QUASI-STATIC NANOINDENTATION STUDY OF PHERITE-MARTENSITE DUAL PHASE STEEL

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Abstract

The paper of authors deals with the quasi static nanoindentation technique of the DP 600 steel. It is dual phase steel consists of martensite and ferite. The phases have different mechanical properties where their optimal combination gives benefit final mechanical properties of the steel. Nanoindentation testing is a method that consists essentially of touching the material of interest whose local mechanical properties as are hardness and elastic modulus are unknown. Nanoindentation testing is simply an indentation test in which the length scale of the penetration is measured in nanometres (10^{-9} m) rather than microns (10^{-6} m) or millimetres (10^{-3} m), the latter being common in conventional hardness tests. Both load and depth of penetration are recorded at each load increment (ultimately providing a measure of modulus and hardness as a function of depth beneath the surface). The presented paper also quantifies the difference in hardness and elastic modulus of both present phases.

Keywords: quasi-static nanoindentation, TI 950triboindenter, dual phase steel, scanning probe microscopy.

1 Introduction

Quasi-static nanoindentation is an experimental method for detecting local mechanical properties of materials. It is particularly useful in assessing the properties of very thin layers or properties of individual structural components and material phases. Nanoindentation consists in pushing a very small diametric tip into the surface of the material under investigation, during which the course of the applied load F and the material deformation h is recorded. These quantities are monitored during the load and release cycles. The result is the graphical course of F-h dependency - nanoindendation curve (Figure 1).

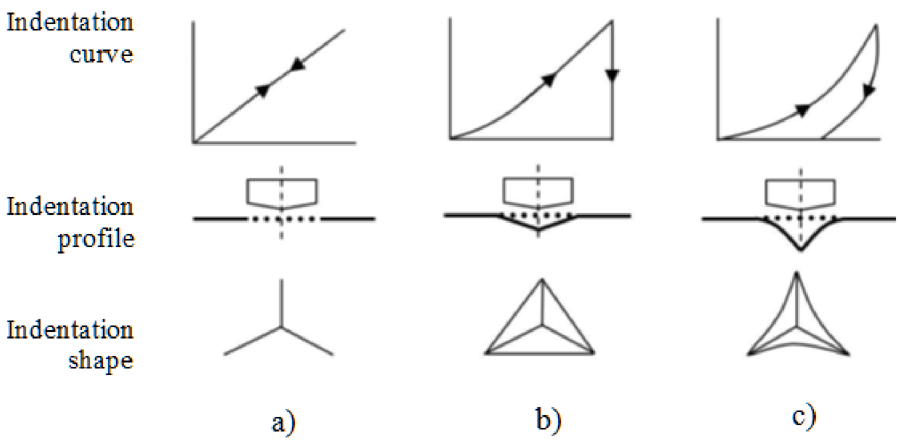

Fig. 1 *Characteristic indentation curves and corresponding imprints after indentation of the Berkovich spikes [2]*

In Fig. 1a, there is an indentation curve of an ideal elastic material where the plastic deformation does not occur and the material returns to its original condition after the load has been released. In Fig. 1b is an ideal plastic material, where, after loosening the load, the overall deformation is only plastic, so the material will be permanently deformed. However, in the real material testing, the overall deformation is a combination of the elastic and the plastic component (Figure 1c). Elastic deformation is eliminated after loosening the load, but the plastic deformation remains in the form of an imprint [1].

A typical nanoindentation curve of real material with a detailed description is shown in FIG. 2. Since the surface of the material is loaded with uniaxial load, the resulting deformation is evaluated and recorded as the depth of the imprint. Interpretation of the indentation curve has already been dealt with by many authors. The most commonly used method for its evaluation is the Oliver-Pharr method [3], which determines the hardness and motility of the nanoindentation curve. The modulus of elasticity is determined by the elastic part of the curve and the hardness of the elastic-plastic part and the imprint parameters. The area bounded by both curves again represents dissipative energy.

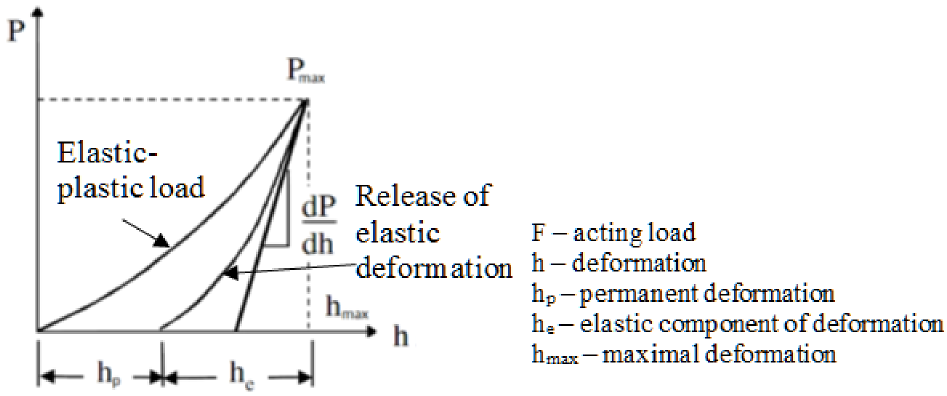

Fig. 2 *Typical load – displacement curve* [1]

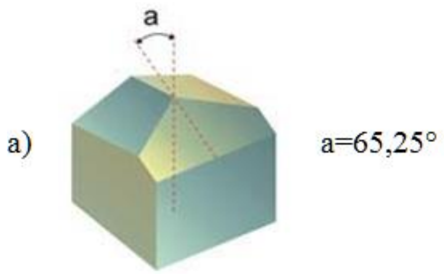

Fig. 3a *Geometry of Berkovich indenation tip* [5]

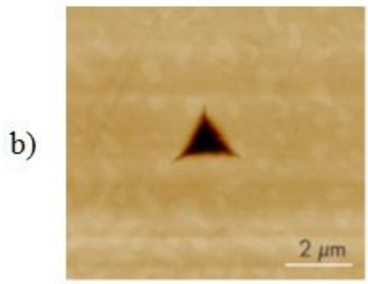

Fig. 3b *The size of indent scanned by the SPM* [5]

Hardness (*H*) is defined as the contact pressure under the indenter

$$H = \frac{F}{A_c} \tag{1}$$

where *F* is the applied load and *Ac* is the contact surface calculated for the indentation depth h. The slope of the tangent to the release portion of the curve is proportional to the modulus of elasticity of the material according to the formula

$$S = \frac{dF}{dh} = \frac{2E_r\sqrt{A_c}}{\sqrt{\pi}} \tag{2}$$

where *S* is the tilt of the tangent (contact stiffness), F is the applied load, and E_r is the so- reduced modulus of elasticity. The total deformation in the nanoindentation test is a combination of sample deformation and deformation of the indentation tip used. The actual modulus of elasticity of the sample (E_s) can then be determined from relation 3, and it is necessary to know the modulator of elasticity by the indenter (E_i) and poisson numbers of sample material (v_s) and indenter (v_i):

$$\frac{1}{E_r} = \frac{1-v_S^2}{E_S} + \frac{1-v_i^2}{E_i} \tag{3}$$

The most commonly used nanoindentation tip is Berkovich's triangular diamond tip, which is shown in Fig. 3a. In Fig. 3b is an example of an imprint after realization of nanoindentation by Berkovich tip. According to some authors [4], due to the properties of the Berkovich diamond tip (large modulus of elasticity, Poisson's small value), the relationship 3 can be reduced to the shape

$$\frac{1}{E_r} = \frac{1-\nu_S^2}{E_S} \quad (4)$$

2 Experimental material

Two-phase steels (DP) belong to the AHSS steel group (advanced high-alloy steels). DPs are characterized by high strength in combination with good plastic properties (ductility), these characteristics being given by their microstructure. The structure of two-phase steels usually consists of ferrite and a hard secondary phase - usually martensite. DP steels are widely used, especially in the automotive industry, and are used in the production of different body parts, various armrests and brackets, car seat parts, wheel disks and under. The DP600 steel sheet with h = 3 mm thickness was used for the experiment. The chemical composition of this steel measured by the spectral analysis and its basic mechanical properties are shown in Table 1.

Table 1 *Chemical composition (wt.%) And basic mechanical properties of DP 600 steel*

C	**Si**	**Mn**	**Cr**	**Mo**	**Ni**	**Al**	**Co**	**Cu**	**Nb**	**P**	**S**
0,1261	0,097	2,362	1,00	0,019	0,03	0,046	0,00	0,025	0,051	0,013	0,017
Fe	**Ti**	**V**	**B**		**R_m [MPa]**		**R_e [MPa]**		**A_{80} [%]**		**HV5**
95,7	0,02	0,012	0,003		650		380		21		370

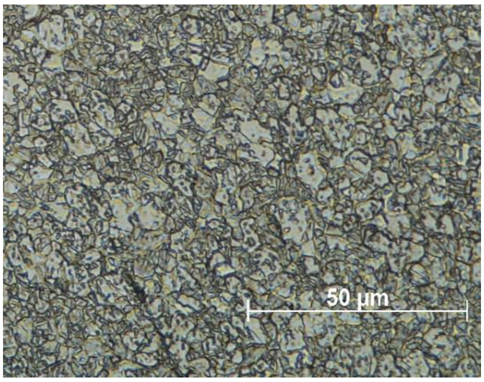

Fig. 4a *Microstructure of experimental sample created by the optical microscopy (etch Nital)*

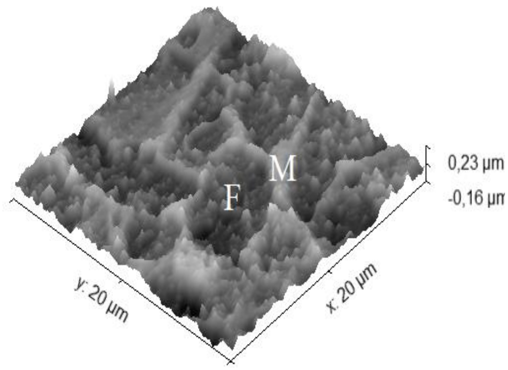

Fig. 4b *Surface of experimental sample created by the AFM microscopy*

In Fig. 4a, there is a photograph of the DP 600 microstructure. The basic components of the microstructure are ferrite (light areas) and darkness (dark areas). In more detail, the structure is shown in Fig. 4b via an AFM microscope. In this case, the lighter elevations of the martensite phase and the lower lower positions of the ferrite regions correspond.

3 Nanoindentaion analysis of phases

Within the nanoindentation test, the quasi-static nanoindent was performed with six injections at the selected microstructure site, the measured area being 20x20 µm. As the load curve, a standard trapezoid with a maximum of 1000 µN and a pushing time of 2 s was used. The Triboindenter TI950 nanoindentor was used for the measurement. The measured values of the nanoindentation hardness *H* [GPa] and the reduced modulus of elasticity *Er* [GPa] in the individual positions are shown in tab. 2. The measured positions are shown in Fig. 5.

Table 2 *Measured nanoindentation characteristics*

Position	Nanohardness *H* [GPa]	Reduced modulus of elasticity E_r [GPa]	Phase
1	2,57	204,71	F
2	1,97	198,61	F
3	2,32	221,92	F
4	6,13	169,09	M
5	5,68	163,76	M
6	5,53	172,62	M

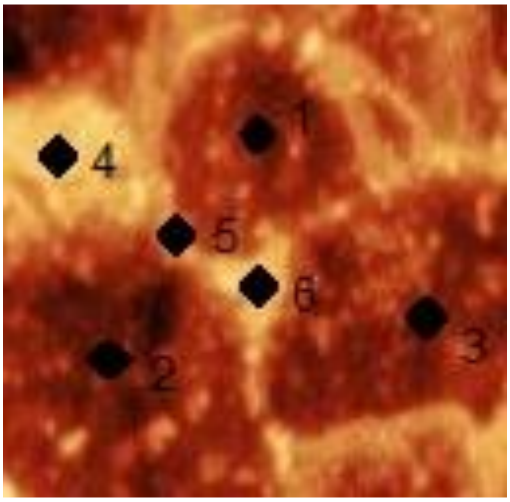

Fig. 5 *Measured positions – the SPM microscopy 10x10µm*

As an example, Fig. 6 of the nanoindentation curves corresponding to the ferrite (position 1) and the martensite (position 4). The shift of the ferrite curve to the right as well as the course of its load cycle confirmed the significantly better plastic properties of this phase compared to martensite.

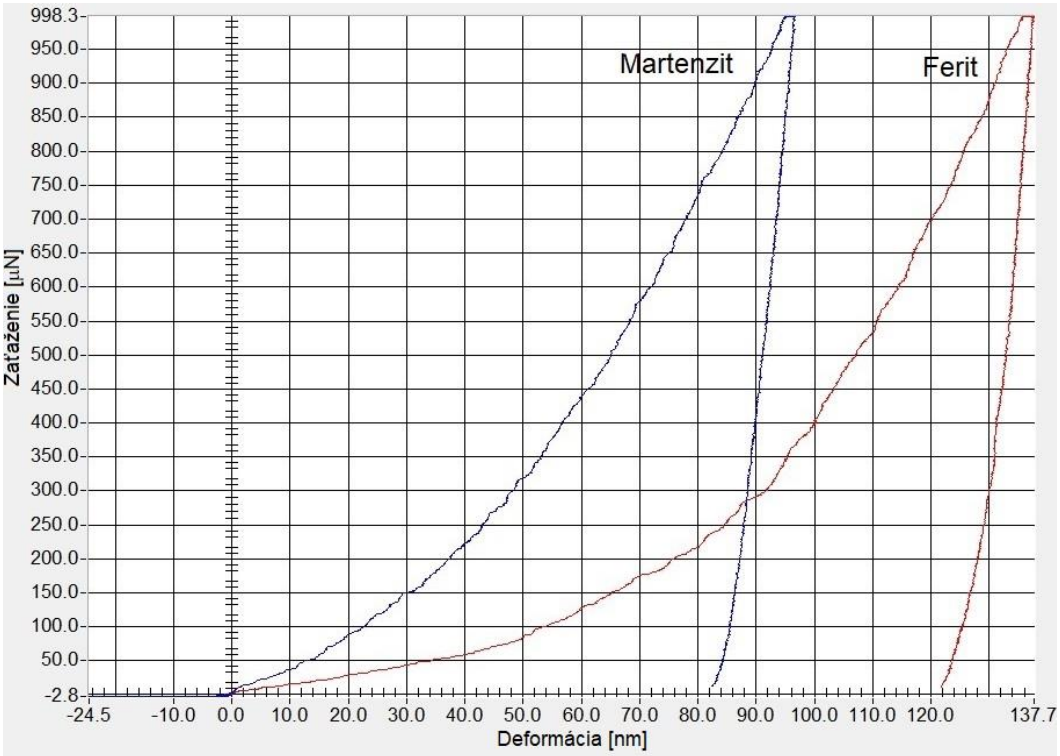

Fig. 6 *The nanoindentastion curves for the ferrite and martensite*

In Table 3 are the average values of nanoindentation hardness *H* [GPa] and reduced modulus of elasticity *Er* [GPa] for ferrite and martensite. A graphical representation of these values is indicated by the column graphs in Fig. 7 and Fig. 8.

Table 3 *The average values of all measured nanoindentation characteristicsfor the frrrite and martensite*

Phase	NanohardnessH [GPa]	Reduced modulus of elasticity E_r [GPa]
Ferrite	2,29	208,41
Martensite	5,78	168,49

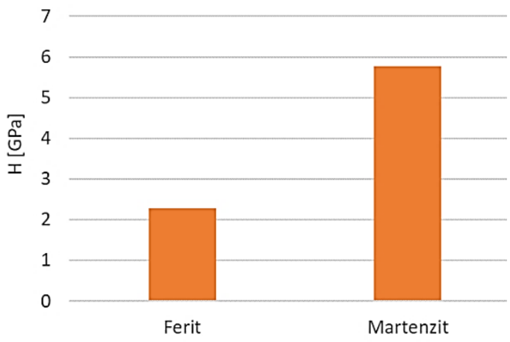

Fig. 7 *Graphical interpretation of nanoindentation hardness H [GPa] fot ferrite and martensite*

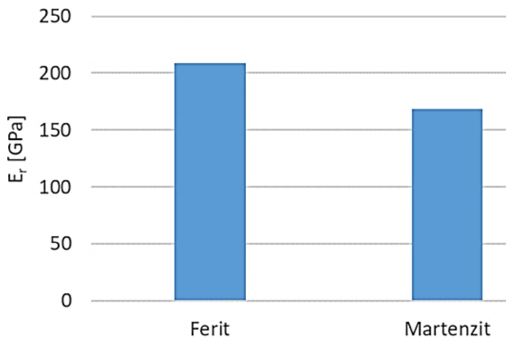

Fig. 8 *Graphical interpretation of reduced modulus of elasticity E_r [GPa] for ferrite and martensite*

4 Conclusion

The aim of the experiment, which is described in the paper, was the quasi-static nanoindentation of the basic structural components of the DP600 steel. The microstructure of this steel is made up of two basic phases - martensite and ferrite. The structure was analyzed by AFM microscopy and then quasi-static nano-indentation of the regions corresponding to the two phases was performed. The resulting nanoindentation hardness and reduced modulus of elasticity quantify the differences between the hard martensitic phase and the plastic ferritic phase. The optimal combination of both is advantageous for the mechanical properties of the DP600 steel. Using a quasi-static nano-indentation with a square-shaped matrix of the truth-distributed large number of prints, it is possible to determine the percentage of both phases present.

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