Peening

Surface nanostructuring induced by NanoPeening®: Effect on the nitriding of high-Cr tool steel X38CrMoV16

Introduction

In the early 2000s, it was discovered that the nanostructuration of metallic materials could be induced by mechanical treatments [1-2]. Mechanical nanostructuring was born, and soon the pages of many scientific and technical papers (including MFN) were filled with articles exploring its potentials [3-4]. Since then it has grown into a whole family of treatments generally referred to as "Severe Plastic Deformation" (SPD) processes. They were listed and extensively described in a two-volume handbook published last year [5]. Some of them affect the whole bulk while others focus on the surface. This is the case of NanoPeening®, a process developed and patented by Winoa [6-7]. Through repeated impacts of blasted media, the surface is plastically deformed with a very high rate and accumulates huge stresses. The resulting strains are such that a great density of dislocations appear to accommodate them. They organize into networks that further evolve into boundaries partitioning the initial grains into much finer, nanometric grains. Their size of around 30nm right under the surface gradually increases towards the core of the material.

This gradient in grain size translates into a gradient in hardness, which on the top surface can reach extremely high values, making the treated parts much more resistant to abrasive wear.

Another consequence of the change in microstructure is the increase in grain boundaries density. Because they act as channels for the diffusion of species, the efficiency of treatment such as nitriding is promoted. It was already observed in the case of austenitic stainless steels (304L, 316L) and tool steels (X38CrMoV5) [8].

Here the case of the grade X38CrMoV16 was studied, whose chemical composition (as controlled using an optical emission spectrometer) is given in Table 1. Its high Cr-content allows it to be considered both as a stainless steel and as a tool steel. As tool steel, it is subjected to wear and finds benefits in treatments able to increase its resistance. On the other hand, its high level of alloying elements makes it somewhat difficult to nitride deeply. It was found out that NanoPeening® performed before nitriding enables almost double the enriched layer.

Experimental procedure

The X38CrMoV16 was provided as a bar (30mm in diameter) in the quenched and tempered state and cut into cylindrical samples having a thickness of 20mm. Some of the latter were processed by NanoPeening® on one flat side; some were kept un-treated as reference. As many projection-based processes, NanoPeening® tends to roughen the surfaces; so, some samples

received a post-treatment intended to smoothen it. The study also aimed at checking whether or not this finishing modified (positively or negatively) the effect of NanoPeening® on nitriding.

The adopted nomenclature is summarized in Table 2. "N" may also be referred to as "reference sample".

The samples were gas-nitrided at 520°C for 50h in an industrial installation. Cross-sections were prepared by cutting, plugging and polishing. The nitrided structures were observed by Optical Microscope (OM) and Field Emission Gun-type Scanning Electronic Microscope (FEG-SEM) after chemical etching using Villela reagent. The diffusion profiles were registered by Glow Discharge Optical Emission Spectroscopy (GD-OES). Hardness profiles, using a 50g loading, completed the characterization.

Results and discussion

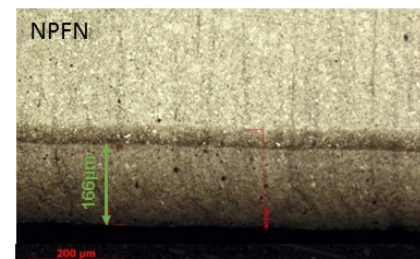The pictures taken with the Optical Microscope are shown in Figure 1. They

Figure 1: OM pictures of N, NPN and NPFN samples in cross-section

Element	C	Cr	Mo	V	Mn	P	S
Wt%	0.4	16.5	1	0.1	0.7	≤0.03	≤0.03

Table 1: Chemical composition of steel grade X38CrMoV16

Sample name	N	NPN	NPFN
Treatment(s) received	Nitriding	NanoPeening® + nitriding	NanoPeening® + finishing + nitriding

Table 2: Nomenclature used in the study
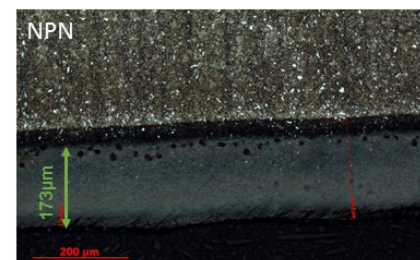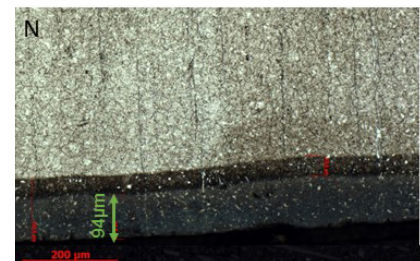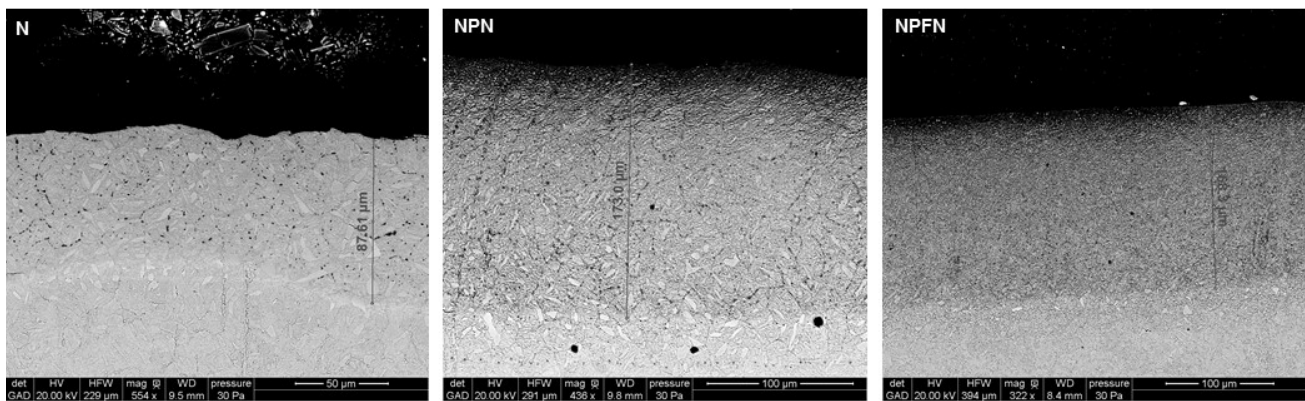
Figure 2: SEM observations in BSE mode (GAD) of N, NPN and NPFN samples in cross-section

allow for an easy comparison of the nitrided layers, which correspond to the light grey part at the bottom (marked off by the grey arrows). The most striking difference lies in the thickness, superior in the case of the NPN and NPFN samples that had both received the NanoPeening® pre-treatment.

The SEM pictures were taken using Back-Scattering Electrons (BSE) detector to highlight, by chemical phase contrast, the N-enriched layers. It is thus possible to observe their morphology and to assess their thickness more precisely than with OM. As can be seen in Figure 2, in all 3 cases (N, NPN and NPFN) the surface is free from compound layer and the interface between the diffusion layer and the bulk is clearly defined, making it possible to evaluate the thickness of the latter. On the N sample, it is not homogeneous but was assessed to less than 88µm at the thickest. In both NPN and NPFN samples, the diffusion layers are not only more regular but also thicker: 173 and 168µm respectively. It might be noticed that the magnifications used slightly differ from each other: this choice was made on purpose in order to show a global view of the layer.

The lower thickness of the diffusion layer in N sample may be ascribed to the many nitrides and carbo-nitrides that appear as black dots on the picture. They form during the process of nitriding by precipitation of nitrogen atoms with the alloying elements, in particular chromium, which is present either in solid solution or as carbides and has a high affinity to nitrogen; but molybdenum and vanadium are also prone to

form nitrides. This phenomenon slows down or even blocks the progression of nitrogen into the material. In the NPN and NPFN samples that were first processed by NanoPeening®, the increase in grain boundaries density - that comes along with the decrease in grain size - provides nitrogen species with many more paths to diffuse through the surface: they are less likely to meet Cr and as a result can move deeper.

Nitrogen concentration as a function of depth was plotted in the graph of Figure 3. A focus on the first 3 µm is shown in the insert. In all three cases the diffusion profile first sharply drops in the first micrometers, then slowly decreases and finally almost stabilizes. Obvious differences however can be seen in the maximum nitrogen content measured at the surface. In GD-OES the

very first points are often subjected to artefacts and should be discarded. This taken into account, the profiles show that in the NPN and NPFN samples the top surface is more enriched than the N sample: 13.7%, 12% and 11%, respectively. Still more remarkable is the concentration measured in depth: close to 5% at 30µm for NPN and NPFN samples while in the reference sample it has gone down to 0.5%.

In terms of hardness, lower values could have been expected on the surface of NPN and NPFN samples, due to a dilution effect caused by the deeper diffusion of nitrogen, but it is definitely not the case: the profiles displayed in Figure 4 show that similar levels around 1300 HV are reached on the top surface for all samples. The differences in nitrogen diffusion, however, have direct
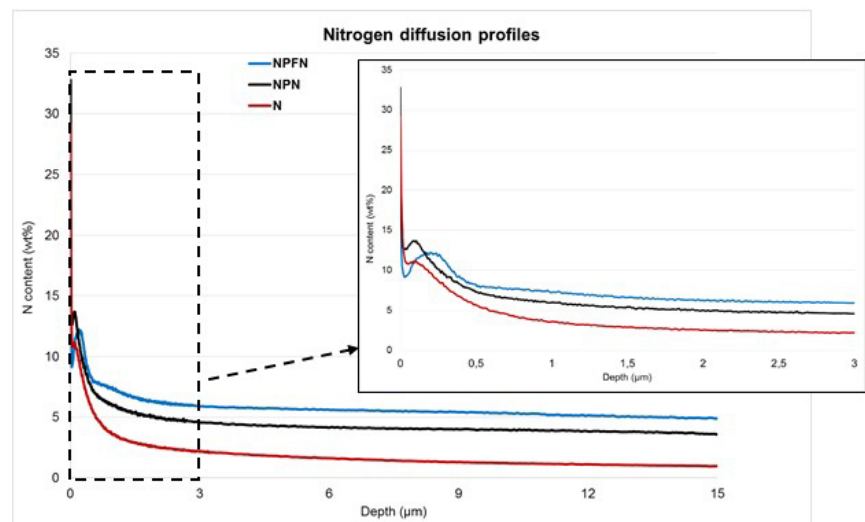

Figure 3: Nitrogen concentration against depth in N, NPN and NPFN samples, analyzed by GD-OES
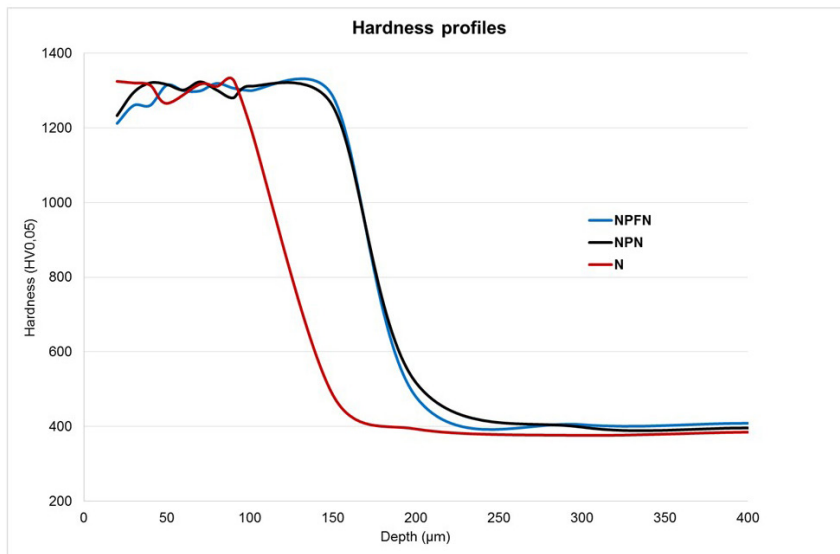
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Figure 4: Hardness against depth for N, NPN and NPFN samples; Vickers indenter / 50g loading were used

consequences on the hardened depth: in the N sample the decrease in hardness starts at 100µm to reach 400 HV at 150µm, while for both NPN and NPFN samples it remains at the maximum value until 150µm.

Such grades as X38CrMoV16 find their main applications in the die casting of low melting point metals (tin, zinc, lead, magnesium, aluminum, copper and its alloys) where they are used for the molds, cores, and inserts. Such tools are quite expensive and their life time is an issue. To make it longer, surface treatments like nitriding can be implemented. The increase in hardened thickness and surface hardness obtained by the association with NanoPeening® could further improve the durability of these parts.

Conclusion

The effect of surface nanostructuration by NanoPeening® on nitriding was studied on a steel grade containing a high amount of chromium. With such grades it is usually difficult to get thick nitride layers because Cr tends to trap nitrogen by forming precipitates. It was found that the extreme refinement of the grains makes it possible to almost double the thickness of the N-enriched layer, from less than 90µm to more than 170µm. Moreover, the surface hardness does not get any lower. It was also shown that the finishing treatment aimed at reducing the roughness is neutral with respect to the influence of NanoPeening® on nitriding.

These results could find interesting applications in the field of tools dedicated to the die casting of low melting points metals but also to cold working and stamping where such steel grades as X38CrMoV16 are used.

References

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Acknowledgement

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