

Determination of Transition Temperatures Ductile-brittle Fracture from the Results of Penetration Tests

Stanovení přechodových teplot tvárný – křehký lom z výsledků penetračních testů

prof. Ing. Karel Matocha, CSc.; Ing. Ondřej Dorazil; Dr. Ing. Zdeněk Kuboň

MATERIAL & METALLURGICAL RESEARCH Ltd., Regional Materials Science and Technology Centre, Pohraniční 693/31, 703 00 Ostrava-Vítkovice, Czech Republic

The principal difference between the SP testing technique and standardized Impact testing lies in the fact that the SP tests carried out in accordance with CWA 15627 use disc-shaped test specimens without a notch. Especially in tough materials, the temperature dependence of fracture energy in the transition area is very steep and it lies close to the temperature of liquid nitrogen. In this case, the determination of T_{SP} can lead to significant errors in its determination. Efforts to move the transition area of penetration testing closer to the transition area of standardized impact tests led to the proposal of the notched disc specimen 8 mm in diameter and 0.5 mm in thickness with a "U" shaped notch 0.2mm deep in the axis plane of the disc.

The paper summarizes the results obtained when determining the transition temperature of Small Punch tests, T_{SP} according to CWA 15627 for material of pipes made of P92 and heat treated 14MoV6-3 steel in the as delivered state and a material of two steam pipe elbows made of 14MoV6-3 steel after long term operation at 540 °C. Although the obtained results confirmed the results of other workers in that the presence of a notch in SP disc is insufficient to increase the transition temperature significantly and certainly not to the values obtained by Charpy testing, comparison of the different behaviours of the tested alloys reveals some evidence that the notch reduces the energy for crack initiation. This implies that the test on a notched disc is more a test of crack growth and would be a useful instrument if included in the forthcoming EU standard for SP testing.

***Key words**: Small Punch Testing; Small Punch transition temperature T_{SP}; notched disc specimen; plane disc specimen; impact energy; Fracture Appearance Transition Temperature (FATT)*

Zásadní rozdíl mezi penetračními testy a standardizovanou zkouškou rázem v ohybu spočívá v tom, že pro penetrační testy je CWA 15627 doporučeno používat zkušební tělesa tvaru disku bez vrubu. Především u houževnatých materiálů je pak teplotní závislost lomové energie v tranzitní oblasti velmi strmá a je blízká teplotě kapalného dusíku. Postup doporučený v CWA pro stanovení T_{SP} může tak vést k významným chybám při jejím stanovení. Snaha posunout tranzitní teplotu T_{SP} blíže k tranzitní teplotě stanovené zkouškami rázem v ohybu vedla k návrhu zkušebních disků o průměru 8 mm a tloušťce 0,5 mm s „U" vrubem o hloubce 0,2 mm v ose roviny disku.

Článek shrnuje výsledky získané při stanovení tranzitní teploty penetračních testů T_{SP} podle CWA 15627 pro materiál trubek vyrobených z oceli P92 a z tepelně zpracované oceli 14MoV6-3 v dodaném stavu a materiál dvou ohybů parovodů po dlouhodobém provozu při teplotě 540 °C vyrobených rovněž z oceli 14MoV6-3. Ačkoliv získané výsledky potvrdily výsledky získané na jiných pracovištích v tom, že přítomnost vrubu v penetračním disku je nedostatečná na to, aby výrazně zvýšila tranzitní teplotu T_{SP} a už vůbec ne na hodnoty dosažené při Charpyho zkoušce rázem v ohybu. Porovnání výsledků získaných pro sledované materiály prokázalo, že přítomnost vrubu snižuje energii pro iniciaci lomu. Při nízké energii pro iniciaci nedochází k posunu tranzitní teploty T_{SP} k vyšším teplotám. To znamená, že penetrační zkouška prováděná na disku s „U" vrubem charakterizuje spíše odolnost vůči šíření lomu a mohla by být užitečným nástroje při interpretaci výsledků, pokud bude zahrnuta do připravované normy EU pro penetrační zkoušky.

***Klíčová slova**: penetrační zkoušky; tranzitní teplota penetrační zkoušky T_{SP}; zkušební vzorek tvaru disku s vrubem; energie rázu; FATT-tranzitní teplota pro 50% houževnatého lomu na lomové ploše zkušebního tělesa Charpy V*

CWA 15627 Part B: A Code of Practice for Small Punch Testing for Tensile and Fracture Behaviour describes the procedures recommended for determination of yield stress (*YS*), ultimate tensile strength (*UTS*), DBTT (measured by *FATT* and/or absorbed energy *TT* (for example 41 J)) and fracture toughness J_{IC} of the metallic materials from the results of Small Punch tests [1, 2].

DBTT expressed as Fracture Appearance Transition Temperature (*FATT*) is correlated according to the Code with T_{SP} (Small Punch transition temperature),

Hutnické listy č. 6/2016, roč. LXIX
ISSN 0018-8069

determined from the results of punch tests in the temperature range -193 °C to +23 °C in the form [1 – 5]

$$T_{SP} = \alpha \, . \, FATT_{Charpy}$$

Typical values for α for structural steels have been reported to be around 0.35 [6, 7] are indicative of much lower transition temperatures realised during the SP test than with conventional Charpy methods. The Small Punch transition temperature T_{SP} is determined according to the Code as the temperature where E^{SP} has its mean value of the highest and the lowest values in the transition region, by intersecting the smooth curve fitted from the energy versus temperature dependence of fracture energy E^{SP} [5].

The SP fracture energy E^{SP} is determined by integration of the force-displacement curve [1, 5]:

$$E_{SP} = \int_0^{u_f} F(u)du$$

The principal difference between the SP testing technique and standardized Impact testing lies in the fact that the SP tests carried out in accordance with the Code use disc-shaped test specimens without a notch. Especially in tough materials, the temperature dependence of fracture energy in the transition area is very steep and lies close to the temperature of liquid nitrogen. The procedure recommended in the CWA for the determination of T_{SP} can, in this case, lead to significant errors in its determination [8]. Efforts to move the transition area of small punch testing closer to the transition area of standardized Impact tests led to the proposal of the notched disc specimen. Up to the present only very few authors have introduced a notch in small punch testing, mostly in relatively recent studies [8 – 11]. However, the introduction of a sharp circular notch with a notch tip radius < 5 μm and of diameter equal to the punch diameter with the intention of maximising the notch tip load equally around the notch circumference did not result in the displacement of SP transition temperature T_{SP} towards the *FATT* obtained from conventional Charpy V notch tests [12]. The most recent attempts in the authors' laboratories to investigate the effect of notch geometry on SP test transition temperature have used both scratched (an orthogonal cross of 50 μm [13]) and a diametral EDM notch 200 μm deep [14] on a disc taken from a reactor pressure vessel steel. Although both types of notch contributed significantly to the initiation of the fracture process, they were found to have little effect on the ductile-brittle transition temperature, even with high strain rate testing closer to Charpy conditions. Nevertheless, it is under consideration to include notched disc testing in the proposed standard on Small Punch Testing and additional testing programmes are required to enable this decision to be taken. For this reason, and based on the results from published efforts cited above, it has been decided to extend the study to additional materials but concentrating on the 0.2 mm deep notched disc.

Fig. 1 shows disc test specimen with a "U" shaped notch 0.2 mm deep in the axis plane of the disc [2, 14].

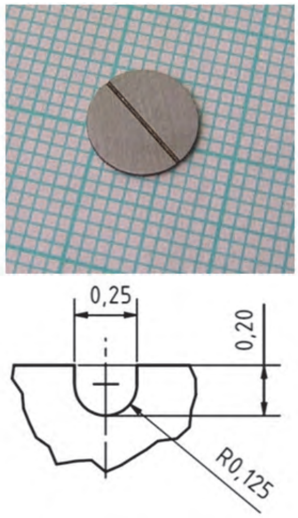

Fig. 1 Disc test specimen with "U" notch in the axis of disc plane
Obr. 1 Zkušební vzorek tvaru disku s „U“ vrubem v ose disku

The first results of SP tests at lower temperatures carried out on 14MoV6-3 steel indicated that the use of the notched specimens could shift to a limited extent the transition temperature T_{SP} to higher temperatures but not to the much higher transition temperatures obtained using Charpy testing (Tab. 2 below). Beyond the maximum fracture energy, the temperature dependence of fracture energy was found also to be less steep (Fig. 2).

In the present paper these results will be compared with the results obtained for tube ø 219 × 22.2 mm in as-received state made of P92 steel, steam pipe elbow ø 245 × 36 mm after 264 200 hours of operation at 540 °C made of low alloy steel 15128.5 and steam pipe elbow ø 245 × 36 mm after 270 000 hours of operation at 540 °C made of low alloy steel 15128.5

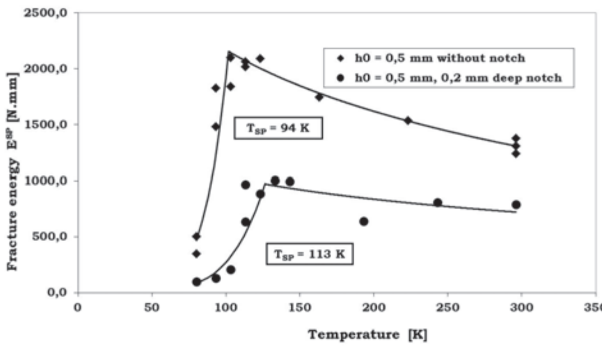

Fig. 2 The effect of the notch on the temperature dependence of fracture energy of penetration test. Pipe ø 457 × 28 mm made of 14MoV6-3 steel after heat treatment 940 °C/1 hour/air + 720 °C/2 hours/air. Crosshead speed 1.5 mm·min^{-1}, punch diameter 2.0 mm [2].
Obr. 2 Vliv vrubu na teplotní závislost lomové energie penetračního testu. Trubka ø 457 × 28 mm vyrobená z oceli 14MoV6-3 po tepelném přepracování 940 °C/1 hod./vzduch + 720 °C/2 hod./vzduch. Rychlost pohybu příčníku 1,5 mm·min^{-1}, průměr razníku 2 mm [2].

1. Test material

Tab. 1 summarizes the tensile behaviour at ambient temperature using round bars 8 mm in diameter. Test specimens were oriented in the longitudinal direction.

Tab. 1 Tensile behaviour of testing material at ambient temperature
Tab. 1 Výsledky zkoušek tahem při laboratorní teplotě

Material	***YS***	***UTS***	***A***	***Z***
	(MPa)		**(%)**	
P92	669	805	21.5	65
14MoV6-3	403	536	30.3	83
15128.5 after 264 200 hours	262	460	30.9	76
15128.5 after 270 000 hours	351	553	25.1	66

The microstructure of the P92 steel is composed of tempered martensite while the microstructure of the steel 14MoV6-3 in the as-received state is formed by a mixture of tempered ferrite and bainite. The ferritic microstructure of the elbow exposed for 264 200 hours at 540 °C was heavily tempered by long-term creep exposure, with carbides precipitated inside the ferritic grains, as well as along the primary grain boundaries. The yield stress of the elbow was quite low, just 65 % of the original value and more than 100 MPa below the requested minimum for this steel in the as-received state. The ferritic microstructure of the elbow exposed 270 000 hours at 540 °C was heavily tempered by long-term creep exposure, with carbides precipitated inside the ferritic grains as well as along the primary grain boundaries. Besides the ferrite, there were found also small blocks of tempered bainite. The actual yield stress of the elbow was found to lie just on the lower limit for the steel 0.5 %Cr-0.5 %Mo-0.25 %V in the as-received state (355 MPa) although the actual tensile strength was still quite high.

Tab. 2 summarizes transition temperatures *FATT* and Impact energies measured at *FATT* temperatures using Charpy V test specimens $(KV)_{FATT}$ and Charpy U test specimens $(KU)_{FATT}$ with notch 2 mm deep. All test specimens were oriented in the tangential direction.

Tab. 2 Results of Impact tests
Tab. 2 Výsledky zkoušek rázem v ohybu

Material	***FATT***	$(KV)_{FATT}$	$(KU)_{FATT}$	$(KV)_{in}$
	(°C)	**(J)**		
P92	-3	62	89	27
14MoV6-3	-10	117	173	56
15128.5 after 264 200 hours	+41	113	163	50
15128.5 after 270 000 hours	+84	74	132	58

The fracture initiation energy $(KV)_{in} = (KU)_{FATT} - (KV)_{FATT}$ of the P92 steel is significantly lower than fracture initiation energies of the 14MoV6-3 and 15128.5 steel after long-term operation.

2. Small punch test results and discussion

SP tests at -193 °C up to ambient temperature were carried out on the servo-mechanical testing machine LabTest 5.10ST following the procedures set out in [1], under crosshead control at crosshead speed of 1.5 mm·min^{-1} with puncher 2 mm in diameter using discs 8 mm in diameter and 0.5 ± 0.005 mm thickness. The load - cross head displacement curve was monitored during each SP test.

Fig. 3 and 4 show the temperature dependences of the fracture energy E^{SP} determined for 15128.5 steel after 264 200 hours of operation at 540 °C and P92 steel respectively using both plane disc specimens and disc specimens with the 0.2 mm deep notch. It can clearly be seen in Fig. 3 that the increase in the Small Punch transition temperature for the 15128.5 steel as a result of the introduction of the notch, namely from 100 K to 129 K, is similar to the result for the 14MoV6-3 shown in Fig. 2. However, the increase in value falls very much below the transition temperature obtained from the conventional Charpy test (Tab. 2).

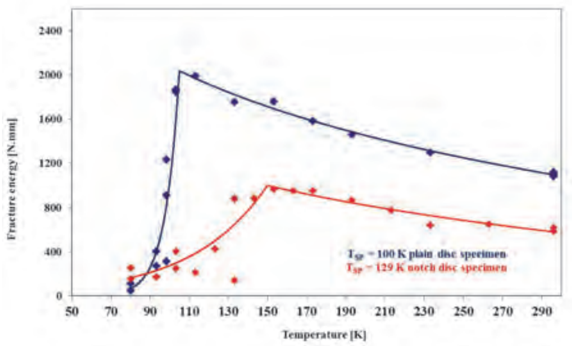

Fig. 3 Temperature dependence of Small Punch fracture energy determined for 15128.5 steel after 264 200 hours of operation at 540 °C using plane disc specimens 0.5 mm in thickness and disc specimens with 0.2 mm deep notch

Obr. 3 Teplotní závislosti lomové energie stanovené pro ocel 15128.5 po 264 200 hodinách provozu při 540°C na hladkých discích o tloušťce 0,5 mm a na discích s vrubem o hloubce 0,2 mm

The fracture results for the P92 steel (Fig. 4) are quite different from those for 14MoV6-3 and 15128.5 steels. The T_{SP} of 146 K derived from the un-notched tests and the maximum fracture energy is reached around 170 K, the transition part of the curve being much less steep than for 14MoV6-3 and P22 but reaching higher temperatures. This higher value for the SP transition temperature compares with the value of 133 K reported by Blagoeva et. al [15] for the comparable alloy P91. In comparison with the two lower alloy steels, there is no shift in the curve observed for the notched test at low temperatures, where the curve appears to follow exactly the same rise in fracture energy with temperature until around 150 K, coincidentally around the T_{SP} for the un-notched test, where the fracture energy commences its fall with further increase in temperature. A similar lack of displacement of the notched curve was also reported

Hutnické listy č. 6/2016, roč. LXIX
ISSN 0018-8069

for the alloy P91 but for 1 mm thick discs with and without 0.5 mm deep notches by Turba et al [12]. As a result of this lack of displacement of the curve to higher temperatures the T_{SP} for the P92 notched test is characterised by the lower value of 124 K. In this case, the introduction of a notch played no role in increasing the SP transition temperature towards the Charpy value, in fact quite the opposite.

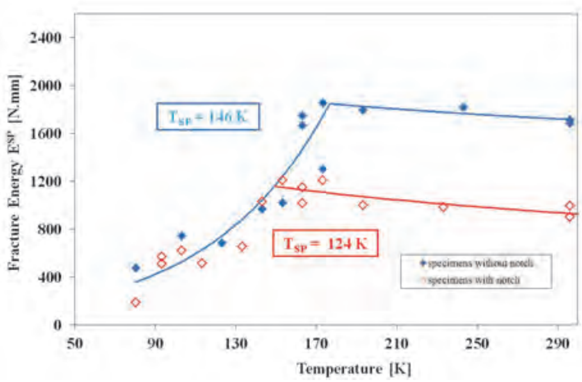

Fig. 4 Temperature dependence of Small Punch fracture energy determined for P92 steel using plane disc specimens 0.5 mm in thickness and disc specimens with 0.2 mm deep notch

Obr. 4 Teplotní závislosti lomové energie stanovené pro ocel P92 na hladkých discích o tloušťce 0,5 mm a na discích s vrubem o hloubce 0,2 mm

For all these types of notched SP test, it is important to understand the role played by the initiation of the crack and the energy for crack growth. Although the presence of the notch will undoubtedly play a role in crack initiation, the results show that the effect of the notch in the Charpy test is much more dominant, indicating that the main effect in the notched SP test lies with the crack propagation. As a result, the transition temperatures found in the notched tests are not so much affected by the notch but the fracture energies are certainly reduced for the notched specimens. This could simply be caused by the difference in ligament length, 0.5 mm for the un-notched and 0.3 mm for the notched specimens, through which the crack must propagate. This is also apparent in the results for P91 [12] where the fracture energy for a 1mm thick disc is clearly greater than for a 0.5 mm thick disc. It is intended to investigate this aspect in future work by testing plain discs of 0.3 mm thickness and notched specimens with 0.1 mm deep notches together with plain specimens of 0.4 mm thickness.

The acceptance of plant operators is of considerable importance in the application of SP fracture testing - whether they can rely on the α values obtained in predicting the *FATT* or DBTT properties for components, in particular where degradation in long-term properties may be expected for example through irradiation, creep or other microstructural damage. As Tab. 3 shows that the ratio of $T_{SP}/FATT$ determined using plane discs is significantly dependent on the type of material, although the differences appear to be smaller for the notched discs. For this reasons it would still appear to be justifiable to include notched SP tests in the forthcoming EN standard, although much more fundamental understanding of the actual role of the notch is required.

Tab. 3 Ratios $T_{SP}/FATT$ obtained using both plane and notched discs

Tab. 3 Poměry $T_{SP}/FATT$ získané na discích bez vrubu a s vrubem

Material	$(T_{SP})_{plane}$ (K)	$\alpha=(T_{SP})_{plane}/FATT$	$(T_{SP})_{notch}$ (K)	$\alpha=(T_{SP})_{notch}/FATT$
P92	146	0.54	124	0.46
14MoV6-3	94	0.40	113	0.43
15128.5 after 264 200 hours	100	0.32	129	0.41
15128.5 after 270 000 hours	124	0.35	149	0.42

Conclusions

On the basis of temperature dependences of Small Punch fracture energy E^{SP} determined for P92, 14MoV6-3 steel in as-received state and two 15128.5 steels after long term operation at 540 °C with different microstructures using both plane and notched disc test specimens and Impact tests for determination of *FATT* temperature it is possible to make the following conclusions:

a) The introduction of a 0.2 mm U-shaped notch into a standard 0.5 mm SP disc specimen raises to a limited extent the measured ductile-brittle transition temperature for 14MoV6-3 and two 15128.5 low alloys steels while marginally reducing the transition temperature for the P92 steel.

b) This confirms the results of other workers looking at different sizes and shapes of notches and indeed different materials that it is not possible to raise the transition temperature using a notched SP disc to the levels measured in a conventional Charpy test.

c) However, with increasing number of data being made available for the correlation of SP and Charpy fracture transition temperature using the relationship $\alpha = (T_{SP})_{notch}/FATT$, it is conceivable that the consistency of α values may be improved by obtaining them with notched SP tests and to this end these tests could usefully be introduced into the EN standard for SP testing presently in preparation.

Acknowledgement

Part of this paper was created within the Project No. LO1203 "Regional Materials Science and Technology Centre - Feasibility Program" funded by Ministry of Education, Youth and Sports of the Czech Republic.

Literature

[1] CEN WORKSHOP AGREEMENT "Small Punch Test method for Metallic Materials" CWA 15627:2007 D/E/F, December 2007.

[2] HURST, R., MATOCHA, K. Experiences with the European Code of Practice for Small Punch Testing for Creep, Tensile and Fracture Behaviour. In *3rd International Conference SSTT 2014 „Determination of Mechanical Properties of Materials by Small Punch and other Miniature Testing Techniques"*, Sept. 23 to 25, 2014, Castle Seggau near Graz, Austria, pp. 1–26, ISBN 978-80-260-6722-2.

[3] HURST, R.C., MATOCHA, K. The European Code of Practice for Small Punch Testing – where do we go from here? *Hutnické listy* (*Metallurgical Journal*), 63 (2010), In *1st International Conference SSTT „Determination of Mechanical Properties of Materials by Small Punch and other Miniature Testing Techniques"*, August 31 to September 2, 2010, pp. 5–11, ISSN 0018-8069, ISBN 978-80-254-7994-0.

[4] HURST, R., MATOCHA, K. Where are we Now with the European Code of Practice for Small Punch Testing? In *2nd International Conference SSTT „Determination of Mechanical properties of Materials by Small Punch and other Miniature Testing Techniques "*, Oct. 2 to 4, 2012, Ostrava, Czech Rep., pp. 4–18, ISBN 978-80-260-0079.

[5] DYMÁČEK, P., JEČMÍNKA, M. Applicability of Small Punch Testing Methods for Material Mechanical Properties at Room and High Temperatures. In *3th International Conference SSTT 2014 „Determination of Mechanical Properties of Materials by Small Punch and other Miniature Testing Techniques"*, Sept. 23 to 25, 2014, Castle Seggau near Graz, Austria, pp. 65–74, ISBN 978-80-260-6722-2.

[6] HA, J.,S., FLEURY, E. *Small punch tests to estimate the mechanical properties of steels for steam power plant*: II. Fracture toughness. Int. J. Pres. Ves. & Piping 1998. 75:707–13.

[7] FOULDS, J. R., VISWANATHAN, R. Small punch testing for determining the material toughness of low alloy components in service, *J. of Eng. Materials and Technology*, 5 (1994), 457–464.

[8] MATOCHA, K. Small Punch Testing for Tensile and Fracture Behaviour – Experiences and way Forward. Application of Miniature Small Punch Test Specimen in Determination of Tensile Properties, Small Specimen Test Techniques, In *6th International Symposium, ASTM STP1576, ASTM International*, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, 2014.

[9] JU, J., B., JANG, J., KWON, D. Evaluation of fracture toughness by small-punch testing techniques using sharp notched specimens. *Int. Journal of Pressure Vessels and Piping*, 80 (2003), 221–228.

[10] CUESTA, I. I., RODRIGUEZ, C., BELZUNCE, F. J, ALEGR, J. M: Analysis of different techniques for obtaining pro-cracked/notched small punch test specimens. *Engineering Failure Analysis*, 18 (2011), 2282–2287.

[11] LACALLE, R., ÁLVAREZ, J. A., CICERO, S., GUTIÉRREZ-SOLANA, F. From Archaeology to Precious Metals: Four Applications of Small Punch test. In *1st Int. Conf. SSTT (Small Sample Test Techniques)*, *Hutnické listy* (*Metallurgical Journal*), 63 (2010), 59–68.

[12] TURBA, K., GŮLCIMEN, B., LI, Y., BLAGOEVA, D., HÄHNER, P., HURST, R. Introduction of a new notched specimen geometry to determine fracture properties by small punch testing. *Engineering Fracture Mechanics*, 78 (2011) Issue 16, Nov., 2826–2833.

[13] ADAMS, J., BACHE, M. R., HURST, R. C., BORRADAILE, J. B. Ductile-brittle transition characterisation of reactor pressure vessel steels using notched small punch specimens. In *3rd International Conference SSTT 2014 „Determination of Mechanical Properties of Materials by Small Punch and other Miniature Testing Techniques"*, Sept. 23 to 25, 2014, Castle Seggau near Graz, Austria, pp. 1–26, ISBN 978-80-260-6722-2.

[14] ADAMS, J. *Development of small specimen test techniques for toughness measurement*. (Ph.D. thesis) University of Swansea, 2015

[15] BLAGOEVA, D, LI, Y.,Z, HURST, R.,C. Qualification of P91 welds through small punch creep testing. *J Nucl Mater*, 409 (2011) 124–30.

Téměř nová vysoká pec za 100 dnů

Westdeutsche Allgemeine *25.11.2016*

Za 120 mil. € je pec B hutních závodů Krupp-Mannesmann uváděna znovu na nejvyšší stupeň techniky. Práce probíhají jak uvnitř, tak venku. Nová vyzdívka je důvodem, proč zde pracuje něco mezi 450 až 550 spolupracovníků z cizích firem a další pracovníci z HKM (Hüttenwerke Krupp-Mannesmann). Vnitřní prostor vysoké pece má 11 m průměr, pec je vysoká 26 metrů.

Importní ceny: Dovoz oceli se citelně zdražil

Stahl Aktuell *01.12.2016*

Importní ceny v Německu se v říjnu silně zvýšily. Podle Spolkového statistického úřadu se index dovozních cen vyšplhal ve srovnání s předcházejícím měsícem o 0,9 %. Ve srovnání s předcházejícím rokem byl ale zaregistrován pokles importních cen o 0,6 %. Roční pokles se tak dále zeslabil: v září to totiž bylo -1,8 %, v srpnu -2,6 % a v červenci -3,8 %. Ocel byla nakupována výrazně dráž: například ceny kvartoplechů ze zahraniční za rok vzrostly o 11,9 % (+1,8 % oproti předcházejícímu měsíci) a široký pás, válcovaný za tepla se zdražil o 6,3 % (+1,8 %).