NITRIDING LAYER STRUCTURE WITH DIFFERENT SURFACE TREATMENT

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Abstract. Two tool steels were employed in this work, AISI H-13 and DIN 12367 the samples were treated to obtain different surfaces finishing. After that the samples were nitride in a gas atmosphere and salt bath environment, the depth and the hardness of the nitriding layer formed was measured and the structure was identified by X-ray diffraction.

Introduction

For a tool steel to perform effectively, it is usually required a quenched and a heat treatment, these treatments will enable the tool steel to develop their properties required for a high hardness with good wear, abrasion and impact resistance. For heat treat a tool steel component requires homogeneous preheating and then raising it to its final hardening temperature. To achieve a high hardness the tool steel then requires to be quenched in oil, air or inert gas. After that tool steel is quenched and needs to be tempered three times before nitriding. Nitriding is a surface-hardening heat treatment that introduces nitrogen into the surface of steel at a temperature range from 500 to 650°C[1,2,3,4,5].

In this work we investigate the relationship between surface finishing and the nitriding layer in a tool steel system and the structure of these materials were investigated by X-ray diffraction the presence of retained austenite was identified and the amount of each phase present in the tool steels were quantified by rietveld method. Two different tool steels (AISI H13 and DIN 1.2367) were investigated in this work.

Method

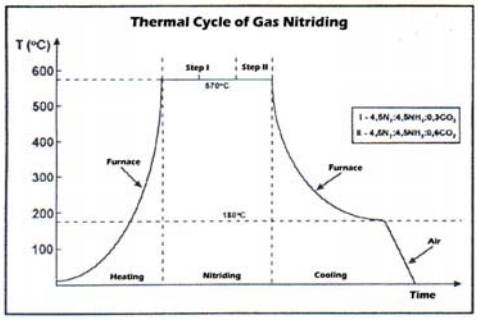
The samples of AISI H – 13 and DIN 1.2367 were quenched, tempered and nitrided in gas atmosphere or in salt bath (2 or 8h)[6,7,8,9,10,11]. The tool steels AISI H13 and DIN 1.2367 in order to obtain different surface finishing, before the nitriding process, they were machined or rectified (Industrial surface finishing). Another surface treatment realized were human ground 240 mesh (HG 240), human ground 600 mesh (HG 600) and polishing 3μm (laboratory surface finishing). All samples were obtained from Aços Böhler, and treated at Brasimet Comércio e Indústria S/A. Initially the samples were analyzed, as received, and the composition, microstructure and hardness were checked.

Thermal treatment:

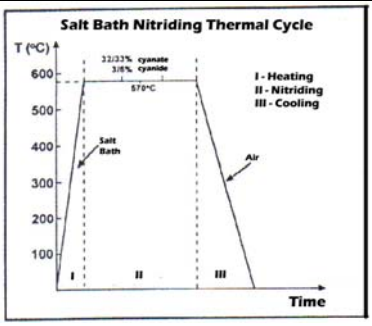
The samples were cleaned and them quenched in a vacuum furnace model VKUQ and followed by three tempering stages. After quenching and tempering the samples, they were nitride by the following process whose thermal treatment cycles are presented in Figure 1:

- Gas nitriding:Deganit® process with working temperature (520-570°C) during 6 hours.

- Liquid nitriding: Tenifer® process with working temperature (550 – 570°C) during 2 and 8 hours.



a) Gas nitriding thermal treatment cycle



b) Salt bath nitriding thermal treatment cycle

Figure 1: Nitriding cycles of the thermal treatment.

Microstructure analysis:

The samples were analyzed by optical microscopy and a thin copper foil was used in the samples to allow the identification of the nitride case[12]. The samples were etched by Nital 3% and analyzed in Karl Zeiss microscopy with magnification of 500x and the micrographs were obtained by Olimpus BX 60M and Sony Video Printer. The depths of the nitriding cases were measured by Vickers hardness according to DIN 50.190.

X-rays diffraction:

The x-ray diffraction were performed in Rigaku Multiplex Model diffractometer with silicon monochromatized CuKα radiation, with 2θ scanned from 10 to 90° and scan rate of 0.05° s^{-1}.

Results and Discussion

In Figure 2 are presented the structures of the AISI H13 and DIN 1.2367 in the different conditions investigated in this work "as received" (annealed, espheroidized), quenched and tempered. In Figure 3 one can see the photomicrographies of the different samples after gas nitriding (a), salt bath nitriding for 2 hours (b) and salt bath nitriding for 8 hours (c). In Figure 4 the relationship between surface finishing before nitriding x roughness (Ra) and the nitriding depth case obtained for gas nitriding, salt bath nitriding during 2 h and salt bath nitriding during 8 h are plotted.

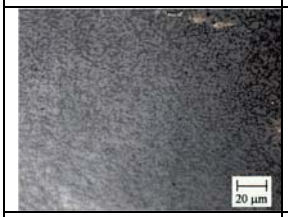
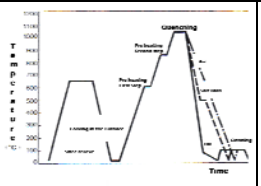
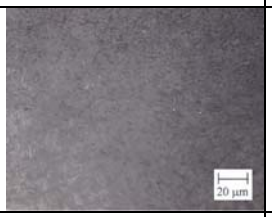
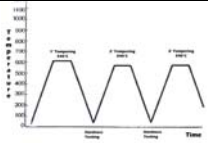
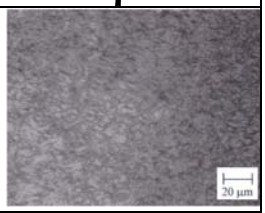
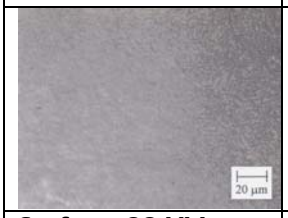
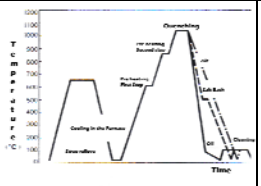
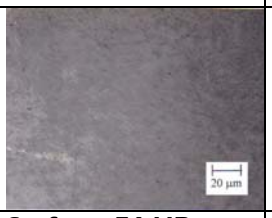
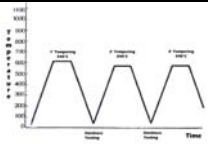
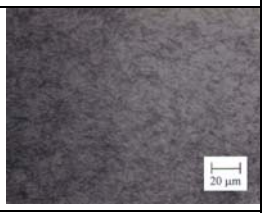
As received	Quenching	Quenched	Tempering	Tempered
				
Surface: 95 HV Center: 94 HV	AISI H - 13	Surface: 53 HRc Center: 52 HRc		Surface: 49 HRc Center: 48 HRc
				
Surface: 88 HV Center: 87 HV	DIN 1.2367	Surface: 56 HRc Center: 55 HRc		Surface: 51 HRc Center: 51 HRc

Figure 2: The structure of the tool steels.

Din 1.2367 Polish	H – 13 Polish		
Din 1.2367 HG 240	H – 13 HG 240	Din 1.2367 HG 600	H – 13 HG 600
Din 1.2367 Machined	H – 13 Machined	Din 1.2367 Rectified	H – 13 Rectified

a) Gas Nitriding

Din 1.2367 Polish	H – 13 Polish		
Din 1.2367 HG 240	H – 13 HG 240	Din 1.2367 HG 600	H – 13 HG 600
Din 1.2367 Machined	H – 13 Machined	Din 1.2367 Rectified	H – 13 Rectified

b) Salt bath nitriding for 8 hours.

Din 1.2367 Machined	H – 13 Machined	Din 1.2367 Rectified	H – 13 Rectified
Din 1.2367 HG 240	H – 13 HG 240	Din 1.2367 HG 600	H – 13 HG 600
Din 1.2367 Polish	H – 13 Polish		

c) Salt bath nitriding for 2 hours.

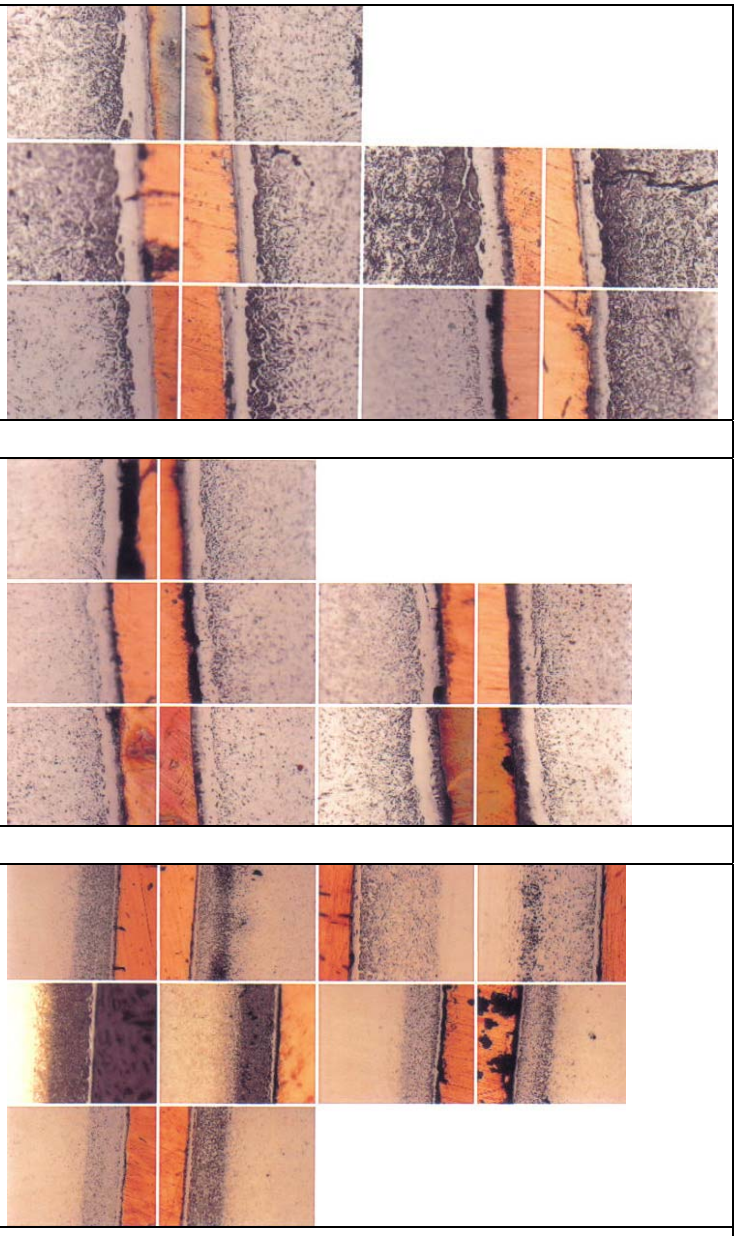
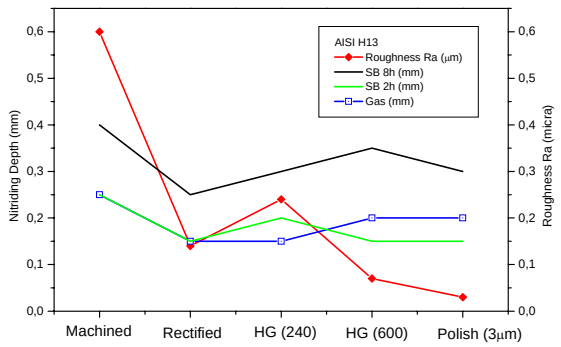


Figure 3: Photomicrographies of the tool steels after nitriding.

One can see in Figure 3 that all the nitriding process formed a white layer followed by a diffusional layer in all samples. The nitriding time of the treatment has a direct effect in the depth case, in fact the depth case formed by nitriding in salt bath during 8h produces the biggest cases for all process.

The depth case formed by nitriding in salt bath during 2h produces similar depth cases for all samples. The results presented in Figure 4 and obtained for salt bath nitriding during 8h indicates that for industrial process of surface finishing the depth case is directly proportional to roughness, while for laboratory surface finishing HG240 and HG600 the depth of the case increases when roughness decreases. The gas nitriding process reveals a tendency in decreasing the depth case for rectified and polish process, this fact is well known by practioners.

In Figure 5 are presented three diffractograms that were obtained for gas nitriding, salt bath nitriding for 2 and 8 hours. The other samples presented similar diffractograms. One can see that for salt bath nitriding some residual austenite were presented indicating that these tool steels will probably not perform well. The other nitriding methods there was no austenite retained. After the phase identification was performed the quantitative analysis of the phases for gas nitriding and for salt bath nitriding during 8 hours[13,14]. The results are presented on Table 1.



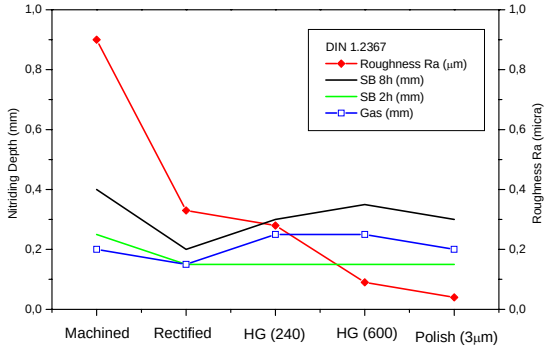
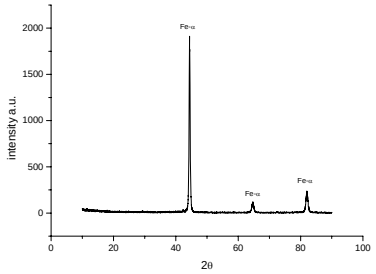
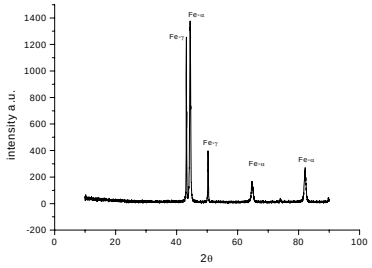


Figure 4: AISI H-13 and DIN 1.2367 roughness and depth nitriding x surface treatment.





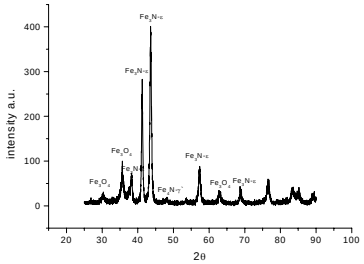


Figure 5: diffractogram of the 3 nitriding process.

a) AISI H13 polished, gas nitriding for 6 hours (Deganit).

b) DIN 1.2367 HG 240 salt bath nitriding for 2 hours.

c) AISI H13 polished, salt bath nitriding for 8 hours.

Table 1: Quantitative analysis of the phases using rietveld method.

Sample	Fe_3O_4 [%wt]	ε-Fe_3N [%wt]	γ'-Fe_4N [%wt]
SB 8h - AISI - H13- Machined	61.5	38.5	n/d
SB 8h - AISI - H13- HG 240	50.7	49.3	n/d
SB 8h –AISI – H13 –HG 600	45.3	54.7	n/d
SB 8h - AISI - H13- Rectified	51.0	49.0	n/d
SB 8h - AISI - H13- Polish	68.4	31.5	n/d
SB 8h - DIN 1.2367- Machined	53.9	46.1	n/d
SB 8h - DIN 1.2367- Rectified	41.3	51.7	n/d
SB 8h - DIN 1.2367- HG 240	48.1	47.7	4.1
SB 8h - DIN 1.2367- HG 600	28.3	65.6	6.2
SB 8h - DIN 1.2367- Polish	28.6	64.4	7.0
Deganit- AISI - H13- Machined	18.7	79.3	1.9
Deganit- AISI - H13- Rectified	2.3	97.6	n/d
Deganit- AISI - H13- HG 240	16.0	84.0	n/d
Deganit- AISI - H13- HG 600	11.2	88.8	n/d
Deganit- AISI - H13- Polish	14.0	86.0	n/d
Deganit-DIN 1.2367- Machined	14.1	85.9	
Deganit--DIN 1.2367- Rectified	14.1	85.9	n/d
Deganit--DIN 1.2367- HG 240	14.2	85.7	n/d
Deganit--DIN 1.2367- HG 600	11.4	88.5	n/d
Deganit-DIN 1.2367- Polish	17.2	82.7	n/d

Nitriding mechanism is generally known, but some specific reactions that occur in different steels and different nitriding media sometimes are unknown. Since nitrogen has partial solubility in iron it can form a solid solution with ferrite when nitrogen concentration is up to 6%. At 6% of nitrogen is formed Fe_4N called (γ'). With nitrogen content greater than 8% the reaction product is Fe3N (ε). Fe_4N were identified only on samples SB 8h - DIN 1.2367- HG 240, DIN 1.2367- HG 600, DIN 1.2367- Polish and Deganit- AISI - H13- Machined. . As the ε zone of the case is hardened by the formation of the Fe_3N compound, and below this layer there is some solid solution strengthening from the nitrogen in solid solution one can identify that the gas nitriding process produces more Fe_3N phase than the others, according to the data of table 1[15].

Conclusions

All the nitriding process formed a white layer followed by a diffusional layer in all samples.

The nitriding time of the treatment has a direct effect in the depth case.

The depth case formed by nitriding in salt bath during 8h produces the biggest cases for all process.

The depth case formed by nitriding in salt bath during 2h produces similar depth cases.

The results obtained for salt bath nitriding during 8h indicates that for industrial process of surface finishing the depth case is directly proportional to roughness. While for laboratory surface finishing HG240 and HG600 the depth of the case increases when roughness decreases.

The gas nitriding process reveals a tendency in decreasing the depth case for rectified and polish process, this fact is well known by practioners.

The salt bath nitriding for 2 hours was the only process that presents retained austenite.

The samples SB 8h - DIN 1.2367- HG 240, DIN 1.2367- HG 600, DIN 1.2367- Polish and Deganit- AISI - H13- Machined presented Fe_4N compound.

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