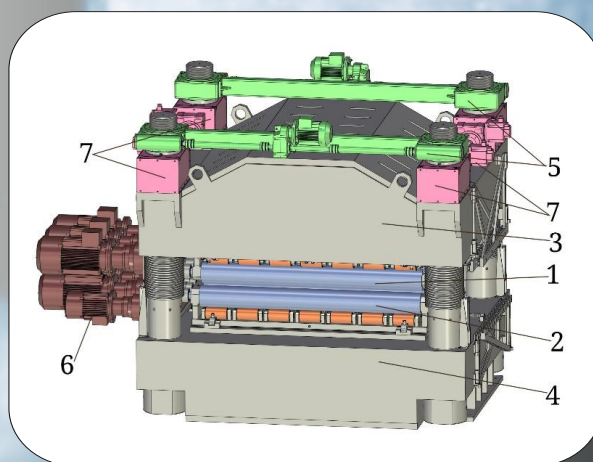


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MATERIALS

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Roll leveler for straightening sheet blanks

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Abstract: The report presents the design of a 13-roll leveler for straightening sheet blanks of complex configuration. The roll leveler is equipped with a mechanism for controlling the roll gap, which during the levelling process compensates for the elastic deformation of the housing and maintains the specified levelling conditions. The levelling process was analyzed using the finite element method (FEM) with the determination of forces acting on the rollers during levelling of sheet blanks up to 50 mm thick.

KEYWORDS: LEVELLING PROCESS, LEVELLING FORCE, MOTOR POWER

1. Introduction

Sheet blanks are widely used in industrial production. For their cutting, laser and plasma cutting is often used. After that, sheet blanks often have insufficient geometric accuracy (flatness). To improve the geometric accuracy of sheet blanks various methods can be used, including roller levelling.

Roller levelling is a technological process of metal forming, used to minimize the flatness of sheet blanks and reduce the level of residual stresses. When levelling, the workpiece is subjected to cyclic alternating bending with decreasing amplitude. In this case, the penetration depth of deformation should be at least 0.7 of the thickness of the workpiece [1].

The existing experience of the world's leading manufacturers of process equipment (ARKU, KOHLER, etc.) shows the feasibility of using multi-roll levelers both for straightening piece sheet parts and for integrating levelers into existing and planned automated lines for the production of piece sheet blanks of a wide range for machine-building, petrochemical, aerospace and other branches of industrial production.

Multi-roll levelers are used for cold straightening of steel sheet blanks and blanks of non-ferrous alloys up to 50 mm thick and up to 5000 mm wide. Hot straightening of steel sheet blanks is carried out at temperatures of 500-700 °C, mainly immediately after leaving the working stand of the rolling mill. Hot straightening cannot provide a sufficiently even surface of the blanks, since they undergo buckling during the cooling process. Sheets with a thickness of over 40 - 50 mm are usually straightened in a cold state under presses.

At present, there is a demand for versatile and productive equipment for levelling sheet blanks with complex contours. These requirements are met by multi-roll levelers.

2. Components of the roll leveler

Roll leveler mainly composed of 6 parts (fig. 1):

- Upper and lower roll frames. They include upper and lower leveling rolls, which are used for cyclic alternating bending of sheet blanks. The roll frames also contain back up rolls which are designed to prevent excessive bending of the leveling rolls under operating loads.
- Upper and lower frames.
- Mechanisms for adjusting the inlet and outlet inter-roll gap. Used for the initial setting of the roll leveler for straightening blanks of a certain thickness.
- Drive unit. The leveling rolls are driven by a drive unit. It consists of electric motors and reducers.
- Compensation mechanisms. Serve to compensate for the elastic deformation of the elements of the roll leveler under the working load.
- Mechanical means for moving blanks.

The roll leveler is designed for straightening blanks with a thickness of 6 to 50 mm with a maximum width of up to 2000 mm. It is equipped with 13 leveling rolls with a diameter of 200 mm and 15 back up rolls. The distance between rolls is 205 mm (Fig. 2). In the roll leveler, a roll arrangement is used, in which the upper frame of rolls is located at an angle to the lower one, which ensures cyclic

bending with decreasing amplitude of the billet. The levelling speed is in the range from 3 to 12 m/min.

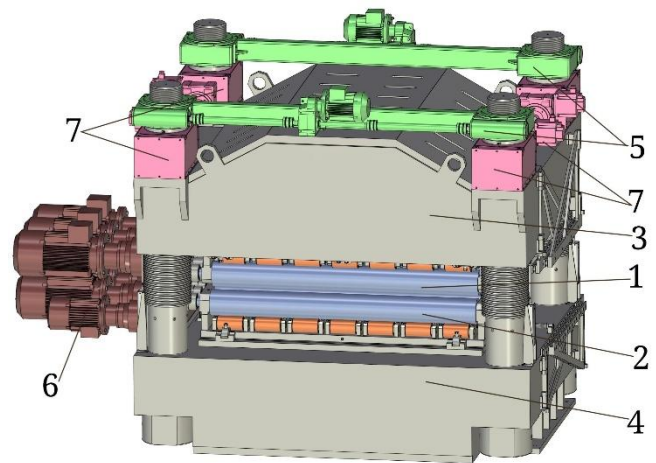


Fig. 1. Components of the roll leveler

1 – upper roll frame; 2 – lower roll frame; 3 – upper frame; 4 – lower frame; 5 – mechanisms for adjusting inlet and outlet inter-roll gap; 6 – drive unit; 7 – compensation mechanisms

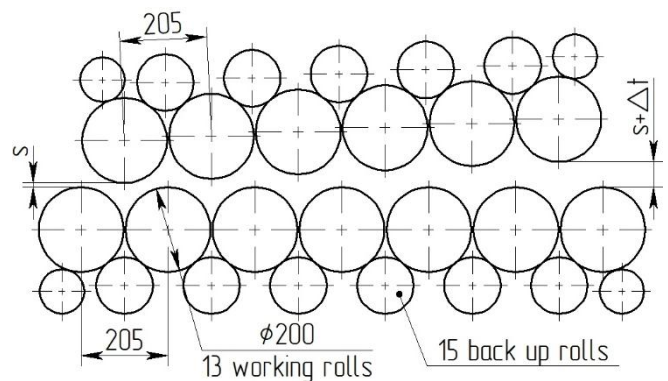


Fig. 2. Roll arrangement

s – inlet inter-roll gap; s+Δt – outlet inter-roll gap

Using the finite element method, the straightening force of a sheet blank 50 mm thick and 650 mm wide was determined. The following characteristics of the material were used: Young's modulus 210000 MPa, Poisson's ratio 0.3, density 7850 kg/m³, yield strength $\sigma_t=390$ MPa, tensile strength 530 MPa, relative elongation $\delta=19$ %. With these parameters, the force on the most loaded leveling roll reaches 5 MN, the total force during leveling is 30 MN.

The total power of the drive unit is 115 kW. Electric motors with a total capacity of 15 kW are also installed to adjust the roll leveler (moving the upper frame and operation of the compensation mechanisms).

When leveling under the influence of the working load, elastic deformation of the roll leveler structure occurs. Back up rolls are used to prevent leveling rolls from bending. Upper and lower frames have a cellular structure. Stretching and bending of the pillars of the roll leveler (leveler has 4 pillars with a diameter of 300 mm) connecting the upper and lower frames are controlled using four inductive sensors. During the straightening process, the deformation of the pillars is constantly monitored and, based on these data, the roll gap is corrected using a compensation mechanism. The maximum elastic deformation of the pillars is 4 mm.

3. Conclusion

When developing sheet-straightening mills, it is necessary to take into account many phenomena occurring in metal subjected to alternating loading with decreasing amplitude (interaction of elastic

and plastic deformation, the Bauschinger effect, etc.). In this process, there are conditions that make straightening difficult (hardening) and facilitating (Bauschinger effect). All this can give rather large differences in the results of calculations performed by different methods. For various materials over the entire range of thicknesses, setting up the leveling rolls can vary significantly. Therefore, for any leveler, it is required to create a correct computer model, taking into account as many necessary parameters as possible.

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Development and implementation of closed loop position control concepts on electromechanical linear system using Beckhoff controller

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Abstract: Electromechanical linear axes are used as actuators in many areas of industrial automation. Such axes are a fundamental building block of any machine tool, manipulator or similar device, whether they implement only one- or multi-axis positioning. Usually, servomotors or stepper motors are used to drive the axes, and the control is performed by a variety of PLC controllers and controller concepts. The paper presents the implementation of a multi-purpose electromechanical linear axis, which enables the study, research and comparison of various linear drive concepts and types of closed loop controllers. The linear axis incorporates two different motor types: a BLDC servomotor as well as a stepper electric motor, and thus also enables comparison of these two motor types and corresponding control concepts. A multi-core Beckhoff soft PLC is used as a controller, which can implement either built-in closed loop controllers or can also implement controllers designed in the Matlab/Simulink environment.

PID closed loop position controller built-in Beckhoff TwinCAT 3 is presented in the paper. Further on, it is compared by PID closed loop controller developed in Matlab/Simulink which was exported and imported into Beckhoff TwinCAT 3 environment in order to confirm the adequacy of importing and using Matlab/Simulink controllers on Beckhoff soft PLCs.

Keywords: ELECTROMECHANICAL LINEAR AXIS, SERVOMOTOR, STEPPER MOTOR, CLOSED-LOOP CONTROL, SOFT-PLC

1. Introduction

The paper presents the planning and implementation of linear axis control with the aim of using our own developed position controller. This allows us to use any position controller to control the system. The system does not allow this by using the standard controllers, which are part of the Beckhoff TwinCAT 3 software environment, as it uses a specific program block for absolute axis travel, which performs motor rotation according to the specified parameters.

The main advantage of standard TwinCAT 3 controllers (program blocks) is that they are as easy as possible to use in industry, as the user does not need to design its own controller, but only select one of the controllers available in the TwinCAT 3 software and set individual parameters. For simple operations, the PID controller is the most commonly used, which is usually sufficient to control industrial applications. The aim of the research was to replace the standard TwinCAT 3 PID controller with custom closed-loop controller developed in Matlab/Simulink and confirm the adequacy of importing and using Matlab/Simulink controllers on Beckhoff soft PLCs.

For the development and implementation of our own closed loop controllers, we first had to build a demonstration system and get familiar with the soft PLC environment. Once we had the system built and developed industry standard closed-loop control of the axis using TwinCAT 3 program blocks, we were able to start creating our own closed-loop controllers using the Matlab/Simulink environment. In order to confirm the consistency of the proposed method, we had compared the functionality of the standard PID closed loop controller in TwinCAT 3 and the functionality of the same controller developed in Matlab/Simulink that was imported into TwinCAT 3 system. As presented in this paper, we have achieved almost identical results, which confirmed the possibility to develop new types of different non-linear controllers in Matlab/Simulink and incorporate them in industrial applications using Beckhoff soft PLCs.

2. System overview

For the purpose of research, it was first necessary to design a linear positioning unit with a linear axis. The design is based on Festo DGE-25 linear axis, which converts rotational motion into linear motion with the help of a toothed belt and is designed for medium loads. Such linear systems are commonly used for transporting, lifting, manipulating and performing feeding movements.

To drive the linear axis, two different Beckhoff motors are mounted on the drive. One is Beckhoff AS1050-0120 stepper motor

and the other is Beckhoff AM8113-0F20 BLDC servo motor, which has an integrated 18-bit absolute incremental encoder. The motors are connected via the shafts to the pulleys of the linear module that drive the linear axis by means of a toothed belt, which allows fast and dynamic movement of the linear axis. A track guide with a ball-bearing carriage is also attached to the linear axis. The carriage is used for mounting weights, which allows the use of ten smaller weights of 500 g each. Weights can be removed or added as needed, and depending on the weight of the load, the dynamics of the system also changes. It should be noted that both motors never drive the linear axis at the same time, but they can be connected and disconnected via the shaft coupling. The appearance of the linear positioning unit with installed motors and weights is shown in Figure 1.

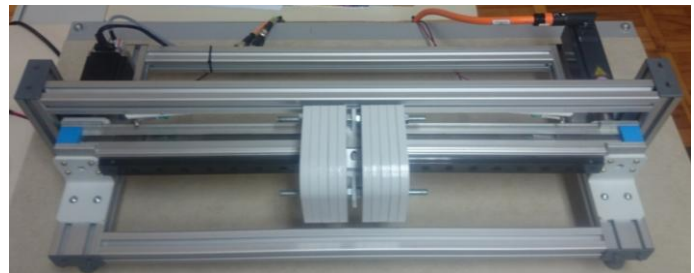


Fig. 1 Electro-mechanical linear axis with servo- and stepper-motor.

The advantage of the toothed belt is the cushioning of sudden shocks and changes to the system, so the dynamics of the system remain without major changes, as the rotational motion is transmitted directly to the belt. Thus, the system is more demanding to control, as we can achieve great external influences on the system.

The axis control system is designed using the TwinCAT 3 software environment, which enables the development and implementation of control programs for Beckhoff soft PLC controllers. In the TwinCAT 3 software environment, we can add various modules for motor and axis control, such as servo motor control and numerically controlled axis modules (NC axis module). NC axis module allows us to change the type of closed loop controller (PID and their combinations) and its parameters, scaling factors for the incremental encoder and other axis parameters.

In order to monitor the response of the system, the Scope function was used in TwinCAT 3, which allows us to monitor and record system data in very short sample times (e.g. 1 ms or 0,1 ms).

3. User interface

In order to create a more complex and sophisticated user interface, the user interface between the user and the TwinCAT 3 software environment (HMI) was created in Visual Studio C # Windows Forms Application, which is designed to build graphical applications. As shown in Figure 2, the user interface (on the left) allows us to select different types of controllers (P, PI, PD and PID), set the controller parameters and define the new set-point for our system. After enabling the motion, the response can be monitored and recorded on the right side of the screen by using *Scope* function that was implemented in the WinForms application.

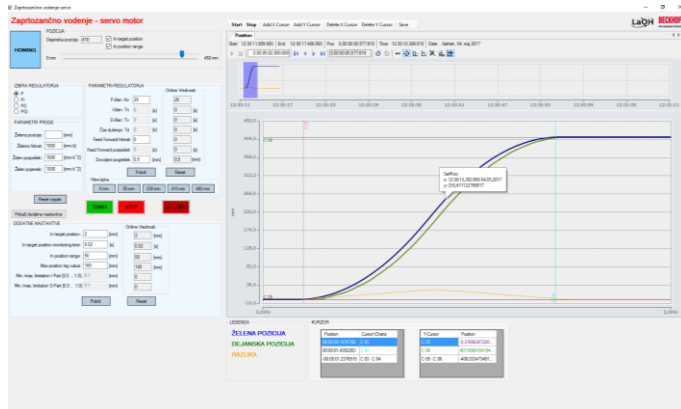


Fig. 2 User interface developed in Visual Studio C# as Windows Forms Application.

In order to access the real-time PLC variables (the program created in TwinCAT 3) from the WinForms user interface created Visual Studio C#, the Beckhoff ADS (Automation Device communication protocol) is used, which defines an interface of an independent device on an independent network. The TwinCAT 3 architecture allows individual software modules to be considered as stand-alone devices. For each task, the software uses a module that acts as a server or as a client. Messages between objects are exchanged through the ADS interface, more precisely in the message center, through which information is distributed across the system via a TCP/IP connection. In this way, all data and commands necessary for the operation of the program are transmitted. The ADS protocol is therefore a communication protocol in Beckhoff TwinCAT 3, developed for data exchange between different software modules, for example for communication between PLC and NC axis.

This protocol allows the use of other communication tools in the TwinCAT programming environment. If you need to communicate with another computer or device, the ADS protocol is used on top of TCP/IP. This means that all data in the network is accessible from any desired point and the user can connect to almost any connection method and connect to a device that changes the parameters [1].

To analyze the responses obtained from the real system, the TwinCAT 3 built-in function for plotting diagrams *Scope* is used. The *Scope* is used to plot the set-point motion line, the actual motion line and the deviation between these two values. The use of *Scope* also proves to be excellent for monitoring and observing responses at various controller parameters, as we can monitor the operation of the linear by using different controllers and controller settings in real time.

Scope settings were generated in the TwinCAT 3 programming environment and imported to the WinForms in Visual Studio C# with program code. In addition to *Scope* itself, we also added the possibility of creating X and Y cursors that measure the value of the difference between responses. We have also added a save function, which saves our response to a CSV file format that we can access at any time.

4. TwinCAT 3 PID controller

The system allows closed loop control of either the servo-motor or the stepper motor using different control concepts. Hereafter, the closed loop position control of the servomotor will be presented more in detail.

Servo motor control is performed by two control systems. One is implemented by a virtual controller and the other is implemented in the servo motor control module (Beckhoff EL7211). For the control system that is operated by a virtual controller, we can also select the controller type in the *Motion* (Axis – Ctrl tab) and set the control parameters in the same way as for a stepper motor.

The control system in the "EL7211" motor control module, which drives and controls the servomotor, is implemented in a cascade procedure. Figure 3 shows the control diagram, which represents the cascade control for the control of the AM8113 servo motor used. Cascade control in this case consists of three controllers. These are the position controller, the speed controller and the current controller.

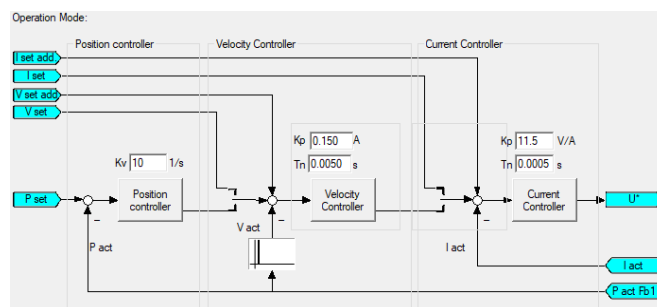


Fig. 3 TwinCAT 3 cascade closed-loop controller for servo-motor.

Complex system control often cannot be achieved with a single feedback loop. The reason for this is that complex systems are managed on the basis of only one information about the system and that is its output size. The controller reacts to changes that occur within the system only when their effect is expressed on the output of the output quantity. This usually results in a slow response to the control error [2]. Therefore, cascade control is often used in automation, where it is necessary to have fast and exact response of the system.

Commonly, the cascade control system consists of two control loops where the internal loop is slave or auxiliary. The outer loop is the superior or the main one. In such a cascade structure, the main controller does not act directly on the actuator, but forms a reference value for the slave controller. The effect of a fault in the auxiliary control loop is practically compensated in this loop [2].

The output of one controller therefore changes the set-point of the auxiliary controller. Cascade control loops can consist of two or more controllers. Each controller has its own variable that is measured in the process. Cascade control is typically used to isolate the primary system from interference, nonlinearity, and drive-related problems.

Cascade control brings improvement only when the dynamics of the internal control loop is fast enough compared to the external control loop. It does not make sense to use cascade control if the inner loop is not at least three times faster than the outer control loop. If the inner loop is not much faster than the outer one, there is a great chance of interaction between the two loops that can cause instability [3].

In the scope of this research we have concentrated only on the external loop of our cascade controller, that is the PID closed loop position controller of the servo-motor.

5. Matlab/Simulink PID controller

In the scope of this research a Matlab/Simulink PID controller was developed in order to confirm the adequacy of importing and using Matlab/Simulink controllers on Beckhoff soft PLCs, which will further allow us to design and implement other types of controllers, such as nonlinear closed loop controllers. The Matlab/Simulink software environment enables us to generate program code for the TwinCAT 3 software environment, in which we design the system control program. Thus, we can directly convert the designed controller model into the TwinCAT 3 program code. Thus, at first step, we have only implemented the PID controller in the Matlab/Simulink software environment and compared the response of the implemented controller with the response of the TwinCAT 3 NC Axis system controller.

The implemented PID controller was designed in the Matlab/Simulink software environment with sampling time of 1 ms, as the execution on the controller is discrete and not continuous. The sampling time had to be the same as set in the TwinCAT 3 software environment project. The PID controller was designed using a transfer function consisting of different blocks such as add, multiply, divide, delay and input and output block. Figure 4 shows a diagram of the implemented PID controller in Matlab/Simulink.

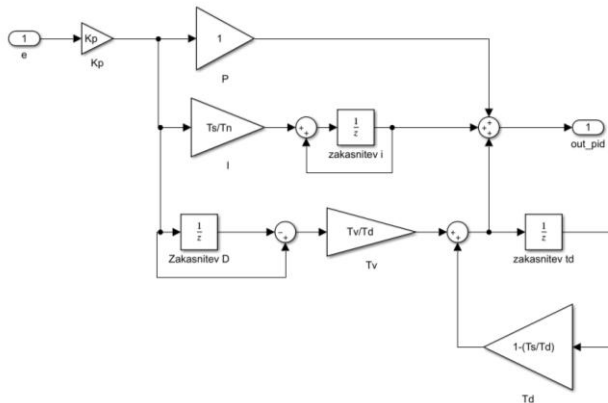


Fig. 4 PID controller developed in Matlab/Simulink.

The created diagram in Matlab/Simulink is then used to create *Subsystem* and prepare it to generate code suitable for import into TwinCAT 3 software environment. The generated code is then imported into the TwinCAT 3 program as *POU* using the import function (*PLCopenXML* format). This generates a program block including the created controller with corresponding input and output variables.

6. Results

The aim of the research was not only to control the linear system using Matlab/Simulink designed and implemented PID closed loop position controller, but also to evaluate its proper operation and response, which was achieved by comparing the responses of the TwinCAT 3 PID controller and the controller developed in the Matlab/Simulink environment. In this way, we were able to verify that the axis control system and the closed loop controller were properly designed and implemented. In case we have implemented everything correctly, the behavior of both systems should be the same. Figure 5 shows the response of the system using both PID controllers (above TwinCAT 3 PID controller, below Matlab/Simulink controller) with the same PID settings and trajectory of set-point change with speed of 1000 mm/s and acceleration/deacceleration of 500 mm/s².

The green line represents the start of the set-point change, while the orange line shows the generated position trajectory representing the set-point value and the red shows the actual axis position when moving relative to the generated trajectory. The difference between the set-point and actual position is represented by blue line. The

Figure clearly shows that the response of the both PID position controller are more or less identical, confirming that the design and the implementation of PID controller in Matlab/Simulink and import into TwinCAT 3 control software was successful.

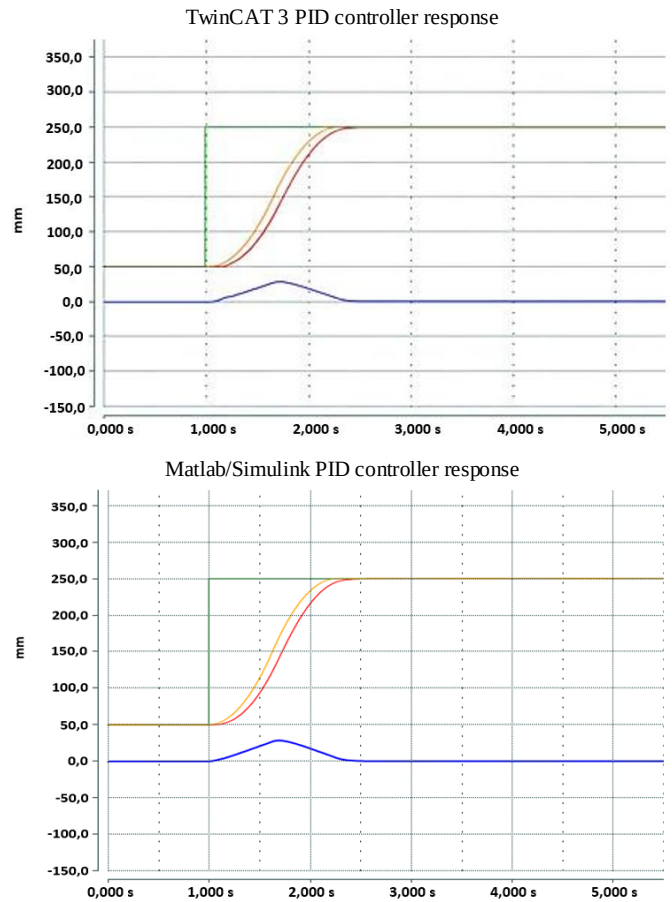


Fig. 5 TwinCAT 3 PID controller response (above) in comparison to Matlab/Simulink PID controller response (below). Green: start point; Yellow: position set-point value; Red: actual position value; Blue: difference between set-point and actual value.

7. Conclusion

The main aim of the research was to build a linear-axis research system consisting of industrial type of servo motor and stepper motor and to test the design and implementation of Matlab/Simulink closed-loop controllers in Beckhoff TwinCAT 3 system. Thus, a positioning axis with a linear guide driven by an industrial servo motor or stepper motor was designed and manufactured. Further on, the TwinCAT 3 PLC program and corresponding user interface for controlling the system was developed in Visual Studio C# WinForms application. This further allowed us to implement and test PID position controller from TwinCAT 3 system and the one built in Matlab/Simulink, which was imported into TwinCAT 3.

The final aim of the research at this point was to confirm the suitability of the PID controller designed in the Matlab/Simulink software environment, which was then imported into TwinCAT 3 software environment. The results of both controller responses fully confirm the adequacy of importing and using Matlab/Simulink controllers on Beckhoff soft PLCs. By confirming the results and obtaining an identical response, we confirmed the adequacy of the process of development and implementation of closed loop controllers from Matlab/Simulink environment.

The design and implementation of PID position controller from Matlab/Simulink software environment into TwinCAT 3 system allows us to use more advanced, more complex, more responsive

and faster controllers. We are able to compare different controllers for each system, so we can choose the most optimal controller for application needs. By adding even more different controllers, we gain the ability to test and compare new high-end controller concepts and are no longer limited to TwinCAT 3 controller types. This further enables us to develop and test different nonlinear controllers, such as a soft logic controllers. Soft logic controllers use experiential rules such as natural language words. In addition to non-linear controllers, linear controllers can also be used by changing the parameters during operation, which has the advantage that the parameters are set to the optimal ones.

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Research of the stress state of a gantry crane track

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Abstract: The technology of work in some industrial enterprises involves the using of the heavy lifting machines moving on a special railway track. The type and dimensions of the gantry crane and the crane track are in accordance to the size and weight of the processed loads and the final product. The gantry cranes and the crane tracks usually work outdoors in all weather conditions and aggressive environmental influences. Most of these facilities were built 30 ÷ 40 years ago. During this period the regulations and requirements for their operation have been changed. Digital technologies now allow more precise modeling and calculation of the structure of the crane track, as well as inspection the impact of individual elements on the stress state of the structure as a whole.

The effect of the present study will have the greatest impact on loading and unloading platforms in railway stations, harbors and intermodal terminals.

Keywords: CRANE TRACK, MODELING OF THE STRUCTURE, STRESS STATE

1. Introduction

With the development of modern computer technology and software, the issue of modeling of the building structures is becoming increasingly important. The subject of this paper is the study of the stress state of a gantry crane's track with the help of SAP 2000 computing software. Gantry cranes are used to carry out loading and unloading operations in ports, railway stations and in some industrial enterprises. These lifting machines usually operate outdoors and the crane roads are exposed to all kinds of weather conditions and aggressive environmental influences. The type and dimensions of the elements inserted in the road depend on the load capacity, working hours and productivity of the gantry crane. The rails and fastenings used in the crane track are standardized elements used as a finished product with fixed geometric and strength characteristics. However, there are different options for the sub-rail base, the suitability of which shall be proven. A variant with a continuous reinforced concrete beam on which the fastenings and rails are attached is considered in the present study. The impact of the rail cross-section, the intensity of the fastenings and the strength characteristics of the ground base on the stress state of the sub-rail beam was traced.

2. Model of the structure

The cross-sections and strength characteristics of rails 49E, 60E1 [1], A55 [2] are introduced in the digital model for the needs of research. The load model is from a gantry crane with a load capacity 12.5t [3]. The rails are point-supported on a continuous reinforced concrete beam. A scheme of the construction is given in Fig.1.

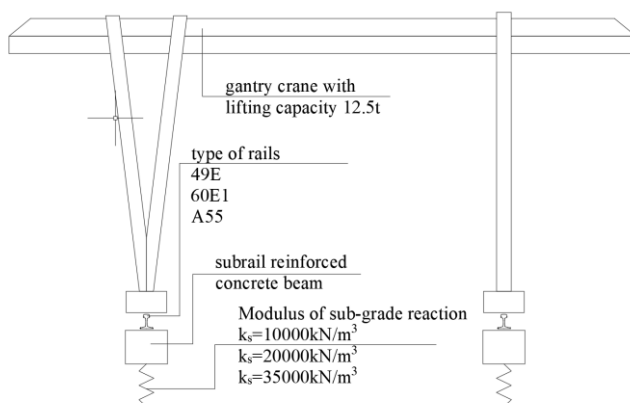


Fig. 1 Scheme of the construction and gantry crane

Model description

A three-dimensional model of the structure was made in the software product SAP 2000. The characteristics of the elements and load as follows:

- Rail → modeled by FRAME - elements with a length corresponding to the distance between the points of support (fastenings). The joints at the beginning and end of each element are modeled with 6 restrains, which means translation and rotation about the X, Y, Z axes of the coordinate system is prevented.

- Fastenings → LINK - elements. The elastic characteristics of the fastenings does not take into account. The directional properties in X, Y, Z are fixed. The study was performed for the distance between the fastenings 0.60, 0.80 and 1.00 m.

- Sub-rail reinforced concrete beam → modeled by FRAME – elements with cross section 0.4/0.5m. The characteristics of the frame elements are the same as the rail frames. Strength characteristics of the material are as for concrete C20/25, according to Eurocode 2 [4]. The springs are putted in the beginning and in the end of every frame element. The stiffness of the springs corresponds to the strength characteristics of the different ground base.

- Ground base – the strength characteristics are represented by the coefficient of subgrade reaction k_s . Three values of coefficient of subgrade reaction are used:

$k_s = 10000 \text{ kN/m}^3$ - weak soil

$k_s = 20000 \text{ kN/m}^3$ - connected soil, medium plastic

$k_s = 35000 \text{ kN/m}^3$ - fine gravel, coarse sand, semi-hard clays [5]

- The load - only load from crane and lifting load are included in the study. Loads from wind, snow, icing and seismic impact does not take into account. The crane load is modeled as concentrated vertical and horizontal forces for each wheel. Direction of the horizontal force is parallel to the axis of the road. This horizontal load represents tractive or braking force. The loads are static because of low speed of the crane in operation 1.25m/s. The load combinations are

1) self-weight + vertical force

2) self-weight + vertical force + horizontal force

The loading and unloading platform are served by two or more cranes very often. The relative position of the cranes must be such as to obtain maximum value of the member force diagram in sub-rail beam.

For this purpose, the already made model of the structure is loaded with a single vertical force and the zero points of the moment's diagram of the sub-rail beam are determined.

The continuous elastic base is modeled by assign of spring supports in joints of the reinforced concrete beam, corresponding to coefficient of subgrade reaction $k_s = 20000 \text{ kN/m}^3$. It has been studied whether the distance between joints affects to the distance A between the applied point of force and the zero point of the moment diagram.

The distance between the joints coincides with the distance between the fastenings. The data are given in Fig. 2.

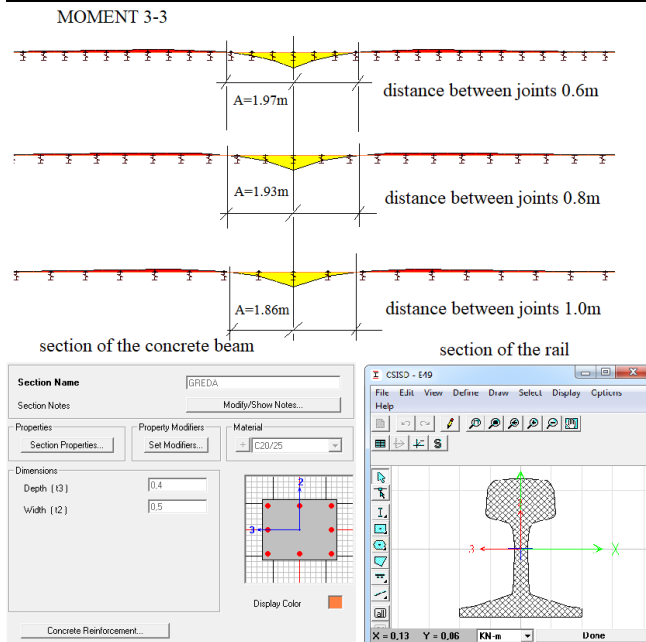


Fig. 2 Moment 3-3 diagram in sub-rail beam

The distance A can be calculated by using of the basic value of the beam L [6].

$$L = \sqrt[4]{\frac{4EI}{bk_s}} \quad (1)$$

By L the continuous beam is convert to the beam of two supports, and distance A is:

$$A = \frac{\pi L}{4} \quad (2)$$

For given on fig.2 cross sections of rail and concrete beam are received as follow

The stiffness of the structure $EI=83903\text{kNm}^2$

Width of the sub-rail beam $b=0.5\text{m}$

The basic value of the beam $L=2.4069\text{m}$

Distance $A=1.89\text{m}$

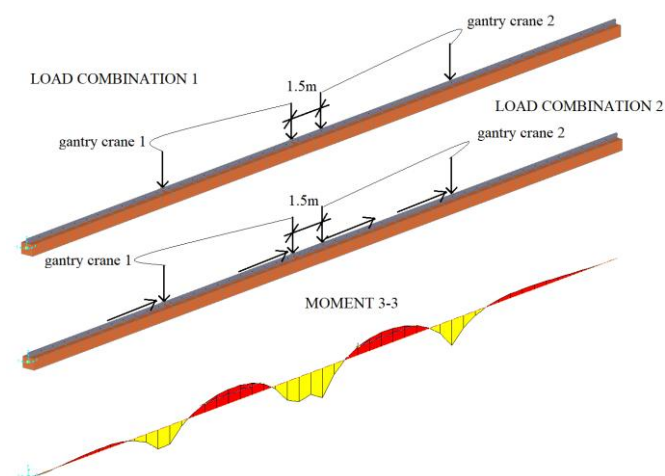


Fig. 3 The scheme of loads and the type of the moment diagram

We can see from Fig.2 that the values of A are approximately the same. The difference between the minimum and maximum value is 5.7%. It can be assumed that the distance between the fastenings does not affect the position of the zero points in the moment diagram. The difference of A value calculated with equation 2 and with the SAP 2000 is less than 5%. There is a good

agreement between the theoretically obtained value and the values calculated using the digital model.

Load combination type using in SAP 2000 for calculation of element forces is Linear Add. In this case for maximum value of moment is necessary distance between wheels of cranes to be shorter than A. The scheme of loads and the type of the moment diagram in the sub-rail beam is given in Fig.3.

3. The results

The values of the bending moment and shear force in rail and sub-rail beam are given on table 1. The values are calculated for different distances between fastenings, rail section 49E and $k_s=35000\text{ kN/m}^3$.

Table 1: The bending moment and shear force

	Distance between joints 0.6m	Distance between joints 0.8m	Distance between joints 1.0m
Moment 3-3 in rail [kNm]	19.00	19.87	28.84
Shear 2-2 in rail [kN]	124.09	214.30	201.78
Moment 3-3 in beam [kNm]	84.85	83.06	88.95
Shear 2-2 in beam [kN]	100.61	98.31	95.26

The dependency between shear force, bending moment and fastenings distance is given in Fig.4.

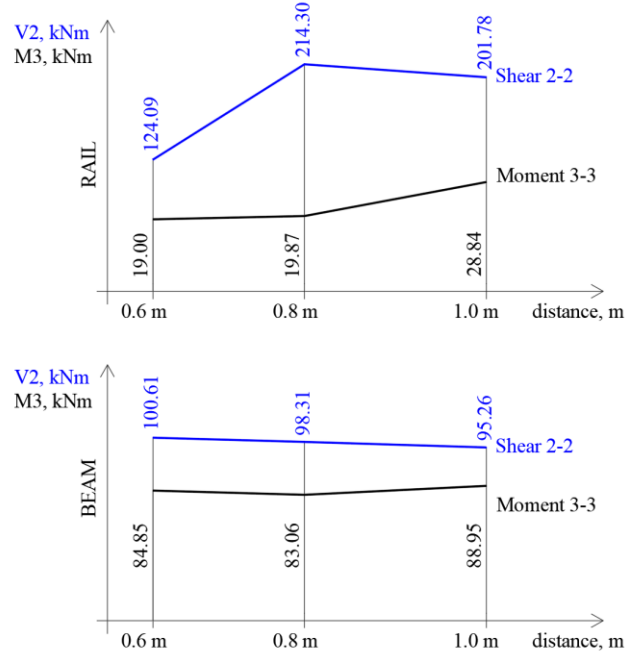


Fig. 4 Moment 3-3 and Shear 2-2 in rail, and in sub-rail beam

The first part of the graph shows that the distance between the fastenings significantly affects the values of the shear forces in the rail. Different options for the distance between the fastenings should be considered when choosing the cross section of the rail. The second part of the graph shows that the distance between the fastenings changes the values of the shear forces and bending moments in the reinforced concrete sub-rail beam slightly. The values of the shear force decrease with increasing distance, but the difference is small 5.2% between the maximum and minimum value. For the bending moment, the difference between the maximum and minimum value is also small 6.6%.

The influence of the rail section on the bending moment and shear force in the sub-rail beam was also studied. The data is given

in table 2. All values are calculated for fastenings distance 0.6m and coefficient of subgrade reaction $k_s=35000 \text{ kN/m}^3$

Table 2: The internal forces in sub-rail beam by different rail sections

	Rail A55	Rail 49E	Rail 60E
Moment 3-3 in beam [kNm]	102.51	84.85	79.06
Shear 2-2 in beam [kN]	119.63	100.61	94.15

The dependency between shear force, bending moment and rail cross section is given in Fig.5

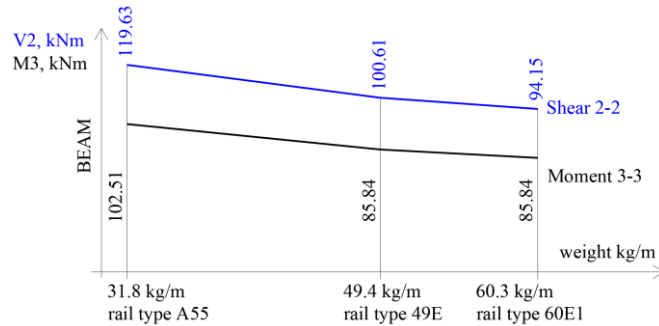


Fig. 5 The dependency between shear force, bending moment and rail cross section

It can be seen that as the weight of the rail increases, the bending moment and the shear force in the sub-rail beam decrease. However, almost doubling the weight of the rail reduces the bending moment by 16% and the shear force by 21%.

To establish the relationships between the coefficient of subgrade reaction and the beam forces in the digital model, bed coefficient values were used for weak soils, for connected soil medium plastic and for fine gravel coarse sand semi-hard clays. The numerical values of the forces are given in Table 3. The study was performed for rail type 49E and distance between the fastenings 0.6 m.

Table 3: The internal forces in sub-rail beam by different values of coefficient of subgrade reaction k_s

	$k_s=10000 \text{ kN/m}^3$	$k_s=20000 \text{ kN/m}^3$	$k_s=35000 \text{ kN/m}^3$
Moment 3-3 in beam [kNm]	109.66	95.56	84.85
Shear 2-2 in beam [kN]	108.97	105.06	100.61

Based on Table 3, it can be concluded that the bending moment and shear in the beam are higher with a weaker ground base. The moment decreases by 23% if the weak base is changed with sustainable soil base. The shear force decreases by 8%.

In Fig. 6 and Fig. 7 are shown the generalized relations of the moment and the shear force.

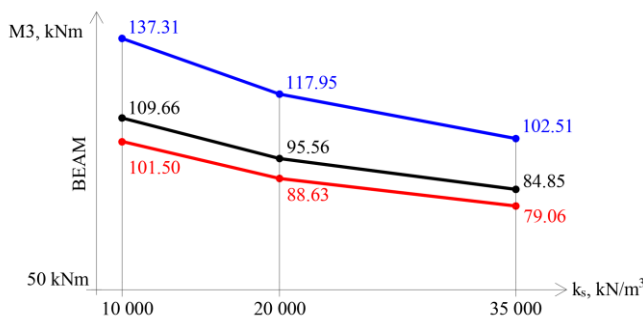


Fig. 6 Bearing moment in sub-rail beam by different values of k_s

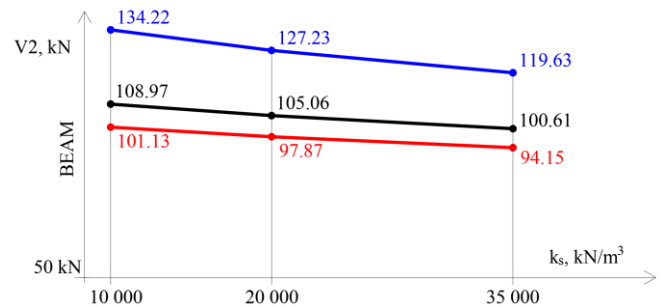


Fig. 7 Shear force in sub-rail beam by different values of k_s

4. Conclusions

The conclusions from the examinations made so far are the following:

- Replacement the cross-section of the crane track rails can reduce the internal forces in the sub-rail beam.
- The distance between fastenings does not affect the values of the internal forces in the sub-rail beam.
- The strengthening of the ground base under the crane road also leads to a reduction of the internal forces in the sub-rail beam.

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Possibilities of reducing the degradation of molds for high-pressure of Al alloys

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Abstract: The paper focuses on the degradation of molds that are used for the technology of high-pressure casting of Al and its alloys. The method of high-pressure casting of aluminum products is one of the widely used production methods, which at the same time meets the requirements for precision and productivity in the production of cars and various mechanical parts. In the high-pressure casting process, the molds are exposed to various thermal and mechanical loads, where the molds and their shaped parts are degraded. The paper presents the results of research focused on the use of duplex PVD coatings to increase the life of shaped parts of molds for high-pressure casting of Al and its alloys.

Keywords: ALUMINUM, DIE CASTING, DEGRADATION, COATINGS, ADHESION, CRACKS

1. Introduction

The high-pressure die casting process is used to produce parts mostly from aluminum, magnesium, zinc, and copper alloys by injection of the molten metal in the mold cavity. The mold cavity that are used to produce those parts are constantly exposed to highly severe conditions, such as high pressure, rapid temperature fluctuations and erosion from fast moving molten metal. The usual molten metal input speed is comprised between 20 and 60 m/s and the temperature, depending on aluminum alloy type is around 700 °C. The maintenance of replace of these molds require a huge cost which implies that producers need to find the best solution to increase their lifespan. The industrial environmental and working conditions increase the capacity to induce some failure mechanisms on hot work tool steel, such as erosion, corrosion, wear, and thermal fatigue. Parts produced by this method conform accurately to the die size, have favorable mechanical features and are low in cost [1,2].

The die casting method of aluminum products is one of the widely used manufacturing methods as it is a technology that can simultaneously satisfy precision and productivity requirements during the production of automobile and various mechanical parts. However, during the rapid solidification process, defects like porosity and shrinkage might remain and significantly reduced the ultimate mechanical properties, e.g., tensile strength, wear resistance and fatigue strength [3]. During die casting process operations, the tools steel used to this process are exposed to a several thermal and mechanical loads, which can lead to damage the molds for high-pressure die casting. The improvement of these structures' lifetime was strongly required due to several economic and environmental reasons. Corrosion, soldering, erosive wear, and thermal fatigue are the primary failure mechanisms that limit die life in aluminum high-pressure die casting. Corrosion and soldering are caused by the physical impingement of the incoming liquid aluminum. Thermal fatigue results from the change in stress caused by alternate heating and cooling of the die surface during the casting process. Under the combined effects of these failure mechanisms, the die will crack; fragments are broken off the die, necessitating die removal and a consequent increase in process costs. One of the major damage mechanisms occurring in die casting process, under cyclic thermal loads is the formation of a network of interconnected cracks [4-6].

The stress cracking, which presents another variant of thermal fatigue cracks, was clearly marked in areas exposed to local stress concentrations and can lead to crack initiation in a die casting mold. Then, these cracks can grow and became, more pronounced driven by several factor including thermal fatigue, erosion, oxidation, soldering of the molten metal to the die surface, deformation of die contact surface and dangerous fracture. The formation of thermal fatigue cracks may lead to a loss of surface material as small fragments splinter off from the surface. To endure these severe conditions the tools are made of hot-work tool steel, designed to have and adequate combination of hot strength, toughness, and ductility, as well as thermal conductivity and thermal expansion. Die and its shaped parts maintenance may be done by grinding or

welding if the surface quality or dimensions of the castings are no longer sufficient [4-6]. However, the tool and service costs constitute a remarkable part of the production costs in die casting and there are numerous approaches to optimize the lifetime of the dies. In general die life may be enhanced by geometric factors in die design (governing stresses and thermal gradients), die material considerations e.g., machinability, heat treatment, toughness, resistance to wear and heat checking, processing conditions (preheating, heating and cooling cycles, machine closing force, lubricants, service intervals) and die surface considerations [5,6]. The Fig. 1 shows the degradation mechanism of the mold part - the mold insert used for high-pressure aluminum die casting due to the repetitive cycles of the casting process.



Fig.1 Degradation of the mold part

Surface treatments, such as nitriding, hard PVD coatings and others are often applied for casting dies to reduce failure mechanisms (abrasive wear) and improved thermal fatigue resistance. In recent years, hard PVD coatings on nitride-based to the surface of the mold and its shaped parts have been applied to increase the overall life of the molds and their parts used in high-pressure die casting of aluminum and its alloys. In Fig. 1 is a comparison of the life of a mold for high-pressure aluminum die casting without the use of PVD coatings and using PVD coating based on nitrides CrN, Ti (C, N). Hard coatings based on nitrides and carbides of transition metals (TiN, CrN, CrAlN, CrC) and duplex treatments that combine surface modification of the die with a hard coating have also been developed. The use of PVD coatings has been shown to increase the service life several times (4 to 17 times). The die coating system to be far superior to that of the uncoated die with respect to cost performance criteria. The die coating system must be [7-9]:

- Non-wetting with liquid aluminum
- Wear and oxidation resistance
- Able to accommodate the thermal residual stresses induced by shot cycling (temperature and pressure) during pressure die casting process

- Adherent to the die material – and engineered interface
- Able to delay the onset of thermal fatigue cracking (heat checking)

The Fig. 2 represents a diagram of an optimized coating architecture for die coatings used in aluminum high pressure die casting [7]:

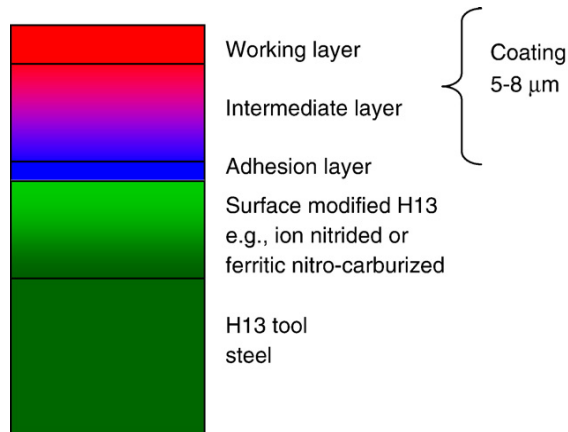


Fig.2 A diagram of an optimized coating architecture for die coatings used in aluminum high pressure die-casting.

2. Materials and Methods

Uddeholm Dievar was used as the base material for experiment, which is used in the production of die casting molds for aluminum and their shaped parts. The chemical composition of the base material is in Tab.1.:

Table 1: Chemical composition of Uddeholm Dievar

Element	C	Si	Mn	Cr	Mo	V
Wt. %	0.38	0.2	0.5	5	2.3	0.6

The mold parts were manufactured according to the drawing documentation and were tempered to a hardness of 48 HRC and subsequently fitted into the mold body for high-pressure casting of aluminum alloy base on Al-Si-Cu realized on machines with a cold filling chamber. The mechanical properties of the base material are shown in Tab.2:

Table 2: Mechanical properties of Uddeholm Dievar

Mechanical properties of Uddeholm Dievar				
Hardness [HRC]	Tensile strength Rm [MPa]	Yield strength Rp _{0.2} [MPa]	Ductility [%]	Relative narrowing [%]
52	1900	1560	12.5	52

AlXN³ and nACRo³ coatings were used to perform PVD coating. For the nano-multilayer coating, ALXN3 (X=Cr) is the basic adhesive coating of CrN, followed by Al/CrN nano-coating and the top coating is AlCrN. It is a tough coating with high resistance to abrasion at high temperatures up to 900 °C.

The nACRo³ is a nanocomposite coating consists of AlCrN nanocrystalline grains that are embedded in an amorphous Si₃N₄ matrix. The coating are three layers: the first adhesive coating consists of CrN, the second coating is AlCrN, and the three final top coating is formed by a nc-AlCrN/a-Si₃N₄ nanocomposite coating. This nanocomposite coating is very tough, resistant to abrasion to high temperature up to 900 °C – 1100 °C. The light microscopy technique was used for crack site analysis. The quality control of the mold surface before coatings process consisted of a visual control, which was carried out in accordance with ISO 13018. The visual control was followed by a capillary control according to ISO

23277. Scanning electron microscopy was used for chemical elemental analysis at the crack site. The Mercedes test (Tab.3) was used to measure the adhesion of the coatings, which was performed on a universal hardness tester UH 250 with a Rockwell indenter at a load of 1500 N according to ISO 18265. The evaluation is performed by assigning it to the appropriate category with the adhesion number HF1 – HF6, which characterizes the degree of cracking and peeling layer.

Table 3: Coating adhesion by the Rockwell indentation test

Evaluation of coating adhesion by the Rockwell C indentation test			
	HF 1- good adhesion a small amount of cracks		HF 4 - reduced adhesion peeling around the edge of the indent
	HF 2 - satisfactory adhesion small peeling between cracks		HF 5- insufficient adhesion peeling even at greater distances from imprint
	HF 3- reduces adhesion peeling over more than two cracks		HF 6- unsatisfactory adhesion complete peeling around the indentation

A Jeol JSM 7000F scanning electron microscope with an EDX analytical unit was used to determine the chemical elemental analysis of the defect environment according to ISO 15362. Microhardness measurements were performed on a Leco LM 700 microhardness tester with a Vickers indenter HV 0.025 according to ISO 6507-1. Scratch test was used to evaluate adhesion of coatings and was performed on an AMI CSEM -Revertest device (feed 10 N/mm, load up to 80 N, track length approx. 8 mm). All samples for analysis were prepared by metallographic procedure. The samples were prepared in conductive dentacryl Polyfaste, then were ground on sandpaper of various grits (240, 400, 600 and 800), moistened with water, polished with 1/0 diamond paste, washed, and rinsed with benzine alcohol. Prior to observation, all samples were purified in methanol in an ultrasonic device. Samples were taken from the molded parts of the mold half according to Fig. 3:



Fig.3 Sampling procedure from the mold part

3. Results and discussion

In Fig.4 is worn solid half of the mold part - mold insert and appearance of cracks of the mould after capillary test.

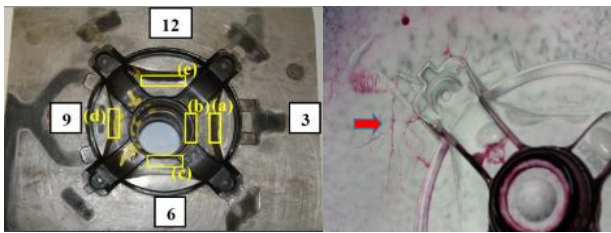


Fig.4 Worn solid half of the mold insert (left); appearance of cracks of the mould after capillary method (right)

Wedge-shaped branched crack were observed in the analyzed places of the mold part due to cyclic stress of the mold parts in the area of elastic deformations. The cracks that formed in the areas of sharp transitions in the corners of the mold parts were filled with oxides and a release agent during the repetitive casting process. It is precisely due to the repetitive elastic deformations during the repetitive cycle of the casting process that the oxide filler and the release agent acted as a wedge, which was pressed into the mold material and caused wedge branched cracks. The analyzed cracks observed by light and electron microscopy are shown in Fig.5:

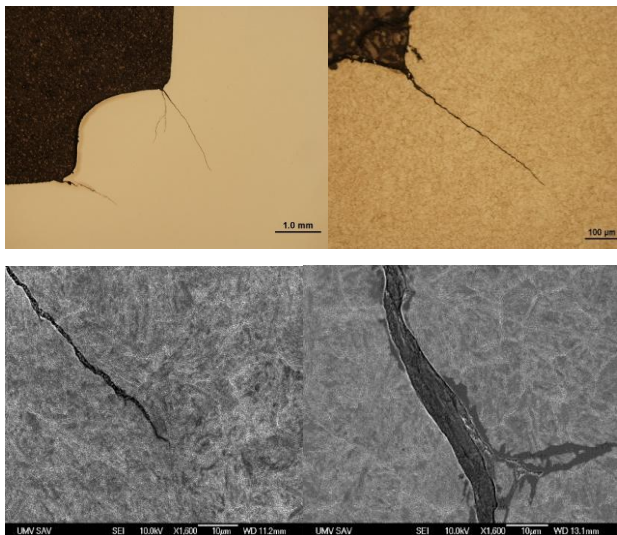


Fig.5 Cracks in the zones of sharp transitions of the surfaces of shaped parts of the mold (light and electron microscopy)

The presence of other chemical elements, namely the presence of oxygen, calcium and silicon, was observed in the filling of wedge shaped cracks formed by oxide and a separating agent. Outside the crack-containing zones, the microstructure was formed by heterogeneous sorbitol together with fine globulite carbides on Fe-Cr-Mo-V.

The presence of other chemical elements, namely the presence of oxygen, molybdenum, chromium and silicon, was observed in the filling of wedge shaped cracks (Fig.6) formed by oxide and a separating agent. Outside the crack-containing zones, the microstructure was formed by heterogeneous sorbitol together with fine globulite carbides on Fe-Cr-Mo-V. EDX analysis was also performed at the crack location according to the Fig.6 on the mold part shown in the Tab. 4:

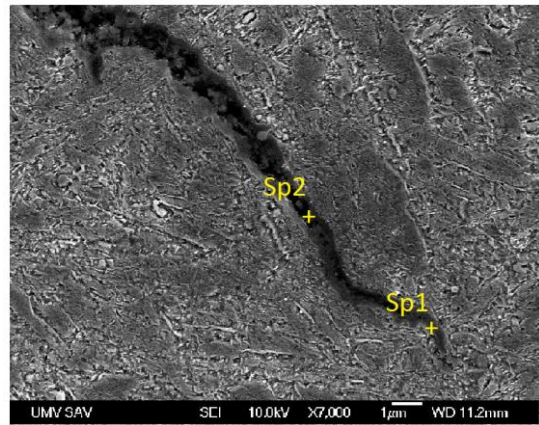


Fig.6 Zone of sharp transitions of the surfaces of the shaped parts of the mold

Table 4: EDX analysis (Sp1, Sp2) at a crack site

	Element	Wt. %	At. %
Sp1	O	24.85	52.57
	Si	3.13	3.78
	Fe	7.2	43.65
	Totals	100	
Sp2	O	23.58	51.02
	Si	3.91	4.82
	Fe	65.83	40.8
	Cr	3.13	2.8
	Mo	3.54	1.28
	Totals	100	

3.1 Adhesion of PVD coatings

Good adhesion of PVD layers to the substrate was confirmed, determined by the ratio cohesive and the degree of HF = 1, which was characteristic for the occurrence of only isolated cracks and minimal disruption of the integrity of PVD coatings around the indentations. Fig.7 shows the morphology the indentations into the coating surfaces, with only isolated radial cracks in both cases reaching of max. 200 µm. It was measured a degree of adhesion HF = 1-2 according to the Tab.3, which means good adhesion of a coating.

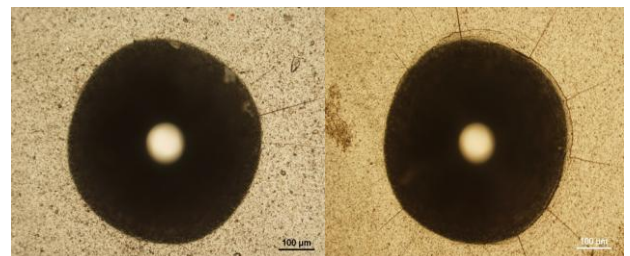


Fig.7 Indentation impression after Mercedes test; ALXN³ (left), nACR³ (right)

3.2 Tribological properties of PVD coatings

The device records the course of the increasing normal F_n and the tangential force F_t acting on the indenter, the values of the coefficient of friction and the acoustic emission signal AE. The output is a graphical record of the AE emission signal and the COF depending on the size of the load. The value of the critical load F_z

at which the substrate. In practice, the value of the critical load $F_z = 40$ N is referred to as satisfactory adhesion. Satisfactory adhesion was recorded on the subject tested samples with applied coatings, because the failure or detection of the substrate occurred at values of about 50 N (Fig.8).

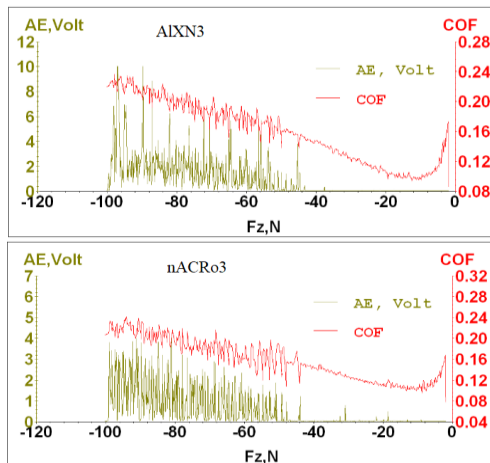


Fig.8 Dependence of acoustic emissions (AE) on the coefficient of friction (COF) of coatings

3.3 Microhardness of PVD coatings

By evaluating the microhardness of the Vickers indenter HV 0.025 of PVD coatings, an increase of about 18% - 25 % compared to the base material of mold was recorded. The microhardness measurement was performed in 16 places, and a graph of microhardness values was subsequently constructed from the measured values. Both minimum and maximum values were measured:

- In the case of the ALXN³ coating, the minimum microhardness value was 600 HV and the maximum microhardness value was 720 HV
- In the case of the nACRo³ coating, the minimum microhardness value was 610 HV and the maximum microhardness value was 810 HV

Fig.9 presents hardness profile of coatings and shows an arrow from the area of the coatings to the base material:

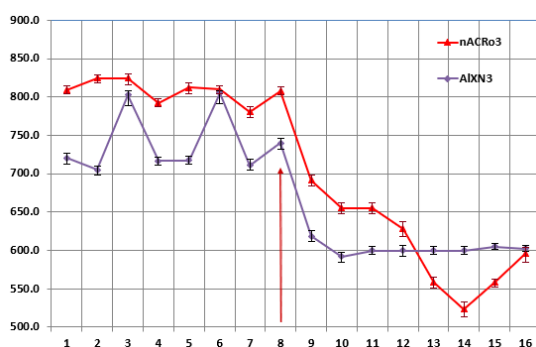


Fig.9 Graph of measured microhardness values

4. Conclusions

The paper is the results of research aimed at finding out the degradation mechanisms of molds for high-pressure aluminum die casting and the possibility of modifying the quality of functional surfaces of molds using PVD nanostructured coatings of a new generation. In the first phase, the degradation mechanisms in the corners of shaped parts (inserts) of the mold in the form of branched cracks due to elastic deformation due to repeated of the casting process were analyzed. In the second phase, a conventional ALXN3

coating and a new generation nanostructured nACRo3 coating were applied to the mold parts. The PVD coatings were of high quality, which was confirmed by tests performed to assess the adhesion, hardness, and COF of coatings. The coatings were compact, intact, and formed a barrier between the base material and the molten metal Al after the high-temperature corrosion test (680 ± 20 °C).

5. Acknowledgments

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Study of the defect closure behaviour during forging in a step-wedge strikers

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Abstract. This work is devoted to the study of the internal defect closure during metal broaching in step-wedge strikers with wedge and depression by FEM in Deform program. A through cylindrical hole with a diameter of 3 mm was considered as an internal defect. At the same time, three variants of the defect vertical location were modeled – in the workpiece center, near the upper face of the wedge and near the lower face of the depression. In all three cases, the internal defect closure is uneven. The most intense defect closure is observed when it is located in the workpiece axial zone. Defects located near the strikers surface zones are not closed well enough. The stress-strain state analysis showed that the defect location affects the distribution nature of stresses and strains along the workpiece section.

Keywords: FORGE, STEP-WEDGE STRIKERS, FEM, DEFECT CLOSURE, STRESS-STRAIN STATE.

1. Introduction

When obtaining blanks for forging and stamping production, one of the main tasks is to eliminate various defects formed at the casting stage. The most typical defects of this class are discontinuity defects – cracks, gas bubbles, non-metallic inclusions, etc. Their presence in the finished forging leads to a significant anisotropy of mechanical properties along the section of the workpiece, which can lead to breakage of the part made of this forging. Therefore, the research of traditional and the development of fundamentally new methods and devices that allow to increase the level of internal processing of metal during forging operations remain relevant.

In the course of previous studies, new designs of forging strikers were proposed, the configuration of which allows to develop both shear and alternating deformations in the metal [1]. A comparative analysis of the strikers of two configurations revealed that the use of a wedge on one striker and a depression on the other is the most optimal solution from the point of view of surface shaping of the metal [2]. This work is devoted to the study of the closure of internal defects during metal broaching in step-wedge strikers of optimal configuration with finite element modeling in the Deform program.

2. FEM-simulation

A square section blank with a side of 30 mm and a length of 15 mm was created as the initial part. The choice of a small length was due to the fact that on each of the three strikers sections, the surface configuration (the nature of the faces inclination) remains unchanged, i.e. the use of a workpiece whose length is obviously less than the length of the smallest section, equal to 20 mm, makes it possible to simultaneously assess the shape of the metal at the front and rear ends, as well as in the central workpiece zone. When using a billet of a longer length, similar results would be obtained during different broaching stages.

As an internal defect, a through cylindrical joint with a diameter of 3 mm was created. At the same time, three variants of the vertical location of the defect were considered – in the center of the workpiece, near the upper face of the wedge and near the lower face of the depression. To optimize the calculation on the workpiece, vertical symmetry was used (Fig. 1). AISI1035 steel heated to 1100°C was chosen as the workpiece material. The compression of the workpiece in each section was regulated by the size of the gap change of 10% during deformation in the first section. The friction coefficient at the contact of workpiece and strikers was assumed to be 0.3.

During the analysis of the models, the shape of the workpiece (the closure degree of the through defect), as well as the main parameters of the stress-strain state – equivalent strain and average hydrostatic pressure were studied.

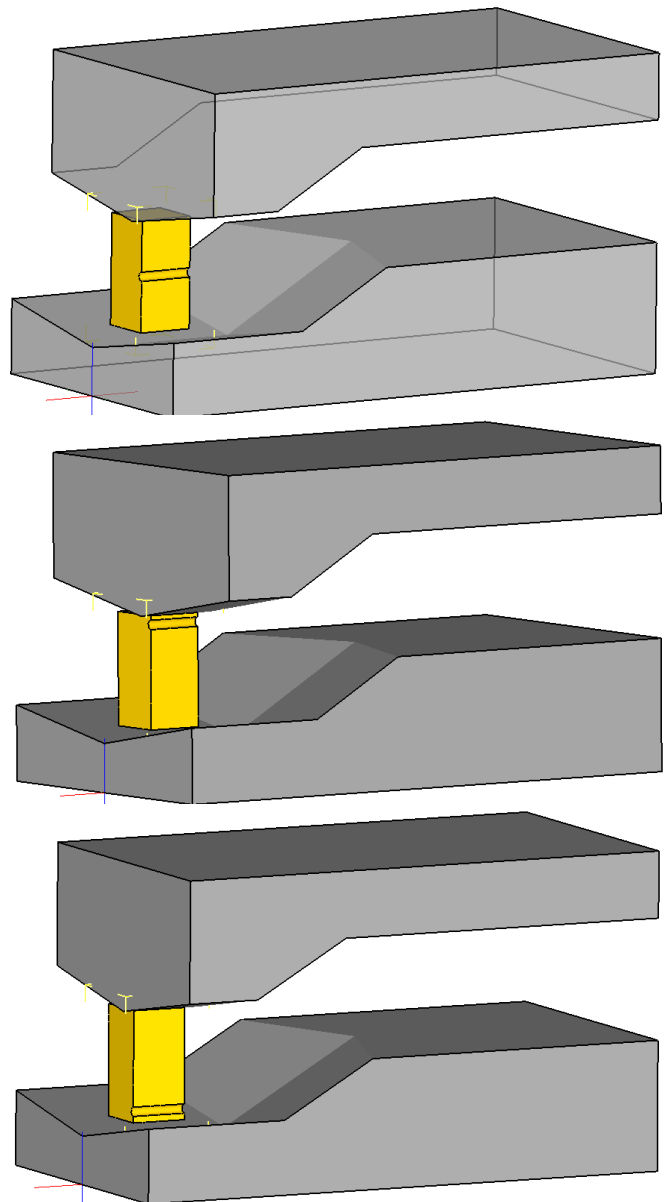


Fig. 1. Initial models

3. Results and discussion

As a result of the calculation of the models, the following results were obtained (Fig. 2).

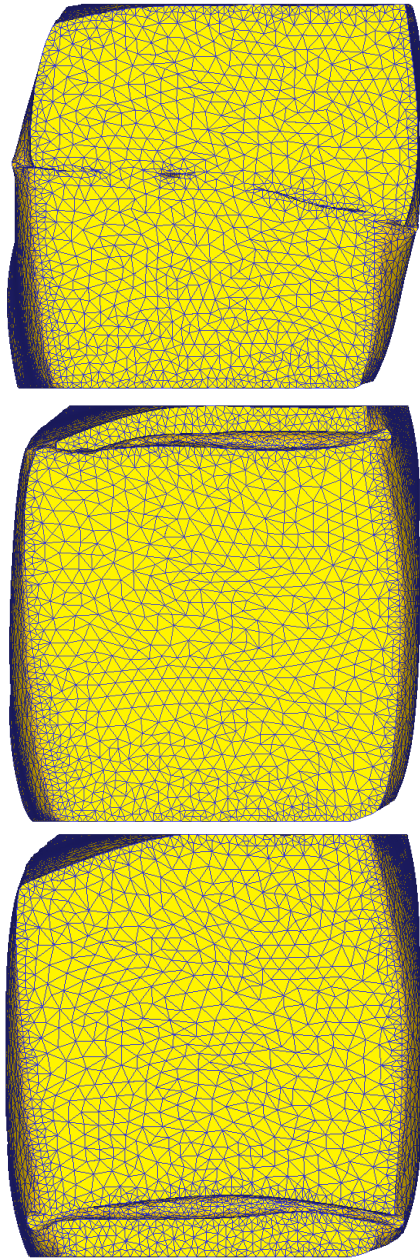


Fig. 2. Defects closing

In all three cases, the closure of the internal defect is uneven. At the same time, the most intense closure of the defect is observed when it is located in the axial zone of the workpiece. Defects located in the surface areas are not closed well enough. This is a consequence of the fact that in the axial zone the workpiece receives deformation from the simultaneous action of the wedge and the depression of both strikers, which is characterized by the slope of the defect generator. In surface defects, the slope is observed only from the side of the nearest configuration element (wedge or depression), in the same zone, the maximum closure of the defect occurs, which is still insufficient due to almost a single impact.

Analyzing the parameters of the stress-strain state, it was found that their distribution over the section of the workpiece also depends on the location of the defect. When analyzing the equivalent strain (Fig. 3), it was revealed that the numerical value of the level of the developed strain in one pass in all three models is approximately the same and ranges from 0.3 in the least processed zones to 0.9 in the most processed zones. At the same time, the nature of the strain

distribution in models with an upper and lower location of the defect has a "mirror" character, i.e. the patterns of the distribution of the strain fields are opposite in the horizontal direction. In the case of the central location of the defect, the distribution of strain fields has a relatively symmetrical appearance.

When analyzing the average hydrostatic pressure in models with the upper and lower location of the defect, the "mirror" nature of the distribution of stress fields was also revealed (Fig. 4). At the same time, the zone of maximum compressive stresses ($-138 \text{ MPa} \div -250 \text{ MPa}$) covers almost the entire workpiece in height, but does not affect the defect area. In a model with a central location of the defect, the specified zone of maximum compressive stresses passes through the defect area, which contributes to its more intensive closure.

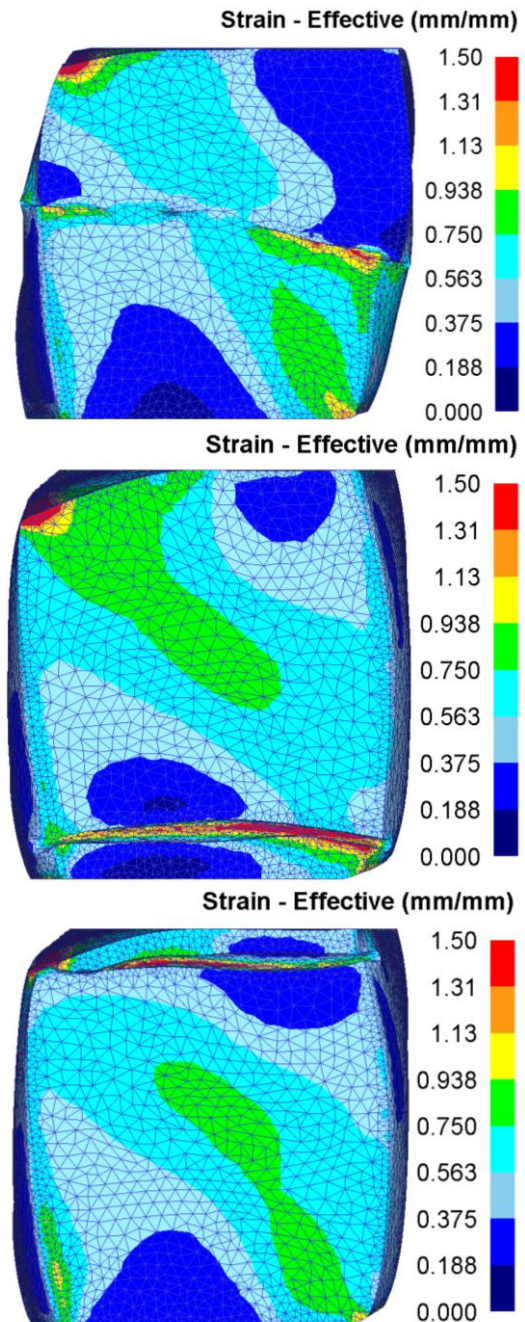


Fig. 3. Equivalent strain

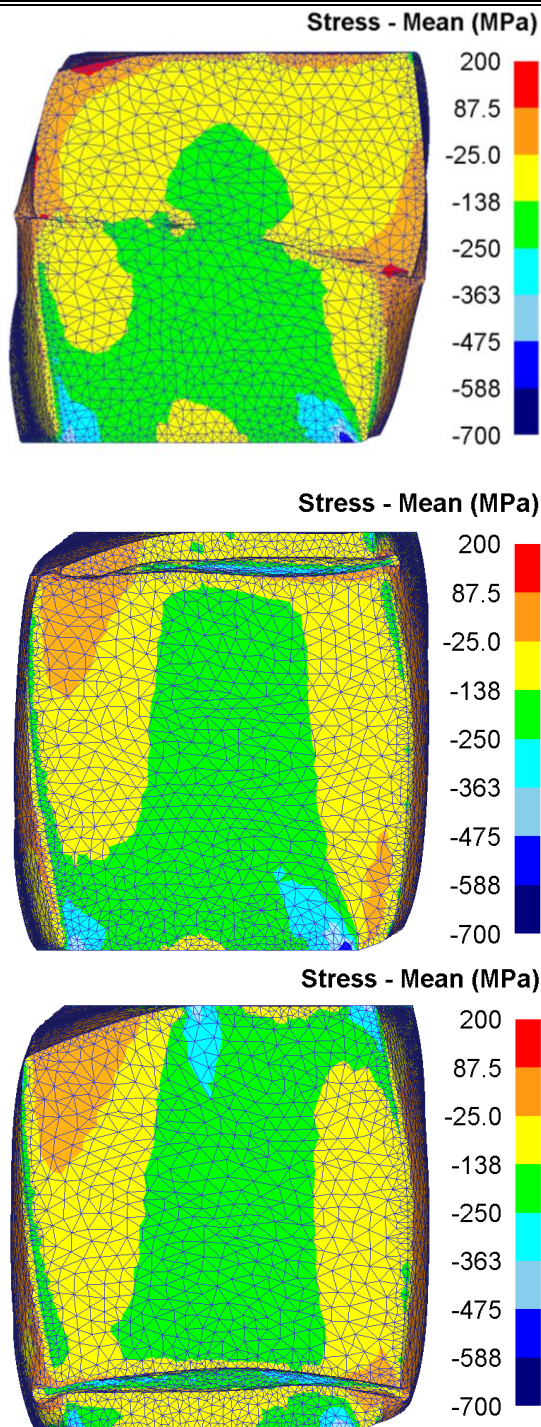


Fig. 4. Average hydrostatic pressure

Conclusion

The study of the internal defect closure during metal broaching in step-wedge strikers with wedge and depression by FEM in Deform program showed that the most intense defect closure is observed when it is located in the workpiece axial zone. Defects located near the strikers surface zones are not closed well enough. The stress-strain state analysis showed that the defect location affects the distribution nature of stresses and strains along the workpiece section. Taking into account the fact that the main share of defects in cast blanks (in particular, forging ingots) is located precisely in the axial zone, this configuration of strikers is quite a preferable choice for the effective study of the inner layers of metal in order to close possible defects.

Acknowledgments

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Method for repair and recovery of defected industrial reinforced concrete structures through an effective thin repair overlay

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Abstract: The bearing monolithic reinforced construction of "Open Fuel Storage" of SOLVAY SODY AD, Devnya, has been operated for more than 40 years in conditions of extreme stress and external aggressive factors. All this has logically led to the presence of serious corrosion damages. The basic requirements for developing a technical solution for repair and restoration of defect structures retaining walls (assembly type), supporting beams and columns, is the bringing of the technical characteristics of the reinforced concrete structure in its initial (design) or better condition. The report discusses the features of the proposals as the most effective technological method for repair, based on the possibilities for the formless construction of a new special thin repair concrete layer restoring the original cross-section, applied with the methods of "wet" shotcreting. A composition of high-tech concrete with the participation of an internal crystallization chemical admixture and a shrinkage-compensating one suitable for shotcrete is proposed. Specific details have been developed for the renovation of the individual structural elements, as well as optimal technological regulations for the implementation of the repair.

Keywords: DAMAGED CONCRETE STRUCTURES, INNOVATIVE CONCRETE MIX DESIGN, INTERNAL CRYSTALLIZATION ADMIXTURES, SHRINKAGE COMPENSATING ADMIXTURES, "WET" SHOTCRETING

1. Introduction

The bearing monolithic reinforced construction of "Open Fuel Storage" of SOLVAY SODY AD, Devnya, has been operated for more than 40 years in conditions of extreme stress and external aggressive factors – marine aerosols, specific chemical aggression, industrial air pollution etc. (Photos 1-3).



Photo 1 Open fuel storage – general view



Photo 2 Storage – inside



Photo 3 Severe structural damages

The basic requirements for developing an optimal technical solution for repair and restoration of defected structures retaining walls (assembly type), supporting beams and columns, is to bring the technical characteristics in their initial (design)/or better state. The latter should be considered as a technical assignment for the design of the relevant technical solution.

2. Possible technical solutions

Retaining walls along the main axes of the storage, built of pre-fab steel-reinforced concrete panels, currently operates under the combined action of operational and external environmental factors, some of which have significant corrosion potential. The latter has had a negative impact on the reinforced concrete, as in some parts

of the structure there are serious corrosion damage, the removal of some of which is urgent.

According to the project assignment, the repair and restoration works should be carried out bilaterally - inside the volume of the warehouse on the surface of the retaining walls (entirely on the entire open surface of the panels), while on the outer surface (outside the volume of the warehouse) they must in certain areas marked as defective after an on-site technical inspection.

The latter also applies to the defective parts of the main supporting structure (columns and beams), which are repaired to restore concrete section only in the areas of established and marked defect.

Design and implementation of the necessary repair and restoration works, based on the development of a technological regulation for the application of a company repair system in the context of the series of standards **BDS EN 1504 Products and systems for the protection and repair of concrete structures**, is a priori rejected. The latter would mean the combined application of a system of compatible companies with different purposes, but of relatively large thickness, which will inevitably increase the unreasonably high cost of repairs, as well as increase the duration of repairs.

The construction by a conventional method (monolithic construction) of an entirely new steel-reinforced concrete shell, increasing the cross section of the retaining walls by 10-12 cm, is also unacceptable, given the impossibility of simultaneous and completely one-sided surface disclosure of internal surfaces, a prerequisite for unacceptable shutdown of external reinforcement, which would lead to a problem with structural stability safety.

As for the application of such methods to the main load-bearing structure (columns and their connecting beams), they can be applied only in case of proving non-repairable complete defect of individual elements.

The most suitable technological method for repair is the formless construction of a new special concrete innovative overlay, restoring the original cross-section, applied with the methods of shotcreting. Exactly such an approach is perceived as a basic one in the developed technical solution. Preliminary technical and economy considerations prove the advantage of so called "wet" shotcreting over dry one.

3. "Wet" shotcreting design solution description

The principal technological sequence of work includes, directed to pre-fab panels, columns and beams is described below. Respective detail needed are proposed (Fig. 1-4):

- treatment of the entire surface of the panels from the inside of the warehouse by blasting with highly abrasive material (slag), resp. the marked areas on the surface of the panels on the outside of the warehouse;
- additional mechanical treatment of visibly defined "weakened" areas "to strong non-carbonized concrete" - if necessary (proven high degree of carbonization of concrete in the depth of the section, according to the requirements of **BDS EN 14630: 2007 Products and systems for protection and restoration of concrete structures. Test methods (Determination of carbonization depth of hardened concrete by phenolphthalein method)**;
- restoration of corroded and/or broken existing steel reinforcement (in details);
- installation by anchoring a new welded steel mesh N6 (15x15 cm) - only on the surface of the panels with damaged steel reinforcement from the inside of the warehouse (in details);
- cleaning and dedusting with compressed air;
- wetting of the surface without the effect of water saturation - 1 day before shotcreting in day-casting zone;
- anti-corrosion protection of the exposed and / or restored existing reinforcement and, respectively, the new steel mesh;
- re-cleaning of the surface with compressed air to remove any fine dust particles from the operation of the warehouse - immediately before laying the shotcrete;
- laying and finishing of a specially developed within the project prescribed mix design of innovative polymer-modified hybrid fibre-reinforced fine-grained concrete with included internal-crystallization and shrinkage compensating chemical admixtures, intended for shotcreting by "wet" method;
- application of a moisture-retaining membrane impregnator within the necessary "concrete curing" after finishing.

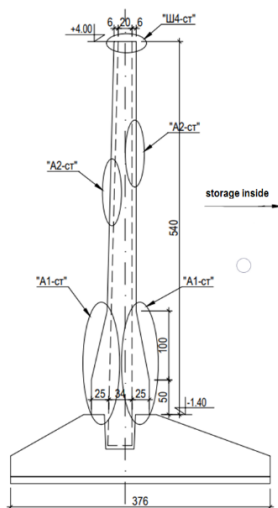


Fig. 1 Detailing zones – retaining wall

The concept for ensuring design durability, based on the specification of minimum requirements for the composition of prescribed concrete and the required steel-reinforcement concrete cover in accordance with the requirements of **BDS EN 1992-1-1: 2005 Eurocode 2: Design of reinforced concrete structures, Part 1-1: General rules and rules for buildings, resp. National Annex (NA: 2011)**, is specified below.

In addition to the operational mechanical effects, the structure is simultaneously subjected to long-term action of environmental and operational factors - physical and chemical influences. The latter are classified in **BDS EN 206: 2013+A1:2016 Concrete. Specification, properties, production and conformity, resp. National Annex / NA:2017**.

The exposure classes are selected depending on the conditions valid at the place of use of the concrete.

Concrete can be subject to more than one impact, and the environmental conditions to which it is exposed can be expressed as a combination of impact classes. For a specific structural element, different concrete surfaces may also be subject to different environmental influences.

In case of chemical exposure, a special study may be required to establish the relevant external conditions. The latter is in force for the industrial area of the town of Devnya, where the combined action of many corrosion factors due to the exploitation environment, affected by the surrounding chemical plants, should be taken into account.

Based on all those factors, in summary it can be considered that the construction of Open Fuel Storage, in operation works is in conditions of the combined action of several aggressive factors. (*Table 1*).

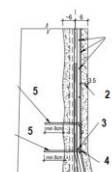


Fig. 2 Zone III4-cm - retaining wall

- 1 – welded mesh reinforcement N6 15x15; 2 – concrete cover;
- 3 - horizontal distribution reinforcement N10/120
- 4 – bend of welded mesh reinforcement;
- 5 – dowels N8x20 bonded with HIT-HY 200R for fastening horizontal distribution reinforcement (2 pcs/m²);

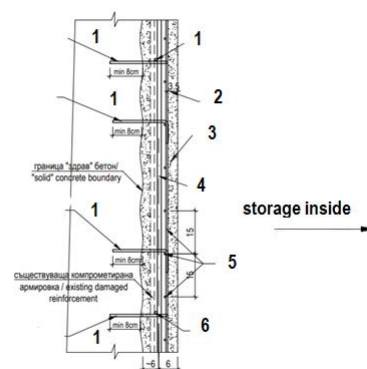


Fig. 3 Zone A2-ct - retaining wall

- 1 – dowels N8x20 bonded with HIT-HY 200R for fastening horizontal distribution reinforcement (1 pcs/m²); 2 – concrete cover;
- 3 - FR sprayed concrete; 4 – new vertical reinforcement N14/120 cm;
- 5 – N6 15x15 welded mesh reinforcement; 6 – N10/120 horizontal distribution reinforcement welded at dowels

BDS EN 206/NA:2017, Appendix F1 (informative) formulates subsequent recommendations to the limit values of the concrete mix design concerning the efficient resistance to various aggressive agents due to the surrounding and working medium. Comparing the specific requirements to concrete in accordance with the degree of impact of individual aggressive factors, the dominated requirement is that prompted by the class reflecting a chemical attack – XA3. The latter yields the use of concrete with compressive strength class

C35/45, minimal sulfate resistant cement contents of 360 kg/m³ and maximal water/cement ratio 0,45.

Table 1 Generalized specification of the aggressive site-factors due to exploitation and surrounding medium

Class specification	Medium description
Corrosion due to carbonation XC4 ^{*)}	Cyclic wetting and drying
Corrosion due to non-sea water chlorides XD1	Moderate humidity
Corrosion due to sea water chlorides XS1	Impacts of air salts without a contact with sea water
Impacts due to freezing/thawing with or without thawing agents XF1	Moderate water saturation in the absence of a thawing agent
Chemical attack XA3 ^{**)}	Environment yielding strong chemical aggression

Note:

^{*)} Intense carbonation due to technological factors and operated by CO₂.

^{**)} Intense complex chemical attack of different character, for instance attack of nitrogen oxides and chlorine compounds in the air

It is necessary to take into account that, in accordance to **BDS EN 1990:2003 Eurocode: Foundations of construction design, Item 2.3 (Designed exploitation term), Table 2.1**, retained walls should be specified as installations with a post-repair exploitation term of "category 4" (corresponding to exploitation term of 50 years).

Here it should be adequately taken into account that the main load-bearing reinforcement of the structure after undertaking the repair and restoration measures envisaged in the project is intact - respectively cleaned of corrosion, restored in detail in case of interruption, reliably protected by applying a new anti-corrosion coating. In addition, on the treated main reinforcement, a steel welded mesh is additionally mounted in detail - for the panels from the inside of the warehouse.

In this case, the following procedure refers to determining the thickness of the concrete cover above the outer surface of the additional steel meshes, which, in practice, provides an additional reserve with respect to the currently obtained new concrete cover of the main supporting steel reinforcement in the structure.

Concrete cover is the distance from the surface of the outer reinforcement (including joints, stirrups and surface reinforcement) to the closest concrete surface. The nominal concrete cover (**C_{nom}**) is found as a sum of the minimal cover (**C_{min}**) plus deviation supplement (**ΔC_{dev}**):

$$C_{nom} = C_{min} + \Delta C_{dev}$$

The minimal concrete cover (**C_{min}**) should transfer cohesion forces, provide steel protection against corrosion (durability) and fire resistance. The adoption of the maximal value is mandatory:

$$C_{min} = \max\{C_{min,b}; (C_{min,dur} + \Delta C_{dur,\lambda} - \Delta C_{dur,st} - \Delta C_{dur,add}); 10 \text{ mm}\}$$

Where:

C_{min,b} - minimal cover providing cohesion (see **Table 4.2. of BDS EN 1992-1-1:2005 – considering individual non-stressed bars, it is equal to bar diameter, 28 mm in this case**);

C_{min,dur} - minimal cover protecting against environmental impacts (see **4.4.1.2. Item 5. of BDS EN 1992-1-1:2005 – considering structures, class S4, with individual non-stressed bars, corrosion class XD1, it is equal to 35 mm**);

ΔC_{dur,λ} - safety addition (see **4.4.1.2. Item 6. of BDS EN 1992-1-1:2005/NA – equal to 0 mm**);

ΔC_{dur,st} - decrease of concrete cover when using stainless steel, **equal to 0 mm**;

ΔC_{dur,add} - decrease of concrete cover by applying additional protection, no protection here, **equal to 0 mm**.

Hence, the minimal necessary thickness of concrete cover is:

$$C_{min} = \max\{28; 35; 10 \text{ mm}\} = 35 \text{ mm}$$

The deviation supplement (**ΔC_{dev}**) = 5-10 mm, or "0" under strict control, **acc. BDS EN 1992: 1-1: 2005, p. 53**.

In summary, design concrete cover has to be:

$$C_{nom} = C_{min} + \Delta C_{dev} = 35+0=35 \text{ mm}$$

4. Prescribed concrete mix design

The concrete mix design developed in the framework of this technical solution is based on the proposed technological system for repair and restoration of individual parts of the structure of the retaining walls. Concrete mix design in terms of strength-deformation ratio and as a protective ability corresponds to the specified requirements set out in **item 3. and Table 1**.

The included special chemical admixtures are aimed at improving the internal structural parameters of concrete mix and hardened concrete. **DYNAMON SX** superplasticizer based on acrylic polymers provides a low water-cement ratio with an optimal consistency of the concrete mixture. **KRYSTALINE Add1 HD** internal crystallization chemical admixture provides a constantly increasing degree of impermeability of the concrete section over time based on additional chemical interactions in the hardened concrete in contact with water of different origin and / or moisture from the air [1,2]. **KEPTONITE** is innovative shrinkage compensating agent, hydro-oxid type [3]. **KMC** is thixotropic polymer modifier increasing mix compactness with shotcrete rebound reducing action.

The mix design is proposed in **Table 2**:

Table 2 PRESCRIBED MIX DESIGN of polymer-modified hybrid fibre-reinforced fine-grained concrete with internal-crystallization chemical admixture and shrinkage compensating agent for "wet" shotcreting (**D_{max}** = 8 mm) with compressive strength class C35/45 and resistant to environmental aggressive agents XC4, XA3, XF4, XD1, XS1

COMPONENTS	QUANTITY, kg/m ³
Sulfate resistant Portland cement CEM I 42,5 SR5, DEVNYA	550
River sand, SILISTRA, "POLARIS - 8", fraction 0-4 mm	745
Crashed washed sand, ESKANA, "Sini vir ", fraction 0-4 mm	300
Crashed stone, PATSTROY, town of Karnobat, fraction 4-8 mm	505
Highly water reducing admixture DYNAMON SX, MAPEI, Italy – 0,6% of cement mass	3,30
PP fibers BelMix 12/18 mm, Belgium	1,0
KMC – thixotropic polymer modifier increasing mix compactness (water solution of 10% dry compound – conforming to a prescription)	10,00
Internal-crystallization chemical admixture KRYSTALINE Add1 HD, Spain	1,00
Shrinkage compensating agent KEPTONITE, BG	15,00
Mixing water for dry aggregates	~190
Consistency (regarding to slump test), cm	S2

The fibre-reinforcement with micro-polypropylene fibers **BelMix 12/18 mm**, in addition to improving all the characteristics of the concrete mixture in the pre-structural stages (homogeneity), also increases the impermeability of the concrete section, increase the frost resistance and surface hardness of hardened concrete, in addition to reducing rebound when shotcreting.

Thus, in a technically expedient and cost-effective combined way, an extremely high quality of one single stage of "wet" shotcreting of approximately 8-10 cm layer, passivating the existing and additional steel reinforcement, with the highest degree of resistance to the specified aggressive factors of the operational and environment.

The steps of repair/recovery (including reinforcement) of steel reinforced structural elements should follow the requirements of the structural design. Hence, adequate technical regulations should be developed based on the following principles - entire systematic approach, good production practice, reasonable adequacy of the offered solutions, use of materials with technical characteristics proved by a systematic control, systematic and appropriately recorded control of the executed technological operations via monitoring of the control parameters and their limit values, development of an approval system of the particular phases and stages of the technological operations and labor safety.

5. Conclusion

The proposed original technical solution is based on the latest world trends for repair and restoration work on defective reinforced concrete building structures – using an effective specially designed thin hybrid-reinforced concrete overlays with purposefully involved innovative chemical admixtures.

The joint use of internal crystallization and shrinkage-compensating admixtures is an original idea with excellent results, providing additional resource and high durability in conditions of intensive operation of load-bearing reinforced concrete structures.

This innovative solution is optimal for the operating conditions and is in full compliance with the requirements for the implementation of repair work during the continuous operation of the equipment in the plant.

Acknowledgments

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Testing of nitinol sensor on bending deformed polypropylene tube: comparison of winding methods

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Abstract: A thin nitinol wire with a diameter of 0.05 mm was used as a surface sensor indicating deformation changes of the polypropylene tube during bending. Two types of nitinol wire windings were compared: parallel placement with the longitudinal axis of the tube and transverse windings around this axis. Changes in the electrical resistance of the wire were measured during the bending deformation. The sensitivity of the nitinol sensor, its comparison with conventional wire metal resistance strain gauge made of constantan alloy, its application possibilities, as well as the advantages resulting from the different winding geometry were discussed.

KEYWORDS: NITINOL, NITINOL SENSOR, BENDING DEFORMATION, WIRE WINDING

1. Introduction

At present, so-called electric strain gauges are used to measure the mechanical stress of loaded machine parts. These are electrical elements placed on the surface of the monitored component or other structural elements that respond to deformation by changing electrical properties. Resistance strain gauges (metal or semiconductor) placed on the surface of structural elements are most often used. There, they convert the deformation caused by the mechanical load into a change in electrical resistance. The cause of changes in the specific electrical resistance of semiconductor resistance strain gauges (silicon) is the deformation of their crystal lattice induced by the action of an external force (so-called piezo resistivity) [1]. Metal strain gauges are most often constructed of constantan [2], an alloy of copper and nickel, in the form of a wire a few hundredths of millimeters in diameter, placed on an insulating pad, which is attached to the surface of mechanically loaded monitored components. Changes in electrical resistivity of constantan resistance strain gauges are caused by changes in wire dimensions (elongation and change in wire cross-section).

Our research task was the experimental development of a deformation sensor made of nitinol wire, which could be used similarly to other wire strain gauges attached to the surface of the monitored objects. This simple sensor could also be built into any part of the composite structure and thus serve to monitor and evaluate the mechanical load or damage to the object. However, the method of placing the nitinol wire on / in the monitored component as well as the winding geometry must be adapted to the expected direction of the deformation forces, the expected range of values, or the required sensor sensitivity (for example to eliminate noise).

This application of nitinol is highly innovative. While a lot of studies deal with the use of nitinol shape memory for medical purposes [3,4,5,6,7], we find only exceptionally work focused on the use of nitinol as a deformation sensor. [8,9]

In this work, we describe and discuss the partial results obtained by a single bending load of PP tubes with two different winding methods of nitinol wire, which led to the development of a sensory composite push rod.

2. Preconditions for resolving the problem

2.1. Nitinol

Nitinol is a metal alloy of nickel and titanium in a different ratio. Its typical properties are shape memory and superelasticity, which are related to the transformation temperature of a particular nitinol alloy and its transitions between the austenitic and martensitic phases [10,11]. The shape memory effect means that it will bend but will return to its original shape when heated. The austenite

transformation finish temperature (A_f) has been specified as a key characteristic and the sole parameter for predicting the nitinol material properties. A_f ranges from about 140 °C to +130 °C [12]. However, the shape memory effect is not desirable for the sensory use of nitinol wire, so it is necessary to select a nitinol alloy for this purpose whose transformation temperature is lower than that at which deformation detection will take place [13]. At temperatures above the transformation temperature of the alloy, a superelastic effect is applied to the nitinol strain gauge sensor, which is an advantage over conventional wire resistance strain gauges made of constantan. The elasticity of nitinol reaches up to 10 % [10,14]. The material relaxes along the hysteresis curve [11] and the value of the hysteresis decreases with increasing temperature [15]. The load can act repeatedly, with very little permanent deformation. It is thus possible to monitor the long-term load of cyclically stressed structural elements with an assessment of the state of fatigue of the material, hidden micro cracks, and other damages.

2.2. Comparison of constantan and nitinol wire strain gauges

A comparison of some properties of nitinol and constantan is shown in **Table 1**. The high modulus of elasticity of the constantan in comparison with the values of the modulus of nitinol confirms the relative rigidity and strength of the constantan, with which it resists loading, while nitinol responds to deformation stress with significant flexibility and elongation, up to the ultimate strength. The maximum tensile strength of nitinol is more than double that of constantan. The specific electrical resistance of nitinol is also higher at room temperature, so smaller deformation changes can be well detected electrically (e.g., using a 0.05 mm diameter wire, depending on the selected geometry, the deformation change of the one micrometer/ meter will result in an electrical change of several milliohms).

Table 1. Selected properties of constantan and nitinol [13,16,17]

Property	Constantan	Nitinol
Melting point	1210 °C	1310 °C
Tensile strength	340–535 MPa	≥ 1070 MPa
Specific electrical resistance	12–49 $\mu\Omega \cdot \text{cm}$	82 $\mu\Omega \cdot \text{cm}$
Young's modulus	115–162 GPa	41–75 GPa
Modulus of elasticity in shear	44 GPa	10.8 GPa

2.3. Strain gauge sensitivity

The sensitivity of the strain gauge is expressed by the constant k (1) [18], which in the case of constantan strain gauges is around 2. These strain gauges are used to measure deformations with relative

elongation up to approximately 2 mm/m. Nitinol wire can be used to measure deformations with relative elongation up to several cm/m. Compared to the conventional metal constantan strain gauge, nitinol shows a high sensitivity to deformation. For example, the sensitivity of a 0.05 mm diameter nitinol wire, expressed by the constant k , is approximately 100 times higher than that of constant strain gauges ($k > 200$), which is typical for semiconductor strain gauges.

$$k = \frac{\Delta R}{R_0 \cdot \varepsilon} = \frac{\Delta R \cdot l}{R_0 \cdot \Delta l} \quad (1)$$

ΔR ... the increase in the resistance of the strain gauge at the relative elongation of the object surface ε

R_0 ... strain gauge resistance at initial mechanical load

l ... the original length of the object

Δl ... elongation of the deformed object

3. Experimental

3.1. Material

The model deformed object was a 20 cm long hollow polypropylene tube resistant to the temperature with the crystalline structure at random (PP-RCT). It was manufactured by a β -nucleation process, which significantly improves the crystal structure of the random PP copolymer. The tube was reinforced with glass fiber in the middle layer, outer diameter 20 mm, wall thickness 2.5 mm. This material is characterized by resistance to wall stress at elevated temperatures, low thermal expansion, high dimensional stability, and resistance up to 90 °C.

Nitinol wire with a diameter of 0.05 mm (Fort Wayne Metals, Indiana, USA/Ireland), superelasticity (not shape memory) is declared by the manufacturer as the main property. This nitinol is intended primarily for medical and other low-temperature applications, i.e., for use at room or body temperatures. It should withstand an elongation of up to 8 % at room temperature without significant permanent deformation, or the permanent deformation after 8 % elongation should be less than 0.5 %. The ultimate tensile strength of a material is 1241 MPa at room temperature, elongation > 10 %, austenite activation temperature 10 - 18 °C. At rest, this wire has electric resistance of approximately 2.1 Ω /cm.

The described nitinol wire about 20 cm long was attached to the first PP tube using epoxy resin as a single fiber in the direction of the longitudinal axis of the tube. An 80 cm long wire was attached to the second PP tube with epoxy resin in several transverse windings symmetrically distributed in the left and right halves of the tube (2 x 6 windings + one wide central winding). To eliminate the possibility of cutting the nitinol wire during tube bending, the central part of the tube was designed so that this part was wireless and the deformation movable crosshead from above hit only the tube body. The free ends of the nitinol sensor wire were held to the screw terminal for measuring the electrical resistance on the oscilloscope.

3.2. Method

The three-point bend test of PP tubes with nitinol sensors was performed on the universal testing machine (dynamometer) Instron using the control SW Tinius Olsen Horizon. For this purpose, a special tube holder was made (Fig.1). The tubes were inserted into grooved holders at both ends so that the middle part of the tubes was in the air, and therefore the samples could bend with a maximum radius in the middle part, which was acted upon by the deformation force of the descending crosshead from above. The deformation crosshead moved from above in a direction perpendicular to the horizontally placed tube. The constant feed rate was chosen to be 0.01 mm/s up to a final load of 250 N. The

starting point was approximately 1 mm above the tubes. The total movable crosshead displacement was approximately 9.7 mm (including 1 mm above the tube), followed by the crosshead return at 1 mm/s. The final deformation, bending of the tubes with nitinol wire fixed in the direction of the longitudinal axis, was simulated in the program Blender 2.9.2.0. The tubes were modeled using Bézier curves and fitted according to reference photographs taken during the three-point bending. The dimensions of the resulting models of the bent tubes were corrected according to the parameters of the real tubes before deformation.

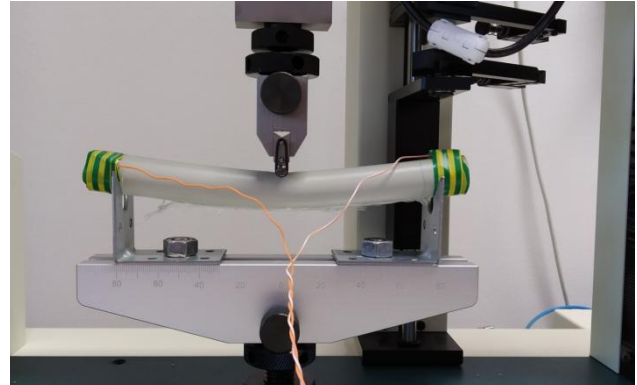


Fig.1. Three-point bend test arrangement

3.3. Results and discussion

3.3.1. Tube with axially placed nitinol wire

The online measurement of the electric resistance during the deformation test of the tube with nitinol wire placed longitudinally with a long axis is recorded in Fig.2. A graphical 3D simulation of the final tube deformation is shown in Fig.3.

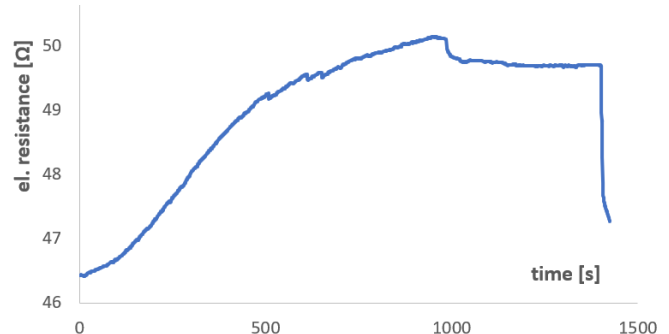


Fig.2. Recording the electric resistance of the nitinol wire placed on the tube longitudinally with its long axis during deformation

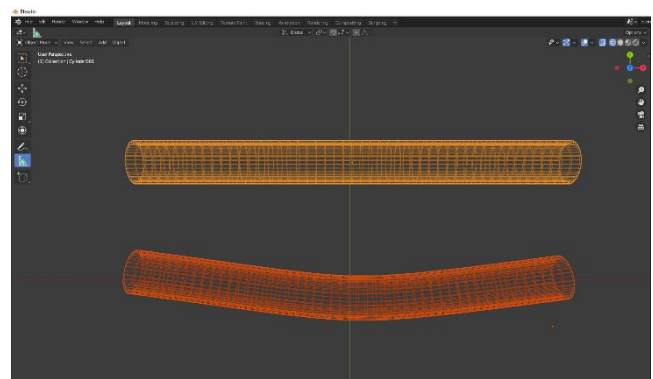


Fig.3. 3D graphic illustration of a tube before and after deformation

The calculation of the maximum bending length of the area furthest from the deformation crosshead can be simply converted to the calculation of the parabola arc length. This shape is closest to the final bend shape, because with a very slight bend (here an angle of approximately 4-7°) the outer parts of the deformed tube essentially copy the shape straight lines, while the central part is approaching the circle. Therefore, this formula (2) can be used to calculate the length of the lower arc of the bent tube:

$$L = \frac{1}{2} \sqrt{b^2 + 16 \cdot a^2} + \frac{b^2}{8 \cdot a} \ln \left(\frac{4 \cdot a + \sqrt{b^2 + 16 \cdot a^2}}{b} \right) \quad (2)$$

a ... distance between the top of the parabola and the center of the chord (here: effective length of the deformation path of the crosshead, 8.7 mm)

b ... chord length (here: original length of the undeformed tube, 200 mm)

L ... the length of the lower arc of the bent tube, L=201 mm

Suppose, therefore, that due to bending, the total elongation of the wire on the lower arc of the deformed tube occurred by approximately 1 mm. This change was recorded as an overall increase in electrical resistance of approximately 3.6 Ω. This means that a change in the length of the tested nitinol wire by 1 μm meant an increase in the value of el. resistance by several milliohms.

3.3.2. Tube with transverse winding

The record of the el. resistance measurement from the bending of the PP tube with the transverse nitinol wire winding is shown in Fig.4.

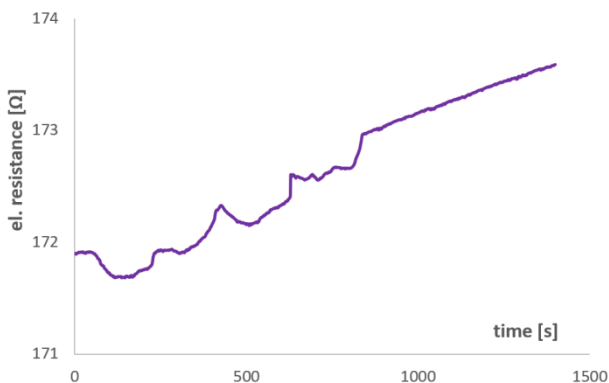


Fig.4. Recording the electrical resistance of the nitinol wire placed on the tube transversely in the wraps during deformation

The fluctuations of the electrical resistance in the first part of the graph correspond to the situation when the bending of the tube stretches more wire in the lower part, i.e., in the area furthest from the point of the descending crosshead pressure. The wire in the winding is stretched unevenly: in the lower part it is stretched, but in the upper part of the tube (in the place with a smaller deflection) the wire is pulled centrally. This means that the wraps tend to move away from each other towards the middle part of the tube and move to the position of the winding at an angle of 45°, which is their relatively most stable position during the deformation. Continued deformation and bending lead to increase tension in the nitinol wire, which was compensated by (accidental) slippage of the wraps into this position. Each such "slip" to a more stable position will temporarily reduce the tension in the wire, which was reflected in another slight decrease in the measured electrical resistance. The maximum deformation occurs in the central part of the tube. The state, which occurred from about 850th second, corresponds to a situation where the wraps closest to the center of the tube are

already in a maximum stable position of 45° and the increase in total resistance is constant and corresponds to increasing tension and elongation of the whole wire.

If we convert the whole system of a tube with a wire winding by unrolling the tube sheath into 2D, we can compile the formula for calculating the wire length based on the Pythagorean equation (3). We calculate the difference between two hypotenuses: in the first term of the equation, the hypotenuse of the deformed system is expressed by a cathetus based on the bending length simulated by the parabola (L) and in the second cathetus expressed by the tube circumference times the number of wraps ($2 \pi r \cdot n$). In the second term of the equation, the first cathetus is equal to the length of the undeformed tube (200 mm) and the second cathetus remains the same, i.e., the circumference of the tube times the number of wraps. It follows from the formula that the more wraps (n), the less the bending of the deformed tube (L) itself influences the change in wire length.

$$\Delta L = \sqrt{(L_2)^2 + (2\pi r \cdot n)^2} - \sqrt{(L_1)^2 + (2\pi r \cdot n)^2} \quad (3)$$

ΔL ... nitinol wire extension

L_1 ... original length of the tube before deformation (200 mm)

L_2 ... length of the tube after deformation (simulated by a parabola according to formula (1), i.e., approximately 201 mm)

r ... tube / wrap radius (10 mm)

n ... number of wraps (13)

L ... length of the tube bent into the shape of a parabola (see (2))

The result is a theoretical extension of the wire of only about 0.25 mm! This change was recorded as a total increase in electrical resistance of approximately 2 ohms during the deformation.

3.3.3. Discussion

Since the total load of the tube was the same for both tested geometries (shift of the crosshead along the same path, at the same speed and to the same end load), it is possible to compare the total increase of electrical resistance during deformation, which should correspond to the total elongation of the nitinol wire. In the first case, the total increase was about 3.6 Ω (from the original 46 Ω), in the second case there was an increase of about 2 Ω (from the original 172 Ω), it means about an increase in resistance by 8 % and 1 %. Thus, the sample with the wraps showed relatively smaller deformation changes than the sample with the linearly placed wire. The reason is the fact that the largest deformation changes during bending occurred in the central part of the tube, and therefore were in fact the most tensile-stressed wire wraps that were closest to the center. Thus, the maximum elongation of the wire occurred at only a fraction of the length of the entire winding. Also, the absolute elongation of the wire in wraps is less than if the wire of the same length was stretched linearly.

4. Conclusion

Several findings emerge from this deformation test:

1. When the nitinol wire (cross-section 0.05 mm) was stretched by 1 micrometer, the electrical resistance increased by 3.6 milliohms. When using this superelastic form of nitinol wire it was possible to achieve up to 8 -10 % of elongation with around 0.5 % of permanent deformation.
2. A shorter nitinol wire placed longitudinally with the long axis of the tube records small deformations with higher sensitivity than a longer wire in the transverse wraps.
3. Nitinol sensor wire in the form of a transverse winding must be wound at an angle of 45° to provide maximum stability of the wire during its loading (tensioning) and to prevent accidental fluctuations

during deformations in the form of slippages of the upper parts of the wraps.

4. The nitinol sensor wire in the form of a transverse winding can react to random deformation deflections of the support tube in all directions. Thanks to the stable winding, the electrical resistance returns to almost the original values according to the load level, during relaxation.

5. We do not find out the direction of deformations of a tube with nitinol wire in the form of a transverse winding. We only record that the deformation has occurred.

6. In the case of a tube with a linearly placed wire, we obtain information about the direction of deformation. We will refine this sensory data when more wires will be placed in/on the tube wall linearly around the circumference and each will have its output.

The proposed nitinol sensor can replace the use of strain gauges wherever it is necessary to directly electrically measure or monitor the deformation of a solid object, as an indicator of damage to cyclically or long-term mechanically stressed parts: for example in aerospace, automotive, pressure and force measurement in pneumatic devices, during short-term and long-term loading of building structures, as sensors to assess the strength of welded or glued joints, as a part of monitored beds (sleep laboratories, home and health care of lying patients), etc.

5. Acknowledgement

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Investigation of Heat-Treated Steels Using the Magnetic Noise Method

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Abstract: This paper presents the results of an investigation conducted on samples of high-quality heat-treated carbon steel using the magnetic noise method. The objective of the work was to find the proper informative parameters for identifying the obtained structures with different hardness. To this end, samples were prepared using quenching and tempering heat treatment at different temperatures, from 150°C to 500°C. After microstructural analysis and hardness measurements, the samples were investigated using the magnetic noise method. The signals were visualized and analyzed using a digital oscilloscope and the Multi Instrument 3.8 software program. The RMS informative parameters and the registration time of the magnetic noise signals were used to identify the obtained microstructures with different characteristics and hardness.

Keywords: MAGNETIC NOISE METHOD, HEAT-TREATED STEELS, HARDNESS, INFORMATIVE PARAMETERS, RMS, REGISTRATION TIME OF MAGNETIC NOISE SIGNALS

1. Introduction

The magnetic noise method is widely used for the investigation of the microstructure of ferromagnetic materials. It is based on the phenomenon that occurs when a ferromagnetic material is magnetized [1]. The structure of a ferromagnetic material consists of magnetic domains separated by domain walls [1, 2, 3]. In the absence of a magnetic field, the domains are randomly oriented [2]. When a magnetic field is applied, the magnetic domains align in the direction of the applied field by moving their domain walls. This movement is strongly affected by the microstructural features of the ferromagnetic material such as grain boundaries, other phases, inclusions, dislocations, residual stresses, etc. [2]. As a result, abrupt magnetization and induction of voltage in the magnetic test coil occur. The resulting magnetic noise was first recorded and explained by Prof. Heinrich Barkhausen in 1919, though it took until the early 1980s to attract the attention of researchers and the industry. The advantages of this method include good sensitivity, straightforward experimental procedure and no requirements for surface preparation [1-3]. The magnetic noise signal depends on the measurement parameters, the magnetizing current and frequency, and the frequency response characteristic in the analysis of the registered magnetic noise [1]. This method also has some disadvantages related to the need to calibrate the magnetic test coil and the non-standardized approach to the measurement and selection of proper magnetic noise parameters for identifying relationships with the microstructural features of the studied material.

The analysis of the literature shows that the magnetic noise method is sensitive to the carbon content [4], the size of the crystallites [5, 6] and the different microstructures such as: ferrite [5, 7], pearlite [10, 11] and martensite [12, 13].

One or more parameters such as root mean square (RMS) voltage, energy, pulse height, position of the maximum signal as well as the parameters of the magnetic noise envelope were used for qualitative and quantitative assessment of the changes in the microhardness and the structure of the materials [2, 3, 11, 12, 13]. Thus, the Barkhausen noise analysis technique can be used to assess the surface integrity of a manufactured part. In recent years, the researchers have focused their efforts on developing scanning techniques such as the Barkhausen continuous noise method [14] or the use of a rotating coil [15], which makes it possible to expand the application of this method in industrial settings.

The objective of this paper is to search for and find the proper informative parameters of the magnetic noise signals for identifying the obtained microstructures of high-quality carbon steel after quenching and tempering heat treatment.

2. Experiments

2.1. Preparation of samples for conducting experiments

Cylindrical samples of high-quality carbon steel with a diameter of 5.5 mm and a length of 100 mm were prepared. The chemical composition of the steel type used was: C 0.42-0.5%, Si- 0.17-0.37%, Mn - 0.5-0.8%, P < 0.035%, Ni < 0.25%, Cr- < 0.25%, S < 0.04%, Cu < 0.25%. Five groups of samples were prepared, four of which were subjected to heat treatment, which consisted of quenching and subsequent tempering at the following temperatures: 150°C /group 2/, 250°C /group 3/, 350°C /group 4/ and 450°C /group 5/. The heat treatment modes are presented in column 2 of Table 1. By the means of metallographic analysis, the type of the obtained microstructure was determined. The microstructure of the non-heat-treated samples was ferrite and pearlite /Fig. 1a/.

The hardness was measured with a Zwick/Roell ZHV 10 hardness test machine under a load of 9.807 N.

Table 1: Heat treatment of investigated samples

	Heat treatment	Microstructure	Vickers hardness HV
Group 1	Non-heat-treated	Ferrite and pearlite	320
Group 2	Quenching T = 850°C Tempering T = 150°C	Tempered martensite	750
Group 3	Quenching T = 850°C Tempering T = 250°C -	Tempered martensite	620
Group 4	Quenching T = 850°C Tempering T = 350°C	Tempered troostite	400
Group 5	Quenching T = 850°C Tempering T = 500°C	Tempered sorbite	340

2.2. Experimental installation for conducting investigations using the magnetic noise method

MULTITEST-MC 04 device [16], which consists of a magnetizing unit, a converter for recording magnetic noise signals and measuring magnetic noise voltage, and a signal amplifier with several bandpass filters was used for preliminary investigations. Sinusoidal magnetization with a frequency of 76 Hz was applied. A cyclic sinusoidal field was induced in the sample using a magnetic-noise converter. The Barkhausen signals were visualized and recorded by the means of a digital oscilloscope VT DSO 2810 at a sampling rate of 100 MSPS and the data acquisition software Multi-Instruments Pro 3.8.

3. Results and Discussion

3.1. Results from the performed heat treatment

The results from the performed heat treatment and metallographic analysis are presented in Table 1. Columns 3 and 4 show the type of the obtained microstructure and the mean values of the measured hardness (HV), respectively.

After quenching and tempering at $T = 150^{\circ}\text{C}$, the obtained microstructure was tempered martensite with a hardness $\text{HV} = 750$ /Fig. 1b/; after quenching and tempering at a temperature of 250°C , the result was tempered martensite with a hardness $\text{HV} = 600$ /Fig. 1c/; after quenching and tempering at temperatures of 350°C и 500°C , the result was tempered troostite /Fig. 1d/ and tempered sorbite /Fig. 1e/, respectively.

The changes in the structure after a low-temperature tempering are related to the decomposition of martensite, the reduction of the carbon content in the main volume of the martensite and the beginning of the formation of iron carbides [17]. The processes that take place during tempering at temperatures of $200\text{--}250^{\circ}\text{C}$ can be explained by the decomposition of residual austenite, which results in the reduction of the internal stresses in the structure and lower hardness. When tempering is performed at temperatures higher than 250°C , the existing martensite decomposes to ferrite and cementite. The troostite formed by tempering at intermediate temperatures / $T = 350^{\circ}\text{C}$ / is a finely dispersed mixture of ferrite and cementite. With tempering at temperatures of 500°C and above, the formed carbides coagulate and the result is a dispersed mixture of ferrite and cementite called tempered sorbite. The hardness decreases, but the impact strength and ductility increase.

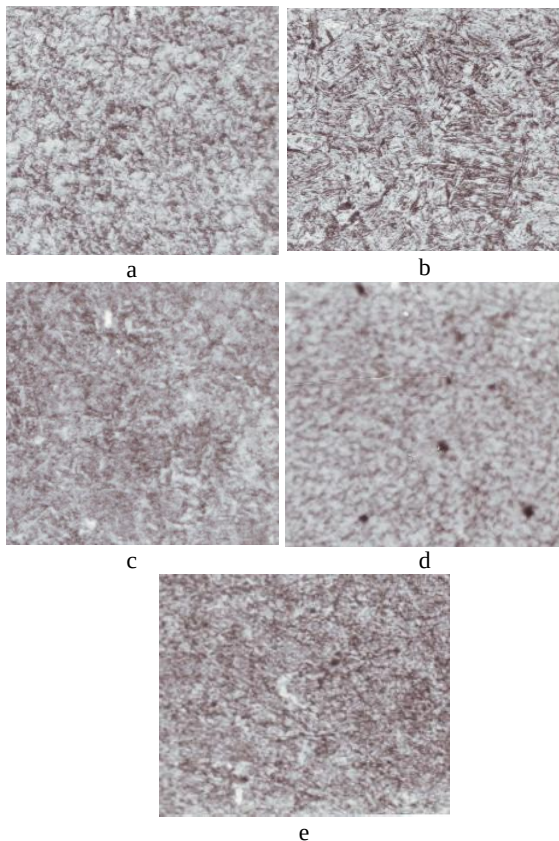


Fig. 1 The microstructure of the tested samples; initial ferritic and pearlitic structure before heat treatment (a), martensitic structure after quenching and low-temperature tempering at $T = 150^{\circ}\text{C}$ (b), martensitic structure after quenching and tempering at $T = 250^{\circ}\text{C}$ (c), tempered troostite - structure after quenching and tempering at $T = 350^{\circ}\text{C}$ (d), tempered sorbite - structure after quenching and tempering at $T = 500^{\circ}\text{C}$ (e).

3.2. Results of a study using the magnetic noise method

Tests were conducted using the magnetic noise method. The samples described in paragraph 2.1 were analyzed. The frequency of the magnetizing field did not change during the tests. In order to select the optimal magnetization level, the samples were magnetized using magnetizing currents ranging from 100 to 250 mA. A series of magnetic noises was recorded during each magnetization. The magnetization parameters had an effect on the count of the pulses as well as on their size and shape.

To analyze the magnetic noise (MN) parameters, 6 signal bursts were used for each sample, each of them corresponding to half a cycle of magnetization. A bandpass filter of up to 100 kHz was used. The following parameters were used for the preliminary assessment of the magnetic noise signals:

- the RMS value - calculated by the following formula:

$$RMS = \sqrt{\frac{1}{n} \sum_{i=0}^{n-1} x_i^2}$$

as x_i is the amplitude of the magnetic noise signal that passes through the bandpass filter.

- Magnetic noise counts
- Mean width of the magnetic noise signal bursts.

Figure 2 shows part of the recording performed during the magnetization of the tested samples and the magnetic noises that occur at a magnetization current of 200 mA.

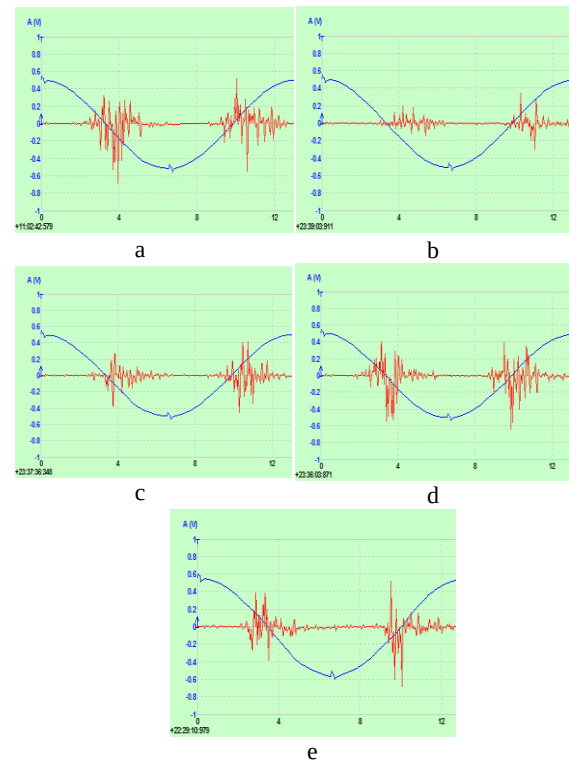


Fig. 2 Recordings of magnetic noises over time obtained from the tested groups of samples at a magnetizing current of 200 mA: specimens group 1 (a), group 2 (b), group 3 (c), group 4 (d), group 5 (e).

The results of the analysis of the magnetic noise parameters for the first group of samples for three magnetization cycles are presented in Table 2. As the magnetizing current increases, the RMS value and the bandwidth of the magnetic noise also increase, but the magnetic noise count decreases /Table 2, Fig. 3/.

In the case of half a cycle of magnetization, the sample is demagnetized and then magnetized in the opposite direction. This activates the movement of the domain walls, which overcome the obstacles posed by the different microstructural phases. Under the alternating magnetic field, the generation and the proliferation of magnetic domains are affected by the magnetic polarity reversal. This process depends on the microstructural features, the different phases, the boundaries between the crystallites, the dislocations, the presence, shape and size of the carbide inclusions, the carbide grains, etc. The maximum amplitude and the RMS values of the magnetic noises depend on the magnitude of the magnetic domain jumps with the corresponding magnetizing current.

Table 2: The results of the analysis of the magnetic noise parameters for the first group of samples

I_n, mA	RMS	MN Count	Pulse width, mks
100	79.84	1425	21
150	129.5	1031	25
200	166.3	930	33
250	191.8	900	29

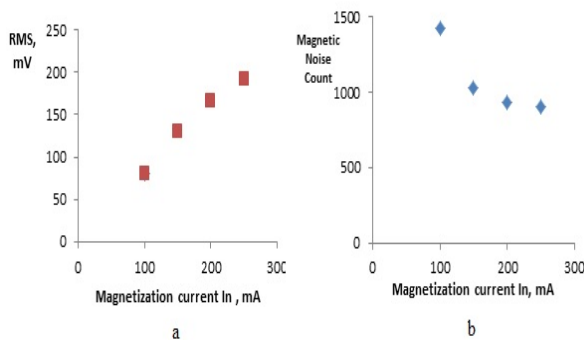


Fig.3 Change in the RMS /a/ and the magnetic noise counts /b/ with the increase of the magnetizing current for one magnetization cycle in samples before heat treatment.

Figure 4 shows the change in the RMS of the magnetic noise depending on the magnetizing current in the tempered samples. A high degree of distinctness is observed at a magnetizing current of 150 mA. The samples subjected to quenching and low-temperature tempering which have tempered martensite structure feature the lowest RMS values. In the other groups of samples /3, 4, 5/, an increase in the RMS value is observed, which can be explained by the type of the obtained microstructures of the tempered martensite, troostite and sorbite.

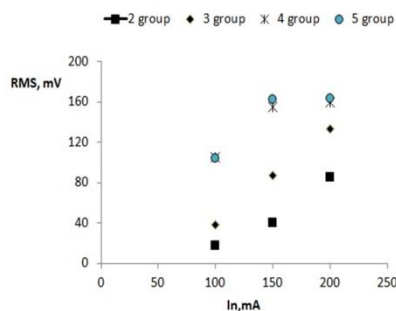


Fig.4 Change in the RMS of the magnetic noises depending on the magnetizing current in samples subjected to heat treatment

The recorded magnetic noise signals feature a different time of noise occurrence depending on the magnetization current sine wave.

Therefore, signal processing should be performed. The signals were smoothed and normalized. After the signal processing, the noise envelopes and the peak values were determined.

The time of magnetic noise occurrence, determined from the maximum value of the signal envelope relative to the zero value of the magnetization field, is defined as the parameter (Δt), which is used to identify the microstructure type. Figure 5 shows the envelopes of the magnetic noises of the samples from the tested groups. There is a shift of the peak values at the field value of zero as well as differences in the shape of the envelope. In the tempered martensite structures, the peak value is small and the magnetic noise envelope is wide. In the transition from tempered martensite with high hardness to troostite and sorbite with hardness close to the initial hardness, there is an increase in the peak values and sharpening of magnetic noise envelope at magnetic field values around zero.

The differences in the shift over time as well as in the shape of the magnetic noise can be explained by the irreversible movement of the domain walls when overcoming obstacles due to the different microstructures, i.e. the microstructure of the material directly affects the dynamic behavior of the domains during magnetization.

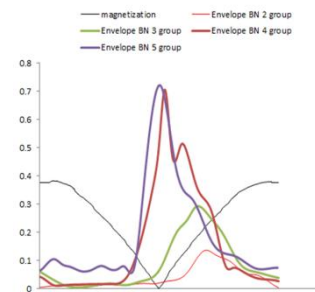


Fig.5 Magnetic noise envelopes determined during magnetic polarity reversal with $I_n = 150 \text{ mA}$ in the tested groups of samples with a different structure

Figure 6a shows the changes in the time of magnetic noise occurrence parameter (Δt) for group 1 which has ferrite and pearlite structure in an equilibrium state, and Figure 6b shows the results for the shift over time in the other groups.

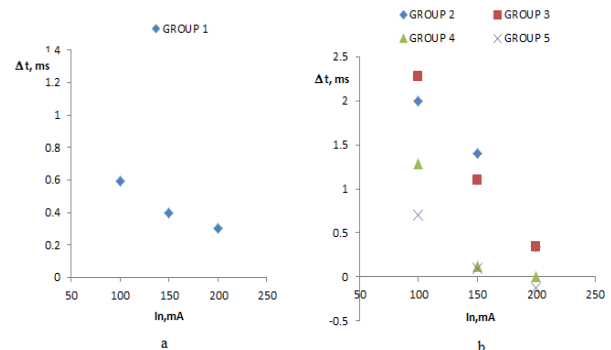


Fig. 6 The changes in the time of magnetic noise occurrence parameter (Δt) for investigated group samples

The greatest shifts over time occur at low magnetizing currents with the martensite structure obtained by low-temperature tempering. According to [12, 13, 14, 18], the magnetization of martensite results in many small domains due to the characteristics of martensite and the presence of multiple martensite needles.

Martensite features a high density of dislocations and high internal stresses. That is why a greater value of the magnetizing field is needed in order to move the domains and create new ones.

The obtained peak value of the magnetic noise is small and it occurs at relatively higher values of the magnetizing current. This also explains the different shapes of the magnetic noise in the martensitic structure shown in Figure 5.

Magnetic polarity reversal requires a higher value of the magnetic field and the displacements of the domain walls are small due to the characteristics of martensite. The morphological change from martensitic needles to ferritic and pearlitic structures in the form of troostite and sorbite makes the magnetic structure larger and the average size of the domain walls increases [18]. Magnetic polarity reversal becomes easier. At a magnetizing current of 200 mA, the amplitude of the magnetic noise signal increases significantly and the values of the parameter (Δt) get close to zero.

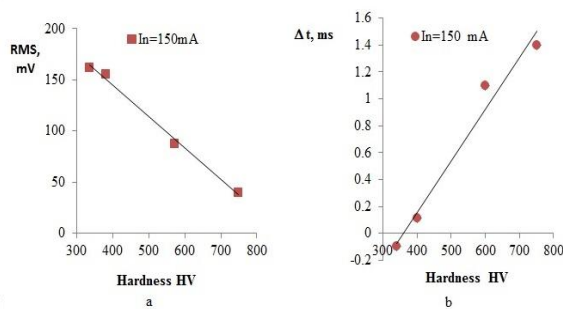


Fig. 7. Change in the RMS parameters /a/ and the shift over time of the occurrence of the magnetic noises (Δt) /b/ depending on the hardness of the obtained structures at a magnetizing current of 150 mA.

Figure 7 shows how the RMS informative parameters and the time of occurrence of magnetic noises, determined relative to the zero value of the magnetic field, change depending on the hardness of the tempered samples. These dependencies can be represented by a linear relationship. The proposed informative parameters characterize the changes in the structure and can be used to assess the hardness as well as for qualitative assessment of the type of the microstructure of structural medium-carbon steels after tempering.

4. Conclusions

By studying tempered samples using the magnetic noise method, data for a series of voltage pulses as a function of time and the related values of the magnetizing field were obtained. Various parameters such as the RMS of the noise, magnetic noise counts per magnetization cycle, etc., were used to characterize the noise signals. It was found that the time of noise registration, as determined from the zero point of the magnetization sine wave to the maximum value of the magnetic noise envelope, is a suitable informative parameter for the analysis of microstructures. After signal processing, the values of this parameter were determined and a relationship in their behaviour was found to exist due to the magnetic polarity reversal processes in the microstructures obtained after tempering. The study found linear relationships between the RMS, the time of magnetic noise occurrence and the hardness of the structures obtained after tempering.

Acknowledgements

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Mesoporous transition metal oxide composites for effective remediation of air from VOCs

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Abstract: This study is aimed at the preparation of mesoporous Cu-Ti-M oxide composites ($M = \text{Ce, Zr, Sn}$) by combination of template assisted hydrothermal technique with the conventional impregnation or novel "chemisorption-hydrolysis" techniques. The obtained materials are characterized by nitrogen physisorption, XRD, SEM, FTIR, HRTEM, XPS, TPR and tested as catalysts for ethyl acetate oxidation, as a probe VOCs molecule. The regulation of the microstructure, surface and redox properties and the related catalytic behavior of the composites by changing of the metal oxide dopant and preparation technique used is discussed in details.

Keywords: VOCs ELIMINATION, CATALYSTS, MESOPOROUS METAL OXIDE COMPOSITES, EFFECT OF DOPANT

1. Introduction

The accelerated urbanization and industrialization provide a permanently increasing problem with the environmental pollution. In response, the recent worldwide conventions related to the climate changes, ratified the need of radical reduction of the greenhouse emissions via adequate economic, social and scientific approaches [1,2]. These conventions determined the volatile organic compounds (VOCs) as the dominant air pollutants due to their toxic, volatile and carcinogenic features [3]. Recently, the application of catalytic and catalytic-hybrid technologies have been recommended as the most effective strategies for the VOCs elimination [4]. Due to the lower price, resistance to poisons and capability of regeneration, the transition-metal based catalysts have been considered as promising alternative to the noble ones [4,5]. Nowadays, nanotechnology opens new prospects in the optimization of the catalysts formula for the effective remediation of air from VOCs. Here, the nanostructured porous metal oxide composites gain a considerable attention due to their unusual texture, morphology and structural characteristics and the related with them, superior redox, acidic-base and catalytic properties [5,6].

This study is aimed at the development of novel strategy for the preparation of nanostructured mesoporous $\text{CuO-TiO}_2\text{-MO}_2$ ($M = \text{Ce, Zr, Sn}$) composites. A template assisted hydrothermal technique was used for the preparation of $\text{TiO}_2\text{-MO}_2$ mixed oxides with equimolar M/Ti ratio, followed by chemisorption, hydrolysis and decomposition of copper ammonia complex on them. Alternatively, the conventional incipient wetness impregnation technique was applied for copper deposition. Special attention was paid on the relation between the microstructure, surface and redox properties of the obtained materials and their catalytic behavior in total oxidation of ethyl acetate, as a representative VOCs. For the purpose, nitrogen-physisorption, XRD, SEM, HRTEM, FTIR of adsorbed pyridine, XPS and TPR analyses were combined and discussed in details.

2. Materials and Methods

2.1. Materials

Ceria-titania, zirconia-titania and tin oxide-titania mesoporous oxides with M/Ti ($M = \text{Ce, Zr, Sn}$) molar ratio of 1 were synthesized using aqueous solutions of TiCl_4 and MCl_4 as precursors and N-hexadecyl-N, N, N-trimethylammoniumbromide (CTAB) as a template according to the procedures described in [7-9], respectively. After the precipitation of the mixture with 25% solution of ammonia, the sludge was subjected to treatment at 373 K for 24 h in a closed container. The obtained solid was calcined at 773 K for 10 h. Copper ammonia complex was prepared from $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ and aqueous solution of ammonia as described in [7]. Then, the $\text{TiO}_2\text{-MO}_2$ solid was added and cooled in ice bath up to 273 K. The product was diluted with water and the solid was subjected to calcination at 773 K for 4 h. The materials obtained by using of this chemisorption-hydrolysis (CH) procedure were denoted as CuTiM_A . Incipient wetness impregnation (WI) of the supports with aqueous solution of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ and calcination at 773 for

4 h was applied for the preparation of the samples from the CuTiM_W series. For all modifications, the overall amount of copper was 8 wt.%.

2.2. Methods

Nitrogen physisorption measurements were performed on a Beckman Coulter SA 3100 apparatus. A Bruker D8 Advance diffractometer with $\text{Cu K}\alpha$ radiation and a LynxEye detector with constant step of $0.02^\circ 2\theta$ and counting time of 17.5 s per step was applied for the powder X-ray diffraction analyses. The SEM investigation was carried out on a FEI Nova NanoSEM450. A FEI Talos F200X transmission electron microscope was applied for the HRTEM analyses. The XPS measurements were carried out on a AXIS Supra electron spectrometer (Kratos Analytical Ltd.) using monochromatic $\text{AlK}\alpha$ radiation with a photon energy of 1486.6 eV. The pyridine desorption spectra were carried out with a BioRadFTS-60 spectrophotometer equipped with mid-IR MCT detector [9]. The TPR/TG (temperature-programmed reduction/thermo-gravimetric) analyses were performed on a Setaram TG92 instrument in a flow of 50% H_2 in Ar with heating rate of 5 $\text{K}\cdot\text{min}^{-1}$.

2.3. Catalytic test

The total oxidation of ethyl acetate was carried out in a flow type apparatus in a flow of 1.21% ethyl acetate (EA) in argon. The obtained products were analyzed on-line by HP 5890 GC, equipped with flame ionization detector. Absolute calibration method and carbon based material balance were used for the elucidation of the conversion and products distribution.

3. Results and Discussion

The texture characteristics of the obtained $\text{TiO}_2\text{-MO}_2$ mixed oxide supports and the corresponding copper modifications were studied by low-temperature nitrogen physisorption (Fig. 1). According to the IUPAC classification, the isotherms are of IV type, which is typical of mesoporous materials.

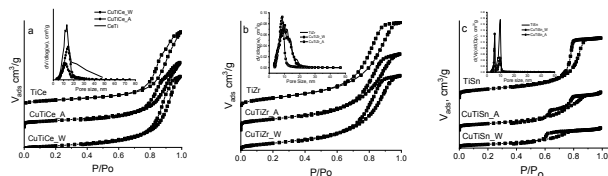


Fig. 1 Nitrogen physisorption isotherms and pore size distribution (inset) of the obtained composites.

The shape of the hysteresis loop was of type H3 for $\text{TiO}_2\text{-CeO}_2$, H1 for $\text{TiO}_2\text{-ZrO}_2$ and H2 for $\text{TiO}_2\text{-SnO}_2$ based composites, which could be attributed to the presence of irregular inter-particle, cylindrical-like or "bottle"-like mesopores, respectively. The BET surface area (Table 1) and total pore volume were much larger for the $\text{TiO}_2\text{-ZrO}_2$ support, evidencing the superior role of ZrO_2 additives as structural promotor of titania. After their modification with copper, the decrease in the BET surface area and total pore

volume (Table 1) combined with some changes in the shape of the desorption branch of the isotherms and pore size distribution (Fig.1), indicate pore blocking due to the deposition of copper particles within the mesopores. This effect was more pronounced for the $\text{TiO}_2\text{-CeO}_2$ and $\text{TiO}_2\text{-SnO}_2$ composites, most probably due to the lower BET surface area and domination of pores with diverse diameter along them, which provokes the agglomeration of CuO particles.

Table 1. BET surface area (S_{BET}), total pore volume (V_{tot}) and phase composition for various samples.

Sample	S_{BET} , m^2g^{-1}	V_{tot} , cm^3g^{-1}	Phase	Particle size, nm
TiCe	99	0.45	cerianite	12
CuTiCe_W	69	0.39	cerianite tenorite	12 45
CuTiCe_A	76	0.33	cerianite	12
TiZr	248	0.69	-	-
CuTiZr_W	187	0.60	-	-
CuTiZr_A	193	0.59	-	-
TiSn	107	0.32	cassiterite	5
CuTiSn_W	70	0.18	cassiterite	5
CuTiSn_A	79	0.20	cassiterite	5

In Fig. 2 are presented XRD patterns of the obtained composites and data for the phase composition and average crystallite size are listed in Table 1.

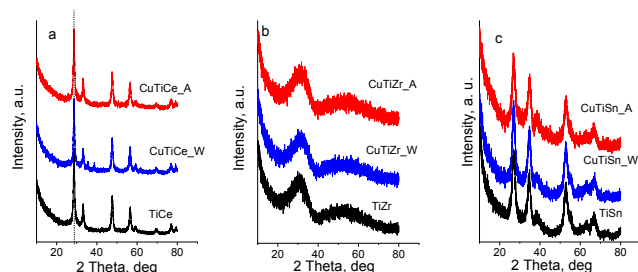


Fig.2. XRD patterns of the obtained composites.

The broad lines in the patterns of all $\text{TiO}_2\text{-ZrO}_2$ based samples (Fig. 2) is attributable to the well-dispersed phase with amorphous structure, which well corresponds to the superior texture characteristics of these materials (Table 1). Small reflections, typical of cassiterite phase with average crystallite size of 5 nm, were detected for the $\text{TiO}_2\text{-SnO}_2$ composites. The well-defined reflections at $2\theta=28.6^\circ$, 33.1° , 47.6° and 56.4° in the patterns of $\text{TiO}_2\text{-CeO}_2$ composites could be attributed to the partially substituted with Ti^{4+} cerianite phase (PDF 00-065-5923). The absence of reflections of any copper-containing phase indicates that it is highly dispersed within the $\text{TiO}_2\text{-MO}_2$ supports. Only in the case of $\text{TiO}_2\text{-CeO}_2\text{-W}$, small reflections of tenorite phase (PDF 00-89-5899) with average crystallite size of 45 nm was observed.

The morphology of the samples was well visualized by SEM (Fig. 3) and HRTEM-EDS (Fig 3, inset) images. A "coral-like" structure of $\text{TiO}_2\text{-ZrO}_2$ support with homogeneously dispersed small copper entities in it was observed for both CuTiZr composites. Cubic-like CuO aggregates are well-distinguished on the $\text{TiO}_2\text{-CeO}_2$ and $\text{TiO}_2\text{-SnO}_2$ supports, when the WI procedure was applied. CuO particles with needle-like morphology were detected for their CH obtained analogues. Nevertheless the similarity in the BET surface area of both supports, the copper particles were much smaller in case of $\text{TiO}_2\text{-SnO}_2$, where more homogeneous penetration of the individual TiO_2 and SnO_2 oxides in solid solution was also observed. All these results were in line with the data, obtained by the XRD and nitrogen physisorption analyses (Table 1).

The variations in the morphology of the copper particles, obtained by diverse deposition procedure, could be associated with different mechanism of their formation [7]. Stabilization of Cu^{2+} ions by the basic oxygen ions in the interstitial positions of the

$\text{TiO}_2\text{-MO}_2$ solid solution is probably realized during the incipient wetness impregnation procedure. Here, segregation of loosely interacted with the support copper entities with the formation of larger CuO crystallites on the external surface, is expected as well.

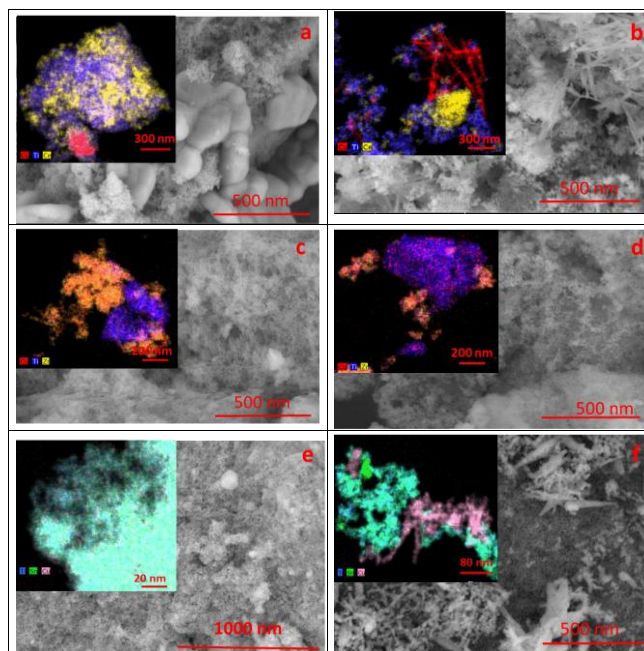


Fig. 3 SEM and HRTEM-EDS (inset) images of CuTiCe (a,b); CuTiZr (c,d) and CuTiSn (e,f) composites prepared by WI (left) and CH (right) techniques.

During the chemisorption-hydrolysis procedure, interaction between the copper ammonia precursor and surface OH acidic groups of the support is could be realized. This provokes growth of the needle-like CuO crystallites [7], as was also demonstrated by the SEM and HRTEM analyses (Fig. 3). Obviously, their dispersion is controlled by the texture characteristics and surface acidity of the $\text{MO}_2\text{-TiO}_2$ support.

In Table 2 are listed data from the FTIR study of adsorbed pyridine for the various $\text{TiO}_2\text{-MO}_2$ mixed oxides [7-9]. The amount of Lewis acidic sites, which dominated in all materials, increased as follows: $\text{TiSn} < \text{TiCe} < \text{TiZr}$. This order was also preserved after the normalization of the acidity per unit surface area. This evidences the specific influence of the dopants in titania not only on its texture characteristics, but also on the variation of the surface acid-base properties.

Table 2. Pyridine adsorption on various supports

Sample	mmolPy/g	mmolPy/ $\text{m}^2 \cdot 10^{-3}$
TiCe	0.08	0.79
TiZr	0.30	1.20
TiSn	0.05	0.47

XPS analyses were performed for the elucidation of the surface composition of the samples. For all of them, Ti 2p, Sn 3d, Ce 3d and Zr 3p XPS spectra were recorded.

The Ti 2p spectra consisted of two peaks at ca. 458.5 and 464.2 eV, attributable to $\text{Ti } 2p_{3/2}$ and $\text{Ti } 2p_{1/2}$ spin-orbit peaks. The $\text{Ti } 2p_{3/2}$ peak was successfully fitted with two components with binding energies (BE) of 458.5 and 455.9 eV for TiO_2 and Ti_2O_3 , respectively [10].

The Ce 3d core level spectra are fitted with ten peaks attributable to Ce $3d_{5/2}$ and Ce $3d_{3/2}$ spin-orbit splitting in CeO_2 and Ce_2O_3 [11].

The peaks with BE of about 331.9-332.1 eV in the Zr 3p spectra correspond to Zr $3p_{3/2}$ in ZrO_2 phase [12].

The Sn 3d spectra exhibited doublet peaks at ca. 486.1 and 494.5 eV, corresponding to Sn 3d_{5/2} and Sn 3d_{3/2} spin-orbit splitting. The Sn 3d_{5/2} peaks were well-fitted with three components with BE of 486.6, 485.9 and 485.0 eV which could be assigned to Sn⁴⁺ and Sn²⁺ in different environment [13].

The Cu 2p spectra in the copper containing materials were deconvoluted in two peaks with BE of 934.8 and 932.8 eV, which are ascribed to copper ions in CuO and Cu₂O, respectively [14].

The presence of ions in different oxidative state for all materials clearly evidences strong interaction between the individual components in them. This could be realized *via* replacement of the lattice metal ions by the foreign ones or *via* insertion of the latter in the interstitial lattice positions. Obviously, the resulted lattice deformations and the need of recovery of the lattice charge balance provoke the formation of oxygen vacancies and this process is controlled by the metal ions characteristics. In addition, electron transfer due to the diverse electron density around the different metal ions is expected.

In Fig. 4a is presented the surface ions distribution, determined by the XPS analyses. Generally, the "chemisorption-hydrolysis" procedure provoked higher exposure of copper phase on the surface, which well corresponded to its higher dispersion, elucidated on the base of the XRD (Fig. 2) and HRTEM (Fig. 3) analyses.

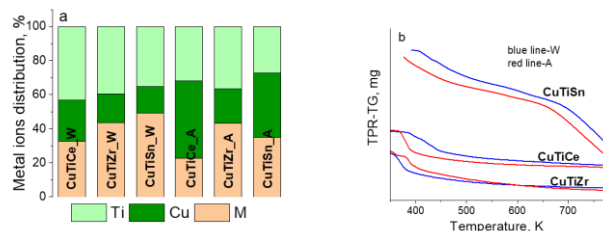


Fig. 4 Metal ions distribution determined by the XPS spectra (a) and TPR-TG profiles (b) for the obtained CuTiM composites.

For these samples, the Zr/Ti ratio was close to the expected equimolar one, indicating formation of homogeneous TiO₂-ZrO₂ solid solution, which is probably provoked by the similarity in the metal ions radius. The preservation of this ratio after the modification evidences almost random distribution of the copper species within the solid solution. In line with the nitrogen physisorption analyses (Fig. 1, Table 1), the extremely lower copper exposure for the CuTiZr composites, despite the modification procedure used, could be attributed to copper blocking deeply into the "cylindrical"-like mesopores of the TiO₂-ZrO₂ support.

The deviation in the M/Ti ratio for the TiCe and TiSn composites could be attributed to "core-shell" deposition of SnO₂ over TiO₂ in the TiSn support, or *vice versa* for the TiCe one, most probably due to diverse precipitation rates of the corresponding precursors. Obviously, the lower density of the surface acidic groups in the TiSn support (Tables 1, 2) ensures higher dispersion of the loaded on it copper species (Figs. 2, 3). In agreement with HRTEM (Fig. 3) and XRD (Fig. 2) analyses, the highest copper exposure in the CuTiCe composites is probably related to the formation of larger CuO aggregates.

The TPR-TG profiles of copper modifications in hydrogen are shown in Fig. 4b. Two main effects with a maximum at ca. 390 and 430 K were detected for CuTiCe_W. They are attributable to the reduction of CuO particles with different dispersion to metallic copper [15]. The peaks were shifted to lower temperature for CuTiZr_W, which well corresponds with the higher dispersion of the copper phase in it (Figs. 2, 3). Only one narrow TG effect was observed for their CH obtained analogues, evidencing presence of uniform, small and well-crystallized CuO particles. The TPR effect for CuTiZr_A was broader, but shifted to lower temperatures. In accordance with the nitrogen physisorption (Fig. 1), XRD (Fig. 2), XPS (Fig. 4a), SEM and HRTEM (Fig. 3) analyses this could be

assigned to the presence of very finely dispersed copper oxide crystallites, which are partially blocked in the mesopores. Rather different are the TPR profiles for both CuTiSn composites. Here, a continuous weight loss is observed above 400 K, and it corresponds to simultaneous reduction transformations with the CuO particles and TiO₂-SnO₂ support. These effects are shifted to about 100 K lower temperature as compared to the pure TiO₂-SnO₂ support (not shown), which probably originates to the synergistic reduction activity of the adjacent CuO and SnO₂ particles.

In Fig. 5a are presented the temperature dependencies of ethyl acetate (EA) oxidation on various CuTiM composites.

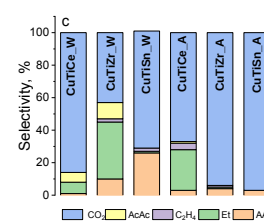
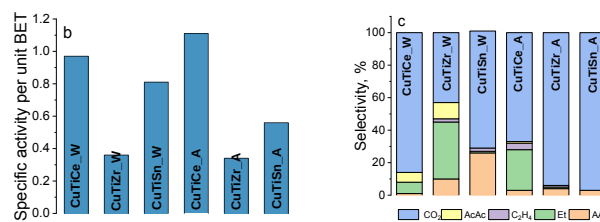
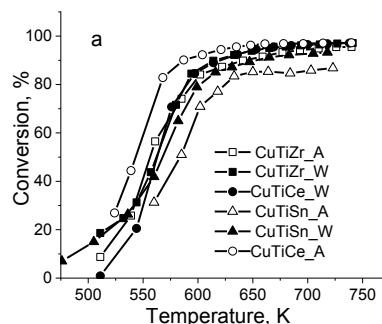


Fig. 5 Temperature dependencies of ethyl acetate oxidation (a); specific catalytic activity per unit surface area at 575 K (b) and products distribution at 50% conversion for various CuTiM composites.

More of the catalysts slightly differed in their catalytic activity despite the variations in their composition. CuTiCe_A demonstrated the highest catalytic activity, while the lowest one was detected for CuTiSn_A.

In order to precise the discussion and to diminish the effect of the differences in the BET surface area of the composites (Table 1), a specific catalytic activity (SA) per unit surface area was calculated (Fig. 5b). Both CuTiCe samples exhibited extremely high specific catalytic activity and here, a facile effect of the CH procedure for the copper deposition was observed. Just the opposite, the traditional incipient wetness impregnation technique provided the production of catalysts with improved SA when TiO₂-SnO₂ mixed oxide was used as a support. The SA of CuTiZr composites was slightly influenced by the preparation procedure and among the studied composites, both of them demonstrated the lowest SA.

For all catalysts, the main product of the EA oxydation was CO₂, but acetaldehyde (AA), ethylene, ethanol (Et) and acetic acid (AcAc) in different proportions were also detected as byproducts (Fig. 5c). The best selectivity to CO₂ was found for CuTiSn_A and CuTiZr_A samples, while their TiCe based analogue possessed higher ability to ethanol formation. The samples, obtained by the conventional incipient wetness impregnation technique produced CO₂ with relatively lower selectivity. Ethanol and acetic acid were the main byproducts for CuTiCe_W and CuTiZr_W, while a significant portion of acetaldehyde was detected on CuTiSn_W.

The variations in the SA of the samples (Fig. 5b) and the products distribution (Fig. 5c) at selected conversion clearly indicated differences in the catalytic sites in them. It is well known that the EA oxidation is a step-wise process, where a hydrolysis of ethyl acetate with the activity of surface acidic sites is realized as an

initial step, followed by oxidation of the produced ethanol and acetic acid to acetaldehyde and CO₂ [7]. Obviously, the strength and the proportion between the surface acidic sites and the redox centers in the composites is of the strategical importance for the optimization of the catalysts performance in total oxidation of EA, with minimum formation of hazardous by-products. A synergistic activity of the TiO₂-MO₂ supports and the loaded on them copper species is expected, which could be controlled by the accessibility of the active sites to the reactants in the mesoporous structure.

Thus, the existence of high amount of Lewis acidic sites in the titanium doped cerianite type TiCe support (Table 2) provokes the hydrolysis of ethyl acetate to acetic acid and ethanol, but the formation of larger CuO aggregates on the support external surface (Figs. 2, 3) suppresses the redox activity (Fig. 4b). As a result, these composites possess a significantly high catalytic activity per unit surface area, but low selectivity in total oxidation to CO₂.

For the CuTiSn composites, the facile electron transfer in the conjugated Sn⁴⁺-Sn⁰ and Cu²⁺-Cu⁰ redox pairs (Fig. 4b) enhances the activity in the oxidation of the ethyl acetate intermediates, which significantly improves the selectivity in total oxidation of ethyl acetate. Obviously, this is facilitated by the higher dispersion of the CuO particles, which are grown during the "chemisorption-hydrolysis" modification procedure. However, the lower density of surface acidic sites on the TiO₂-SnO₂ support (Table 2) provides a relatively low ability in ethyl acetate hydrolysis, which decreases the catalytic activity per unit surface area.

Surprisingly, the CuTiZr composites, which were characterized with the highest surface acidity (Table 2) and excellent CuO dispersion, possessed extremely low specific catalytic activity (Fig. 5b). This was also accompanied with about 50 % selectivity to ethanol and acetic acid formation for the sample obtained by the impregnation procedure. Taking into account the N₂ physisorption analyses (Fig. 1, Table 1) and XPS data (Fig. 4b) this could be attributed to the dominant activity of the acidic sites of the TiO₂-ZrO₂ solid solution and lower accessibility of the CuO particles, blocking in the long cylindrical mesopores of the support. According to the XPS analyses (Fig. 4a) the improved selectivity to CO₂ for CuTiZr_A could be related to higher exposure of the copper species, provoked by the interaction of the copper ammonia with the surface hydroxyl groups on the TiO₂-ZrO₂ support.

4. Conclusions

The template assisted hydrothermal synthesis of TiO₂-MO₂ (M=Ce, Zr, Sn) mixed oxides combined with their modification with CuO produces high surface area mesoporous nanocomposites. The phase composition, dispersion, morphology, surface and redox properties of the composites are controlled by the nature of the MO₂ dopant and the peculiarities of the modification procedure used.

The novel modification procedure including of chemisorption of copper ammonia complex on the TiO₂-MO₂ mixed oxides, followed by its hydrolysis and decomposition, provokes the formation of more finely dispersed, needle-like CuO particles. In comparison with the cubic-like CuO aggregates, formed during the conventional incipient wetness impregnation procedure, they improve the catalytic activity and CO₂ selectivity in total oxidation of ethyl acetate. The loaded CuO nanoparticles act in a synergistic mode with the acidic sites on the TiO₂-MO₂ surface, but the catalytic performance strongly depend on their location in the porous matrix.

The high density of surface acidic sites in TiO₂-CeO₂ supports promotes the catalytic activity *via* acceleration of the ethyl acetate hydrolysis, but the lower redox ability of the relatively large CuO aggregates on it, suppresses the total oxidation of the intermediates to CO₂.

The SnO₂ additives to TiO₂ decrease the density of the surface acidic groups and provides the formation of more finely dispersed CuO crystallites. As a result, the CuTiSn composites possessed lower catalytic activity, but improved selectivity in total oxidation

to CO₂, especially in the case of the chemisorption-hydrolysis modification procedure.

The superior surface acidity and CuO dispersion in the CuO-TiO₂-ZrO₂ composites did not provide the expected improvement of the catalytic activity due to the significant blocking of the active copper nanoparticles in the low-accessible cylindrical mesopores of the support.

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Strain controlled fatigue of 40X steel

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Abstract: The fatigue strength of 40X steel at 25 °C was determined following ASTM E606M standard for strain-controlled tests. The studied experimental dependence was modelled with a power function as well as with the functions and coefficients described in the standard. The material expresses plastic properties for cyclic loadings above 0.2% strain amplitude.

Keywords: 40X ALLOY, FATIGUE, ASTM E606-M

1. Introduction

We have a new hydraulic testing machine designed for both static and cyclic (dynamic) testing of materials at different temperatures. Below are shown results from fatigue tests of cylindrical specimens of 40X steel, performed according to ASTM E606M standard [1], i.e. with strain-controlled cycles. The 40X steel is a structural alloy steel used in heavy-duty components like shafts, springs and gears [2].

1.1 Short literature survey

Vakulenko et al. studied effect of the quality of the surface layer on the fatigue characteristics, [3]. They published data for fatigue tests of normalized 40X steel, obtained under fatigue cycles with an asymmetry factor of 0.3. The published data ranges from 7500 cycles of life at 600 MPa maximal stress to 500 million cycles at 400 MPa maximal stress. They have found that surface treatment of the samples increases their longevity on average by 3–5 times for loading amplitudes up to 530 MPa and more for higher amplitudes.

Shetulov et al. show fatigue data for 40X steel in the range 200 MPa – 650 MPa, [4]. They analyze the dependence of the fatigue curve on the test frequency, as the cycles to failure increase with increase the frequency.

Loktev et al. also analyze the influence of high frequency (20 kHz) on fatigue properties of 40X steel [5]. They obtained yield strain of 604 MPa and 924 MPa strength.

C.M. Sonsino holds the view that physically a knee point does not exist, as the Woehler-curves change their slope fluently in the transition range (from finite to high-cycle fatigue strength). The knee point is solely a parameter for strength estimations [6]. In this paper, he reviews strength and fatigue properties and gives recommendations for fatigue life design of different materials.

Chapter [7] describes most of the theory taught to the students. We met there the ideas for damages, cracks, fracture of specimens, fatigue, notches etc.

Most articles about materials fatigue talk about Woehler curves and the dependency of the number of cycles to failure on the stress amplitude and shape. However, in [7, p. 677] and in the standard ASTM E606M, an alternative – strain-based approach is explained for fatigue life determination.

Significant dispersion of results for Woehler curves can be seen in many articles. However, B. Boardman in ASM Handbook [7] shows a few diagrams from a strain-controlled approach to determine cycles to failure that have low dispersion.

1.2 Purpose

The aim was to master the work with the new machine in fatigue tests according to the ASTM E606M standard and to study the fatigue destruction of 40X steel.

2. Material and specimens

The specimens from 40X steel are cylindrical with 8.0 mm test diameter, 30 mm cylindrical test section, 130 mm long, 10 mm

gripping diameter and 16 mm transition radius. The working diameter has to have $R_z = 6.3 \mu\text{m}$ roughness. These specimens were used for both static test and cyclic tests.

With their gauge length of 25.0 mm, these test pieces are non-proportional.

Published properties usually refer to quenched state. However, material for the specimens is in its delivery state, not quenched.

3. Testing equipment



Fig. 1 Testing machine Zwick-Roell HA-250.

The hydraulic machine was equipped with a 250 kN load cell and 100 kN hydraulic grips. The strain was measured with a strain gauge Sandner EXA 25-2.5o with a 25 mm base length.

The testing machine is equipped with threaded holders designed for fatigue tests. However, the present data were obtained using the mentioned specimens designed for the hydraulic grips.

The hardware and software realizing machine control, measurement of monitored parameters and data registration are advanced and sophisticated.

4. Experimental plan

Determination of the tensile strength properties using 3 specimens and test procedure described in standard ISO 6892 – part 1.

Determination the number of cycles to failure for alternating sinusoidal cycle with strain amplitudes in the following order: 0.18%, 0.20%, 0.22%, 0.25%, 0.30%, 0.35%, 0.40%, 0.50% and 0.60%. The middle strain is zero.

We think that all tests should be done with equal maximal accelerations of the moving machine parts. For an oscillating system, acceleration is proportional to the strain amplitude and on the square of frequency:

$$\varepsilon_a v^2 = \text{const.} \quad (1)$$

For the used fixtures, this $\text{const.} = 70 \text{ \%Hz}^2$. This means that the cyclic frequencies were set between 20 Hz and 11 Hz for the planned strain amplitudes.

Two series were tested with mentioned maximal strains. For the first one, we have set the machine to stop cycles when the maximal force begins to decline due to the development of a big crack. For the second series, we set the machine to stop the test when the specimen breaks and the force becomes zero.

All tests were carried out in a temperature chamber at 25 °C.

5. Results and discussion

5.1 Tensile test properties

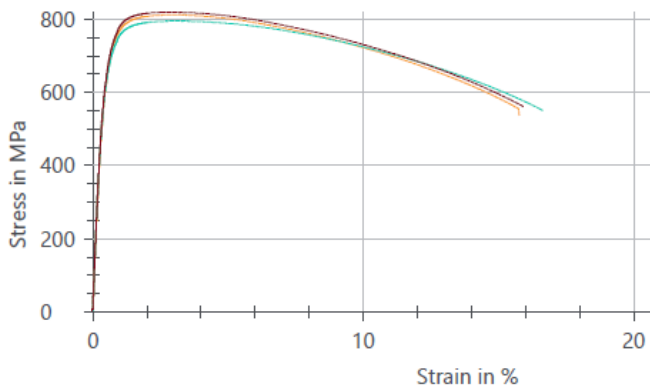


Fig. 2 Stress-strain diagram of 40X steel in tension, $T = 25 \text{ }^{\circ}\text{C}$.

From the **static tensile test**, made by standard ISO 6892-1, were obtained $195 \pm 5 \text{ GPa}$ elastic modulus, $649 \pm 11 \text{ MPa}$ proof stress at 0.2% plastic deformation, $809 \pm 12 \text{ MPa}$ ultimate strength, $(15.8 \pm 0.5)\%$ elongation at break and $(51 \pm 2)\%$ reduction of area in the neck.

Pay attention that these deformations were obtained using an extensometer with 25 mm gauge length and non-proportional specimens.

5.2 Results from cyclic tests

Tests were conducted following standard ASTM E606M, i.e., strain-controlled fatigue tests. We set the cycling frequency, a strain amplitude, the mean strain is zero, and the machine follows these instructions and records the stress for each cycle until the specimen breaks.

The following data is processed on Excel. Figure 3 shows the stress amplitude recorded for cyclic loading with 0.4% alternating strain. Authors usually divide similar diagrams into III stages. In this case, the first and second stages contain 8170 cycles. The IIIrd stage contains only 280 cycles, or 3.3 % of the time to break completely. In the test with 0.3% alternating strain, the IIIrd stage is 5.5% from total cycles to complete break.

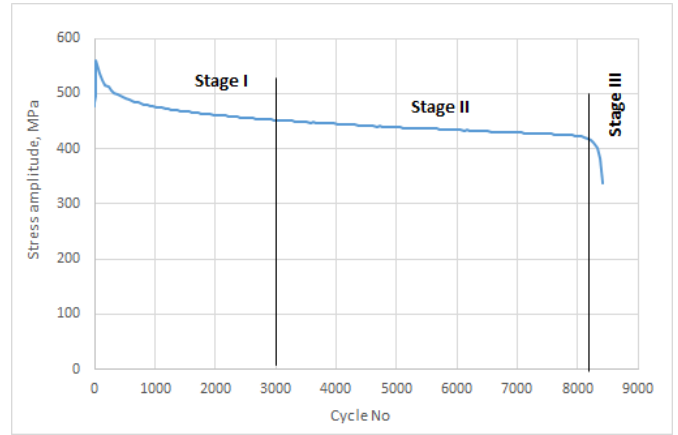


Fig. 3 Recorded stress amplitude at $\pm 0.4\%$ strain amplitude cyclic loading.

We accept the end of the second stage as a failure criteria. The resulting diagram is shown in Fig. 4.

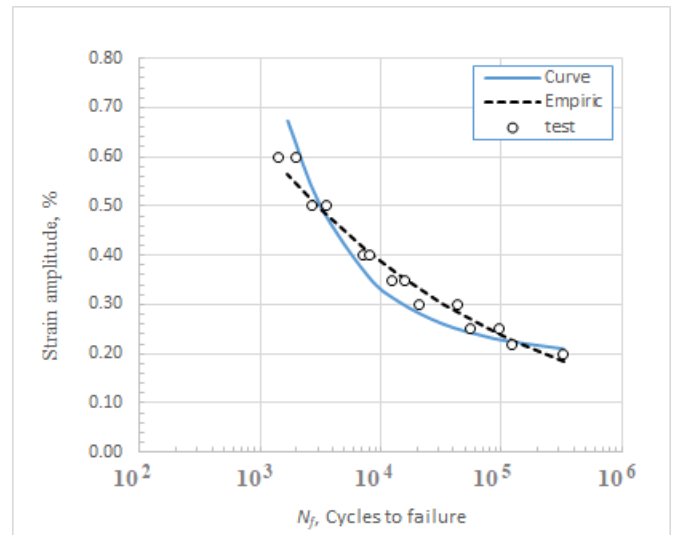


Fig. 4 Number of cycles to failure according to the strain amplitude.

There is one specimen tested at 0.18% strain amplitude. It endured 500 000 cycles, and the test was stopped without fracture.

5.3 Mathematical interpretation

The standard ASTM E606M describes a mathematical apparatus for interpreting such tests. Main equations and definitions [1, Appendix X1]:

$$\frac{\Delta \varepsilon}{2} = \frac{\Delta \sigma}{2E} + \frac{\Delta \varepsilon_p}{2} \quad (2)$$

Where $\Delta \varepsilon$ is total axial strain for a cycle loop and $\Delta \varepsilon/2$ is strain amplitude, $\Delta \sigma/2$ is stress amplitude and $\Delta \varepsilon_p$ is plastic strain for the whole cycle.

Recognizing that

$$\frac{\Delta \sigma}{2} = K' \left(\frac{\Delta \varepsilon_p}{2} \right)^{n'} \quad (3)$$

we may express the cyclic stress-strain curve by the following equation:

$$\frac{\Delta \varepsilon}{2} = \frac{\Delta \sigma}{2E} + \left(\frac{\Delta \sigma}{2K'} \right)^{1/n'} \quad (4)$$

Equations for Fatigue-Life relationships:

$$\frac{\Delta\sigma}{2} = \sigma'_f (2N_f)^b \quad (5)$$

$$\frac{\Delta\varepsilon_p}{2} = \varepsilon'_f (2N_f)^c \quad (6)$$

$$\frac{\Delta\varepsilon}{2} = \frac{\sigma'_f}{E} (2N_f)^b + \varepsilon'_f (2N_f)^c \quad (7)$$

Where N_f are cycles to failure and $2N_f$ is the number of reversals. These constants have the following names:

n' = cyclic strain hardening exponent,

b = fatigue strength exponent,

c = fatigue ductility exponent,

K' = cyclic strength coefficient,

σ'_f = fatigue strength coefficient,

ε'_f = fatigue ductility coefficient,

E = Young's modulus.

5.4 Discussion

The software calculates for each cycle the plastic strain using Eq. (8), which follows from Eq. (2):

$$\Delta\varepsilon_p = \Delta\varepsilon - \frac{\Delta\sigma}{E} \quad (8)$$

The check showed that software calculates elastic modulus for each cycle and presents the plastic strain in millimetres. For the test shown in Fig. 3, the obtained modulus at the end of IInd stage is 165 GPa, and the development of the plastic deformation, expressed in %, is shown in Fig. 5.

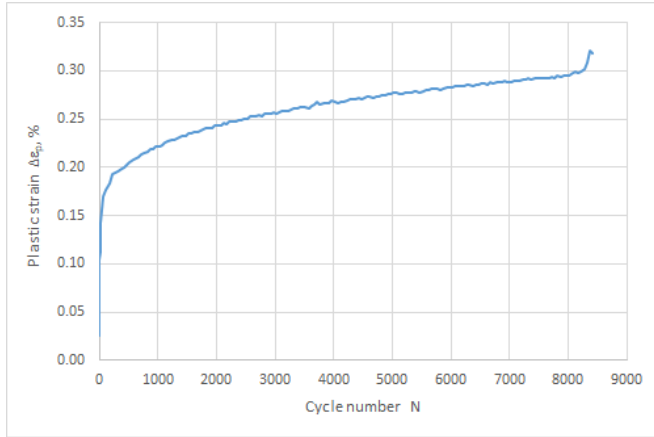


Fig. 5 Development of plastic strain for a test with 0.4% strain amplitude.

The typical three stages of fast growing, steady growing and catastrophic failure are well visible in Figs. 3 and 5. For each test there are no definite (constant) amplitudes of the stress and the plastic strain, representative for use in Eqs. (5) and (6).

We decided to use the values of stress and plastic strain at the end of the IInd stage, as representative values for use in Eqs. (5), (6) and (7).

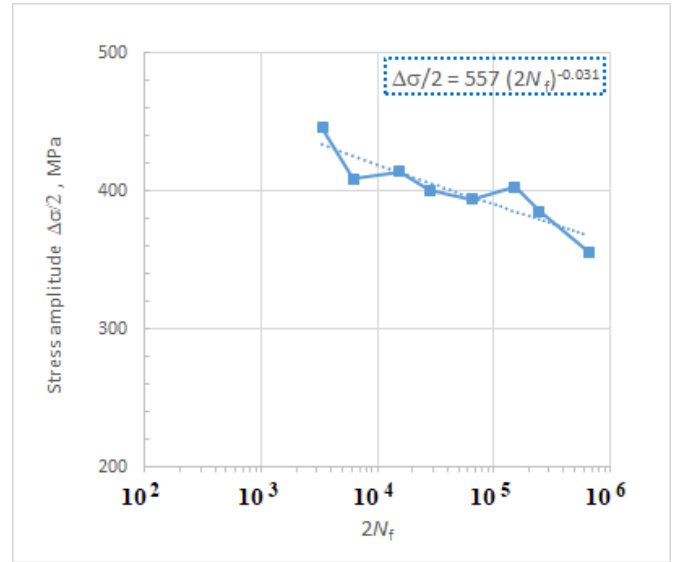


Fig. 6 Stress amplitude at the time of failure expressed with eq. (5) up to 0.33 million cycles

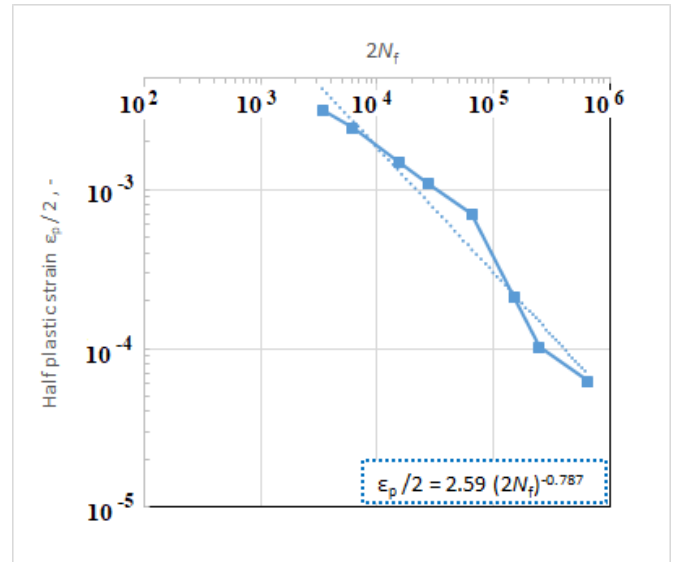


Fig. 7 Half of the plastic deformation at the time of failure expressed with eq. (6)

From Figs. (6) and (7) is visible that Eqs. (5) and (6) do not fit very well the whole diagrams. Probably, we should use one set of constants for $N_f \leq 10^5$ cycles and another for bigger fatigue lives.

Finally, Eq. (7) fits this experimental data best when the equation is used with $E = 181$ GPa. The relationship of strain amplitude on cycles to failure calculated with Eq. (7) and the mentioned coefficients ($\sigma'_f = 557$ MPa, $b = -0.031$, $\varepsilon'_f = 2.59$, $c = -0.787$, and $E = 181$ GPa) is shown in Fig. 4 as "Curve".

The mentioned curve does not look well despite the complicated model with 5 constants. The power function (9) fits better this experimental data (up to 0.33 mil. cycles) and its graph is shown in Fig. 4 as "Empiric".

$$\frac{\Delta\varepsilon}{2} = 2.75 N_f^{-0.213} \quad [\%] \quad (9)$$

6. Conclusion

The proof stress $R_{p0.2} = 649 \pm 11$ MPa was obtained from tensile tests.

The fatigue strength of 40X steel was determined following ASTM E606M standard for strain-controlled fatigue tests.

The studied experimental dependence, Fig. 4, was modelled with a power function - Eq. (9), as well as with equation (7), according to the dependences and coefficients described in the standard.

At 0.2% strain amplitude, the stress amplitude is 355 MPa and it almost does not change till the brittle fracture after 0.33 million cycles. For higher cyclic loads the plastic strain grows, like in Fig. 5, and the stress amplitude relaxes, similar to shown in Fig. 3.

Assuming that Eq. (7) and determined coefficients are correct enough, we can predict strain amplitude of 0.183% and endurance limit $\sigma_{-1} = 332$ MPa for 10^7 cycles to failure and 50% survival.

7. Acknowledgments

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