SELF-AFFINE PATTERNS OF BORIDE LAYERS

Morales, O. [*1], Campos, Iván .[1], Martínez J.[1], Tejeida, R.[12], Balankin, A. [1]

[1]Instituto Politécnico Nacional, SEPI-ESIME, U.P. Adolfo López Mateos, Zacatenco. México D.F. 07738. México.

[2]Instituto Politécnico Nacional, SEPI-EST, Av. Miguel Bernard #39, Frac. La Escalera. México D.F. 07630. México

ABSTRACT

In this study, the evaluation of interfaces on iron boride Fe_2B layers obtained by paste boriding process was carried out. Fractal geometry is used like a powerful tool for the roughness analysis present during iron boride growth. Experiments were performed in AISI 1045 steel at temperatures of 1193 K for exposure times of 2, 4 and 6 h, and 1223 K for treatment times of 2, 4, 5 and 6 h, varying the boron paste thicknesses in the range of 1–5 mm for each temperature and time. The fronts of the interfaces in iron boride coatings were characterized and digitized with mean of an optic microscope and Scion Image software. Self-affine methods were applied to the interface growths for validate the fractality of the system. It was established that the interface width, ω , scales to $\omega(L) \sim L^{H}$, where H represents the roughness exponent of the boride layers.

Keywords: boriding; self-affine; roughness interface; fractal geometry; boride coatings.

INTRODUCTION

In recent years, to understand the nature of disordered structures and their formation by means of random processes, fractal concepts, introduced by Mandelbrot (1982) in a theory called fractal geometry have been developed. The concepts of fractals have been applied to the natural sciences for several reasons. Self-similarity and self-affinity are the concepts that unify areas such as fractals, laws of potency, and chaos. Self-similarity is one of the fundamental symmetries that govern the universe. Likewise, self-affinity, or invariance under changes in anisotropic scale or size, is an attribute of many surfaces and interfaces found in certain natural and economic phenomena (Balankin, Morales & Campos, 2000), (Balankin, 1997). The fractal dimension is a parameter that relates structure properties and can be applied in the growth of interfaces, where its merit lies in the fact that this parameter is related to measurement of the roughness of the interface (Holten et al, 1997).

During the thermochemical treatment of paste boriding, it has been confirmed that the growth of iron boride, Fe_2B, on low alloy steel substrate presents a rough morphology between the layer/substratum interfaces (Matsschka, 1980). The characteristic of this growth is due to the tendency of Fe_2B crystals to grow following a pattern of minimal perpendicular resistance on the substrate surface. Also, it has been shown that as the thickness of the boron paste increases on the substrate surface, while maintaining treatment time and temperature constant, the iron boride layers are more compact and continuous, increasing the diffusion rate of boron in the borided layer (Campos et al, 2005). Furthermore, the mathematical models used to describe the diffusion coefficients of boron on borided phases are limited in measuring rough interfaces, which contributes to dispersion of values for the diffusion coefficient.

The present study establishes that growth fronts of the Fe_2B phase on AISI 1045 borided steels may be treated as self-affine patterns. Roughness fronts were measured by means of the Hurst exponent, *H*, using two techniques of self-affine measurement: roughness/length and range-rescaling. The influence of the experimental parameters of time, boron paste thickness, and treatment temperature on interface roughness was verified based on the Hurst exponent values.

EXPERIMENTAL DETAILS

Rectangular samples of AISI 1045 steel with dimensions of 20 x 20 mm and height of 25 mm were cut and annealed for 1 h at 873 K. The samples were placed in acrylic molds for the impregnation of boron paste with thickness of 3, 4 and 5 mm, over one side of the sample surfaces. The paste consists of B_4C with cryolite mixed with water as activator. The water/powder ratio for the paste preparation has been fixed in 0.2 (for more details see (Campos et al, 2005)). Covered samples were dried at 393K to eliminate any water residues, and borided at 1193 K for 2,4, and 6 h, also at 1223 K for 2, 4, 5 and 6 h under a pure argon atmosphere in a conventional furnace. After the treatment, each piece was quenched in oil and cross-sectioned by electrical discharge machining. The metallographic preparation employed a sequence of abrasion, until 1000-grit silicon carbide paper, followed by polishing with a diamond paste and ethylene glycol. The interfaces of the boride layers were obtained by means of Olympus GX51 optical microscopy in clear field at magnification of 500x (Fig. 1a). Then, the images were digitalized with the aid of MSQ PLUS and Scion Image softwares (Fig. 1b and Fig. 1c). The presence of boride Fe_2B formed in the borided layer at the surface of the AISI 1045 steel was determined by XRD analysis, using CoK_ radiation with _ = 1.54 A.

On other hand, roughness measurement of the boride layers was obtained using the BENOIT computational package. The program focuses on the different evaluations and methods for determining the Hurst exponent of self-affine patterns.

RESULTS AND DISCUSSIONS

The presence of the Fe_2B monophase layer at the surface of the AISI 1045 steel was presented in the X-Ray diffraction pattern (Fig. 2). In the borided layers, in zones corresponding to the boundaries, it is normally possible to find mixed crystals of different phases. Crystals of the Fe_2B type orientate themselves with the z-axis perpendicular to this surface. Consequently, the peaks of the Fe_2B type phases corresponding to crystallographic planes, with deviation from zero of the *l* index, show increased intensities in the X-ray diffraction spectra (Badini & Mazza, 1998).

The growth fronts of the Fe_2B layer were characterized by two methods of self-affine fractals. The Hurst exponent for the self-affine methods allows us to determine whether a phenomenon presents fractal behavior, by measuring the intensity of long-term dependency of a series of data at a width *L* of the image digitalized by ScionImage. Accuracy in determining *H* for the two self-affine methods depends on the number of data used in the calculation. If the number is reasonably large, the curves for each method can be expected to provide information on self-affinity in all intervals of the image width. All microphotographs of the diffusion fronts have a resolution of 2048 X 1536 pixels. Also, 20 photographs were taken in different sections of each borided sample for the full set of experiments to guarantee the reliability of the data for the roughness exponent.

The roughness/length method accounts for the standard deviation, or roughness of the root-mean-square (RMS) of the data in size windows w, instead of vertical range. For a self-affine line, roughness SD (where SD is standard deviation) measured in a size window w is related to the Hurst exponent as follows:

$$SD\,\alpha\,L^{H}\,. \qquad (1)$$

In the case of the rescaled range method, for a series of data for width n, R/σ is defined as the quotient of the maximum standardized range of the integrated signal $R(n)$ over the standard deviation $S(n)$:

$$S(n)=\left[\frac{1}{n}\sum_{t=1}^{n}\left(X_t-\frac{1}{n}\sum_{t=1}^{n}X_t\right)^2\right]^{1/2} \qquad (2)$$

If the pattern is self-affine, the expected value of R/σ has an scale L^{H} when $L\rightarrow\infty$:

$$R/\sigma\,\alpha\,L^{H}\,. \qquad (3)$$

Table I shows the values of the Hurst exponent obtained for the set of experiments at temperatures of 1193 and 1223 K using the two self-affine methods.

Variations in the roughness values obtained with each method are to be expected due to statistical errors associated with each analytic approach. Fig. 3 shows the functions of SD and $R/$_ for diffusion fronts at a temperature of 1223 K, with 4 h of treatment and a 5 mm thickness of boron paste. In both methods, the value of H is not sensitive to the minimum, maximum, or average values of the heights of the chosen diffusion fronts.

The growth process of an interface typically starts with a soft front that increment its roughness over time. Experimental observations (Meakin,1988) have revealed that in an initial stage interface height grows in accordance with a time power law $h\,\alpha\,t^{\beta}$, where _ is the growth exponent. However, after a time the interface becomes saturated and the overall average of spatial fluctuations obeys an escalating performance $h(L,t\rangle t_s)\alpha\,L^{\alpha}$, where L is the lateral size of the system and _ is the overall roughness exponent. Saturation time, t_s, increases as the lateral size of the interface increases, as $t_s\,\alpha\,L^{H}$, where H is the statistically self-affine dynamic exponent.

To determine the fractality of growth interfaces it needs to be shown that the interface variation values across the rough profile present a normally distributed behavior (Riesel, & Heimann, 2004). Fig. 4 shows Gaussian distributions for temperature of 1193 K, 6 h of treatment and boron paste thicknesses of 4 and 5 mm respectively, confirming the self-affine behavior of the interfaces.

The experimental parameters of treatment time and boron paste thickness for each boriding process temperature are compared with the Hurst exponent values obtained with the rescaled range method. For both temperatures, the Hurst exponent did not vary with the different treatment times or boron paste thicknesses used in the process, which indicates that there is no functional dependency between these parameters (Fig. 5). This proves that the growth morphology of the iron boride Fe_2B layer does not depend on experimental process conditions, and that the value of *H* may be universal in rough fronts of iron boride over an AISI 1045 steel substrate.

CONCLUSIONS

The quantitative characterization of the morphology of growth fronts of the iron boride Fe_2B layer is an important step toward a better understanding of the physical mechanisms of boride layer growth during the boriding process. The fact that interface growth in non-equilibrium systems presents complex scaling properties tell us nothing about the reasons why that is so. Therefore, a crucial point to understand this phenomenon is the origin of the general scale invariance of interface roughness. On the other hand, the rescaled range and roughness/length measurement methods have shown that the growth of an iron boride layer on AISI 1045 steel presents a self-affine behavior, where the Hurst exponent does not depend on the experimental parameters of the process.

It is feasible that the roughness parameter essentially depends of the substrate where iron boride grows. The Hurst exponent is also an indicator to determine whether a phenomenon presents a fractal behavior and measures the intensity of long-term dependency of a series of data.

The experimental results show that the morphology associated with iron boride Fe_2B growth fronts during paste boriding process can be described in terms of self-affine fractal geometry and introduce an alternative to model the growth of these layers by means of fractal equations.

ACKNOWLEDGMENTS

This work has been supported by the research grants 20070130 20070131, 20051570 and 20071284 of Instituto Politécnico Nacional at Mexico.

REFERENCES

Badini, C.& Mazza, D. (1988). *Mat. Sci*. Lett. 23, 3061-3066.
Balankin, A. (1997): *Eng. Fracture Mech*. 57 135-203.
Balankin, A., Morales, D.& Campos, I. (2000). *Phil. Mag. Lett.* 80 165-172.
Campos, I. O.et al. (2005): *Appl. Surf. Sci.* 243 429-436.
Holten, T. et al. (1994). *Phys. Rev*. E 50 754.
Mandelbrot, B. (1982). *The Fractal Geometry of Nature*, first ed., W.H. Freeman,
Matuschka, A. (1980). *Boronizing*. first ed., Carl Hanser Verlag, Munich.
Meakin, P. (1998): *Fractals, scaling and growth far from equilibrium*, Cambridge Nueva York.
Riesel, G.& Heimann, R. (2004). *Surf. Coat. Technol.* 185 215-221.
University Press, United Kingdom.

Table I. The Hurst exponent (H) measured from the two self-affine methods at two different temperatures of boriding process.

Measure Hurst exponent (H)	**1193 K**	***1223 K***
SD	0.85 ± 0.002	*0.89 ± 0.002*
R/_	*0.80 ± 0.008*	*0.80 ± 0.008*

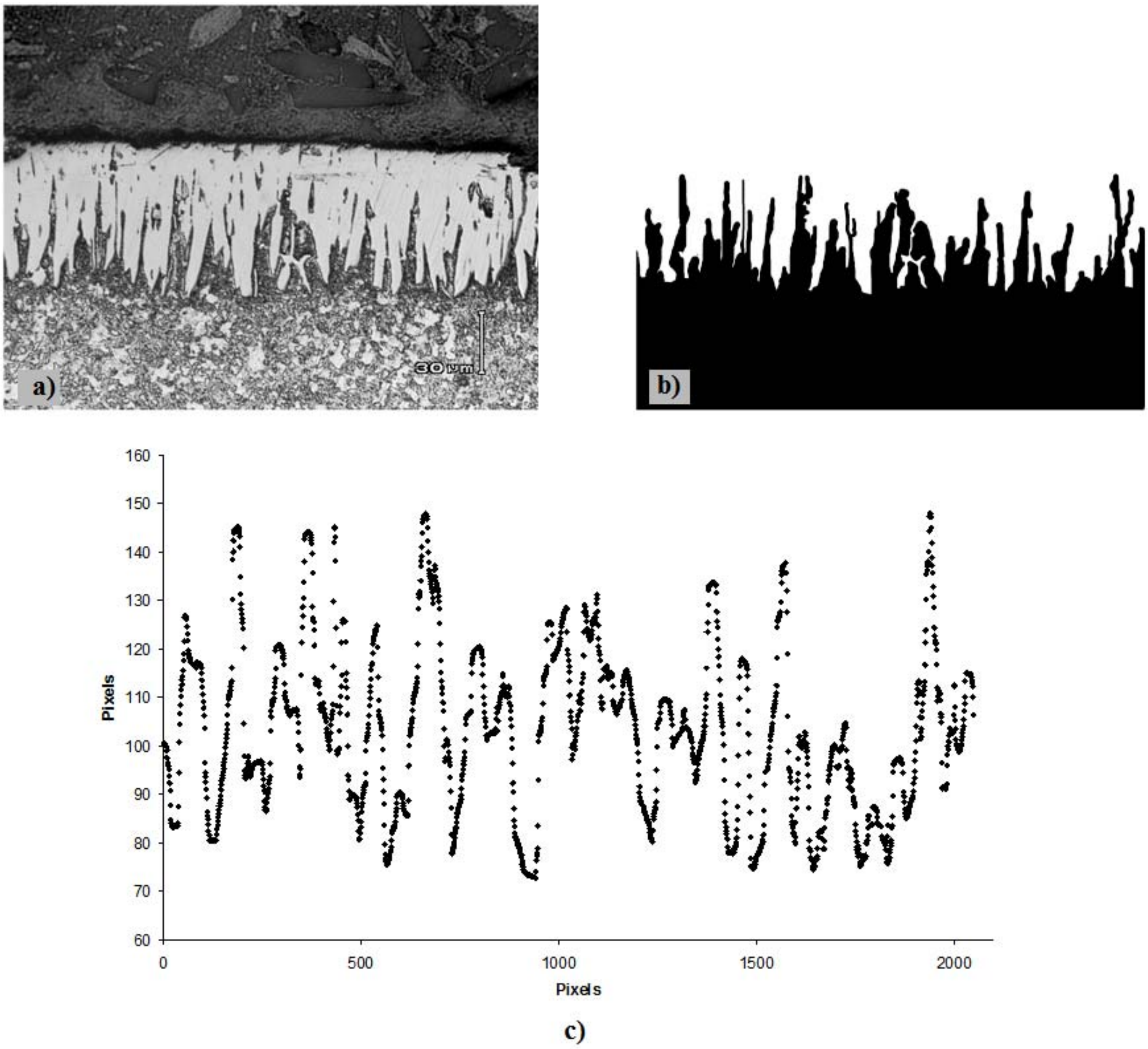


Figure 1. a) Cross sectional view of the borided sample at 1223 K, with exposure time of 4h and boron paste thickness of 5mm, b) the same image digitalized in binary colours c) image interpretation with aid of Scion Image Software.

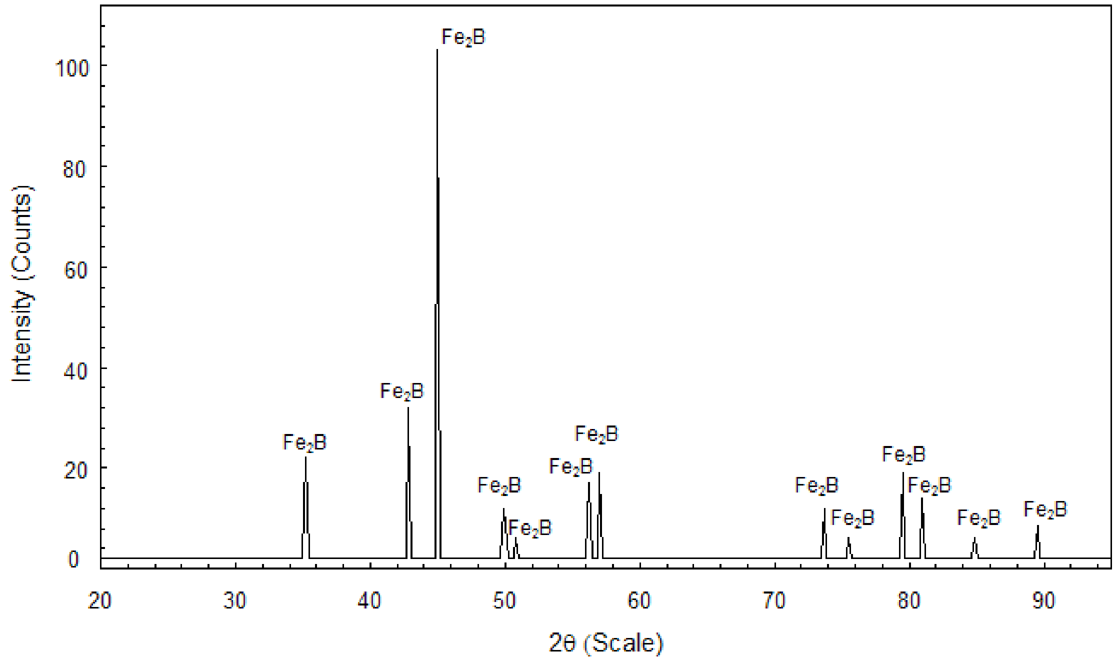
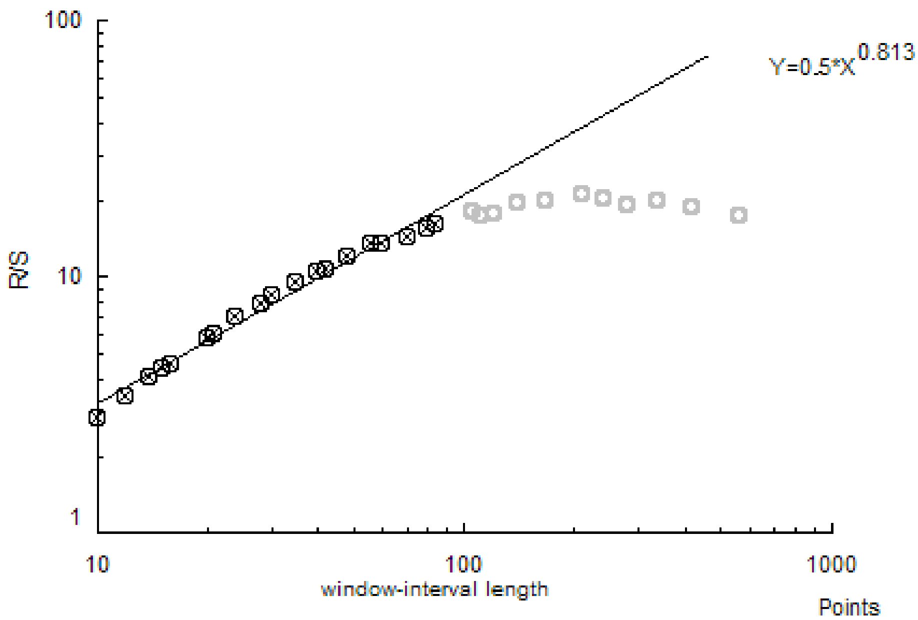
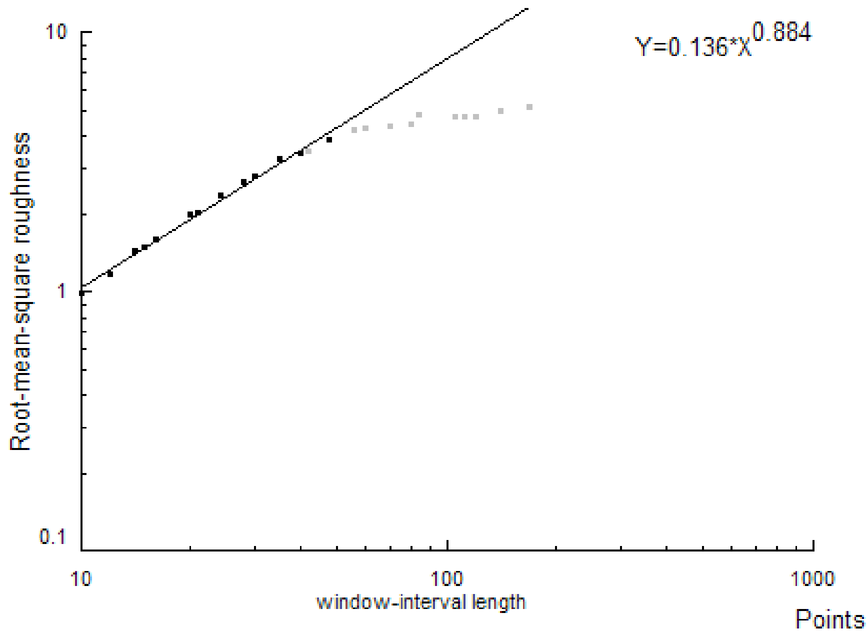


Figure 2. XRD pattern for AISI 1045 steel borided at 1223 K with 6 h of treatment and 5 mm of boron paste thickness.



a)



b)

Figure 3. Determination of Hurst exponent in borided layers applying the methods: **a)** rescaled range, **b)** roughness/length.

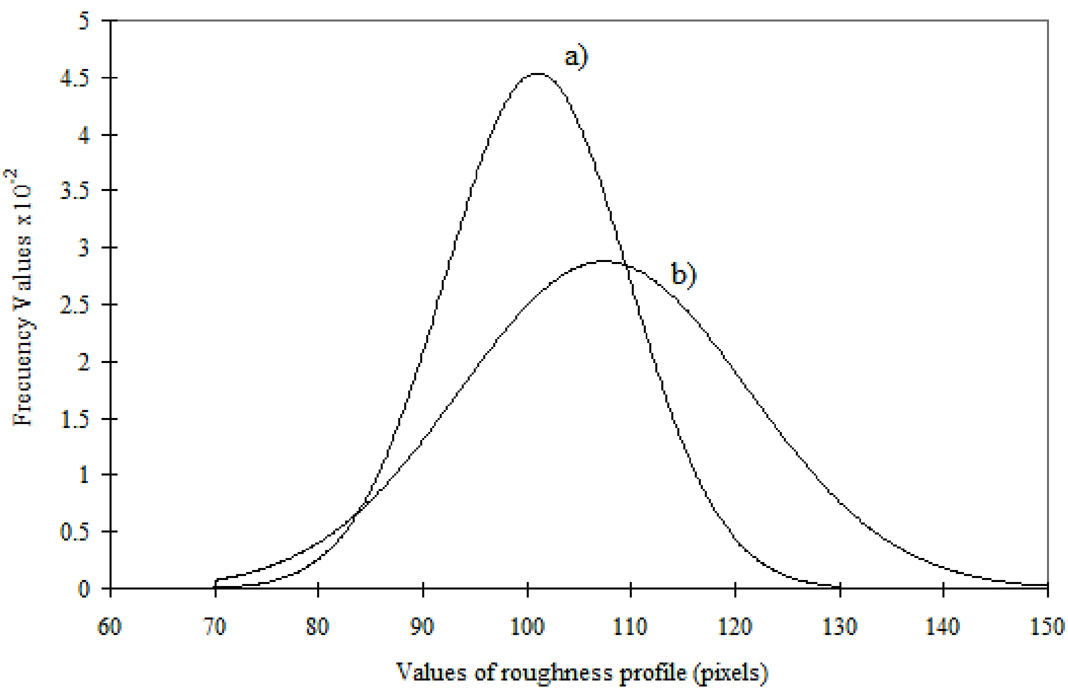
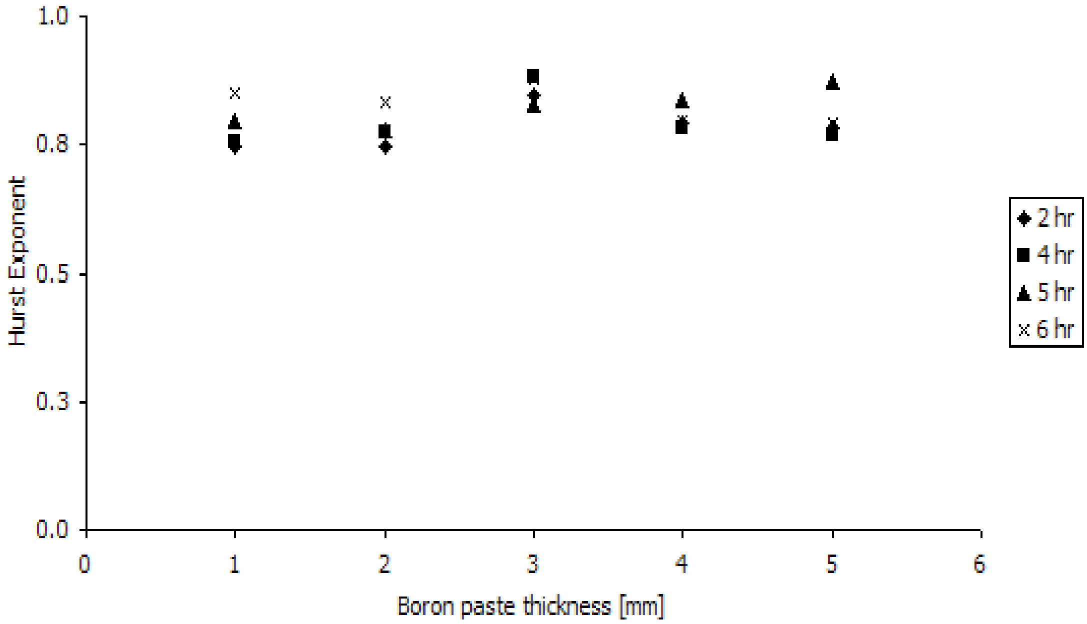
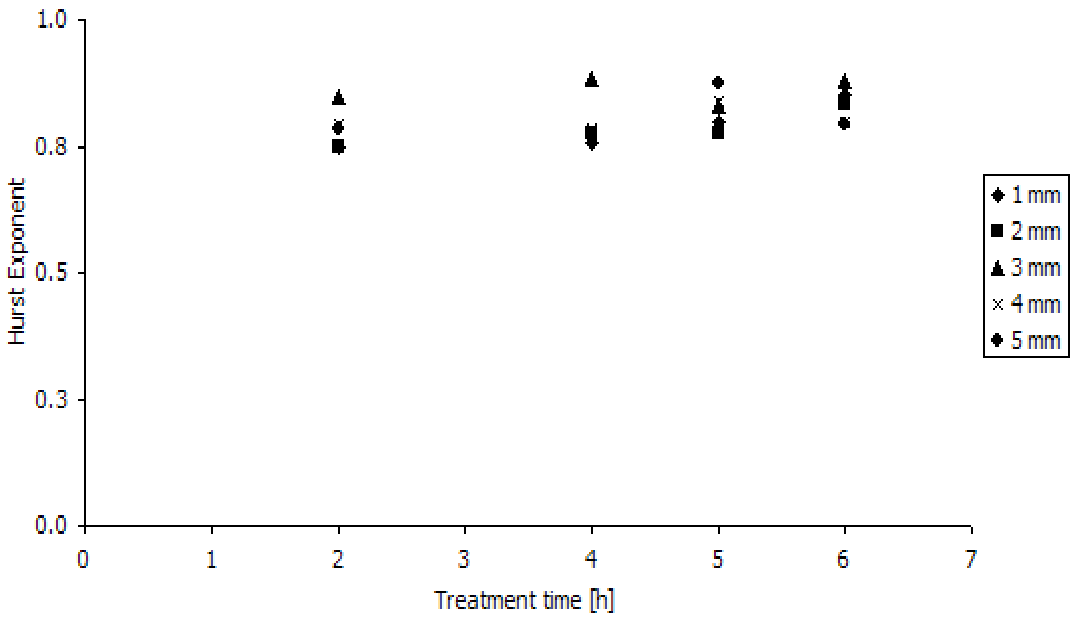


Figure 4. Gaussian distribution of the interfaces roughness profiles in the borided samples at temperature of 1193 K and 6 h of treatment time, with boron paste thickness of: **a)** 4 mm, **b)** 5mm.



a)



b)

Figure 5. Influence of Hurst exponent in the experimental parameters of **a)** boron paste thicknesses, and **b)** treatment time. Boriding temperature of 1223 K.