The Influence of the Temperature and Ageing Time on the $NiCr_{23}Co_{12}Mo$ Alloy Microstructure

MAREK SROKA[1]*, MARCIN NABIALEK[2], MICHAL SZOTA[3], ADAM ZIELINSKI[4]

[1] Institute of Engineering Materials and Biomaterials, Silesian University of Technology, Faculty of Mechanical Engineering, Konarskiego 18a Str., 44-100 Gliwice, Poland

[2] Institute of Physics, Faculty of Production Engineering and Materials Technology, Czestochowa University of Technology, 19 Armii Krajowej Str., 42-200 Czestochowa, Poland

[3] Institute of Materials Science and Engineering, Faculty of Production Engineering and Materials Technology, Czestochowa University of Technology, Al. Armii Krajowej 19, 42-200 Czestochowa, Poland

[4] Institute for Ferrous Metallurgy, K. Miarki 12-14, 44-100 Gliwice, Poland

The NiCr23Co12Mo alloy (Inconel 617) is a material used for example in the production of superheater boilers with ultra-cool supercritical parameters of work. The paper presents the results of microstructure changes after the long-term impact of temperature and strain. Analysis of the microstructure was carried out using scanning electron microscopy. Qualitative and quantitative identification of precipitations were conducted using X-ray analysis of the phase composition. Was studied the influence of ageing on tested alloy precipitations. The results of the study are the database alloy material of the new generation, which is used in the activities of the diagnostic elements of pressure parts of boilers.

Keywords: NiCr23Co12Mo, Inconel 617,microstructure, precipitations, creep test

In the light of European legal requirements, domestic resources of brown and hard coal, depreciation of most of operating power units in Poland, as well as energy security, trends in research and development of energy aim to build a modern power units with ultra-cool supercritical parameters, e.g. a temperature of 650-740 °C and pressure 30-35 MPa [1-6].

It is recognized that fossil fuels, especially coal, will remain the main source for electricity generation for the next decade. While the plans to build nuclear power plants in Poland, according to the latest data from the Ministry of Economy are for 2030 [7-11].

To reduce the level of CO_2 in the atmosphere and the savings of fossil fuels to improve the efficiency of electricity generation are necessary. This enables the development of new steels and alloys for elevated and high temperature [12-22].

The history of the construction of supercritical boilers reaches 50 years of the last century. Then, the first prototype units in the US in the Eddystone power plant, owned by Philadelphia Electric Co and in Ukraine with parameters row 30MPa/649/566 °C were created. At that time the basic austenitic steels and technologies that do not perform the quality requirements were used. Due to the lack of experience, technical errors and insufficient knowledge concerning the austenitic steel properties and their way of using caused large operation problems and low blocks availability. Only in the middle of 90's of the last century, there was a return to the blocks supercritical [23-29].

The materials used in the construction of boiler elements have to provide possibly the longest stability of tensile properties: yield strength, creep strength, a low tendency to brittleness, corrosion resistance at the time up to 200,000 h. This is possible by maintaining the structure stability and physical properties during operation. It should be noted that during the operation these parameters are continuously decreasing. However, they should not be lower than the permissible lower limit determined at the design stage [30-34].

Except for the high strength properties, these materials should be characterised by good weldability and ability to cold and heat plastic deformation, as well as the uncomplicated heat treatment.

At the moment in the country and the world in the field of interest of materials for the construction of modern boilers are the high-nickel and high-chromium austenitic steels HR3C, Super 304H, HR6W, CR30 and nickel superalloys type Inconel 617, 625 and 740 [35, 36]. The use of these materials for the construction of boilers depends not only on their high strength properties in the state of initial but mainly on their behaviour during long-term operation.

Experimental part

Materials for investigations

The material for investigations included the specimens from the sections of finished products in the form of tubes with dimensions of Ø 31.5 x 5 mm for DMV617 grade. Chemical composition is presented in table 1.

Methodology

The microstructure investigations were carried out using an Inspect F scanning electron microscope (SEM) and a TITAN 80-300 transmission electron microscope (TEM). The SEM observations were carried out on metallographic

Table 1

CHEMICAL COMPOSITION OF THE EXAMINED MATERIAL, wt%

C	Fe	Mn	Cr	Ni	Mo	Co	B	Ti	Al	Nb	N
0.06	1.10	0.02	21.70	56.00	8.40	11.40	0.002	0.48	0.68	0.10	0.05

* email: marek.sroka@polsl.pl

Table 2
THE REQUIRED VALUES OF TENSILE STRENGTH AT ROOM TEMPERATURE ACCORDING TO THE STANDARD PN - EN ISO 6892

Product shape	Product thickness	Test direction	$YS_{0,2}$ min. [MPa]	$YS_{1,0}$ min, [MPa]	TS [MPa]	El.[%]
tube	all	longitudinal	300	330	700÷950	35

Table 3
THE REQUIRED VALUES OF IMPACT ENERGY AT ROOM TEMPERATURE IN INITAL STATE

Product shape	Kind of sample	Sample direction	KCVm J/cm^2
all	with V notch	transverse	100
		longitudinal	150

microsections electrolytic etching, while the TEM observations were done using thin foils.

The analysis of precipitation processes was performed using X-ray isolates carbide on an Empyrean diffractometer PANalytical company using filtered radiation of cobalt and microdiffraction techniques in the configuration of the Pixcel detector and by means of thin films using a selective electron diffraction.

The quantitative analysis of precipitation was carried out using the image analysis system NIKON EPIPHOT200 & LUCIA G v.5.03. The scale marker as shown in the photos was used for calibration of the image analysis system. Calibration coefficient: 1 pixel = 0.040 μm.

Results and discussions

The required values of tensile strength at room temperature according to PN-EN ISO 6892-1 are shown in Table 2, while the required values of impact energy at room temperature in the delivery state according to EN ISO 148-1: 10.2010 are shown in table 3.

The results of the obtained tensile strength TS, yield strength YS, elongation and contraction in the static tensile test at room temperature and raised the temperature for a tested alloy in the initial state are shown in figure 1. However, the changes in these properties after annealing for 1,000 h at 700, 750 and 800 °C are shown in figure 2. The results of impact energy after annealing for 1,000 h at 700, 750, and 800 °C are presented in figure 3.

The annealing process conducted at temperature simulating long-term use causes changes in the microstructure which are acting on the strength and plastic material properties which are rated based on the results of static tensile testing, hardness and dynamic breaking work.

Ageing at a temperature of 700°C and time to 1,000 h resulted in a significant increase in the yield strength and the tensile strength (fig. 3). The value of the yield point (YS) after annealing for 1,000 h increased by 26%, while the tensile strength (TS) increased by 17%. After annealing at higher temperature – 750°C values of these parameters,

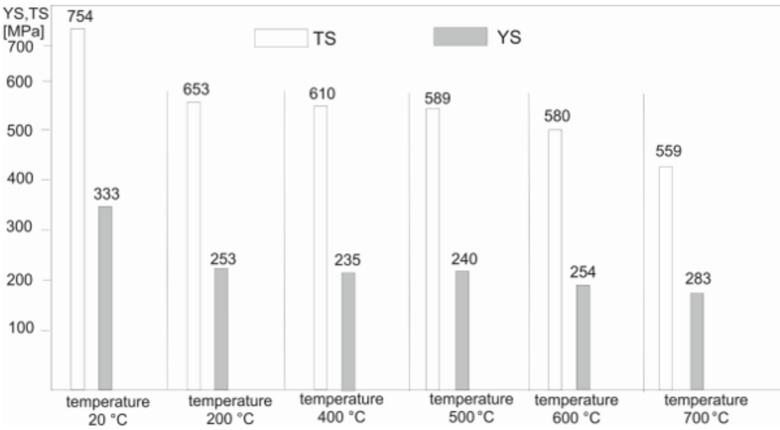

Fig. 1. Change in mechanical properties of Inconel 617H alloy in the room and elevated temperature

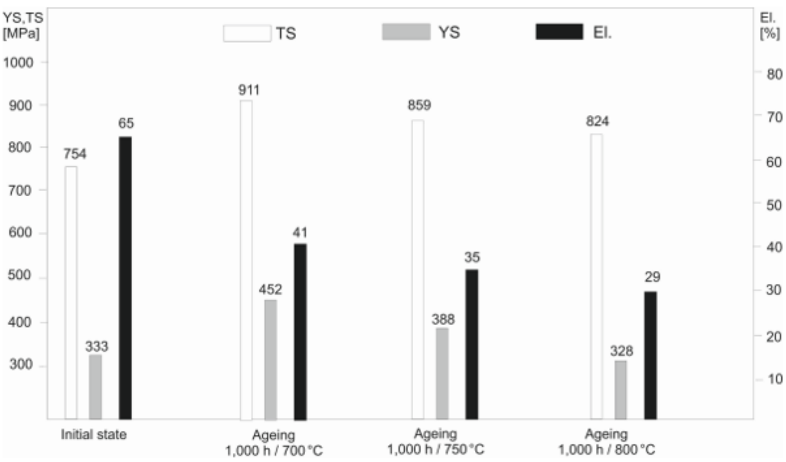

Fig. 2. Change in mechanical properties of Inconel 617H alloy during 1,000 h ageing at 700, 750 and 800°C

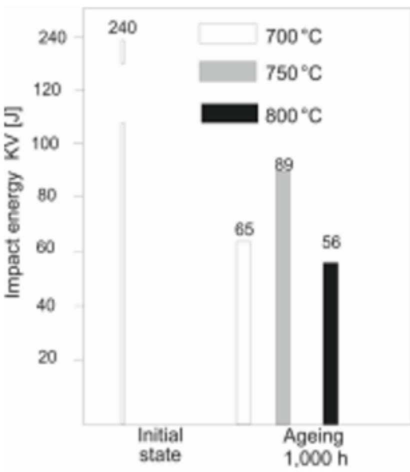

Fig. 3. Change in impact energy of Inconel 617H alloy during 1,000 h ageing at 700, 750 and 800°C

similar like after ageing at 700 °C significantly exceed the condition at delivery state. After ageing for 1,000 h at 750 °C the yield stress is around 15% and tensile strength is 13% higher in relation to the baseline. Ageing at a temperature of – 800 °C resulted in an increase in the tensile strength and yield strength of about 8%.

For all three levels of the annealing temperature up to 1,000 h an increase of tensile and yield strength were observed. While the elongation after annealing at 700, 750 and 800°C has constantly been decreased over the annealing time. Similar changes in the mechanical properties of Inconel 617 alloy were observed in [37,38].

The ductility properties of the material have been evaluated by measuring the breaking work for non standard samples of dimensions 55x10x2,5 mm which is a relative measure of the impact energy, the results of which are shown in figure 6. Annealing for 1,000 h results in a rapid decrease in the level of impact energy from 240 J for the level of delivery state to the level of 65 J for 700°C, and then from 89 and 56 J, respectively for the temperature of 750 and 800 °C.

The microstructure of Inconel 617 alloy in initial state observed using light and scanning electron microscopy is shown in figure 4. The tested alloy characterised by a coarse-grained austenitic microstructure with a grain size of 3-5 according to PN-EN ISO 643: 2013-06 with a small number of primary carbides. In the initial state of the tested steel may occur the precipitations of MX, $M_{23}C_6$ and M_6C type [38]. The precipitates identification in a transmission electron microscope revealed of the investigated the steel presence the occur of primary precipitates - evolved in the last phase of solidification [38] - of MX, $M_{23}C_6$ and M_6C type.

Images of the microstructure observed in a scanning electron microscope, proving the changes in the Inconel 617 alloy after ageing at 700, 750 and 800 °C at the time of 1,000 h are shown in figure 5.

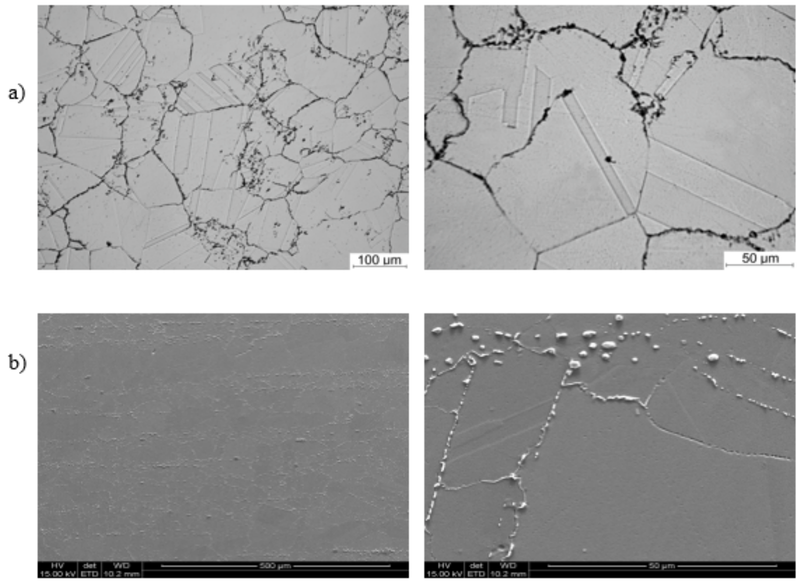

Fig. 4. Microstructure of Inconel 617H alloy in the as-received state a) light microscope (LM), b) scanning electron microscope (SEM), hardness 175 HV10

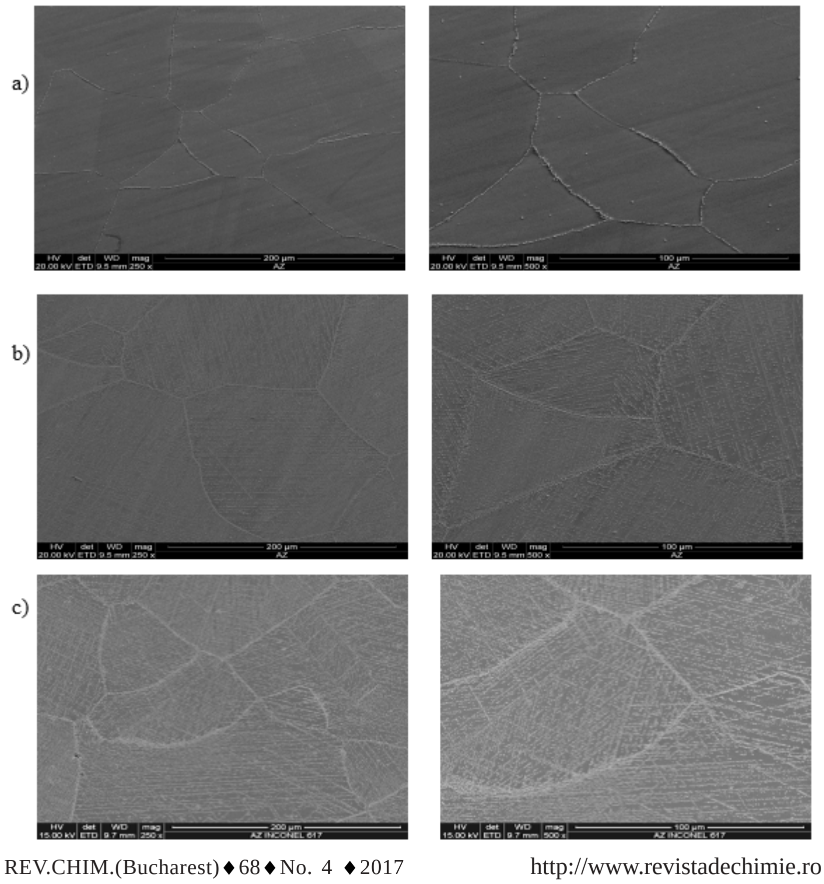

Fig. 5. Microstructure of Inconel 617H alloy after 1,000 h ageing at a) 700°C, b) 750°C, c) 800°C

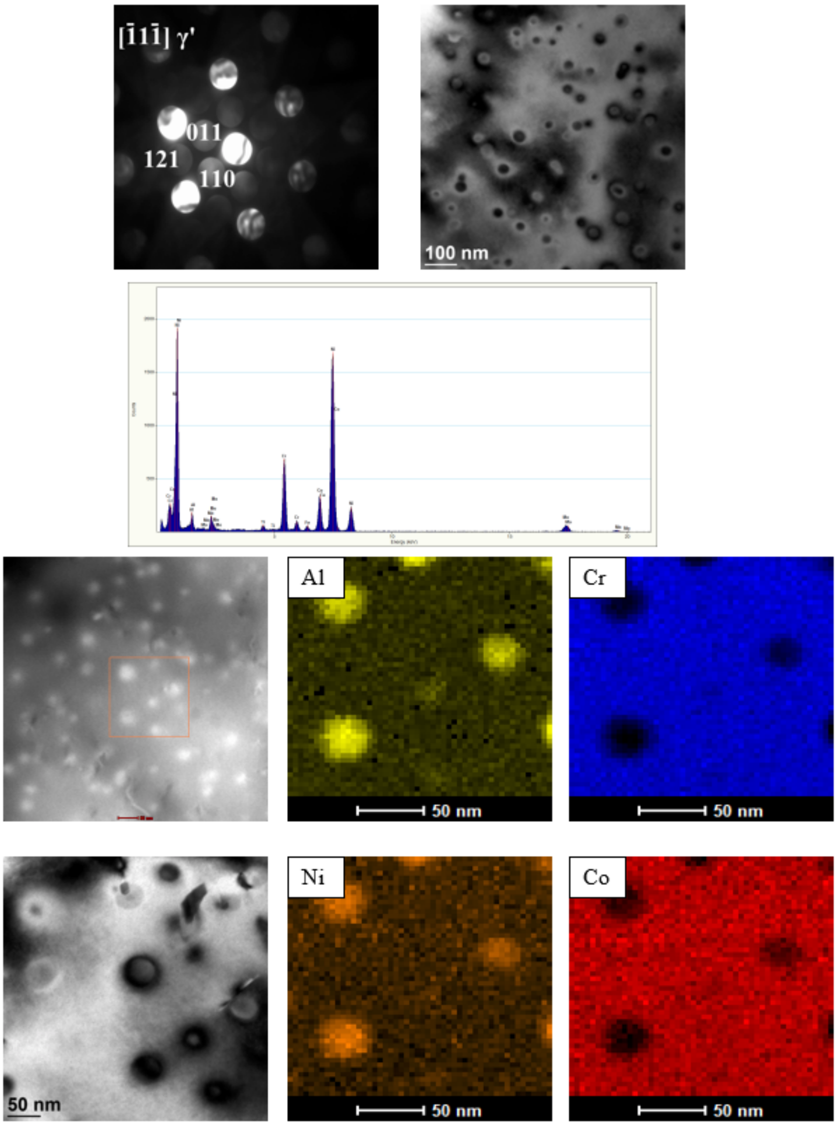

Fig. 6. The γ' precipitation in Inconel 617H alloy after 1,000 h ageing at 700 °C

The changes in the microstructure of steels with an austenitic matrix by long-term annealing at elevated temperature manifest themselves through processes of precipitation of secondary phases, both the austenite grain boundaries, twins, and inside the particles ageing for 1,000 h at 700 °C resulted in the secretion and increase the size of carbides, and M_6C $Cr_{23}C_6$ at grain boundaries of austenite, twins and slip planes within the grains and the austenite γ' phase (fig. 5a).

The intensification of the precipitation process of Inconel 617 alloy is noticeable with increasing annealing temperature (fig. 5b). This is confirmed by an increase in the size of the γ' phase precipitates (fig. 6), which amounted to 700 °C temperature and time of 1,000 h 28 nm, and the temperature 750 °C 54 nm. While for ageing temperature of 800°C increase in the average size of the γ' phase precipitates in relation to a temperature of 700 and 750 °C it is quite significant and is 185 nm for a time ageing 1,000 h similar character of changes in the size of the precipitates in the Inconel 617 alloy was observed in [38].

Conclusions

The Inconel 617 alloy due to high creep strength at elevated temperatures, and very good resistance to high-temperature corrosion and oxidation in steam provided by the formation of passivating chromium oxides Cr_3O_2 is recommended for long-term operation at a temperature of 700-800 °C.

The characteristics of heat-temperature materials, indicating their suitability for use at a specific temperature recognise the results of the stability of microstructure and mechanical properties in conditions of laboratory annealing at the temperature close to the temperature of potential use. The studies of ageing at 700-800 °C for up to 1,000 h revealed changes in microstructure consisting mainly in the tendency to create a finely dispersed, uniformly distributed γ' phase inside grains, whose average diameter increases with temperature and ageing time. In addition, in the microstructure the $M_{23}C_6$ and M_6C type precipitations, which are dispersed mainly in the austenite grain boundaries and twins, whose size also increases with the level of the test temperature.

Research of the tensile properties and impact energy test up to 1,000 h of ageing shows the high stability of the Inconel 617 alloy in the temperature range 700-750 °C. Annealing at 800°C contributes to an increase in the tensile strength and yield strength in the initial stage of annealing (100 h), and after long-time ageing, these values are close to the baseline. This shows the changes identification in the microstructure, as evidenced by the significant increase in the size γ' of phase.

References

1.SNOPINSKI, P., TANSKI, T., LABISZ, K., RUSZ, S., JONSTA, P., KRÓL, M., Int. J. Mater. Res., 107, no. 7, 2016, p. 637.

2.JONDA, E., BRYTAN, Z., LABISZ, K., DRYGALA, A., Arch. Metall. Mater., 61, no. 2, 2016, p. 963.

3.ZIELINSKI, A., GOLANSKI, G., SROKA, M., Mat. Sci. Eng. A-Struct., 682, 2017, p. 664

4.TOMICZEK, B., DOBRZANSKI, L.A., MACEK, M., Arch. Metall. Mater., 60, no. 4, 2015, p. 3029.
5.ZIELINSKI, A., MICZKA, M., BORYCZKO, B., SROKA, M., Arch. Civ. Mech. Eng., 4, 2016, p. 813.
6.SLIWA, A., MIKULA, J., GOLOMBEK, K., KWASNY, W., PAKULA, D., Arch. Metall. Mater., 61, 2016 pp. 1371.
7.ZIELINSKI, A., SROKA, M., HERNAS, A., KREMZER, M., Arch. Metall. Mater., 61, no. 2, 2016, p. 761.
8.ZIELINSKI, A., SROKA, M., MICZKA, M., SLIWA, A., Arch. Metall. Mater., 61, no. 2, 2016, p. 753.
9.ZUKOWSKA, L.W., SLIWA, A., MIKULA, J., BONEK, M., KWASNY, W., SROKA, M., PAKULA, D., Arch. Metall. Mater., 61, 2016, p. 149
10.SROKA, M., ZIELINSKI, A., DZIUBA-KALUZA, M., KREMZER, M., MACEK, M., JASINSKI, A., Metals, 7, no. 3, 2017, p. 82.
11.PAKULA, D., STASZUK, M., GOLOMBEK, K., SLIWA, A., Arch. Metall. Mater., 61, 2016, p. 1265.
12.DOBRZANSKI, L.A., MACEK, M., TOMICZEK, B., NUCKOWSKI, P., NOWAK, A.J., Arch. Metall. Mater., 61, no. 2, 2016, p. 1229.
13.DRYGALA, A., DOBRZANSKI, L.A., SZINDLER, M., SZINDLER, M.M., PROKOPIUK VEL PROKOPOWICZ, M., JONDA, E., Int. J. Hydrogen. Energ., 41, no. 18, 2016, p. 7563.
14.SLIWA, A., BONEK, M., Metalurgia, 65, 2017, p. 223.
15.DOBRZANSKI, L.A., CZAJA, M., BOREK, W., LABISZ, K., TANSKI, T., Int. J. Mater. Prod. Tec., 51, no. 3, 2015, p. 264.
16.TANSKI T., PAKIELA W., JANICKI D., TOMICZEK B., KROL M., Arch. Metall. Mater., 61, no. 1, 2016, p. 199.
17.TOMA, S.L., GHEORGHIU, D.A., RADU, S., BEJINARIU, C., Applied Mechanics and Materials, 371, 2013, p. 270.
18.MOGA, I.C., PRICOP, F., IORDANESCU, M., SCARLAT, R., DOROGAN, A., Industria Textila, 64, no. 4, 2013, p. 222.
19.PRICOP, F., SCARLAT, R., IORDANESCU, M., GHITULEASA, C., POPESCU, A., MOGA, I.C., Industria Textila, 64, no. 1, 2013, p. 40.
20.NEJNERU, C., CIMPOESU, N., STANCIU, S., VIZUREANU, P., SANDU, AV., Metalurgia International, 14, no. 7, 2009, p. 95.
21.TOMA, S.L., Surface & Coatings Technology, 220, 2013, p. 261.
22.PUSKAS, A., MOGA, L.M. International Journal of Sustainable Development and Planning, 10, no. 2, 2015, p. 165.
23.SROKA, M., ZIELINSKI, A., HERNAS, A., KANIA, Z., ROZMUS, R., TANSKI, T., SLIWA, A., Metalurgija, 65, 2017, p. 356.
24.ZIELINSKI, A., MICZKA, M., SROKA, M., Mater. Sci. Tech-Lond., 32, no. 18, 2016, p. 1899.
25.KREMZER, M., DZIEKONSKA, M., SROKA, M., TOMICZEK, B., Arch. Metall. Mater., 61, 2016, no. 3, p. 909.
26.SLIWA, A., BONEK, B., MIKULA, J., App. Sur. Sci., 388, 2016, p. 174
27.DRABCZYK, K., KULESZA-MATLAK, G., DRYGALA, A., SZINDLER, M., LIPINSKI, M., Sol. Energy., 126, 2016, p. 14.
28.SLIWA, A., MIKULA, J., GOLOMBEK, K., TANSKI, T., BONEK, B., KWASNY, W., BRYTAN, Z., App. Sur. Sci., 388, 2016, p. 281.
29.DOBRZANSKI, L.A., SLIWA, A., SITEK, W., Surf. Eng. 5 ISEC, 2006, p. 26.
30.SROKA, M., ZIELINSKI, A., MIKULA, J., Arch. Metall. Mater., 61, no. 3, 2016, p.969.
31.SLIWA, A., KWASNY, W., SITEK, W., BONEK, M., Arch. Metall. Mater., 61, 2016, p. 481.
32.TORRES, E., UGUES, D., BRYTAN, Z., PERUCCA, M., J. Phys. D. Appl. Phys., 42, no. 10., 2009, 105306.
33.WESZKA, J., SZINDLER, M., SLIWA, A., HAJDUK, B., JURUSIK, J., J. of Achiev. in Mat. and Manuf. Eng., 48, no. 1, 2011, p. 40.
34.TANSKI, T., SNOPINSKI, P., PAKIELA, W., BOREK, W., PRUSIK, K., RUSZ, S., Arch. Civ. Mech. Eng., 16, no. 3, 2016, p. 325.
35.SALZGITTER MANNESSMANN STAINLESS TUBES, Boiler Grade DMV 304 HCu, 2008
36.NIPPON STEEL & SUMITOMO METAL, HR3C, 2013
37.MASUYAMA, F., ISIJ Int., 41, 2001, p.612.
38.LEE, H.J., KIM, H., KIM, D., JANG, C., Mater. Charact., 106, 2015, 283.

Manuscript received: 16.12.2016