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THE INFLUENCE OF THE SOAKING TEMPERATURE OF NITRIDED WCL TOOL STEEL (EN-X37CrMoV5-1) ON THE MICROHARDNESS AND TRIBOLOGICAL WEAR OF THE SURFACE LAYER

WPŁYW TEMPERATURY WYGRZEWANIA AZOTOWANEJ STALI NARZĘDZIOWEJ WCL (EN-X37CrMoV5-1) NA MIKROTWARDOŚĆ ORAZ ZUŻYCIE TRIBOLOGICZNE WARSTWY WIERZCHNIEJ

Key words:

tool steel, X37CrMoV5-1, nitriding, upper layer, microhardness, tribological wear

Słowa kluczowe:

stal narzędziowa, X37CrMoV5-1, azotowanie, warstwa wierzchnia, mikrotwardość, zużycie tribologiczne

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Abstract

The present paper refers to the evaluation of the influence of soaking temperature of nitrided hot work tool steel, X37CrMoV5-1 (WCL), intended for dies for extruding aluminium profiles, on the structure, microhardness, and tribological wear of the nitrided layer.

The research involved nitrided steel specimens (X37CrMoV5-1) soaked for 8 hours in an industrial furnace at temperatures of 450°C, 480°C, 520°C, 560°C, and 600°C. For comparison purposes, a REFERENCES material was used, which was not soaked after nitriding. Initially, as the soaking temperature raised, the microhardness of the nitrided layer increased by ca. 10%; however, a further increase in the soaking temperature to more than 450°C caused a decrease in the microhardness of the nitrided layer. The results of tribological tests showed that soaking nitrided steel at a low temperature (450°C) and high temperature (600°C) caused a decrease in tribological wear. Out of the tested materials, the highest microhardness of the upper layer was observed in the samples soaked at 450°C, while the highest resistance to tribological wear was obtained for the samples soaked at 600°C. The conducted tests indicate the possibility of extending the lifetime of dies made from the investigated nitrided steel.

INTRODUCTION

In industrial applications, such as extrusion and forging, the equipment (moulds, dies, etc.) is repeatedly subjected to cyclic thermal and mechanical loading. These technologies are characterized by operating temperatures above 600°C, and very high values of surface load and specific tribological pairs. As a result of these difficult working conditions, damage to the tool surface appears in a form of, e.g., abrasive wear, corrosion, or thermal cracking **[L. 1, 2]**. Due to the large number of products manufactured in these industries with a relatively short life cycle of the tooling, even slight improvements in this area bring a positive economic effect.

Tool life can be improved in two ways: The first method consists in improving the base material by adding alloying elements, such as boron or niobium **[L. 3, 4]**. The second method involves the improvement of surface properties so as to maximize its efficiency **[L. 5–7]**. Nitriding is a commonly used process of tool surface treatment in the manufacturing industry. It enhances the hardness of the surface and, at the same time, induces residual compressive stresses on it **[L. 8, 9]**, which is advantageous from the viewpoint of increasing the fatigue life of the material. Among the methods of nitriding, gas nitriding is the most common method for the surface treatment of extruding dies due to its simplicity, reliability, and cost effectiveness **[L. 8, 10]**.

The die is a key element in the aluminium extrusion. It must withstand high stresses and high temperatures, which is necessary for an aluminium billet to be squeezed through the die channel in the non-lubricated sliding contact conditions **[L. 11, 12]**. The wear of calibration rings is inevitable in such an environment, which cannot be tolerated, for it has a negative effect on the dimensions and surface quality of the extruded material.

The most widely used materials for tools to work at high temperatures are steels such as 5% Cr and 1% Mo (X37CrMoV5-1/WCL). This is due to their relatively low content of expensive alloying components, a not too high temperature of hardening, small dimensional changes, a resistance to oxidation (the content of Cr and Si), very good ductility, and a resistance to cracking **[L. 13]**. Steel X37CrMoV5-1 is also the most resistant to tempering, since it contains large amounts of chromium and molybdenum. In addition, it exhibits the least sensitivity to rapid temperature changes during operation and high hardenability **[L. 14]**.

RESEARCH MATERIAL AND METHODOLOGY

Nitrided hot work tool steel (X37CrMoV5-1) specimens with the following chemical composition (mass concentration of elements): 5% Cr, 1% Mo, 0.4% V, and 0.37% C, where subjected to tests. Before the nitriding process, they were quenched and tempered to the desired hardness of 48–50 HRC. Next, the specimens were subjected to automatically controlled regulated gas nitriding, using the NITREG® (Canada) technology, in the following conditions: a two-step nitriding; nitriding temperature – 530°C; nitriding time – 18 h; nitriding atmosphere – ammonia + nitrogen; nitric potential = 3. The prepared specimens were soaked for 8 hours in an industrial furnace at temperatures of 450°C, 480°C, 520°C, 560°C, and 600°C. The input (REFERENCES) material, not soaked in the process of nitriding, was also used for comparison purposes. The tested specimens were delivered by ALUFORM Sp. z o.o. from Tychy, which carried out the processes of nitriding and soaking.

The soaking that followed the nitriding process resulted from the application nature of the research commissioned by the supplier of the analysed material. Namely, the dies for aluminium extrusion made from the tested material are subjected to the pre-heating to a temperature ranging from 400°C to 600°C before the extrusion process. This procedure lasts a few hours, and in order to maintain continuity of the extrusion process, there are several dies being soaked in the furnace at the same time (according to the approved production plan). Since there may be a variety of unplanned situations in the production process (such as standstill, failure, etc.), it may happen that a die will be held in the furnace longer than planned. Therefore, the aim of the study was to investigate the changes in the nitrided layer after soaking the dies in the furnace at various temperatures, before starting their extrusion, and thus, to determine at what temperatures dies may be held in the furnace for a predetermined time (8h) without undesirable changes in the nitrided layer.

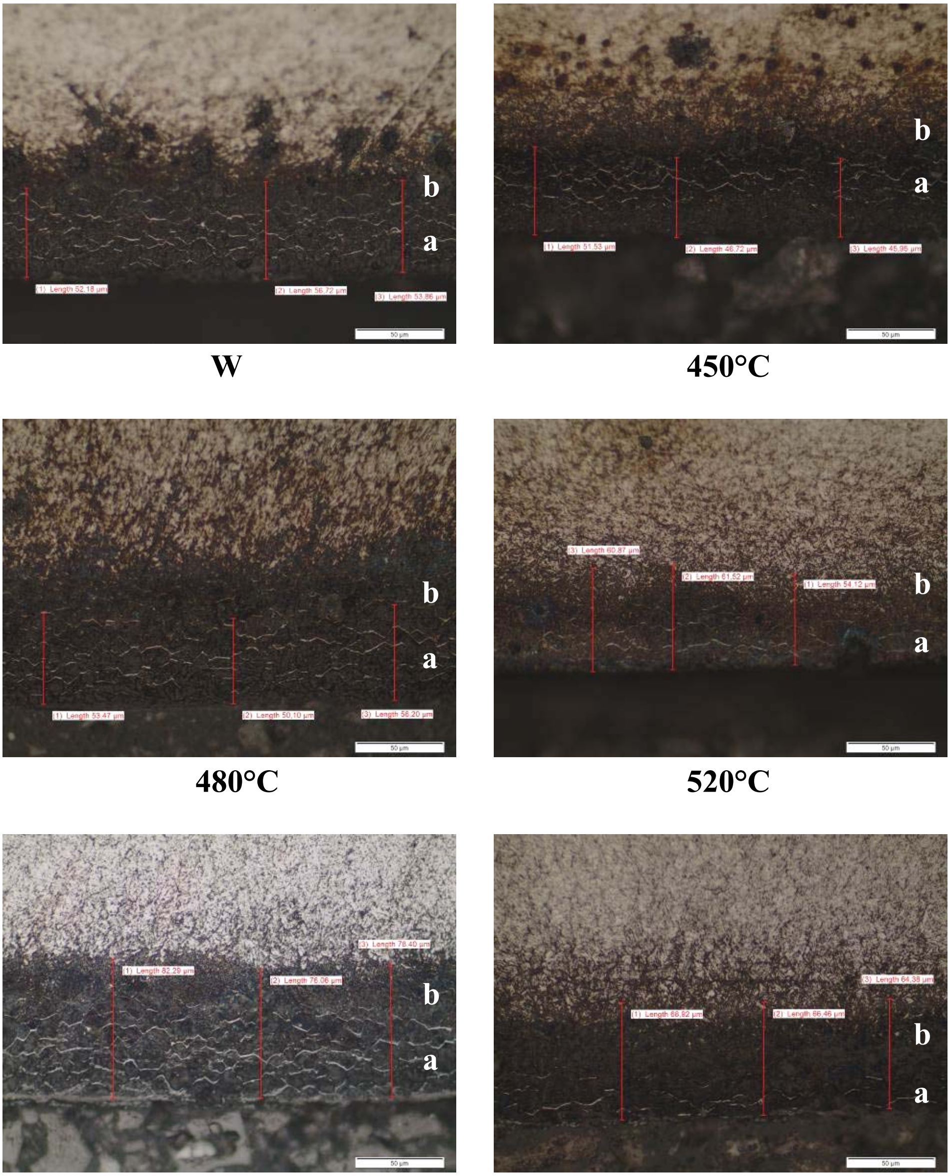
The analysed specimens were cylinder-shaped, 20 mm in diameter, and 25 mm in height. Before the test, they were cut transversely into two parts and micro-sections of $R_a = 0.1$ μm were made on their cross sections. The surface of micro-sections was etched with 1.5% Nital in order to make the nitrided layer visible. Microscopic images were taken on the etched surfaces of the micro-sections, using a light microscope OLYMPUS GX51. The thickness of the nitrided layer was measured on the photographs (9 measurements for each specimen) with the Stream Essentials software. Micromechanical tests were performed using a Vickers microhardness tester of Wolpert Wilson Instruments (model 401MVD). Nine indentions were made for each of the specimens in the precipitation zone. The following parameters were determined: a Vickers microhardness (HV) of 0.5, a load of 4.9 N (500 gF), and a time of withstanding the load – 10 s. Tribological tests were conducted using the Tribometer TRN tester (Anton-Paar) in the ball-on-disc system. For each of the specimens, the tests were carried out on the front surface of the cylinder. An Al_2O_3 ball, 6 mm in diameter, was used as the counter-specimen. The tests were carried out at a friction distance of 2 km with the sliding speed of 0.1 m/s. The load applied to the sliding contact was 10 N. The ambient temperature was maintained within the range of 21±1°C with relative humidity of 50±5%. Volumetric wear W_v was determined using averaged profilographometric measurements by measuring the area of wear trace along the specimen's radius in four places located at an angle of 90° relative to each other. The Mitutoyo Surfiest SJ-500 profilograph was used in this research. The volumetric wear W_v was determined from the following formula:

$$W_v = \frac{V}{F_n \cdot s}\left[\frac{mm^3}{N \cdot m}\right] \tag{1}$$

where V – volume of the wear trace of the disc, F_n – pressure force, s – friction distance.

RESEARCH RESULTS AND THEIR ANALYSIS

The microscopic images of the tested specimens made it possible to observe the structure of the nitrided layer. Examples of the pictures for each of the soaking variants and the REFERENCES specimen are shown in **Figure 1**.



W

450°C

480°C

520°C

560°C

600°C

Fig. 1. Nitride precipitates zone (a) and diffusion zone (b) of soaked specimens
Rys. 1. Strefa wydzieleń azotków (a) oraz strefa dyfuzyjna (b) wygrzewanych próbek

There are two layer zones visible in the photographs: the zone of nitride precipitates γ', that is closer to the surface (white lines along the primary austenite grain boundaries), and the diffusion zone, which is a solid solution of nitrogen in iron α, that is closer to the core. According to literature **[L. 15–17]** layers with such a structure are usually obtained in two-stage processes of regulated gas nitriding in industrial conditions, i.e. like the ones used in this study. One of the purposes of the analysis of microscopic images was to determine the possible adverse changes in the nitrided layer, such as cracks / microcracks, under the influence of elevated temperature. The conducted analysis did not reveal defects of this type.

The dependence of the thickness of the nitrided layer on soaking temperature is shown in **Figure 2**.

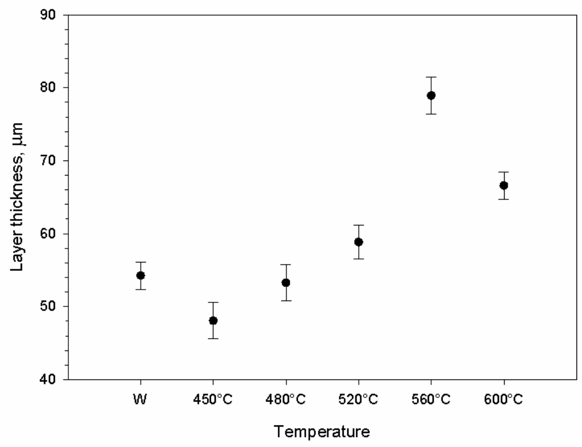


Fig. 2. Thickness of nitrided layer measured for the tested samples
Rys. 2. Grubość warstwy azotowanej zmierzona dla badanych próbek

The measurements have shown that the nitrided layer did not undergo degradation under the influence of elevated temperature, and even with increasing temperature, the thickness also increased. There was a slight decrease in its thickness only at a temperature of 600°C. It was observed that the diffusion phenomenon took place in the layer under the influence of temperature and the thickness of the diffusion zone changed.

The measurement of the microhardness of the nitrided layer was aimed to investigate the effect of temperature on the mechanical properties of the upper layer. The obtained results of microhardness tests **(Fig. 3)** indicate its initial growth in relation to the REFERENCES material by ca. 10% for the material soaked at 450°C. However, a further increase in temperature caused a decrease in microhardness to ca. 20% of the initial value for the material soaked at 600°C.

The biggest differences (decreases) in microhardness were observed for the specimens soaked in the lowest and highest temperatures, i.e. 480°C and 600°C. The differences in the values set for the intermediate temperatures are insignificant and fall within the margin of error. These values are also close to the microhardness of the REFERENCES material. The decrease in microhardness can be associated with a decrease in the amount of nitride precipitates that ensure the high hardness of the upper layer. It can also result from a decrease in the degree of the dispersion of nitrides, because, together with an increasing temperature the thickness of the nitrides increases, the hardness of the layer decreases. A similar dependence was also observed in paper **[L. 18]**, where the decrease in hardness of the nitrided layer, occurring as a result of oxidation, was attributed to the decomposition of the nitrides responsible, at least partly, for hardening the surface of the material.

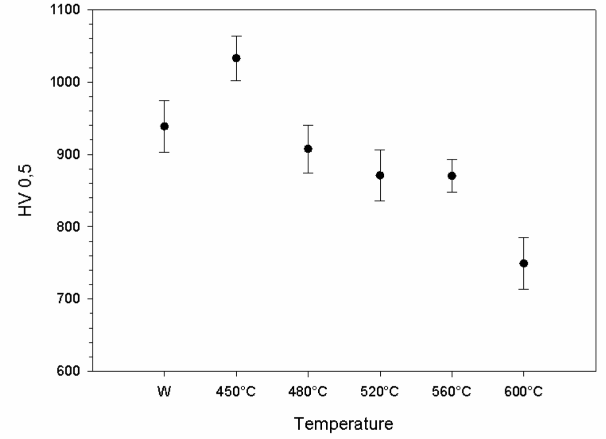


Fig. 3. Microhardness of HV 0.5 determined for the tested specimens
Rys. 3. Mikrotwardość HV 0,5 wyznaczona dla badanych próbek

The results of tribological tests are presented in **Figures 4** and **5**.

The determined values of tribological wear **(Fig. 4)** for specimens soaked at 450°C exhibited a slight decrease by ca. 12% in relation to the REFERENCES material. As the soaking temperature increased, an increase of tribological wear of the specimens was observed up to a value by approx. 78% higher in relation to the initial material in the case of temperature of 560°C. For the material soaked at 600°C, a visible increase in the resistance to tribological wear was observed, and out of all the materials analysed, this one exhibits the lowest wear. The tribological wear that grows together with the temperature and thickness of the nitrided layer indicate that the diffusion zone grows at the expense of the hard nitride precipitates zone γ'. The obtained values of the friction coefficient **(Fig. 5)** are similar for the REFERENCES material and for the material soaked at temperatures of 450°C and 520°C, and they fall within the range of 0.88 to 0.89.

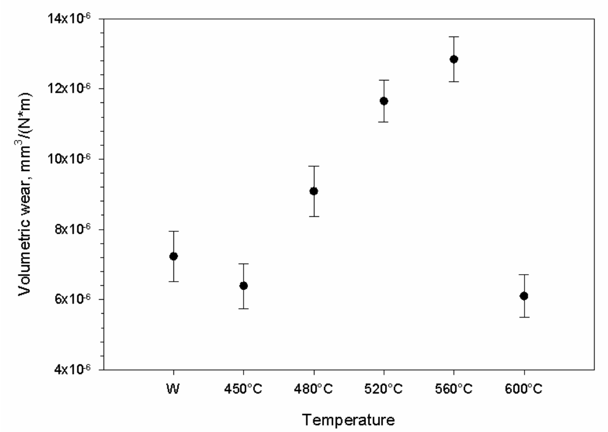


Fig. 4. Tribological wear of the nitrided layer determined for the tested samples
Rys. 4. Zużycie tribologiczne warstwy azotowanej wyznaczone dla badanych próbek

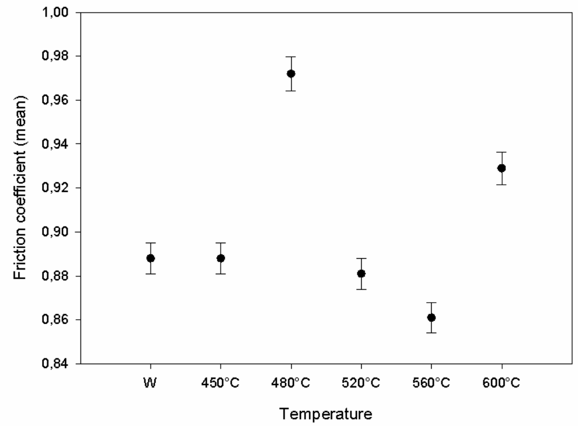


Fig. 5. Values of the average friction coefficient determined for the tested specimens
Rys. 5. Wartości średniego współczynnika tarcia wyznaczone dla badanych próbek

The specimens soaked at 560°C showed a slightly lower friction coefficient, while, in the case of the specimens soaked at 480°C and 600°C, the coefficient rapidly increased by approx. 9% and approx. 5%, respectively.

CONCLUSIONS

- Soaking of nitrided steel at temperatures in the range of 480°C–600°C for 8 hours causes a decrease in the microhardness of the near-surface zone of the upper layer.
- As the soaking temperature increases, the tribological wear increases as well. This trend does not apply to the highest temperature of soaking (600°C) for which a rapid decrease in wear was observed.

- The nitrided layer did not undergo degradation under the influence of elevated temperatures, and its thickness increases with temperature to approx. 560°C.
- The decreasing microhardness of the layer together with the increasing tribological wear, with an increasing thickness of the nitrided layer, may indicate a decrease in the thickness of the nitride precipitates zone γ' in favour of the growing diffusion zone, and it may indicate a decrease in the amount and/or enlarging (coagulation) of the nitride precipitates of alloying elements in the diffusion zone.
- In the majority of cases, the friction coefficient is maintained at a similar level (0.88 to 0.89). Only the specimens soaked at 480°C and 600°C exhibit a rapid growth in the friction coefficient.
- The conducted analysis of microscopic images did not reveal the presence of any defects (such as cracks or microcracks) in the upper layer.

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Streszczenie

Niniejsza praca dotyczy oceny wpływu temperatury wygrzewania azotowanej stali narzędziowej do pracy na gorąco X37CrMoV5-1 (WCL) przeznaczonej na matryce do wyciskania profili aluminiowych na strukturę, mikrotwardość oraz zużycie tribologiczne warstwy azotowanej.

Badaniom poddano próbki azotowanej stali X37CrMoV5-1 wygrzewane przez 8 godzin w piecu przemysłowym w temperaturze: 450°C, 480°C, 520°C, 560°C oraz 600°C. W celach porównawczych zastosowano także materiał wzorcowy, który nie był wygrzewany po procesie azotowania. Ze wzrostem temperatury wygrzewania początkowo odnotowano wzrost mikrotwardości warstwy azotowanej o ok. 10%. Z kolei podwyższenie temperatury wygrzewania powyżej 450°C powodowało jej obniżenie. Wyniki testów tribologicznych wykazały, że wygrzewanie stali azotowanej w niskiej temperaturze (450°C) oraz w wysokiej (600°C) powoduje spadek zużycia tribologicznego. Spośród badanych materiałów największą mikrotwardość warstwy wierzchniej odnotowano dla próbek wygrzewanych w temperaturze 450°C, natomiast najwyższą odporność na zużycie tribologiczne uzyskano dla próbek wygrzewanych w temperaturze 600°C. Przeprowadzone testy wskazują na możliwość wydłużenia czasu użytkowania matryc wykonanych z badanej stali azotowanej.