

IMPROVEMENT OF THE WEAR BEHAVIOUR OF HIGHLY-LOADED COMPONENTS AND TOOLS BY MULTI-COMBINED SURFACE TREATMENT

Gundis Grumbt^{1,*}, Rolf Zenker^{1,2}, Heinz-Joachim Spies¹, Rainer Franke³, Ingrid Haase³

¹ TU Bergakademie Freiberg, Institute of Materials Engineering, Gustav-Zeuner-Str. 5, 09599 Freiberg, Germany

² Zenker-Consult, Johann-Sebastian-Bach-Str. 12, 09648 Mitteida, Germany

³ IMA Materialforschung und Anwendungstechnik GmbH, Wilhelmine-Reichard-Ring 4, 01109 Dresden, Germany

* corresponding author: tel.: +49 3731 39-2898, fax.: +49 3731 39-12898, e-mail: grumbt@ww.tu-freiberg.de

Resume

These multi-combined surface treatments lead to a significant improvement in load-supporting capacity. Critical load values of cohesive failure measured by scratch tests are tripled when compared to the individual treatment of PVD, and increased by at least 20% in comparison to the duplex treatments of EBH+PVD or PN+PVD.

The metallurgical compatibility of the single treatments is essential for the success of combined treatments. Material-specific limitations are defined, which exclude failure due to crack initiation, the occurrence of retained austenite, and tempering effects.

Based on the model wear-test assembly block-on-cylinder, it was proved, that the specific wear rate of multi-combined treated specimens is reduced about 20-50% while wear of counterpart components is decreased as well. The triplex surface heat treatment introduced opens up new prospects for highly-loaded components and tools.

Available online: <http://fstroj.uniza.sk/journal-mi/PDF/2014/01-2014.pdf>

Article info

Article history:

Received 15 March 2013

Accepted 8 September 2013

Online 8 January 2014

Keywords:

Combined surface treatment;

Electron beam hardening;

Plasma nitriding;

Physical vapour deposition;

Tool steel.

ISSN 1335-0803 (print version)

ISSN 1338-6174 (online version)

1. Introduction

High-performance tools for plastics processing, e. g. forming and injection-moulding tools, are subjected to complex loading conditions characterised by enormous differences and steep gradients between demands on material surfaces and bulk materials. Due to ever-increasing demands on these materials, conventional treatment technologies like vacuum hardening, thermochemical treatments, such as nitriding and boronizing, the deposition of hard layers by PVD or CVD, or thermal surface hardening using induction, electron beam or laser beam techniques, are not sufficient for realising optimised property profiles. A promising way to solve this challenge is to generate surface treatment combinations (duplex- or even triplex

surface treatments) which create property complexes that are not attainable by employing only one of these individual treatments. For basic knowledge on duplex surface treatments see Ref. [1-6].

Since the beginning of the 1980s numerous duplex surface technologies have been investigated and, to varying extents, successfully implemented in industrial applications, in particular nitriding with subsequent PVD coating (see, e. g. [7, 8]). Combinations are known, too, including EB surface-treatment technologies. The duplex treatments of nitriding + EB surface hardening and vice versa have been used as standard series applications for tools and components since 1996 [9].

It is well known that wear protection coatings, such as hard, wear-resistant coatings

deposited by physical vapour deposition (PVD) improve surface properties, but not without an additional heat treatment of the base material before or after PVD. In the case of using bulk heat treatment, large-scale and high-cost processing equipment is normally necessary. Furthermore, treatment of the component as a whole is accompanied by risks in relation to distortion [10] and other undesirable effects.

Electron beam surface hardening is a progressive, economical and ecological alternative to bulk heat treatment technologies, because the heat treatment can be limited to the highest-loaded areas only and energy deposition is exactly definable [9, 11-15]. Because of the very short interaction time and the process-related treatment in vacuum conditions, even a subsequent heat treatment of nitrided and hard-coated steels is possible without undesirable changes in composition, structure and properties of the layers [16-21].

For a successful combination of two or more individual treatments, it is essential to know the effects of interaction between the surface treatment and material, i. e. the material's reactions as a result of the sequence of treatments. Therefore, this paper deals with the basic relationships between combinations of hard protective coatings from PVD based on CrN, with plasma nitriding and EB surface hardening as a fundamental requirement for future industrial application. Based on results of the duplex treatments of plasma nitriding with

electron beam hardening (PN+EBH and EBH+PN), the material-specific limitations for successful triplex surface treatment technologies (PN/EBH/PVD) in differently-sequenced steps of processing are defined.

2. Experimental

For these investigations the steels 40CrMoV13-9 (DIN 1.8523) and X54CrMoVN17-1 (M340 Isoplast[®]) were used as substrate materials in a previously heat treated (hardened and tempered) state. The chemical composition of these steels is shown in Table 1.

EBH was carried out in the electron beam facility (type K26-15/80) at an acceleration voltage (U_a) of 60 kV and an output power of 12 kW. A high-speed beam deflection technique was used for energy transfer. Because of its good deflectability, the electron beam moves with frequencies of up to 100 kHz within a previously-programmed energy transfer field, so that an isothermal energy distribution is guaranteed. This results in track-shaped hardening with the adjacent areas remaining almost unaffected. For the present investigations, the track width was limited to 20 mm and the transformation depth varied between 0.2 mm and 1.5 mm.

Nitriding was carried out as plasma nitriding. The process parameters depend on the sequence of treatments ($T_N = 420-600$ °C; $t_N = 8-24$ h; composition of processing gas: 15-70% N_2 + 85-30% H_2). The formation of a closed compound layer was avoided.

Table 1

Chemical composition of steels

Element (wt%)	Fe	C	Si	Mn	Cr	Mo	V	others
40CrMoV13-9	main	0.40	< 0.4	0.5	3.2	1.0	0.2	-
X54CrMoVN17-1	main	0.54	0.45	0.4	17.3	1.1	0.1	+N

The diffusion layer thickness was varied between 15-200 μm .

For PVD coating, the commercially-available multilayer Cr/CrN coating system MAXIT[®] CrN-modified (Sulzer Metaplas) was chosen. The specific characteristic of the so-called 'CrN-mod' is a thin, glass-like Cr₂O₃ top layer, with very low degree of surface energy. This property causes good wear resistance with an extremely low tendency of adhesion to polymers and elastomers. The layer thickness generated was in the range of 3.2-3.5 μm .

The results were characterised with regard to the effects of nitriding and/or hard coating on the EB-hardened or nitrided layer using light microscopy and mechanical tests of the layer-matrix compounds (hardness depth profiles HV 0.05, surface hardness HV 1, critical loads in scratch tests). For wear testing, a block-on-cylinder model wear test assembly was used. The combination-treated specimen (base body) was tested against an iron-based hard facing alloy Reiloy 121 counter body, containing 10% Cr, 6% Mo, 4% Ni, 3,8% B, and 2% C, with particles of glass fibre in between at $F_N = 50 \text{ N}$, $v = 0.2 \text{ ms}^{-1}$ and $T = 200 \text{ }^\circ\text{C}$ for $t = 2 \text{ h}$.

3. Results and Discussion

3.1. Duplex treatments

3.1.1. Plasma nitriding + EB hardening

A typical metallographic cross section of a two-ply layer resulting from the duplex treatment PN+EBH can be seen in Fig. 1a. The effect of the combination of nitriding and EB hardening is illustrated by the hardness depth profiles (Fig. 1b). Because of the formation of nitrogen-enriched martensite in the previously nitrided layer, higher hardness occurs after PN+EBH when compared to the state following the individual treatments.

The thickness of the martensitic transformed layer increases with increasing energy input during EBH (from EBH1 - lowest energy input to EBH3 - highest energy input).

The formation of small cracks near the surface (Fig. 1c) must be noted, however, this occurs if the energy input exceeds a certain amount.

Because of the high chromium content, the high alloy tool steel has an excellent chemical affinity to carbon and nitrogen. As a result of plasma nitriding, the carbon of the chromium carbides is displaced by nitrogen and is enriched interstitially below the diffusion line of nitrogen. For this reason, the formation of retained austenite is supported during the subsequent EB hardening. High hardness of the PN+EBH layer only occurs with small energy inputs (Fig. 3, PN+EBH1). With increasing energy input, the amount of retained austenite (the white layers in Fig. 2) increases, too. This causes a pronounced minimum in hardness, which is clearly shown in Fig. 3, PN+EBH3, that limits the application of this technology to hardening depths lower than SHD (550 HV) < 0.5 mm.

3.1.2. EB hardening + plasma nitriding

Subsequent nitriding of EB-hardened specimens of the steel 40CrMoV13-9 required low nitriding temperatures, because nitriding as the second treatment step had a tempering effect on the hardness of the EBH layer. Nevertheless, a nitriding temperature of only 420 $^\circ\text{C}$ / 24 h, which resulted in an acceptable nitrogen diffusion depth (0.1 mm), caused a decrease in hardness of about 150 HV 0.05 (Fig. 4a). The achieved hardness depth profile was characterised by a two-stage gradient, with high case hardness in the thin layer enriched with nitrogen and a thicker EBH supporting layer underneath.

With regard to PVD hard coating, it was necessary to avoid the formation of a closed compound layer. The formation of iron nitrides at the surface is controllable by the nitrogen content in the processing gas. During nitriding with a concentration of 70% N, a compound layer of approx. 4 μm was formed (Fig. 4b), whereas nitriding with decreased nitrogen

content (e.g. 15% N, Fig. 4c) avoided the formation of a compound layer without a significant reduction of the diffusion layer thickness.

Thermal stability is not critical for the high alloy tool steel X54CrMoVN17-1. However, high internal stresses occur during nitriding which cannot be dissipated by lower nitriding temperatures, and cracks are thus formed in the nitrided layers. According to experience, the tendency towards crack formation decreases with decreasing nitriding depth. Therefore, the nitriding depth was limited to 20-40 μm . On the other hand, higher nitriding temperatures impede crack formation, because the yield strength of the nitrided layer decreases at temperatures in the range of 530-550 $^{\circ}\text{C}$.

The usage of higher nitriding temperatures is beneficial: With increasing energy input during EBH and, therefore, increasing hardening depth, the high alloy steels strongly tend towards forming retained austenite. Subsequent nitriding at temperatures in the third annealing range of the steel causes the transformation of retained

austenite. In Fig. 5b, the metallographic cross section of the sample nitrided at 570 $^{\circ}\text{C}$ shows untempered martensite (white). At the same time a tempering of the EBH layer has to be accepted, so that the plateau-like hardness depth profile changes into one with a continuous hardness gradient (Fig. 5a).

3.2. Triplex treatments

3.2.1. EB hardening + plasma nitriding + PVD coating

Steel 40CrMoV13-9

Fig. 6 presents the results of hardness measurements after single, duplex and triplex treatments involving EBH, nitriding and PVD coating of the steel 40CrMoV13-9. Because of its limited tempering stability, the hardness in the EBH layer was reduced by subsequent thermal treatments (nitriding and PVD hard coating). The subsequent hard coating did not have any effect on the previously nitrided steel because both treatments exhibited good compatibility with the time-temperature cycles.

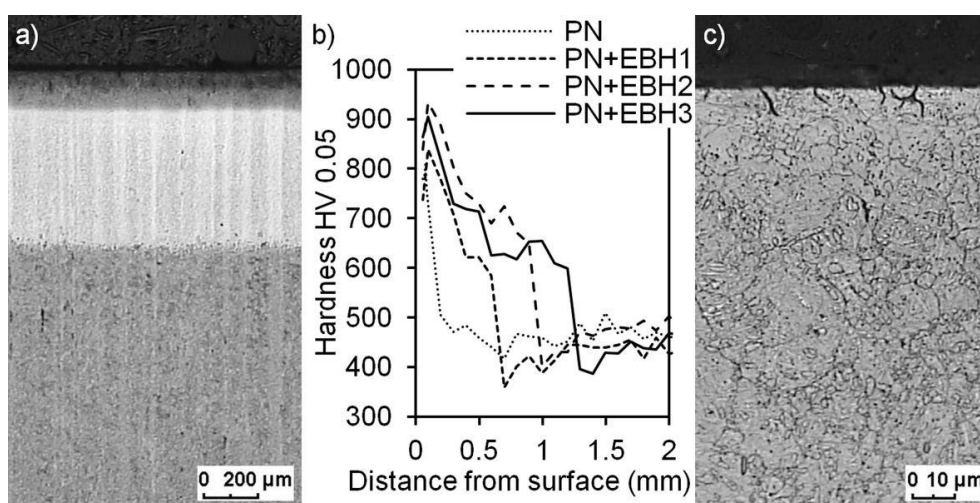


Fig. 1. Results of duplex treatment PN+EBH of the steel 40CrMoV13-9,
 a) Metallographic cross section after treatment PN+EBH1,
 b) Hardness depth profiles of nitrided and subsequently EB hardened specimens,
 c) Microcracks occurring at higher energy input (EBH3).

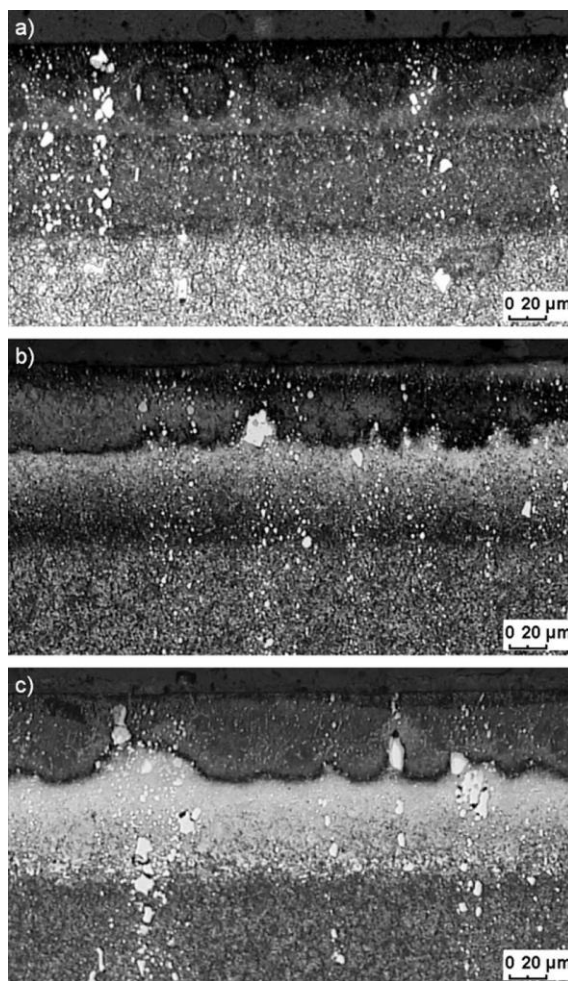


Fig.2. Metallographic cross sections after the duplex treatment PN+EBH of the steel X54CrMoVN17-1 with varying energy input during EBH, a) PN + EBH1, b) PN + EBH2, c) PN + EBH3.

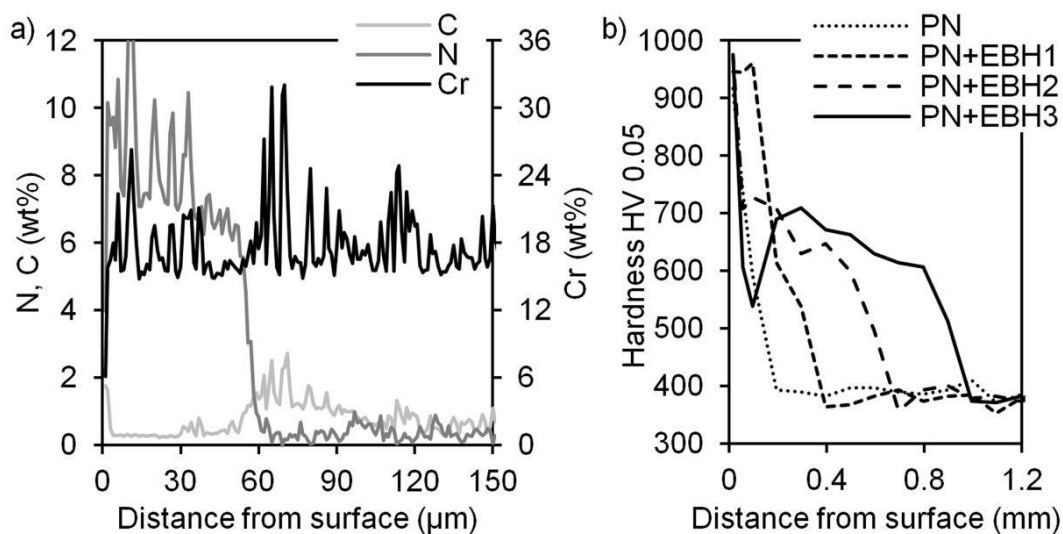


Fig. 3. Influence of interstitial elements on the formation of retained austenite during EBH of nitrided specimens of the high alloy steel X54CrMoVN17-1.

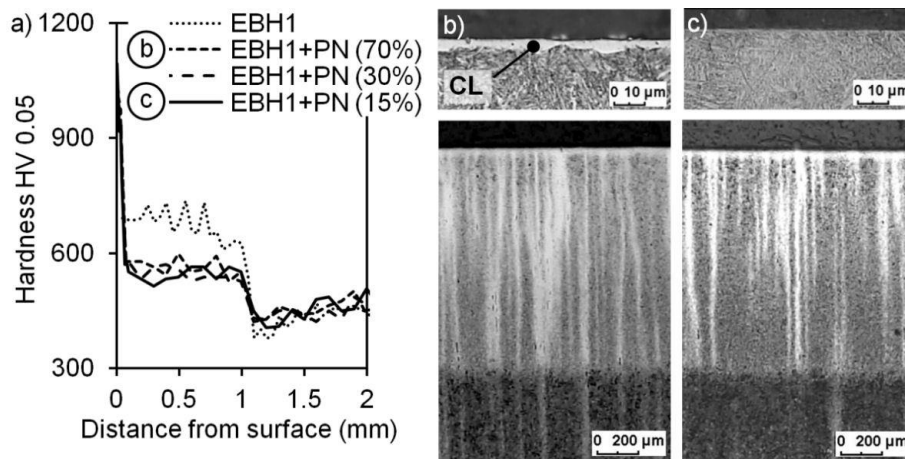


Fig.4. Influence of subsequent plasma nitriding with differing nitrogen contents on the hardness depth profile and structure of EB-hardened specimens of the steel 40CrMoV13-9, a) hardness depth profiles, b) formation of compound layer (CL), c) no (closed) compound layer.

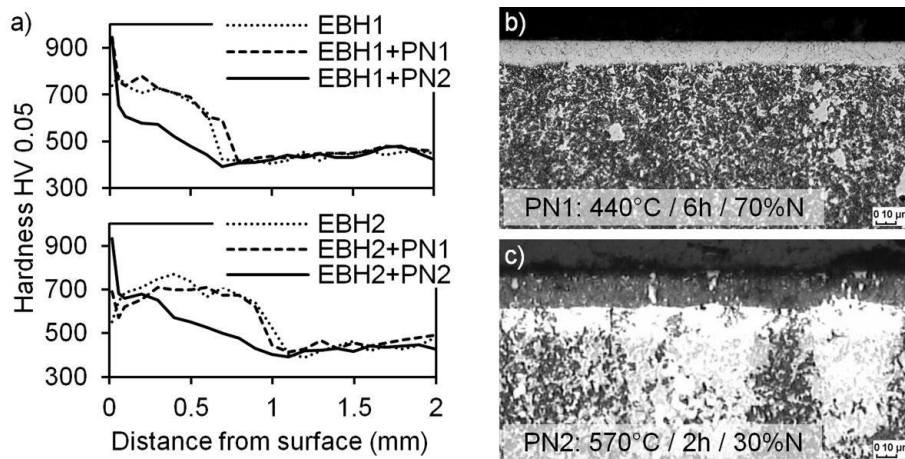


Fig. 5. Hardness depth profiles and metallographic cross sections of X54CrMoVN17-1 after EBH+PN at different nitriding temperatures, a) hardness depth profiles, b) metallographic cross section: EBH2+PN1 ($T_N = 440\text{ }^\circ\text{C}$, $t_N = 6\text{ h}$, 70% N), c) metallographic cross section: EBH2+PN2 ($T_N = 570\text{ }^\circ\text{C}$, $t_N = 2\text{ h}$, 30% N).

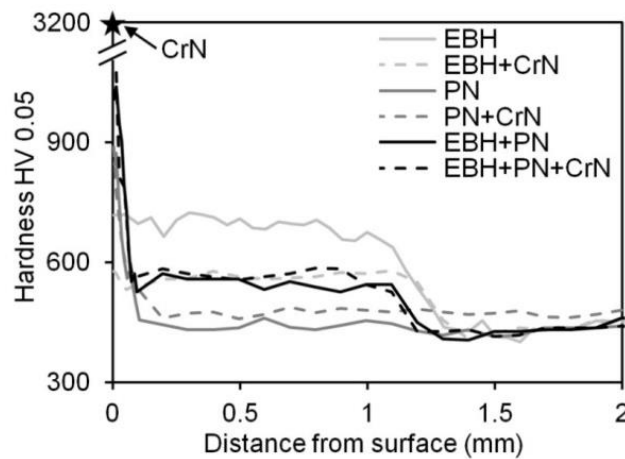


Fig. 6. Hardness depth profiles of single, duplex and triplex treatments of the steel 40CrMoV13-9.

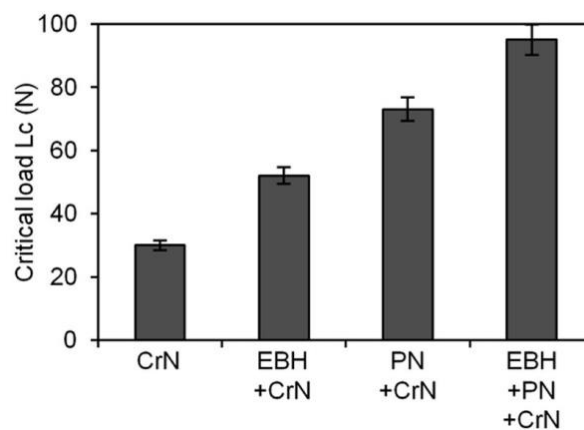


Fig. 7. Critical load for first adhesive failure (scratch test) after hard coating for duplex and triplex treatments (steel 40CrMoV13-9).

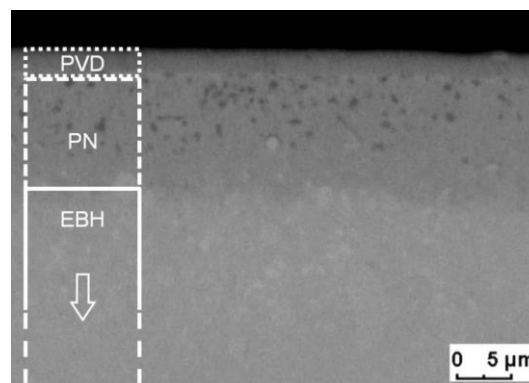


Fig. 8. Triple-graded surface layer of the steel X54CrMoVN17-1.

In Fig. 7, the critical loads for first adhesive failure are given, which show a good correlation to the base material's load support capacity for the hard coating. If the base material has only been hardened and tempered, and is coated with CrN, the critical loads are very low. Using a duplex treatment with EBH or nitriding, a significant improvement in load support capacity occurred. Best results in scratch tests were achieved for the triplex-treated specimens. The best load support capacity for the hard coating was exhibited by the graded properties of the EB-hardened and nitrided surface layer.

Steel X54CrMoVN17-1

A typical three-ply surface layer of the high alloy steel X54CrMoVN17-1 is shown in Fig. 8 and quantified on the basis of hardness depth profiles (Fig. 9). Considering the single

treatments first, the EB-hardened layer exhibited a good depth effect, though the surface hardness was relatively low. The very thin nitrided layer of this steel (20-30 μm) exhibited excellent case hardness (ca. 1100 HV 0.05). The combination of both treatments resulted in a cumulative curve from both single treatments, so that an optimal load support for the final coating occurred.

Fig. 10 confirms this tendency by means of the results of model wear tests. Compared to the normally-used M390 steel grade (chemical composition of PM steel Böhler M390 Microclean[®]: 1.9% C, 0.7% Si, 0.3% Mn, 20% Cr, 1% Mo, 4% V, 0.6% W), the wear rate after triplex treatment was reduced by half. It should be pointed out that the wear rate of the test counterpart was reduced, too.

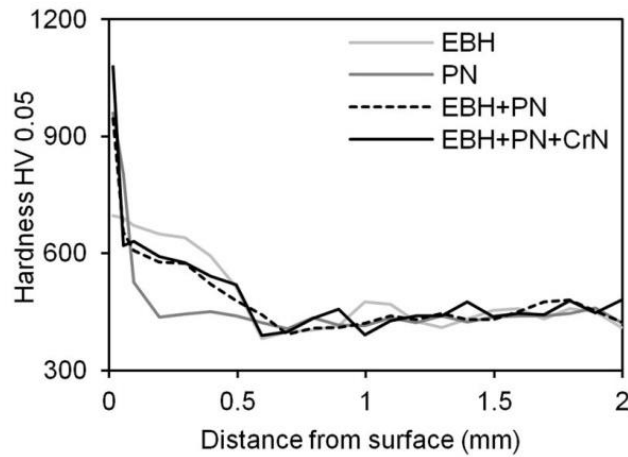


Fig. 9. Hardness depth profiles of single, duplex and triplex treatments (steel X54CrMoVN17-1).

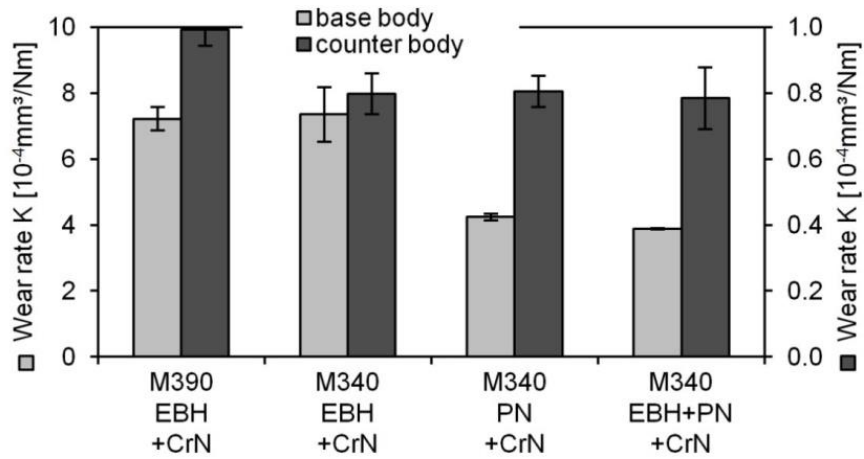


Fig. 10. Results of the wear tests (steel X54CrMoVN17-1).

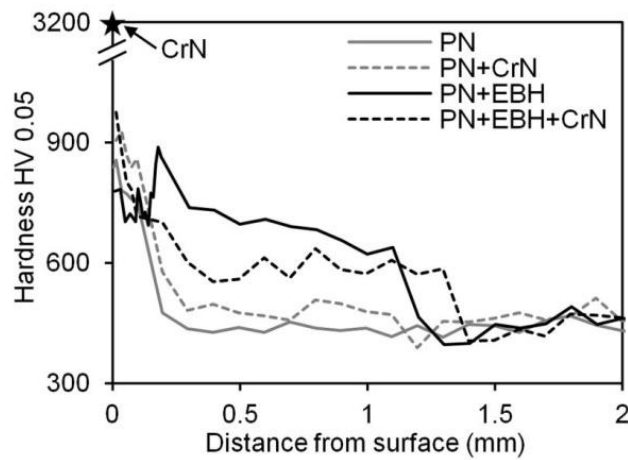


Fig.11. Hardness depth profiles of single, duplex and triplex treatments of steel 40CrMoV13-9.

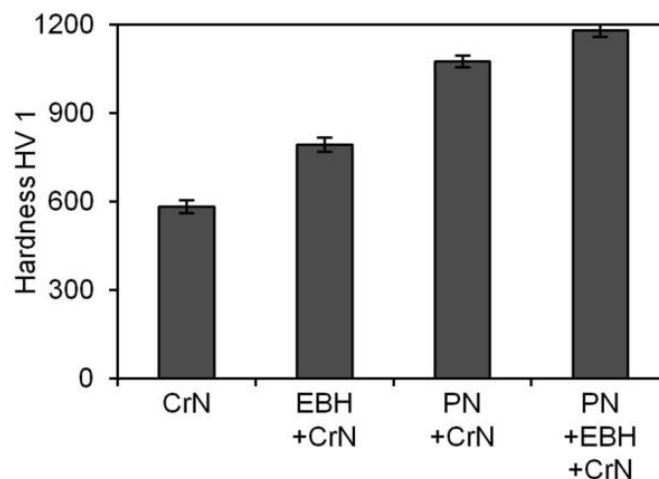


Fig. 12. Surface hardness after hard coating for duplex and triplex treatments (steel 40CrMoV13-9).

3.2.2 Plasma nitriding + EB hardening + PVD coating

Steel 40CrMoV13-9

In contrast to a triplex treatment with EBH as the first process step, hardness in the EBH layer and, therefore, the metallurgical compatibility of this triplex treatment, depends on the temperature time cycle of the hard coating deposition. Because the PVD process was carried out at temperatures between 350 and 410 °C, the hardness in the EBH layer decreased for steel 40CrMoV13-9 by ~100 HV 0.05. Compared to the duplex treatment PN+EBH (Fig. 11, black solid line), the hardness in the combined treated areas (PN+EBH) increases after PVD (Fig. 11, black broken line). It is assumed that, due to the temperature cycle during the deposition process, retained austenite may have been transformed into martensite.

Surface hardness measurements HV 1 were used to evaluate the base materials load support capacity (Fig. 12). It has to be noticed that surface hardness represents a mixed value based on the hardness of the matrix material and the hard coating. Highest hardness values are achieved for the triplex treatment PN+EBH+PVD.

4. Conclusions

Combining two or more surface technologies, e. g. EBH, nitriding and PVD hard coating, broadens the field of application of tools and components by new structure/property relations of the layer-matrix compounds. By thermal or thermochemical surface treatment, a hardened and strengthened layer is produced, resulting in a significant improvement of the base materials load support capacity for the hard coatings. Additionally, the property gradient is optimized.

Attention has to be paid to the metallurgical compatibility of the single processes. For electron beam hardening before nitriding and hard coating, respectively, tempering effects have to be taken into account if the layer deposition temperature and time exceed the tempering stability of the base material. For EBH after nitriding, the hardness in the areas treated with combined processes (the diffusion layer) is increased to a level that is higher than the level achieved by any of the single treatments, because of the formation of nitrogen-containing martensite.

The required load support capacity for the hard coating depends on the loading conditions and the base material. For the high

alloy steel X54CrMoVN17-1, a significant improvement in load support capacity is achieved even by a thin nitrided layer of 20-30 µm. Despite the lower nitriding depth in the case of PN+PVD, the improvement in load support capacity is higher than for a combined EBH+PVD process, because of the higher surface hardness. Therefore, the hardening depth is not an adequate criterion for load support capacity.

According to the wide range of different application characteristics, there are different requirements for the case structure, too. A triplex treatment offers the opportunity to generate a multiplicity of application-specific layers. First investigations of a triplex treatment combining nitriding, EB hardening and PVD hard coatings suggest a great potential for complex surface treatments, which are useful for highest-loaded components and, in particular, for injection-moulding tools and tools used for forming.

Acknowledgment

This research work was kindly supported by AiF Pro-Inno II (projects KF0600401TN7, KF0467202TN7 and KF0592801TN7).

The authors wish to thank their project partners pro-beam systems GmbH, Arburg GmbH & Co. KG and Sulzer Metaplas GmbH for their helpful support and successful cooperation.

References

- [1] R. Zenker: *Neue Hütte* 28(10) (1983) 379-385.
- [2] T. Bell: *Surf. Eng.* 6(1) (1990) 31-40.
- [3] A. Matthews, A. Leyland: *Surf. Coat. Technol.* 71 (1995) 88-92.
- [4] H.-J. Spies: *Stahl und Eisen*, 117(6) (1997) 45-52.
- [5] O. H. Kessler, F. T. Hoffmann, P. Mayr: *Surf. Coat. Technol.* 108-109 (1998) 211-216.
- [6] Kessler: *Surf. Coat. Technol.* 201 (2006), 4046-4051.
- [7] M. Sokovic, P. Panjan, R. Kirn: *J. Mater. Process. Technol.* 157-158 (2004), 613-616.
- [8] K. S. Klimek, A. Gebauer-Teichmann, P. Kaestner, K.-T. Rie: *Surf. Coat. Technol.* 201 (2007), 5628-5632.
- [9] R. Zenker, A. Buchwalder: *Elektronenstrahl-Randschichtbehandlung. Innovative Technologien für höchste industrielle Ansprüche, Monograph pro-beam AG & Co. KGaA*, 2010.
- [10] O. Keßler, F. Hoffmann, P. Mayr: *Surf. Coat. Technol.* 89 (1997) 299-304.
- [11] R. Zenker, M. Müller: *Heat Treatm. Met.* (4) (1988) 79-88.
- [12] R. Zenker, W. John, D. Rathjen, G. Fritsche, B. Kamfe: *Heat Treatm. Met.* (2) (1989) 43-51.
- [13] S. Schiller, S. Panzer: In: *ASM Handbook*, Vol. 4, 1991, pp. 297-311.
- [14] R. Zenker: *Adv. Eng. Mater.* 6(7) (2004) 581-588.
- [15] R. Zenker: *Int. Heat Treat. Surf. Eng.* 5(2) (2011) 50-56.
- [16] H.-J. Spies, R. Zenker, K. Bernhard, H. Buschbeck: In: *ECLAT96*, Stuttgart, Germany, Sept 16-18, 1996, pp. 319-328.
- [17] H.-J. Spies, R. Zenker, K. Bernhard, H. Buschbeck: *Härtereitechn. Mitt.* 53(4) (1998) 222-227.
- [18] H.-J. Spies, S. Friedrich, A. Buchwalder: *Mat.wiss. u. Werkstofftech.* 34 (2003) 128-135.
- [19] R. Zenker, G. Sacher, A. Buchwalder, J. Liebich, A. Reiter, R. Häbeler: *Surf. Coat. Technol.* 202 (2007) 804-808.
- [20] G. Sacher, R. Zenker, N. Frenkler, T. Kimme: *HTM J. Heat Treatm. Mat.* 64(1) (2009) 20-27.
- [21] G. Sacher, R. Zenker, H.-J. Spies: *Mater. Manuf. Process.* 24 (2009) 800-805.