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CONTROLLED GAS NITRIDING OF 40HM AND 38HMJ STEEL GRADES WITH THE FORMATION OF NITRIDED CASES WITH AND WITHOUT THE SURFACE COMPOUND LAYER, COMPOSED OF IRON NITRIDES

Keywords

Controlled gas nitriding, nitriding potential, iron nitrides, nitriding atmosphere, diffusion layer.

Abstract

The article describes the results of investigations of controlled gas nitriding processes of alloyed structural steel grades 40HM and 38HMJ, used for machine components exposed to corrosion, wear and contact fatigue in service.

Examples are given of the process design enabling the formation of nitrided cases on alloyed steels with an iron nitride compound layer at the surface, designated for fatigue applications and as substrate for duplex processes.

Introduction

The article is dedicated to problems connected with the formation of nitrided cases on structural alloyed steel grades 40HM (4140 per AISI) and 38HMJ (Nitalloy 135M) with the presence of a surface iron nitride layer of

varying phase composition or without this layer, designated for application for precision components of machines, equipment and vehicles subjected to corrosion, wear and fatigue in service. The aim of this project was to develop nitriding processes enabling the formation of three types of nitrided cases:

- with an iron nitride layer with a dominant ϵ phase and a microstructure composed of $\epsilon + (\epsilon + \gamma'_{\text{precip.}})$, porous in its external zone, which facilitates its impregnation with a rust inhibitor,
- with a compact layer of iron nitrides and with a lesser proportion of the ϵ phase and a composition of $\epsilon + \gamma'_{\text{precip.}}$, with an external porous zone limited to a minimum ($\leq 2.5 \mu\text{m}$),
- without an iron nitride compound layer.

All nitrided cases, taking into account their expected exposure to corrosion, tribological or contact fatigue hazards, should be characterized by high hardness as well as appropriate thickness of the iron nitride layer and its particular zones for corrosion resistance (first two cases), or by an appropriately deep effective case depth, to counteract contact fatigue (third type of case). Cases containing the compound nitride layer should also be characterized by a limited thickness of the latter, on account of the narrow dimensional tolerances of the components for which they are designated. They should, moreover, meet the requirements regarding corrosion resistance.

It should be emphasized that literature concerning anti-corrosion nitriding of alloyed steels contains substantially less publications than in the case of carbon steels. This is especially true of publications dealing with the formation on alloyed steels of thin, corrosion-resistant iron nitride compound layers [1-5]. Similarly, the problem of formation of nitrided cases without compound layers on alloyed steels, despite a definite interest in them for fatigue applications, is limited to only very few mentions, these being mainly concentrated on Nitralloys [6-8].

1. Materials used

For the investigations, two alloyed steels were used, the 40HM and 38HMJ grades, widely used for nitriding. These steels were quenched and tempered prior to nitriding. The heat treatment parameters and hardnesses are given in Table 1.

Table 1. Parameters of heat treatment

Steel grade	Heat treatment parameters	Hardness	
		HV10	HRC
40 HM	Q. 860°C/ oil		
	T. 490°C	395	40
	T. 570°C	329	33
	T. 600°C	327	33
38HMJ	Q. 920°C/ oil		
	T. 490°C	473	47
	T. 570°C	390	40

For the investigations, coupons, sized $\phi 24 \times 4$ mm, ground to $Ra = 0.32 \mu\text{m}$ were used.

2. Nitriding equipment

Nitriding of the coupons was carried out in an industrial pit-type furnace, model Nx609, manufactured by Nitrex, with a retort of $\phi 600 \times 900$ mm dimensions and computer process control. This furnace is equipped with an ammonia dissociator, as well as a system of fast cooling of the load after the process, as well as with a neutralizer of the effluent atmosphere. Computer process control enables precise control of the composition, flow rate and nitriding potential of the atmosphere, as well as of the temperature and time of the cycle. It also enables monitoring and full recording of all process parameters during each of its stages, very useful in the analysis and design of nitriding cycles.

3. Nitriding processes

Nitriding processes were carried out at temperatures within the range of 470 – 580°C and times of 4 – 28 h in atmospheres composed of ammonia or a mixture of ammonia and dissociated ammonia. Process parameters varied, depending on the type of cases and layers produced.

Nitriding with the formation of layers with a porous iron nitride zone were carried out at the higher end of the temperature range (570 – 580°C) for 4 h, in atmospheres composed of ammonia with the cycle ending on a nitriding potential $N_p = 1.97 - 2.76$, which facilitated the rapid formation of nitride compound layers.

For the formation of nitrided cases with a compact iron nitride layer, lower nitriding temperatures were implemented (560 and 530°C), times used were 4 – 12 h and the atmospheres were composed of ammonia and dissociated ammonia, with a lower nitriding potential of 1.38 – 2.56.

For the formation of cases free of the iron nitride layers, the nitriding temperatures were even lower (530 – 470°C), while times were longest, reaching 28 h, and atmospheres composed of ammonia and dissociated ammonia had very low nitriding potentials (0.23 – 0.38). The implementation of low, non-conventionally used temperatures (470°C) facilitated the retention of a high core hardness ($\geq 40\text{HRC}$), advantageous from the point of view of contact fatigue strength.

In the first two instances (i.e. cases with iron nitride compound layers at the surface), atmospheres with a fixed nitriding potential throughout the cycle or its stage were used. In the third instance (nitrided cases without a compound layer at the surface), the nitriding potential was gradually lowered until it reached a

very low-end value. During the running of the process, control of the atmosphere was implemented throughout the cycle, including the heat-up stage. Changes in the nitriding potential during the heating up and at temperature were selected, based on the Lehrer diagram in such a way that they were as close as possible to the boundary values of the potential between the α , γ' and ϵ phases on that diagram [9, 10]. They were also selected, depending on the needs, below and above the boundary values. Such potential variations were verified experimentally, choosing the most advantageous running of the process from the point of view of strengthening the nitrided case and the kinetics of its growth.

4. Post-nitriding investigations

Following the nitriding processes, investigations encompassed the following:

- surface hardness (H_s), maximum hardness (H_{\max}) and core hardness (H_{core}),
- hardness traverses across the nitrided case,
- microstructure of nitrided cases,
- phase composition of the iron nitride compound layers,
- compound layer thickness (CLT) and of its porous zone (PZT),
- effective hardened case depths ($ECD_{\text{core}+50}$, ECD_{400} , ECD_{600}).

Hardness measurements were conducted on a semi-automatic hardness tester manufactured by Zwick.

Investigations of microstructure of nitrided cases were carried out on cross-sections of the coupons, with the aid of an optical microscope manufactured by Zeiss. The optical microscope was also used for the measurement of compound layer thickness, with its particular zones. The phase composition of iron nitrides was determined with the aid of an X-ray diffractometer, employing $\text{CoK}\alpha$ radiation. Effective case depths were determined based on hardness distribution curves.

5. Examples of nitriding processes

The chosen examples were three processes which enabled the formation on 40HM and 38HMJ grades of three types of nitrided cases with the most advantageous structural and usable features (case depth and hardness) from the point of view of expected applications.

Nitrided cases without the external iron nitride compound layer, designated for applications exposed to contact fatigue stresses were produced on both grades of steel in a process carried out at a temperature of 470°C (Nx616). On the other hand, nitrided cases with a compact iron nitride compound layer (Nx610), or a porous layer (Nx613), designated for anti-corrosion and tribological applications, were produced in processes carried out at 560°C (compact layers) and 570°C (porous layers). Nitriding times of the successive processes were 28, 12 and 4 h.

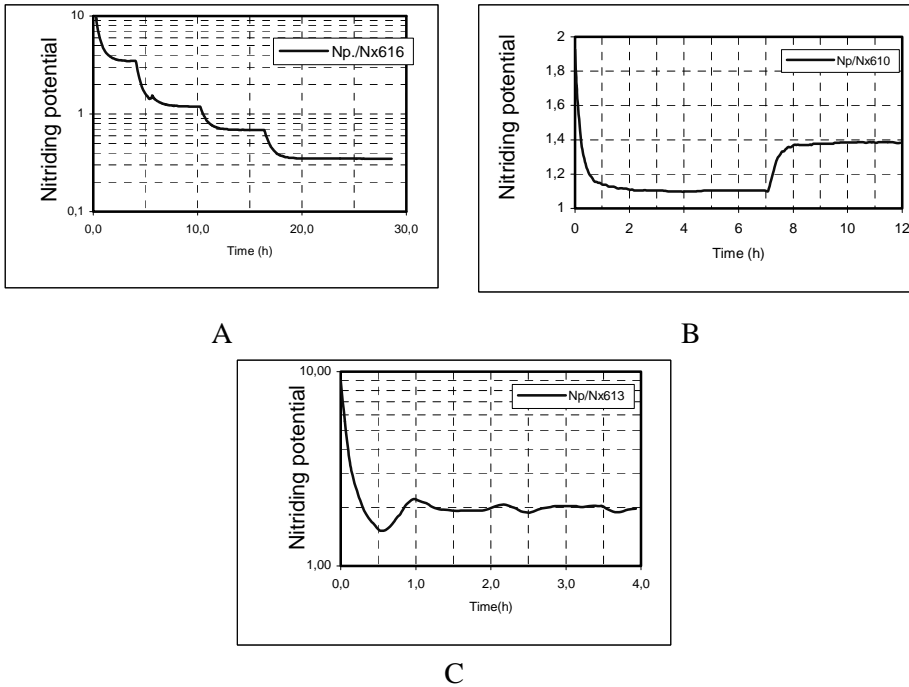


Fig. 1. Variations of the atmosphere nitriding potential value with time for: controlled gas nitriding, enabling the formation, on 40HM and 38HMJ steel grades of nitrided cases of the following composition: A – without a compound layer (proc. Nx616), B – with a compact compound layer (proc. Nx610), C – with a porous compound layer (proc. Nx613)

In each of the three processes, a significant role was played by the nitriding atmosphere, the potential of which was designed appropriately to microstructure of the layer, required of that process. Variations of the nitriding potential during each of the processes are shown in Fig. 1. The most complex course of variation of the nitriding potential was that in the process coded Nx616, facilitating the formation of nitrided cases without the external iron nitride compound layer. In this process, the nitriding potential was gradually lowered from the initial value above 10, through intermediate values of 3.5, 1.2 and 0.7 down to the end value of 0.34, slightly lower than the boundary potential between the α and γ' phases on the Lehrer diagram, which, at a temperature of 470°C is equal to 0.37.

In the case of the second process, conducive to the obtaining of a nitrided case with a compact iron nitride compound layer (process Nx610), the values of the nitriding potential were varied in two steps from the initial value of 1.8, through the intermediate value of 1.1 to a final value of 1.32, higher than the boundary potential between the γ' and ϵ phases on the Lehrer diagram, the value of which is 1.13.

In the case of the third process (Nx613), the purpose of which was to obtain a nitrided case with a porous surface layer of iron nitrides, the nitridding potential during the entire process, with the exception of the initial short lowering of its value from 10 to 1.5, remained at a fixed value of 2. This potential was clearly higher than the boundary potential between the γ' and ϵ phases, which according to the Lehrer diagram, is 1 at the temperature of 570°C.

The consequence of processes carried out in such a manner was the obtaining of cases with the desired microstructure, as proven by x-ray diffraction studies, the results of which are shown in Fig. 2 for the example of

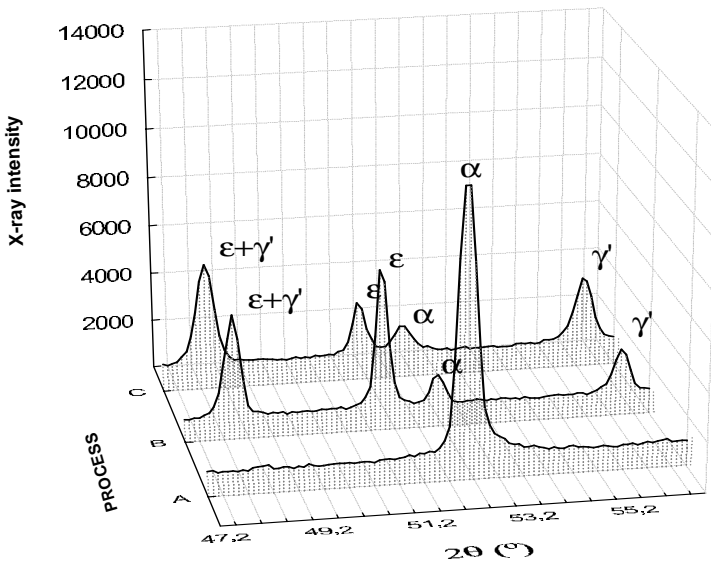


Fig. 2. Results of investigations of phase composition of nitrided cases on 40HM grade steel: A – without a compound layer, B – with a porous compound layer (ϵ phase = 67%), C – with a compact compound layer (ϵ phase = 35%)

the 40HM grade. In subsequently run processes, a nitrided case was obtained with a microstructure of the solid solution of $Fe_{\alpha}(N)$ without an iron nitride compound layer (A), with a compact iron nitride compound layer (C) and a phase composition of $\epsilon + \gamma'$ and a smaller proportion of the ϵ phase (35%), as well as with a porous compound layer (B) with a phase composition of $\epsilon + (\epsilon + \gamma')$ and a greater amount of the ϵ phase (67%). This phase composition was also confirmed by metallographic investigations (Fig. 3) which comprised, besides hardness and effective case depth, also the thickness of the compound layers and its zones (Fig. 3 c, d).

As it can be seen from results of measurements of nitride layer thicknesses, put together in Fig. 3 c, d, for the process Nx616, no iron nitride compound

layer was formed on either 40HM or 38HMJ steels. On the other hand, in the remaining processes (Nx613 and Nx610) a nitrided case was obtained with a porous compound layer and a thickness of the order of 16.5 and 18.5 μm (Nx613), as well as one with a significantly thinner compact compound layer of 10 and 12.5 μm with no porous zone on the 38HMJ grade and a 2 μm thick porous zone for the 40HM grade (Nx610).

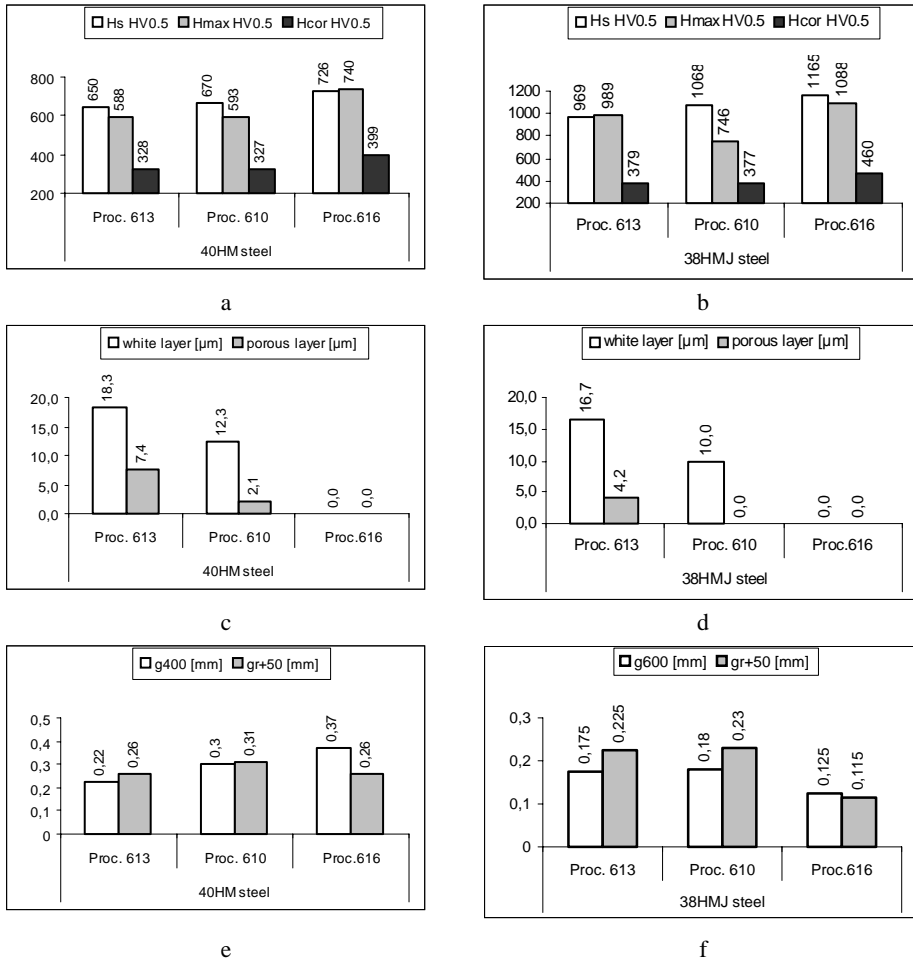


Fig. 3. Comparison of surface maximum hardness values and core hardnesses (H_S , H_{max} , H_{core}), compound layer thickness (CLT) and porous zone thickness (PZT), as well as effective case depths ECD_{400} and ECD_{c+50} for steel grade 40HM and ECD_i ECD_{cor+50} for steel grade 38HMJ. Nitrided cases:

- without a compound layer (proc. Nx616)
- with a compact compound layer (proc. Nx610)
- with a porous compound layer (proc. Nx613)

A comparison of hardnesses of the nitrided cases obtained as a result of the three processes discussed (Fig. 3 a, b) indicates that the highest surface hardness and maximum hardness values (exceeding 700 HV0.5 for the 40HM grade and exceeding 1100 HV0.5 for the 38HMJ grade) were exhibited by nitrided cases without compound layers obtained in the process Nx616 at the low temperature (470°C). In the remaining two processes (Nx610 and Nx613), carried out at temperatures of 560 and 570°C, which yielded compact and porous compound layers, surface and maximum hardness values were lower.

It should be emphasized that satisfactory effective case depths were obtained on both grades of steel (Fig. 3 e, f), especially in the case of the low temperature process (Nx616), designated for these applications, which were to be exposed to contact fatigue.

Conclusions

1. Process parameters assumed here, in particular those of the nitriding atmosphere, as well as variation of the nitriding potential with time, allowed the obtaining of nitrided cases without the iron nitride compound layer at the surface, as well as with either a compact or a porous compound layer with a controlled phase composition.
2. Nitrided cases, in particular those without the compound layer at the surface, were characterized by a high hardness, which is of significance when considering the expected application with fatigue hazard.
3. The obtained thicknesses of iron nitride compound layers and of their porous zones were conformable with thicknesses required of iron nitride compound layers to be used in corrosion applications (minimum 8 μm of the compact zone).
4. A significant result of the investigations was the demonstration that it is possible to effectively nitride alloyed steels such as 40HM and 38HMJ at a low temperature (470°C), making it possible to obtain high core hardnesses ($\geq 40\text{HRC}$), advantageous when applied to contact fatigue stresses.

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Regulowane azotowanie gazowe stali 40HM, 38HMJ z wytwarzaniem warstw azotowanych z i bez powierzchniowej warstwy azotków żelaza

Słowa kluczowe

Regulowane azotowanie gazowe, potencjał azotowy, azotki żelaza, atmosfera azotowania, warstwa dyfuzyjna.

Streszczenie

W artykule omówiono wyniki badań procesów regulowanego azotowania gazowego konstrukcyjnych stali stopowych gatunków 40HM i 38HMJ, używanych na części maszyn, narażone w eksploatacji na korozję, zużycie przez tarcie i zmęczenie stykowe. Do azotowania stosowano piec wglębny z automatycznym sterowaniem komputerowym procesem, wyposażony w dysocjator amoniaku i system przyspieszonego chłodzenia wsadu. Procesy azotowania prowadzono w zakresie temperatur: 470–580°C i czasów 4–28 h, w atmosferach z NH_3 bądź z $\text{NH}_3 - \text{NH}_{3\text{zdys}}$. z regulowaniem ich składu i potencjału azotowego w okresie nagrzewania i w temperaturze procesu. Podano przykłady rozwiązań procesu umożliwiające wytwarzanie warstw azotowanych na stalach stopowych, z warstwą azotków żelaza przy powierzchni, przeznaczone do narażeń korozyjnych i bez warstwy azotków żelaza, przeznaczone do narażeń zmęczeniowych i procesów podwójnych typu duplex.