Evaluation of mechanical properties by instrumented indentation of Duplex treated steels

R. M. Buzdugan [a, b*], **I. Mitelea** [a]

[a] Politehnica University of Timişoara, Materials and Manufacturing Engineering Department, Timişoara, Romania
[b] National R&D Institute for Welding and Material Testing - ISIM Timisoara, Romania

*) *E-mail: mdobra@gmail.com*

Keywords

Carburizing, Duplex treatments, bulk modulus, hardness

1. Introduction

Nowadays, many mechanical components demand a combination of dimensional stability, superior mechanical strength, as well as moderate wear and good corrosion resistance [1]. Therefore, designers are continually stretching the limits of existing materials. To extend the use of current materials, beyond their conventional limits, manufacturers are exploring the concept of surface treatment, such as gas carburizing, plasma nitriding [2, 3], deposition of hard, protective ceramic coatings [4], etc.

But, in an increasing number of cases, the resistance of metals against complex loads is no longer high enough. Combining single processes into one treatment can result in an even higher resistance of metals against complex loads, such as superimposed wear, fatigue and corrosion, due to the addition of the single process advantages. Processes combined in this way, known as duplex or hybrid processes, have a high potential for the future treatment of metals. The number of possible combined processes is very large. An important aspect of the Duplex treatments is the substantial choice of technical and economical promising combinations [5].

Carburizing is a remarkable method of enhancing the surface properties of shafts, gears, high stressed machine parts, in order to obtain very high fatigue and wear resistance. Tempered martensite is the dominant microstructure constituent of properly carburized steel. However, the martensite changes in morphology, amount, and properties as a function of distance from the surface. Other micro-structural constituents, such as retained austenite, massive carbides, prior austenite, grain boundaries carbides, and surface oxides may also be present and significantly affect the performance of carburized parts [6].

The carburizing treatment does not ensure a radical change of the treated layer properties. Therefore, it is necessary to realize a hardening and tempering treatment in order to obtain the desired properties of both the core and the surface layer. Induction hardening allows the selective hardening of a part, in order to achieve desired hardness over a specific area and depth. Because the part is selectively heated, the heat-affected zone can be adjusted to minimize distortion and other problems. Surface hardening with induction creates parts that have excellent resistance to fatigue. A hard outer case is created over a ductile core, with high compressive forces at the surface. These compressive forces at the surface improve fatigue properties by delaying crack initiation and propagation during service [3].

The aim of this study is to determine the mechanical properties of Duplex treated EN 16MnCr5, consisting in carburizing followed by surface induction quenching and low tempering using instrumented indentation tests. The micro-indentation technique [7] has been developed in some decades, the mechanical properties within a sub-micron or nanoscale being widely discussed. The techniques are expected to be useful for the measurement of mechanical properties of thin films or local structure of various materials. The instrumented indentation technique can directly determine the hardness, bulk modulus and the deformation characteristics [8]. From the loading and unloading curve, the nature of elastic–plastic transition can be analyzed.

2. Experimental procedures

The Duplex treatment consisting in gas carburizing followed by surface induction quenching was carried out on EN 16MnCr5 cylindrical samples. This steel is widely used in industry, especially in automotive industry. Also, it was selected for these experiments due to its high mechanical and hardenability properties and lower cost, compared to other carburizing steels.

The first step in realizing the Duplex treatment consisted in a gas carburizing, without effectuate a further martensite quenching. The carburizing temperature was around 920 ± 10 °C. After the carburizing treatment, that lasted 8 hours, the samples were air cooled. The thickness of the carburized layer was about 0.6 mm.

The second step of the Duplex treatment consisted in surface induction hardening the thermochemically treated specimens by means of high frequency induction heating. To obtain optimal results, the induction coil has to be adjusted to the samples geometry. Therefore, a special induction coil has been made. The distance between the samples and the induction coil was around 10 mm. The parameters of the treatment are: the specific power, ΔP = 0,9 kW/cm^2, the heating time t = 4 s and the frequency f = 32 kHz. Using these parameters, a layer thickness of 0.8 - 1 mm was obtained. The cooling was realized in water. To reduce the internal stress appeared as a result of the surface induction hardening, the samples were subjected to a low tempering treatment, at 180 °C for 90 minutes.

The mechanical properties, such as hardness and bulk modulus, were determined by instrumented indentation tests. The indentation tests consist of performing a print on the surface of a material by the penetration of an indenter at a specified load. The mechanical properties mentioned before were determined by analysing the load-depth curve [7].

Before micro indentation testing can be performed, it is necessary to create a high-quality surface in order to ensure both accuracy and repeatability of the tests. The samples have been metallographically mounted, grinded and polished.

To get more accurate values twelve measurements, with the applied load ranging from 100 to 1000 mN, were performed on a Dynamic Ultra Micro Hardness tester, equipped with a diamond Berkovich indenter tip. The indentation prints are presented in Figure 1.

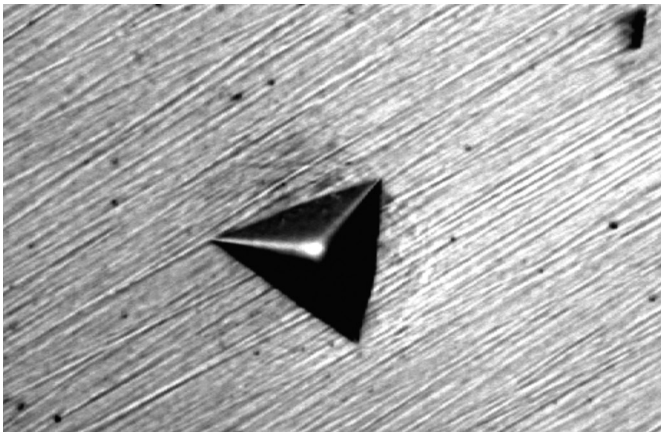


Figure 1. The micro indentation print.

The four key parameters needed to determine the hardness and the bulk modulus from the load-depth curves are the maximum load, P_{max}, the maximum depth, h_{max}, the contact depth, hc and the elastic unloading stiffness, also called the contact stiffness S = dP/dh, defined as the slope of the upper portion of the unloading curve during the initial stages of unloading.

The accuracy of hardness and bulk modulus measurement depends on how well these parameters can be measured experimentally [16].

3. Results and discussions

From instrumented indentation test, which allows the plot of a load-depth curve, Figure 2, the calculation of the bulk modulus can be done by Oliver and Pharr method. They proposed that the bulk modulus can be calculated from the total compliance of the specimen and of the instrument, which results from the contribution to the depth measurement deflections of the load frame, added to the displacement into the material [7-8].

The bulk modulus can be calculated from the total compliance Ct of the sample and of the instrument [8], figure 2.

When a load is applied, the reaction force is taken up by deflection of the load frame and added to the depth registration. The corrections are made using the formula:

$$h = h_m - C_f \cdot P \quad (1)$$

Where h is the indentation depth into material, h_m is the measured indentation depth, C_f is the instrument compliance and P is the load applied [7-8].

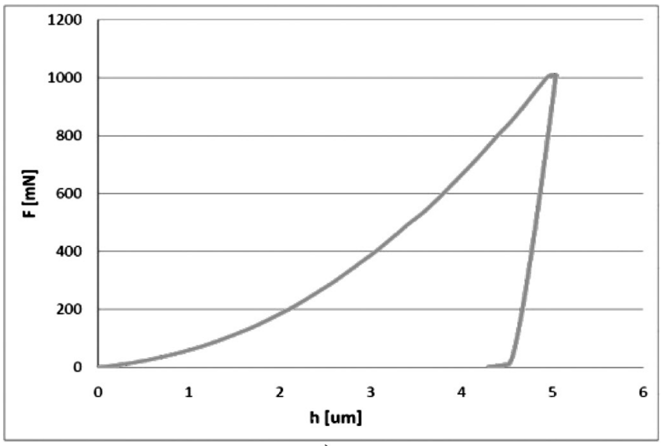
The contact stiffness is very important, since it is required to calibrate the indentation depth prior to the calculations. The contact stiffness is given by:

$$\frac{1}{S} = C_f + \sqrt{\frac{\pi}{24{,}5}} \cdot \frac{1}{2 \cdot \beta \cdot \gamma \cdot E_R} \cdot \frac{1}{h_c} \quad (2)$$

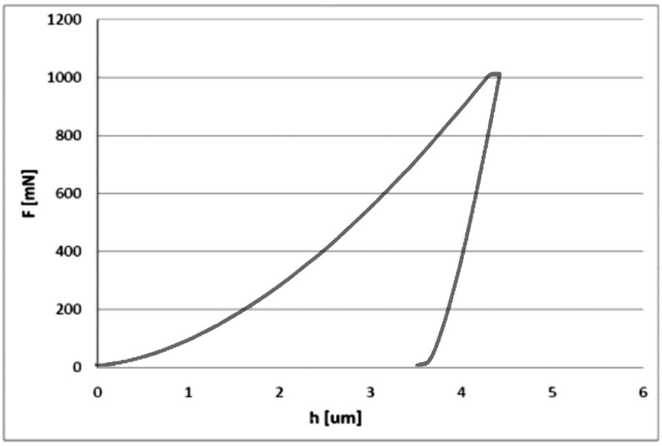
Where β is a correction factor which depends on the shape of the indenter, h_c is the contact depth and E_R is the reduced modulus defined as:

$$\frac{1}{E_R} = \frac{1-\nu_m^2}{E_m} + \frac{1-\nu_i^2}{E_i} \quad (3)$$

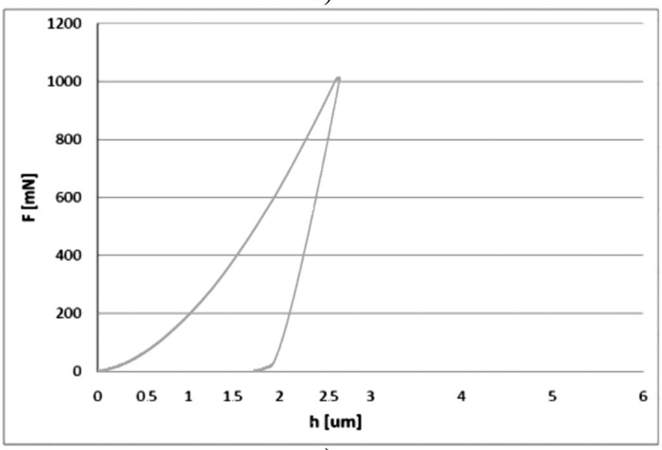
Where E_m and ν_m are the bulk modulus and Poisson's ratio of the material and E_i and ν_i are the same parameters for the indenter.



a)



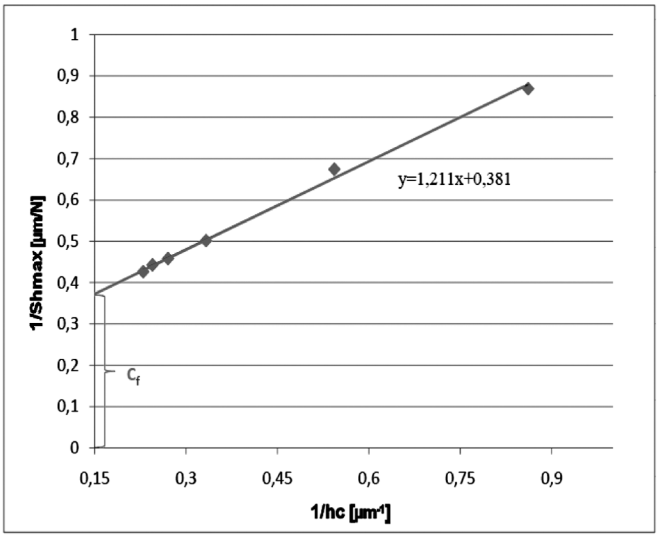
b)



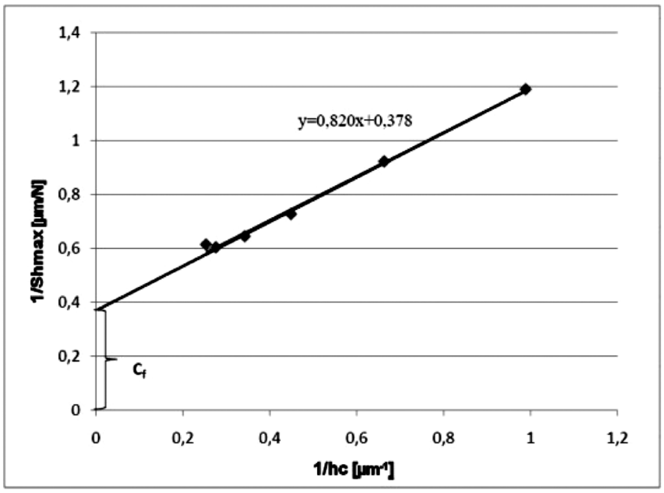
c)

Figure 2. Load – penetration depth curve: a - annealed state; b – Carburized state; c – Duplex treated state.

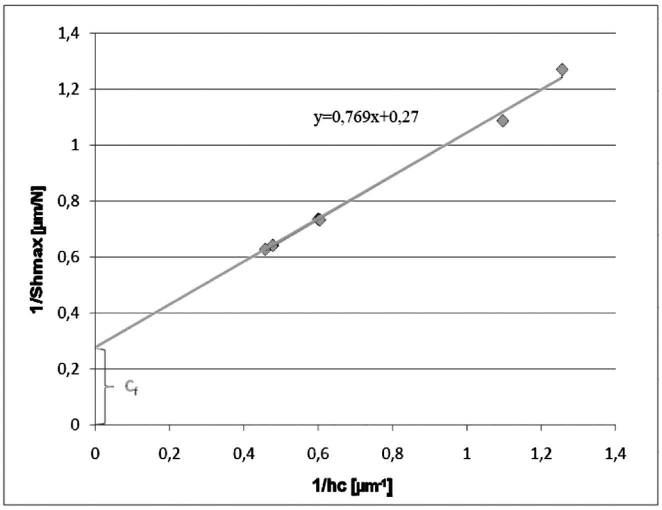
Figure 3 represents the inverse of the contact stiffness as a function of the inverse of the contact indentation depth obtained from Berkovich indentation realized on Duplex and non-Duplex samples.



a)



b)



c)

Figure 3. 1/S as a function of 1/hc performed on: a - annealed state; b - carburized state; c - Duplex treated state.

The representation is linear, so the slope is directly linked to the bulk modulus of the material according to equation 2. By considering the elastic properties of the indenter, 1140 GPa for the bulk modulus and 0.07 for the Poisson's ratio and by taking 0.3 for the Poisson's ratio of the material, 1.067 for the correction factor γ we obtain the value for the reduced modulus. The Martens hardness can be calculated using the formula:

$$H = \frac{P}{A_C} = \frac{P}{26.43h_c^2} \qquad (4)$$

Using the contact depth for calculation, an average value of 2.07 GPa (annealed state), 2.96 GPa (carburized state) and 7.41 GPa (Duplex treated state) was obtained for the Martens hardness.

The hardness values could be independent of load, it could increase or deacrease with load, and it could show a complex variation with load changes depending on the material.

The values of bulk modulus and Martens hardness of the Duplex and non-Duplex samples, are presented in Table 1.

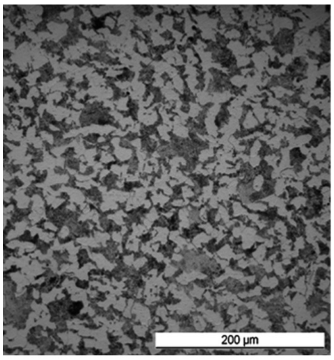
Table 1. Values obtained during micro indentation tests.

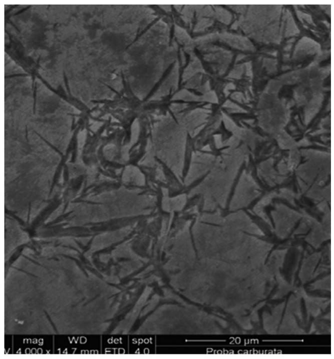
Structural state	Reduced modulus E_R [GPa]	Bulk modulus E_m [GPa]	Martens Hardness [GPa]
Annealed state	131	135	2.07
Carburised state	194	213	2.96
Duplex treated state	207	230	7.41

From table 1, it can be observed that the values of the reduced and bulk modulus are higher for the Duplex treated samples (207, respectively 230 GPa), compared to the untreated ones (131, 135 GPa).

The values of the Martens hardness are considerably increased from 2.07 GPa (annealed state) to 7.41 GPa (Duplex state).

The microstructure of the examined samples confirms the microindentation results. Therefore, in the annealed state, the investigated steel presents a ferito-pearlitic structure (Figure 4a), with a ratio of about 80% ferrite and 20% pearlite; After the carburizing treatments, the structure consists of P + Ce_{II} (Figure 4b), and after surface hardening by means of high frequency currents the structure consists in a fine martensitic structure with a small percentage of residual austenite (Figure 4c).





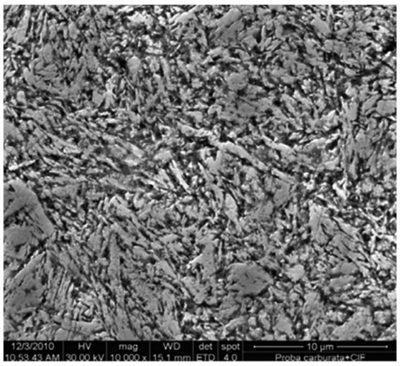


Figure 4. The microstructure of the examined samples: a- annealed state; b- carburized state; c- Duplex treated state.

4. Conclusions

The objective of these experiments was to determine the mechanical properties using instrumented indentation of Duplex treated EN 16MnCr5 alloyed steel consisted in gas carburizing followed by surface induction quenching.

Instrumented indentation technique proves to be very sensitive to the structural changes and permanent induced tension by the Duplex treatment consisting of carburizing followed by surface induction quenching and low tempering.

The load-depth curves show an increase of 70 % of the bulk modulus values obtained for the Duplex treated samples compared to the annealed ones.

The value of Martens hardness increases from 2.07 GPa (annealed state) to 7.41 GPa (Duplex treated state), which represents 250% increment. This increase can be justified by a fine microstructure and a reduced proportion of residual austenite.

Acknowledgement

This work was partially supported by the strategic grant POSDRU/88/1.5/S/50783, Project ID50783 (2009), co-financed by the European Social Fund – Investing in People, within the Sectoral Operational Programme Human Resources Development 2007 – 2013.

References

[1]. G.M. La Vecchia, N. Lecis, Impact test and tribological behaviour of duplex-treated low-alloy steel, Surface and Coatings Technologies, Vol. 205, 2010, pp. 614-619.

[2]. H. Kato, T. S. Eyre, B. Ralph, Surface Engineering, Vol. 10, 1994, pp.65.

[3]. T. Lampe, S. Eisenberg, G. Lauden, Surface Engineering, Vol. 9, 1993, pp. 69.

[4]. A. Matthews, K. Holmberg, S. Franklin, in: Thin Films in Tribology, Tribology Series, Vol. 25, Elsevier, Amsterdam, 1993, pp. 429

[5]. F. Gürer, The effect of retained austenite on abrasive wear resistance of carburized SAE 8620 steel, Master thesis, Middle East Technical University, 1988.

[6]. D.R. Dobra, I. Mitelea, The effect of Duplex treatments on corrosion properties of steels, Scientific Bulletin of Politehnica University of Timişoara, Timişoara, November 2011, pp. 33-38, ISSN 1224-6077.

[7]. T. Bell, H. Dong, Y. Sun, Realising the potential of duplex surface engineering, Tribology International, nr. 1–3, 1998, pp. 127–137.

[8]. K. Pantleon, O. Kessler, F. Hoffann, P. Mayr, Induction surface hardening of hard coated steels, Surface and coatings technologies, Vol. 120-121, 1999, pp. 495-501.