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STRESS–STRAIN CURVES UNDER COMPRESSION FOR CMSX4 NICKEL BASED SUPERALLOY

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Summary

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 (900-1200°C), were conducted on superalloy – CMSX-4, to study the effect of temperature and strain rate ($\dot{\varepsilon}$ =10^{-4} and 4x$10^{-4}$$s^{-1}$) on its flow stress. On the basis of received flow stress values activation energy of a high-temperature deformation process was estimated. Mathematical dependences ($\sigma_{pl} - T$ *i* $\sigma_{pl} - \dot{\varepsilon}$) 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, CMSX4, superalloys, activation energy

Krzywe odkształcania nadstopu CMSX-4 po próbie ściskania w wysokiej temperaturze

Streszczenie

Zachowanie się materiału podczas odkształcania plastycznego na gorąco charakteryzują krzywe zmiany naprężenia uplastyczniającego w funkcji odkształcenia. Do ich oceny stosowane są próby skręcania, ściskania lub rozciągania. Pozwalają określić dane niezbędne do prowadzenia procesu przeróbki plastycznej materiału z zastosowaniem odpowiednich parametrów odkształcania – temperatury
i prędkości chłodzenia. W pracy przedstawiono analizę wyników badań wpływu temperatury i prędkości odkształcania ($\dot{\varepsilon}$ =10^{-4} i 4x$10^{-4}$$s^{-1}$) na wartość naprężenia uplastyczniającego nadstopu niklu – CMSX-4 w zakresie wartości temperatury wydzielania cząstek faz umacniających (900-1200°C) uzyskane w jednoosiowej próbie ściskania. Ustalone wartości naprężenia uplastyczniającego były podstawą do wyznaczenia energii aktywacji *Q* procesu odkształcania wysokotemperaturowego. Na podstawie uzyskanych danych oraz odpowiednich zależności (σ_{pl} – *T* i $\sigma_{pl} - \dot{\varepsilon}$) określono wartości stałych materiałowych oraz ustalono zależność prędkości ($\dot{\varepsilon}$), temperatury odkształcenia (*T*) i naprężeniem ustalonego płynięcia plastycznego σ_{pl} - $\dot{\varepsilon} = A_1\sigma^n \cdot \exp(-Q/RT)$.

Słowa kluczowe: krzywe płynięcia, odkształcenie plastyczne, nadstopy niklu, CMSX4, energia aktywacji

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1. Introduction

With their many exceptional properties, CMSX-4 has a high potential to become important high-temperature structural material. CMSX-4 is Ni-based alloy. Nickel based superalloys CMSX-4 is 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 superalloys 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 CMSX-4.

One of the principal aim of a modern materials engineering is to prepare for each, as regard a chemical composition, type of superalloy potentially universal mechanical characteristic (true stress-true strain curves). These may allow to run a safe plastic deformation process of given precipitation hardened superalloy 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 superalloy. 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 ($\alpha\sigma$) ≤ 0,39; the equation (1) can be simplified (value of sinh($\alpha\sigma$) can be approximated by ≈ $\alpha\sigma$ 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 ($\alpha\sigma$) ≈ 0,5exp($\beta\sigma$)) 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 [11-15].

2. Material and experimental

Investigation was carried out on CMSX-4 – chemical composition (wt %): Cr – 6.5, Co – 9, Mo – 0.6, W – 6, Ta – 6.5, Ti – 1, Al – 5.6, Hf – 0.1, Fe – 3, Ni – 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).

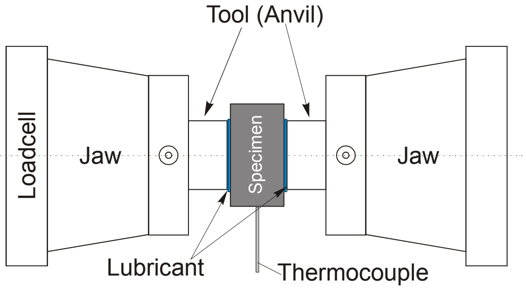


Fig. 1. Outline of the hot compression test and measured parameters in Gleeble thermo mechanical simulator

The cubicoidal samples (20x10x20 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 (900-1200°C) were measured. The value of activation energy was determined in the following way:

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 superalloy after different heat-treatment deformed at each of the two strain rates (10^{-4}; $4x10^{-4}s^{-1}$) and temperature of 900-1200°C is shown in Fig. 2-4.

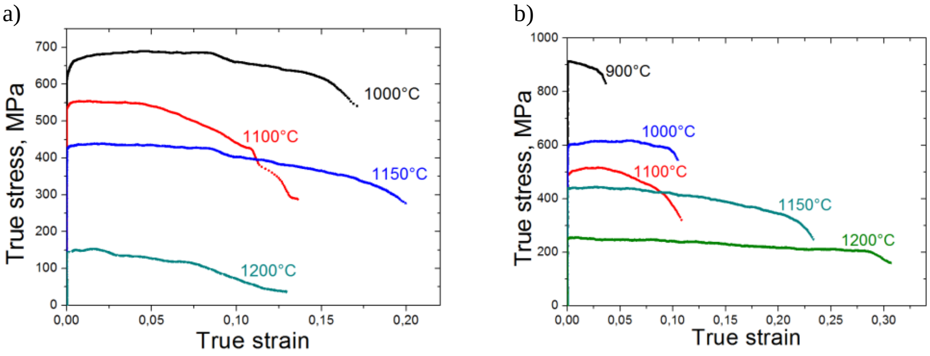


Fig. 2. True stress-true strain curves CMSX-4 deformed at different temperatures and a strain rate of a) 10^{-4} s^{-1} and b) $4{\cdot}10^{-4}$ s^{-1} (the deformation temperature was indicated in the figure)

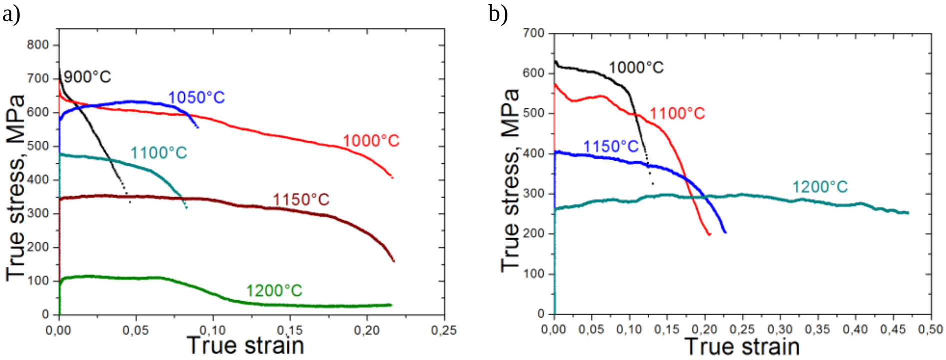


Fig. 3. True stress-true strain curves CMSX-4 after solution heat treatment and aged, deformed at different temperatures and a strain rate of: a) 10^{-4} s^{-1} and b) $4{\cdot}10^{-4}$ s^{-1} (the deformation temperature was indicated in the figure)

A plot of the change in the peak strain with inverse temperature is shown in Fig. 5. The slopes of the peak strain and 1/*T* plots for the CMSX-4 alloy are approximately equal at higher temperatures and shift to slightly higher values at 1200°C for the alloy deformed at higher strain rate. The same effect was observed after deformation of CMSX4 after solution heat treatment and both stage of aging. This change or transition in the slope may be attributed to the influence of hardened precipitation on dynamic recrystallization.

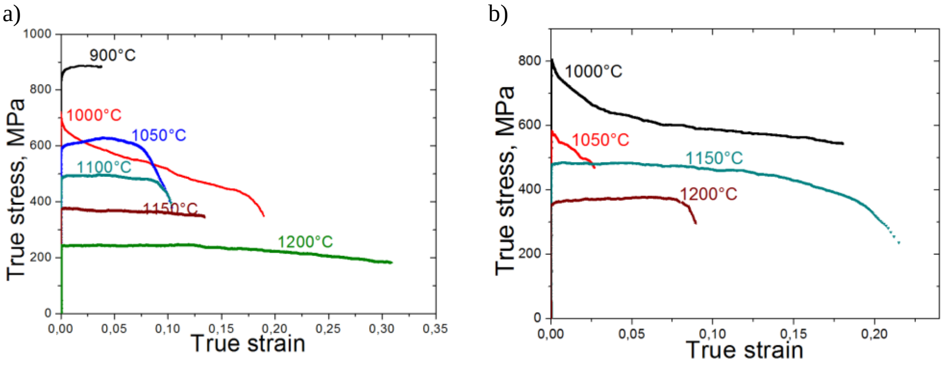
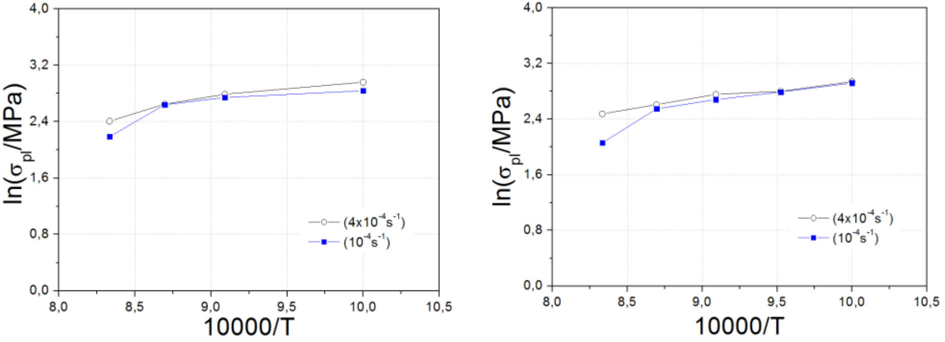


Fig. 4. True stress-true strain curves CMSX-4 after solution heat treatment and two stage of aging, deformed at different temperatures and a strain rate of: a) 10^{-4} s^{-1} and b) $4{\cdot}10^{-4}$ s^{-1} (the deformation temperature was indicated in the figure)



c)

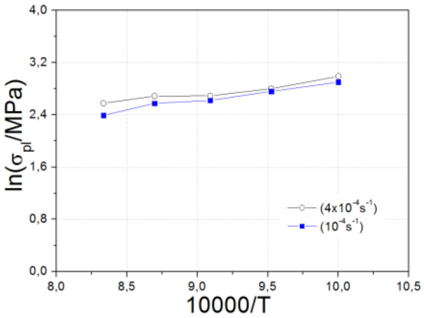
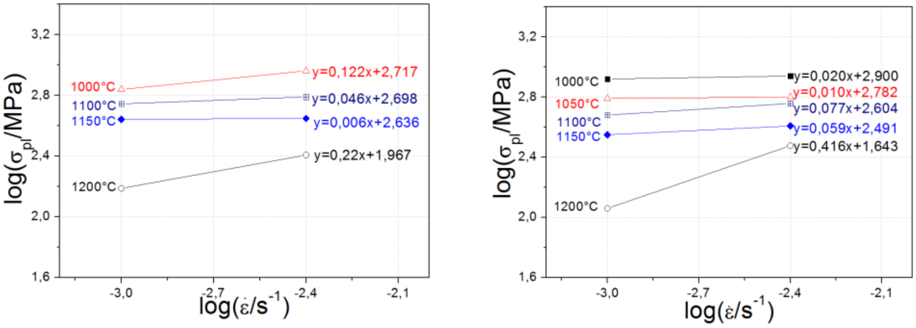


Fig. 5. Relationship between the flow stress and deformation temperature for CMSX4: a) solution treated b) after 1st step of aging and c) after 2nd steps of aging

Figure 5 and 6 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 behaviour is similar to the results obtained by Guimaraes and Jonas [5].

In the present work, activation energy of deformed superalloy was determined in the way: the steady state stress values evaluated from flow stress obtained within the range of 1000-1200°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.



a)

b)

c)

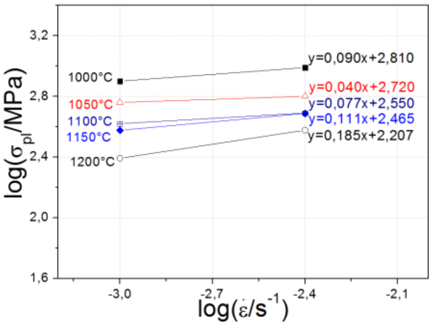


Fig. 6. Relationship between the flow stress and strain rate for CMSX4: a) solution treated, b) after 1st step of aging and c) after 2nd steps of aging

The activation energy of deformation was then determined from the slopes of these curves. The values of $\left.\frac{\partial \ln\sigma}{\partial T^{-1}}\right|_{\dot{\varepsilon}}$; $\left.\frac{\partial \ln\sigma}{\partial \ln\dot{\varepsilon}}\right|_{T}$ were used to estimate the activation energy for high-temperature deformation process of the investigated nickel based alloy. For the range of deformation conditions employed, flow stress as a function of deformation temperature and strain rate was analysed using a hyperbolic-sine Arrhenius type equation [6].

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

Activation energy Q for the deformation process of CMXS4 is strongly affected by temperature and strain rate (Table 1).

The mean value of activation energy for high-temperature forming in the temperature range of 1000-1200°C is for CMSX-4 equal to 779,46 kJ/mol. Estimated value of activation energy (Q) has a physical meaning and can be used in practice for modelling many types of engineering processes, e.g. analysis of plastic forming using the Finite Element Method.

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. 5 and 6).

$$\dot{\varepsilon} = 3.09x10^{14}[\sinh(\alpha\sigma)]^{2,34} \cdot \exp\left(\frac{-Q}{RT}\right) \tag{10}$$

Activation energy Q for the deformation process of CMSX-4 is strongly affected by temperature and strain rate (Table 1).

Table 1. Activation energy for the CMSX-4 deformed in the temperature range of 1000-1200°C

Strain rate $\dot{\varepsilon}$, s^{-1}	**Temperature, °C**				
	1000	1050	1100	1150	1200
	Activation energy Q, kJ/mol				
10^{-4}	**Solution Heat Treated CMSX-4**				
	741	-	786	828	911
	CMSX-4 after S1 aging				
	749	784	796	824	841
	CMSX-4 after S2 aging				
	755	794	816	834	794
$4x10^{-4}$	**Solution Heat Treated CMSX-4**				
	689	-	732	771	848
	CMSX-4 after S1 aging				
	697	730	741	783	802
	CMSX-4 after S2 aging				
	703	738	760	777	801

The mean value of activation energy for high-temperature forming of in the temperature range of 1000-1200°C is for CMSX-4 = 779,46 kJ/mol. 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. Figure 7 shows relationship between the flow stress and the Z parameter. The flow stress value increased with increasing the Z parameter.

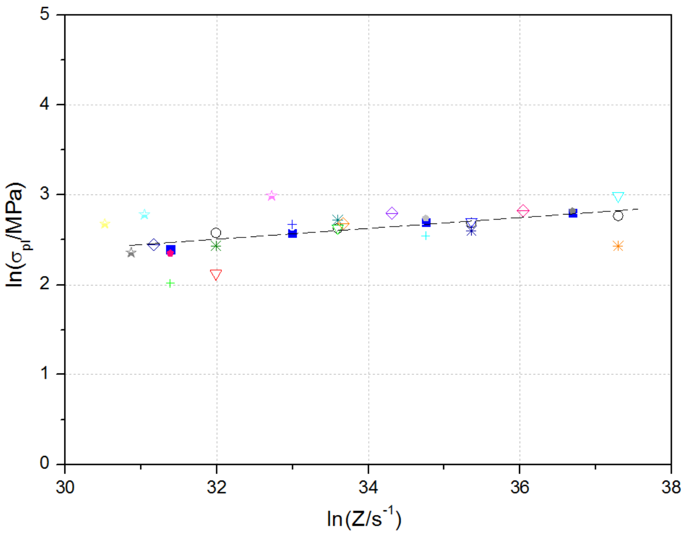


Fig. 7. Relationship between flow stress and the Z parameter for deformed of solution treated CMSX-4

3. Discussion

True stress-true strain curves (Fig. 2-4) confirmed, that increasing deformation temperature, or strain rate decreasing result in decreasing of flow stress value σ_{pl}. Decreasing the value of strain rate by eight (from $4x10^{-4}$ to $10^{-4}s^{-1}$) results in a 15% flow stress reduction. Increase of deformation temperature was found to have a greater influence on the flow stress value reduction. One must notice two distinguishable regions on the flow stress curves of the heat treated samples deformed at relatively lower temperatures, namely: 1000 and 1050°C and with strain rate of 10^{-4} s^{-1}. The first region, which occurs at an applied strain not exceeding 0.05, 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 $\varepsilon \approx 0.1$. However, the flow stress of the samples deformed at these temperatures with higher strain rate ($4x10^{-4}$ s^{-1}) increased to a peak value and then rapidly decreased as the strain further increased.

Basing on the diagrams – Fig. 5 and 6, the factors were determined in order to estimate the mean value of activation energy (Q) of high-temperature deformation process of examined alloy – Q = 779,46 kJ/mol. The results (Table 1) 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 [4, 6]. 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 CMSX4 alloy 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 $\sigma_{pl} = f(Z)$.

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References

[1] H. MC QUEEN: Dynamic recovery and its relation to other restoration mechanisms. Zeszyty Naukowe AGH Kraków. *Metalurgia i Odlewnictwo*, **3**(1979)8, 421-450.

[2] C.M. SELLARS: Dynamic recrystallization. Zeszyty Naukowe AGH Kraków, *Metalurgia i Odlewnictwo*, **5**(1979)3, 377-403.

[3] T. SAKAI, J.J. JONAS: Dynamic recrystallization: mechanical and microstructural consideration. *Acta Metallurgica*, **32**(1984), 189-208.

[4] F. GROSMAN: Problemy doboru naprężenia uplastyczniającego do programów komputerowej symulacji procesów przeróbki plastycznej. Mat. Konf. „Plastyczność materiałów”, Ustroń 1996, 11-18.

[5] F. GROSMAN: Kryteria doboru charakterystyk technologicznej plastyczności materiałów do symulacji procesów obróbki plastycznej. Mat. XVI Międzynarodowej Konf. Naukowo-Technicznej „Konstrukcja i technologia wytłoczek i wyprasek”. Poznań-Wąsowo 2004, 157-168.

[6] J.J. JONAS, C.M. SELLARS, W.J. MCTEGART: Recrystallization of metals during hot deformation. *Metallurgical Review*, **14**(1969), 1-24.

[7] T. SAKAI, J.J. JONAS: Dynamic recrystallization: mechanical and microstructural considerations. *Acta Metallurgica*, **32**(1984), 198-209.

[8] W. ROBERTS: Deformation, processing and structure’ (ed. G. Krauss). Metals Park, American Society for Metals, Ohio 1985.

[9] C.M. SELLARS, W.J. MCTEGART: On the mechanism of hot deformation. *Acta Metallurgica*, **14**(1966), 1136-1138.

[10] L. BŁAŻ: Dynamiczne procesy strukturalne w metalach i stopach. Wydawnictwo AGH, Kraków 1998.

[11] R. SANDSTÖRM, R. LAGNEBORG: A model for static recrystallization after hot deformation. *Acta Metallurgica*, **23**(1975), 481-489.

[12] A.J. MC LAREN, C.M. SELLARS: Modelling distribution of microstructure during hot rolling of stainless steel. *Materials Science and Technology*, **8**(1992), 1090-1098.

[13] A. NOWOTNIK, L. BŁAŻ, J. SIENIAWSKI: Interaction of phase transformation and deformation process during hot deformation of 0.16%C steel. *Defect and Diffusion Forum*, **237-240**(2005), 1240-1245.

[14] J. SIENIAWSKI: Nickel and titanium alloys in aircraft turbine engines. *Advances in Manufacturing Science and Technology*, **27**(2003), 23-34.

[15] A.A. GUIMARAES, J.J. JONAS: Recrystallization and aging effects associated with the high temperature deformation of waspaloy and inconel 718. *Metallurgical Transaction*, **12**(1981), 1655-1666.

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