UDK 669.14.018.252:621.785.5
Izvirni znanstveni članek

ISSN 1580-2949
MTAEC9, 37(3-4)145(2003)

PLASMA NITRIDING OF P/M M2 TOOL STEEL - STRUCTURE AND PROPERTIES

NITRIRANJE ORODNEGA JEKLA P/M M2 V PLAZMI - MIKROSTRUKTURA IN LASTNOSTI

Peter Jurči[1], Pavel Stolař[1], František Hnilica[2], Tomáš Blaštík[3]
[1]ECOSOND s.r.o., Křížová 1018, Prague, CZ – 150 00, Czech Republic
[2]ŠKODA-ÚJP Praha, a.s. Nad Kamínkou 1345, Prague, CZ – 156 10, Czech Republic
[3]Brano a.s., Hradec na Moravicí, CZ – 747 41, Czech Republic
jurci@ecosond.cz

Prejem rokopisa - received: 2002-11-18; sprejem za objavo - accepted for publication: 2003-03-28

Plasma nitriding is a well-known method that enables us to modify some of the surface properties of metallic materials. The application of this thermo-chemical heat-treatment for the surfacing of tools can bring many benefits for the end-user because it allows to improve the quality of manufactured tools. This is due to the strengthening of the near-sub-surface region and to the occurrence of compressive stresses in these areas.

In this investigation specimens and tools from P/M M/2 ledeburitic steel were heat treated to 730 HV 10, then plasma nitrided and submitted to a structural analysis. The presence of nitrides Fe_3N and Fe_4N was determined in the near-surface areas. These phases increased the hardness above that provided by the hardening and tempering: and the hardness of about 1000 HV 10, depending on the processing parameters is achieved. Nitrided and non-nitrided tools (punches) were used on a production line, and plasma nitriding led to the prolongation of the tools lifetimes for an order of magnitude. This phenomenon was explained by changes in the location of the fracture initiation and the crack propagation. Whereas the crack started at the surface in the case of the non-nitrided tools, the fracture initiation was shifted to a certain depth for the nitrided material. As was reported previously, these alterations may initially be due to the compressive stresses in the plasma-nitrided layer.

Key words: plasma nitriding, P/M high-speed steel, fracture, tool lifetime

Nitriranje v plazmi je dobro poznana metoda, ki omogoča spreminjanje lastnosti površine kovinskih materialov. Uporaba te toplotno-kemične tehnike za obdelavo površine orodja, omogoča nekatere prednosti za končnega uporabnika, ker se s to metodo izboljša kvaliteta orodja. To je posledica utrjevanja površine in podpovršinskega področja ter pojava tlačnih napetosti v utrjenem področju.

Vzorci in orodja, izdelani iz ledeburitnega jekla P/M M2, so bili toplotno obdelani na trdoto 730 HV 10 in nitrirani v plazmi. Po tem postopku je bila izvršena strukturna analiza. Potrjena je bila prisotnost Fe_3N- in Fe_4N-nitridov v območju pod površino. Odvisno od parametrov postopka, navzočnost teh faz poveča trdoto na 1000 HV 10. Nitrirana in nenitrirana orodja (prebijači) so bila uporabljena na proizvodni liniji. Orodja, nitrirana v plazmi, so pokazala podaljšano zdržljivost, izboljšanje je bilo za red velikosti. To si razlagamo s spremembo mesta iniciacije razpoke in njene propagacije. Medtem ko je bila iniciacija razpoke pri nenitriranem orodju na površini, se je iniciacija razpoke pri nitriranem orodju pomaknila na določeno globino. Kot je bilo že objavljeno, je ta premik posledica delovanja tlačnih napetosti v plasti, nitrirani v plazmi.

Ključne besede: nitriranje v plazmi, hitro rezno jeklo P/M, prelom, zdržljivost orodja

1 INTRODUCTION

The use of fine-grained high-speed steels (HSSs) and ledeburitic steels produced using powder metallurgy (P/M) techniques can bring many benefits to the tool industry because these materials are able to prolong considerably the service lifetime of manufactured cold-worked tools. This is due to the excellent combination of microstructure and mechanical properties as result of the production method used [1-3].

Tools made from P/M tool steels have to be heat treated before use. The heat processing involves vacuum austenitising, gas quenching and multiple tempering, which results in a typical hardness ranging between 700 and 800 HV. As previously reported, the service lifetime of stamps was prolonged more than seven times when the conventionally produced AISI M2 steel was replaced with P/M material of the same chemical composition and hardness [4].

Plasma nitriding was established as a convenient method of pre-treatment prior to PVD coating [5-7], or as a final treatment of the material [8]. The use of plasma nitriding as the final operation leads to:

1) a significant surface hardness increase in comparison with the core material.
2) the formation of nitrides in the sub-surface region, which may increase the wear resistance significantly and lower the friction coefficient [10].
3) the formation of compressive stresses in the diffusion layer [11], due to the saturation of martensite with nitrogen.

In previous experimental reports the investigation of the optimum processing route for achieving the best combination of wear resistance, hardness and other structural parameters were described. These investigastions led also to practical application of the technique, and in this paper attempts some of the aspects of the applications of plasma nitrided tools made from AISI M2 P/M high-speed steel in industry.

2 EXPERIMENTAL

P/M AISI M2 grade high-speed steel with a chemical composition of 0.9 % C, 6.1 % W, 5 % Mo, 4.14 % Cr, 2.02 % V, Fe bal. was used for the investigation. Three sets of punches were made, net-shape machined and then heat treated (hardened and triple tempered) to a hardness of 730 HV 10. Cylinders with a diameter of 10 mm and a length of 12 mm were added to each processed set of tools as reference specimens for the structural analysis and related measurements. The plasma nitriding (unless otherwise designated*) was carried out with the following combinations of processing parameters:

*I: without plasma nitriding

II: T = 500 °C, atmosphere with N_2:H_2 = 1:3, t = 60 min.

III: T = 530 °C, atmosphere with N_2:H_2 = 1:3, t = 120 min.

The structural analysis was carried out with optical and scanning electron microscopy. The phase constitution of the nitrided regions was investigated with X-ray diffraction. The surface hardness and microhardness depth profiles were tested using the Vickers method: a load of 10 kg (HV 10) was used for the surface hardness measurement and a load of 50 g (HV 0.05) for the in depth profiles.

For the practical testing, the punches were installed in a standard production line at the Brano, a.s. plant, working with austenitic stainless steel sheet (0.07 % C, 17.5 % Cr, 8 % Ni) with a thickness of 4 mm. The lifetime of the punches was evaluated from the number of punched pieces. After the tools failed they were subjected to a failure analysis using scanning electron microscopy.

3 RESULTS

The thickness of the nitrided layer developed using processing route II was approximately of 30 µm. This corresponds to the recommended values according to [5]. The microstructure of the layer (**Figure 1**) consisted of tempered martensite, original carbides and the nitrogen-rich Fe_4N phase (**Figure 2**) that induces the optical contrast with the non-nitrided substrate. The layer formed at 530 °C for 120 min differs from that shown in **Figure 1**, mainly in the thickness, but also in the formation of a high-nitrogen portion containing Fe_3N nitrides (ε-phase), see **Figure 3**. Both nitrided layers can be divided into two areas. Close to the surface there is the "white" region, where the enhanced-nitrogen reduced-carbon contents were determined [8] and the cause for the carbon redistribution, which results in the occurrence of a dark region in the micrograph was discussed. Close to the surface the microhardness of the nitrided layer formed at 500 °C was 1400 HV 0.05, but it dropped down noticeably with an increasing distance from the surface. This fact is reflected in the surface hardness, which was 1084 HV 10. No difference in the near-surface microhardness was found for the specimen processed at a higher temperature. However, the microhardness values decreased very slightly with the

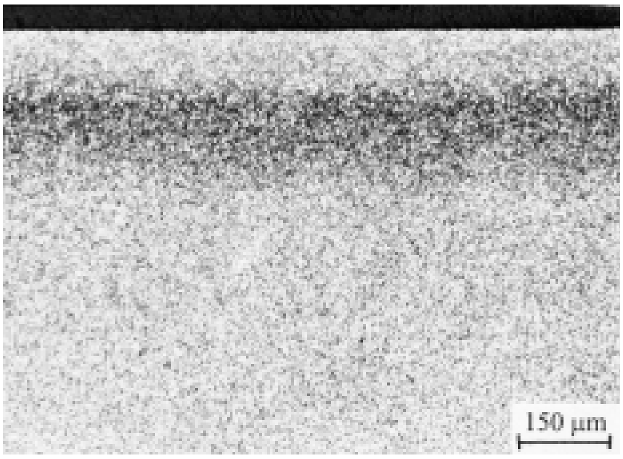

Figure 1: Microstructure of the P/M M2 tool steel, nitrided at 500 °C for 60 min

Slika 1: Mikrostruktura orodnega jekla P/M M2, nitriranega 60 min pri 500 °C

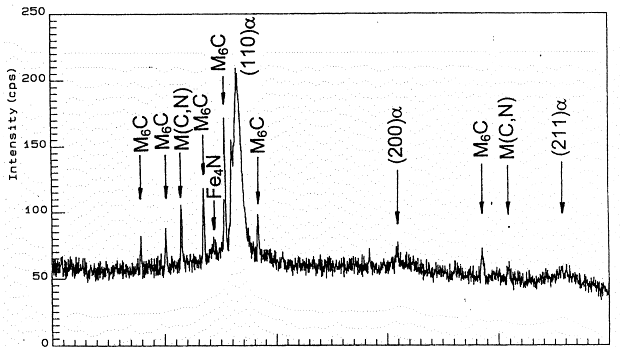

Figure 2: Diffraction patterns for the nitrided layer formed at 500 °C for 60 min

Slika 2: Primer difrakcije nitrirane plasti, nastale v 60 min pri 500 °C

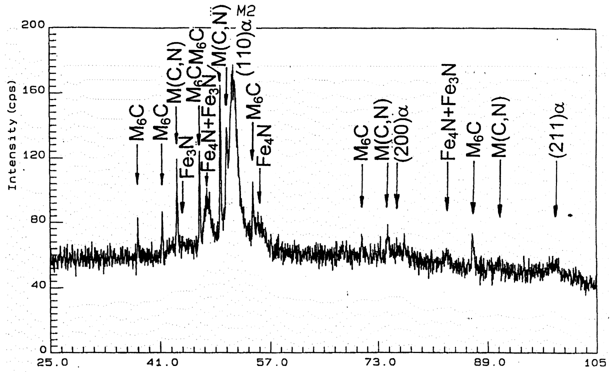

Figure 3: Diffraction patterns for the nitrided layer formed at 530 °C for 120 min

Slika 3: Primer difrakcije nitrirane plasti, nastale v 120 min pri 530 °C increasing distance from the surface. As a result, the surface hardness significantly exceeded 1200 HV 10.

As reported previously, plasma nitriding induces an improvement in the wear resistance of P/M AISI M2 tool steel [9]. Plasma nitriding can thus play an important role in some industrial processes, where high strength and/or thick sheets are worked and, as a result, the tools are heavily loaded and wear stressed. However, the tools are not only wear stressed, the following loadings can also occur: global compressive stress in the tool axis direction, and fatigue and local tension when the tools are loaded by forces acting perpendicularly to the tool axis. The last of these is met very often in industrial processes and can lead to failure before any wear occurs.

Such a cause of failure was observed before with many materials (12 % Cr ledeburitic steel, M2 conventionally produced, etc.). The tools were mostly damaged by chipping and complex breakdown of the punch was also observed. In any case the lifetime did not exceed 2750 pieces. Such situation is undesirable because it results in frequent downtimes in the production line and increased production costs and the attempt to minimise early tool failure were a logical step.

First, tools without a plasma nitrided layer were tested. Compared to the materials used before, the lifetime was prolonged to 3100 pieces. This improvement was for economic reasons insufficient for the end-user. To achieve economic efficiency, at least 7500 pieces without tool failure were required. Plasma nitriding led to the following results: the diffusion layer formed at 500 °C (for 60 minutes) induced a prolonging of the average lifetime to 65,000 pieces. The use of a higher temperature and a longer processing time led to a less-significant improvement - the average lifetime was of 46,000 pieces. There can be no doubt that the application of plasma nitriding has a strongly positive technical and economic impact on production.

Figure 4 presents an overview of the fracture surface of a tool without the plasma nitrided layer. The fracture was initiated at more places on the surface and taking into account the number of pieces made, a fracture mechanism like "low-cycle fatigue" led to the damage to the tool. The detail micrograph, **Figure 5**, shows that a slight plastic deformation occured at the fracture surface. This is rather surprising at first sight; however, the symptoms of plastic deformation of ledeburitic steels were observed previously, and confirmed in ref. [12].

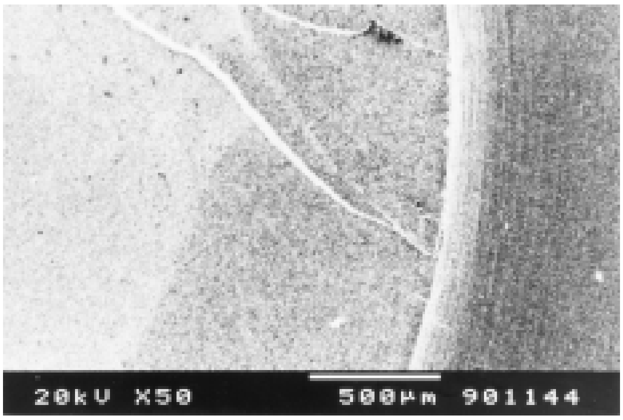

Figure 4: Fracture surface of a non-nitrided tool
Slika 4: Površina preloma orodja, ki ni bilo nitrirano

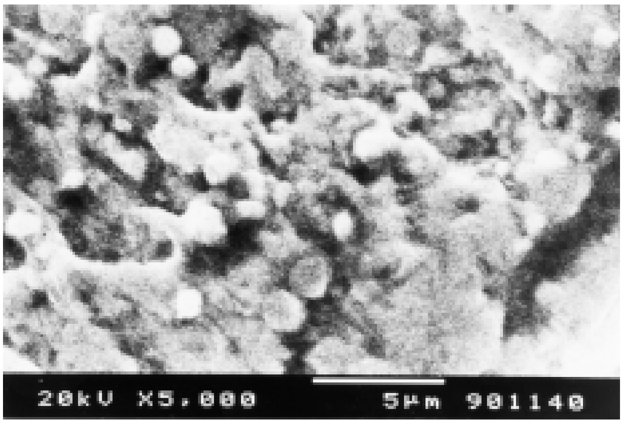

Figure 5: Detail
Slika 5: Detajl

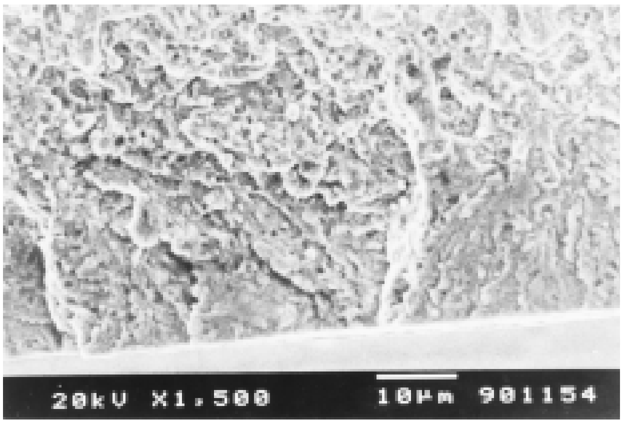

Figure 6: Fracture surface of nitrided tool (500 °C, 60 min) - close the surface
Slika 6: Površina preloma nitriranega orodja (500 °C, 60 min) - blizu površine

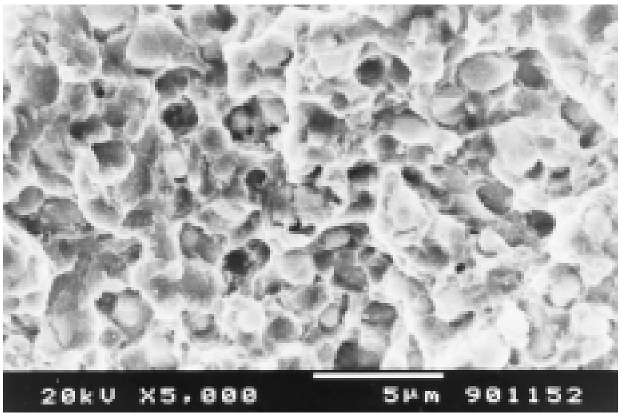

Figure 7: Fracture surface of nitrided tool (500 °C, 60 min) - core matrix
Slika 7: Površina preloma nitriranega orodja (500 °C, 60 min) - osnova

The situation in the case of the plasma nitrided punch (processing route II) differs clearly from that of the non-nitrided punch. It is evident that the fracture is initiated at a certain depth below the surface and not at the surface (**Figure 6**). This depth corresponds to approximately 75 % of the total thickness of the nitrided layer. The fracture surface of the core material can be described in a similar way to that of the non-nitrided material, i.e. the material behaves as a brittle steel with local plastic deformation, **Figure 7**.

4 DISCUSSION

The reason why the fracture of the nitrided material is not initiated at the surface, but below, can be explained as follows: Fox-Rabinovich [5] and Holemar [11] independently reported that compressive stresses mostly occur in the nitrided surface of M2-type high-speed steel. They can reach up to 750 MPa during the initial stage of the nitriding, i.e. when only the saturated α-solid solution is formed. The appearance of the first nitride particles is connected with a decrease of compressive stresses, which is initially slow and after a certain holding time, when the phase Fe_3N starts to grow, the decrease is noticeable.

In most of the sheet-working processes the tools are not only loaded with axial forces, but also with forces acting perpendicularly to the tool axis (this is very probable with respect to the nature of the industrial process where the punches were used). The occurrence of tensile stresses in the near-surface region can thus also be expected. They may be compensated by the compressive stresses due to the nitriding, when the material was nitrided up to a certain depth below the surface. At a larger distance from the surface, the compressive stresses are too low, or are not present, and the tool is only loaded by the cyclic tensile loading. This may lead to the initiation of the fracture below the surface, and after a significantly longer period than in the case of the non-nitrided material.

The reason why the application of higher nitriding temperature and/or longer time is deleterious can also be explained with the consideration above - the higher the nitrogen saturation the lower the compressive stresses. Therefore, the compensation of the cyclic tensile strain is smaller for the tools processed at 530 °C than for those nitrided at 500 °C and, as a result, the lifetime is shorter.

5 CONCLUSIONS

1) Nitrided layers exhibit a structure containing Fe_4N and Fe_3N nitrides, tempered martensite and carbides. The presence of these phases increased the hardness to a level significantly exceeding the values typical for the non-nitrided M2-grade steel.
2) Plasma nitriding of the punches that work in difficult conditions leads to a substantial improvement in the lifetime of the tools.
3) This may be due to the occurrence of compressive stresses in the nitrided layers. These stresses can compensate for the radial loading, leading to the cyclic tensile stresses in the near-surface region.
4) The last assumption was demonstrated by an analysis of the fracture surfaces, which exhibit the so-called "ductile low-energetic fracture" typical for heavily loaded ledeburitic tool steels. On the other hand, the surfaces differ significantly in terms of the initiation mechanism: in the case of non-nitrided tools, the fracture is initiated directly on the surface, while the initiation is shifted to a certain depth below the surface for the nitrided steel.

Acknowledgements

The authors wish to thank the Ministry of Education, Youth and Sport of Czech Republic for the financial support of the E2718 project.

6 REFERENCES

[1] P. Jurči, Research and development of high alloy tool materials prepared by rapid solidification in gas atomisation of the melt, Research report, SVÚM Prague, 1996 (In Czech)

[2] P. Jurči, Powder Metallurgy, 41 (**1998**) 269-27

[3] H. Jones, J. Mater. Sci., 19 (**1984**) 1043-1076

[4] P. Jurči, P. Stolař, J. Suchánek, Potentialities for prolonging the life - time of cold work tools, Proc. Conf. METAL 99, Vol. IV. Ostrava, Czech Republic, 1999, 78-85

[5] G.S. Fox - Rabinovich, Wear, 160 (**1993**) 67-76

[6] M. Van Stappen, et al., Mater. Sci. Engng. A140 (**1991**) 554-562

[7] A. Leonhardt, I. Endler, Ceramics International, 21 (**1995**) 421-425

[8] P. Jurči, J. Suchánek, P. Stolař: Effect of various plasma nitriding procedures on surface characteristics of P/M high speed steel, Proc. of the 5th ASM Heat Treatment and Surface Engineering Conference in Europe, Gothenburg, Sweden, 2000, 197-208

[9] P. Jurči, J. Suchánek, P. Stolař, F. Hnilica, V. Hrubý: Surface characteristics of duplex coated P/M M2 grade high speed steel, Proc. of the European PM 2001 Congress, Nice, France, 2001, 303-308.

[10] Proceedings of the European PM 2001 Congress, October 22-24, 2001, Nice, France, 303 - 308.

[11] P. Jurči, P. Stolař, J. Suchánek: Effect of various plasma nitriding procedures on surface characteristics of P/M ledeburitic tool steels, Proc. of the 14th Int. Conf. on Surface Modification Technologies, Paris, France, 2000, 233-239.

[12] A. Holemář, V. Hrubý, V.: Plasma nitriding in practice, SNTL, Prague, 1989 (In Czech)

[13] P. Jurči, F. Hnilica, J. Suchánek: Improvement of wear resistance of the P/M Cr - V ledeburitic steel by duplex coating, Proc. of the Int. Conference Deformation and Fracture of Structural PM Materials, High Tatras, Slovakia, 2002, 294-301