, Fig. 8). Initial surface microhardness was increased due to the presence of a higher concentration of chrome in A2 sample which is related to the formation of nitride and carbide elements. Consequently, the concentration of chrome in-

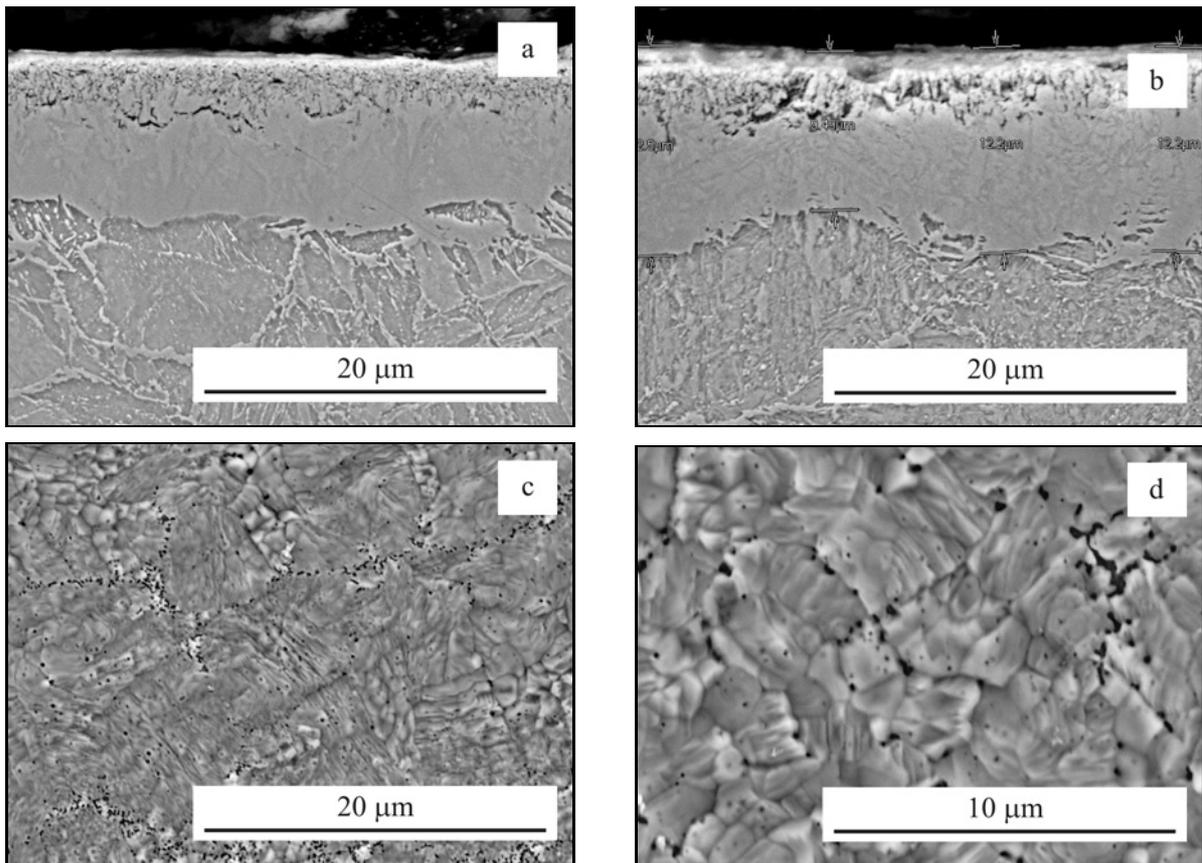


Fig. 7. Porosity in compound layer in a cross-section: sample A1 after gas nitriding, magnification 5000× (a); sample A4 after gas nitriding, magnification 5000× (b); morphology of compound layer, porosity on boundary of grains, sample A1 after gas nitriding, magnification 5000× (c); detail of compound layer morphology, porosity on surface, sample A4 after gas nitriding, magnification 10000× (d).

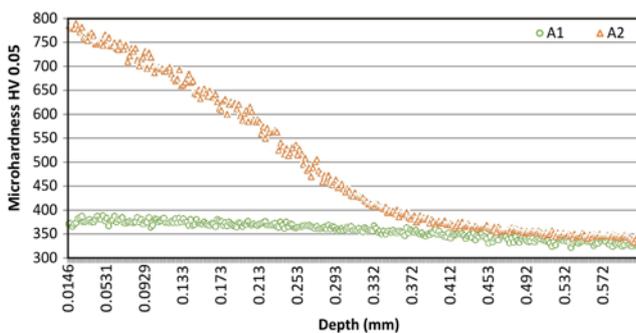


Fig. 8. Dependence of microhardness on the depth of diffusion layer sample A1 and A2 after gas nitriding process, samples tempered; the influence of chromium concentration on the hardness and depth of diffusion layer.

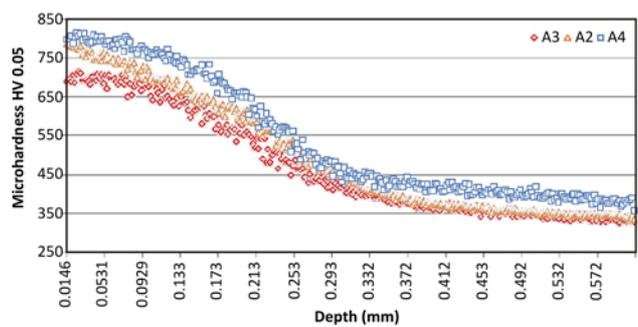


Fig. 9. Dependence of microhardness on the depth of diffusion layer sample A2, A3 and A4 after gas nitriding process; samples tempered, the influence of chromium and nickel concentration to the hardness and depth of diffusion layer.

creased the thickness (depth) and microhardness of the diffusion layer. The concentration of chrome positively affects the diffusivity of nitriding process (compare Fig. 8).

The increase in surface microhardness and thickness of the diffusion layer of A3 sample after nitriding process was achieved due to higher chromium concen-

tration than in case A2 sample. The presence of nickel caused the decrease of surface hardness which is visible in Fig. 9. Generally, nickel has an inauspicious impact on the creation of carbides and nitrides and negatively influences the diffusion process in the crystal lattice.

Another possibility of increasing the surface microhardness and thickness of diffusion layer and im-

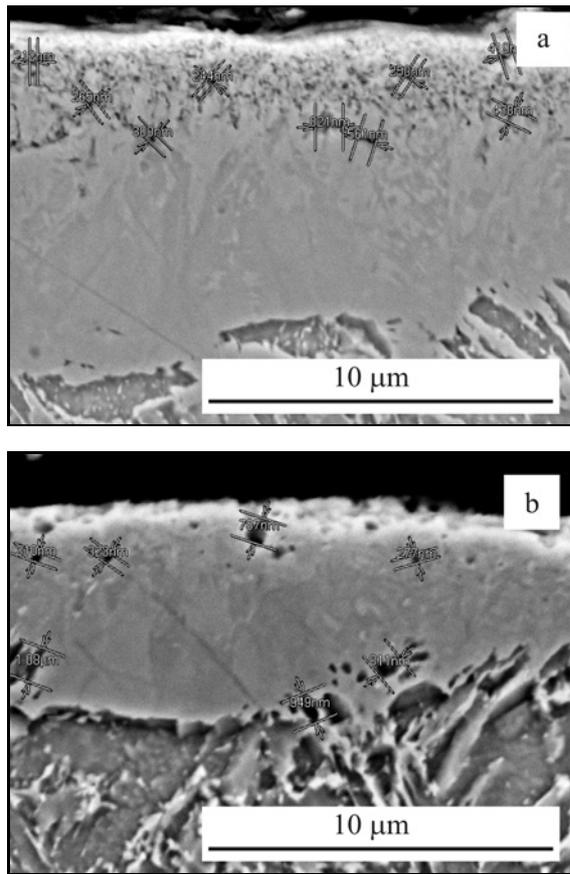


Fig. 10. Compound layer after gas nitriding, porosity in the surface layer (a); reduced compound layer after plasma nitriding, reduced porosity (b).

provement of mechanical properties is to optimize the concentration of molybdenum. In fact, it is visible in Fig. 9. In case of sample A4 with a concentration of molybdenum about 0.2 wt.%, higher values of surface microhardness of diffusion layer were reached. Sample A4 is represented by the blue curve in Fig. 9. All mentioned measurements of surface microhardness of diffusion layer were performed on cross-sections. The evaluation of compound layer morphology was studied from the cross-section and surface as well.

In compound layers, also known as white layers,

the pores were formed. The microstructure of steel and the porosity of compound layer in cross-section and surface view is documented in Fig. 7.

The pores were massively formed in compound layers after gaseous technologies. The formation of pores is possible to reduce by plasma technologies (compare Figs. 10a,b). The porosity of surface is in many applications very positive phenomenon due to storage of nanoparticles of lubricants. Typical marks of surface morphology after use of gaseous technologies are given in Figs. 7a–d.

The occurrence of pores is irregular; some pores form cavities in different depths. This phenomenon decreases the wear resistance of the surface. The most significant disadvantage of compound layer creation is their connectivity with changing dimensions of components. Due to changing dimensions, many parts must be finely ground to achieve the initial dimension accuracy [19, 20].

4. Conclusions

The influence of alloying elements on diffusion technologies is well-known. It is necessary to know the interdependence of each other alloying elements in Fe-C system on the diffusion process. The experiments proved the influence of nickel, chromium, and molybdenum on the case depth and surface microhardness of the diffusion layer. An important task was to obtain an optimized microstructure before application of diffusion process. Optimized microstructure ensures to obtain maximum surface hardness after nitriding. During experiments, higher case depths of nitriding on quenched steels were reached. It is necessary to perform the chemical heat treatment immediately after quenching. It was proven that chemical heat treatment process, esp. due to time and temperature, causes the decrease of hardness after quenching process to the lower values after tempering (Figs. 3–5). However, remember that the surface microhardness of quenched steel after nitriding process is more than about 30% harder than in case of nitriding of tempered steel. By nitriding in gaseous atmospheres, there is a possibility to increase the surface hardness

Table 3. Changing of roughness parameters R_a after gas nitriding

Sample	R_a (μm)	
	Initial state (before gas nitriding)	Final state (after gas nitriding)
A2	0.064 ± 0.003	0.178 ± 0.006
A1	0.086 ± 0.005	0.116 ± 0.002
A3	0.064 ± 0.002	0.158 ± 0.004
A4	0.072 ± 0.005	0.125 ± 0.003

about 250 HV0.05 independent on chemical composition.

The influence of alloying elements on the formation of the compound layer was not proved. The thickness of a compound layer formed on all surfaces of samples A1 to A4 was approx. the same value, from 3.01 to 4.89 μm .

It has been said that the compound layer decreases the quality of surface esp. roughness [21]. Of course, the main disadvantage of the created compound layer is still the degradation of surface topography, especially the increase in roughness which is given in Table 3. Another possibility is to optimize technology for reducing the compound layer creation due to reducing the risk of dimensional accuracy changing.

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