

Effect of shot peening on the gas nitriding of iron compounds produced by powder metallurgy

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Abstract

The aim of this work was to evaluate the gas nitriding behavior of iron sintered samples as received and previously submitted to shot peening process. The results have shown that the as received samples did not present white layer only at the surface but also around the porous in the bulk of the sample. This fact suggests that the gaseous atmosphere goes through interconnected porous resulting in the embrittlement of the component. Samples previously submitted to shot peening process before gas nitriding showed an external white layer but also permitted the access of nitriding atmosphere to the bulk of the sample. In this case white layer around internal porous did not form and numerous acicular nitrides were observed.

Introduction

Sintered materials are produced by the compaction of one or more powders, metallic or not, in dies, that can have the final dimension of products, followed by sintering at high temperatures. Materials produced by this route have as advantage the need of low transformation energy, but the specimen is porous (1).

Nitriding is a well-known thermochemical process that consists in the introduction of atomic nitrogen by diffusion in the lattice of ferrous alloys in the temperature range from 500-590°C. The nitrogen species that diffuse combine with iron and alloy elements to form nitrides that modify physical and mechanical properties of the surface of the alloys. Nitriding is known to modify properties such as wear, fatigue and corrosion resistance, besides microhardness (2, 3). This treatment allows producing, for example, materials with high superficial hardness and a toughness nucleus that has the advantage of being resistant to cracks propagation. The most widely used nitriding processes are gas nitriding, salt bath nitriding and plasma nitriding.

Many difficulties are found when sintered samples are nitrated and it varies according to the process utilized. Gas nitriding is one of the most widely used surface modification technique to improve surface hardness, anticorrosive properties, and wear resistance of metallic materials by formation of a surface nitrated layer (4). In gas nitriding of sintered iron the gas penetrates not only in the surface, but in a large volume of the sample where high surface specific area is found and forms nitrides randomly in all this volume. It results in a high distortion and embrittlement of the component.

Salt bath nitriding has as advantage, the possibility of control of the chemical composition of the bath and of lowering the operating temperature by changing the chemical composition (5). For sintered samples, liquid salt penetrates along the interconnected pores and are of difficult removal at the end of the process (6). In addition, there are environmental restrictions on the use of these salts

(7). The solution found for nitriding these materials is plasma nitriding, which has only a superficial effect. In recent years, this process has been extensively used to improve the surface properties of a wide variety of materials such as screws and cylinders of plastic-processing machines (8). Plasma nitriding, although the better control over the metallurgic properties of the layer has as disadvantage the expensive costs of implantation and operation.

Shot peening is a mechanical process, widely used in the industry to improve the fatigue resistance of components and structures (9). Residual stresses, surface roughness, and work hardening are the main beneficial effects inducted in the surface layer from shot peening, which depend on the choice of the peening parameters (10). In this process, impacting a metal surface with small steel balls at high velocity causes large plastic deformation. Overlapping dimples develop a uniform layer of residual compressive stress (9). According to K.Funatani (5), nitriding is favored by shot peening.

The aim of this work was to evaluate the microstructural changes resulting from gas nitriding of sintered iron specimens previously submitted to shot peening process.

Methods and Materials

The materials studied are sintered specimens produced from a 50%-mixture of iron powders water atomized. The chemical compositions of these powders in wt % are shown in Table I. Rectangular-shaped bars with densities of 6.5 g/cm³ were used. This material was selected due to its large use in automotive components submitted to high attrition operation conditions.

Table I – Chemical composition of the powders

	Fe (min)	C	O	S	Mn
Powder 1	99.4%	0.05%	0.17%	0.005%	0.008%
Powder 2	99.4%	0.003%	0.08%	0.007%	0.20%

Samples with 12 x 15 x 85 mm were peened on two opposite faces that presented 85 x 12 mm exposed area, at room temperature.

Nitriding of as received and after shot peening specimens was carried out in a gas furnace at 520°C, following the cycle indicated in Table II.

Table II: Nitriding cycle carried out at 520°

Description	Time (h)
Heating in N ₂	1
CO ₂ flux = 0.3 m ³ /h N ₂ flux = 4.5 m ³ /h NH ₃ flux = 4.5 m ³ /h	4
CO ₂ flux = 0.6 m ³ /h N ₂ flux = 4.5 m ³ /h NH ₃ flux = 4.5 m ³ /h	2
Cooling in N ₂	1

The metallographic characterization was performed in an optical microscope, model BX60, and the images were registered by using the software Image Pro-express Version 5.1. Cross section of samples was observed. Nital 1% was used to reveal the microstructure.

The hardness tests were performed in a micro-Vickers HXD 1000TM Pantec – Panambra equipment.

Results and Discussion

Figure 1a shows the as received material. It can be seen large and numerous pores near the surface of the sample. This fact results from de attrition between the powder and the internal surface of the mould during the compaction process. The attrition turns difficult the compaction and thus the volume of powder that is in a large distance from the surface of the mould during this process tends to be less dense in the sintered sample. Figure 1b presents the same material after shot peening process. It can be seen the region affected by the shot peening, by the observation of elongated grains and by the closure of superficial pores.

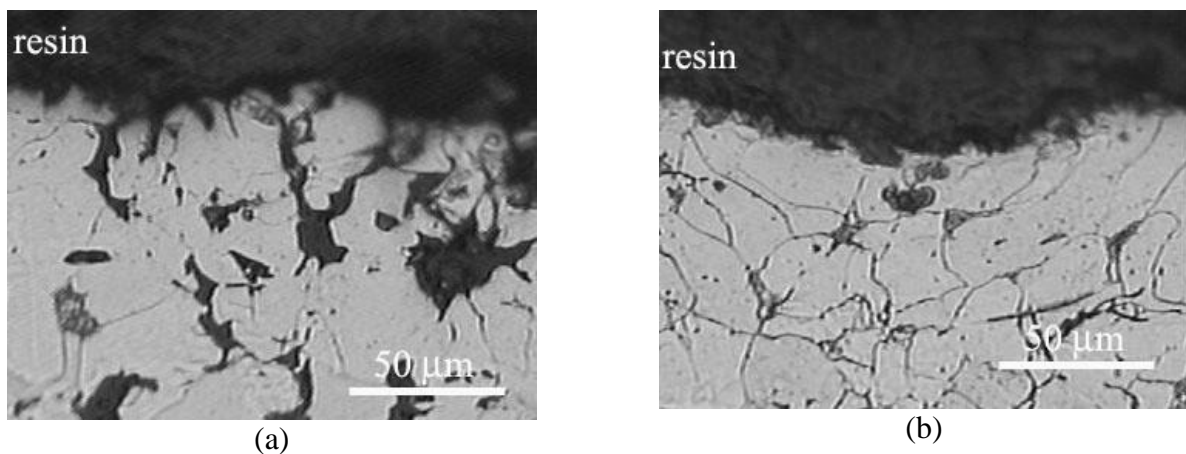


Figure 1: Specimen cross section in a region near surface. (a) as received; (b) peened.

The plastic deformation resulting from shot peening process creates defects in the surface of the material and increases its internal energy. The difference of energies between the deformed material and the same material without any deformation is the driving force to recrystallization (11). For commercially pure iron, the temperature of recrystallization is about 450°C (11).

J.F.Gu (2) observed that in cast nitrided samples, the growth rate of white layer obey a linear law. As the process goes on, the growth rate of the nitride layer obeys a parabolic rate law. Otherwise, the same material previously submitted to shot peening process do not present linear kinetics, but a parabolic one since the beginning, controlling the diffusion rate of nitrogen. Nitrogen diffusivity depends on many factors including lattice structure, grain size, chemical composition and defect density (12, 13).

The cross section near the surface of the peened and nitrided at 520°C sample is shown in Figure 2a. The nitriding atmosphere passes through interconnected pores resulting on the formation of white layer surrounding the pores in the bulk of the sample, as it can be seen in Fig. 2b, suggesting that the nitriding atmosphere access the nucleus of the sample.

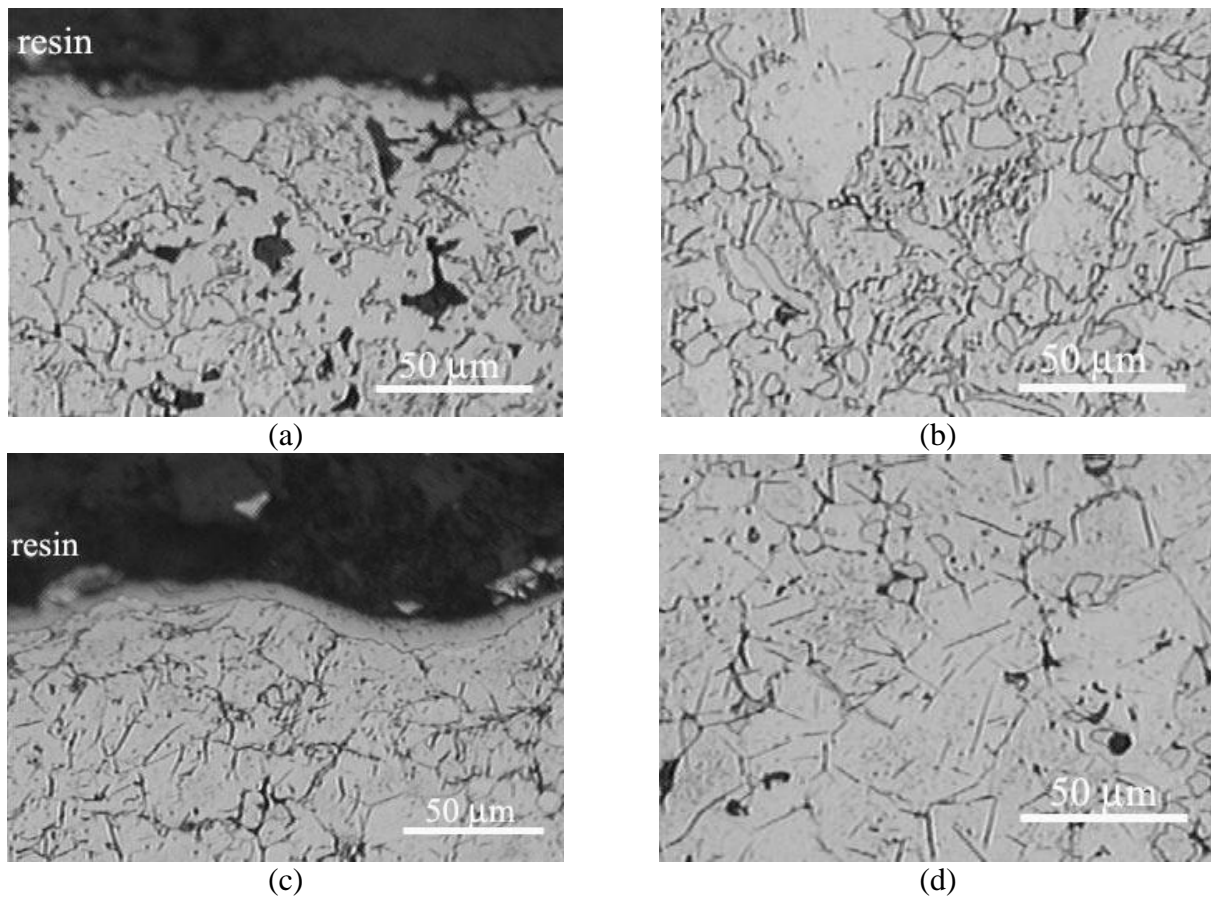


Figure 2: (a) Cross-section of the sample nitrided at 520°C, near surface; (b) Cross-section of the sample nitrided at 520°C, nucleus; (c) Cross-section of the sample peened and nitrided at 520°C, near surface; (d) Cross-section of the sample peened and nitrided at 520°C, nucleus.

Figure 3a presents the sample peened and nitrided at 520°C. The micrograph shows the cross section near the surface of the sample. The white layer presents irregular thickness, with a maximum value of 12 μm . Acicular nitrides are also observed.

Figure 3b presents the nucleus of the sample peened and nitrided at 520°C. Acicular nitrides are seen until the nucleus of the specimen. The establishment of a superficial continuous nitride layer in the surface was responsible for closing the opened pores and for limiting the access of the nitriding atmosphere, inhibiting the formation of white layer in the surface of internal pores in the bulk of the sample.

Figure 4 presents the diffraction diagram of peened sample nitrided at 520°C. It suggests the presence of the phases Fe_3N , hexagonal and Fe_4N , cubic.

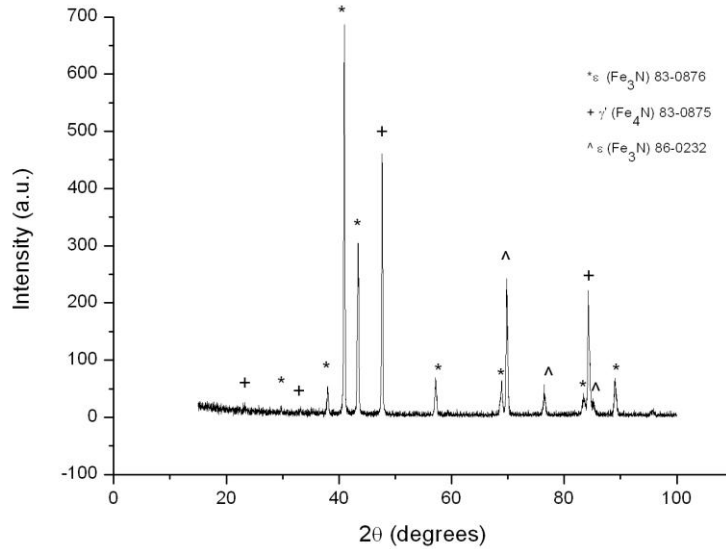


Figure 4: X-ray diffraction spectrum of the specimen peened and nitrided at 520°C.

Table III – Hardness of the samples

Sample condition	50µm depht	Nucleus
As received	60 HRF	60 HRF
Peened	218 HV	80 HV
As received + nitrided	202 HV	172 HV
Peened + nitrided	255 HV	148 HV

Table III presents the values of hardness obtained at 50 µm depth under the top of the surface, and in the nucleus of the sample. A superficial hardness enhance was observed and it was attributed to the shot peening process. The difference in hardness between the region near the surface and core material in nitrided samples can be attributed to the hardening of the matrix, which contains nitrogen in solid solution.

Conclusions

1) In the nitriding of sintered materials, the interconnected porous, characteristic of materials produced by this process, allow the penetration of the nitriding atmosphere inside the sample, forming nitrides in a high depth of the sample or even in the whole sample.

2) The hardness in the nucleus of samples not previously peened is higher than for the peened samples after nitriding. This fact suggests that the shot peening process influences the nitriding process and difficults the access of nitrogen ions to the bulk of the sample.

3) Shot peening process used in sintered samples difficults the nitrogen access and favors the white layer formation on the surface, but did not inhibit the formation of acicular nitride in the whole sample.

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