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DEVELOPMENT OF A TEST DEVICE FOR TESTING MINIATURE SAMPLES BY THE SPT METHOD

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The paper presents the results of the development and manufacture, as well as assembly and installation of a test device for testing miniature samples by the SPT method. The results of mechanical tests by tensile and SPT methods of 35X steel material after heat treatment (quenching + tempering) at different tempering temperatures. The evaluation of changes in the strength characteristics of steel grade 35X, depending on the heat treatment mode. The correlation equations between the *t/10* method and the yield strength of each 35X steel sample obtained during tensile tests were determined and obtained.

Keywords: *SPT tests, development, tensile tests, yield, strength.*

INTRODUCTION

The bending test of miniature disks, introduced in the 1980s by Manahan, was intended as an alternative characterization method for obtaining the mechanical properties of irradiated steels from nuclear reactor vessels [1, 2]. The novelty of this method was its miniaturized geometry, allowing the use of small disks for microstructural analysis in transmission electron microscopy. The main advantage of miniature disks was the short duration of sample irradiation and the possibility of obtaining a larger number of samples from a small volume of test material.

Data on the mechanical properties of materials of structural elements are important for design, integrity assessment and remaining service life. In this regard, small sample test methods (hereinafter referred to as SSTM) are preferable to typical tests in two cases:

1) when a volume of material is limited;

2) when it is difficult to work with a huge volume of material, for example, if the material is radioactive.

The SSTM are more promising for assessing the integrity and residual life of components during their operation, as they are easy to perform and are non-destructive in nature. Of the available SSTMs, the Small Punch Test (hereinafter referred to as SPT) method is mainly used to determine the mechanical properties of thin samples. The sample size required for characterization is similar to that required for transmission electron microscopy (TEM), i.e. ~3 mm in diameter [2]. This method was primarily developed to study post-radiation effects at nuclear power plants [2–7]. Nowadays, this method has been extended to studies of other mechanical properties: Young's modulus [8, 9], ultimate strength [10, 11], and ultimate tensile strength [12], transition temperature from plastic to brittle state [13], fracture properties [14] and creep behavior [15]. In 2006, *The CEN Code of Practice* was introduced and revised in 2007 to standardize the SPT method [16].

The main advantages of this methods are:

- use as a non-destructive testing method, since the production of samples from metal of the operated equipment does not reduce its performance due to its diminutive size (thickness less than 1 mm);
- the ability to study such objects as elements of welded joints, parts of small sizes or complex shapes, parts made of materials that are unreasonable for tensile testing due to their high brittleness, destroyed parts from which full-size macrosamples cannot be cut in accordance with GOSTs, etc.

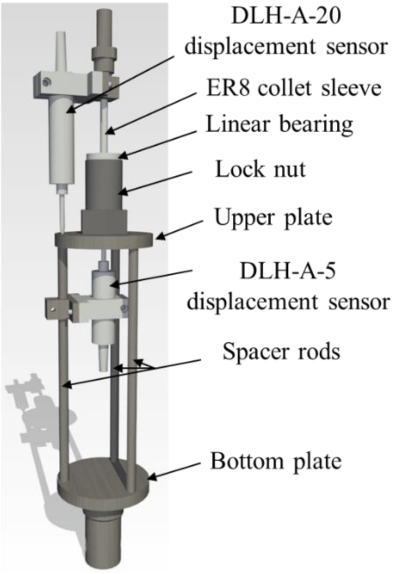
Compared to non-destructive testing methods such as ultrasonic, magnetic and X-ray flaw detection, SPT is a direct measurement method, which allows obtaining information about the mechanical properties of the material directly, while other methods are based on evaluation of indirect indicators.

Thus, to date, development and implementation of a new method for testing miniature samples for indentation will allow evaluating the strength and plastic characteristics of highly radioactive materials and other materials without requiring the manufacture of massive standard samples.

DESIGN AND MANUFACTURE OF A TEST DEVICE. ASSEMBLY AND INSTALLATION OF THE TEST DEVICE

The test device consists of a frame consisting of top and bottom plates, three spacer rods (Figure 1). The top plate houses the loading jig and the bottom plate is rigidly connected to an Instron 5966 testing machine equipped with a 0.5 kN load sensor using a 1/2 inch pin and lock nut.

The loading jig for testing miniature samples by the SPT method was designed in accordance with the recommendations [16–17], and consists of an upper and lower die, a rod and a ball (Figure 2). The dies are made of 30X13 hardened chromium steel with a hardness of 50 HRC. The ball (∅ 2.5 mm) and the rod (∅ 2.5 mm) are made of materials with high hardness and modulus of elasticity – zirconia ZrO_2 and tungsten carbide WC, respectively. Combination of the rod and ball forms a punch that passes through the sample, held by the upper and lower dies.



a)

b)

Figure 1. 3D model (a) and outer view of the test device, mounted on a test machine (b)

Some authors use a solid punch with a hemispherical tip instead of a separate ball and rod (punch), arguing that it is relatively easy to simulate deformation processes. However, in our opinion, manufacture of a solid tip from hard materials is costly and it is impossible to provide the required sphericity on it.

The punch rod is connected with a precision ER8 collet sleeve to a larger diameter rod (∅ 6 mm). To ensure coaxial translational motion of the rod with the punch, the closed LMF6UU linear rolling bearing is installed on the lock nut. Thus, the lock nut not only fixates the test sample between the upper and lower dies, but also guides the translational motion of the punch.

One of the features of the testing device is a semi-rigid connection of the punch to the lock nut with one degree of freedom, rather than a rigid connection to the traverse of the testing machine. This combination does not require the alignment of the punch motion relative to the sample after disassembly/assembly of the loading jig.

The test device additionally contains two LVDT transformer sensors (DLH-A-20 and DLH-A-5, Dacell), operating on a bridge connection, one of which is used to continuously record the movement of the punch relative to the top plate, and the second registers the axial extrusion of the sample from the side of the lower die (Figure 3).

a) DLH-A-5

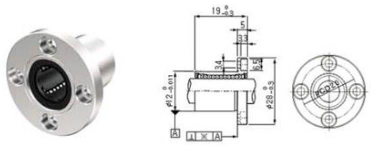
b) DLH-A-20

Figure 3. Outer view of linear displacement sensors

DESIGN AND ASSEMBLY OF INFORMATION AND MEASURING SYSTEM

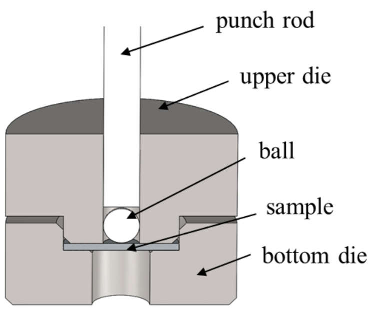
To ensure continuous data collection during testing, an external measuring system was designed and assembled on the testing machine, which allows recording data simultaneously from four different primary transducers (Figure 4).

The measuring system consists of primary converters, analog-to-digital converters (ADC), and a personal computer with software. Analog-to-digital converters and power supplies are placed in a measuring unit with a metal case. The primary transducers are connected to the unit via DE-9 connectors. Data output from the measuring unit is performed using the RS232-USB converter to a portable computer (laptop). The received data is displayed in a software window created in the Trace Mode 6 environment and written to a tabular XLS file.

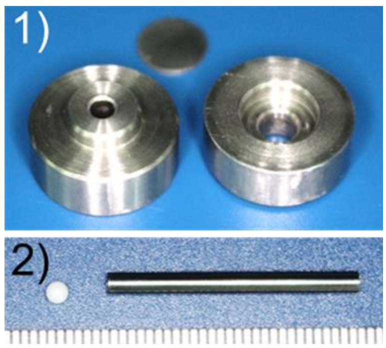


a) LMF-UU linear bearing

b) ER8 collet sleeve



c) load unit section sketch



d) the upper and lower dies, test sample ∅ 8 mm (1); ceramic ball ∅ 2.5 mm and tungsten carbide rod (2)

Figure 2. Sketches and pictures of the components of the loading jig

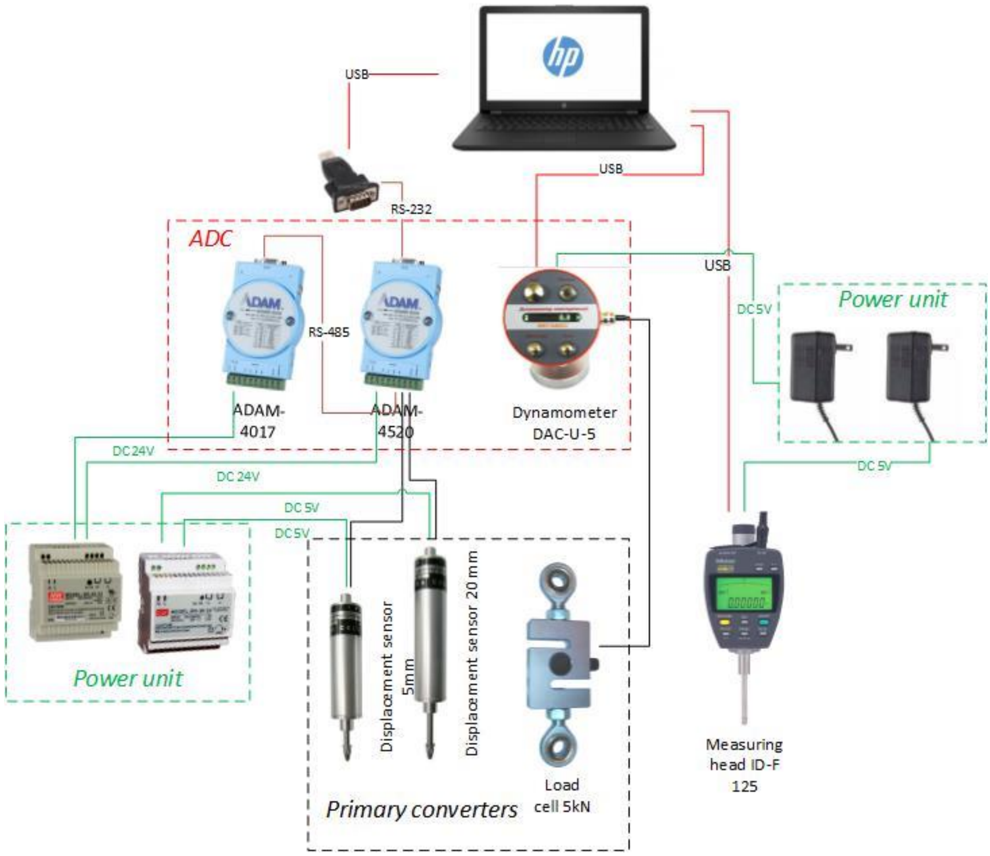
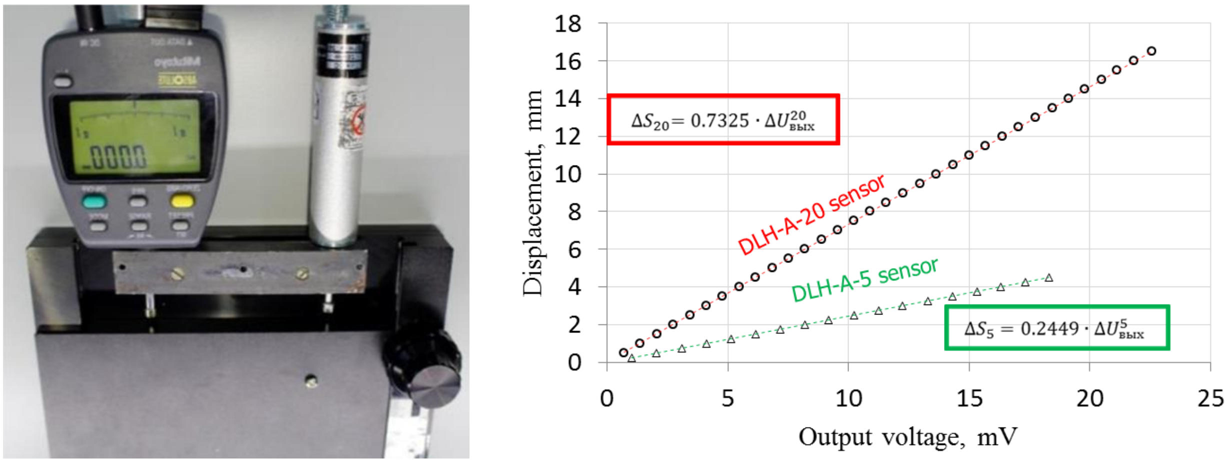


Figure 4. Schematic diagram of information and measuring system connection



a) device for calibration of linear displacement sensors

b) dependencies of output voltage on linear displacement of the rod of DLH-A-20 and DLH-A-5 sensors

Figure 5. Calibration of linear displacement sensors

CALIBRATION OF LINEAR DISPLACEMENT SENSORS

To calibrate linear displacement sensors, a high-precision digital indicator Mitutoyo ID-F 25 with a measurement resolution of 0.001 mm in the range of 0–25 mm was used. To calibrate the linear displacement sensors, each sensor with a digital indicator was rigidly fixed horizontally to the surface of a two-axis microscope object table, which had a coaxial feed with a microscrew (Figure 5, a). Then, with a resolution of 0.5 mm and 0.25 mm, the output voltage readings were taken from the DLH-A-20 and DLH-A-5sensors, respectively. Figure 5, b shows the dependence of linear displacement on the value of the output voltage for sensors with different measurement limits.

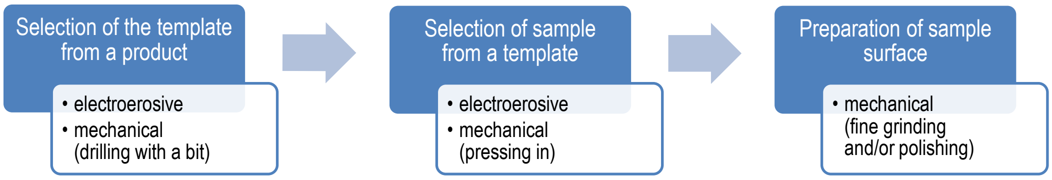


Figure 6. Stages of selection and preparation of miniature samples

Based on calibration measurements, the linear dependences of the displacement (ΔS_L) of the rod for each sensor on their output voltage were determined. The equation of linear dependencies for each sensor is as follows:

$$\Delta S_5 = 0.2449 \cdot \Delta U_{OUT}^{5} \text{ – for DLH-A-5 sensor,} \quad (1)$$

$$\Delta S_{20} = 0.2449 \cdot \Delta U_{OUT}^{20} \text{ – for DLH-A-20 sensor,} \quad (2)$$

where, ΔS_L – linear movement of the sensor rod, mm (L – sensor measurement limit); ΔU_{OUT}^{L} – output voltage, mV (L – sensor measurement limit).

RESEARCH METHOD

Method of Miniature Sample Manufacture

The procedure for selecting and preparing miniature test samples consists of three main stages (Figure 6).

To select the template and the sample, a wire-cutting method is used on an ARTA 123 PRO CNC electro-erosive machine. Surface is prepared by mechanical fine grinding with a final abrasive grit P1200.

Method for Identifying Correlation Dependence by the t/10 Method

To identify the correlation dependence, the method of correlation between the yield strength σ_y and the yield load P_y was applied with the following empirical equation [10]:

$$\sigma_y = \alpha_1 \cdot \frac{P_y}{t^2} + \alpha_2 , \quad (3)$$

where, t – sample thickness, a_1 and a_2 – correlation factors that are obtained from regression analysis of test results of different materials that should be correlated.

Currently, there are several different methods for obtaining a value of the yield strength P_y from the SPT curve [10, 16, 18], one of the most accurate is the *t/10* method [19].

According to the *t/10* method, the yield load P_y is obtained in a similar way to the method for determining the yield strength σ_y (displacement: 0.2%) in standard tensile tests. A parallel line tangent to the elastic zone of the SPT curve is drawn with a shift equal to t/10 along the shift axis. The intersection of this line with the SPT curve is defined as the yield load P_y (Figure 7).

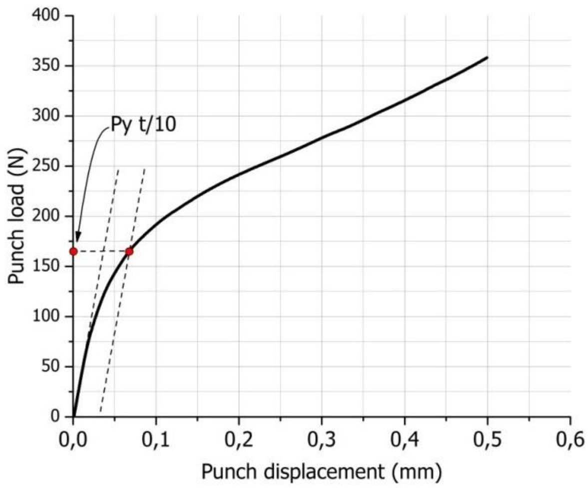


Figure 7. Calculating P_y with the t/10 Method from the SPT Curve

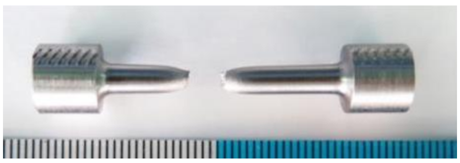
RESULTS AND DISCUSSION

Results of Tensile Test by Conventional Method

The sample material was 35X low-alloy steel with various heat treatment to obtain a wide range of strength and plastic characteristics. Heat treatment of the samples was performed by quenching (at a temperature of 860 °C) and subsequent tempering at various temperatures of 200 °C, 300 °C, 400 °C, 500 °C.

For testing, standard cylindrical samples of type III, No. 8 according to GOST 1497-84 were prepared from heat-treated blanks (Figure 8). Determination of the actual mechanical properties of heat-treated samples were performed according to the conventional tensile test method at a constant strain rate of 0.0025 s^{-1} according to GOST 1497 (Figure 9).

a)



b)

Figure 8. Outer view of standard 35X steel samples before (a) and after (b) tensile testing

The results of determining the strength and plastic characteristics are presented in the Table.

Table. The results of tensile tests of 35X steel samples

Sample #	Tempering temperature, °C	Temporary resistance σ_v, MPa	Conventional yield strength $\sigma_{0.2}$, MPa	Relative elongation after rupture δ_5, %	Relative contraction after rupture ψ, %
35X-1.1	without HT	764	444	14	60
35X-1.2		771	447	17	56
Mean value		**768**	**446**	**15**	**58**
35X-2-1	200	1393	1251	10	44
35X-2-2		1419	1282	11	48
Mean value		**1406**	**1267**	**10**	**46**
35X-4-1	300	1186	1090	13	53
35X-4-2		1162	1067	13	50
Mean value		**1174**	**1079**	**13**	**51**
35X-6-1	400	1056	946	14	56
35X-6-2		1091	980	15	56
Mean value		**1074**	**963**	**14**	**56**
35X-7-1	500	758	606	23	69

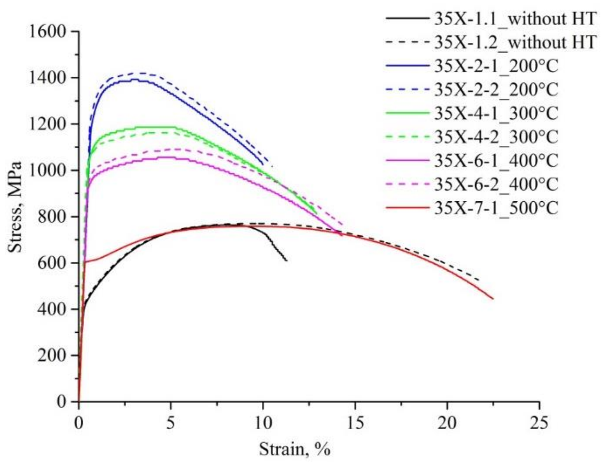
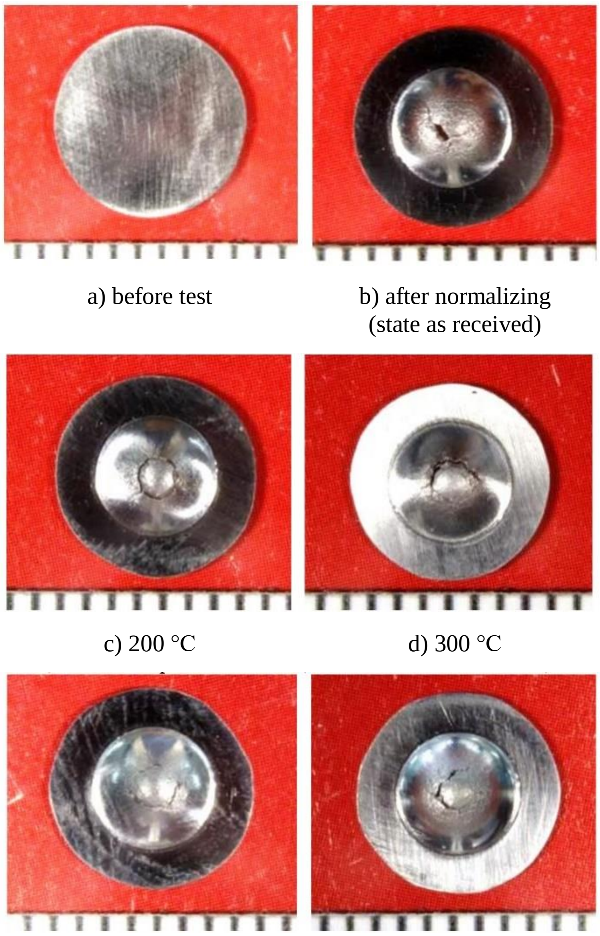


Figure 9. Tensile diagram of 35X steel before and after heat treatment (quenching + tempering) at different tempering temperatures

Results of testing miniature samples by the SPT method

For testing according to the SPT method, disk-shaped miniature samples with dimensions of ∅ 8×0.5 mm were made from a head of the destroyed samples (Figure 10). Using the Instron 5966 test machine with designed test device, miniature samples were tested by the SPT method (Figure 11). Miniature samples were tested by the SPT method with a punch loading speed of 0.2 mm/min and constant recording of the load-displacement curve data.



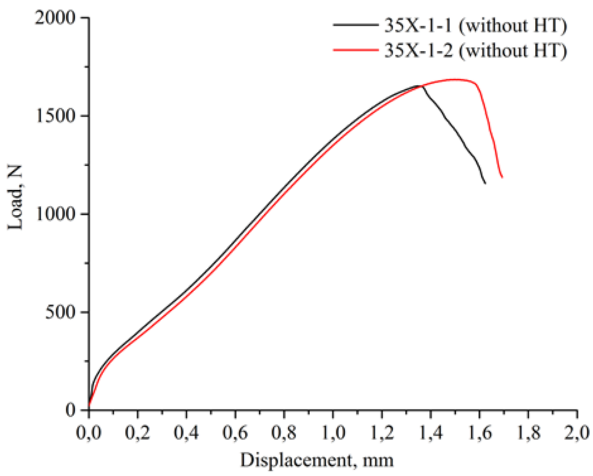
a) before test b) after normalizing (state as received)

c) 200 °C d) 300 °C

e) 400 °C f) 500 °C

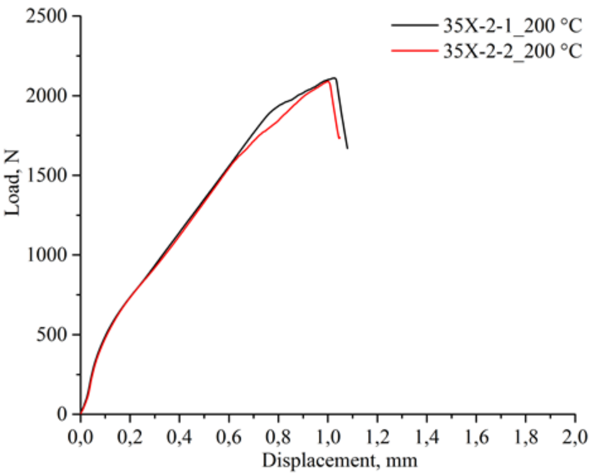
Figure 10. Outer view of miniature 35X steel samples before and after testing by the SPT method

As the SPT load-displacement curves show, the designed test device shows good repeatability of results.

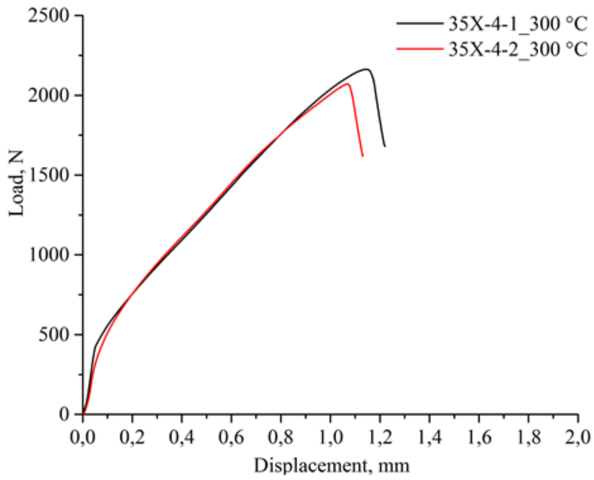


a) after normalizing (state as received)

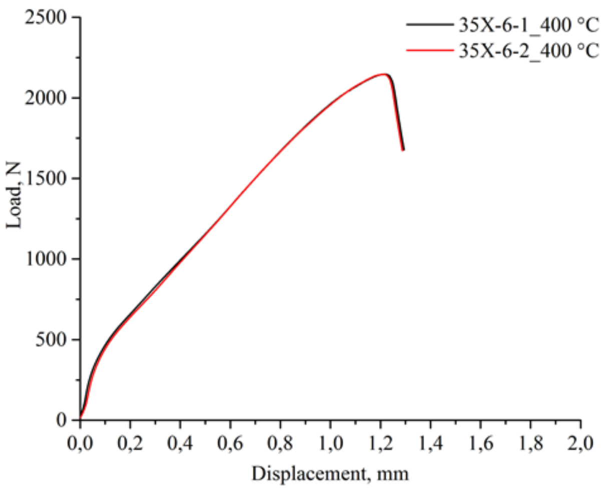
Figure 11. Load-displacement curves of SPT testing of 35X steel after heat treatment



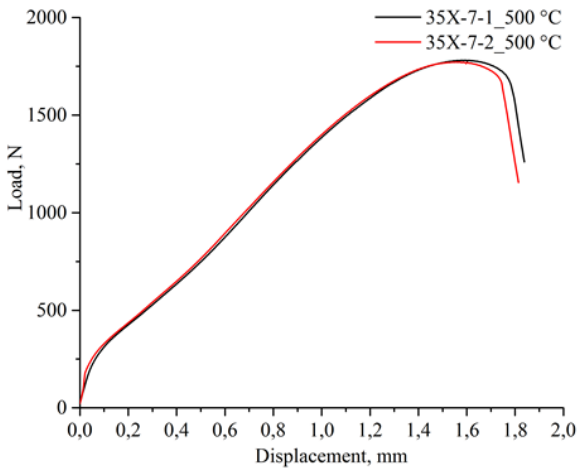
b) after quenching and tempering at 200 °C



c) after quenching and tempering at 300 °C



d) after quenching and tempering at 400 °C



e) after quenching and tempering at 500 °C

Figure 11 (continued). Load-displacement curves of SPT testing of 35X steel after heat treatment

ANALYSIS AND GENERALIZATION OF RESEARCH RESULTS

Based on the results of testing miniature samples made of heat-treated 35X steel, typical zones of elastic, elastic-plastic and plastic deformation were determined. After analyzing the load-displacement curves, the P_y/t^2 values were determined by the *t/10* method, which is similar to the method for determining the yield strength $\sigma_{0.2}$ (0.2% displacement) in standard tensile tests. Between the actual values of the yield strength $\sigma_{0.2}$ (tensile test) of the 35X steel material and the value of P_y/t^2 (SPT method), the following dependency was established (equation 1) with a high level of correlation ($R^2 = 0.99$) (Figure 12).

$$\sigma_y = 0.5482 \cdot \frac{P_y}{t^2} - 158.79 . \quad (4)$$

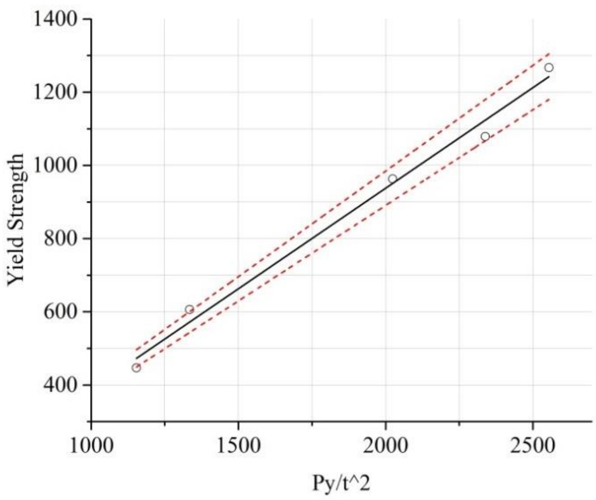


Figure 12. Correlation between the yield strength and the value of $P_{y/t2}$ according to the SPT method for the 35X steel material

CONCLUSION

As part of this work, the following activities have been conducted:

− The test device for testing miniature samples using the SPT (Small Punch Test) method has been designed and manufactured.

− The procedure for manufacturing miniature samples according to the requirements of the methodology has been tested. A series of methodical tests by the SPT method of samples made of heat-treated 35X steel with strength characteristics in a wide range has been performed.

− The obtained results of methodical tests were analyzed and a linear relationship was established between the actual values of the yield strength $\sigma_{0.2}$ (tensile test) of the 35X steel material and the value of P_y/t^2 (SPT method).

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SPT ӘДІСІ БОЙЫНША ШАҒЫН ҮЛГІЛЕРДІ СЫНАУҒА АРНАЛҒАН СЫНАҚ ҚҰРЫЛҒЫСЫН ӘЗІРЛЕУ

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Жұмыста шағын үлгілерді SPT әдісімен сынау үшін сынақ құрылғысын әзірлеу және дайындау, сондай-ақ құрастыру нәтижелері келтірілген. Әр түрлі температурада термиялық өңдеуден кейін (шыңдау+жұмсарту) 35Х болат материалын созу және SPT әдістерімен механикалық сынау нәтижелері көрсетілген. Термиялық өңдеу режиміне байланысты 35Х маркалы болаттың беріктік сипаттамаларының өзгеруін бағалау жүргізілді. *t/10* әдісі мен созылу сынақтары кезінде алынған әрбір 35Х болат үлгісінің аққыштық шегі арасындағы корреляциялық теңдеулер анықталды және алынды.

Түйін сөздер: *SPT әдісі, әзірлеу, созу сынағы, аққыштық, беріктілік.*

РАЗРАБОТКА ИСПЫТАТЕЛЬНОГО УСТРОЙСТВА ДЛЯ ПРОВЕДЕНИЯ ИСПЫТАНИЯ МИНИАТЮРНЫХ ОБРАЗЦОВ МЕТОДОМ SPT

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В работе приведены результаты разработки и изготовления, а также сборки и монтажа испытательного устройства для проведения испытания миниатюрных образцов методом SPT. Приведены результаты механических испытаний методами на растяжение и SPT материала стали 35Х после термообработки (закалка+отпуск) при различных температурах отпуска. Выполнена оценка изменений прочностных характеристик стали марки 35Х в зависимости от режима термообработки. Определены и получены корреляционные уравнения между методом *t/10* и пределами текучести каждого образца стали 35Х, полученными при испытаниях на растяжение.

Ключевые слова: *метод SPT, разработка, испытания на растяжение, текучесть, прочность.*