FLOW STRESS VALUE AND ACTIVATION ENERGY OF HOT DEFORMED INCONEL SUPERALLOYS

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S u m m a r y

Variations of a flow stress vs. true strain illustrate behavior of material during plastic deformation. Stress-strain relationship is generally evaluated by a torsion, compression and tensile tests. Results of these tests provide crucial information pertaining to the stress values which are necessary to run deformation process at specified deformation parameters. Uniaxial compression tests at the temperature through which precipitation hardening phases process occurred (720-1150°C), were conducted on superalloys - Inconel 718 and X750, to study the effect of temperature and strain rate ($\dot{\varepsilon}$ =10^{-4} and $4x10^{-4}s^{-1}$) on their flow stress. On the basis of received flow stress values activation energy of a high-temperature deformation process was estimated. Mathematical dependences (σ_{pl} -*T* i σ_{pl} - $\dot{\varepsilon}$) and compression data were used to determine material's constants. These constants allow to derive a formula that describes the relationship between strain rate ($\dot{\varepsilon}$), deformation temperature (T) and flow stress σ_{pl} – $\dot{\varepsilon} = A_1\sigma^n \cdot \exp(-Q/RT)$.

Keywords: flow stress, plastic deformation, Inconel, superalloys, activation energy

Krzywe odkształcania i energia aktywacji nadstopów Inconel odkształcanych w wysokiej temperaturze

S t r e s zc z e n i e

Zachowanie się materiału podczas odkształcania plastycznego na gorąco charakteryzują krzywe zmian naprężenia uplastyczniającego w funkcji odkształcenia. Do oceny są stosowane próby skręcania, ściskania lub rozciągania pozwalające określić dane niezbędne do prowadzenia procesu przeróbki plastycznej materiału z zastosowaniem odpowiednich parametrów odkształcania – temperatury i prędkości chłodzenia. W pracy przedstawiono wyniki badań wpływu temperatury i prędkości odkształcania ($\dot{\varepsilon}$ = 10^{-4} i 4 x $10^{-4}s^{-1}$) na wartość naprężenia uplastyczniającego nadstopów niklu – Inconel 718 i X750 w zakresie temperatury wydzielania faz umacniających (720-1150°C) uzyskane w jednoosiowej próbie ściskania. Otrzymane wartości naprężenia uplastyczniającego umożliwiły wyznaczanie wartości energii aktywacji Q procesu wysokotemperaturowego odkształcania. Wyniki z próby ściskania oraz zależności matematyczne (σ_{pl} – *T* i σ_{pl} –$\dot{\varepsilon}$) były podstawą do ustalenia

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zależności pomiędzy prędkością ($\dot{\varepsilon}$), temperaturą odkształcania (T) i naprężeniem ustalonego płynięcia plastycznego σ_{pl} – $\dot{\varepsilon} = A_1 \sigma^n \cdot \exp(-Q/RT)$.

Słowa kluczowe: Inconel 718, Inconel X750, nadstopy, Inconel, odkształcenie plastyczne, krzywe płynięcia, energia aktywacji

Introduction

With their many exceptional properties, Inconel 718 and Inconel X-750 have a high potential to become important high-temperature structural materials. Both Inconel 718 and Inconel X-750 are precipitation-hardened Ni-Cr-Fe-based alloys. Nickel based superalloys Inconel 718 and X750 are extensively used in aero gas turbine for critical assemblies such as discs and shafts which are subjected to severe service conditions at high temperature. The need of thermomechanical processing of Inconel's alloys – Inconel 718 and X750 for higher properties is known and published by mainly for forged products. There is lack information for the alloys subjected to deformation at high temperature under dynamic condition. Keeping this in view, compression testing within temperature characteristic for precipitation of hardening phases were taken up. This paper reports mechanical properties of the deformed Inconels – 718 and X 750.

One of the principal aim of a modern materials engineering is to prepare for each, as regard a chemical composition, type of superalloys potentially universal mechanical characteristic (true stress-true strain curves). These may allow to run a safe plastic deformation process of given precipitation hardened superalloys and make possible to predict suitable microstructures from overcooled austenite after heat treatment with application of various cooling rate, what is to determine final properties of such superalloys. Subsequently chemical and phase composition, microstructure and method of deformation or even its parameters play decisive role in the plastic deformation process. These factors affect a strengthening kinetics and changes of the microstructure and in the end the mechanical properties of a material. The hardening due to deformation process is relaxed by the softening process, namely recovering and (or) recrystallization. It is assumed that at high deformation temperatures, materials with a high stacking fault energy (SFE) such as aluminium, nickel etc. dynamic recovery becomes the sole softening process, however dynamic recrystallization may play significant role in the softening of deformed materials with a low SFE [1-3].

One of the most fundamental feature that characterize plastic workability of material is flow stress σ_{pl} – i.e. stress essential for a plastic flow initiation and following continuation in a one-dimensional stress state [4]. The flow stress value is strongly affected by temperature, deformation value, strain rate and the history of the course of deformation process [5]. To describe the relationship between of deformation parameters and stress value, following mathematical formula can be used [6-10]:

$$\dot{\varepsilon} = A\left[\sinh\left(\alpha\sigma\right)\right]^n \cdot \exp\left(\frac{-Q}{RT}\right) \tag{1}$$

where: A, α, n – material constants, $\dot{\varepsilon}$ – effective strain rate, Q – experimental activation energy for deformation process, T – deformation temperature, σ – flow stress value (σ_{pl}), or stress coressponds to the first maximum at a true stress/true strain curve (σ_{max}), R – universal gas constant (8,314 J/mol·K).

In the case of the small stresses ($\alpha\sigma$) $\leq$ 0,39; the equation (1) can be simplified (value of sinh($\alpha\sigma$) can be approximated by $\approx$ $\alpha\sigma$ with an accuracy ±1%) and expressed as:

$$\dot{\varepsilon} = A_1\sigma^n \cdot \exp\left(\frac{-Q}{RT}\right) \tag{2}$$

where $A_1 = A\alpha^n$ – material constant.

For a large value of the flow stress – $\alpha\sigma > 3,9$ (sinh ($\alpha\sigma$) $\approx$ 0,5 exp($\beta\sigma$)) the equation (1) can be modified as follows:

$$\dot{\varepsilon} = A_2 \cdot \exp\left(\beta\sigma\right) \cdot \exp\left(\frac{-Q}{RT}\right) \tag{3}$$

where: $A_2 = A/2^n$; $\beta = \alpha n$.

It is known that temperature has a significant effect on the flow stress variations during hot forming process. In addition, commonly used in description of high-temperature deformation process, Zener-Holloman parameter (usually known as a flow stress expressed in terms of temperature-compensated strain rate or as a deformation intensity) is thought to influence the course of $\sigma = f(\varepsilon)$ function, the flow stress value – maximum σ_{max} and at stationary state – σ_{pl}, dynamic recrystallization kinetic and size of dynamically recrystallized grains.

$$Z = \dot{\varepsilon} \cdot \exp\left(\frac{Q}{RT}\right) \tag{4}$$

An activation energy for deformation process Q (equation (1)-(3)) is characteristic for an examined material and deformation parameters. In the practice, the value of Q is constant, and depends on the method of forming process and deformation parameters [8]. Therefore its value can be used only for mathematical description of the relation: stress-deformation-temperature. In general, during hot-deformation of a material with high stacking fault energy (Al alloys and ferritic Fe alloys) stabilized value of the flow stress results from softening due to dynamic recovery, Q value is close to the value of activation energy for the self-diffusion process. For materials with low SFE (Ni, Cu and stainless steel), in most cases undergoing both dynamic recovery and recrystallization, Q value exceeds the value of activation energy for the self-diffusion process [6, 8]. The aim of the research was to obtain empirical relationship between the flow stress and phase composition and deformation parameters (deformation temperature, strain rate and cooling during deformation). The obtained values of a flow stress allow us to estimate the activation energy for high-deformation process of the examined superalloys.

Material and experimental

Investigation was carried out on Inconel 718 and X750 superalloys – chemical composition is shown in Table 1.

Table 1. Chemical composition of Inconel 718 and Inconel X750 (wt.%)

	Inconel 718	**Inconel X750**
C	0.0200	0.040
Cb	-	0.851
S	0.0064	-
Cr	17.957	14.740
Si	0.0640	-
Nb	4.5880	0.793
Co	0.0527	0.011
Zr	0.0159	-
Ta	0.0221	0.010
Ti	0.8734	2.732
Fe	16.665	8.451
Mo	2.6400	0.690
Al	0.4917	0.690
Mn	0.0537	0.060
V	0.0137	0.208
W	0.0132	-
Ni	balance	balance

In order to determine the effect of temperature and strain rate on flow stress value, isothermal high-temperature compression tests were performed on computer-controlled, Gleeble test equipment was used for the compression test (Fig. 1).

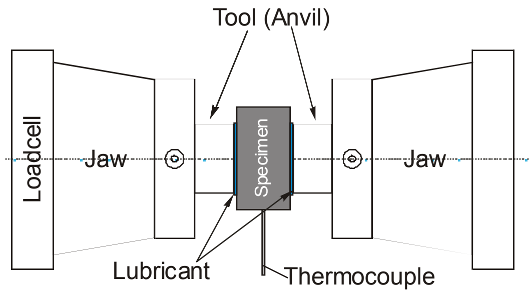

Fig. 1. Outline of the hot compression test and measured parameters in Gleeble thermo mechanical simulator

The cubicoidal samples (20 x 10 x 20 mm) were conductive heated to 1150°C at heating rate of 3°C/s, held for 300 s and finally cooled to the compression temperature. The temperature was controlled by a type K thermocouple inserted and welded in an opening hollowed out in the central part of the sample by spark erosion technique. Three additional thermocouples were used to acquire the distribution of the temperature from one of the faces to the centre of the specimen. A combination of graphite and molybdenum foils was used to reduce the friction between the anvils and the specimen as well as the gradient of temperature along the specimen. The deformation for all the tests was controlled by the stroke and measured by means of a loadcells attached to the jaws. The tests were carried out in an argon atmosphere.

The flow stress values under two different low strain rates over a range of temperatures (720-1150°C) were measured. The value of activation energy was determined in the following way [11-14]:

By finding logarithm the following equation has been obtained:

$$\ln \dot{\varepsilon} = \ln\left(A\left[\sinh\left(\alpha\sigma\right)^{n}\right]\right) - \frac{Q}{R} \cdot T^{-1} \tag{5}$$

hence:

$$\left.\frac{\partial \ln \dot{\varepsilon}}{\partial T^{-1}}\right|_{\sigma} = -\frac{Q}{R} \tag{6}$$

This relation (6) allow us to evaluate the activation energy value for a forming process carried out at σ = constans, e.g.: creep test. For the processes run at constant strain rate the equation (6) is rearranging to the following form:

$$\left.\frac{\partial \ln \dot{\varepsilon}}{\partial T^{-1}}\right|_{\sigma} = -\left.\frac{\partial \ln \dot{\varepsilon}}{\partial \ln\left[\sinh\left(\alpha\sigma\right)\right]}\right|_{T} \cdot \left.\frac{\partial \ln\left[\sinh\left(\alpha\sigma\right)\right]}{\partial T^{-1}}\right|_{\dot{\varepsilon}} = -\frac{Q}{R} \tag{7}$$

hence:

$$Q = R \cdot \left.\frac{\partial \ln \dot{\varepsilon}}{\partial \ln\left[\sinh\left(\alpha\sigma\right)\right]}\right|_{T} \cdot \left.\frac{\partial \ln\left[\sinh\left(\alpha\sigma\right)\right]}{\partial T^{-1}}\right|_{\dot{\varepsilon}} \tag{8}$$

The value of activation energy can be also estimated by applying following formula:

$$Q = \frac{\left.\dfrac{\partial \ln \sigma}{\partial T^{-1}}\right|_{\dot{\varepsilon}}}{\left.\dfrac{\partial \ln \sigma}{\partial \ln \dot{\varepsilon}}\right|_{T}} \tag{9}$$

The effect of deformation temperature on the true stress-true strain curves for the superalloys deformed at each of the two strain rates (10^{-4}; 4 x $10^{-4}s^{-1}$) and temperature of 720-1150°C is shown in Fig. 2 and 3.

a) b)

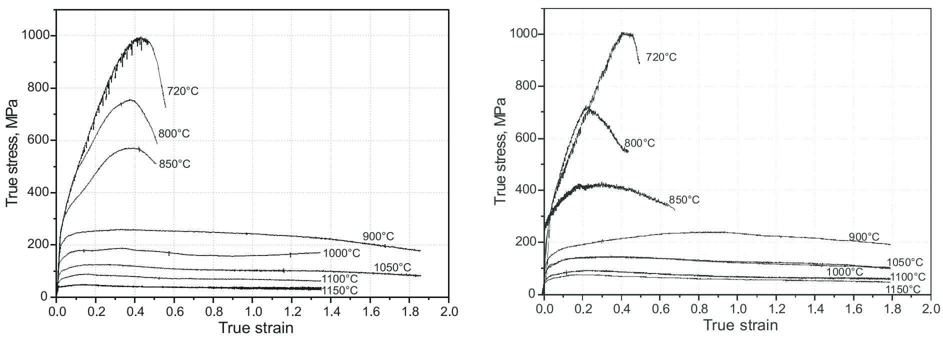

Fig. 2. True stress-true strain curves for: a) Inconel 718 and b) X750 superalloys deformed at different temperatures and a strain rate of 10^{-4} s^{-1} (the deformation temperature was indicated in the figure)

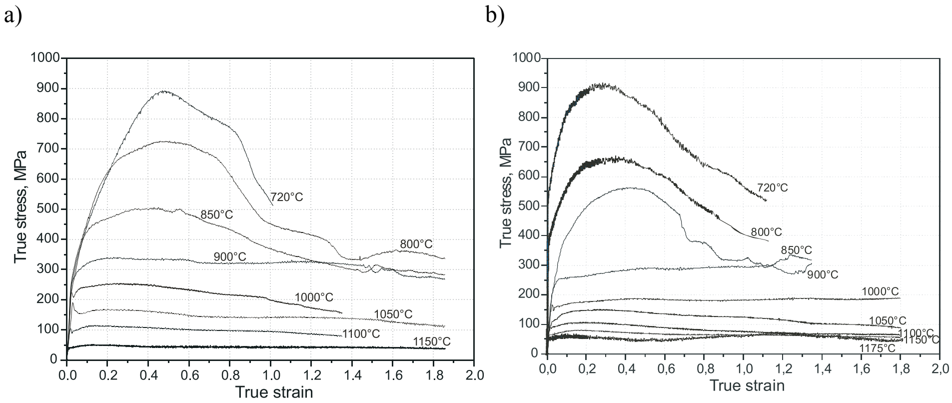

Fig. 3. True stress-true strain curves for: a) Inconel 718 and b) Inconel X750 deformed at different temperatures and a strain rate of $4 \cdot 10^{-4}$ s^{-1} (the deformation temperature was indicated in the figure)

A plot of the change in the peak strain with inverse temperature is shown in Fig. 4. The slopes of the peak strain and 1/T plots for these Inconel alloys are approximately equal at higher temperatures and shift to slightly higher values at 1150°C for the Inconel X750 deformed at higher strain rate. This change or transition in the slope may be attributed to the influence of hardened precipitation on dynamic recrystallization.

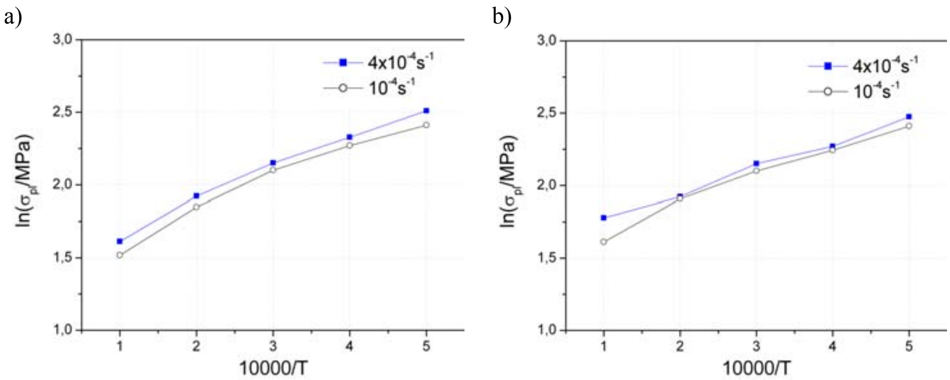

Fig. 4. Relationship between the flow stress and deformation temperature for solution treated: a) Inconel 718 and b) Inconel X750

The stress-strain curves for both strain rates and for both examined Inconel superalloys evaluated do not give a true indication of a stationary plastic flow in the temperature range (720-850°C) due to fracture of the samples. Therefore, for an evaluation of coefficients required for determination of the Q value of deformed Inconels superalloy, only data obtained in the limited temperature range, where plastic flow was stable, namely 900-1150°C, have been used. The flow stress can be selected as the representative stress of each true strain-true stress curve. Figure 4 and 5 show the relationship between flow stress on deformation temperature and strain rate, respectively. The classic interdependence of the flow stress and deformation parameters can be seen, namely: the flow stress increased with decreasing deformation temperature and increasing strain rate. This behavior is similar to the results obtained by Guimaraes and Jonas [15].

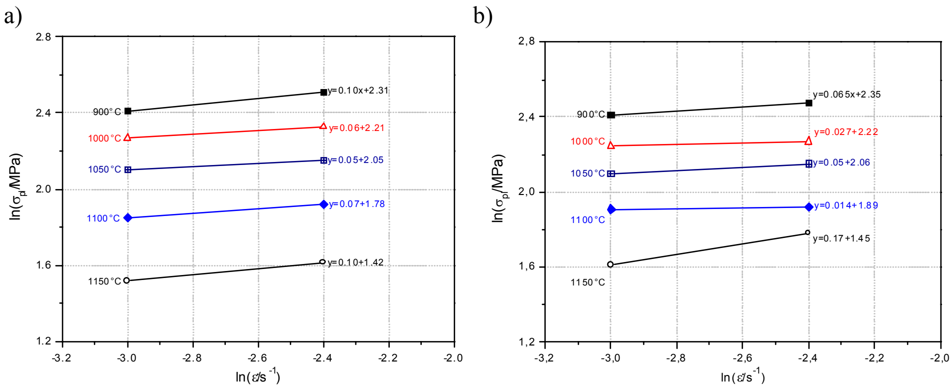

Fig. 5. Relationship between the flow stress and strain rate for solution treated: a) Inconel 718 and b) Inconel X750

In the present work, activation energy of deformed Inconel superalloys was determined in the way: the steady state stress values evaluated from flow stress obtained within the range of 900-1150°C were plotted as a function of inverse temperature and the logarithm of strain rate $\ln\sigma_{max} = f(1/T)$ and $\ln\sigma_{pl} = f(\ln\dot{\varepsilon})$. This gives the relationship between the peak strain and steady state stress and strain rate at constant temperature. The activation energy of deformation was then determined from the slopes of these curves. The values of $\left.\frac{\partial \ln\sigma}{\partial T^{-1}}\right|_{\dot{\varepsilon}};\left.\frac{\partial \ln\sigma}{\partial \ln\dot{\varepsilon}}\right|_{T}$ have were used in the equation (9) to estimate the activation energy for high-temperature deformation process of the investigated Inconels. For the range of deformation conditions employed, flow stress as a function of deformation temperature and strain rate was analyzed using a hyperbolic-sine Arrhenius type equation [14].

Flow stress vs. deformation temperature and strain rate dependence $\sigma_{pl} = f(\dot{\varepsilon}\ , T)$ was determined based upon both equations (1), (2), (4) and experimental data (Figs. 4 and 5).

For Inconel 718:

$$\dot{\varepsilon} = 3.93 \text{ x } 10^{14}\left[\sinh\left(\alpha\sigma\right)\right]^{2.21} \cdot \exp\left(\frac{-Q}{RT}\right) \tag{10}$$

and for Inconel X750:

$$\dot{\varepsilon} = 3.18 \text{ x } 10^{14}\left[\sinh\left(\alpha\sigma\right)\right]^{1.98} \cdot \exp\left(\frac{-Q}{RT}\right) \tag{11}$$

Activation energy Q for the deformation process of Inconel 718 and Inconel X750 is strongly affected by temperature and strain rate (Table 2).

Table 2. Activation energy for the Inconel 718 and Inconel X750 deformed in the temperature range of 900-1150°C

Strain rate $\dot{\varepsilon}$ **, s^{-1}**	**Temperature, °C**				
	900	1000	1050	1100	1150
	Activation energy Q, kJ/mol				
10^{-4}	**Inconel 718**				
	590.1	495.7	443.0	424.9	380.05
	Inconel X750				
	520.4	456.4	399.2	391.0	362.5
$4x10^{-4}$	**Inconel 718**				
	550.1	461.3	412.4	395.5	353.7
	Inconel X750				
	509.8	411.4	388.7	352.8	334.7

The mean value of activation energy for high-temperature forming of in the temperature range of 900-1150°C is for Inconel 718 = 450,8 and Inconel X750 = = 412.7 kJ/mol. For comparison, the activation energy evaluated by Weis [16] was 423 kJ/mol and Medeiros et al. [17] – 400 kJ/mol for Inconel 718. Estimated value of activation energy (Q) has a physical meaning and can be used in practice for modeling many types of engineering processes, e.g. analysis of plastic forming using the Finite Element Method. The effect of deformation temperature and strain rate on the flow stress can be expressed by the Zener-Hollomon parameter Z. Fig. 6 shows relationship between the flow stress and the Z parameter. The flow stress value increased with increasing the Z parameter.

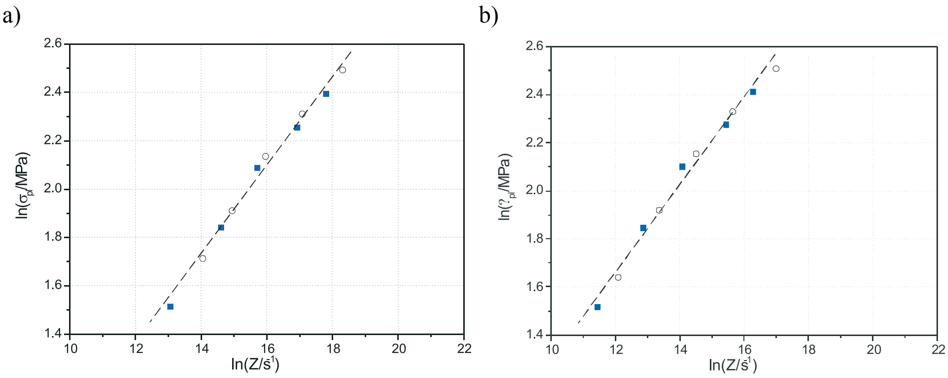

Fig. 6. Relationship between flow stress and the Z parameter for deformed of solution treated: a) Inconel 718 and b) Inconel X750

Discussion

Results of high temperature deformation of Inconel 718 and Inconel X750 superalloys include data concerning the effect of deformation temperature and strain rate on the flow stress value. True stress-true strain curves (Fig. 2) confirmed, that increasing deformation temperature, or strain rate decreasing cause result in decreasing of flow stress value σ_{pl}. Decreasing the value of strain rate by eight (from 4 x 10^{-4} to $10^{-4}s^{-1}$) results in a 10% flow stress reduction. Increase of deformation temperature was found to have a greater influence on the flow stress value reduction (Figs. 2 and 3). One must notice two distinguishable regions on the flow stress curves of the solution treated samples of Inconel alloy deformed at relatively low temperature, namely: 720, 800 and 850°C and with strain rate of 10^{-4} s^{-1} (see figs. 2 and 3). The first region, which occurs at an applied strain not exceeding 0.4, is characterized by almost uniform work hardening to a hump due to effective static precipitation within the alloy. The second region is characterized by a rapid flow softening followed by sample fracture at strain not exceeded the value of $\varepsilon \approx 0.6$ (Fig. 2). However, the flow stress of the samples deformed at these temperatures with higher strain rate (4 x 10^{-4} s^{-1}) increased to a peak value and then rapidly decreased as the strain further increased (Fig. 3).

The relationship between deformation parameters and a maximum flow stress value was described by equation (2). Basing on the diagrams – Fig. 4 and 5, the factors from the equation (9) were determined in order to estimate the mean value of activation energy (Q) of high-temperature deformation process of examined Inconel 718 – Q = 450.7 kJ/mol and Inconel X750 Q = 412.7 kJ/mol. The results (Table 2) show that activation energy depends on temperature and strain rate. Analogous dependence of deformation parameters on the value of activation energy was observed in other alloys systems and metals [11-14, 18]. One cannot, however, generalize in respect to the strain rate and temperature, the tendency in the change of activation energy for all type of materials. It is worth to emphasize that any particular single structural process could not be ascribed to the value of energy activation. Change of fraction of thermally activated dynamic recovery process and dynamic recrystallization in depreciation of the flow stress value is due to variation of the deformation parameters. It would be almost impossible to draw distinction between this processes on the grounds of an energy activation value. Therefore, averaging Q values obtained from the deformation within wide temperature and strain rate range should be treated as a mathematical treatment allowing to determine on of a constant in the equation describing the relationship between deformation parameters and flow stress value.

The results of high-temperature deformation of the examined Inconel alloys may possibly find some practical use in the workshop practice to predict a flow stress values, but only within particular temperature and strain rate ranges. Dissimilar energy activation values obtained under various conditions (depending on a research centre) or for a variety of materials make impossible to do a direct comparison of measurements, e.g. by means of plotting them on one common graph $\sigma_{pl} = f(Z)$. One should stress that a mathematical description of the relationship between deformation conditions is attributed for the range characteristic for the presence of particular phase. Thus, the equation (10) solely refers to the range of temperature 900-1150°C.

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