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STRESS RELAXATION OF ZINC MONOCRYSTALS DEFORMED ON THE SYSTEM (0001)<112̄0>

RELAKSACJA POD NAPRĘŻENIEM MONOKRYSZTAŁÓW CYNKU ODKSZTAŁCANYCH W SYSTEMIE (0001)<112̄0>

Basing on a stress relaxation test, the activation volume V^* of Zn (99.995%) monocrystals deformed in the system (0001)<112̄0> at the temperatures 77 K and 293 K was determined. The density of forest dislocation on the plane (0001) was 10^4 cm^{-2}. The investigations have shown that the relaxation process in zinc monocrystals proceeds in two stages. Each stage is characterized by a different value of the activation volume V^*. The kinetics of the process depends to a great degree on the temperature of the test and the stresses at the beginning of relaxation.

W oparciu o próbę relaksacji pod naprężeniem wyznaczano objętość aktywowaną V^* monokryształów Zn (99,995%) odkształcanych w systemie (0001)<112̄0> w temperaturach 77 K i 293 K. Gęstość dyslokacji lasu na płaszczyźnie (0001) była rzędu 10^4 cm^{-2}. Badania pokazały, że proces relaksacji w monokryształach cynku przebiega dwuetapowo. Każdy etap charakteryzuje różna wartość objętości aktywowanej V^*. Kinetyka procesu zależy w dużym stopniu od temperatury próby i naprężenia początku relaksacji.

Key words: Zn monocrystals, stress relaxation, activation volume

1. Introduction

In the course of plastic deformation the moving dislocations meet on their way several obstacles. The most often encountered lattice obstacles are the forest dislocations of the slip plane, dislocations pile-ups, dislocation jogs, alien atoms and precipitates [1]. The difficulties in the dislocation motion cause the formation, in the deformed metal, of dislocation structures in which the energy supplied to the system becomes

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stored in the form of internal stresses. The normal tendency is to attain the state in which the internal energy is as small as possible. The phenomen consisting in unloading the internal energy of the material has been called relaxation under stress [1, 2].

Several relaxation mechanisms are distinguished [1]. The activity of the given mechanism is mainly determined according to the type of the lattice obstacles occuring in the structure (the crystal lattice) of the metal, the temperature and the relaxation stress.

The character and the magnitude of the obstacles occuring in the lattice of the examined material are analyzed by examining the change of its thermodynamic parameters depending on the applied stress, temperature changes, deformation rate. On the basic parameters, determined experimentally, is Gibbs thermodynamic potential [1–5]:

$$\Delta G = \Delta g - \tau * lb\Delta R, \tag{1}$$

where: ΔG — change of thermodynamic potential of Gibbs (activation energy),
  Δg — change of Helmholtz free energy,
  $\tau*$ — thermal component of the sharing stress in the slip plane,
  ΔR — extent of activation (dimensions of the obstacle),
  l — mean distance between the obstacles,
  b — Burger's vector of a displacing dislocation.

The member $l\Delta R$ is described by the term: activation surface. It is the area contained between the dislocation line before overcoming the obstacle and the same line after passing through the obstacle.

Generally the concept of activation volume ($V*$) is used.

It is a member of eqation 1:

$$V* = lb\Delta R. \tag{2}$$

The value of $V*$ can be determined experimentally in several ways. Investigations carried out on pure Zn monocrystals by various authors gave the following results:

— Korbel and Łatkowski [6] determined the value of the activation volume by the method of jump change of the deformation rate in a tensile test. The obtained value of $V*$ was contained in the interval $(0.7–1.0) \times 10^{-18}$ cm^3 for the deformation rate in the range $10^{-5}–10^{-3}$ s^{-1}. The density of forest dislocations was of the order of 10^6 cm^{-2}.

— Lukac [8] determined the value of the activation volume by the method of jump change of the deformation rate in a compression test at various temperatures. He obtained $V* = (0.9–3.0) \times 10^{-19}$ cm^3 for temperatures in the range 78–423 K and the deformation rate $10^{-4}–10^{-3}$ s^{-1}. The mean distance between the forest dislocations was of the order 10^{-4} cm.

— Wielke [7] determined the value of the activation volume by the stress relaxation method in a tensile test. The initial deformation rate was 7.7×10^{-4} s^{-1}. The mean distance between the forest dislocations was of the order of 10^{-4} cm. The obtained values were as follows: $V* = (0.2–3.0) \times 10^{-19}$ cm^3 for temperatures in range $4.2–323$ K.

2. The author's own investigations

To determine the value of the activation volume, the stress relaxation in a compression test was used. Zn (99.995%) single crystals obtained by the Bridgeman's method with the initial orientation $\lambda_o = 39°$, $\chi_o = 51°$ were examined. It enabled to obtain a great deformation range in the system of easy slip (0001)<11$\bar{2}$0>. The density of forest dislocations (0001) was 10^4 cm^{-2} (Fig.1). Samples measuring $7 \times 7 \times 15$ mm were examined.
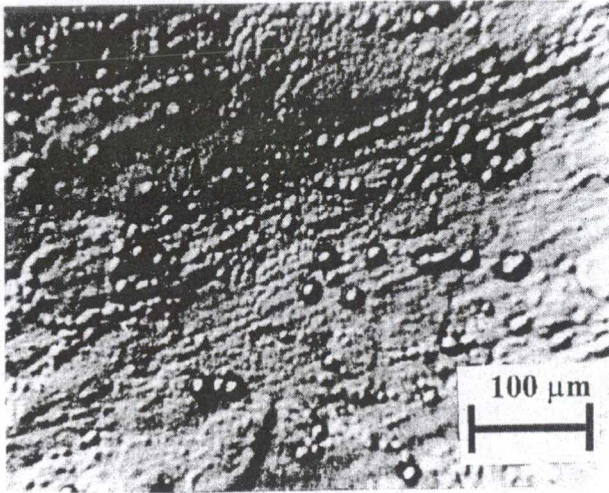
Fig. 1. Etch pitches on the plane (0001)

TABLE

Parameters at which stress relaxation was initiated

	Temp. [K]	Shape deformation γ	τ [MPa]
1.	77	0.005	0.402
2.	77	0.075	0.94
3.	77	0.383	7.43
4.	293	0.0077	0.37
5.	293	0.093	0.955
6.	293	0.435	4.67

Before the compresion test, each sample was annealed at the temperature 653 K for 20h. The initial deformation rate before relaxation was 1.1×10^{-3} s^{-1}. The investigations were carried out at the temperatures 77 K and 293 K. In order to reduce the coefficient of friction the compression test were carried out on teflon pads.

When determining the value of the activation volume V^* the formula [2, 10] was used:

$$V^* = kT \left(\frac{\delta \ln \dot{\varepsilon}_p}{\delta \tau} \right)_T \cong kT \left(\frac{\Delta \ln \dot{\varepsilon}_p}{\Delta \tau} \right),$$ (3)

where:

$$\dot{\varepsilon}_p = -\frac{\cos \lambda_0 \cos \chi_0}{E} \left(\frac{\delta \sigma}{\delta t} \right)_T,$$ (4)

where: $\delta\sigma/\delta t$ — elementary change of stress
[MPa] in the period of 1 sec.,
$\Delta \tau$ — change of shear stress in the system

(0001)<11$\bar{2}$0> [MPa],
E — Young's module [MPa],
$\cos \lambda_0 \cos \chi_0$ — orientation factor.

For each temperature of the investigations there was carried out relaxation at various deformation degrees:
— at the beginning of the range of easy slip
(samples 1 and 4)
— in the middle range of easy slip
(samples 2 and 5)
— in the range of the operation of secondary systems
(samples 3 and 6).

3. Discussion of the results

The analysis of the distribution of the density of forest dislocations on the slip plane, carried out by the method of each pits, has revealed both single dislocations and their agglomerations (Fig.1). This observation allows to assume that in the examined metal the forest dislocations form a differentiated sort of obstacles for the motion of slip dislocations in the system (0001)<11$\bar{2}$0>, differing considerably in their diensions:
— low-energy obstacles — single forest dislocations,
— high-energy obstacles — agglomerations of forest dislocations.
According to Evans and Rawlings [1], in the case of the presence in the lattice of obstacles of various types („series processes"), the value of the activation volume, determined experimentally, refers exclusively to long-range obstacles. Thus determined value V^* is valid only for agglomerations of forest dislocations which are high-energy obstacles.
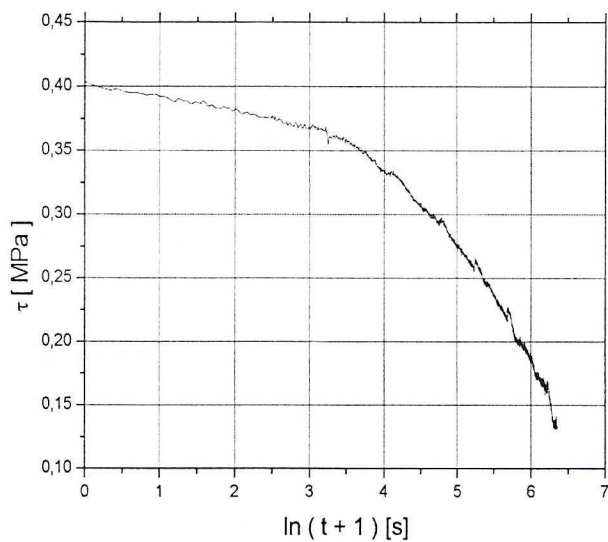
Fig. 2. Relaxation curve $\tau = f(\ln(t+1))$ of zinc monocrystal (sample 1), subjected to stress relaxation at the temperature 77K
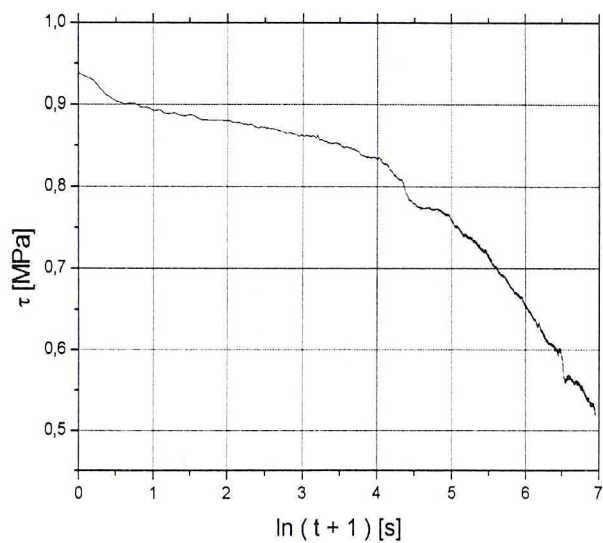

Fig. 3. Relaxation curve $\tau = f(\ln(t+1))$ of zinc monocrystal (sample 2), subjected to stress relaxation at the temperature 77K
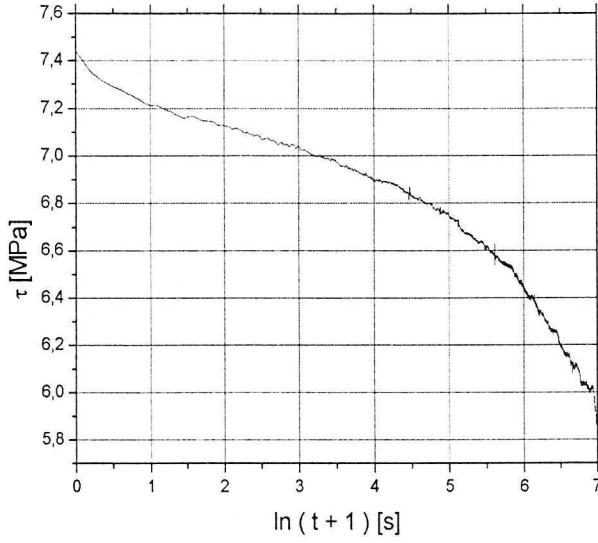
Fig. 4. Relaxation curve $\tau = f(\ln(t+1))$ of zinc monocrystal (sample 3), subjected to stress relaxation at the temperature 77K
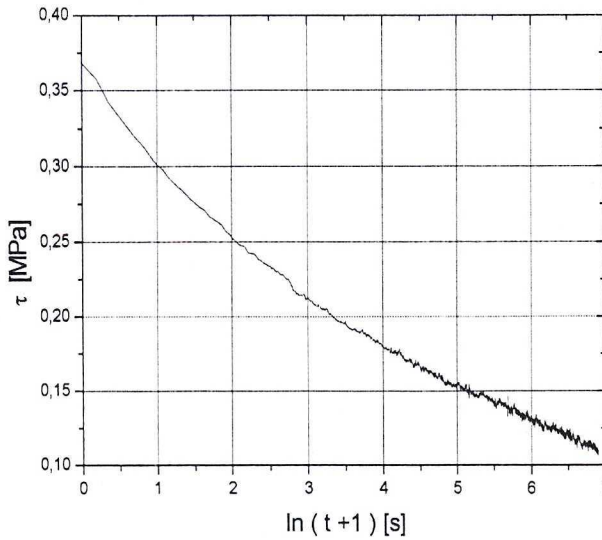

Fig. 5. Relaxation curve $\tau = f(\ln(t+1))$ of zinc monocrystal (sample 4), subjected to stress relaxation at the temperature 293K
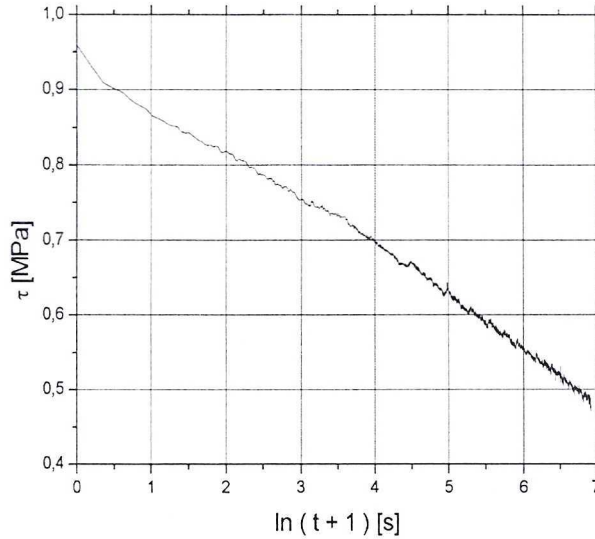
Fig. 6. Relaxation curve $\tau = f(\ln(t+1))$ of zinc monocrystal (sample 5), subjected to stress relaxation at the temperature 293K
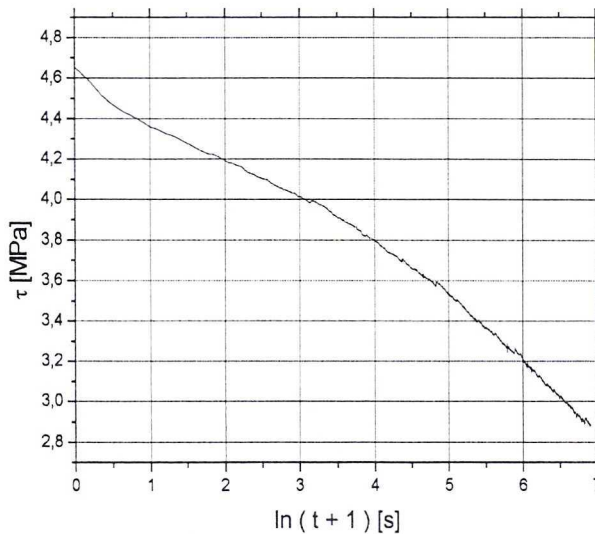

Fig. 7. Relaxation curve $\tau = f(\ln(t+1))$ of zinc monocrystal (sample 6), subjected to stress relaxation at the temperature 293K

The obtained relaxation curves for monocrystals of pure Zn can be divided into two stages (Fig. 2–7) characterized by two rectilinear sections on the curve $\tau = f(\ln(t+1))$. Basing on the experimental data it can be concluded that in the course of stress relaxation in zinc monocrystals the value of activated volume undergoes a change. As it has been demonstrated in the course of investigations, the kinetics of this change depends greatly on

temperature and the applied stress at the beginning of relaxation. The duration of the first step of relaxation for 77 K increases with increase of the applied stress at the beginning of relaxation. For the temperature 77 K the value $V*$ decreases in the second step of relaxation, whereas for the temperature 293 K it increases at the second stage of relaxation (Fig.8).
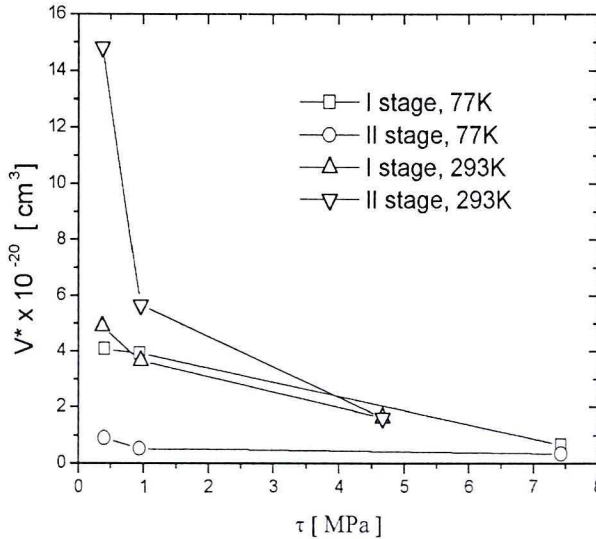

Fig. 8. Activation volumes determined for various relaxation stages and various temperatures

This is an indication of the existence of an intermediate temperature, at which the value of the activation volume remains constant during the whole relaxation process.

Increase of stress at the beginning of relaxation causes a strong reduction of the value $V*$. With stress increase to the values sufficient to activate secondary slip systems, the value of the activation volume becomes less and less dependent on temperature.

The above results allow to define certain features of the operating mechanism of relaxation. At the first stage the values of the activation volume, determined for both temperatures, are comparable. This is an indication of the operation of a relaxation mechanism independent of temperature. Significant qualitative and quantitative differences appear at the second stage of relaxation. It can be concluded that the mechanism operating here depends strongly on temperature and stress, which additionally requires period of incubation.

4. Summary

— The relaxation process in monocrystals of pure Zn proceeds in two stage,

— the duration of the first stage of relaxation depends on the relaxation stress and increases with increasing stress,

— the value of activation volume at the first stage of relaxation is independent of temperature and dependent on stress (decreases with stress increase),

— the value of the activation volume at the second stage strongly depends on stress and temperature.

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