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New method of nitrocarburizing of X37CrMoV5-1 hot working tool steel in chemically active fluidized bed

A. Ciski [a,*], T. Babul [a], K. Lankiewicz [a], Z. Obuchowicz [a], P. Šuchmann [b]
[a] Institute of Precision Mechanics, ul. Duchnicka 3, 01-796 Warsaw, Poland
[b] COMTES FHT a.s., Dobřany, Czech Republic
* Corresponding e-mail address: konrad.lankiewicz@imp.edu.pl

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ABSTRACT

Purpose: This paper presents a new method of nitrocarburizing in chemically active powders fluidized by mechanical vibrations.

Design/methodology/approach: The research in the field of microstructure (light microscopy), hardness, wear resistance and impact strength was carried out on specimens made of X37CrMoV5-1 alloy tool steel subjected to nitrocarburizing at a temperature 560°C with thermo-chemical treatment time 4 and 8 hours.

Findings: The obtained results were related to the steel subjected to conventional heat treatment consisting of quenching and tempering. Conducted nitrocarburizing processes enabled the formation of layers with parameters similar to the layers obtained using other nitrocarburizing techniques, such as box methods in chemically active powders or in gaseous atmospheres.

Practical implications: As a result of nitrocarburizing strengthened steel had significantly higher hardness and wear resistance than the material subjected to conventional quenching and tempering.

Originality/value: Obtained diffusion layers confirmed the possibility of the practical use of studied fluidized bed nitrocarburizing technology for thermo-chemical treatment especially for tools designed for plastics processing.

Keywords: Nitrocarburizing; Fluidized bed; Chemically active powder

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MANUFACTURING AND PROCESSING

1. Introduction

The nitrocarburizing of steel is used in the machine and tool industry in order to obtain unique tribological and fatigue parts properties, impossible to achieve by carburizing or carbonitriding. Nitrocarburizing is subjected to the most common parts such as extrusion dies, extruders screws and nozzles, piercing and cutting dies, dowels and others. High mechanical properties of machined parts accompanied with their high corrosion resistance, are especially important e.g. for dies working in chemically aggressive environment during the plastics processing such as polyvinyl chloride PVC, when the parts are exposed to chlorine. The reasons for the increase of the properties of parts subjected to nitrocarburizing can be understood by observing the steel microstructure. The outer layer known as compounds zone is a several micrometers to several dozens of micrometers phase layer – an iron, nitrogen and carbon compound, mainly in the form of a carbonitride ε - $Fe_{2-3}(C,N)$ with different carbon and nitrogen content. The compound layer is called white or ceramic layer and is responsible for tribological and corrosion properties. Diffusion zone properties differ significantly from the compounds zone properties. Diffusion zone has approximately several hundred micrometers thickness. This layer is responsible for material bearing capacity and also static and fatigue strength properties.

During the processes of thermo-chemical treatment the source of nitrogen and carbon is mainly carbon- and nitrogen-bearing gas (e.g. ammonia, endothermic atmosphere). In recent years, plasma nitriding technique is gaining popularity [1,2]. Nitrocarburizing medium may also be a salt bath, but this method is used less frequently because of cyanate bath high toxicity [3]. Despite the relatively widespread use of the gas nitrocarburizing method, this technique is dangerous, both for the employees and natural environment, due to the produced exhaust gases and explosion risk of flammable atmosphere in the absence of appropriate precautions behavior. Disadvantages of gas nitrocarburizing are substantially reduced in the atmospheric fluidized-bed method, where for example there is no risk of explosion in the furnace workspace. Above drawbacks are devoid in box-method [4], but this method is inconvenient to handle. Discussed in this article nitrocarburizing method in fluidized chemically-active bed, combines the most important advantages of the box-method and atmospheric fluidized-bed method. The source of carbon and nitrogen in this case is based on a special mixture of active carbon and potassium ferrocyanide, which is also a heat accumulator, which ensures uniform heat distribution. For the bed fluidization are used vibrations resulted from the forced mechanical vibrations which are subjected to furnace retort. In this method retort with fluidized bed is equipped with electro-vibrator and the whole system is mounted elastically in the body of the furnace [5]. Because of vibrations, bed has similar characteristics to those obtained by introducing fluidization gas into it. Similar to gas, vibrations make that the fluidized bed moves in a furnace retort, heats up uniformly and is in direct contact with the entire surface of the workpiece, regardless of its shape [6]. The advantage of this technique, compared to the already used conventional methods in thermo-chemical treatments in the retort, continuous, vacuum and others furnaces, is that the workpiece can be removed and placed in the furnace at any time. This means that in one furnace can be carried out independently in parallel processing multiple parts treatment that require different processing time by inserting and removing them, depending on the technological requirements, without waiting for the completion of the processes carried out on other elements.

This article present the results of the properties of hot working tool steel, important from the point of view of manufacturers and tools users.

2. Purpose and scope of work, research methodology

The article presents the results of the microstructure, hardness, wear resistance and impact strength studies of samples made from hot working tool steel X37CrMoV5-1 subjected to nitrocarburizing at a temperature of 560°C with treatment time 4 and 8 hours.

Nitrocarburizing attempts were carried out in order to assess the suitability of this method to practical use in the tools manufacturing processes. There was carried out control analysis of the samples chemical composition using the analytical spectrometer Foundry Master 01D0058 (Table 1). Before the processes of thermo-chemical treatment, samples were subjected to heat treatment consisting of quenching (austenitizing temperature 1030°C, cooling medium: nitrogen at a pressure of 0,6 MPa) and double tempering (temperature 525°C/2 hours and 580°C/2 hours).

Carbonitriding process was carried out in a fluidized furnace FP700 with retort diameter ø110 mm with attached electric vibrator type BM110/10, which was forcing mechanical vibrations of the circular type with amplitude approx. 4 mm (motor power 140 W and rotation speed 1500 rpm). Retort with vibration motor was placed in furnace axis and was based on a horizontal furnace plate with use of three brackets with coil springs, whose task was to suppress the vibration transmission to the body of the furnace.

Metallographic observations of prepared specimens (etched with 3% Nital) were made with aid of metallurgical light microscope Nikon Eclipse LV150 at 500x magnification. Hardness measurements were made using Struers DuraScan-70 hardness tester under load of 0.5 and 1 kG. Hardness profiles were determined with CV Instruments hardness tester on metallographic sections. Measurements were performed under a load of 0.2 kG, at depths from 0.03 to 0.24 mm.

Table 1.
Chemical composition of X37CrMoV5-1 steel

Chemical composition, wt. %	C	Si	Mn	Cr	Mo	V	Ni	W
	0.38	0.99	0.47	4.86	1.29	0.37	0.24	0.18

Wear resistance studies were conducted using a 3-rollers and cone method using a I-47-K-54 device. Tests were performed with continuous oil (Lux-10) lubrication, at a constant counter-sample speed of 526 rpm and a constant unit pressure of 100 MPa. The depth of abrasion, which was defining linear wear, was determined by measuring the diameters of the ellipses formed by wiping the surface of each of the rollers and the average results. Total friction test time was 100 minutes. Abrasion was measured while breaking the attempt every 10 minutes and was followed by increased load corresponding to increased surface abrasion. Abrasive wear was characterized by total linear wear (after 100 minutes of test) and by the wear intensity I_I in a predetermined period of wear. Conic counter-specimen was made of 41Cr4 steel and was quenched and tempered to 30 HRC hardness and grinded for roughness R_a=0.32 μm.

The impact measurements were performed with Charpy method for samples with a length of 55 mm, square cross section with a side of 10 mm, with U-shape notch depth 2 mm and the notch bottom fillet radius 1 mm. The test was performed on the WPM impact hammer, with the pendulum initial energy 50 J.

The results of samples properties were referred to the variant without nitrocarburizing (samples heat treatment only).

3. Nitrocarburizing of X37CrMoV5-1 steel

3.1. Metallographic observations

Microstructure images of nitrocarburized (4 and 8 hours) samples are shown in Figs. 1a and 1b.

In the pictures can be visible not-etching and closely bonded with the substrate subsurface layers of iron carbonitrides which thickness depends on the nitrocarburizing time and is equal respectively approx. 2.5 μm and 5 μm, for the process time 4 and 8 hours. Obtained white layers are compact with low porosity visible in the outer parts, which may indicate low activity of nitrogen and corresponding carbon activity [7]. The compounds layers are continuous over the entire samples cross section and consist probably mainly from the carbonitrides ε - $Fe_{2\text{-}3}(C,N)$. Below the white layer is highly visible strong-etching diffusion zone, with dark color due to the strongly accumulated at the surface of the grain boundary nitrides or carbonitrides of type γ - Fe_4N. In the microstructure of the diffusion zone and below this zone can be seen presence of small globular carbides located in the matrix of tempered martensite. Presumably these are carbide of alloying elements such as molybdenum and vanadium and their presence is associated with precipitation processes, resulting from the chemical composition of the steel subjected to nitrocarburizing.

In [7] it was observed, that the dispersion carbides containing chromium during the nitrocarburizing process of X40CrMoV5-1 steel easily dissolve, directly transforming into the nitride (or carbonitrides [8]) Cr(C,N). Depending on the processes duration, diffusion layer thickness was approx. 70 μm (4 h) and 100 μm (8 h). The accurate characterization and analysis of obtained layers requires testing using X-ray diffractometer XRD.

3.2. The hardness of the surface and layer cross-section

The substrate material subjected to nitrocarburizing has after heat treatment hardness approx. 507-540 HV (Fig. 2 – variant HT). Nitrocarburizing processes, depending on their duration, allowed to achieve a hardness approx. 796-808 HV0.5 (Fig. 2a) or 713-755 HV1 (Fig. 2b). Hardness differences between the loads may result from the law of variable hardness or may result from the impact of white layer high hardness, which was pierced when measuring under higher load (Fig. 2b).

Hardness of samples in the cross-section subjected to nitrocarburizing is shown in Fig. 3. Maximum measured hardness in the subsurface zone, depending on the time of thermo-chemical treatment was between 790-820 HV, whereas the hardness of the core material was approx. 510 HV.

Obtained hardness of the surface and layers cross-section are slightly lower than the hardness of tool steel with similar chemical composition subjected to nitrocarburizing by powder mixture box-method [4]. The lower hardness can be resulted from a carried out different heat treatment of samples, especially tempering operation, which was carried out at a much lower temperature of 520°C. Related hardness values were obtained by the authors [7] in the case of gas nitrocarburizing of H13 steel in an atmosphere consisting of $N_2/NH_3/CO_2$. Analyzing the hardness obtained by the authors for other steels, especially in the case of nitrocarburizing of alloy tool steels, it should be in mind the possible impact of the substrate material alloying elements solubility in the structure of the compound layer and the formation of hard phases, affecting on the achievable hardness of the surface layer.

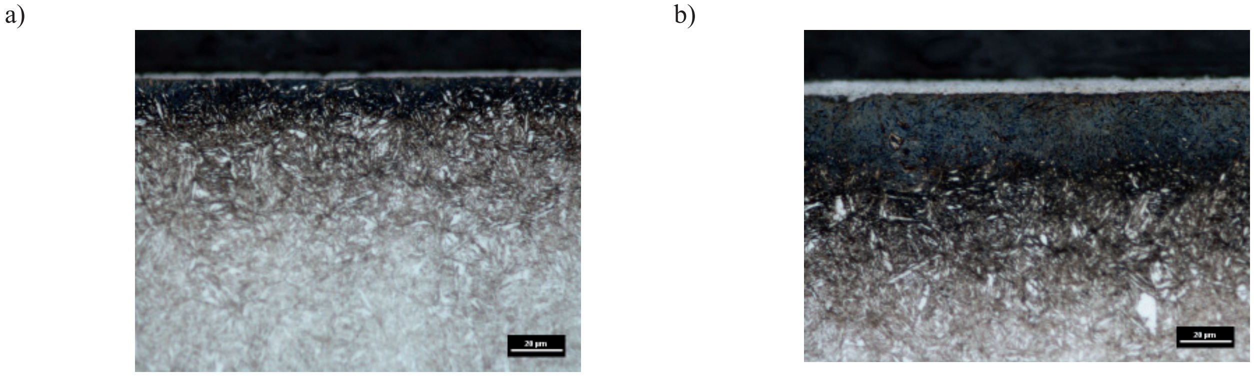

Fig. 1. Microstructure of X37CrMoV5-1 steel after 4 h (a) and 8 h (b) nitrocarburizing process

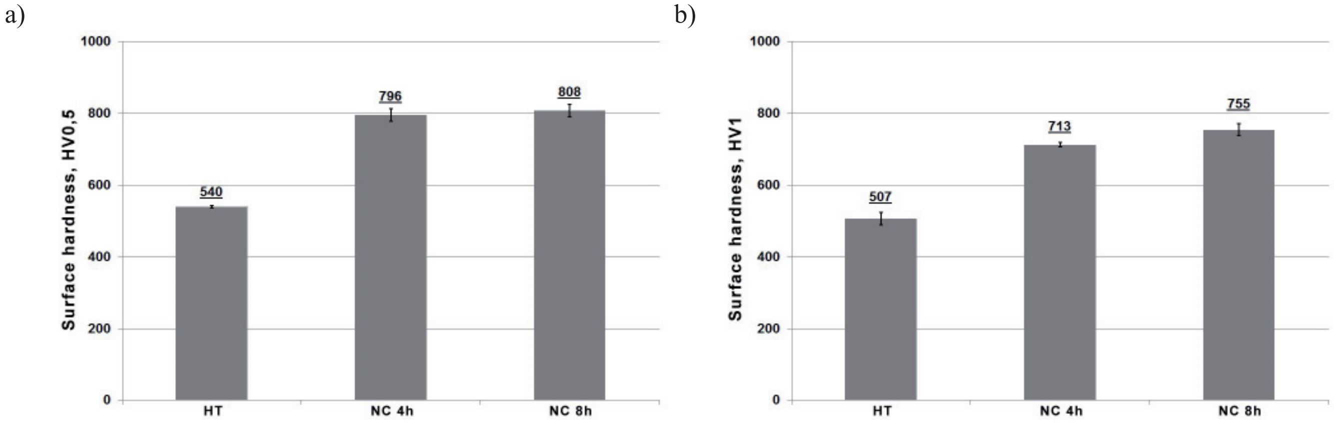

Fig. 2. HV0.5 (a) and HV1 (b) hardness of X37CrMoV5-1 steel after nitrocarburizing process

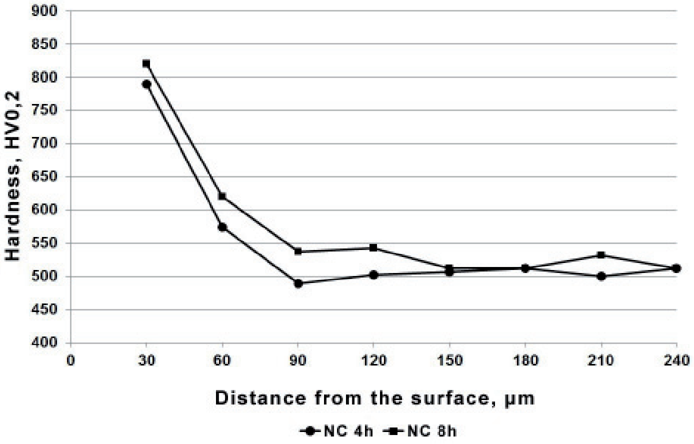

Fig. 3. HV0.2 hardness profile at the cross section of X37CrMoV5-1 steel after nitrocarburizing process

3.3. Wear resistance

Wear resistance of samples subjected to heat treatment and nitrocarburizing closely correlates with the hardness of material surface which resulted from the subsurface area microstructure (occurrence of hard compounds layer and diffusion zone).

All nitrocarburized samples were characterized by a regular wear degree, revealing the presence of steady-rate wear phenomenon in the entire wear test, without the initial break-in (lapping) period.

Analysis of the data shown in the graph of linear wear in a function of friction time, allow to observe that the lowest wear values and the lowest values of wear intensity are correlated with the samples subjected to nitrocarburizing for 4 hours. Mentioned dependence was revealed at both 100 MPa and 400 MPa unit pressures (Figs. 4a, 4b). Higher wear resistance of samples subjected to nitrocarburizing in a shorter time (4 hours) is probably associated with the presence of white layer in the structure, although its thinner thickness, with the predominant ε phase with high nitrogen content, preferably affecting the tribological properties [9] and other steel properties [10, 11]. The lowest wear resistance values were associated with samples subjected to quenching and tempering. In addition, for friction test with 400 MPa unit pressure, samples underwent seizure after 30 minutes (Fig. 4b).

Analyzing the obtained friction tests results together with the microscopic compounds layer thickness it may be noticed that in the case of 100 MPa unit pressure and samples nitrocarburized for 8 hours, wipe formed on the samples surface does not exceed the thickness of white layer (Figs. 1b and 4a) and the obtained linear wear course, despite the presence of the two intensity ranges, is practically a straight line. For samples nitrocarburized in shorter time (4 hours), with the white layer thickness approx. 2.5 μm (Fig. 1a), after reaching by emerging wipe this value (after 60 minutes of test), there was a significant change in wear intensity, illustrated on wear course graph with a visible change of the wear curve inclination angle (Fig. 4a).

For four-times higher unit pressures reaching by emerging wipe values of compounds layers thickness followed after approx. 20-30 minutes of test (Fig. 4b). It should be mentioned, that despite exceeding this thickness, friction process, apart from the obvious counter-sample contact with the exposed diffusion zone, took place also within the wear resistant white layer, placed on the elliptical shape wipe circumference. This fact explains the lack of a drastic increase of wear intensity after piercing by wipe the white layer.

3.4. Impact strength

As it was expected, the nitriding process resulted in a significant reduction in the steel impact strength, associated with a creation of hard and thus brittle diffusion zone and white layer on the material surface. Along with prolongation of thermo-chemical treatment time there was a significant decrease in material impact strength (Fig. 5). For this reason, nitrocarburizing should be used where high steel impact strength is not a key feature from the point of view of tools or machine parts durability.

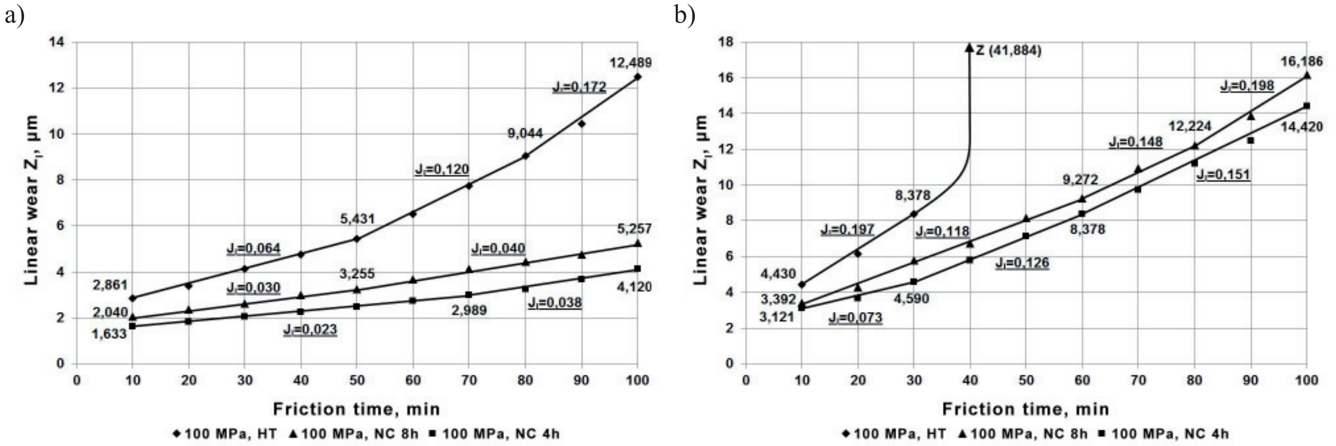

Fig. 4. Courses of linear wear vs. time of friction of X37CrMoV5-1 steel after nitrocarburizing process, 100 MPa (a) and 400 MPa (b) contact unit pressures

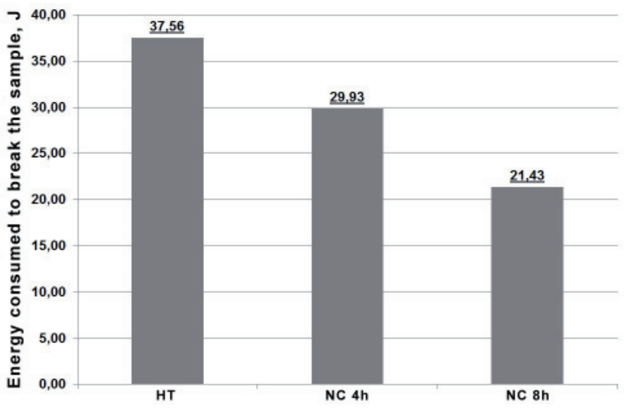

Fig. 5. Impact strength of X37CrMoV5-1 steel after nitrocarburizing process

4. Summary and conclusion

Based on the analysis of layers characteristics formed during nitrocarburizing process and designated strengthened steel properties, it can be said that the present method allows to obtain a layer with correct structure, it means obtaining compounds layer with phase structure consisting mainly ε type carbonitrides having a relatively thin porous zone and diffusion zone with a suitable thickness.

With the nitrocarburizing process strengthened steel has a much higher hardness and wear resistance than the material subjected to conventional quenching and tempering. Independently on the nitrocarburizing time, obtained layers had a similar wear resistance, while thermo-chemically treated samples had significantly different impact strength and, in a small way, hardness. Selection of nitrocarburizing time 4 hours seems to be appropriate compromise between the derived parameters such as relatively high surface and over cross-layer hardness, wear resistance and impact strength.

Obtained diffusion layers confirm the practical applicability of the studied nitrocarburizing method in fluidized bed of X37CrMoV5-1 steel. With the intended application they could be used to strengthen the surface of tools used for plastics processing e.g. polymers for chemical, medical and food industry from which, besides its high wear resistance, is required high corrosion resistance, due to its work in medium containing acids and chlorides formed from the decomposition of thermoplastics such as for example PVC.

In the further stages of conducted studies it is expected to be carried out test of nitrocarburizing of specific tools or machinery parts and carried out comparative performance test.

Mentioned nitrocarburizing method offers a number of advantages and benefits for its use in industrial practice, in comparison to bath, gas and plasma techniques. Among the advantages, the first to mention is an easy operation of furnace and the lack of liquid or gaseous technological media, because for a proper treatment is necessary only powder mixture with specified chemical composition. This feature provides also a low-cost equipment operation. This method is also harmless to the environment.

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