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Modulus of Rupture Evaluation on P/M Cold Worked Tool Steels Submitted to Gas Nitriding

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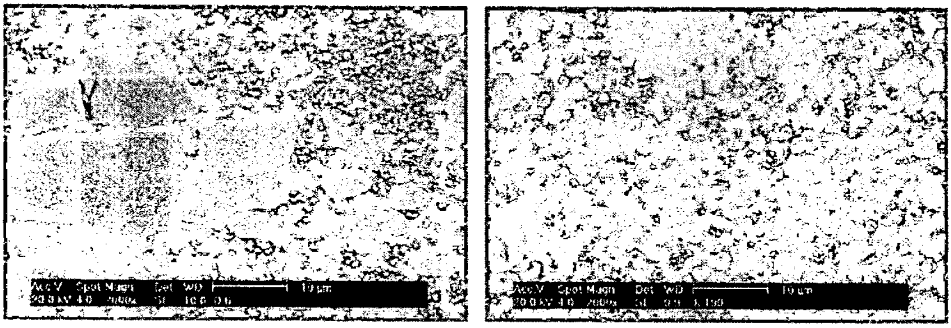
Abstract: Cold work tool steels have been successfully applied for decades. Modern Powder Metallurgy (P/M) techniques are considered important for tool steels in general since high alloy projects could be development. An important example is P/M cold work tool steel X 220 Cr Mo V 13 4. Although tribological properties are quite important to be studied, mechanical properties are also relevant since tools are submitted to high static and dynamic loads. Present work has investigated the effect of gas nitriding process on modulus of rupture, in modern cold work tool steel above mentioned. Nitriding process applied has affected mechanical properties significantly, reducing by 30 % of modulus of rupture decreasing obtained from 4-point bending test. The high C content of those modern steels induces ε mono-phase composition in compound layer. Although ε nitride, due to its high strength, increases some tribological properties, ductility of those tool steels is clearly affected.

Introduction

In general, tool steels in the industrial manufacturing processes appear with economic importance. An important family is called cold work tool steels, mainly applied as forming dies, gages, cold extrusion, mandrels, forming and bending rolls.

Tool steels present a structure showing carbides embedded in a tempered martensitic matrix, resulting in a hard and wear resistant material.

It is established that tool steels produced from conventional Metallurgy routes, present limitation in contents of C and alloy elements, since during solidification, segregation and a coarse structure tend to be developed. That coarse microstructure leads to a brittle tool steel, as well as uneven wear resistance property. Powder Metallurgy (P/M) is the most significant development in the manufacturing methods for tool steels. In fact, P/M techniques appear as a solution for complex tool steels production, as high C and Cr alloys [1]. P/M tool steels due to metallic atomization and rapid cooling might present a fine, even and isotropic structure. Fig. 1 presents conventional (a) and P/M (b) structure of cold work tool steels.



(a) (b)

Fig.1: Structures of cold work tool steels. (a) 2.1%C, 12%Cr, 0.2%V from conventional metallurgy and (b) P/M tool steel 2.3%C, 12.5%Cr, 4%V.

Fine and homogeneous carbides distribution, which is presented in Fig. 1 (b), is remarkable concerning P/M tool steel, leading to an isotropic structure. Since tool steel performance is directly related to wear resistance, they benefit from nitriding processes. This thermochemical treatment introduces nitrogen into surfaces of steels in order to increase surface hardness, improving wear resistance in the tool surface. Although nitriding has been applied for many years, this process has been largely applied lately due to advantages, as low cost and low temperatures during the process [2].

Gas nitriding is usual process applied for cold work tool steels, consisting by exposure the tool to ammonia gas atmospheres at temperatures between 500 to 580 °C. As a result, a thin layer is formed consisting of fine and dispersed nitrides particles in a tempered martensite. This layer produces very high surface hardness (up to HV 1200) for cold work tool steels, increasing wear resistance. The obtained layer is subdivided in two zones: external (compound or white layer) and internal (diffusion layer), both presented in cold work tool steel shown in Fig. 2.

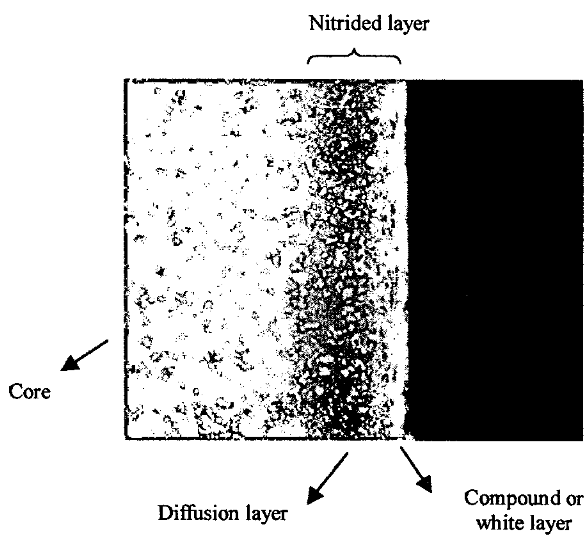


Fig. 2: Nitrided layer showing compound and diffusion zones after gas nitriding of cold work tool steel AISI D6.

In general, compound layer for steels is dominantly composed of $Fe_{2\text{-}3}N$ (ε), Fe_4N (γ') or even a mixture of both (ε + γ'). Although Fe-N equilibrium diagram would suggest that γ' phase should always be present, some factors as C content in the alloy, can favor ε nitride nucleation, resulting a mono-phase compound layer developed [3,4]. Due to its high strength, ε phase determines some tribological properties, whereas diffusion layer, which is associated to γ' phase presence, principally determines fatigue properties [5].

Present paper investigates gas nitriding effect on mechanical properties of a modern P/M cold work tool steel, named K190 (Böhler trade mark) with composition 2.3%C – 12.5% Cr – 1.1%Mo – 4%V, DIN 1.2380, X220CrMoV134. This effect was evaluated after submitting samples to 4-point bending test, suitable for hard materials as quenched and tempered cold work tool steels [6]. A correlation between layer structure and mechanical properties are also aimed.

Experimental Procedures

Annealed samples of P/M K 190 were prepared to four-point bending test, using usual mechanical machines as lathe, mill, cylindrical grinder and electro-erosion. Samples measurements were 5mm outside diameter and 60 mm length. After that, they were submitted to quenching / tempering and gas nitriding. Five samples were prepared and submitted to each chosen condition. Samples were quenched and tempered by: preheating 400°C for 2 hours and 840°C for 15 minutes; austenitizing at 1100°C for 10 minutes; cooling 500°C for 30 minutes then salt bath cooling; tempering at 560°C for 60 minutes then oil cooling. Those parameters have been chosen to obtain matrix hardness of HV 780 up to 820, suitable values according to the steel producer. Gas nitriding was *Deganit®* process (NH_3 / N_2 / CO_2), 6 hours at 570 °C, usual parameters for cold work tool steels.

As-quenched and tempered condition samples, not submitted to gas nitriding process, were tempered for the second time in order to achieve same structure and hardness condition of nitrided samples.

For the four-point bending test, a tension-compressing-bending machine has been used, with a special adapter for four-point load application. Nitrided microstructures were observed using Philips XL 30 Scanning Electron Microscopy (SEM), after Nital 2% etching. Vickers hardness measurements on nitriding layer were carried out in Shimazu equipment, using load of 0.050 Kg.
X- ray diffractograms were performed in Rigaku equipment.

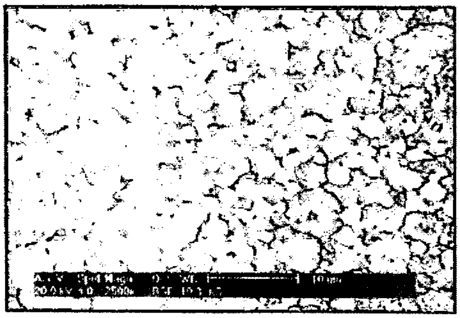
Results and Discussion

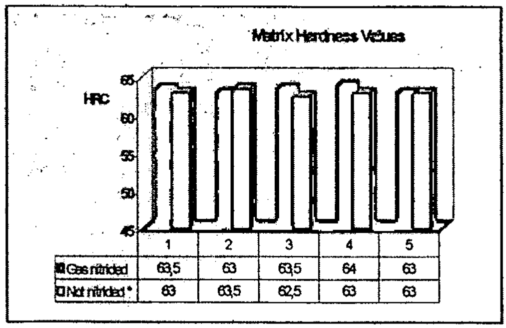
Table 1 shows the chemical composition of P/M cold work tool steel K 190, DIN 1.2380, X220CrMoV134.

Table 1: P/M K 190 chemical composition (wt%)

%C	%Si	%Mn	%P	%S	%Cr	%Mo	%V
2.31	0.41	0.36	0.027	0.026	12.23	1.15	3.99

As mentioned, samples not submitted to gas nitriding process were tempered for the second time, in order to obtain iso-matrix samples with those nitrided. Since gas nitriding (Deganit process) and tempering treatment are performed at similar temperatures, nitriding process has a tempering effect on the steel matrix. Fig. 3 shows typical core structure after gas nitriding and samples not submitted to this process, as well as hardness values.





(a) (b)* Twice tempered in order to achieve iso-matrix.

Fig. 3: (a) typical matrix structure observed by SEM; (b) matrix hardness values.

Typical matrix for both situations are represented in Fig. 3(a), consisting in a fine martensitic tempered structure with M_7C_3 (gray) and MC (black) carbides. It was possible to achieve matrix with same hardness level for samples nitrided and not nitrided, as shown in Fig. 3(b). That point was important to compare layer effect on mechanical property of cold work tool steel K 190.

Fig. 4 shows typical layer obtained for P/M K 190 after gas nitriding process.

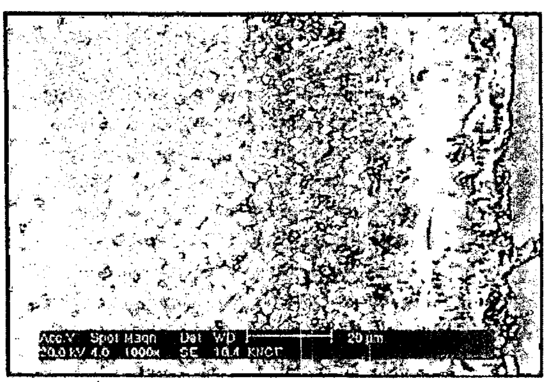


Fig. 4: Typical layer after submitted K 190 to gas nitriding (Deganit process).

P/M K 190 submitted to Deganit process has shown about 20 μm of compound layer as seen in Fig. 4. Total nitrided layer was estimated in about 100 μm due to carbide concentration, lately confirmed by Vickers hardness profile as shown in Fig. 5.

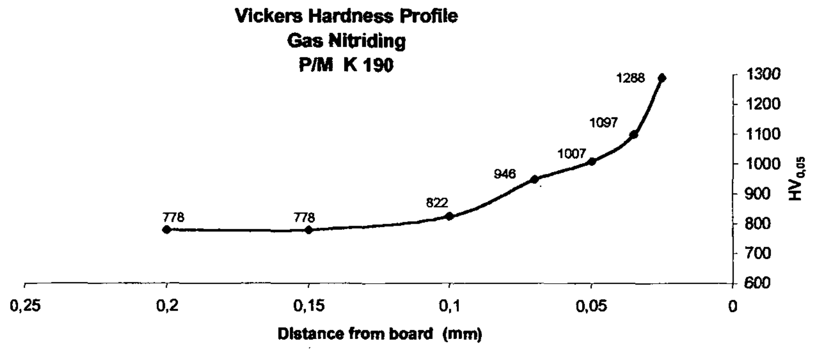


Fig. 5: Layer Vickers hardness profile.

P/M K 190 has shown hardness increasing of HV 500 from core to harder area measured in the layer. After that, samples submitted and not submitted to gas nitriding process, but presenting same matrix hardness level, have been tested in 4-point bending equipment. Fig. 6 shows modulus of rupture obtained for both conditions.

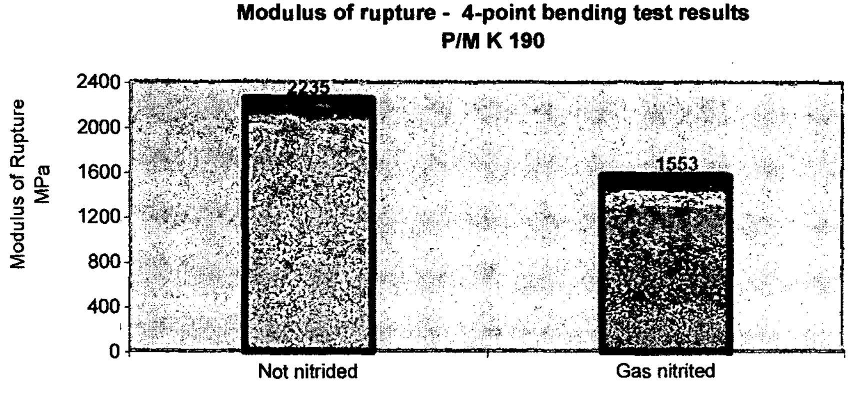


Fig. 6: Four-point bending test results for P/M K 190 samples.

Samples of P/M cold work tool steel K 190, submitted to gas nitriding process (Deganit) have presented about 30% of reduction in modulus of rupture, showing an important decreasing in mechanical property. In order to detect phase information, nitrided samples were submitted to X ray diffraction. The results are shown in Fig. 7:

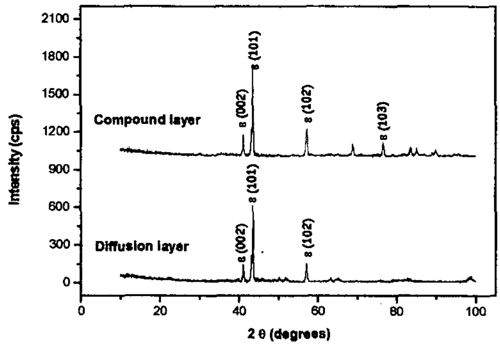


Fig. 7: X ray diffractograms from compound and diffusion layer.

No presence of γ' phase in both studied areas was detected. Although compound layer usually consists of a mixture of ε and γ' nitrides, high carbon contents have favored ε phase formation. That is the case concerning present P/M alloy, which is considered high C, Cr and V cold work tool steel.

Conclusions

Usual gas nitriding process applied in P/M X 220CrMoV134 has reduced mechanical property, measured in 4-point bending test. Reduction of 30% in average of the modulus of rupture was detected. X-rays diffractograms have revealed just ε nitrides probably due to high C contents of mentioned alloy. P/M routes are considered the most significant development for tools steels, giving chance to produce complex alloys with high carbon and carbide formers. Although modern P/M techniques lead to isotropic structures, concerning compound layer formation after gas nitriding, ε mono-phase zone might be responsible for even more reduction on mechanical properties of tools.

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