



## Indentation size effect on the Fe<sub>2</sub>B/substrate interface

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### ABSTRACT

This study evaluated the indentation size effect on the Fe<sub>2</sub>B/substrate interface using the Berkovich nanoindentation technique. First, the Fe<sub>2</sub>B layers were obtained at the surface of AISI 1018 borided steels by the powder-pack boriding method. The treatment was conducted at temperatures of 1193, 1243 and 1273 K for 4, 6 and 8 h at each temperature. The boriding of AISI 1018 steel resulted in the formation of saw-toothed Fe<sub>2</sub>B surface layers. The formation of a jagged boride coating interface can be attributed to the enhanced growth at the tips of the coating fingers, due to locally high stress fields and lattice distortions. Thus, the mechanical properties achieved at the tips of the boride layer are of great importance in the behavior of borided steel.

Applied loads in the range of 10 to 500 mN were employed to characterize the hardness in the tips of the Fe<sub>2</sub>B/substrate interface for the different conditions of the boriding process. The results showed that the measured hardness depended critically on the applied load, which indicated the influence of the indentation size effect (ISE). The load-dependence of the hardness was analyzed with the classical power-law approach and the elastic recovery model. The true hardness in the tips of the Fe<sub>2</sub>B/substrate interface was obtained and compared with the boriding parameters. Finally, the nanoindentation technique was used to estimate the state of residual stresses in this critical zone of the Fe<sub>2</sub>B/substrate interface.

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### 1. Introduction

Boriding is a thermochemical surface-hardening process in which boron diffuses into a well-cleaned base metal (steel) surface at a high temperature. The boriding process takes place at temperatures between 1123 and 1273 K over a period of 1 to 10 h. The resulting metallic boride has improved hardness, high wear resistance, high temperature resistance and high corrosion resistance [1]. Boriding steel alloys form either a single or double-phase layer with definite compositions. The outer layer is FeB (with an orthorhombic crystal structure), with a boron content of approximately 16.4 wt.%B, while the inner layer (Fe<sub>2</sub>B) has a tetragonal crystal structure with a boron content of approximately 9 wt.%B [2]. In borided low-carbon steels, the morphology of the boride layers at the surface of the material displays a saw-toothed shape that reflects the anisotropic nature of the layers. The formation of a jagged boride coating interface can be

attributed to the enhanced growth at the tips of the coating fingers, due to locally high stress fields and lattice distortions [3] and this growth is expected to enhance the adhesion to the substrate steel [4]. Therefore, the evaluation of the mechanical properties at the tips of the coating/substrate interface is of great importance in the application of borided steel.

The indentation test has been used extensively to measure the mechanical properties of materials. Nanoindentation combines the process of recording an indentation at a high resolution and analyzing the accompanying data to determine the mechanical properties directly from the load-displacement data without imaging the indentation. In recent years, the depth-sensing indentation technique has been employed to estimate the mechanical properties of FeB layers produced on the surface of borided low-carbon steels [5,6]. The results obtained with the Berkovich nanoindenter showed the load-dependence of the Young's modulus, yield strength, and indentation depth on FeB layers over a constant boriding time.

Typically, the measured hardness is very high when a very low test load is applied to a ceramic material, while the measured hardness decreases as the load increases [7]. This phenomenon, known as the indentation size effect (ISE), depends on the size of the indentation that results from an applied load. Unfortunately, the existence of ISE

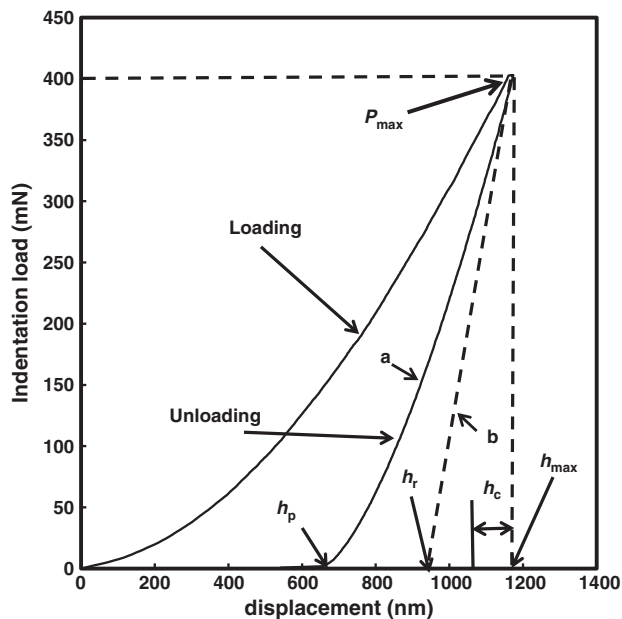
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**List of symbols**

$P_{\max}$	indentation peak load
$A_c$	projected area of the hardness impression
$H$	hardness
$h_c$	contact depth of the indenter with the sample at $P_{\max}$
$h_p$	permanent indentation depth after removal of the test force
$h_r$	point of the tangent b to curve a at $P_{\max}$ with the indentation depth axis
$h_{\max}$	maximum indentation depth at $P_{\max}$
$A$	Meyer's constant
$n$	Meyer's index
$A^*$	standard hardness constant
$H_o$	true hardness
$h_o$	correction factor in the indentation size $h_c$ due to the elastic recovery
$k$	constant dependent on the indenter geometry
$c$	crack length emanated at the corner of the indentation mark
$K_{IC}$	fracture toughness
$\sigma$	residual stress
$E$	Young's modulus
$a$	diagonal-length mark

has not been considered in the measured hardness of boride layers. Therefore, the hardness value is clearly insufficient to be used as a selection criterion for a particular application.

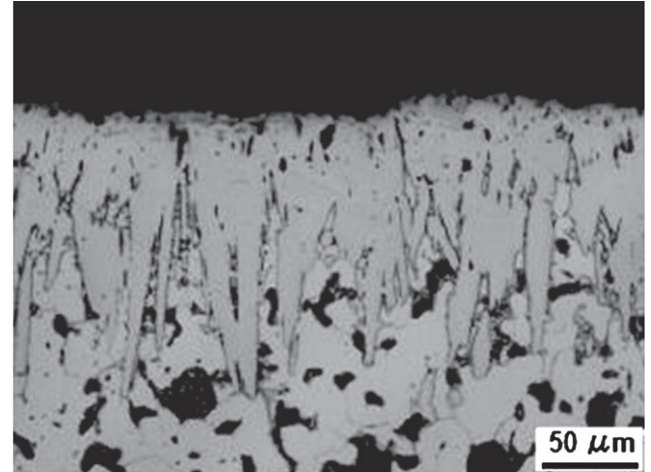
The purpose of the present work was to evaluate the nanoindentation hardness at the tips of the jagged boride layer obtained in AISI 1018 borided steel exposed to different boriding temperatures and treatment times. The measured hardness in this zone of the boride layer exhibited a peak load-dependence in the range of 10 to 500 mN, and the results were expressed by two classical approaches to



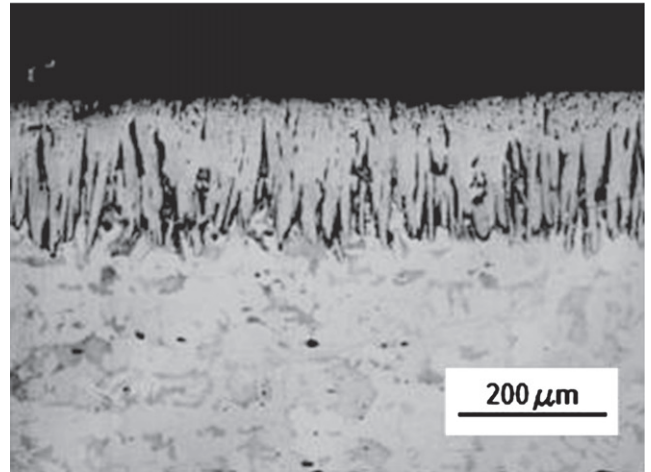
**Fig. 1.** Schematic representation of a typical load versus indenter displacement data for an indentation experiment.

describe the ISE. In addition, the residual stresses and fracture toughness at the tips of the  $Fe_2B$ /substrate interface were estimated using the length of the cracks that originated from the corners of the Berkovich nanoindentations site as a result of the different applied loads.

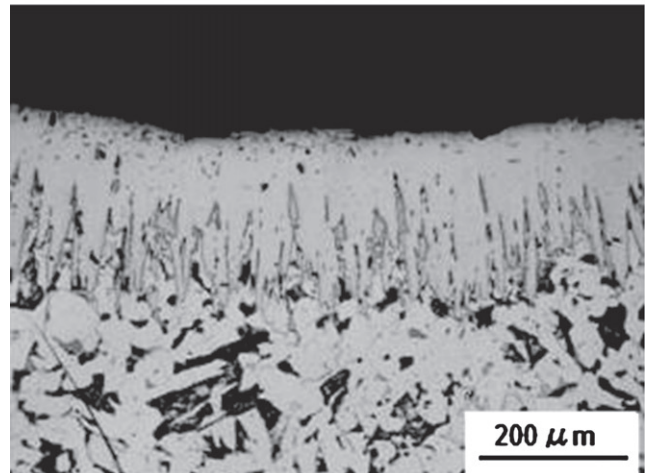
(a)



(b)



(c)



**Fig. 2.** Boriding of AISI 1018 steels at temperatures of: (a) 1193 K (50 $\times$ ), (b) 1243 K (20 $\times$ ), (c) 1273 K (20 $\times$ ) with 6 h of exposure.

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