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APPLICATION OF SMALL PUNCH TEST METHOD IN STUDIES OF THE 14MoV63 STEEL BEFORE AND AFTER REVITALISATION

ZASTOSOWANIE METODY SMALL PUNCH W BADANIACH STALI 14MoV63 PRZED I PO REWITALIZACJI

The paper presents the results of studies of the chromium-molybdenum-vanadium steel designed for operation at elevated temperatures. The material was examined after long-term operation and after the revitalising heat treatment. Studies were performed by conventional methods and small punch test. Curves of ductile-brittle transition were plotted from the Charpy V-notch impact tests and were next compared with the curves of sudden loss of ductility obtained in the small punch test. Additionally, for the material before and after revitalisation, the values of temperatures T_{pk} and T_{nuc} were calculated using the method of inflection point.

Keywords: steel for operation at elevated temperatures, unconventional small punch test, impact strength, ductile-brittle transition temperature, temperature of sudden loss of ductility

W pracy przedstawiono wyniki badań stali chromowo-molibdenowo-wanadowej przeznaczonej do pracy przy podwyższonych temperaturach. Materiał badano w stanie po wieloletniej eksploatacji i po obróbce cieplnej rewitalizującej. Badania prowadzono z zastosowaniem metod konwencjonalnych oraz przy pomocy metody small punch. Sporządzono krzywe przejścia plastyczno-kruchości z próby udarnośći Charpy V i porównano je z krzywymi nagłej utraty ciągliwości z próby small punch. Dla materiału przed i po rewitalizacji określono wartości temperatur T_{pk} i T_{nuc} stosując metodę punktu przegięcia.

1. Introduction

Steels operating at elevated temperatures suffer microstructure degradation. Phase transformations that occur in the microstructure adversely affect the mechanical properties. This clearly shows the impact of long-term operation on reduced fracture toughness of the material [1,2,3,4,5] Monitoring of these changes is done during periodical inspections of the whole construction and is sometimes associated with the need to collect a large volume of material (so-called “plugs”) from which the specimens for mechanical tests are prepared. In the case of positive results, the places where the test material has been taken are repaired, which is time-consuming and can adversely affect further operation of the structure. These problems are avoided using the technique of small punch test (SPT), where only a small volume of material is collected by the scoop sampling method (SSAM) [6,7], and samples have the shape of mini discs with a diameter of 6.35 mm and a thickness of 0.50 mm. There is no need to repair the places where samples have been collected from the thick-walled components.

The mechanical properties of steel which suffered degradation during long-term operation can be recovered by revitalising heat treatment.

2. Scope and purpose of the study

This paper presents the results of tests carried out on a seamless pipe made from the 14MoV63 steel grade operating for over 100 000 hours at a temperature of 520°C and a pressure of 10 MPa. The steel was investigated after long-term operation and after the revitalising heat treatment. The heat treatment was typical for this grade of steel and consisted in normalising at a temperature of 950°C/20minutes followed by annealing at a temperature of 680°C/3 hours with air cooling. The material was tested by a conventional method and by SPT.

The aim of the study was to determine the ductile-brittle transition temperature T_{pk} and the temperature of sudden loss of ductility T_{nuc} for the 14MoV63 steel. After analysis it has been proposed to adopt as these characteristic points, the points of inflection observed on the curves plotted by both research methods.

3. Test results

Hardness and tensile strength tests of 14MoV63 steel before and after revitalisation showed no significant differences. Average values of these parameters were similar. On the other hand, the results of impact tests (Fig. 1) and energy measurements by SPT (Fig. 2) showed much larger discrepancies.

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3.1. Ductile-brittle transition curves from the impact test

Tests were performed on standard ISO Charpy V specimens taken parallel to the direction of plastic working. The curve plotted in solid line in Figure 1 shows the results obtained on steel after long-term operation, while the dashed line curve represents the revitalising heat treatment. It is easy to note that part of the curve corresponding to the ductile-brittle transition of the steel after revitalisation is shifted towards lower temperatures. At ambient temperature, the toughness of the material in service has assumed the value of 13J/cm², which is beyond any acceptable criteria. This value was increasing with the increasing temperature, to reach at a temperature of + 200°C the level of about 175J/cm². After revitalisation, these values were much higher. At ambient temperature they amounted to 126J/cm², but at about +100°C they increased to 179J/cm².

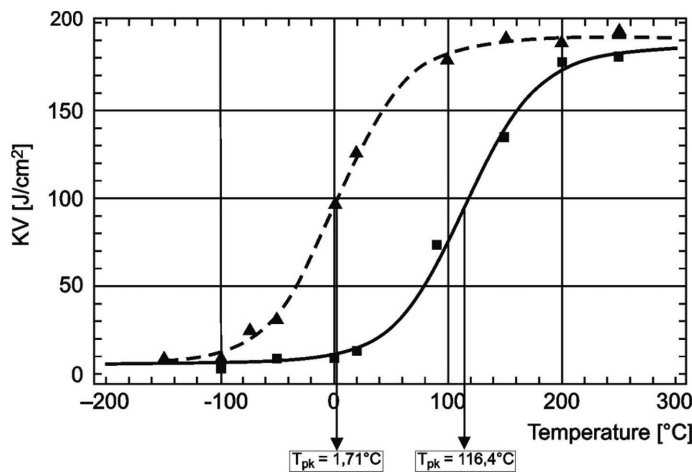


Fig. 1. Measuring points and graphs of approximation functions used to describe ductile – brittle transition phenomena; solid line – before revitalisation, dashed line – after revitalisation

The approximation function well describing the ductile-brittle transition phenomena has the following form:

$$y = a + \frac{b}{1 + \exp\left(-\frac{x-c}{d}\right)} \quad (1)$$

In equation (1), the value of the regression coefficient "a" corresponds to the level of toughness for the lower shelf energy in the range of brittle fracture and the sum of the "a + b" terms to the upper shelf energy in the range of ductile fracture [8]. The constant "c" denotes the ductile-brittle transition temperature T_{pk} .

3.2. Sudden loss of ductility curves from the SPT measurements

Mini discs for SPT were cut out from the broken halves of Charpy impact specimens. From each broken specimen about 20 mini samples were obtained for the small punch test. The method of cutting out mini discs is shown in Figure 2.

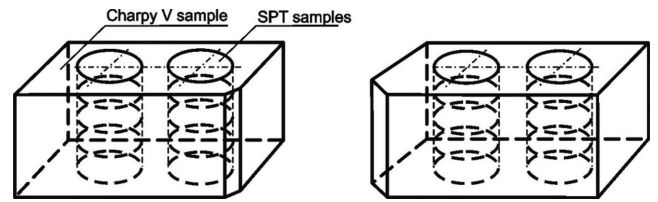


Fig. 2. The method of collecting mini SPT samples from Charpy V impact specimens

A chart from small punch test is schematically shown in Figure 3. The SPT curves were plotted using two parameters, i.e. the force with which the punch of a semi-spherical outline is pressed into the surface of a mini disc and displacement of the bulged central part in the bottom of the sample [3,9,10].

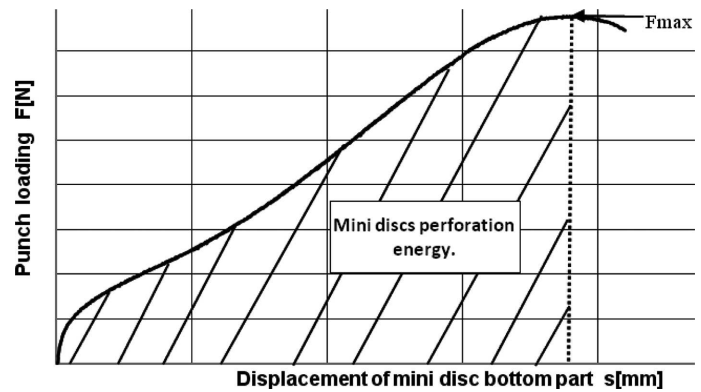


Fig. 3. Typical SPT curve plotted at ambient temperature

Using available data, the perforation energy needed to puncture the mini disc sample was calculated. It corresponds to an area under the curve calculated in respect of the maximum force F_{max} .

The experience gathered in earlier works has indicated that in studies of the small punch fracture toughness very important are tests conducted at low temperatures [11,12,3]. The sudden loss of ductility in the small punch test is to be expected at temperatures lower than the ductile-brittle transition temperature in Charpy impact test. This is due to the specific nature of small punch test, i.e. smaller dimensions of the samples which exhibit relatively higher resistance to cracking and the deformation rate lower than in the impact test [13]. For this reason, tests were carried out in the temperature range from +25°C to -196°C.

Average values of the energy used to puncture the mini discs at selected temperatures are shown in Figure 4.

With decreasing temperature, the energy of perforation increases for the steel after both long-term operation and revitalisation. For the steel after long-term operation it reaches a maximum of 2.05J at a temperature of -75°C. In the material after revitalisation, the maximum value of the energy of perforation is higher and amounts to 2.60J, occurring additionally at a lower temperature of -125°C. Having reached its maximum, the value of the energy is observed to undergo a sharp decline. Both materials become very brittle.

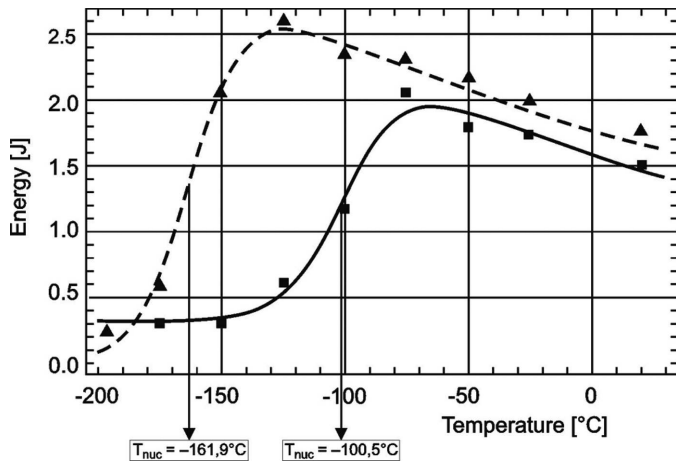


Fig. 4. Measuring points and graphs of approximation functions describing the phenomena of sudden loss of ductility according to SPT method; solid line-before revitalisation, dashed line-after revitalisation

These phenomena are well described by approximation functions of the following type:

$$y = a + \frac{b}{g} \left[1 + \exp\left(\frac{x + d \ln g - c}{d}\right) \right]^{-\frac{g+1}{g}} \exp\left(\frac{x + d \ln g - c}{d}\right) (g + 1)^{\frac{g+1}{g}} \quad (2)$$

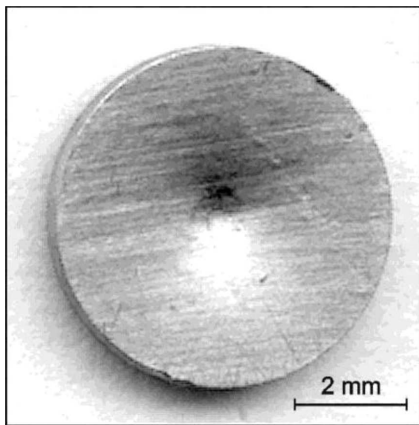


Fig. 5. The temperature of -196°C – concave punched surface of mini disc

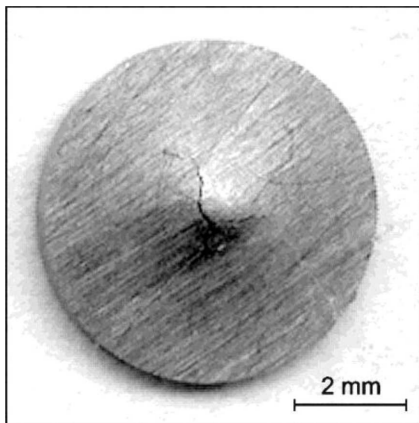


Fig. 6. The temperature of -196°C – convex bulged surface of mini disc

Different modes of fracture observed in mini discs after testing at the two extreme temperatures, i.e. -196°C and

+20°C, are presented on photographs. At temperatures close to the temperature of liquid nitrogen, even a small deformation causes cracking of the disc (Figs. 5 and 6). As the temperature increases, cracks on the mini disc surface occur only when a large plastic deformation is being reached (Figs. 7 and 8).

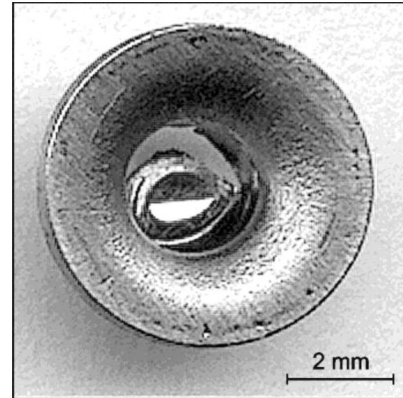


Fig. 7. The temperature of +20°C – concave punched surface of mini disc

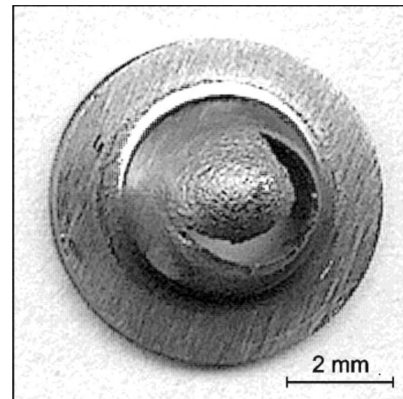


Fig. 8. The temperature of +20°C – convex bulged surface of mini disc

4. Discussion of results

On the approximation curves shown in Figures 1 and 4, there are ranges of sudden transition of material into brittle state, accompanied by changes in convexity on both curves combined with the occurrence of inflection points. It has been assumed that the values of temperature corresponding to the inflection points are mutually equivalent for both methods and characterise the steel susceptibility to cracking. The coefficients of regression for functions (1) and (2) and the corresponding statistical parameters are given in Tables 1 and 2.

TABLE 1
The values of the coefficients in equation (1) and of the statistical parameters R^2 , F and σ_{st}

The coefficients for steel:	a	b	c	d	R^2	F	σ_{st}
	J/cm ²	J/cm ²	°C	°C	-	-	J/cm ²
before revitalisation	5,295	181,332	116,432	32,967	0,999	1403,2	3,349
after revitalisation	4,925	187,027	1,719	31,334	0,998	1134,0	4,186

TABLE 2

The values of the coefficients in equation (2) and of the statistical parameters R^2 , F and σ_{st}

The coefficients for steel:	a	b	c	d	g	R²	F	σ_{st}
	[J]	[J]	[°C]	[°C]	-	-	-	[J]
before revitalisation	0,321	1,637	-64,903	11,816	17,421	0,984	60,27	0,129
after revitalisation	0.000	2,544	-123,404	11,206	28,108	0,986	116,39	0,122

TABLE 3

The values of the temperatures T_{pk} and T_{nuc} corresponding to the points of inflection

Material	Impact toughness		SPT	
	before revitalisation	after revitalisation	before revitalisation	after revitalisation
Temperature of inflection points	$T_{pk} = 116,4^{\circ}\text{C}$	$T_{pk} = 1,71^{\circ}\text{C}$	$T_{nuc} = -100,5^{\circ}\text{C}$	$T_{nuc} = -161,9^{\circ}\text{C}$

To verify the correctness of the selected mathematical model, the correlation coefficient R^2 , Fisher-Snedecor factor F for $\alpha = 0.01$ and the standard error σ_{st} were calculated.

The level of statistical parameters confirms the correctness of the selected model. The values of inflection points corresponding to the temperature T_{pk} in equation (1) for the impact test, and to the temperature T_{nuc} in equation (2) for the SPT method are given in Table 3.

Studies conducted by both methods clearly indicate that revitalising heat treatment significantly improves the properties of the 14MoV63 steel. The ductile-brittle transition temperature T_{pk} calculated by the inflection point method for the steel after long-term operation and after revitalising treatment amounts to 116.4°C and 1.71°C , respectively. Similar relationships were also observed using the SPT method. For the steel after long-term operation, the temperature of sudden loss of ductility T_{nuc} is -100.5°C , while for the steel revitalised the value of T_{nuc} is shifted by about 61°C towards the lower level and amounts to -161.9°C . In both cases, shifting of T_{pk} and T_{nuc} towards lower level indicates a favourable impact of revitalisation on the mechanical properties of the investigated material.

It can be assumed that there is a correlation between the temperature of sudden loss of ductility and the ductile-brittle transition temperature; the lower is the temperature of sudden loss of ductility, the lower is the ductile-brittle transition temperature obtained in conventional trials. It appears that the SPT method can be used in comparative studies of the metallic materials, including an estimation of their sensitivity to brittle crack formation.

REFERENCES

[1] A. Shekhter, S. Kim, D.G. Carr, A.B.L. Croker, S.P. Ringer, Assessment of temper embrittlement in an ex-service 1Cr-1Mo-0.25V power generating rotor by Charpy V-Notch testing, K_{IC} fracture toughness and small punch test, International Journal of Pressure Vessels and Piping 79, (2002).

[2] G.C. Stratford, F.DI Persio, J.Kłaput, Miniaturised creep testing using the small punch test technique, Turin Italy 2005.
 [3] J.Kłaput, Niekonwencjonalne sposoby badania właściwości mechanicznych materiałów metalowych Dozór Techniczny nr 2, 2003.
 [4] T. Linse, M. Kuna, J. Schuhknecht, H.W. Viehrig, Usage of small punch test for the characterization of reactor vessel steels in the brittle-ductile transition region, Engineering Fracture Mechanics 75, 3520-3533 (2008).
 [5] Kaishu Guan, Li Hua, Qiongqi Wang, Xiaohui Zou, Ming Song, Assessment of toughness in long term service CrMo low alloy steel by fracture toughness and small punch test, Nuclear Engineering and Design 241, 5, May, 1407-1413 (2011).
 [6] D.W. Mercaldi, Surface sampling Device, US Patent 4,845,896, Failure Analysis Associates, Inc., Menlo Park, California, 1989.
 [7] I. Klevtsov, A. Dedov, A. Molodtsov, Using of small punch test for determination of tensile properties for power plant steels 6th International DAAAM Baltic Conference Industrial Engineering, 24-26 April 2008, Tallinn, Estonia.
 [8] W. Volk, Statystyka stosowana dla inżynierów, WNT Warszawa 1973.
 [9] M.A. Contreras, C. Rodriguez, F.J. Belzunce, C. Betegon, Use of the small punch test to determine the ductile-to-brittle transition temperature of structural steels, Fatigue & Fracture of Engineering Materials & Structures, Blackwell Publishing 31, 2008.
 [10] E. Cárdenas, F.J. Belzunce, C. Rodríguez, I. Peñuelas, C. Betegón, Application of the small punch test to determine the fracture toughness of metallic materials, Fatigue & Fracture of Engineering Materials & Structures 35, 5, 441-450, May 2012.
 [11] J. Kłaput, Opracowanie niekonwencjonalnej metody badawczej dotyczącej oceny odporności na pękanie materiałów dla energetyki – praca dla ZREW S.A., Warszawa 2001 – praca niepublikowana.
 [12] J. Kłaput, Porównawcza ocena odporności na pękanie materiału pochodzącego z elektrociepłowni Żerań – opracowanie na zlecenie ZREW Warszawa S.A. 2001 – praca niepublikowana.
 [13] Y.Q. Wang, X.Y. Liu, Z.W. Hu, Y.J. Shi, Experimental study on mechanical properties and fracture toughness of structural thick plate and its butt weld along thickness and at low temperatures, Fatigue & Fracture of Engineering Materials and Structures 36, December 2013 – Wiley Online Library.