TOOL STEEL QRO90 SUPREME AFTER FOUR TYPES OF HEAT TREATMENT

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Abstract

Paper deals with tool steel of QRO90 Supreme type that was treated by four different heat treatments. Firstly only laser quenched, afterwards laser quenched with followed-up plasma nitridation, the third material was only nitrided by plasma and the last one was nitrided by plasma and subsequently laser quenched. Microstructures with purity and micro-hardness in different depths under surface were investigated, so that to find the optimised heat treatment leading to the most favourable mechanical properties of the studied steel with taking tribological properties into consideration. Obtained results were compared mutually including results from material after conventional quenching. Laser quenched material with followed nitridation shapes up as the most optimised heat treatment variant

Keywords: QR090 Supreme tool steel, laser quenching, plasma nitriding, microhardness, microstructure

1.   INTRODUCTION

Tool steels of new generation should show longer life time, higher hardness that can be reached using laser quenching (LQ) and/or plasma nitriding (PN). The LQ makes diffusion process in surface layers faster and the PN is able to make the treatment time by 2-8 hours shorter. By combination of both HT processes it is possible to increase material hardness, wear level and elastic modulus of nitrided layer [1]. Simultaneously, porosity has been decreased and hardness has been increased without thickness change. Combination of both mentioned HT has been object of research [1] and tool steel of QRO90 Supreme is one of materials belonging to the target group.

Tool steel of QRO90 Supreme is applied for stamping and mould casting. Both technologies are characterized in that the tool gives final product shape and are plenteously used for complicated products in shape without any other working. Such exact tools are expensive for production and must be made from special steel type. Steel used for hot forming must show a resistance against high temperatures and simultaneously against pressures and abrasion [2,3]. All tool steels for hot working are used in quenched and tempered state and tool steel must keep a high heat strength, plasticity, toughness, heat conductivity, creep strength, resistance against tempering and low heat expansion up to 550/650 °C [4,5]. For preservation of such properties up to higher temperatures effect of secondary hardness is used that can be reached by addition of carbides formed elements as Cr, W, Mo and V [5,6] and making softening lower.

The aim of presented work is study of surface, resp. under surface layers of QRO90 Supreme tool steel after different combinations of surface laser quenching and/or plasma nitriding and finding an optimised way of surface heat treatment for mentioned steel with high mechanical parameters and finding the impact of hardness on tribological properties.

2.   EXPERIMENTAL

In dimension the same five samples of QRO90 Supreme tool steel (AISI H10) after different heat treatments (HT) for microstructure analyses were labelled as it follows. The BM represents basic material without any HT, sample labelled 1 laser quenching (LQ) up to 0.7 - 0.9 mm depth only, while sample 2 was after laser quenching up to 0.7 - 0.9 mm depth with followed plasma nitriding up to 0.2-0.3 mm depth (LQ+PN). Sample 3 was nitrided by plasma up to 0.2-0.9 mm depth (PN) unlike sample 4 that was nitrided by plasma and subsequently laser quenched up to 0.7-0.9 mm depth (PN+LQ). Conditions for heat treatments were in all cases the same. All samples were metallographically grinded and polished for study of their surfaces, resp. under surface areas, including purity, microstructure with grain size, hardness HV0.2 (LECO 2000) and diffusional layers. For qualitative linear chemical analysis SEM QUANTA 450 with EDAX Octane FEG was used. For light microscopy device Olympus IX 70 was at disposal. Chemical composition of QRO90 Supreme tool steel was followed (in wt%): 0.38C, 0.3Si, 0.75Mn, 2.6Cr, 2.25Mo and 0.9V. With exception of sample 1 (LQ) tribological tests were also carried out by means of PIN-on-DISC method. Wear coefficient W ranged from 1.9 mm^3/N·m (BM) to (2.48 - 2.9 mm^3/N·m - all heat treated materials with the same character of abrasion, with even ruts and acoustical effects).

Table 1 Results of micro hardness HV0.2 of investigated material BM after LQ, LQ1+PN, PN and PN+1LQ

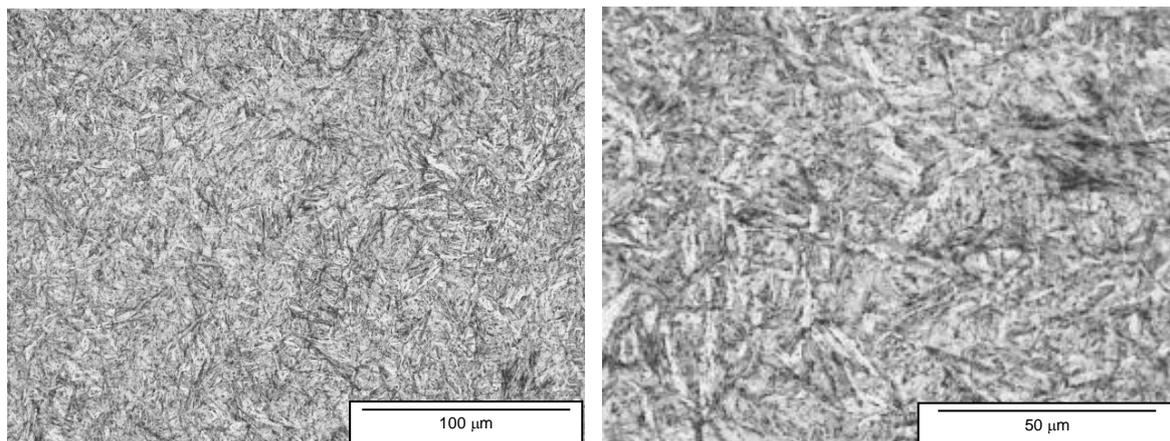| Sample after heat treatment /distance from surface (mm)	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
BM	595	586	587	570	589	600	578	576	593	584
LQ (1)	681	644	619	614	600	581	488	494	481	484
LQ+PN (2)	968	952	648	618	599	599	572	555	531	561
PN (3)	880	723	694	581	590	572	581	563	580	547
PN+LQ (4)	933	659	560	544	547	539	535	523	531	524

Figure 1 Microstructure of basic material (BM) – two magnifications

3.   RESULTS AND THEIR ANALYSIS

In BM globular oxides of D1 and DS0.5 types were found. Hardness ranged from 570 to 600 HV0.2, on average it was 586 HV0.2 as it from **Table 1** can be seen. According ARA-diagrams [2] microstructure should be martensitic-bainitic one, however only martensitic microstructure with fine martensitic needles was detected, as it **Figure 1** demonstrates and results from work [7] support. Average austenite grain size corresponded to 7 ± 4 µm, including also the highest sporadic occurrence of 15 µm grains.

Microstructure of LQ material shows **Figure 2**.¨In laser zone ultrafine microstructure formed. White part of LQ layer is named molten zone and under that one heat affected zone (HEZ) can be detected. The LQ zone shows high homogeneity, abrasion and corrosion resistance. Hardness of that part showed 681 HV0.2. In depth of approx. 710 μm (**Figure 2a**) the HEZ was observed with important fall of HV, under level of BM, as it from above mentioned BM results and **Table 1** follows. Molten zone turned to martensitic heat affected zone. In molten zone extremely ultrafine martensitic microstructure with ultrafine carbides (of Mo and V type) was seen. On the surface approx. 10 $\pm$ 1 μm thick Fe oxide layer formed by $Fe_{1-x}O$, Fe_3O_4 and Fe_2O_3 as an effect of high temperature and/or contamination of environment was observed [8].
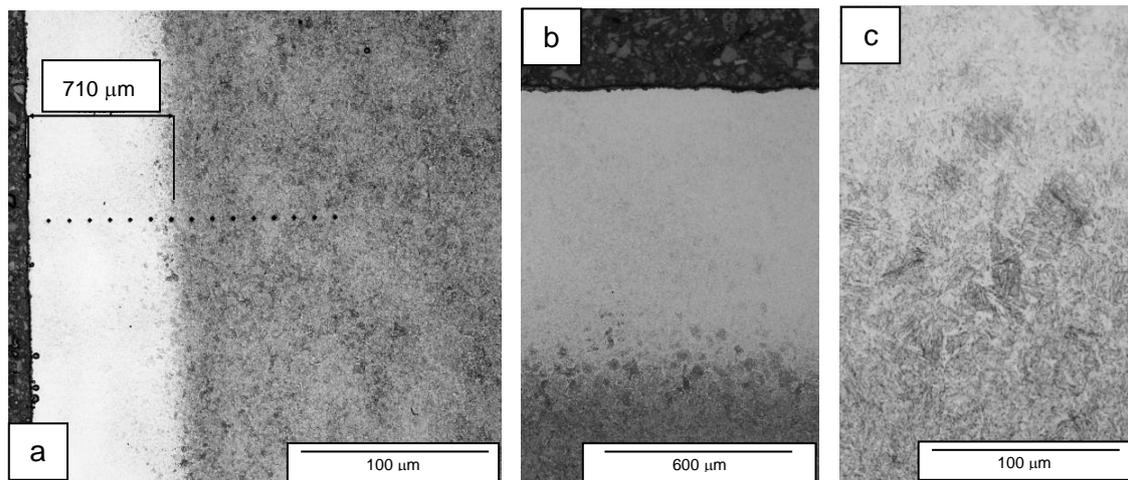

Figure 2 Microstructure of LQ material a) formed layers under surface, b) molten zone in detail, c) transition of molten zone into the HAZ and the BM
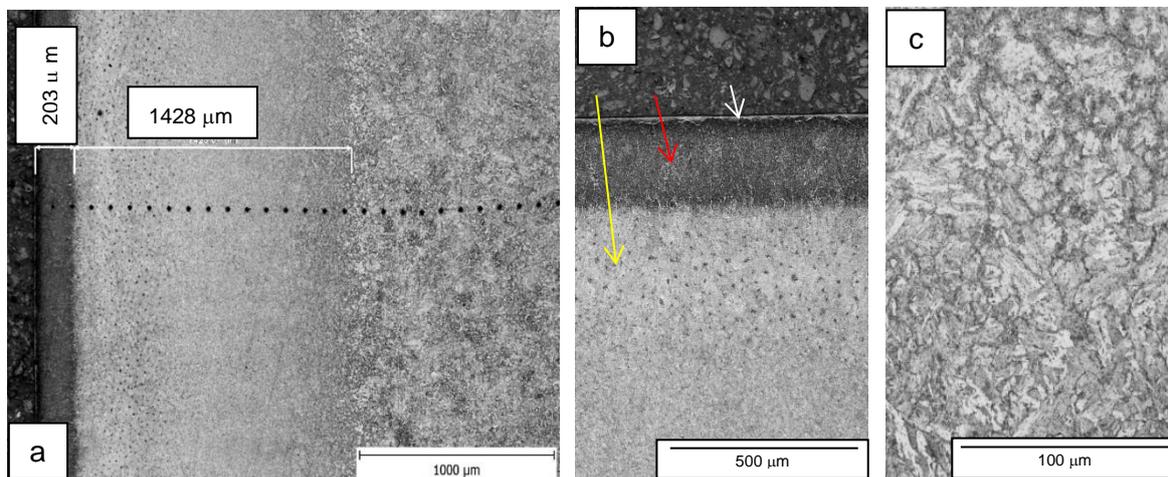

Figure 3 Microstructure of LQ+PN material a) formed layers under surface - 203 μm represents mixed layer, 1428 μm layer includes all deeper detected zones b) white layer (white arrow), mixed layer (red arrow), diffusion zone (yellow arrow), martensite and HAZ bellow diffusion layer c) transition to the basic material from undersurface formed zones

Figure 3a shows course of layers in LQ+PN material being in agreement with results of work [9]. Thickness of mixed layer under surface was approx. 203 μm subsequent diffusion nitride layer turned to laser quenched one and this layer to the HAZ being totally 1428 μm in thickness as it **Figure 3a** demonstrates. Next layer represents unaffected material as it from **Figure 3a** follows. Maximal hardness was found in depth of 100 μm

(see **Table 1**), in which also maximal volume fraction of N was detected (5.8 wt%), together with ε and γ phases as it linear chemical analysis revealed. Up to 200 μm distance away surface fall of HV0.2 was detected, where N level was stabilized on 3 $\pm$ 1 wt%. In distance of 300 μm hardness was stabilized on the lowest level as well as N portion (1.5-2 wt%).

Under mixed layer a net of carbides and/or carbide nitrides can be observed. Nitrided layer (15 $\pm$ 1μm)¨is formed by Fe and N and this layer can be seen as a white one on the polished section and can be influenced by composition of nitridation atmosphere. In case of addition of C to mentioned atmosphere intermetalic phase ε - $Fe_{2-3}N$ is formed. This layer corresponds to range from 8 to 30 μm in thicknes. In given case mentioned layer was 15 μm. During standard gas nitridation more phase layers of ε-$Fe_{2-3}N$ and γ-Fe_4N are formed. [10, 11]. Dark zone under above described layer represents diffusion part, when N atoms occupy the bcc lattice interstitial positionsins [10]. Diffusion layer is noticeably thicker (see yellow arrow in **Figure 3b**). The grain size of the HAZ oscillated about 18 μm.
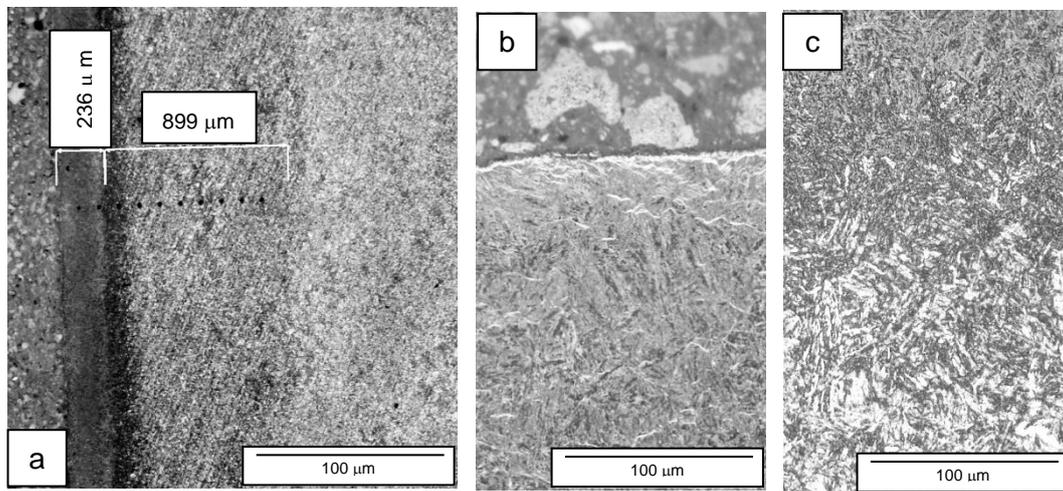

Figure 4 Microstructure of **PN** material a) formed layers under surface, b) transition white layer-diffusion zone in detail, c) transition from nitrided layer to BM

Figure 4 shows material microstructure after PN. In 100 μm depth maximal hardness (880 HV0.2) was detected. This one was increasing, up to 300 μm faster and then was practically stagnated, lightly above the basic material level, as it from **Table** follows. Investigated steel falls into class of medium or high alloyed materials being able to reach hard layers, however thickness of diffusion zone is never higher than 0.5/0.6 mm. Steels with Cr-Mo-V addition have achieved higher lifetime (max. 250 %) generally [11]. The PN sample showed the highest portion of N on the surface and directly under surface (over 9 wt%). Each 0.5 wt% N decrease represented hardness fall by 50 HV0.2 approximately. In distance of 30-35 μm only 3 wt% of N was revealed, while in distance of 350 μm away surface it was 1.5 wt%. That level was practically stable up to 1 mm depth. Diffusion layer corresponded to 236 $\pm$ 20 μm (**Figure 4a**), white layer to 5-9 μm (**Figure 4b**). Average microstructure grain size of diffusion layer was 8 μm, while transition to basic material revealed 12 μm grain size. Generally, properties of nitrided surface depend on applied temperature, because with decreasing temperature also hardness is going down, however not on nitriding time, because that takes 15 hours approximately. Optimized tribological properties are the reason [11].

Figure 5 represents microstructure of PN+LQ sample. Micro-hardness of that sample went down significantly. Higher micro-hardness was in depth of 100 μm. After transition from diffusion zone to LQ one micro-hardness was stabilized on the half level and that corresponded to the BM. In this heat treatment variant nitrogen concentration was higher than in case of LQ+PN sample. In depth of 100 μm micro-hardness was 933 HV0.2

afterwards it was 659 HV0.2 in depth of 200 μm. With further nitrogen fall micro-hardness was evenly going down up to 560/540 HV0.2 in depth of 300/400 μm. White layer length corresponded to 8 ± 2μm (**Figure 5b**), while diffusion zone was 316 μm in thickness (**Figure 5a**). Micro-hardness of this sample was lower as it was already mentioned above. The reason has been nitrides formation after nitriding and their follow-up dissolution after LQ procedure and/or a change their interatomic distances after the LQ. The white layer has been also thinner (1.9 times) than in case of above mentioned sample LQ+PN. The grain size of diffusion zone microstructure corresponded to 6 μm, unlike the layer of LQ (HAZ) microstructure being 12 μm approximately.
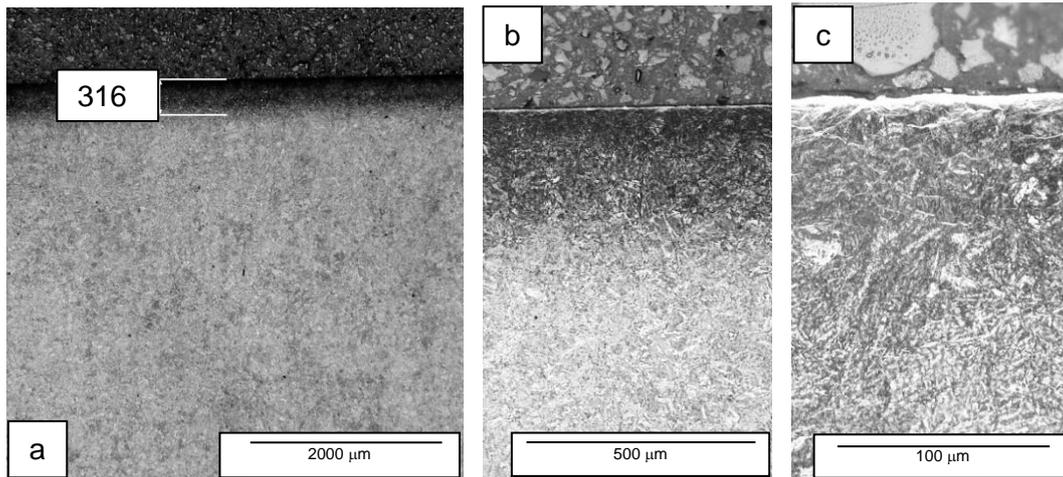

Figure 5 Microstructure of PN+LQ material a) formed layers under surface, b) transition white layer-diffusion zone, LQ, c) white layer and diffusion zone in greater detail
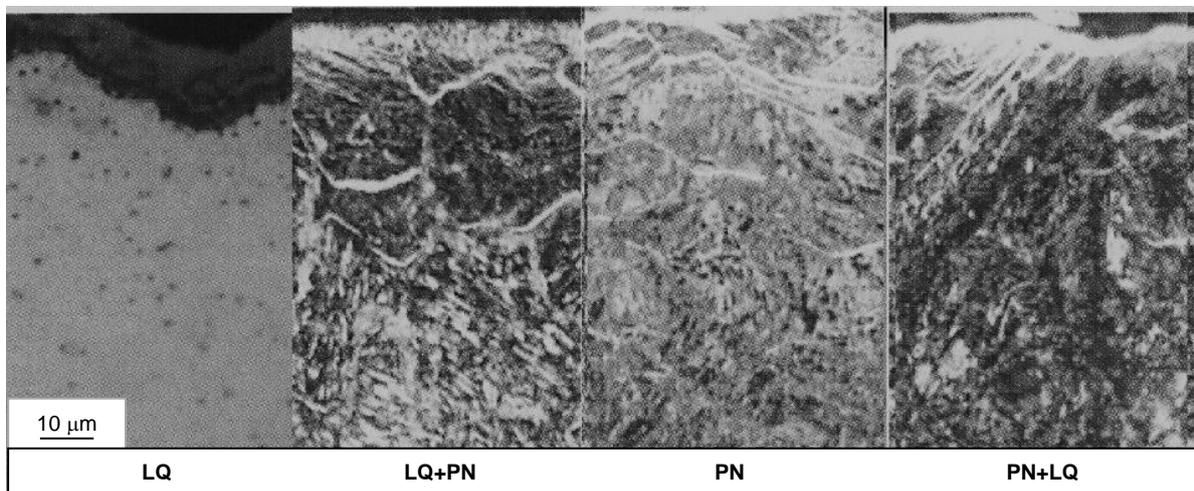

Figure 6 Comparison of formed microstructures after 4 heat treatment variants on the surfaces, resp. directly under surfaces (magnification is the same in all cases)

From the results follows, the most favourable variant of heat treatment could be taken LQ+PN material showing high hardness after LQ treatment by means of the surface/under surface white layer of extremely fine martensite of 22 μm in local thickness (**Figure 6**) being further hardened after the PN treatment at temperature of 480 - 500 °C during 15 hours leading to a change of primary microstructure and to nitrides, resp. carbonitrides formation of fine size as it from **Figure 6** follows. The primary under surface hardness (681 HV0.2) after LQ process was increased by 42 % (968 HV0.2) – see **Table 1.** The second best results the PN+ LQ variant revealed on the surface, resp. under surface up to 100 μm depth. In given case the primary micro-

hardness of 880 HV0.2 was changed slightly on 933 HV0.2 (see **Table 1**) representing 6 % growth only, however in depth of 300 μm fall of micro-hardness (560 HV0.2) was found already. This week effect of double heat treatment is not economical. It is necessary to state, all variants of heat treatments showed practically the same level of tribological results. In case of LQ+PN wear coefficient W = 2.62 mm^3/N·m, while the PN+LQ variant revealed lightly better result (W = 2.48 mm^3/N·m).

4.   CONCLUSION

Beside basic treatment representing conventional quenching four heat treatment variants were applied on surface and under surface of tool steel of QRO90 Supreme including laser quenching (LQ), further LQ with plasma nitriding (LQ+PN), plasma nitriding (PN) and PN +LQ. Results were compared mutually. The lowest surface resp. under surface micro-hardness was generally detected in basic material (600 HV0.2).

The highest micro-hardness was found on the surface resp. directly under surface in case of LQ+PN variant. The second highest micro-hardness on the surface resp. directly under surface corresponded to the PN+LQ variant, however in depth of 300 μm approx. 40 % fall of micro-hardness was already revealed what can be elucidated by dissolution of primary formed nitrides after laser quenching in more slowly cooled region. The other in order was PN material, while the LQ one showed the lowest micro-hardness on the surface (681 HV0.2). From point of tribological tests the best was basic material (wear coefficient W = 1.9 mm^3/N·m), while the other treated materials showed practically comparable level of W = 2.48-2.9 mm^3/N·m).

ACKNOWLEDGEMENTS

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