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## Full length article

# The effects of laser surface modification on the microstructure and properties of gas-nitrided 42CrMo4 steel



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## **ABSTRACT**

Gas nitriding, together with gas carburizing and gas carbonitriding, was the most commonly used thermochemical treatment, resulting in many advantageous properties: high hardness, enhanced corrosion resistance, considerably improved wear resistance and fatigue strength. However, an unfavorable increase in the thickness of compound layer ( $\epsilon$ + $\gamma'$ ) close to the surface was observed after conventional gas nitriding. This was the reason for undesirable embrittlement and flaking. Therefore, a controlled gas nitriding was developed, reducing the thickness of compound layer. In this study, laser modification with or without re-melting was carried out after the controlled gas nitriding in order to change microstructure and to improve wear resistance. The effects of laser beam power on the dimensions of simple laser tracks were analyzed. It enabled to control the obtained microstructure and to select the laser processing parameters during producing the multiple tracks. Such a treatment was necessary to investigate wear resistance. Laser re-melting resulted in dissolving the majority of nitrides as well as in producing the martensitic structure in re-melted and heat-affected zones. This treatment required argon shielding in order to protect the surface against uncontrolled oxidation. Laser heat treatment without re-melting caused a modification of ε nitrides which became less porous and more compact. Simultaneously, it provided heat-affected zone with the partially martensitic structure of increased hardness below compound zone. Argon shielding was not necessary in this case because of the resistance of nitrides to oxidation during rapid heating and cooling. All the laser-modified layers, irrespective if the nitrided layer was re-melted or not, were characterized by the improved wear resistance compared to the typical gasnitrided layer.

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## 1. Introduction

Nitriding, as a well-known process of thermo-chemical treatment, was often applied in order to improve the tribological properties of machine parts and tools as well as their corrosion behavior [\[1,2\].](#page--1-0) The most commonly used nitriding technologies were as follows: controlled gas nitriding  $[1-8]$  $[1-8]$  $[1-8]$ , low-pressure gas nitriding [\[9](#page--1-0),[10\],](#page--1-0) plasma nitriding or ionic nitriding [\[11](#page--1-0)–[17\]](#page--1-0).

Gas nitriding was the process of a special significance. The typical nitrided layer consisted of a compound zone (including iron nitrides  $ε$  and  $γ'$ ) and diffusion zone (the zone of internal nitriding) with precipitates of  $\gamma'$  nitrides. Conventional gas nitriding caused an unfavorable increase in the thickness of compound layer  $(\varepsilon + \gamma')$  close to the surface. This was the reason for undesirable embrittlement and flaking. As a consequence, the working parts were exposed to accelerated destruction and wear. Therefore, the

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<http://dx.doi.org/10.1016/j.optlastec.2016.02.021> 0030-3992/& 2016 Elsevier Ltd. All rights reserved. controlled gas nitriding was intensively developed in recent years. By using such a process, the kinetics of the layer growth (especially, the growth of compound layer) was controlled by changeable value of the nitriding potential. The nitriding potential influenced the obtaining the various types of nitrided layer on a steel substrate. The layers differed in microstructure, especially, close to the surface. The microstructure could be composed of  $\varepsilon+(\varepsilon+\gamma')$ iron nitrides with a predominant percentage of  $\varepsilon$  phase. Such a layer was very porous. However, these pores could also occur in the diffusion zone, beneath the compound layer  $[8]$ . Wear resistance as well as a corrosive behavior of the layer were worsened in such a case. The second type of the layer could consist of a compact compound zone, composed of  $\varepsilon + \gamma'$  iron nitrides with a lesser percentage of ε phase. The next layer didn't contain a compound layer with  $\varepsilon$  phase and was composed of only  $\gamma'$  single phase at the surface and diffusion zone below  $\gamma'$  nitrides [\[5\].](#page--1-0) Finally, only the diffusion zone could appear in the surface layer without compound zone close to the surface. Phase composition of the nitrided layer close to the surface influenced such properties as wear resistance by friction and resistance to corrosion  $[1-8]$  $[1-8]$  $[1-8]$ . In order to improve wear resistance, the compound layer with a diminished percentage of ε phase was advisable. The tribological properties could also be improved if the compound layer didn't occur as a consequence of gas nitriding. The improved corrosion resistance required the presence of compact  $\varepsilon$  phase (with limited porosity) in compound layer. The attractiveness of controlled gas nitriding consisted primarily in repeatability of the phase composition of nitrided layers as well as in easy control of the gas process. Gas nitriding was also particularly attractive because of its relative high efficiency with low cost. During a ZeroFlow nitriding [\[6\]](#page--1-0) the control of nitriding potential was performed by temporarily stopping and reactivating NH3 feeding into the furnace. Such a nitriding process proved to be much more economical in gas consumption than others.

In order to accelerate the diffusion of nitrogen into the steel substrate, low-pressure gas nitriding was recently developed [\[9,10\]](#page--1-0). This process had a non-equilibrium nature. As a consequence, the control of nitriding potential was impossible. Lowpressure nitriding consisted of two stages: "boost stage" and diffusion stage [\[9\]](#page--1-0). The "boost" stage was carried out at the constant total pressure of 26 hPa, at the ammonia supplying flow that has been proportional to the total area of nitrided charge. The diffusion stage was carried out in a vacuum in order to separate the nitrogen reserve in nitrides from any external interactions. These two stages were alternately repeated. The suitable division into "boost" and "diffusion" stages enabled the formation of the layers with the compound zone (ε+γ′) as well as without formation of ε and γ' nitrides close to the surface  $[9]$ . Owing to a new approach to low pressure nitriding, based on artificial intelligence methods, it was possible to have a better control over the process [\[10\].](#page--1-0)

However, there were materials which could not be easy gasnitrided. Although the plasma or ionic nitriding processes were initially carried out on constructional steels [\[11,15\],](#page--1-0) their applications concerned mainly such materials as: high-chromium steels [\[12\],](#page--1-0) titanium alloys [\[13\]](#page--1-0) as well as austenitic steels [\[14,16\]](#page--1-0). These materials were characterized by a propensity to passivation. The formation of oxides close to the surface hindered the nitrogen adsorption and its diffusion into these materials. The first advantage of plasma nitriding was the ability of the ionized gas to activate these most difficult to nitride alloys, containing significant quantities of chromium or titanium and enabling chemisorption of the active nitrogen species on the surface [\[11\].](#page--1-0) During the plasma nitriding process, the energy of ions and neutrals bombarding the cathode workpiece was sufficient not only to remove the native chromium or titanium oxides, passivating the surface, but also to cause significant sputtering of the alloy. Such a situation, together with a low partial pressure of nitrogen in plasma, was the reason for the limited thickness of the compound zone. Therefore, the plasma nitriding was often called a low nitriding potential process [\[11\]](#page--1-0).

During the conventional plasma nitriding, the material to be treated was subjected to a high cathodic potential. The positive ions, generated by the glow discharge, accelerated in the cathode fall region near the cathode surface and bombarded the surface of the treated material. This resulted in some inherent disadvantages such as "edging effect" and damage caused to parts by glow discharge  $[16]$ . The above difficulties were overcome by decoupling plasma generation from the treated material using active screens [\[14,17\]](#page--1-0). Another proposed method was anodic nitriding in a plasma atmosphere [\[17\].](#page--1-0) During this process, the material was placed in an anodic potential. Such a situation caused that treated part was mostly bombarded with electrons of low energy. This way also prevented the inherent shortcomings of conventional plasma-nitrided layer [\[17\]](#page--1-0). Nitriding process was also often used as a first step of hybrid treatment preceding post-oxidizing process (in order to improve the corrosion behavior of plasma-nitrided layer) [\[18\]](#page--1-0) or PVD process (in order to deposit TiN or CrN coatings on gas-nitrided layer) [\[19\]](#page--1-0).

In recent years, laser processing was being used for a wide range of applications in order to modify the microstructure and properties of the metals and their alloys [\[20,21\]](#page--1-0). Laser technology was also applied in order to produce the nitrided layers using a process called laser nitriding [\[22](#page--1-0)–[30\]](#page--1-0). This process consisted in the irradiation of a metal surface by short laser pulses in an atmosphere containing nitrogen. Sometimes, before the laser nitriding, the irradiation chamber was usually evacuated to the pressure lower than  $10^{-3}$  Pa [\[22,23\]](#page--1-0). The obtained phase composition, especially the presence of the compound zone with iron nitrides, depended on laser beam fluence and the pressure of atmosphere [\[23\]](#page--1-0). A special interest was also oriented to materials, such as titanium alloys  $[24-29]$  $[24-29]$  $[24-29]$  or nickel alloys  $[30]$ , which were difficult to the nitriding with conventional methods.

Other applications of laser technology, regarding the nitrided layer, used a laser beam in order to modify microstructure and properties of previously nitrided or nitrocarburized layers [\[15,31](#page--1-0)– [33\]](#page--1-0). Such a laser-heat treatment (LHT) was carried out with remelting of the surface  $[31,32]$  as well as without re-melting [\[15,33\].](#page--1-0) All these papers indicated the improved wear resistance of the nitrided (or nitrocarburized) and laser heat-treated layers. LHT with re-melting resulted in decomposition of the compound zone close to the surface  $[31,32]$ . In that case, the protection against uncontrolled oxidation was very important. LHT without shielding gas could result in the formation of oxides on the treated surface. However, there were no data regarding the use of shielding gas during LHT reported in the papers [\[31,32\].](#page--1-0) XRD patterns were also not shown. In paper [\[15\]](#page--1-0), the laser quenching was applied to modify the microstructure of a plasma-nitrided layer. The authors mentioned, that the treatment was carried out without re-melting. However, the lack of ε phase, identified on the surface before laser quenching (directly after nitriding), the decreased hardness close to the surface as well as the presence of iron oxides after laser modification raised doubts regarding this thesis. Probably, the laser quenching of nitrocarburized layer [\[33\]](#page--1-0) also caused re-melting of the surface, despite the opposite declarations of authors. Therefore, the laser-quenched nitrided layers seemed to be worthy of next investigation.

In this study, the controlled gas nitriding process was followed by various laser heat treatments. LHT of the nitride layer was carried out with and without re-melting as well as with and without shielding gas in order to explain the above doubts. The influence of LHT parameters on the microstructure, phase composition, hardness profiles and wear resistance of laser-modified nitrided layer was shown.

#### 2. Experimental details

#### 2.1. Material

In the present study, 42CrMo4 steel was used as a material of the specimens. The chemical composition of this steel was shown in [Table 1.](#page--1-0) The specimens were roller-shaped with an external diameter of 14 mm and a length of 100 mm.

## 2.2. Controlled gas nitriding

Prior to nitriding, the specimens were austenitized at 860 °C (1123 K), quenched in oil and high-temperature tempered (toughened) at  $600 \degree C$  (873 K) for 2 h. Controlled gas nitriding process was carried out in an atmosphere, consisting of ammonia (NH3). Nitriding parameters were as follows: temperature 570 °C (843 K), time 4 h, a changeable nitriding potential. The

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