Recenzované výzkumné články

Determination of Actual Material Properties of Converter Vessel Using the SPT Method

Stanovení aktuálních materiálových vlastností nádoby konvertoru pomocí metody SPT

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The SPT (small punch test) method is very advantageous for evaluating the mechanical and brittle fracture properties of components, especially in cases where there is not enough material available for standard mechanical testing or it is necessary to evaluate the material non-destructively. The tested material is removed using a special device that ensures an absolutely minimal removal of material, and therefore no repairs are necessary after removal. In addition to the mechanical properties, it is possible to determine the chemical composition or to perform metallographic analysis of the samples taken, thus it is possible to provide a truly comprehensive material analysis with minimal material removal. Material and metallurgical research, Ltd. has more than 20 years of experience with the SPT method and its industrial application, one of the solved problems will also be presented in this article.

***Key words**: Small punch test; current material properties; remaining life; oxygen converter*

Metoda SPT (Small punch test) je velmi výhodná pro hodnocení mechanických, ale i křehkolomových vlastností komponent, obzvlášť v případech, kdy není k dispozici dostatek materiálu pro standardní mechanické zkoušení nebo je nutné materiál hodnotit nedestruktivně. Testovaný materiál je odebírán pomocí speciálního zařízení, které zajišťuje naprosto minimální odběr materiálu, a proto nejsou po odběru nutné jakékoliv opravy. Z odebraných vzorků, je mimo mechanické vlastnosti, možné stanovit chemické složení či metalografickou analýzu, tím je možné poskytnout opravdu komplexní materiálovou analýzu při minimálním odběru materiálu. Materiálový a metalurgický výzkum, s.r.o. má více než 20 let zkušeností s metodou SPT a její průmyslovou aplikací, jeden z řešených problémů bude prezentován i v tomto článku.

***Klíčová slova**: Small punch test; aktuální materiálové vlastnosti; zbytková životnost; kyslíkový konvertor*

1. Introduction

Small punch test (SPT) is a relatively recently developed method for evaluating the material properties of components that minimizes the amount of test material needed. It finds its use not only in power industry, for which it was originally developed, but also in many other industries, and it is also newly implemented in European standards [1]. A great advantage of this method is the use of miniaturized test specimens, which are produced from samples taken with a special sampling device (Fig. 1) directly on the operating component without negatively affecting its function and also without any repairs, only with the necessity of downtime. This method can thus be used in cases where it is necessary to obtain test specimens from very narrow layers of material, for example from decarburized layers, segregations, coatings, etc. Another typical example of the use of the SPT method is the evaluation of the material characteristics of specific areas of weld joints or deposit layers. Considering that the chemical composition can be determined from the samples taken and they can be subjected to metallographic analysis, the SPT method can be successfully used for comprehensive analyzes of actual material properties or for determining the remaining service life of the given component.

The test specimens of the small punch test method are small discs with a diameter of d = 8 mm and a thickness of t = 0.5 mm [1-3], which are punched in the matrix by a punch with a hemispherical top or a ceramic ball with a diameter of d = 2 or 2.5 mm [1, 4]. Schematically, this principle is captured in Fig. 2.

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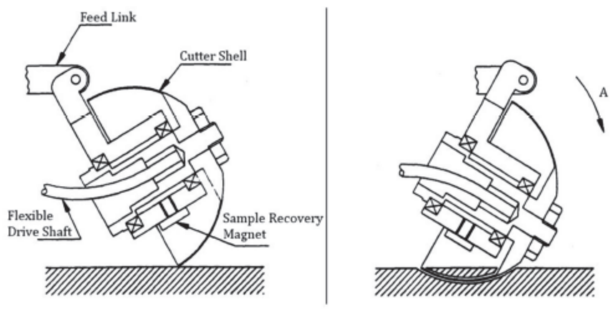

Fig. 1 Scoop for on-site SPT sampling [1]
Obr. 1 Odběrové zařízení pro SPT [1]

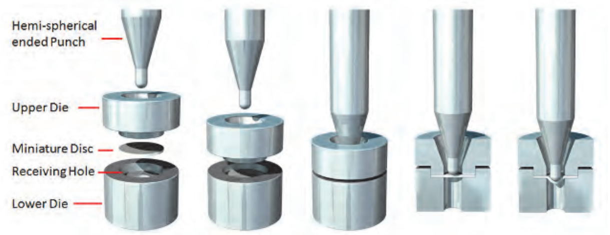

Fig. 2 SPT method principle [5]
Obr. 2 Schematicky znázorněný princip metody SPT [5]

During the test, the force applied to the sample (F) and the displacement of the punch (v) or the deflection of the sample (u) are recorded. The dependence of force – punch displacement (specimen deflection) created in this way can be divided into five characteristic areas [5], see Fig. 3:

(1) Elastic region
(2) Elastic-plastic transition (departure from linearity)
(3) Local bending, transition into membrane stress regime
(4) Membrane stress regime
(5) Final failure

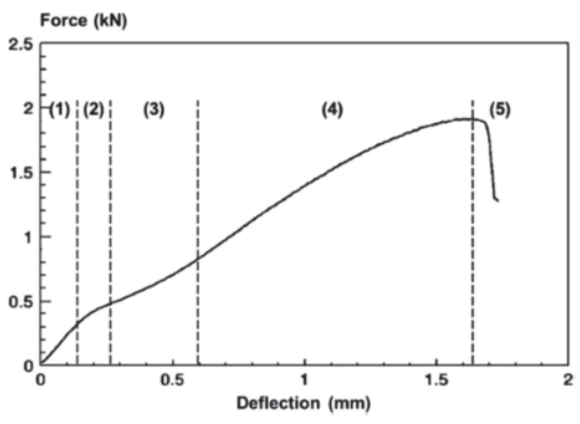

Fig. 3 Typical force-displacement curve obtained from the SPT [5]
Obr. 3 Typická křivka získaná pomocí SPT [5]

Following characteristics used for mechanical and other properties estimation are determined from the abovementioned curve [1, 6]:

- F_e – the elastic-plastic transition force in the small punch test which characterizes the transition from linearity to the stage connected with the development of plastic deformation through the whole thickness of the sample. This value corresponds to the yield strength in the conventional tensile test and it is defined as the point of intersection of two constructed tangents (in Fig. 3 one would be in area 1 and the second one in area 3) [N].
- F_m – the maximum force during the test which corresponds to the load at the tensile strength in the conventional tensile test [N];
- d_m – the displacement of punch tip which corresponds to the force Fm [mm];
- E_{SP} – surface below the force-displacement curve, calculated from 0 up to the F_m point [mJ]. ESP is important for transition temperature evaluation.

Determination of mechanical properties from SPT test values is realized in the form of correlations against a standard tensile test [1]. Equations (1) and (2) give the basic relationships for determining yield strength and ultimate strength.

$$R_{p0.2} = \alpha \cdot \frac{F_e}{{h_0}^2} \quad (1)$$

$$R_m = \beta \cdot \frac{F_m}{h_0 \cdot v_m} \quad (2),$$

where: $R_{p0,2}$ – yield stress [MPa]
R_m – tensile strength [MPa]
F_e – elastic-plastic transition force [N]
h_0 – initial thickness of test specimen [mm]
v_m – punch displacement [mm]
α, β – correlation coefficients

2. Experimental material and procedures

The subject of interest was a converter vessel for steel processing made of P460NH steel, when after the lining was broken there were concerns whether the charge could thermally degrade the material of the converter vessel as well. In an effort to minimize downtime, small samples were taken and the SPT method was used, as it eliminates the need for repair at the point of sample removal. The samples were removed by a special sampling device SSam^{TM}-2 from Rolls-Royce, Fig. 4. The material was sampled in three places, twice in the area where the lining was broken (A, B), the third reference sample was taken at the opposite wall (C).

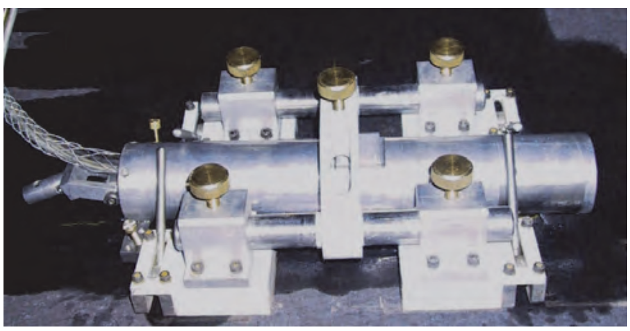

Fig. 4 Sampling device SSam^{TM-2} by Rolls-Royce with magnetic holder
Obr. 4 Odběrové zařízení Rolls-Royce SSamTM^{-2} s magnetickou upínkou

The chemical composition of the samples was subsequently determined by the combustion and ICP OES method, a metallographic analysis was performed to verify the higher level of structure degradation and its possible thermal influence, and last but not least, the mechanical properties of the converter vessel were evaluated by the SPT method.

The results of the analysis of the chemical composition (Tab. 1) confirmed the declared quality of P460NH steel with only a small positive deviation in the carbon content, which, however, undoubtedly could not negatively affect the mechanical properties of the converter the degradation of microstructure.

Tab. 1 Chemical composition of the converter vessel
Tab. 1 Chemické složení nádoby reaktoru

Element	C	Mn	Si	P	S	Cu	Cr	Ni	V
[wt. %]	0.24	1.63	0.51	0.015	0.02	0.33	0.05	0.20	0.17
P460NH	0.20	1.10 1.70	0.60	0.025	0.010	0.70	0.30	0.80	0.20

Three test specimens for penetration tests at laboratory temperature were prepared from each of the samples. From their results, the mechanical properties (yield strength, tensile strength) were subsequently determined using known correlation relations, which are shown in Table 2. Due to the very similar level of mechanical properties achieved, regardless of the place of sampling, it is almost certain that the thermal influence of the converter vessel could not happen. Although the exact thickness of the casing at the sampling point is not known, it falls within the interval of 60 to 100 mm, against which the obtained results are compared in Table 2. It can thus be concluded that the mechanical properties meet the limits prescribed by the standard throughout this interval. The higher yield strength can be caused by the increased carbon content.

Tab. 2 Mechanical properties of the converter vessel
Tab. 2 Mechanické vlastnosti nádoby reaktoru

Sample	Specimen No.	$R_{p0.2}$	R_m
		[MPa]	[MPa]
A	1	538	611
	2	540	651
	3	535	637
B	1	526	656
	2	541	661
	3	554	663
C	1	534	652
	2	555	670
	3	553	646
P460NH [7]			
60 mm < t ≤ 100 mm		**400**	**540-710**

Microstructural analysis was performed on a cut parallel to the plane of sampling. This orientation is associated with the atypical appearance of the structure, especially at a smaller magnification, although it is still the usual linearity of the ferritic-pearlitic microstructure, Fig. 5. At higher magnification, it became clear that while the original lamellar pearlitic structure still prevailed in the dark areas, even though these lamellae were already degraded there, in the light areas the degradation of the original ferritic-pearlitic structure was more pronounced and was accompanied both by the dissolution of pearlite and mainly by the formation of coarse cementite particles, which precipitated primarily at grain boundaries, Fig. 6. However, it should be emphasized that the microstructure of all three analyzed samples was qualitatively identical, none of them showed a more advanced stage of microstructure degradation, a mixed structure that would indicate overheating associated with the transition over the A_{c1} temperature, or signs of creep damage.

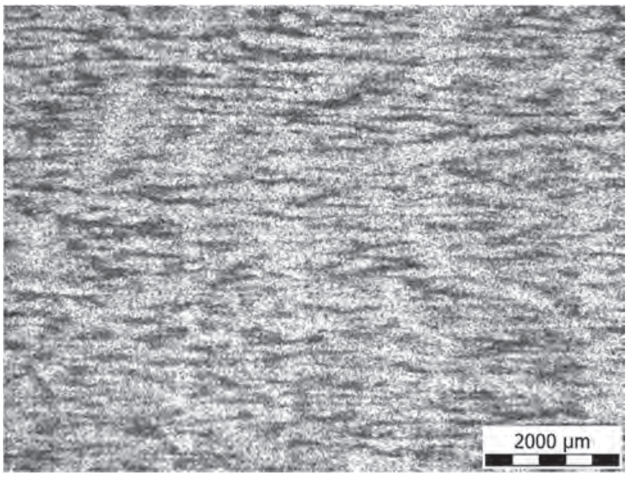

Fig. 5 Sample B – fine-grained ferritic-pearllitic structure
Obr. 5 Vzorek B – jemnozrnná feriticko-perlitická struktura

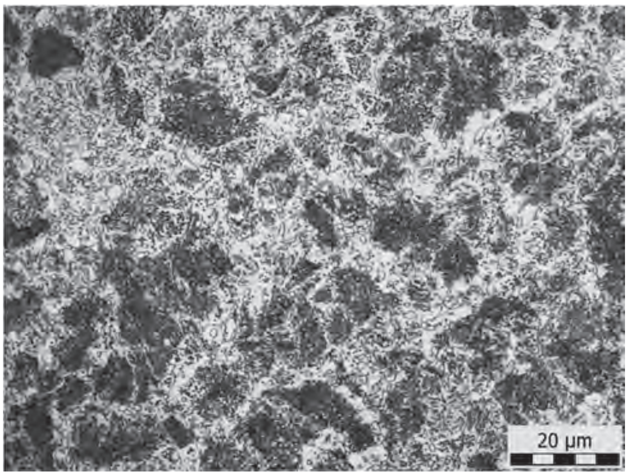

Fig. 6 Sample B –detail of the fine-grained ferritic-pearllitic structure
Obr. 6 Vzorek B – detail jemnozrnné feriticko-perlitické struktury

The changes in the microstructure as well as the state of the individual phases are better seen in the detailed high-magnification images obtained by scanning electron microscopy on a JEOL JSM 5510 in secondary electron mode. In Figs. 7 and 8, remnants of the original pearlite lamellar arrangement, signs of spheroidization of cementite lamellae, and presence of coarser spherical cementite particles at the grain boundaries can be seen, no

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structural differences between the potentially damaged and reference samples were found.

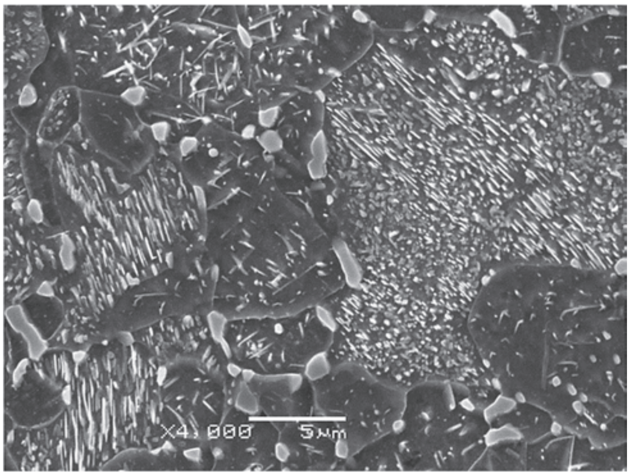

Fig. 7 Sample B –ferritic-pearllitic structure and coarse cementite particles on grain boundaries
Obr. 7 Vzorek B – feriticko-perlitická mikrostruktura a hrubé částice cementitu na hranicích zrn

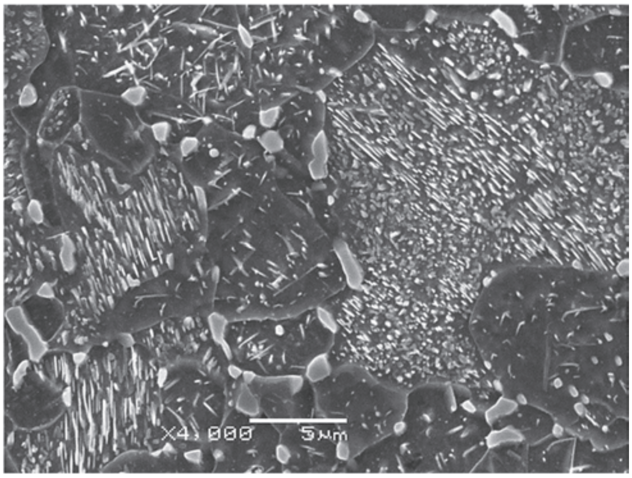

Fig. 8 Reference sample C – microstructure and coarse cementite particles on grain boundaries
Obr. 8 Referenční vzorek C – perlitická mikrostruktura a hrubé částice cementitu na hranicích zrn

3. Experimental results and discussion

Both the chemical composition of the converter vessel and its mechanical properties are (except for an insignificant positive deviation in the carbon content) in full compliance with the material standard. The relief that was observed in the monitored samples (no banding but alternation of light and dark areas) is caused by the fact that the sampling plane is parallel to the component surface which is usual for plates and sheets with ferritic-pearlitic structure. However, the microstructure has already shown signs of decomposition. With some exceptions, P460NH steel is supplied after normalization annealing and should therefore have a microstructure consisting of a mixture of ferrite and lamellar pearlite, possibly with a certain proportion of bainite. However, long-term exposure of steels at elevated temperatures leads to microstructural changes and thermally activated processes (non-conservative movement of dislocations, diffusion, precipitation and dissolution of secondary phases) which precede nucleation and growth of defects. Evaluation of the decomposition and transformation of the ferritic-pearlitic and ferritic-bainitic structure due to long-term exposure at elevated temperature can be assessed using standards for low-alloy heat-resistant steels [8]. This transformation escalates in six stages (A to F) and the extent of decomposition and corresponding microstructure changes during long-term creep exposure are clearly shown in Fig. 9 for both ferritic-pearlitic and ferritic-bainitic microstructures. Based on this comparison, the actual state of the converter vessel structure can be classified as C/D. However, the evaluated mechanical properties are still high enough, the decomposition of the microstructure and mainly the precipitation of coarse cementite particles at the grain boundaries can be a certain threat to the toughness of the steel and can cause an increase in the ductile-brittle transition temperature (FATT).

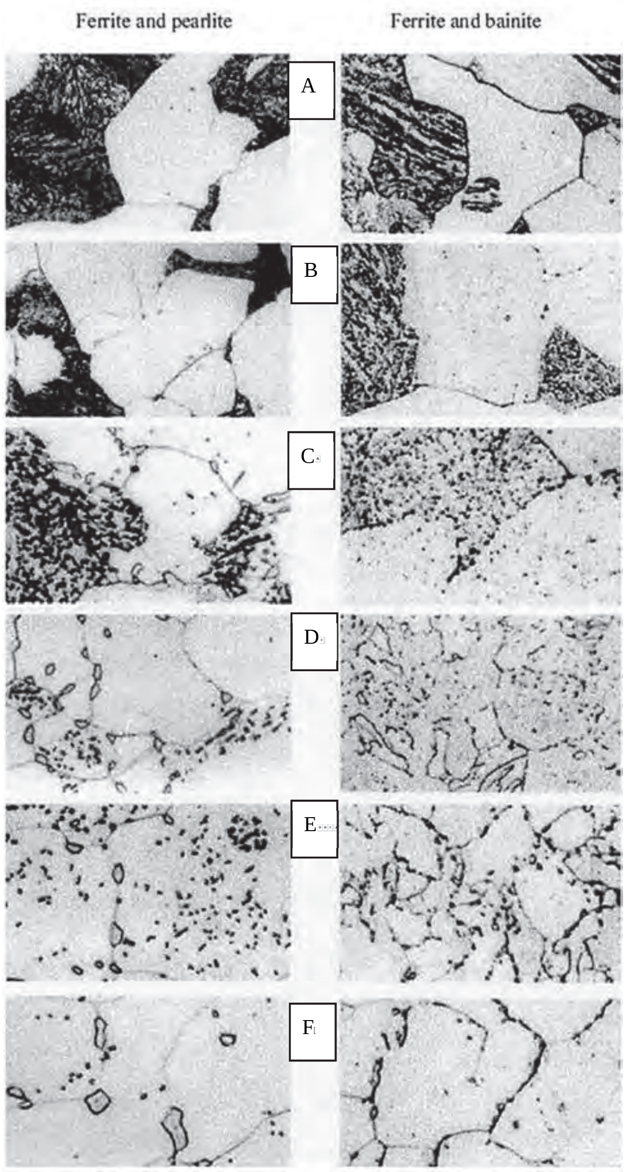

Fig. 9 Classification of structural state based on spheroidization of structure
Obr. 9 Klasifikace strukturních stavů podle sferoidizace struktury

4. Conclusion

Above-mentioned analyses demonstrate that SPT proved as useful evaluation method. The converter vessel does not show any signs of excessive heat load and following degradation of the microstructure beyond the normal structural changes that occur during its standard operation. The observed structural changes have not yet had any negative effect on the properties of the converter vessel therefore it could be put in operation without major disruptions.

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Evropská unie dovezla v prvním čtvrtletí 1,11 milionu tun ruských výrobků ze železa a oceli

V lednu až březnu 2024 snížila Evropská unie dovoz těžebních a kovových výrobků z Ruska o 18,4 % ve srovnání se stejným obdobím roku 2023 na 1,11 milionu tun. Náklady na dovoz ruských těžebních a kovových výrobků během tohoto období klesly o 11,9 % ve srovnání s lednem až březnem 2023 – na 583,3 milionů EUR.

Největší podíl na dovozu měly polotovary – 78,7 % z celkového dovozu. Za 3 měsíce EU dovezla z Ruska 874,1 tis. tun příslušných výrobků, meziročně o 4,2 % více. Největší podíl na dovozu polotovarů měla Belgie – 351,38 tis. tun (+2,2 % r/r).

Dodávky železné rudy z Ruska do EU dosáhly 9,36 kt, meziročně o 95,3 % méně. Celý objem surovin byl expedován na Slovensko. Dovoz feroslitin činil 22,42 tis. tun (+63,8 % r/r), surového železa – 198,75 tis. tun (-34,3 % r/r) a kovového šrotu – 6,38 tis. tun (-21,3 % r/r).

V lednu až březnu 2024 byli hlavními spotřebiteli těchto ruských výrobků ze železa a oceli:

- surové železo – Itálie – 137,8 tis. tun (-44,4 % r/r);
- feroslitiny – Nizozemsko – 16,34 tis. tun (+73,1 % r/r);
- šrot – Litva – 5,8 tis. tun (-17,8 % r/r).

V březnu 2023 EU zvýšila dovoz železných a ocelových výrobků z Ruska o 58,4 % m/ma 11,4 % r/r – na 534 tisíc tun. Dovozní náklady vzrostly o 63,9 % m/ma 28,6 % r/r – na 291,5 milionu EUR.

Navzdory sankcím uvaleným na Rusko ruský těžební a hutnický komplex nadále významně profituje z vývozu produktů do Evropské unie. Přestože se čísla ve srovnání s rokem 2023 výrazně snížila, dodávky jsou stále vysoké.

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