Nitriding problems on rolled rings made of 42CrMo4

S. Barella, M. Boniardi, S. Cincera, M. Bellogini

Even though the existing nitriding processes allow precise parameters control, some components could not achieve sufficient hardening characteristics. The aim of this work is the determination of the main cause of the incorrect nitrided case formation on some large gears made of UNI EN 42CrMo4 steel, realized from rolled rings. Metallographic analyses were conducted on nitrided specimens made of different heats. Moreover different oil-quenching and tempering were performed on partial finished components (supplied in normalized conditions). Several maintenance time and temperature conditions were tested to understand the effects of grain-size and carbides precipitation on the nitrogen diffusion. Both segregation bands and excessive carbide precipitations near the surface lead to low surface hardness and insufficient effective case depth.

KEY WORDS:
Nitriding, band structure, gear, effective case depth

INTRODUCTION

Nitriding is used on mechanical components to improve the wear resistance and the material fatigue strength. This process has the attraction that it results in a component having different properties in its core and case. Nitrogen, which has partial solubility in iron, is introduced through the surface of steel by reaction with an ammonia rich gas. As the nitrogen diffuses into the surface the solubility limit is soon reached and, in presence of alloying elements, hard and finely dispersed compounds are formed. These compounds (nitrides) give the adequate hardness to the surface [1].

Nitriding is very effective only when applied to a limited range of alloy steel containing a selection of elements that form stable nitrides, e.g. aluminum, chromium, molybdenum, vanadium and tungsten. The carbon content varies from 0.1 to 0.5% in relation to the required core strength.

The nitriding results are more favourable in those steels that contain one or more of the major nitride-forming alloying elements (i.e. Aluminum). Chromium-containing steels can approximate the aluminum alloyed steel behaviour if their chromium content is high enough [2].

For this reason UNI EN 42CrMo4 steel is often used for mechanical components that require case-hardening, in particular for all mechanical applications that require high wear and fatigue contact resistances. These applications also rely on standard surface hardness of about 600 HV and effective case depth greater than 200 μm.

Nitriding is a thermo-chemical treatment. Some heat is needed to enhance the diffusion of hardening species through the surface and subsurface regions of a part. The depth of diffusion exhibits a time-temperature dependence such that:

$$\text{Case depth} \propto K\cdot\sqrt{t} \qquad (1)$$

where the diffusivity constant, K, depends on temperature, chemical composition of the steel, and concentration gradient of a given hardening species. In terms of temperature, the diffusivity constant increases exponentially as a function of absolute temperature. Concentration gradients depend on the surface kinetics and reactions of a particular process [2]. Also grain size [3] and carbide distribution influence case depth as suggested by many authors [1][4][5][6].

Case depth is one of the most important parameters related to the component application. For example the choice of the right case depth is important in the design of gears, as they must guarantee high wear resistance toμgether with fatigue contact resistance (pitting).

In some cases it is not possible to reach the desired superficial properties due to particular steel microstructures. The first problem could be a small grain size, as small grains slow the diffusion of interstitial elements. In this case a close net of grain boundaries hinders the atomic nitrogen movement. Moreover, it has been found in a majority of continuous casting steel products that rolling bands are present. If the ingot structure is not homogenized during annealing and hot working, a banded microstructure with highly anisotropic mechanical properties may persist. It is well known that diffusion preferentially occurs in homogeneous regions, while microstructural inhomogeneities hinder the interstitial elements diffusion. [7]. Moreover interstitial elements diffusion varies in relation to the orientation of these inhomogeneities. The diffusivity coefficient is greater along the rolling direction then across the transverse one and the thickness [8] [9]. The rolling bands effect may be eliminated only with a significant annealing heat treatment, able to homogenise the component microstructure.

The aim of this work is to identify the mechanisms reducing the nitriding effective case depth on plastic deformed mechanical components. The study is divided into two parts: in the first one nitrided crown gears were analyzed; in the second part different selected heat treatments were carried out to better understand the influence of grain size on the efficiency of the nitriding process.

EXPERIMENTAL PROCEDURE

a) Part 1

The analysed crown gears are made of UNI EN 42CrMo4 steel, the pitch diameter is 480mm, the modulus is m=4. They were ob-

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Crown	C	Mn	Si	P	S	Cr	Ni	Cu	Mo	Al
1	0,45	0,85	0,25	0,006	0,026	1,24	0,12	0,18	0,19	0,022
2	0,44	0,75	0,22	0,005	0,028	1,18	0,11	0,18	0,19	0,022
3	0,44	0,77	0,25	0,015	0,028	1,15	0,11	0,06	0,21	0,019

TAB. 1 ***Chemical composition (% wt.) of analyzed samples.***

Composizione chimica (% peso) dei campioni analizzati.

tained by hot plastic deformation (rolling ring) of a 500mm diameter round billet. The billet origin is a continuous casting process. The employed rolling temperatures range from 1200 °C to 900 °C.

After finishing operations, crown gears were quenched (austenitizing at 900°C for 3 hours and oil cooling), tempered (640°C) and nitrided for 42 hours with a gas process (p_N = 6 for 6 hours at 525°C and p_N = 2 for 36 hours at 540°C).

Crown gears chemical compositions are reported in Table 1.

Metallographic samples were taken from every crown gear.The tested specimens were prepared by mechanical polishing and chemical metallographic etching (Picral, 10 s). Microstructural analyses were conducted by optical and electronic microscopy. Moreover, on these samples several Vickers microhardness tests (300g) were carried out to evaluate the microhardness profile and thus the effective case depth.

b) Part 2

Several heat treatments (quenching) were carried out on normalized semi-finished components.

The banded structure is aligned to the rolling direction and for this reason the samples were obtained in order to perform the nitriding normal to this stucture (the worst direction in relation to nitrogen diffusion). Heat treatments were conducted with three different austenitizing temperatures (950°C, 1000°C e 1050°C) and, for every temperature, with two holding time: one and three hours.

Three samples were obtained for every parameters couple. One of these samples was normalized (cooling in air) in order to determine the austenitic average grain size (in accordance with ASTM E112). The other two samples were quenched in oil and tempered. One was tempered at 640°C and the second one at 590°C. Table 2 illustrates the test plan.

Quenched and tempered samples were subsequently nitrided with a double stage treatment (p_N = 6 for 6 hours and p_N = 2 for 30 hours).

All samples were analyzed by optical microscopy. The normalized ones were also analyzed by SEM. Microhardness profiles were realized on nitrided samples.

RISULTS AND DISCUSSION

a) Part 1

Microhardness profiles are performed on samples taken from crown gears (figure 1). Effective case depth is always lower than the required one, despite the fact that chemical composition and nitriding process parameters are correct.

Figure 1 shows the three different profiles: Sample 1 has the best characteristics although they don't satisfy the requests.

Metallographic analyses were conducted on the sample in order to understand this behavior. Figure 2 shows the crown gears microstructure. At lower magnification (Figure 2 on the left) "band" structure is evident. Bands are the result of a micro-segregation process, which causes a local different chemical composition. Bands origin is the continuous casting process and they became more marked after the plastic deformation process. In spite of the quenching carried out on the component, the band structure remains. This structure is detectable in all samples but in sample 2 and 3 bands appear more prominent.

Figure 2 also shows the particular bands distribution; their orientation is normal to the nitrogen diffusion direction during the nitriding process. At higher magnification (figure 2 on the right) a fine precipitate dispersion is detectable, mainly at grain boundaries.

A SEM-EDS analysis was carried out in order to evaluate the precipitates type. The analyses were conducted in two zones: near the white layer and at the crown gears core. All samples present a close net of nitrides near the white layer. This net is more evident in Sample 3. The net origin is probably related to the rolling bands presence and their orientation in the metallic matrix. These micro-segregations behave as a diffusion barrier and thus they slow down the nitrogen diffusion. Nitrogen tends to saturate the zones near the segregation bands interface and therefore a nitrides rich region develops. These regions block nitrogen diffusion into the metallic matrix and for this reason only a thin case depth is formed. Some authors [1, 7] suggest that the presence of inhomogeneities could delay nitrogen diffusion.

Moreovr SEM analysis was conducted distal to the surface. Fi-

Austenitizing condition	Normalized Sample	Tempering temperature [°C]	
		640	590
950°C 1h	#AN	#A1	#A2
950°C 3h	#BN	#B1	#B2
1000°C 1h	#CN	#C1	#C2
1000°C 3h	#DN	#D1	#D2
1050°C 1h	#EN	#E1	#E2
1050°C 3h	#FN	#F1	#F2

TAB. 2 ***Heat treatment test plan.***

Piano di prova dei trattamenti termici.

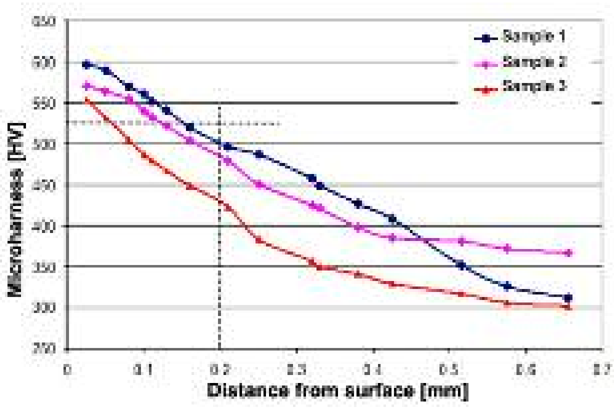

FIG. 1 ***Microhardness profiles of crown gears. Dashed lines represent the specific request on the effective case depth (sample 1, 2, 3 refer to crown 1, 2 and 3).***

Profili di microdurezza delle corone dentate. Le linee tratteggiate rappresentano la specifica richiesta sullo spessore efficace.

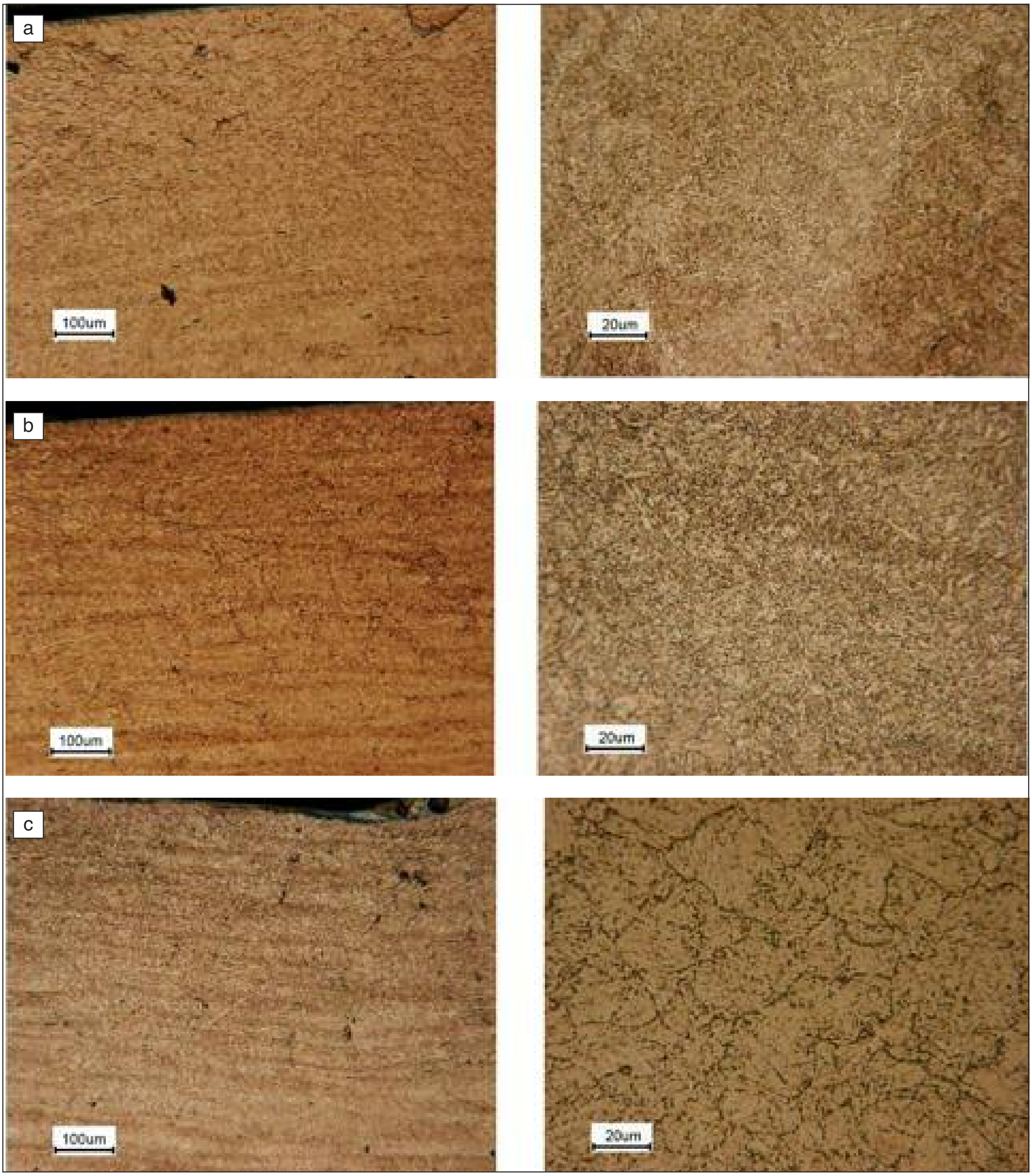

FIG. 2 ***Metallographic analysis of Samples 1, 2 and 3.*** *Aspetto metallografico dei campioni relativi alle corone 1, 2 e 3.*

gure 4 shows a fine dispersion of carbides, about 1µm wide; the EDS analysis shows that they are chromium carbides.

Microstructural inhomogeneities (segregation and chromium carbides) should be assumed as the causes of the thin case depth. Oriented bands hinder adequate nitrogen diffusion into the metallic matrix. Nitrogen accumulates in the superficial region and it does not diffuse beyond 200 µm. Moreover, chromium carbide precipitation limits the hardness increase. Chromium, together with other alloying elements, must be available for nitrides formation. If chromium is already bound to form carbides, nitrogen compounds, which give hardness to the surface, could not form [4] [9].

Finally, the microstructure shows a very fine grain size. This should be a further problem in relation to nitrogen diffusion [10].

b) Part 2

Several heat treatments were carried out on several semi-finished products (ring). These heat treatments were conducted in order to evaluate the influence of different time-temperature parameters on ring microstructure and thus on the nitrogen diffusivity into the metallic matrix.

First, quenching parameters (austenitizing temperature and

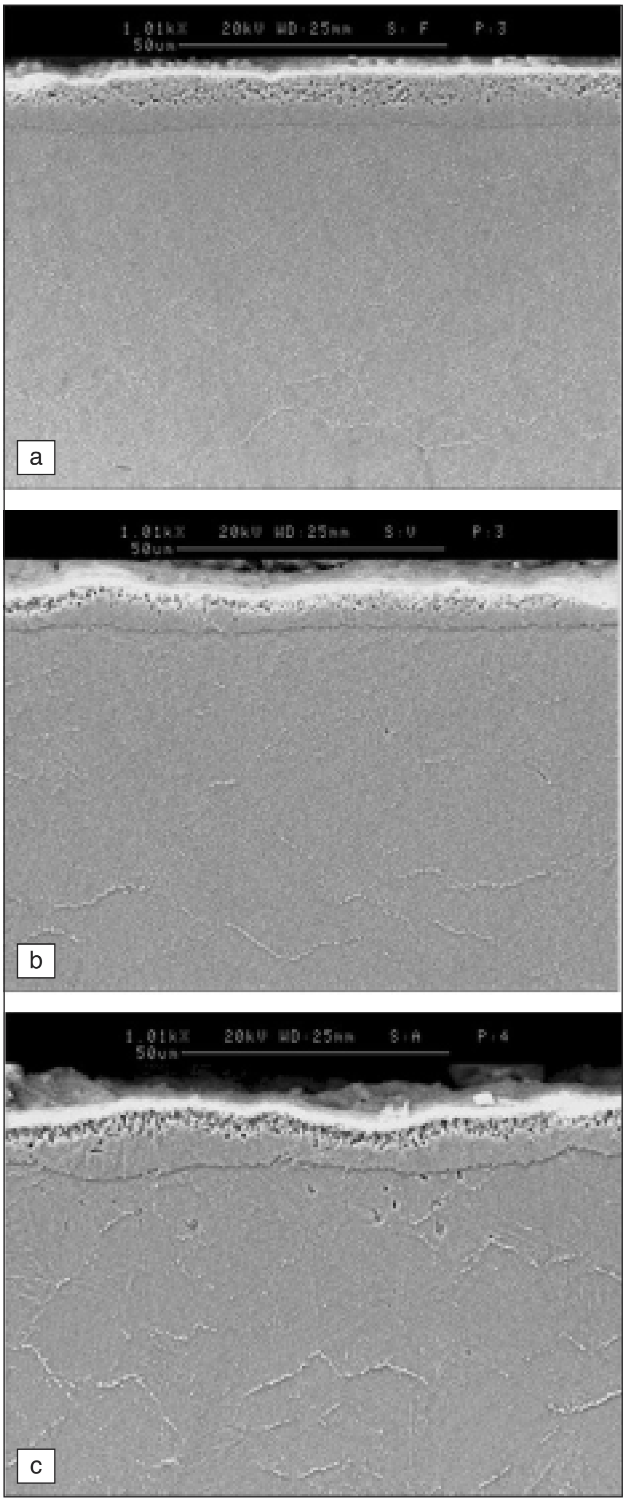

FIG. 3 ***SEM analyses of sample 1 (a), 2 (b) e 3 (c) nitrided layer.***

Analisi SEM dello stato niturato dei campioni 1 (a), 2 (b) e 3 (c).

maintenance time) were varied to analyze their effect on the grain size and on the microsegregation reduction.

The grain size was measured on normalized samples. An increase in austenitizing temperature or time maintenance causes grain size growth (Figure 5). Despite different time-temperature condition the band structure remains unchanged in all the samples.

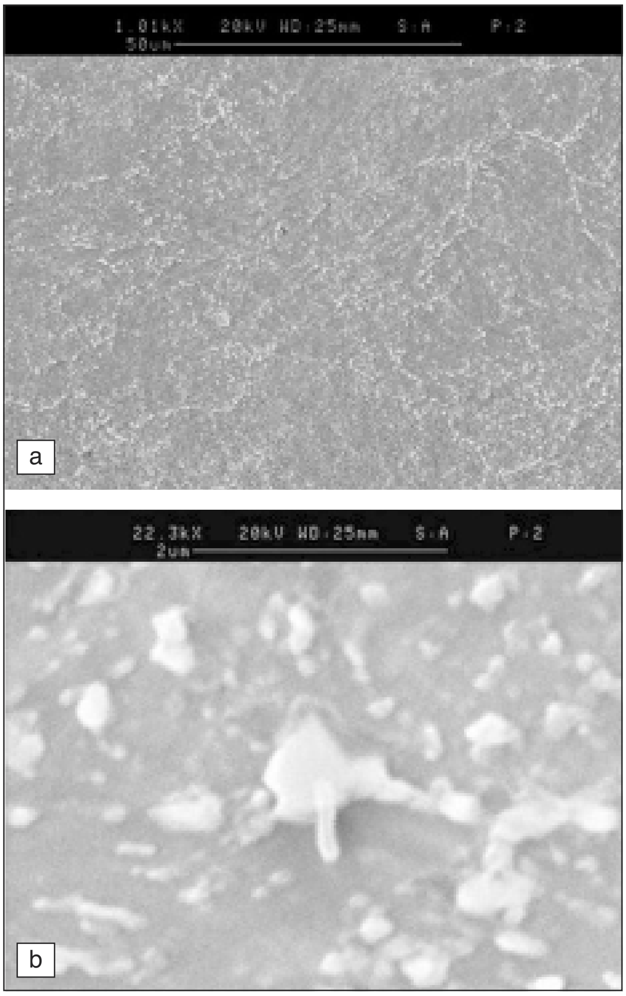

FIG. 4 ***Sample 3 microstructure: core (a) and magnification of precipitate present into metallic matrix (b).***

Microstruttura del campione 3 a cuore (a) e ingrandimento dei precipitati presenti nella matrice metallica (b).

The quenched and tempered samples were subsequently nitrided. Although microhardness profile were obtained for all nitrided samples, a selection of more meaningful values are reported in Figure 7 and 8 (samples #B, #D and #F) . Microhardness profiles on sample maintained for 1 hour are quite similar. Samples tempered at 640°C present a case depth not thick enough regardless of austenitizing temperature (Figure 7). Surface hardness is adequate, in relation to industrial specifications, only if the austenitizing temperature is 1050°C.

Figure 8 shows the microhardness profiles of samples tempered at 590°C. These samples reach the required effective case depth and the microhardness profiles are similar for all austenitizing temperatures.

By considering the tempering temperature and its influence on the chromium carbide formation this behavior could be explained. Tempering performed at 640°C for 3 hours, promotes chromium carbides precipitation. In this case chromium nitrides couldn't form during nitriding and therefore the resulting case depth is not satisfactory for mechanical purposes. On the other hand, tempering performed at 590°C inhibits chromium compounds precipitation and, for this reason, chromium remains available for nitrides formation. In addition, microhardness profiles are similar for all austenitizing temperatures (as shown in

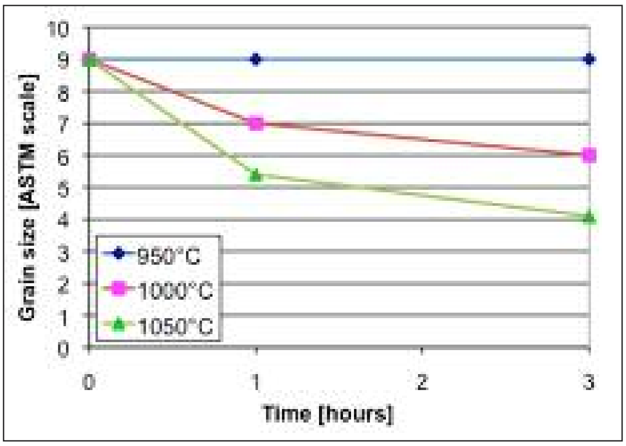

FIG. 5 ***Grain size in relation to maintenance time at austenitizing temperature.***

Dimensione del grano in funzione del tempo e della temperatura di mantenimento.

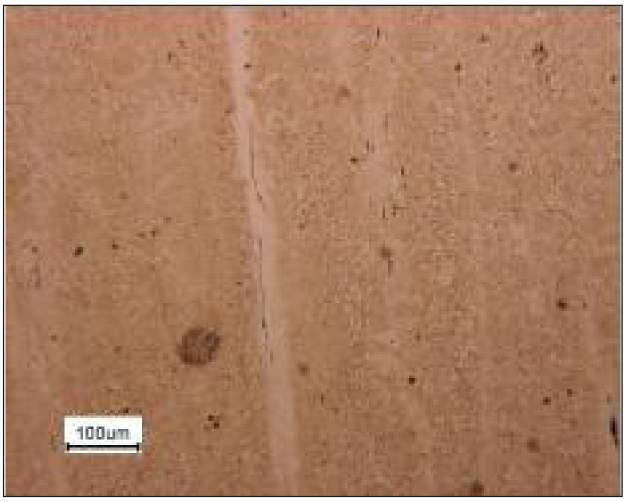

FIG. 6 ***Sample #11 microstructure: the band structure is clearly visible.***

Microstruttura di un campione normalizzato a 1050°C per 3 ore.

Fig. 8). This result confirms that the carbides precipitate during tempering and the austenitization process is adequate even if it is performed at 950°C.

SUMMARY AND CONCLUSIONS

In the present study nitriding problems on rolled rings were analyzed. Several nitrided samples were analyzed to better understand the cause of a too thin case depth, inadequate for the component industrial use.

The second aim of the work was to find an industrially applicable solution.

Microhardness and metallographic analyses conducted on the treated samples show rolling bands presence; these bands are oriented only along the rolling direction. These samples also show a close nitrides net near the surface and precipitated carbides at the component core.

Several normalized samples were heat treated (with different time-temperature parameters) and nitrided in order to identify the microstructural elements impeding the nitriding process. Performed tests underline that band structure and chromium carbides are responsible for the failure of the nitriding process.

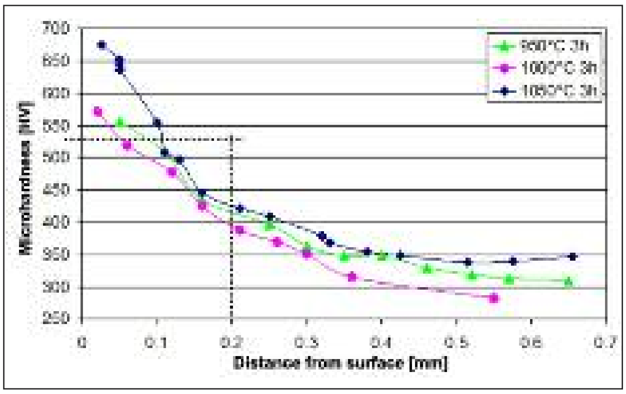

FIG. 7 ***Microhardness profiles of quenched, tempered (640°C) and nitride samples. Dashed lines represent the specific request on the effective case depth.***

Profili di microdurezza eseguiti su campioni temprati alle temperature indicate, rinvenuti a 640°C e nitrurati. Le linee tratteggiate danno un'indicazione dello spessore efficace.

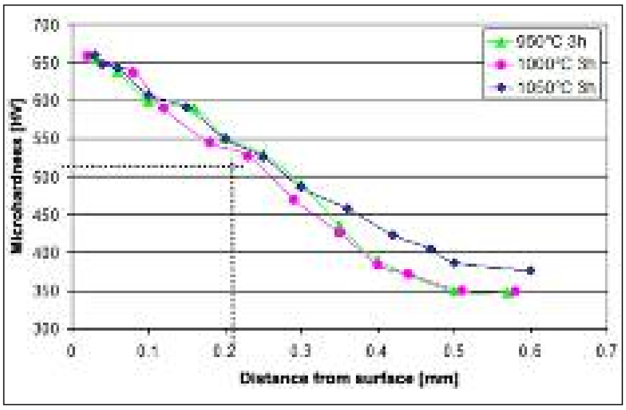

FIG. 8 ***Microhardness profiles of quenched, tempered (590°C) and nitride samples. Dashed lines represent the specific request on the effective case depth.***

Profili di microdurezza eseguiti su campioni temprati alle temperature indicate, rinvenuti a 590°C e nitrurati. Le linee tratteggiate danno un'indicazione dello spessore efficace.

Particularly band structure doesn't permit a regular and deep nitrogen diffusion; this structure couldn't be removed by common industrial heat treatments.

With regard to carbide formation instead, a microstructural control is possible. A solubilization treatment (high temperature austenitizing) is able to strongly reduce nitriding problems. The following tempering treatment shall be carried out at low temperature in order to avoid the chromium carbide re-precipitation.

This solution is able to resolve nitriding problems of specific components.

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Abstract

Problemi di nitrurazione su anelli laminati in 42CrMo4

Parole chiave: Trattamenti superficiali, Precipitazione, Acciaio

Per applicazioni meccaniche in cui è richiesta buona resistenza all'usura e alla fatica da contatto viene spesso impiegato l'acciaio 42CrMo4 e vengono normalmente richieste durezze superficiali dell'ordine di 600 HV e spessori efficaci di indurimento maggiori di 200 μm.

Tuttavia, nonostante gli attuali impianti di nitrurazione permettano un preciso controllo dei parametri di processo, accade talvolta che alcuni componenti presentino caratteristiche di indurimento inferiori a quelle attese per quel particolare acciaio.

Scopo di questo lavoro è stato determinare le cause che non hanno permesso di raggiungere le proprietà desiderate nello strato nitrurato realizzato su una corona dentata prodotta a partire da anelli laminati.

Sono state condotte analisi metallografiche su differenti campioni nitrurati provenienti da diverse colate. Sui componenti semilavorati (forniti allo stato normalizzato) sono stati eseguiti trattamenti di tempra in olio e rinvenimento. I diversi parametri di tempo e temperatura di trattamento hanno permesso di comprendere l'effetto della solubilizzazione dei carburi e dell'ingrossamento del grano cristallino. Le analisi eseguite, inoltre, hanno evidenziato come la scarsa durezza superficiale e la limitata profondità di penetrazione siano dovute alla particolare microstruttura in zone prossime alla superficie, alla struttura a bande e ad una precipitazione eccessiva di carburi nella matrice.