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## PHYSICAL MODELLING OF THE PLASTIC WORKING PROCESSES OF ZIRCONIUM ALLOY BARS AND TUBES IN THERMOMECHANICAL CONDITIONS

### MODELOWANIE FIZYCZNE PROCESÓW PRZERÓBKII PLASTYCZNEJ PRĘTÓW I RUR ZE STOPU CYRKONU W WARUNKACH TERMOMECHANICZNYCH

The article presents results of physical modelling of processes of plastic working of the modified Zr-1%Nb zirconium alloy, obtained using different methods of the plastometric testing. The “Gleeble 3800” metallurgical process simulator, a DIL805 A/D dilatometer with a plastometric attachment, and a “Setaram” plastometer were used for testing. Based on the obtained testing results, the values of the yield stress and limiting plasticity of the tested alloy were determined for wide ranges of temperature variation ( $T = 20 \div 950^\circ\text{C}$ ) and strain rate variation ( $\dot{\epsilon} = 0.1 \div 15.0 \text{ s}^{-1}$ ) under continuous loading conditions. It was found that by using different testing methods, different alloy properties, characteristic for a given plastic working process, could be obtained.

*Keywords:* Zr-1%Nb, rods, tube, physical modeling, hot, cold deformation

W artykule przedstawiono wyniki modelowania fizycznego procesów przeróbki plastycznej modyfikowanego stopu cyrkonu Zr-1%Nb, otrzymane za pomocą różnych metod badań plastometrycznych. Do badań zastosowano symulator procesów metalurgicznych “Gleeble 3800”, dylatometr DIL805 A/D z przystawką plastometryczną i plastometr “Setaram”. Na podstawie otrzymanych wyników badań określono wartość naprężenia uplastyczniającego i plastyczności granicznej stopu dla szerokiego zakresu zmian temperatury ( $T = 20 \div 950^\circ\text{C}$ ) i prędkości odkształcenia ( $\dot{\epsilon} = 0.1 \div 15.0 \text{ s}^{-1}$ ), w warunkach obciążania ciągłego. Stwierdzono, że stosując różne metody badawcze można uzyskać inne własności stopu, charakterystyczne dla danego procesu przeróbki plastycznej.

### 1. Introduction

From the zirconium alloy plastometric testing results reported in references [1-4, 15] it can be found that the selection of the testing method (tension, compression or torsion) when modelling plastic working processes will significantly influence both the yield stress magnitude and the shape (behavior) of the determined plastic flow curves for the zirconium alloys under consideration. Each of the above-mentioned methods not only enables the determination of rheological properties of materials being deformed in a wide range of thermomechanical parameters, but also allows the mode of increasing the presetting strain in time to be allowed for. This provides the possibility of simulating the actual plastic working processes of metals (such as rolling, extrusion, forging, etc.), but, at the same time, does not allow data obtained by a testing method inappropriate for a given preset strain pattern to be formally used when modelling individual technological operations.

The correct selection of the testing method in a physical modelling of plastic working processes assures the accurate shape of plastic flow curves depending on the process parameters to be obtained. Results of such tests can be used both

during the design of a new technology and in improving the existing technological operations of manufacturing rods, tubes and other fuel rod elements of modified zirconium alloys [5, 12, 13].

### 2. Test material and testing methodology

Tests were carried out for conditions prevailing during the plastic working of the modified zirconium alloy (Zr-1.0%Nb-0.7%Fe-0.9%O), hereinafter referred to as Zr-1%Nb. A physical modelling of plastic working processes of the Zr-1%Nb modified zirconium alloy had to be performed with respect to each working process in the technological sequence of producing either rods or tubes, and this determined the selection of one method or another. Three testing methods based on tension, compression and torsion tests, respectively, were employed. The “Gleeble 3800” metallurgical process simulator, a DIL805 A/D dilatometer with a plastometric attachment, and a “Setaram” torsional plastometer were used for the tests. Each of the methods reflected a different technolo-

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gical operation, hence different alloy properties were obtained, which were characteristic of a specific type of testing.

However, a vast majority of plastometric test results were obtained by the compression test, as the cylindrical specimen upsetting method is the most universal in terms of the similarity of this deformation pattern to that of plastic working processes used in the industry. Uniaxial tension tests allow the plasticity of metal in the plane of metal exit from the die in the extrusion process to be sufficiently assessed. The magnitude of the specimen deformation at the failure point determined in the torsion test is always greater than that determined from the tension and compression tests. For this reason, the torsion method is the most effective for determining values of the yield stress and limiting plasticity with the occurrence of large plastic strains that exist, for example, in the skew rolling process of zirconium alloy semi-finished products.

For plastometric tests subjected to the compression method using the “Gleeble 3800” plastometer, specimens of a working part diameter of 10 mm and a height of 12 were used, while for tests on the DIL805 A/D dilatometer, 5 mm-working part diameter and 10 mm-high specimens were employed. Tension test specimens had a working part diameter of 10 mm and a length of 85 mm. They were made of an alloy that had not been subjected to plastic working processes yet. It should be noted that tension tests using the “Gleeble 3800” plastometer involve a methodological peculiarity, whereby the working part of the test specimens equals the length of their local heating section, being 20 mm [6, 14]. Specimens of a working part diameter of 6 mm and a length of 15 mm intended for torsion tests were made from 14 mm-diameter bars.

Prior to the physical modelling of the plastic working processes of the semi-finished products of the alloy under investigation, the analysis of the basic parameters of each process making up the technological sequence was performed to determine possible ranges of variations of these parameters. On this basis it was determined that the experiments would be conducted in a wide range of temperature variations ( $T = 20 \div 950^\circ\text{C}$ ) and strain variations ( $\dot{\varepsilon} = 0.1 \div 15.0 \text{ s}^{-1}$ ).

During testing on the “Gleeble 3800” simulator and the DIL 805 A/D device, specimens were resistance heated with the direct current, together with controlling the rate of the heating (at  $5^\circ\text{C/s}$ ). For the control and monitoring of specimen temperature, a rhomel-niopol thermocouple welded to the central specimen part was used. A function of the thermocouple was to monitor and control the heating and cooling rates of the specimen being tested.

During compression tests, thin pads of a graphite-based material were used as a lubricant. After completion of each test, the ISO-T type working anvils were additionally coated with the OKS 255 brand graphite lubricant.

The torsion test specimens were heated in an electric furnace at an average heating rate of  $0.5^\circ\text{C/s}$ . During the tests, the specimens were held by two horizontal axial clamps, of which one was fixed and connected with a cylindrical sensor, while the other rotated around its axis. The rotational speed was controlled by a velocity sensor, while the torque – by a torque and axial force sensor.

All tests were conducted in vacuum in order to prevent the test specimen surface from oxidation and saturation with gas during the specimen heating.

Cooling of specimens during the tests was effected at a cooling rate of  $10^\circ\text{C/s}$  by blowing an inert gas, and in the instances where the alloy structure required to be fixed, water cooling was used.

Test results were processed using the Microsoft Office Excel application and were used for plotting “temperature-stress-strain” diagrams.

Based on the tension tests, after employing the methodology developed by Kolmogorov [4], the diagrams of limiting plasticity  $\Lambda_p$  were plotted. In the case of uniform deformation (up to the point of neck formation), the limiting plasticity value can be determined using the unit elongation  $A$ :

$$\Lambda_p = 1.73 \ln[100/(100 - A)] \quad (1)$$

while for non-uniform deformation (after the formation of the reduction of area), using the percentage reduction of area,  $Z$ :

$$\Lambda_p = 1.73 \ln[100/(100 - Z)] \quad (2)$$

The limiting plasticity (limiting strain at shearing),  $\Lambda_p$ , can be determined from the compression test, using the following relationship:

$$\Lambda_p = \sqrt{3} \ln(h_0/h_p), \text{ with } (\sigma_m/\tau_i)_m = -0.5 \quad (3)$$

where:  $h_0$  – initial specimen height,  $h_p$  – specimen height at the point of failure occurrence,  $\sigma_m$  – mean stress,  $\tau_i$  – shearing stress intensity.

The limiting plasticity value for specimens subjected to torsion is determined on their surface from the following formula:

$$\Lambda_p = \gamma, \text{ where } \gamma = \pi dN/L \quad (4)$$

where:  $\gamma$  – redundant strain (torsional angle up to specimen failure);  $L$ ,  $d$  – specimen working part length and diameter respectively;  $N$  – number of specimen rotation up to the specimen failure.

### 3. Testing results

Results of the tests carried out are shown in Figs. 1÷7. It can be seen from the data in Fig. 1 that with an increase in the specimen temperature, the strain hardening ratio of the examined alloy decreases, and more and more clear dynamic recovery processes are observed in the plastic flow curves. The kinetics of the dynamic recovery and recrystallization process occurring in the metal is largely reflected by the position of the maximum in the  $\sigma_p - \varepsilon$  curves. With the increase in specimen temperature and decrease in strain rate, this maximum shifts towards lower strain values, which is particularly clearly visible for the temperature range of  $580 \div 770^\circ\text{C}$ . A fixed plastic flow section (when  $\sigma_p = \sigma_{pu}$ ) occurs in the curves obtained at temperatures of  $700^\circ\text{C}$  and  $770^\circ\text{C}$  for a strain value of  $\varepsilon < 0.4$ .

The position of the maximum point in the plastic flow curves corresponds to the value of so called characteristic strain  $\varepsilon_{ch}$ , when the stage  $d\sigma_p/d\varepsilon > 0$  passes into the stage  $d\sigma_p/d\varepsilon < 0$ , that is when the recovery processes intensify and dominate over the strain hardening processes. The  $\sigma_p - \varepsilon$  curves have a dome-like shape. This shape of the plastic flow curves indicates that with increasing metal temperature the dynamic recovery processes play an increasing role due to the

considerable increase in the rate of diffusion processes within the entire range of variation of temperature  $T$ .

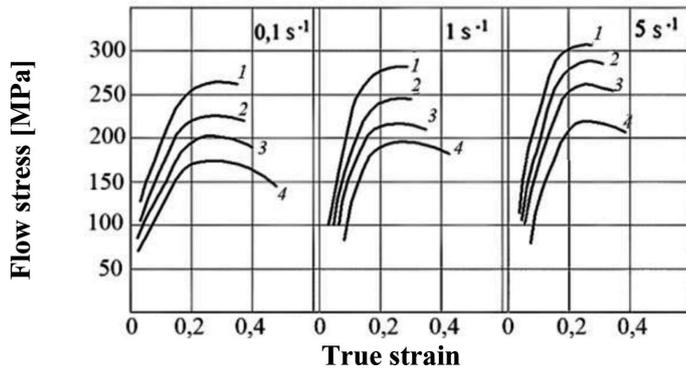


Fig. 1. Strain hardening curves for Zr alloy, obtained from tension tests on the "Gleeble 3800" plastometer; at a temperature of, respectively: 1 – 580; 2 – 650; 3 – 700; and 4 – 770, in °C

The limiting plasticity  $\Lambda_p$  for the tension specimens of a diameter of 10 mm and a total length of 116 mm was determined by the Kolmogorov method from formulas (1) and (2) based on the determined values of elongation,  $A$ , and the reduction of area,  $Z$ .

The data presented in Fig. 2 shows that with the increase in specimen temperature  $T$  from 650°C to 950°C, the limiting plasticity  $\Lambda_p$  of the Zr-1%Nb alloy varies not uniquely. At a strain rate of  $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$ , the limiting plasticity of the alloy increases monotonically, in calculations using both the elongation  $A$  and the reduction of area  $Z$ . For  $\dot{\varepsilon} = 2.5 \text{ s}^{-1}$  and with the use of elongation  $A$  in calculation, the limiting plasticity decreases in a temperature range of up to  $T = 750^\circ\text{C}$ . In the temperature range of  $T = 650 \div 680^\circ\text{C}$  and at a strain rate of  $\dot{\varepsilon} = 2.5 \text{ s}^{-1}$ , the limiting plasticity value of the alloy under examination is greater than that determined for the strain rate of  $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$ . This phenomenon is caused by the occurrence of a thermal effect that inhibits the formation of the test specimen plastic flow location and has the effect of increasing the alloy plasticity.

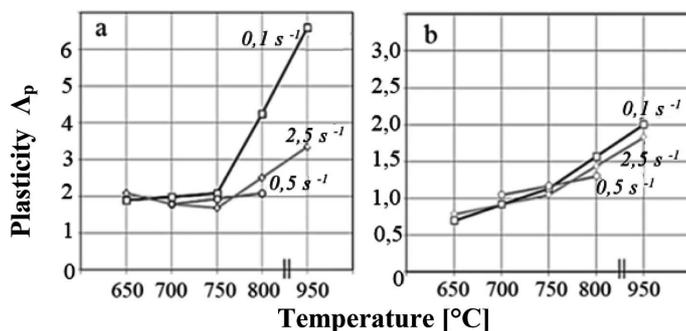


Fig. 2. The effect of temperature and strain rate on the limiting plasticity of the Zr alloy; as calculated based on: a) elongation,  $A$ ; b) reduction in area,  $Z$

The occurrence of the considerable difference between the limiting plasticity values determined using the indices  $A$  and  $Z$  can be explained by the inequality of specimen deformation in the transverse and the longitudinal directions.

Figure 3 shows  $\sigma_p - \varepsilon$  plastic flow curves for the Zr-1%Nb alloy, obtained with continuous loading of the specimens. The

strain hardening ratio of the examined alloy at a temperature of  $T = 20^\circ\text{C}$  clearly decreases with the strain increase (Fig. 3). This curve section takes the parabolic shape, which is caused by multiple slip in the material and it could be stated that the Drucker postulate [7] becomes violated.

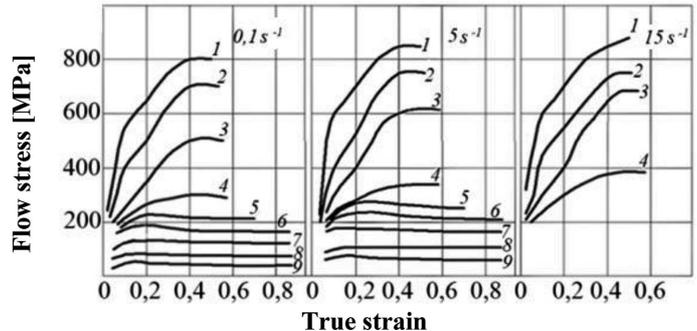


Fig. 3. Strain hardening curves for the Zr alloy, (compression test - Gleeble 3800); at a temperature of, respectively: 1 – 20; 2 – 200; 3 – 350; 4 – 500; 5 – 580; 6 – 650; 7 – 770; 8 – 850; and 9 – 950, in °C

In the region of  $\varepsilon = 0.3 \div 0.4$ , the ratio  $d\sigma_p/d\varepsilon$  is equal to zero. This phenomenon is characteristic of metals and alloys having the h.c.p. lattice. These materials are distinguished not only by a considerable thermal effect occurring in both cold and hot deformation at large rates, but also by an anisotropy of their properties, associated with their textural inhomogeneity.

At a temperature of  $T = 200^\circ\text{C}$ , the behaviour of variation of the flow curves does not change substantially. At strain rates of  $\dot{\varepsilon} = 0.1 \div 0.5 \text{ s}^{-1}$ , with the increase in the preset strain, the flow curves for the examined alloy pass into the stage of steady flow,  $\sigma_p = \sigma_{pu}$ . Under such deformation conditions, the yield stress value lies in the range of  $\sigma_p = 700 \div 800 \text{ MPa}$ .

With the continued increase of temperature, the region in which strain hardening of the examined alloy reduces, and at a temperature of  $T = 500^\circ\text{C}$  the steady flow stage starts already from the value of  $\varepsilon \geq 0.3 \div 0.4$ .

In the range of the test specimen temperature  $T$  from 500°C to 580°C, a dome-like shape of the Zr-1%Nb alloy flow curves becomes prominent, with a heavily extended steady flow section, which clearly indicates the initiation of dynamic recovery processes in the specimen being deformed. In the deformation range preceding the stage at which  $\sigma_p = \sigma_{pu}$ , a cellular structure forms in the metal. The strain value at which this structure starts forming is the lower, the higher the specimen temperature and the lower the strain rate. In the high strain region, during steady flow, the metal structure is characterized by a greater homogeneity, with a relatively small dislocation density [8, 9].

In the temperature range corresponding to the hot deformation ( $T = 650 \div 950^\circ\text{C}$ ) and in the strain rate range of  $\dot{\varepsilon} = 0.1 \div 15.0 \text{ s}^{-1}$ , the maximum value of  $\sigma_p$  decreases when using small strains of  $\varepsilon \approx 0.1 \div 0.2$ . A steady flow section,  $\sigma_p = \sigma_{pu}$ , comes up in the plastic flow curves. With the increase of temperature, this section becomes even more extended.

Figure 4 shows  $\sigma_p - \varepsilon$  flow curves for the examined alloy, as determined under continuous loading using a DIL805A/D plastometer-dilatometer.

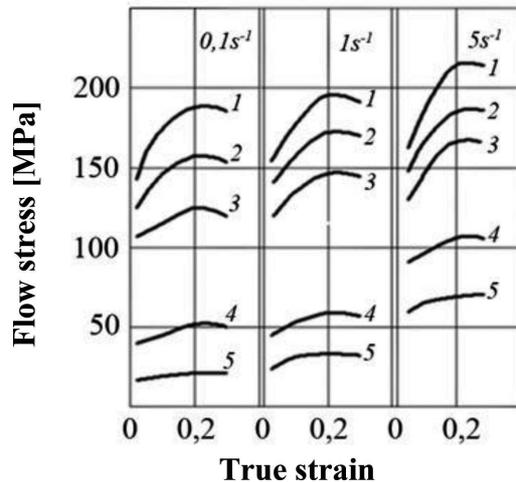


Fig. 4. Strain hardening curves for the Zr-1%Nb alloy, determined from the compression test on the DIL805A/D device; at a temperature of, respectively: 1- 580; 2- 650; 3- 770; 4-850-; and 5- 950, in °C

The shape of the curves obtained by both methods (Figs. 3 and 4) is very similar, which confirms the high accuracy of the obtained test results. However, when conducting these tests, a non-uniform deformation was observed to have occurred at  $\varepsilon > 0.2$ , which manifested itself by the occurrence of a large barreling and loss of stability of the specimen being upset.

The limiting plasticity (limiting plasticity at heating,  $\Lambda_p$ ) in the compression tests of 10 mm-diameter and 12 mm-long cylindrical specimens was determined from formula (3) following the Kolmogorov methodology.

The data shown in Fig. 5 indicates that the limiting plasticity of the alloy under consideration increased monotonically with an increase of the specimen temperature. An influence of strain rate on the limiting plasticity is definite. In the temperature range from 20°C to 650°C and at considerable strain rates ( $\dot{\varepsilon} = 5 \div 15 \text{ s}^{-1}$ ), the limiting plasticity is significantly influenced by the thermal effect of plastic deformation. For these reasons, the plasticity curves determined for the given strain rate range lie above the curves determined for the strain rate of  $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$ . In the temperature range of  $T = 650 \div 950^\circ\text{C}$ , the impact of the thermal effect is smaller, and with the increase of the strain rate, the plasticity of the examined alloy decreased.

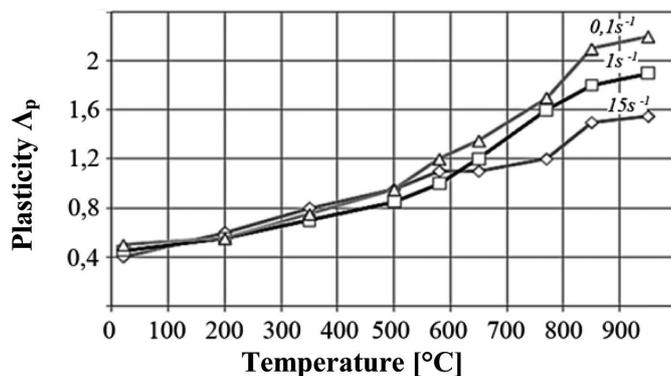


Fig. 5. The influence of temperature and strain rate on the limiting plasticity of the Zr alloy for the temperature range of  $T = 20 \div 950^\circ\text{C}$  (compression test - Gleeble 3800)

Figure 6 shows flow curves for the Zr-1%Nb alloy for three temperature ranges: 1- from 20°C to 200°C; 2- from 580°C to 700°C; and 3- from 750°C to 950°C.

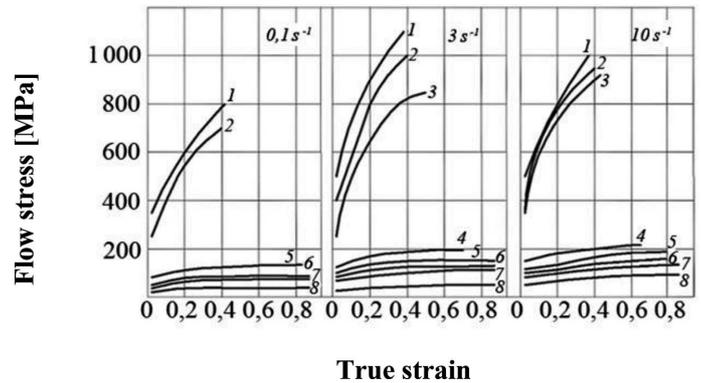


Fig. 6. Strain hardening curves for the Zr alloy, (torsion tests - Setaram); at a temperature of, respectively: 1 - 20; 2 - 100; 3 - 200; 4 - 580; 5 - 650; 6 - 700; 7 - 750; and 8 - 950, in °C

At ambient temperature and after the specimens have been heated up to a temperature of 200°C, the plastic flow curves of the examined alloy are characterized by a high value of the strain hardening index and can be described using the classic dislocation interaction models (such as those of Taylor, Zieger, Mott, etc.). The difference in the flow curve distributions in the temperature range from 20°C to 200°C is caused by an influence of strain hardening due to the increase in strain rate. Thus, if at  $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$  the value of  $\sigma_p$  at 20°C does not exceed 800÷820 MPa, then in the range from 3  $\text{s}^{-1}$  to 10  $\text{s}^{-1}$  the  $\sigma_p$  value attains values from the range of 980÷1000 MPa. The  $\sigma_p - \varepsilon$  curves essentially have the same behavior in terms of both shape and value, which is associated with the occurrence of a thermal effect at higher rates of conducting the loading process.

In the hot deformation range (at  $T = 200 \div 580^\circ\text{C}$ ), the plastic flow curves of the examined alloy change their behavior, which is due to the dynamic recovery process coming up. The course of the initial section of the  $\sigma_p - \varepsilon$  curves, especially at high strain rates, is associated with a considerable dynamic strain hardening, and with an increase of temperature and the preset strain, the flow curves takes on a parabolic shape.

At a temperature of 700°C and the lowest strain rate of  $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$ , a section of steady flow  $\sigma_p - \sigma_{pu}$  can be observed to appear in the flow curves. The occurrence of this section in the  $\sigma_p - \varepsilon$  curves is indicative of the process of dynamic recovery and dynamic polygonization occurring in the metal. With the continued temperature increase, the section becomes more extended and shows up even at higher strain rates.

Transition to the stage of the steady polygonization and formation of a stable structure occurs in the metal. The rheological behavior of materials in the conditions of steady dynamic polygonization is most commonly explained by the Sellars-Tegart hyperbolic relationship [10]:

$$\dot{\varepsilon} = A \left[ \sinh(\alpha \sigma_p) \right]^n \exp(-Q/RT) \quad (5)$$

where:  $Q$  - activation energy;  $R$  - gas constant;  $A, \alpha, n$  - constants.

At this deformation stage, the dislocation metal structure is defined by the subgrain size, which does not subject to any

major changes with increasing value of strain  $\varepsilon$ . This allows a relatively stable structure to be obtained in the region of large plastic strains.

In the temperature range of  $750^{\circ}\text{C} \div 950^{\circ}\text{C}$ , a maximum  $\sigma_p$  value can be found to occur at  $\varepsilon \approx 0.6 \div 0.8$  in the plastic flow curves of the alloy. This evidences the occurrence of the dynamic recovery process in the large strain region in the metal after the  $\varepsilon_{ch}$  (characteristic strain) has been attained, when  $d\sigma_p/d\varepsilon < 0$ .

The limiting plasticity value of specimens deformed by the torsion method was determined using the formula (4). From the diagrams of limiting plasticity variation of the examined Zr-1%Nb alloy (Fig. 7) it can be found that it is characterized by the occurrence of some peculiarities when examined at varying temperatures and strain rates.

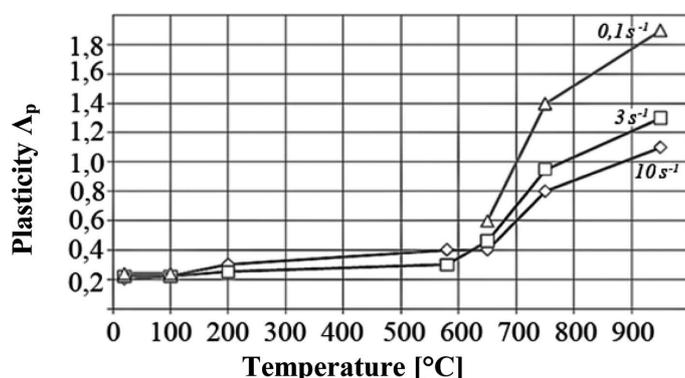


Fig. 7. The influence of temperature and strain rate on the limiting plasticity of the Zr alloy for the temperature range of  $T = 20 \div 950^{\circ}\text{C}$  in torsion tests

In the temperature range of  $20 \div 200^{\circ}\text{C}$ , the limiting plasticity of the examined alloy is low ( $\Lambda_p < 0.3$ ). These characteristics, together with the considerable strain hardening value, determines a given alloy to be classified into a group of materials of low limiting deformability, which requires the single reductions to be reduced at room temperature and in the temperature range of  $100 \div 200^{\circ}\text{C}$ , when conducting the plastic working process under the actual industrial conditions.

In the temperature range of  $580 \div 650^{\circ}\text{C}$ , the  $\Lambda_p$  value is not very large, either. An increase in the limiting plasticity occurs only in the temperature range of  $700 \div 950^{\circ}\text{C}$ . In this range, an influence of strain rate on the value of  $\Lambda_p$  cannot be uniquely determined. The examined Zr-1%Nb alloy at  $580^{\circ}\text{C}$  and for  $\dot{\varepsilon} = 10 \text{ s}^{-1}$  exhibits higher plasticity than for lower strain rates. At a temperature of  $650^{\circ}\text{C}$  and for the same strain rate, a reduced plasticity of the examined alloy can be observed.

With the continued increase of specimen temperature, the value of the limiting plasticity  $\Lambda_p$  for  $\dot{\varepsilon} = 10 \text{ s}^{-1}$  is smaller than for other strain rates. Starting from  $T = 700^{\circ}\text{C}$ , a considerable increase in the limiting plasticity occurs at all strain rates, and by reducing the strain rate  $\dot{\varepsilon}$ , it is possible to positively influence the deformability of the examined alloy.

For the Zr-1%Nb alloy in the temperature range from  $580^{\circ}\text{C}$  to  $650^{\circ}\text{C}$ , an inhibition for the plasticity increase is found (Fig. 7) at a strain rate of  $10 \text{ s}^{-1}$ , which is associated with the allotropic change  $\alpha \rightarrow \alpha + \beta$  in the alloy [11].

In torsion test specimens at  $T = 950^{\circ}\text{C}$ , a surface forms, which is characteristic of large shearing strains in plastic metal flow localization conditions were observed.

#### 4. Conclusions

Based on the investigations carried out, the following findings and conclusions have been formulated:

The presented data shows that:

1. The  $\sigma_p$  yield stress curves plotted in the  $\sigma_p - \varepsilon$  system, determined from the tension tests, lie above the curves determined from the compression and torsion tests, which is due to the fact that the plastic flow curves developed based on the tension tests reproduce conditions that fundamentally differ from the uniaxial stress and strain state scheme.
2. In order to determine the actual values of the yield stress  $\sigma_p$  of the alloys examined by the specimen tension method, a correction to the experimental  $\sigma_p - \varepsilon$  curves should be made according to the methodology of Siebel, Davidenkov – Spiridonov and Bridgman.
3. The shape of the plastic flow curves of the investigated alloy in the temperature range from  $20^{\circ}\text{C}$  to  $580^{\circ}\text{C}$  (during cold and hot deformation) is most influenced by the thermal effect of plastic deformation.
4. With increasing plastic strain rate, the value of the yield stress of the Zr-1% Nb alloy changes only slightly.
5. In the temperature range from  $580^{\circ}\text{C}$  to  $650^{\circ}\text{C}$  in torsion tests at a strain rate of  $10 \text{ s}^{-1}$ , an inhibition of the limiting plasticity increase can be observed, which is associated with allotropic changes taking place.
6. With the increase in the temperature of test specimens from  $700^{\circ}\text{C}$  to  $950^{\circ}\text{C}$ , the plasticity of the investigated alloy increases.

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