Combining Nanoindentation with Atomic Force Microscopy to Characterize the Mechanical Properties of each Microconstituent of Low Carbon Steel

Article *in* Microscopy and Microanalysis · December 2005

DOI: 10.1017/S1431927605050981

CITATION
1

READS
29

4 authors, including:

David J. Shuman
Fundação Centro Tecnológico de Minas Gerais
9 PUBLICATIONS **107** CITATIONS
SEE PROFILE

Margareth Spangler Andrade
144 PUBLICATIONS **1,354** CITATIONS
SEE PROFILE

André Cota
Universidade Federal de Ouro Preto
52 PUBLICATIONS **437** CITATIONS
SEE PROFILE

Microsc Microanal 11 (supp 3), 2005
doi:10.1017/S1431927605050981

Combining Nanoindentation with Atomic Force Microscopy to Characterize the Mechanical Properties of each Microconstituent of Low Carbon Steel

D. J. Shuman*, F. L. G. Oliveira**, M. S. Andrade*, and A. B. Cota**

*Fundação Centro Tecnológico de Minas Gerais - CETEC, Av. José Cândido da Silveira, 2000, Belo Horizonte, MG, 31170-000, Brasil. e-mail: david.shuman@cetec.br
**REDEMAT, Universidade Federal de Ouro Preto, Rua Diogo de Vasconcelos, 122, Ouro Preto, MG, 35400-000, Brasil.

Keywords: atomic force microscopy, instrumented indentation testing, depth-sensing indentation, nanoindentation, microindentation, hardness, elastic modulus, low carbon steel, heat-treatment

In the last decades several investigations have been conducted with the objective of studying the microstructural characteristics and mechanical properties in low carbon microalloyed steel [1,2]. These steels contain a polyphase microstructure, with complex mixture of bainite, MA constituent (martensite-austenite residual), pearlite, ferrite and martensite. They are often used in the automobile industry, in the production pipelines for the transport of gas and oil in areas of sub-zero temperature and in the naval ship construction, because they possess high strength, high toughness at low temperature and good weldability.

It is beneficial to measure the mechanical properties of each microconstituent of multi-phase materials to have control over the final bulk mechanical properties. Sometimes various microconstituents will have the same image appearance but greatly different mechanical properties and hardness. Instrumented indentation testing (IIT) coupled with atomic force microscopy (AFM) can be used in to characterize the mechanical properties of materials. When the residual indents are less than a few micrometers across an optical microscope does not provide enough detail to determine its exact location and shape. An AFM was therefore useful to image the residual indents inside the various microconstituents. The elastic modulus and hardness at the indents was calculated using the Oliver-Pharr method [3]. This work was then applied to characterize the mechanical properties of different phases present in a low carbon steel submitted to different intercritical heat-treatment performed by dilatometric tests.

A Shimadzu™ ultra-micro dynamic hardness tester (DUH-W201S) was used to make the indents with a standard Berkovich indenter. This IIT records the displacement and force during loading and unloading (Figure 1). Many indentation tests were done using a maximum force of 5.0 mN, 0.47 mN/s approach rate, and a 5 s hold time. From these load-unload curves the Martens hardness (Equation 1) and elastic modulus (Equation 2) were calculated.

$$HM = \frac{P_{\max}}{26.44(h_{\max})^2} \qquad (1)$$

The Martens hardness (HM) is the maximum load, P_{max} ,divided by the contact area at the maximum depth h_{max}.

$$E = \frac{(1-v^2)}{\frac{2\sqrt{24.56 \cdot h_c^2}}{a \cdot m(h_{\max} - h_f)^{m-1} \cdot \sqrt{\pi}} - \frac{(1-v_i^2)}{E_i}} \qquad (2)$$

The v is the Poisson's ratio for the steel sample (0.3), h_c is the contact depth, v_i and E_i are the Poisson's ratio and elastic modulus of the diamond indenter. The unload slope coefficients a, m, and final depth h_f were found by doing a non-linear power law fit to the unloading curve.

The chemical composition of the steel under investigation, expressed in wt%, is 0.15C, 1.42Mn, 0.37Si, 0.052Al, 0.031Nb, 0.023P, 0.009S and 0.0042N. The samples were austenitized at 840 ^{o}C, 860 ^{o}C and 900 ^{o}C, using a constant rate, in an Adamel Lhomargy (LK2) dilatometer, and immediately quenched after reaching these temperatures, with the aim to obtain complex microstructures. The samples were prepared by mechanical polishing. The indents were imaged with a Digital Instrument Dimension 3100 AFM using height Tapping Mode.

Figure 2 shows the 840^{o}C heat-treated sample using both an optical microscope and an AFM. All of the indents were placed inside the ferrite region and along the grain boundaries. The lowest hardness was measured at the grain boundary HM 1866 MPa but the elastic modulus at this point was 269 GPa approximately equal to the other two indents in this grain 238 GPa and 268 GPa. The adjacent grain had a lower elastic modulus from 152 GPa in the center to 248 GPa near the grain boundary.

Figure 3 presents results of the same sample material austenitized at 860 ^{o}C and at 900 ^{o}C. These images show a much more complex microstructure than the 840 ^{o}C process. The 860 ^{o}C heat-treatment developed large regions of distinctly different elastic modulus and hardness. The 900 ^{o}C microstructures were finer with an elastic modulus range from 138 GPa to 308 GPa and hardness from 1683 to 12290 MPa in a complex mixture of martensite, bainite, small islands of residual austenite and a small amount of polygonal ferrite constituents.

Figure 4 shows the frequency distribution histogram of the Martens hardness and elastic modulus for the three heat-treated samples. The 840 ^{o}C sample hardness concentrated around 3600 MPa whereas the 860 ^{o}C and 900 ^{o}C plotted together showed a wider distribution of hardness. The elastic modulus for each sample ranged between 138 GPa to 300 GPa. One of the 900 ^{o}C indentations showed elastic modulus results above 300 GPa.

It was concluded that instrumented indentation testing together with atomic force microscopy was a useful method to calculate the elastic modulus and Martens hardness of small microconstituents in complex microstructures. The mechanical properties of the microconstituents were correlated through AFM imaging. The limit of this technique depends on the quality of the unloading curve data. Further research should be done using lower indentation forces to produce nanoindentations in an effort to characterize even smaller microconstituents. [4]

References

[1] Q. Furnémont, et al., Materials Science and Engineering A328 (2002) 26–32.
[2] R. Rodríguez and I. Gutierrez, Materials Science and Engineering A361 (2003) 377–384.
[3] W.C. Oliver and G.M. Pharr, J. Mater. Res., Vol. 7. No. 6, June (1992) 1564-1582.
[4] This work was supported by a grant from CNPq and RHAE/CNPq, Brazil.

doi:10.1017/S1431927605050981
Microsc Microanal 11 (supp 3), 2005

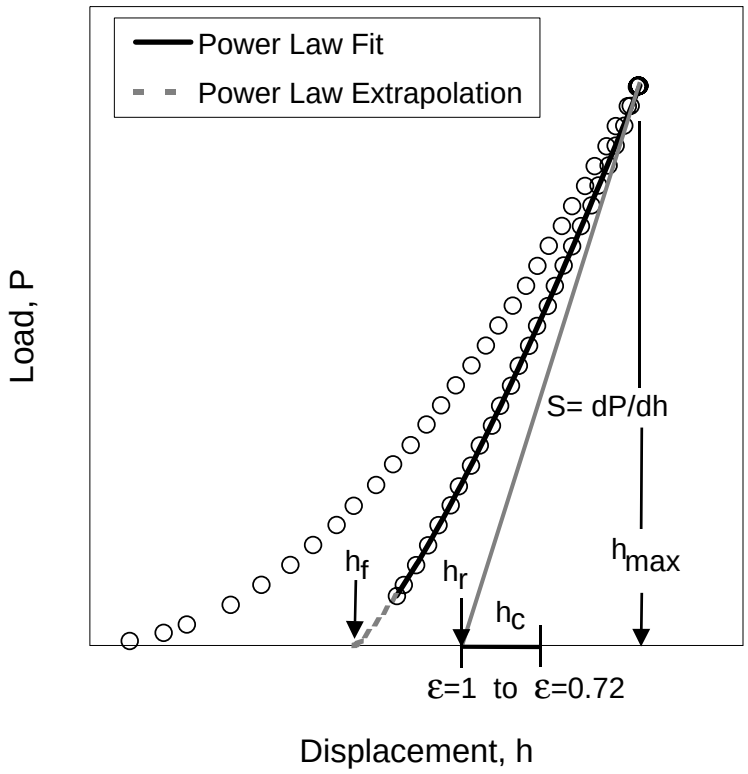

Fig. 1. Schematic of the typical instrumented indentation test load-unload curve.

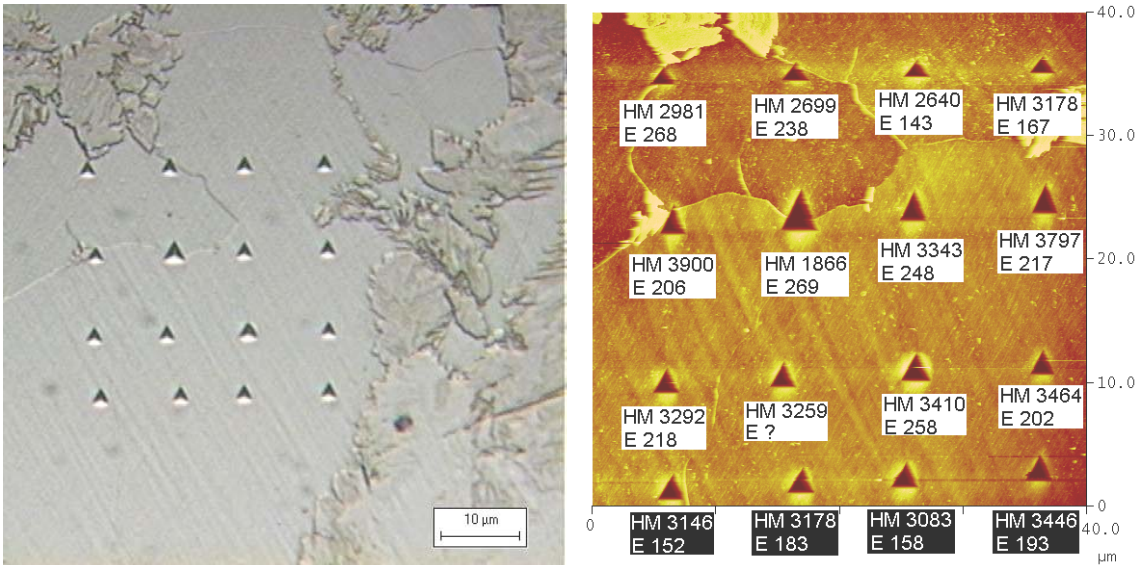

Fig. 2. Optical and AFM height image of the same indentations for the 840°C heat-treated sample. The indentation Martens hardness (HM) (MPa) and elastic modulus (E) (GPa) are indicated.

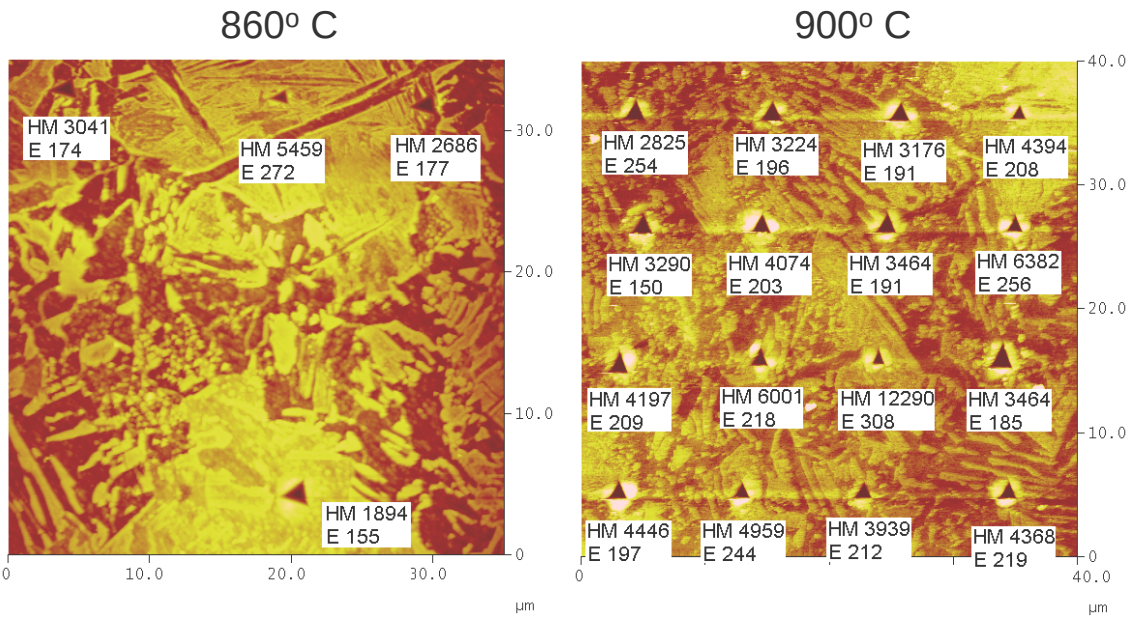

Fig. 3. AFM height image of the 860°C and 900°C austenitized samples. The indentation Martens hardness *HM* (MPa) and elastic modulus *E* (GPa) are indicated.

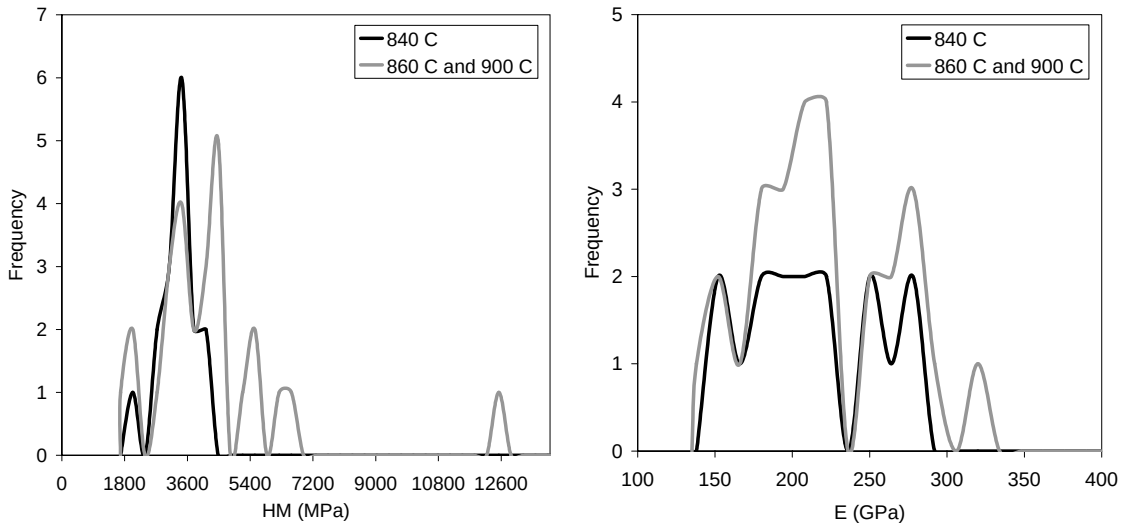

Fig. 4. Frequency distribution of hardness (left) and elastic modulus (right) for the 840°C, 860°C and 900°C samples.