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USE OF SLIDING INDENTATION FOR TRIBOLOGICAL CHARACTERIZATION OF SEVERAL METALLIC MATERIALS

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ABSTRACT
The paper presents the using of sliding indentation for tribological assessment of several metallic materials. The indentation profile and the friction coefficient were measured. One of the tested materials has a laser cladding surface layer and the other has a hydrogenation treatment.

Keywords: sliding indentation, indentation profile, friction coefficient, cladding surface layer, hydrogenation

1. INTRODUCTION

The sliding indentation test was used to make a connection between several tribological properties (shape and size of the sliding indentation trace profile, friction coefficient) and mechanical characteristics of the material. Due to the fact that the sliding traces have a certain spreading range for the length, it may be made an average of the tribological properties. Therefore, this test can be used for inhomogeneous materials. The test is used for the studies of tribological properties of the materials surface layer [1, 2]. Also it is useful for evaluating the coatings [3, 4, 5]. There are two forces that act during the test: a normal force on the work piece surface, and a tangential one over the piece surface. They induce a complex state of tension into the surface layer. The indenter may have various shapes: usually sphere and cone [6]. Depending on shapes, dimensions, kinematics, energy, microgeometry, metallurgical characteristics (chemical composition, purity, microstructure) and mechanical parameters (hardness, tension) the deformations are elasto-plastical and plastical [7].

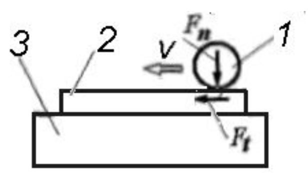


Fig. 1. The tribomodel for the sliding indentation: *1*- indenter, *2* - sample, *3*- support surface, *v* - horizontal sliding speed, F_t - tangential force, F_n - normal indentation force

In the paper, a review of two researches where the sliding indentation test is used for the surface layers investigations is presented. Tests were done on two grades of steel samples. Some of them have laser cladding surface layer and the other were made of X65 steel (hydrogenated and nonhydrogenated), cut from continuous casting slabs.

The tribomodel for this test is presented in Fig. 1. The experimental parameters are, as one may see from the figure, the speed (v), the normal indentation force (F_n) and the tangential force (F_t).

Driveline mechanical components and the presence of a frequency converter make the horizontal speed (v) to be low, adjustable within the limits from 0 to 0.17 mm/s. So, the application of the tangential force may be considered quasi-static.

Both normal and tangential force values were monitored and recorded with two resistive transducers and a data acquisition system.

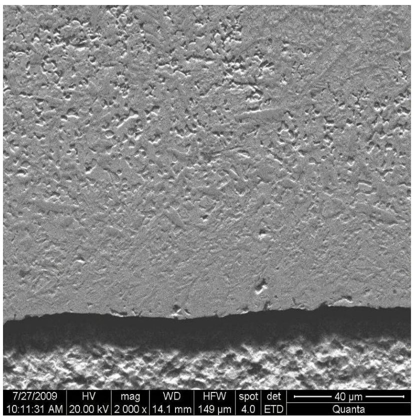
It is used a spherical indenter, a 12.7 mm diameter rolling ball. The ball does not rotate, having only a sliding moving on the surface of the sample. The hardness of the indenter is 62-63 HRC. So it can be considered a rigid one. The samples have a parallelepiped shape, with different dimensions.

2. TESTS OF LASER CLADDING SURFACE LAYER SAMPLES

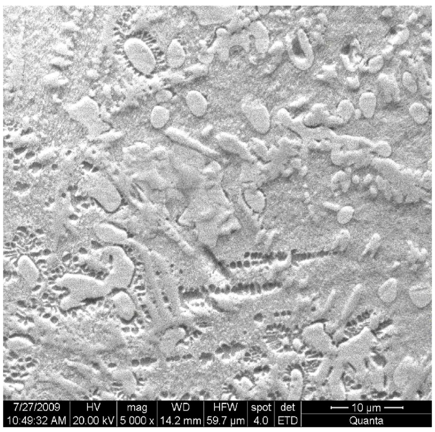
Laser cladding is a technique that improves the mechanical properties of the surface [8, 9, 10, 11]. There were several multi-layer claddings obtained by powder injection of Ni alloy from the Ni-Cr-B-Fe-Al system, in the bath melt by CO_2 laser, in continuous wave [12].

Cladding was performed on a 1C45, according to SR EN 10083-1:1994, with the following chemical compositions (wt. %): 13.75 Ni, 2.72 Mo, 0.019C, 0.50 Si, 1.87 Mn, 0.012 S, 0.022 P, 17.43 Cr, 0.002 Ni, 0.002 Al. The laser is a GT 1400 W type CO_2 continuous wave equipment, with x-y-z coordinate running table and computer programmed running. There were hardness differences between the centre and the edge of the laser clad layer. The centre has higher hardness than edges due to the presence of the intermetallic components (borides, carbides). The cause is a more intense heating of the edges, with an increased evaporation process. The deformation behaviour of the three specimens will be different.

The specimens were coded MB for those made of substrate material, coded A for those from the centre and B for specimen from an edge of the ingot. It was obtained the following values for the Vickers hardness HV_5: 3400 MPa for MB sample, 9270 MPa for a A sample and 9385 MPa for B sample. The microstructural analyses of clad layer and surface layer is presented in Fig. 2.



a) base of clad layer



b) surface layer. Electrolyte attack, solution 50% HNO_3

Fig. 2. Microstructure of clad nickel-based alloy

The normal indentation forces were F1=2886 N, F2=4330 N, F3=5773 N, F4=7216 N. After sliding indentation on the sample surface, traces like those in Fig. 3 were obtained.

The transversal profile of the sliding indentation trace was obtained by measuring six depth profiles across each trace with the stylus digital profilometer “SURTRONIC 3+”.

For the specimen code MB, the material is in an advanced plastic state and the penetration is deep. For the specimen A, the plastic deformation (the deep of the trace) is lower due to higher amounts of precipitates as comparing to the case of the substrate material. For the specimen B, having higher hardness, due to the large amount of borides that determines a minimal plastic deformation. The penetration deep is the lowest as compared to other two cases. All these aspects are presented in Fig. 4 [13].

The sliding indentation allowed a brief characterization of a laser cladding surface layer based on the study of the indentation traces. The shallower depth of the laser treated samples as compared to the untreated ones, reveals the superiority of this treatment.

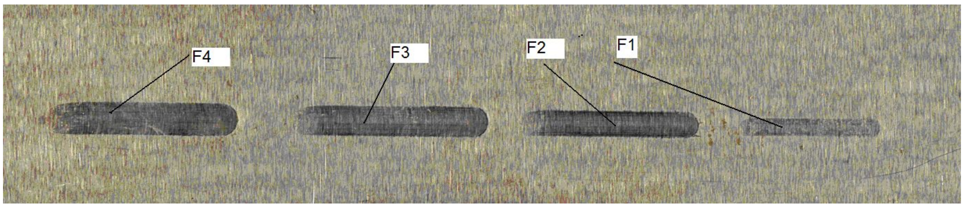
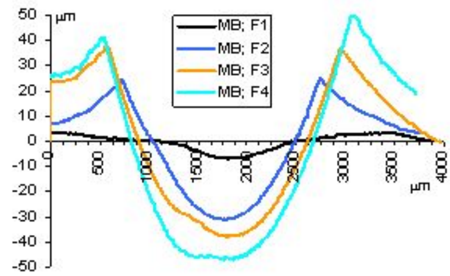
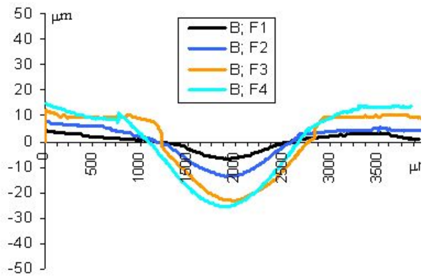


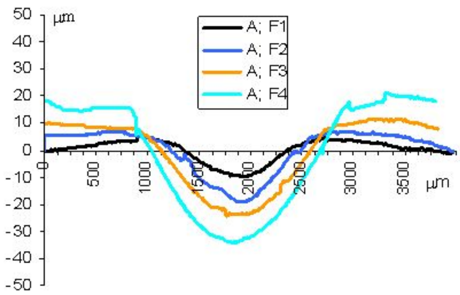
Fig. 3. Sliding indentation traces on specimen MB



a)



b)



c)

Fig. 4. Indentation traces depth profiles, for normal forces: F1=2,886 kN, F2=4,330 kN, F3=5,773 kN, F4=7,216 kN
a) sample MB;
b) sample B;
c) sample A;

3. TESTS OF HYDROGENATED AND NONHYDROGENATED SAMPLES MADE OF X-65 STEEL

X-65 steel is used for oil facilities (pipes and pressure tanks). Usually, it is made by continuous casting and that is the case of these samples. The samples were cut from the surface and from the middle of a slab.

One of the most commonly used test methods for these steel grades is the determination of the hardness. The sliding indentation may be a better test method than hardness. It can determine some characteristics of the bulk and superficial layer of the steel, like plastic deformations, friction coefficient, type of plastic deformation etc.

In the paper, the tribological behavior is determined by the friction coefficient developed during a sliding indentation test.

The hydrogen into steel through by the embrittlement that causes has negative implications in environment, health and economic life [14], [15]. The danger is greater especially for the steels for oil facilities. Such a steel is the grade X65 PSL 2 (according to API 5L standard).

Besides the existing hydrogen due to steel fabrication and casting process, a surplus hydrogen from petroleum products can appear. It causes the embrittlement of steel. Mechanical stresses and embrittlement lead to damages in pipelines and oil facilities [16], [17]. The chemical composition of the X 65 steel grade used in this research is presented in Table 1.

Table 1. Chemical composition of the slab

Chemical composition [wt %]											
C	Si	Mn	P	S	Al	Cr	Ni	As	Ti	Nb	Ca
0.065	0.29	1.59	0.013	0.008	0.037	0.250	0.155	0.005	0.018	0.058	0.0010

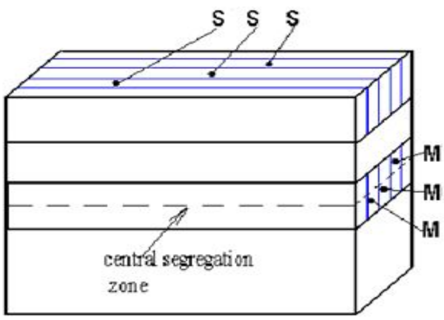


Fig. 5. The position of the samples related to the slab

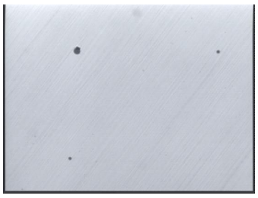
The test specimens were cut from the surface and the middle of a slab. They are coded S those cut from the surface and M for those cut from the middle of the slab (Fig. 5). Some of them were hydrogenated by bubbling sulphured hydrogen into acidulate salt solution for 96 hours (coded S_96 and M_96) [18]. The specimens coded S_0 and M_0 are nonhydrogenated.

The cooling conditions during the casting make the slab heterogeneous. The imagines in Fig. 6 illustrate this.

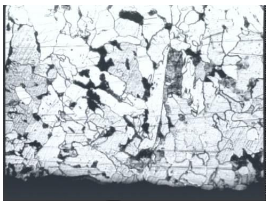
The analysis of nonmetallic inclusions reveals that the samples taken from the slab surface (Fig. 6a) generally have some small nonmetallic inclusions, only and gas holes. The nonmetallic inclusions are mainly oxides. For the samples taken from the middle of the slab (Fig. 6b), there are many gas holes and larger and denser nonmetallic inclusions. They are like a net. The inclusions are oxides, sulphides and nitrides.

The microstructure analyze (2% nital treated samples) shows a ferrite-pearlite structure with smaller and inhomogeneous grains, right on the slab surface (Fig. 6c). The ferrite is lamellar and globular.

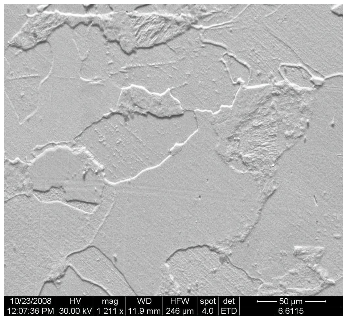
In the middle of the slab (Fig. 6d), the structure is ferrite-pearlite, as results due to the casting process, with bigger and inhomogeneous grains. Also, there are many gas holes, specific to casting and chemical inhomogeneity.



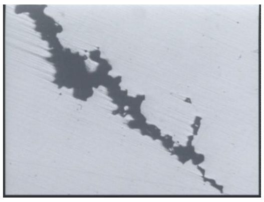
a) Sample S_0 (100X) metallographic microscope nonchemical treated sample



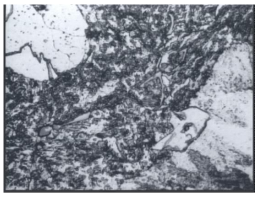
c) Sample S_0 (200X) metallographic microscope 2% nital treated samples



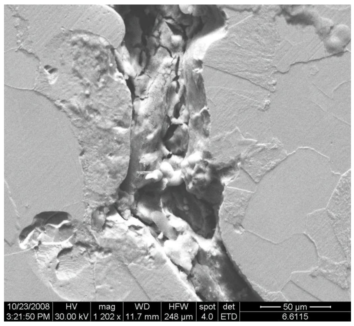
e) Sample S_0 (1211X) scanning electron microscope 2% nital treated samples



b) Sample M_0 (50X) metallographic microscope nonchemical treated sample



d) Sample M_0 (500X) metallographic microscope 2% nital treated samples



f) Sample M_0 (1202X) scanning electron microscope 2% nital treated samples

Fig. 6. The metallographic and scanning electron microscopy images for S_0 and M_0 samples [19]

The same things may be observed from the scanning electron microscope images (Fig. 6e and f).

The surface roughness has an average value of 2 μm with greater values for hydrogenated sample (2.2 μm). The sliding indentation was performed perpendicular to the grinding profile.

For the purpose of this paper the normal forces used for indentation of each sample were: 722 N, 1443 N and 2165 N. For each sample, the average tangential forces that oppose to the motion of the indenter were determined. The values of those forces are shown in table 2. It was computed the friction coefficient as the most synthetic characterization of the two forces (normal and tangential).

Table 2. The values of the tangential forces

Normal indentation forces F_n (N)	The averages of the tangential forces F_t (N)			
	Sample S_0	Sample S_96	Sample M_0	Sample M_96
722	101	30	59	45

1443	176	183	117	95
2165	332	331	220	175

The friction coefficient was calculated with well-known formula:

$$\mu = \frac{F_t}{F_n} \tag{1}$$

where μ is the friction coefficient, F_t is the tangential force, F_n is the normal indentation force. The values and the averages of the friction coefficients are shown in Table 3. The graphical representation of the friction coefficient values is shown in Fig. 7.

Table 3. The values of the friction coefficient

Normal indentation forces F_n (N)	Sample S_0	Sample S_96	Sample M_0	Sample M_96
722	0.140	0.042	0.082	0.062
1443	0.122	0.127	0.081	0.066
2165	0.153	0.153	0.102	0.081
Average values	0.138	0.107	0.088	0.07

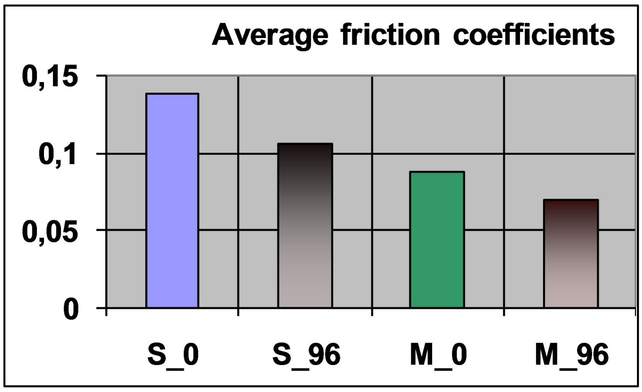


Fig. 7. Average value of friction coefficients

After testing, it was observed a decrease of the friction coefficient for the hydrogenated samples (S_96 and M_96), as compared to nonhydrogenated ones (S_0 şi M_0). The cause is the embrittlement of the X65 steel grade. Also, for the surface samples (S), the values of the friction coefficient are bigger than those for the middle samples (M). The explanation is the structure with big grains and the association of the nonmetallic inclusions and gas holes (see Fig. 6).

The tests shows that the slab is inhomogeneous and the hydrogenation impaired the mechanical properties of steel.

4. CONCLUSIONS

Through sliding indentation test, traces with different depths and corresponding tangential forces are obtained. These are directly related to the normal forces of indentation and the indented material quality.

The sliding over a certain distance allows for computating average value for the material characteristics. This is of particular importance, especially for inhomogeneous materials, such as those analyzed in this article. With the two forces, normal and tangential ones, the friction coefficient, the most synthetic characterization of the tribological behaviour, can be computed.

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