Surface Patterning of Low Carbon Steel via Dc Plasma Nitriding

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Abstract

The surface patterning by plasma nitriding treatment was applied in low carbon steel SAE 1004 and presented in this paper. The same grids used in sample preparation to Transmition Electron Microscopy (TEM) were placed on the surface of steel and DC plasma nitriding treatment was performed following the parameters: temperature of 540 °C, gas mixtures with $5\%N_2/95\%H_2$ *and* $95\%N_2/5\%H_2$*, and time treatment of 2 hours. The samples were characterized by optical microscopy (OM), scanning electron microscopy (SEM) and, optical interferometry. The results showed presence of grid caused a patterning of the surface. Besides, the compound layer presented a discontinuity, which characterizes the feasibility of technique in the production of patterned low carbon steel parts.*

Keywords: surface patterning, DC plasma, nitriding

Introduction

In the last decade, it has been developed products with surface regular arrangements called patterned surfaces. They have a wide application in various areas of engineering and their surface properties cannot be compared with non patterned treated products (Evans & Brian, 1999)(Bruzzone, H., Lonardo, & Lucca, 2008). Structured surfaces are also widely recommended in literature to reduce friction and wear between moving parts. For these surfaces, micro-topographies have shown better results tribological when compared with usual finishes(Etsion & Sher, 2009).

According to the classification propose by Bruzzone et al(Bruzzone, H., Lonardo, & Lucca, 2008), techniques for surface transformation can be categorized into four main groups: adding material, removing material, moving material and self-forming. The third technique is remarkable for promoting patterning surfaces through plastic and elastic deformation and by redistributing of materials. This redistribution can be accomplished by plasma-assisted treatment diffusive (Czerwiec, Marcos, Thiriet, Guo, & Belmonte, 2009) (Marcos, Guilet, Cleymand, Thiriet, & Czerwiec, 2011).

Developing new ways of patterning surfaces is one of current challenges of engineering, aiming to make its processing scale production, reducing costs and increasing productivity. Thus, the goal of nidriding of steels to produce patterning surfaces is an alternative to be studied, since it responds the requirements mentioned above. In this work, the feasibility of DC plasma nitriding in the process of surface patterning of low carbon steel SAE 1004 was studied. The effect of gas mixture also is presented below.

Experimental Procedure

Samples of SAE 1004 of 18x10x5 mm were prepared through metallography allowing the good accommodation of the mask on the sample. The masks used were copper grids used in TEM. These grids have 3 mm in diameter and holes of approximately 95 µm. All samples were placed on the anode of discharge and cathode was negatively biased at 500V.

Before nitriding, the samples were plasma sputter-cleaned in a gas mixture of 100%H_2, under a pressure of 400 Pa (3 Torr), at 400°C, for 0.5 h. This step of process aims to remove oxide layer from sample surface. Plasma nitriding was carried out during 2 hours, at 540 °C temperature, under a pressure of 400 Pa (3 Torr) in two different gas mixture compositions: 95% N_2 and 5% N_2, both balanced with hydrogen. The samples were named N95N2 for those nitrided in an atmosphere rich in nitrogen and N5N2 for those in poor atmospheres nitrogen.

The characterization were made through a scanning electron microscope (SEM, model Philips XL30), an optical microscope (OM, model Leica- MD4000M) and an optical interferometer Zygo, model New View 7300. For microstructural analysis, samples were prepared by conventional metallographic procedure. After polishing, the cross-sectioned samples were etched using Nital 2% reagent.

Results and Discussion

After nitriding process was observed the formation of a pattern of squares on the sample surface, figure 1a and figure 1b. This pattern coming from the portions unprotected and protected by mask. In fact, dimensions of the square regions are in the order of spacing between holes confirming the occurrence of a patterning due to use of masks.

It was also observed samples nitrided in nitrogen-rich atmosphere, the surface had the pattern more visible and better defined. In regions exposed to plasma, there is a change on the surface characteristics of plasma nitrided samples and the formation of precipitates where surface was protected by grid can be seen in figure 1c. These precipitates have a form of plates and needles and are present in large quantities. A possible explanation for the appearance of these plates would be an "outcrop nitrides", probably via diffusion in these locations.This phenomenon has been observed in other studies and refers to places where compound layer is very thin or nonexistent (Bendo, Pavanati, Klein, Martinelli, & Maliska, 2011). Furthermore, the morphology of nitrides shown is typical of steels. The same behavior was not found in samples N5N2.

The pattern formed on the surface can be better visualized with analysis of optical interferometry. Note that for samples N5N2, the surface was expanded and quadrangular patterns are barely visible, figure 2a. With samples N95N2 the same behavior does not happen and the pattern formed by using masks is possible to identify, figure 2b.

With a larger amount of nitrogen in gas mixture, saturation of nitrogen in the surface is higher. The high compressive residual stresses induced by introduction of large amounts of nitrogen produces a nitrided layer expansion in aperpendicular direction to the surface.This expansion was measured and the values for samples N95N2 have averaged 1.2 µm. On the other hand, for samples N5N2 the amount of expansion was only 0.4 µm (approximately).

Microstructure morphology shown in figure 3a corroborates formation of precipitates observed on surface. For conditions of nitriding in an atmosphere rich of N_2, it was observed the compound layer presents a discontinuity due to the presence of the mask, figure 3b. This absence layer, or thickness, apparently makes evident precipitates in forms of plates or needles on the surface, as previously discussed. It was not able to visualize the compound layer formed during process for poor nitrogen atmosphere. There is a possibility that it is not formed in this specific case of thermochemical treatment (low amount of N_2 and sample positioned at anode).

Conclusions

Due to the large amount of atoms that settle in the interstices of the lattice occurs an accumulation of compressive residual stresses. As a result, there is an expansion of nitrided layer from the original surface of steel, perpendicular to the surface. This expansion can be used to perform texturing surface using a mask. Due to the large amount of nitrogen present in atmosphere, samples N95N2 showed better results compared with samples N5N2. The process is viable, but a better understanding of mechanisms involved is necessary to allow better control of features shown by this recent surface texturing process.

Acknowledgments

This work was supported by CNPq, LCM and LabMat.

References

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Figures

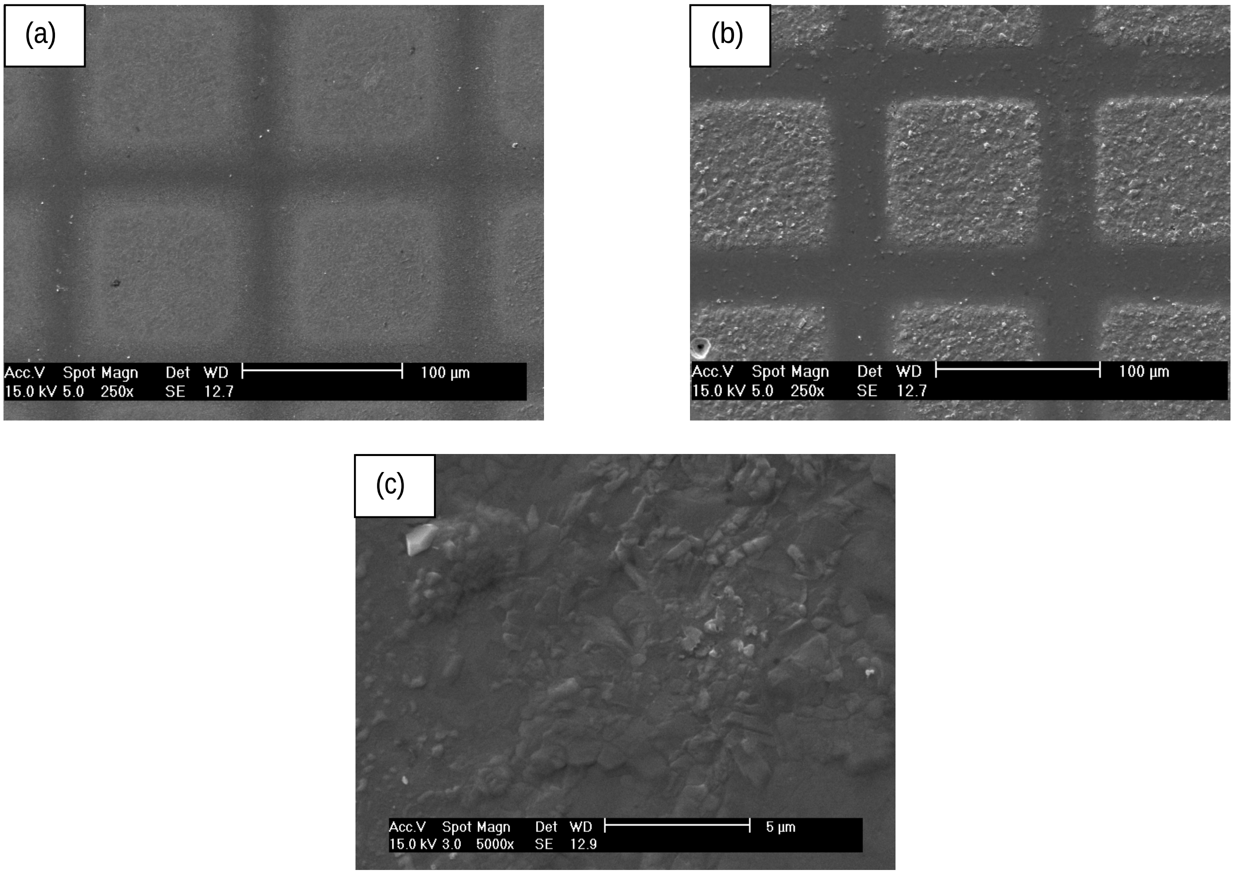

Figure 1: SEM images of patterns formed on the surface of samples (a) N5N2 and (a) N95N2 – 250x. (a) Detail of precipitates, Sample N95N2 – 5000x.

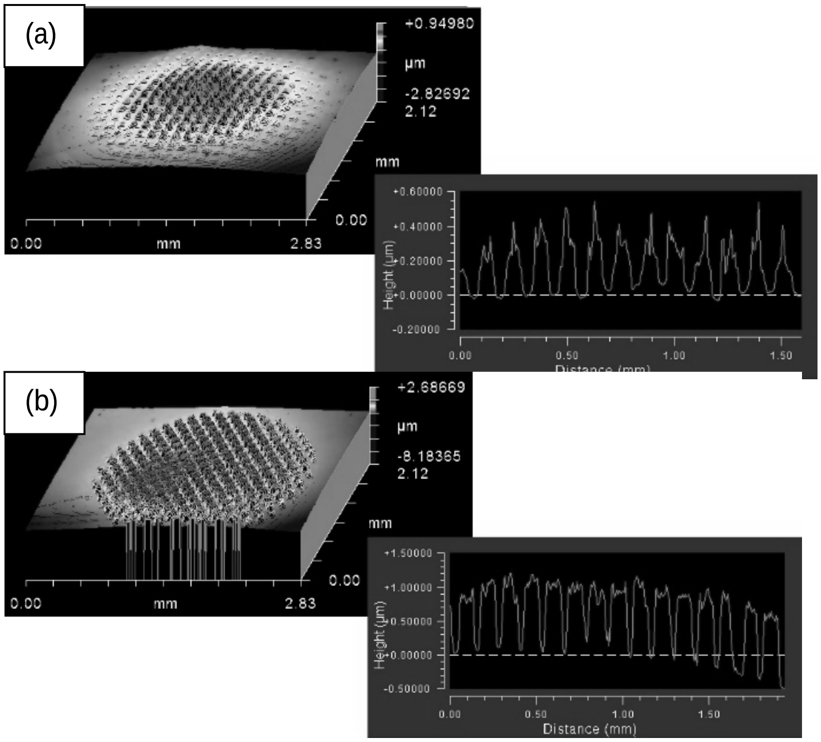

Figure 2: Topography and measures of height of a patterned surface. (a) Sample N5N2, (b) N95N2.

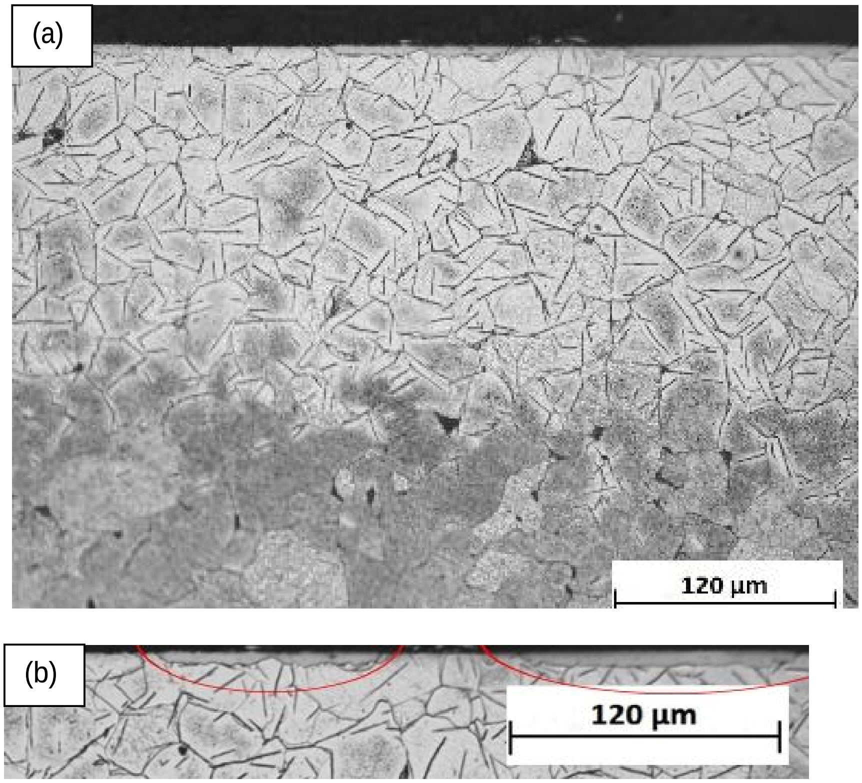

Figure 3: (a) OM image of nitrided layer of N95N2 sample, 200x. Etching with Nital 2%. (b) Detail to discontinuity of compound layer.