Improvement of surface properties by combined processes - A comparison: CVD plus induction hardening against carburizing plus CVD

O. Kessler, F. Hoffmann, P. Mayr

The properties of tools and components can be improved by the combination of heat treatment and surface engineering processes due to the addition of the single process advantages and due to the utilization of process interactions. A well known and clear example is the combination CVD-coating with wear resistant films + quench hardening (through-hardening) of high alloyed tool steels for forming tools. The thin wear resistant coating (e.g. TiN) is supported by the high strength of the hardened substrate. This combination can be further improved by adding surface heat treatment processes like induction hardening or carburizing. Several low and high alloyed, structural and tool steels (AISI 4140, 52100, H13, A2, D2, etc.) have been treated by CVD TiN-coating + induction hardening respectively carburizing + CVD TiN-coating + gas quench-ing. Both combined processes resulted in surfaces with very promising properties. The results were compared concerning chemical compositions, micro-structures, hardness, residual stresses and scratch tests. Homogeneous, dense TiN-coatings with high hardness, high compressive residual stresses and good adhesion were supported by high strength substrate surfaces. An extra advantage of these combined processes compared to CVD + through-hardening were compressive residual stresses in the substrate surfaces.

Parole chiave: trattamenti termici, trattamenti superficiali, acciaio

INTRODUCTION

High resistance of metal tools or components against wear, fatigue and corrosion can be achieved by several different treatments, like thermal, mechanical, thermochemical and coating processes. But in an increasing number of cases, the resistance of metals against complex loads is no longer high enough. Combining successful single processes into one treatment can result in an even higher resistance of metals against complex loads, e.g. superimposed wear, fatigue and corrosion. Processes combined in this way, sometimes called duplex or hybrid processes, have a high potential for the future treatment of metals [1-3]. The improvement is due to the addition of the single process advantages and due to the utilization of process interactions.
A task in the future is the substantial choice of technical and economical promising combinations. As a first step, a classification for combined processes was set up [4-6]. The single processes were tabulated into main sections according to the German standard DIN 8580. Actually the system for combined processes contains the main sections coating and changing properties, but it can be extended straight forwardly. The principle of the classification for combined processes is a matrix out of the single processes A1-Ai and B1-Bi, A in lines, B in columns (Table 1). In the intersections of lines and columns of the matrix the different combined processes A+B can be found. Table 1 also contains some characteristic examples of combined processes. A well known and clear example is the combination CVD-coating with wear resistant films + quench hardening (through-hardening) of high alloyed tool steels for forming tools. The thin wear resis-tant coating (e.g. TiN) is supported by the high strength of the hardened substrate [7,8,25]. This combination can be further improved by adding surface heat treatment processes like induction hardening or carburizing (highlighted in Table 1).

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EXPERIMENTAL

Several low and high alloyed, structural and tool steels (AISI 4140, 52100, H13, A2, D2, etc.) have been treated by CVD TiN-coating + induction hardening respectively carburizing + CVD TiN-coating + gas quenching. The tool steels A2 (DIN X100CrMoV5-1) and H13 (DIN X40CrMoV5-1) are suitable for a comparison, because the combined processes CVD + induction hardening of A2 and carburizing + CVD + quench hardening of H13 lead to similar chemical compositions and microstructures in the substrate surface, underneath the coating. Additionally some results of the CVD-coated and induction hardened bearing steel 52100 (DIN 100Cr6) are presented.
CVD-coating + induction hardening of A2 was done in a two-step-process. Cylindrical samples Ø 27x23 mm with chamfers were used for this treatment. HT-CVD TiN-coating of A2 was done for 5 h at 950°C in a $TiCl_4/H_2/N_2$-atmosphere. Induction hardening was realized in a redeveloped equipment, which enables heating in protective atmosphere and quenching in oil or gas nozzle fields [17-19]. Stationary inductive heating of the sample shell was done with a HF-generator (10 kW, ca. 200 kHz) and a cylindrical coil for 15 s in a nitrogen atmosphere. Quenching was done in a gas nozzle field with 2000 l/min nitrogen flow. Carburizing +

			5 coating, B↓				
			5.1 from gaseous state			5.2 from liquid state	5.3 from ionized state
			CVD	PACVD	PVD	thermal spraying	electro(less) plating
6 changing properties, A →	6.1 rearranging particles	quench hardening	CVD + through-hardening, CVD + induction hardening				
		mechanical strengthening				thermal spraying + mechanical strength. [9]	
		surface remelting				thermal spraying + surface remelt. [10]	
	6.3 introducing particles	carburizing	carburizing + CVD				
		nitriding		nitriding + PACVD [11]	nitriding + PVD [12,13]		electroplating + nitrid. [14]
		ion implantation			PVD + ion implantation [15]		
		surface alloying			surface alloying + PVD [16]		

Table 1: System of combined processes with examples (no. according German standard DIN 8580)

Table 1: Sistema di processi abbinati con esempi (no. secondo la norma tedesca DIN 8580)

CVD-coating + gas quenching of H13 was also done in a two-step-process, because carburizing + CVD-coating could be realized as one step in the CVD-reactor [20-22]. Disc samples Ø 40x5 mm were used for this treatment. Low pressure carburizing and HT-CVD TiN-coating of H13 were done continuously in the CVD-reactor for 40 min at 1000°C in a methane/nitrogen-atmosphere respectively for 2 h at 1000°C in a $TiCl_4/H_2/N_2$-atmosphere. Quench hardening was done in a vacuum chamber at 980°C, 20 min and gas quenching with 6 bar nitrogen.

The treated surfaces and substrates were investigated regarding their chemical composition by GDOS (glow discharge optical emission spectroscopy) / OES (optical emission spectroscopy), microstructure by light microscopy, substrate hardness by the Vickers method HV1, coating hardness HV_f by evaluating the compound hardness [23], and residual stresses by XRD (X-ray diffraction, Cr-K_α, {311} respectively {220} TiN, {211} α-Fe). The behavior in scratch tests, especially adhesive coating failure, was examined by evaluating the scratch tangential force. Both treatments were compared regarding their microstructures and properties to show similarities as well as differences and to demonstrate the advantages and potentials of these combined processes.

RESULTS AND DISCUSSION

The chemical composition depth profiles of A2/CVD TiN/induction hardened and H13/carburized/ CVD TiN/quench hardened are shown in Figs. 1 and 2. Both samples exhibit an almost stoichiometric TiN-coating along most of the coating thickness of ca. 8 µm respectively ca. 4 µm. The A2/CVD TiN/induction hardened is characterized by a high oxygen amount near the surface, due to inductive heating in an oxygen containing atmosphere [19], and a monotonic increase of the carbon content to its bulk value. The H13/carburized/CVD TiN/quench hardened is characterized by a carbon maximum in the coating near the interface, due to carbon diffusion from the substrate [21], and by the introduced carbon gradient in the substrate surface. The maximum carbon content in the substrate surface is ca. 0.8 wt. %, the carburized depth ca. 2 mm.

The microstructures of surface and core of A2/CVD TiN/induction hardened and H13/carburized/ CVD TiN/quench hardened are shown in Figs. 3 and 4. The coating of A2/CVD TiN/induction hardened appears ca. 7 µm thick, homogeneous and dense (bright) with a thin (ca. 1 µm) dark layer on top. This top layer at least partially consists of TiO_2 rutil, due to inductive heating in an oxygen containing atmosphere [19]. The substrate A2 underneath the coating consists of martensite, retained austenite and carbides. A slight increase in inductive heating time (austenitizing temperature) may result in a more homogeneous substrate surface microstructure. The core A2 is not affected by inductive heating and keeps the microstructure after slow cooling from CVD-temperature, which exhibited a mixture of martensite,

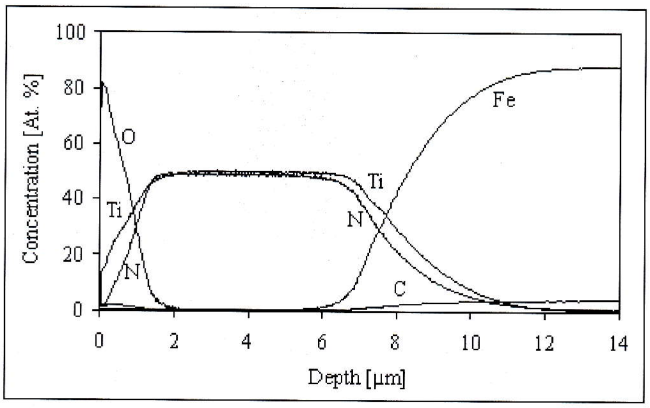

Fig. 1: Chemical composition depth profile of A2/CVD TiN/induction hardened, by GDOS

Fig. 1: Curva di penetrazione della composizione chimica di acciaio A2/rivestito CVD TiN/temprato a induzione, mediante GDOS

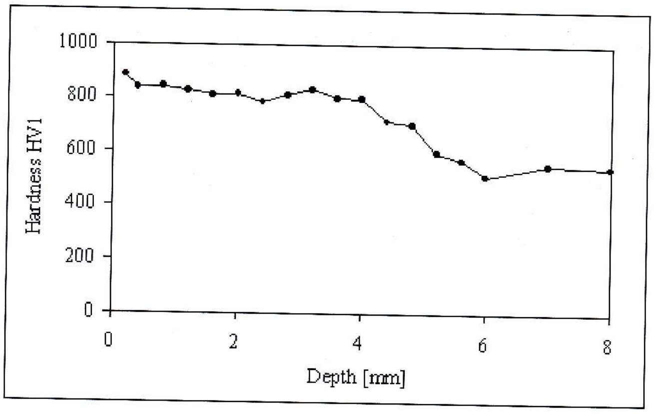

Fig. 5: Substrate hardness depth profile of A2/CVD TiN/induction hardened

Fig. 5: Curva di penetrazione per la durezza del substrato di acciaio A2/CVD TiN/ temprato superficialmente a induzione

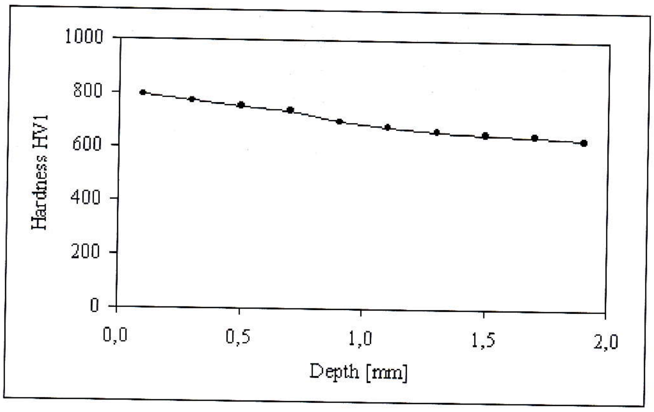

Fig. 6: Substrate hardness depth profile of H13/carburized/CVD TiN/quench hardened

Fig. 6: Curva di penetrazione per la durezza del substrato di acciaio H13/cementato/CVD TiN/ temprato a induzione

hardness of ca. 820 HV1 and a depth of ca. 4 mm. Then the substrate hardness decreases to ca. 530 HV1 at the not inductively heated core. The depth of the induction hardened layer can strongly be influenced by the inductive heating power, frequency and time [17,18]. The carburized and quench hardened substrate surface H13 also exhibits a high hardness of ca. 800 HV1 and a depth of ca. 2 mm. Then the substrate hardness decreases to ca. 640 HV1 at the not carburized core. The depth of the carburized layer can strongly be influenced by the carburizing atmosphere, temperature and time as well as the CVD-duration [22]. In both cases the lower core hardness underneath a strengthened surface may be an advantage for tools or components regarding ductility. The coating hardness of CVD TiN-coatings is only slightly decreased after induction hardening in air/nitrogen-atmospheres. The coating hardness e.g. of steel 52100/CVD TiN amounted ca. 2900 HV_f, after induction hardening ca. 2700 HV_f – similar results were found for other substrates [17,18]. The coating hardness of CVD TiN-coatings is almost unchanged after through-hardening in vacuum, which was proven for steel substrates like 4140, 52100 and D2 [7]. This should also be true for carburized + CVD-coated + quench hardened steels.

An important advantage of the combined processes CVD + induction hardening and carburizing + CVD + quench hardening is the residual stress state compared to the common process CVD + through-hardening. Fig. 7 and Table 2 show the coating and substrate surface residual stresses of 52100/CVD TiN/induction hardened and H13/carburized/CVD TiN/quench hardened. In both cases the CVD TiN-coatings are under high compressive residual stresses, due to the volume change differences between coating and substrate caused by thermal contraction and phase transformations during cooling from CVD-temperature [8]. Those high compressive residual stresses in the coatings can strongly be influenced by post quench hardening, but usually they remain in the high compressive range (Fig. 7 and Table

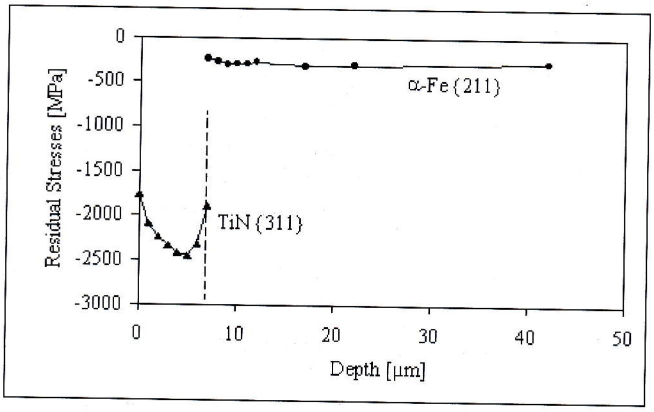

Fig. 7: Residual stress depth profile of 52100/CVD TiN/induction hardened, axial stress in cylinders

Fig. 7: Curva di penetrazione per la tensione residua in acciaio 52100/CVD TiN/temprato superficialmente a induzione, tensione assiale in cilindri

2). In the substrate surface the common process CVD + through-hardening usually produces tensile residual stresses (Table 2 and [8]). Whereas the combined processes CVD + induction hardening and carburizing + CVD + quench hardening produce compressive residual stresses in the substrate surface (Fig. 7 and Table 2), because the hardened substrate layers of high carbon martensite exhibit larger specific volumes than the unaffected substrate cores.

Compound coating/substrate-properties were evaluated by scratch tests. The critical loads for adhesive failure of A2/CVD TiN/induction hardened and H13/carburized/CVD TiN/quench hardened are given in Table 3. The as-deposited state of A2/CVD TiN respectively the as-quench hardened state of H13/CVD TiN already show relative high critical loads for adhesive failure. But post-induction hardening respectively pre-carburizing lead to a further improvement of

substrate	coating/heat treatment	residual stresses in the coating surface, TiN {220}, [MPa]	residual stresses in the substrate surface, α-Fe {211}, [MPa]
H13	CVD TiN/quench hardened	-2020 ± 130	0
H13	carburized/CVD TiN/ quench hardened	-1900 ± 130	-380
DIN 17CrNiMo6	CVD TiN/quench hardened	-1480 ± 160	110 ± 30
DIN 17CrNiMo6	carburized/CVD TiN/ quench hardened	-3590 ± 110	-320 ± 90

Table 2: Residual stresses in coating and substrate of H13/carburized/CVD TiN/quench hardened

Table 2: Tensioni residue in rivestimento e substrato acciaio in H13/cementato/CVD TiN/temprato

Table 3: Critical loads for adhesive failure in scratch tests of A2/CVD TiN/induction hardened and H13/carburized/CVD TiN/quench hardened

Table 3: Carichi critici di adesione in prove di penetrazione per abrasione di acciaio A2/CVD TiN/temprato a induzione e H13/cementato/rivestito CVD TiN/temprato

substrate	coating/heat treatment	critical loads for adhesive failure in scratch tests [N]
A2	CVD TiN	73 ± 7
A2	CVD TiN/induction hardened	> 100
H13	CVD TiN/quench hardened	89 ± 5
H13	carburized/CVD TiN/ quench hardened	> 100

	A2/CVD TiN/induction hardened	H13/carburized/CVD TiN/quench hardened	common tool steel/CVD TiN/through-hardened
coating			
• chemical composition	thin oxidized layer at the surface	increased carbon content near the interface	almost constant
• microstructure	homogeneous, dense	homogeneous, dense	homogeneous, dense
• hardness	slight decrease	almost unchanged	almost unchanged
• residual stresses	slight decrease of compressive stresses	depends on substrate and dimensions	decrease of compressive stresses
substrate surface			
• chemical composition	almost constant	increased carbon content in the substrate surface	almost constant
• microstructure	martensite, retained austenite, carbides	martensite, retained austenite, carbides	martensite, retained austenite, carbides
• hardness	increase in the substrate surface	extra increase in the substrate surface	increase
• residual stresses	compressive	compressive	tensile
substrate core			
• microstructure	martensite, bainite, pearlite	lower carbon martensite	martensite, retained austenite, carbides
• hardness	lower than substrate surface, ductility	lower than substrate surface, ductility	almost equal to substrate surface
compound			
• behavior in scratch tests	improved	improved	improved
• dimensional changes	low, due to small heated surface volume	must be controlled	must be controlled

Table 4: Comparison of chemical compositions, microstructures and properties of A2/CVD TiN/ induction hardened and H13/carburized/CVD TiN/quench hardened

Table 4: confrondo di composizioni chimiche, microstrutture e proprietà di acciaio A2/CVD TiN/ temprato a induzione e H13/cementato/rivestitoCVD TiN/termprato

the behavior of CVD TiN-coated steels in scratch tests, which exceeded the test capabilities of 100 N. Reason is the increased load support of the surface strengthened substrates for the thin hard coatings.

A crucial point of every coated and heat treated tool or component is distortion, because the low coating thickness complicates machining to restore the desired dimensions. For the common process CVD + through-hardening of tool steels a lot of experiences with distortion exist, which allow to control dimensional changes of tools [25]. The same way should be possible for the com-bined process carburizing + CVD + quench hardening. Especially the combined process CVD + induction hardening offers the possibility to reduce distortion, due to the small heated surface vol-ume, which was proven at steel cylinders 4140/CVD TiN [17-19].

Summarizing the discussed chemical compositions, microstructures and properties of A2/CVD TiN/induction hardened and H13/carburized/CVD TiN/quench hardened as well as common tool steel/CVD TiN/through-hardened are compared in Table 4.

The characteristic features of A2/CVD TiN/induction hardened are the thin oxidized layer at the surface, the softer substrate core consisting of martensite + bainite + pearlite, the compressive residual stresses in the substrate surface and the low distortion due to the small heated surface volume. The characteristic features of H13/carburized/CVD TiN/quench hardened are the increased carbon content in the coating near the interface, the softer substrate consisting of lower carbon martensite, and the compressive residual stresses in the substrate surface.

Compared to common tool steel/CVD TiN/through-hardened, three main advantages of the combined processes CVD + induction hardening and carburizing + CVD + quench hardening were found (highlighted in Table 4):

- the possibility, to introduce microstructure and property gradients into the substrate,
- compressive residual stresses in the substrate surface,
- the possibility to reduce distortion (induction hardening).

Besides these technical advantages the combined processes CVD + induction hardening and carburizing + CVD + quench hardening posses further economical advantages:

- Induction hardening of CVD-coated steels can be done very quickly in some seconds or some ten seconds compared to through-hardening in a vacuum chamber, which lasts several ten min-utes to hours and it can be done locally in highly loaded areas of tools or components.

- Carburizing + CVD-coating can be done continuously in the CVD-reactor and needs only little extra time, because in a common boost/diffusion carburizing process the necessary diffusion time can be used for CVD-coating.

These advantages demonstrate the high potential of the combined processes CVD + induction hardening and carburizing + CVD + quench hardening for components or tools under complex superimposed loads, e.g. high wear plus high mechanical loads.

CONCLUSION

The properties of tools and components can be improved by the combination of heat treatment and surface engi-neering processes due to the addition of the single process advantages and due to the utilization of process interactions. A well known and clear example is the combination CVD-coating with wear resistant films + quench hardening (through-hardening) of high alloyed tool steels for forming tools. The thin wear resis-tant coating (e.g. TiN) is supported by the high strength of the hardened substrate. This combination can be further improved by adding surface heat treatment processes like induction hardening or carburizing. Several low and high alloyed, structural and tool steels (AISI 4140, 52100, H13, A2, D2, etc.) have been treated by CVD TiN-coating + induction hardening respectively carburizing + CVD TiN-coating + gas quenching. CVD-coating + induction hardening was done in a two-step-process; induction hardening was realized in a redeveloped equipment, which enables heating in protective atmosphere and quenching in oil or gas nozzle fields. Carburizing + CVD-coating + gas quenching was also done in a two-step-process, because carburizing + CVD-coating could be realized as one step in the CVD-reactor.

Both combined processes resulted in surfaces with very promising properties. The results were compared concerning chemical compositions, micro-structures, hardness, re-sidual stresses and scratch tests. Homogeneous, dense TiN-coatings with high hardness, high compressive residual stresses and good adhesion were supported by high strength substrate surfaces. An extra advantage of these combined processes compared to CVD + through-hardening were compressive residual stresses in the substrate surfaces. A crucial point of every coated and heat treated component is distortion. Especially CVD + induction hardening offers the possibility to reduce distortion, due to the small heated surface volume.

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ABSTRACT

MIGLIORAMENTO DELLE PROPRIETÀ SUPERFICIALI MEDIANTE PROCESSI COMBINATI. UN CONFRONTO FRA CVD + TEMPRA A INDUZIONE E CEMENTAZIONE + CVD

Le proprietà di utensili e componenti possono essere migliorate mediante la combinazione di processi superficiali con trattamenti termici sia per la somma dei vantaggi dei singoli trattamenti che per la loro interazione. Un esempio ben noto e chiaro è l'uso in abbinamento di riverstimenti CVD con strati resistenti all' usura più tempra a cuore (through-hardening) negli acciai altolegati per utensili da formatura. Il rivestimento sottile resistente all'usura (per esempio TiN) è supportato dall'alta resistenza del substrato indurito. Questo abbinamento può essere ulteriormente migliorato mediante l'aggiunta di processi superficiali di trattamento termico come la tempra a induzione o la cementazione.
Diversi acciai strutturali e da utensili (AISI 4140, 52100, H13, A2, D2, ecc.) sono stati trattati tramite rivestimento CVD TiN + tempra a induzione e in alternativa cementazione + rivestimento CVD TiN + tempra a gas. Il rivestimento CVD + la tempra a induzione sono stati effettuati mediante un processo a due stadi; la tempra a induzione è stata realizzata con un' apparecchiatura che permette il riscaldamento in atmosfera protetta e tempra a gas o olio. Anche cementazione + rivestimento e CVD + tempra a gas sono stati eseguiti mediante un processo a due stadi. Entrambe le combinazioni di processi hanno dato luogo a superfici con proprietà molto promettenti. I risultati sono stati confrontati in termini di composizione chimica, microstruttura, durezza, tensioni residue e prove di scalfittura.
Si sono ottenuti rivestimenti TiN omogenei, densi, con elevata durezza, elevate tensioni residue di compressione e buona adesione, abbinati ad un substrato ad alta resistenza. Un ulteriore vantaggio di questi processi combinati rispetto a CVD + tempra a cuore consiste nell'ottenimento delle tensioni residue di compressione anche al di sotto del rivestimento. Un punto cruciale di qualsiasi componente rivestito e trattato termicamente è la distorsione. In particolare il trattamento CVD + tempra superficiale a induzione risulta offrire la possibilità di ridurre la distorsione, grazie al limitato volume superficiale riscaldato.

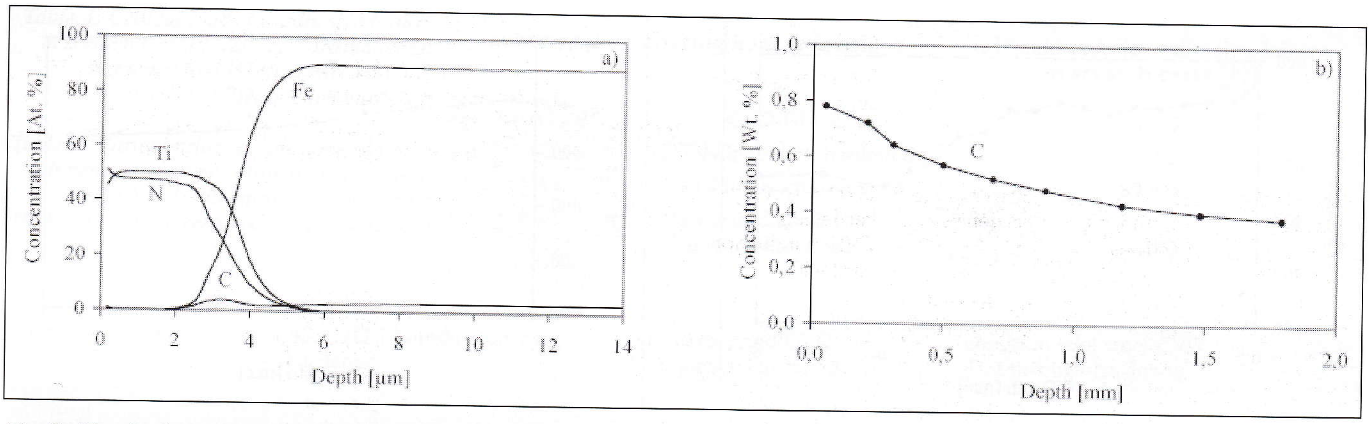


Fig. 2: Chemical composition depth profile of H13/carburized/CVD TiN/quench hardened, a) coating and interface by GDOS, b) substrate surface by OES

Fig. 2: Curva di penetrazione per la composizione chimica di acciaio H13/cementazione/CVD TiN/temprato a cuore, a) rivestimento e interfaccia mediante GDOS, b) superficie del substrato mediante OES

Fig. 3: Microstructure of A2/CVD TiN/induction hardened, a) coating and interface, b) substrate core, ←→ 10 µm.

Fig. 3: Microsctruttura di acciaio A2/CVD TiN/ temprato a induzione, a) rivestimento e interfaccia, b) cuore del substrato, ←→ 10 µm.

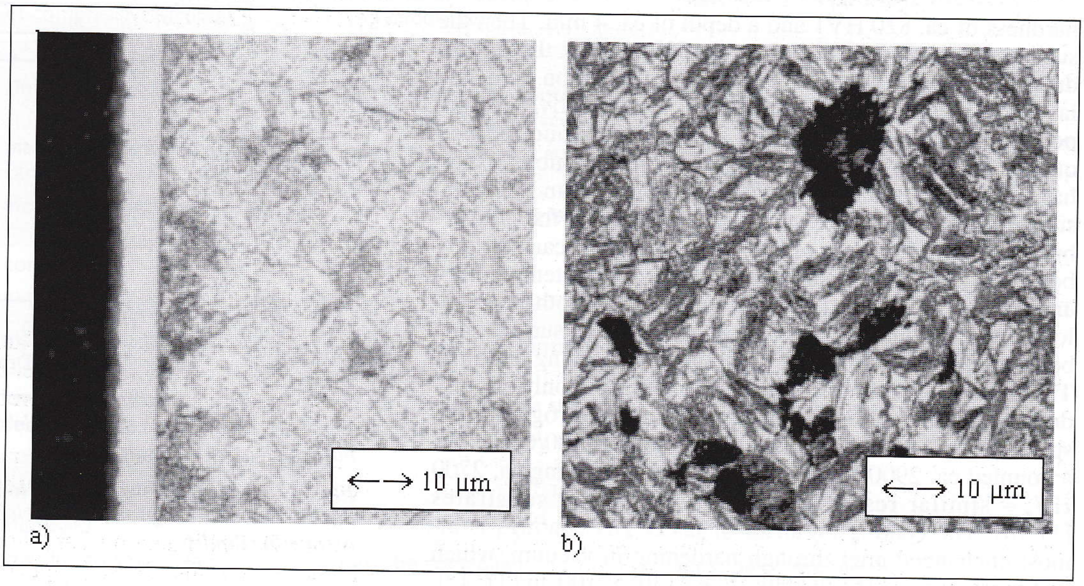
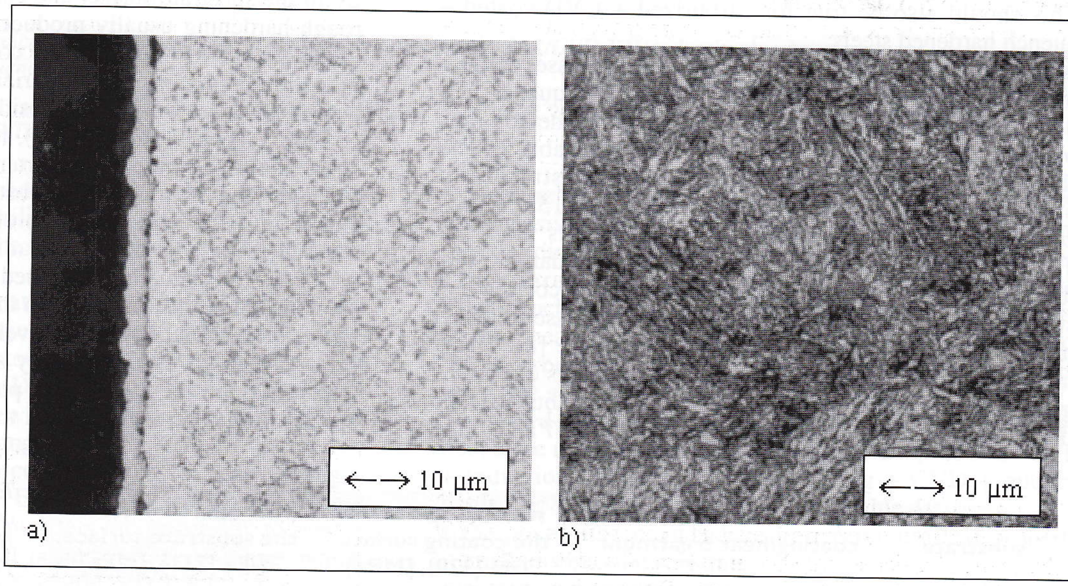


Fig. 4: Microstructure of H13/carburized/CVD TiN/quench hardened, a) coating and interface, b) substrate core, ←→ 10 µm.

Fig. 4: Microsctruttura di acciaio H13/cementato/CVD TiN/ temprato, a) rivestimento e interfaccia, b) cuore del substrato, ←→ 10 µm.



bainite and pearlite. The coating of H13/carburized/CVD TiN/quench hardened appears ca. 4 µm thick, homogeneous and dense. In the interface some small defects occur, which may be caused by interdiffusion between substrate and coating during deposition [24]. The carburized substrate H13 underneath the coating also consists of martensite, retained austenite and carbides. The core H13 shows the usual microstructure after quench hardening, which mainly is martensitic.

The substrate hardness depth profiles of A2/CVD TiN/induction hardened and H13/carburized/ CVD TiN/quench hardened are shown in Figs. 5 and 6. They confirm the described carbon depth profiles and substrate microstructures. The induction hardened substrate surface A2 exhibits a high