Hutnické listy č. 2/2016, roč. LXIX
ISSN 0018-8069

Mechanical Properties of Oxidized Steel Samples by Small Punch Test

Stanovení mechanických vlastností okují metodou small punch test

prof. Ing. Miroslav Raudenský, CSc.; prof. Ing. Jaroslav Horský, CSc.; **Ing. Jozef Hrabovský, Ph.D.**; **Ing. Petr Kotrbáček, Ph.D.**; **Ing. Michal Pohanka, Ph.D.**

Brno University of Technology, Faculty of Mechanical Engineering, Heat Transfer and Fluid Flow Laboratory, Technická 2, 616 69 Brno, Czech Republic

The aim of this work was to determine mechanical properties of oxidized samples made of two different types of steel, low-carbon steel (DC 01) and low-silicon steel (M47-FP). The small punch test (SPT) was chosen to examine several sheet specimens with an initial thickness of 0.5 mm previously exposed to oxidation at 1 000 °C. The formed oxide scale layers were mainly composed of hematite and magnetite and they had thicknesses according to oxidation time. The paper presents a method of determining suitable material parameters for transformation of force-deflection curves to the stress-strain curve for defined steel at elevated temperature (from 600 °C to 800 °C) using Ansys Workbench. For determination of the appropriate transformation, the Chaboche model was used. The presented results show the advantages and disadvantages of considered method that to can be employed in many engineering applications.

Key words: *small punch; oxide scale; Chaboche kinematic hardening; DC 01; M47-FP; stress-strain curve*

Cílem této práce bylo stanovit mechanické vlastnosti okují vytvořených oxidací za vysokých teplot na ocelovém povrchu. Studie byla provedena pro dva typy oceli: nízkouhlíkovou ocel DC 01 a křemíkovou ocel M47-FP s nízkým obsahem křemíku. Nízkouhlíková ocel reprezentovala typický materiál pro běžné aplikace. Křemíková ocel s nízkým obsahem křemíku odpovídala svým chemickým složením plechům používaným v elektrotechnice. Pro stanovení mechanických vlastností byl použit small punch test (SPT). Při tomto testu je do upnutého tenkého vzorku definovaného materiálu vtlačován hrot s kuličkou nebo zaoblený hrot. Kruhový vzorek je držen po svém obvodu mezikruhovými čelistmi a je deformován zvoleným hrotem ve vertikálním směru. Experiment a tedy celou úlohu je možné zjednodušit na osově symetrickou.

Vzorky s původní tloušťkou 0,5 mm, byly před provedením testu zokujeny při teplotě 1 000 °C. Oxidické vrstvy dosahovaly na uhlíkové oceli tlouštěk přes 100 μm a na křemíkové oceli kolem 25 μm. Vzniklé okuje se skládaly převážně z hematitu a magnetitu a jejich tloušťka závisela na době oxidace. Současně s testy zoxidovaných vzorků byly provedeny testy základního neoxidovaného materiálu. Všechny testy byly provedeny pro několik teplot deformovaného vzorku v teplotním rozsahu od 600 do 870 °C

Článek popisuje metodu pro stanovení materiálových parametrů pro transformaci změřených křivek síla vs. posunutí na křivku napětí vs. přetvoření pro definovanou ocel za zvýšených teplot. Jako nástroj pro získání odpovídajících materiálových parametrů byl zvolen software Ansys Workbench. K popisu materiálového chování měřených vzorků byl použit Chabocheho model s kinematickým zpevněním.

Výpočet probíhal iteračně, protože současně s hledanými materiálovými parametry bylo nutné stanovit součinitele tření mezi nástrojem a vzorkem při dané teplotě. Zjištěné součinitele tření jsou jedním z výsledků tohoto výzkumu. Výsledné závislosti napětí - přetvoření jsou silně teplotně závislé a jsou popsány vztahem, pro který jsou materiálové konstanty zjištěny postupem uvedeným v článku. Tyto závislosti je možné využít například při výpočtech, ve kterých se řeší deformace tenkých ocelových vzorků s vrstvou oxidů. Popisovanou metodu je možné použít i v dalších technických aplikacích.

Klíčová slova: *small punch test; vrstva okují; Chabocheho materiálový model; DC 01; M47-FP; deformační křivka*

In steel processing, such as continuous casting, hot rolling, and furnace heating, steel is frequently processed in relatively high temperatures and oxygen-rich environment (by water spraying or in wet ambient conditions). The growth of scale layers on the steel surface becomes an integral part of the production of steel. However, scale layers are the most undesirable by product, because the growth of scale layers could deteriorate the surface and material properties of steel. For example, the growth of scale layers can initiate micro cracks on the steel surface, which can cause the degradation of the physical and mechanical properties of semi-finished or finished products.

For these reasons, it is essential to understand the effect of the scale layers growth on the associated physical and mechanical properties. The proper understanding can lead a better control of scale layer growth, or more specifically, to provide appropriate schedule for descaling to eliminate the crack initiation or to predict the possible degradation due to the growth of scale oxide for maintaining the required integrity of the steel processed.

Because the inherent brittleness and inhomogeneity of oxide structures and the high processing temperatures at which the scales are formed and tested, the quantification or measurement of the mechanical properties of oxide scales can be very problematic and complicated. Furthermore, different steel compositions and processing conditions can create different types of oxide scales; as a result, a great variety of oxide layers with different morphological and chemical conditions can be obtained; this large variability of the oxides can add the complexness and difficulty of the measurement of the mechanical properties. Many techniques, such as tensile tests [1], bending tests [2, 3] and small punch tests (SPTs) [4], have been applied for studying the mechanical properties of the oxide scale formed on the steels or other metals. This paper continues in work presented in Punch Tests at Oxide Scales Surface of Structural Steel and Low Silicon Steel [5]. Methodology and results presented in this paper are based on the measurements described in [5]. Results measured by SPT methodology were processed and evaluated and are presented in this paper. The evaluation was focused on the mechanical properties of the tested specimens. This paper provides comprehensive view on the methodology of mechanical properties obtaining.

1. Method

1.1 The Small Punch Test

In the present study, the small punch test (SPT) is selected for the quantification of the mechanical properties of the steel specimens with different oxide scales. In SPT, as shown in Fig. 1, a punch with a semispherical (or ball) head is pushed at a constant speed through the bore of the dies to press the center portion of the specimen to rapture, where the specimen periphery is clamped and fixed between an upper and a bottom die. The tester is instrumented to provide punch load-displacement data until the specimen is fractured. The deflection in the center of the specimen at the maximum load (at fracture) is frequently measured as the major parameter to represent the material property.

The main advantage of SPT is its miniaturized specimen size. As compared with the dimensions of specimen for the standard tensile test (ASME E8/E8M) [6] or the standard bending test (ASTME855) [7], the size of the SPT specimen is approximately one-order of magnitude smaller. Thus, the SPT becomes less destructive and allows mechanical property changes to be determined locally, which can accommodate to the problems arisen from the inhomogeneity and brittleness of the steel with the oxide scale. The SPT is especially convenient for testing needed to be conducted at elevated temperatures, such as the test temperatures required in the present study, since its size is small enough to fit to most of the furnaces that have good high-temperature controls.

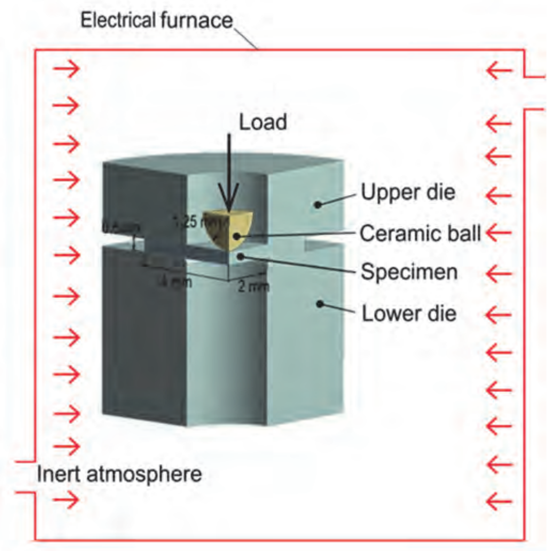

Fig. 1 Schematic illustration of the small punch test SPT
Obr. 1 Ilustrační schéma small punch testu

One of the first attempts to use the miniaturized SPT was the study of Baik et al. [8], who applied a SPT to study the degradation of the mechanical properties after long time exposure to an irradiation environment and correlated the SPT results to those based on the standard Charpy V-notch test. A test specimen much smaller than the standard Charpy specimen would be extremely desirable since small specimens take less space within a reactor in studying the neutron damage. Following this study, many Japanese investigators started to develop the SPT to evaluate the mechanical properties of different materials. Komazaki et al. [4] studied the influence of oxidation on the mechanical properties of Cu alloys used in rocket combustion chamber to understand the material integrity for the long-time service. The Cu alloy specimens were oxidized at 825 °C for 15 hours and then loaded for SPT at temperatures ranging from room temperature to 600 °C. Their testing results indicated that strength and ductility of oxidized specimen gradually decreased with increasing testing temperature accompanying intergranular brittle fracture. No specific values of the mechanical property degradation were reported. The SPT test together with X-ray diffraction of oxide scale was studied in [5].

Over the last two decades, the SPT has been developed to become a versatile and popular technique for studying mechanical properties for various types of materials using specimens with reduced sizes. It has become an ASTM standard for the determination of the

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ISSN 0018-8069

sheet metal failures under forming processes [9] and for studying the integrity of ultra-high molecular weight polyethylene used for surgical implant applications [10]. Recently, it has been under considerations to become a European standard for metallic materials [11].

1.2 The Inverse FE Analysis

The result of the SPT is relationship between the punch force and specimen deflection. The recorded load-displacement data serve for the inverse finite-element analysis (FE) to get stress vs. strain curve which characterizes the material response to mechanical loading. The basic and most important parameters of this curve are then yield stress and ultimate stress.

In the inverse FE analysis, a specific optimization algorithm is adopted to search the best-fit material properties, so that the load-displacement behavior calculated by the FE analysis matches well with the recorded experimental data [12]. The material properties to be searched should be well-defined in the material constitutive model used by the FE analysis. Note that the inverse technique used in the present purpose for material properties is also known as "parameter identification" [12 – 14], and was carried out by Screening, optimization method in toolbox Direct Optimization of computational program Ansys Workbench 14.0.

The material constitutive model used in this paper is originally developed by Chaboche [15, 16] and assumes that the materials are elasto-viscoplastic, which fits very well with the material behavior of steel under the SPT at the elevate temperatures.

Many authors deal with searching suitable parameters to convert force at the yield point and force at the ultimate point into the yield stress respectively ultimate stress. Several empirical parameters were presented [17, 18]. However, the empirical relationships are obtained for a specific type of specimens with defined thickness and tested under given conditions.

2. Experiment

2.1 Specimens Base Material

Two types of steel are chosen to investigate the composition effects on the mechanical properties of steel with oxide scales. The first type is silicon steel, DC01 (European Material No. 1.0330) [19], while the second one, M47-FP steel (ASTM and AISI designation), is non-oriented electrical steel [20]. The DC01 steel is one of the most popular low-carbon structural steels used by industries; its oxide scales are relatively easily removed from its surface, therefore can be considered as a benchmark for gauging the quality of steel with oxidescaled surfaces. The M47-FP steel is selected because of its high content of silicon and popularity used in magnetic core applications. Also, the oxide scale of the M47-FP is relatively difficult to be removed from its surface. Composition of both used materials is presented in Tab. 1.

Tab. 1 Chemical composition of the studied materials, (wt. %)
Tab. 1 Chemické složení studovaných materiálů, (hm. %)

Specimen	C	Mn	Si	P	S	Cr	Cu	Sn
DC 01	0.035	0.290	0.005	0.006	0.009	0.028	0.130	0.010
M47-FP	0.010	0.240	1.140	0.006	0.011	0.050	0.030	0.010

2.2 Specimens Preparation

The steel samples were cut into circular specimens with diameter of 8 mm and thickness of 0.5 mm and heated by an electrical furnace to 1 000 °C. As an oxidizing atmosphere, ambient atmosphere without addition of special gases was chosen. The values of the oxidation time and temperature for both materials are listed in Tab. 2.

Tab. 2 Oxidation time and temperature
Tab. 2 Časy oxidace a teploty

No.	Specimen	Temperature	Oxidation Period	Steel Thickness	Scale Thickness
		(°C)	(s)	(μm)	
1	Silicon steel	1 000	14 400	410	47
2	Carbon steel	1 000	1 200	183	230
3	Silicon steel	No oxidation	No oxidation	500	0
4	Carbon steel	No oxidation	No oxidation	500	0

By oxidation, four specimens were obtained: specimen of low silicon steel with an oxide scale layer, specimens of carbon steel with an oxide scale layer and two reference specimens of base materials without any scales (see Tab. 2).

2.3 Experiment Description

The oxidized specimens were loaded for the SPT at four different temperatures varying from 600 to 880 °C, where the tester was located inside of the electrical furnace as shown in Fig. 2. The temperature during testing was kept constant within ± 0.5 °C. The small punch tests have been performed on the experimental machines constructed by IPM ASCR at a constant speed until the specimen is ruptured. To validate the measurement, the test of low silicon steel with oxide scale layer at 800 °C was repeated.

Fig. 2 The apparatus used for the small punch test (SPT)
Obr. 2 Zařízení pro small punch test (SPT)

3. Numerical Simulation

3.1 Model of Geometry

Model of geometry for oxidized specimens was adjusted to match the actual model for SPT measurements. Modification consisted in distribution of the geometry of the samples into three parts. The top and the bottom corresponded to a layer of the oxide scales; the middle section represented the base material. Thicknesses of each layer were defined according to measurements by electron microscope. All layers were modeled as continuous and homogeneous. The corresponding geometry models are presented in Fig. 3.

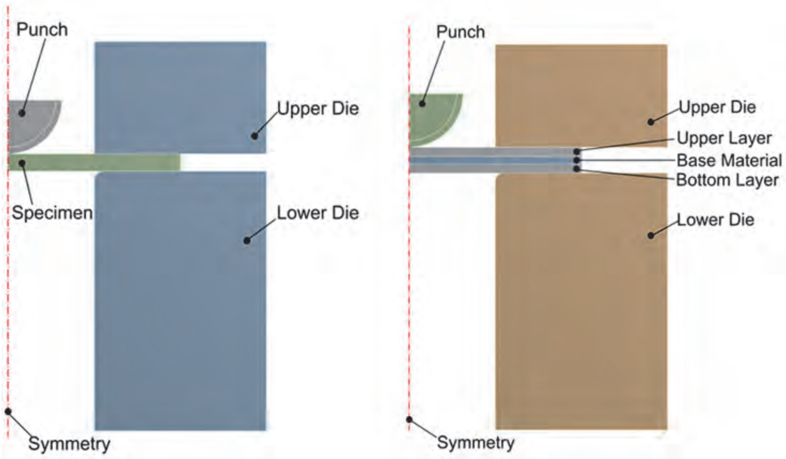

Fig. 3 Model of geometry for the non-oxidized (left) and oxidized (right) specimen
Obr. 3 Model geometrie vzorku bez okují (vlevo) a s okujemi (vpravo)

Individual components of the numerical model of geometry were connected through the contact surfaces. Contact areas were thus defined between the bottom and the top of the sample with bottom respectively upper and lower die. Further contact was then defined between the top of the sample and the puncher. All contacts were set up as contacts with friction. Frictional coefficient between the specimen and the die was defined by the value of 0.5. Frictional coefficient between the indenter and the specimen was considered as unknown parameter and was sought in experiment evaluation process.

3.2 Material Model

Chaboche´s kinematic hardening material model [21] was developed for a monotonic uniaxial loading. Chaboche model is a superposition of multiple kinematic models. Simplified form for the description of monotonic loading is published in [16], see eq. (1). This equation is used for description of material behavior during the SPT test. Individual terms of the equation determine the slope and the course of the stress (σ_x) – strain (ε_{px}) curve.

$$\sigma_x = \sigma_y + \frac{C_1}{\gamma_1}\left(1 - e^{-\gamma_1 \varepsilon_{px}}\right) + C_2 \varepsilon_{px}\,, \qquad (1)$$

where σ_y is yield stress and C_1, C_2 and γ_1 are the unknown material parameters to be found. For the formation of Chaboche´s model it is necessary to have experimental data from which the coefficients of Chaboche´s model are identified. Estimation of parameters can be performed by using mathematical approaches such as nonlinear regression with initial estimate of required coefficients. In numerical modelling and finding the optimal parameters for the specimen measured during the SP test the value of stress depending on the strain were not known. Therefore, the necessary parameters of Chaboche´s model were parameterized and searched by optimization method based on several values of force and corresponding to the value of deformation selected from the whole measured process.

Two basic parameters as Young´s modulus, frictional coefficient between ceramic ball and specimen and four parameters as yield strength, C_1, γ_1, C_2 which represent the Chaboche´s material model were selected for optimization process.

3.3 Evaluation

The unknown parameters (parameters in eq. (1) and interface friction coefficient) were searched by optimization, loop of numerical simulations. Optimization algorithm changes the unknown inputs so at the corresponding measured displacement the most accurate values of force were achieved. The input measured data for evaluation are shown in Fig. 4 and 5. The optimization task was carried out for all the measured curves based on several values of force and corresponding deformation.

However, the approach in this paper is general; the presented resultant values are based on some assumptions as crack initialization was not involved, the thermal component of Chaboche´s material model was neglected, material of steel and oxides were taken as homogenous.

4. Results and Discussion

Experiments with 4 samples were carried out at temperatures of 600, 700, 800 °C and for maximum temperature achievable for each sample (maximum temperatures varied from 870 to 880 °C). The records of force vs. displacement for each elevate temperature are shown for base (clean) material in Fig. 4 and for specimen with oxide scale in Fig. 5.

Evaluated material parameters for individual specimens at appropriate temperatures calculated by optimization are presented for base material of structural steel M47-FP in Tab. 3, for base material of silicon steel DC 01 in Tab. 4. The material parameters of samples with oxide scales are shown for structural steel M47-FP in Tab. 5 and for silicon steel DC 01 in Tab. 6.

The stress-strain curves for obtained material parameters based on Chaboche´s material model equation are plotted for base material in Fig. 6, for material with oxide scales in Fig. 7.

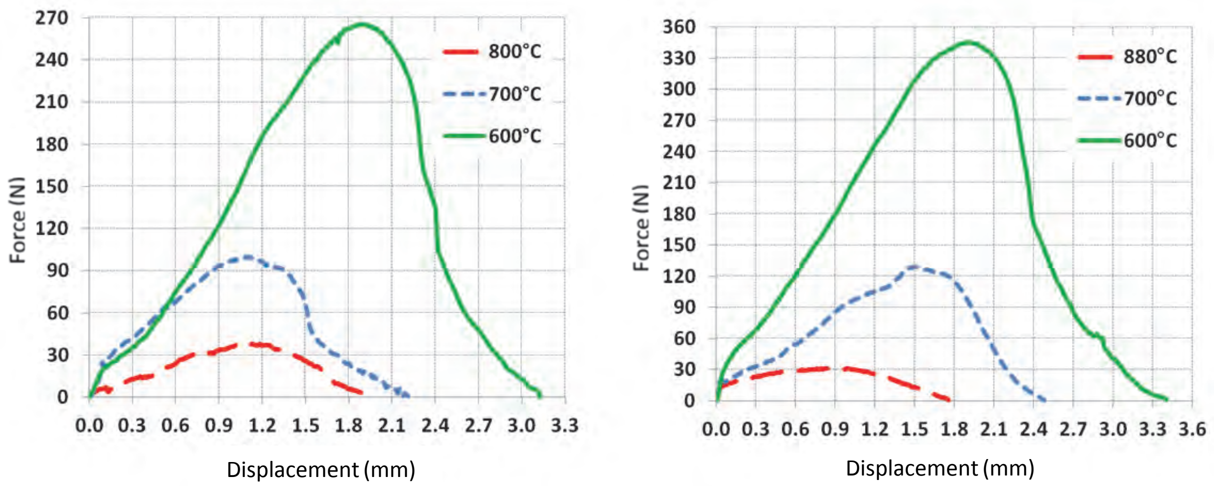

Fig. 4 Force vs. displacement for the samples without oxide scale layer in structural steel M47-FP (left), in silicon steel DC 01 (right)
Obr. 4 Závislosti síly na posunutí pro nezokujené vzorky z konstrukční oceli M47-FP (vlevo) a pro křemíkovou ocel DC 01 (vpravo)

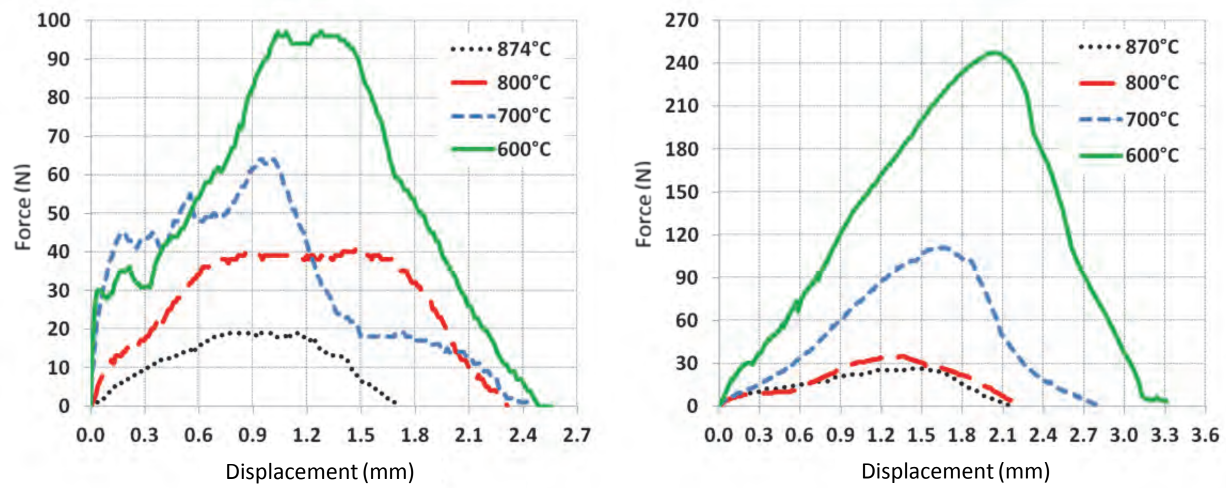

Fig. 5 Force vs. displacement for the samples with oxide scale layer in structural steel M47-FP (left), in silicon steel DC 01 (right)
Obr. 5 Závislosti síly na posunutí pro zokujené vzorky z konstrukční oceli M47-FP (vlevo) a pro křemíkovou ocel DC 01 (vpravo)

Tab. 3 Calculated parameters of the Chaboche´s material model equation for the base material of structural steel M47-FP

Tab. 3 Spočtené parametry Chabocheho modelu pro konstrukční ocel M47-FP

Temperature	Frictional Coefficient	Young´s Modulus	Yield Stress σ_y	C_1	C_2	γ_1
(°C)	(-)	(MPa)				(-)
600	0.26	6 800	85.0	800.0	105.0	200.0
700	0.26	6 700	62.0	1 200.0	2.5	250.8
800	0.28	2 120	19.0	870.0	2.2	190.2

Tab. 4 Calculated parameters of the Chaboche´s material model equation for the base material of silicon steel DC 01

Tab. 4 Spočtené parametry Chabocheho modelu pro křemíkovou ocel DC 01

Temperature	Frictional Coefficient	Young´s Modulus	Yield Stress σ_y	C_1	C_2	γ_1
(°C)	(-)	(MPa)				(-)
600	0.28	15 500	75.0	3 670.0	116.0	148.0
700	0.32	13 500	25.7	1 640.0	76.5	155.7
800	0.33	12 850	24.4	1 255.0	38.3	1 477.8
880	0.34	12 200	23.1	870.0	0.1	2 800.0

Tab. 5 Calculated parameters of the Chaboche´s material model equation for the specimen with oxide scale layer in structural steel M47-FP

Tab. 5 Spočtené parametry Chabocheho modelu pro zokujený vzorek z konstrukční oceli M47-FP

Temperature	Frictional Coefficient	Young´s Modulus	Yield Stress σ_y	C_1	C_2	γ_1
(°C)	(-)	(MPa)				(-)
600	0.26	12 000	21.0	620.0	0.07	1 200.0
700	0.26	11 000	20.0	600.0	3.50	2 011.2
800	0.28	1 130	15.0	112.0	1.07	1 255.7
874	0.31	450	5.1	96.9	0.05	2 870.7

Tab. 6 Calculated parameters of the Chaboche´s material model equation for the specimen with oxide scale layer in silicon steel DC 01

Tab. 6 Spočtené parametry Chabocheho modelu pro zokujený vzorek z křemíkové oceli DC 01

Temperature	Frictional Coefficient	Young´s Modulus	Yield Stress σ_y	C_1	C_2	γ_1
(°C)	(-)	(MPa)				(-)
600	0.26	1 480	25.1	302.0	15.0	602.1
700	0.26	1 110	7.6	277.0	4.6	1 579.7
800	0.28	656	3.2	261.0	9.5	1 869.4
870	0.31	655	2.6	875.0	4.0	466.5

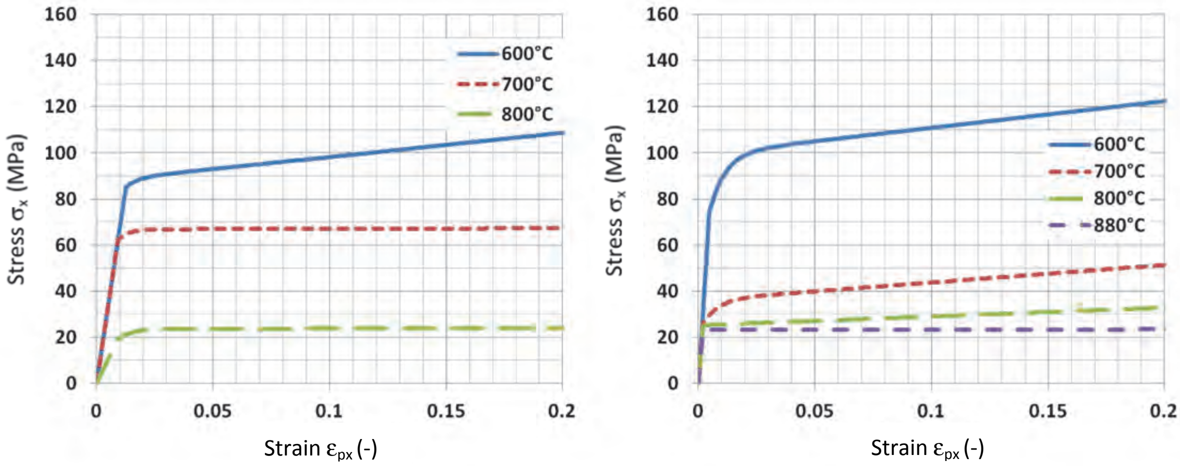

Fig. 6 Stress vs. strain determined for the samples without oxide scale layer in structural steel M47-FP (left), in silicon steel DC 01 (right)

Obr. 6 Závislost napětí na přetvoření stanovená na vzorku bez okují pro konstrukční ocel M47-FP (vlevo) a křemíkovou ocel DC 01 (vpravo)

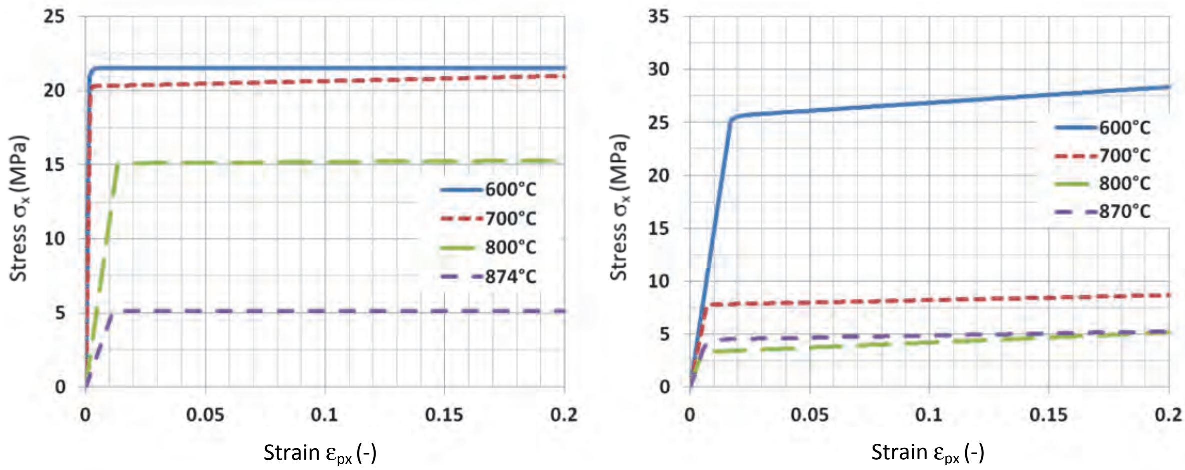

Fig. 7 Stress vs. strain determined for the samples with oxide scale layer in structural steel M47-FP (left), in silicon steel DC 01 (right)

Obr. 7 Závislost napětí na přetvoření stanovená na vzorku s vrstvou okují pro konstrukční ocel M47-FP (vlevo) a křemíkovou ocel DC 01 (vpravo)

5. Conclusions

Mechanical behavior of specimens with and without oxide scale layer was studied on two types of steel. The first type of specimens was made from structural steel, which is characterized by high oxide scale growth and its relatively low adherence of scale on the steel surface. The second type of specimens were prepared from silicon steel, it is characterized by slower oxide scale growth and its difficult descaling. Initial thickness of the samples was 500 µm, the final thickness of oxidized samples of silicon steel was 410 µm (covered both sides by 23 µm of oxides). The final thickness of oxidized carbon steel samples was 183 µm covered both sides by 115 µm scale layers. The presented results documents well sensitivity of the material parameters on the thickness, chemical composition and homogeneity of the oxide layer.

Small punch test at constant strain rate was chosen for analysis of mechanical behavior of the oxidized and clear samples. These tests were carried out on all types of specimens at elevated temperature (600 – 800 °C). Force-displacement curves at corresponding temperature were recorded.

The advantage which was presented in this paper is methodology of direct material parameters estimation through the Ansys Workbench without additional programs. In this case the fully-fledged Chaboche´s material model was applied. The numerical model was used in loop mode to get friction coefficient and coefficients to equation describing stress – strain behavior of the samples.

The presented results allow comparing behavior of structure (carbon) and silicon steels under deformation and are rare by providing deformation data for steel samples covered by oxides. The results are unique because the material properties of the oxide scales are very hard to identify.

Acknowledgements

The research leading to these results has received funding from the Ministry of Education, Youth and Sports under the National Sustainability Programme I (Project LO1202).

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