A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 54 2009 Issue 1

B. MIKUŁOWSKI*, G. BOCZKAL*

# Zn-Ti SINGLE CRYSTALS DEFORMED ALONG THE BASAL SLIP SYSTEM

# MONIKRYSZTAŁY Zn-Ti ODKSZTAŁCANE W SYSTEMIE PODSTAWY SŁUPA HEKSAGONALNEGO

Zn-Ti single crystals of three different chemical compositions grown by the Brigman method were investigated. The microstructure of the materials was determined by scanning electron microscopy. The chemical composition of the matrix and of the second phase was also investigated. The second phase was identified as an intermetallic phase $Zn_{16}Ti$ exhibiting a strongly anisotropic distribution in the matrix. Transmission electron microscopy and electron back scattering diffraction showed that the [11 $\bar{2}$ 0] direction in the matrix is parallel to the [100] direction in $Zn_{16}Ti$ phase.

Sample orientation allowed on the deformation to $\gamma$=0.2 only in one slip system (0001)<11 $\bar{2}$ 0>. Mechanical properties (critical resolved shear stress and hardening coefficient in the range where easy slip operates) were obtained on the basis of compression test. Investigations were made in the temperature range of 77 K to 513 K and at two strain rates of $10^{-3}$ $s^{-1}$ and $10^{-4}$ $s^{-1}$.

*Keywords*: single crystal, second phase, crystal structure, precipitation hardening

Badano monokryształy Zn-Ti o trzech składach chemicznych, wyhodowane metodą Brigmana. Mikrostruktura materiałów była obserwowana przy użyciu skaningowego mikroskopu elektronowego (SEM). Badano także skład chemiczny osnowy oraz określono wydzielenia drugiej fazy. Faza ta została zidentyfikowana jako faza międzymetaliczna $Zn_{16}Ti$, która wykazuje stałą zależność krystalograficzną względem osnowy. Badania wykonane na transmisyjnym mikroskopie elektronowym (TEM) oraz przy użyciu techniki EBSD pokazały, że kierunek [11 $\bar{2}$ 0] w osnowie jest równoległy do kierunku [100] w fazie $Zn_{16}Ti$.

Orientacja badanych próbek pozwalała na zadanie deformacji postaciowej do wielkości $\gamma$=0.2 przy aktywności tylko jednego systemu poślizgu (0001)<1 $\bar{2}$ 0>. Własności mechaniczne (krytyczne naprężenie ścinające KNS oraz współczynnik umocnienia w zakresie działania łatwego systemu poślizgu $\Theta_A$) wyznaczano na podstawie próby ściskania. Badania prowadzono przy temperaturach w zakresie od 77K do 513K, przy dwóch prędkościach odkształcenia; $10^{-3}$ $s^{-1}$ oraz $10^{-4}$ $s^{-1}$.

## 1. Introduction

The study of the influence of second phase particles on the strength of single crystals was the subject of various studies by authors including Orowan [1], Ashby [2] and Ebeling [3]. These studies concerned first of all the hardening of A1 and A2 type lattice single crystals. Phases generated within the microstructure as a result of supersaturation and ageing ($CuAl_2$ – Dew-Huges and Robertson [4]) or introduced from outside ($SiO_2$ – Ebeling and Ashby [3]) constituted an obstacle to dislocations. The deformation range of single crystals with a Cu and an Al matrix, as it was established for the primary slip system, does not exceed a few percent [3]. Therefore metal single crystals showing a hexagonal lattice with a wide range of primary slip system operation are more suitable for the study of the hardening process. This applies for hexagonal metals where the c/a ratio is greater than 1.633. A low activation stress for easy slip in zinc and the lack of transverse slip systems enable a deformation up to a few dozens of percent (a shear strain e.g. $\gamma \cong 0.4$) [8,9].

The reason, which enables such a wide deformation range over the primary basal slip system, is the high stress required to activate secondary slip systems (pyramidal and prism systems) [10]. To activate secondary slip in zinc (c/a=1.856), deformed at 293 K, a shear stress of 10 to 15 MPa is required, whereas the critical resolved shear stress (CRSS) for the basal system reaches a value of 0.35 MPa [10-13]. Large strains along in a single easy slip system make it possible to perform studies of the hardening process in zinc single crystals

* AGH-UNIVERSITY OF SCIENCE AND TECHNOLOGY, DEPARTMENT OF STRUCTURE AND MECHANICS OF SOLIDS, 30-059 KRAKÓW, 30 MICKIEWICZA AV., POLAND

containing second phase particles. This is the subject of the present work.

## 2. Methods

Zn-Ti single crystals were obtained by the Bridgeman method [14], using 99.995 wt.% pure zinc. Titanium addition were 0.0094 wt.%, 0.0230 wt.% and 0.1001 wt.%. For the purpose of the study we have adopted the following simplified designation of the consecutive Zn-Ti single crystals, correspondingly: ZnTi0.01, ZnTi0.02 and ZnTi0.10. The crystals were oriented by the Laue back reflection method. Measurements were performed at both ends and at in the middle of a crystal. Then, samples were cut out for structural characterization and mechanical tests. Examination of the single crystal microstructure included the scanning electron microscopy (SEM) observations and measurements of the crystallographic orientation of individual phases by electron back scattering diffraction (EBSD) and transmission electron microscopy (TEM) as well. Including the titanium content in the matrix of individual single crystals was measured using an X-ray microanalyser.

The characterization of the mechanical properties by compression testing included the determination of the CRSS and the hardening coefficient ($\Theta_A$) within the easy slip range. Samples used were cut out in such a way as to ensure a wide deformation range along the $(0001)<11\bar{2}0>$ system. The compression axis orientation corespond to $\lambda_0 = 44°$, $\theta_0 = 45° \pm 1.5°$ ($\lambda_0$ – angle between the compression axis and the slip direction, $\Theta_0$ – angle between the compression axis and to the slip plane normal). The samples were deformed up to a shear strain $\gamma = 0.2$ at two strain rates: $\dot{\varepsilon}_1 = 10^{-4}s^{1}$ and $\dot{\varepsilon}_2 = 10^{-3}s^{-1}$, at temperatures of 77 K, 150 K, 293 K, 373 K, 473 K, 513 K.

## 3. Results of the study

The chemical composition of the Zn-Ti single crystals is presented in Fig.1 [15,16].

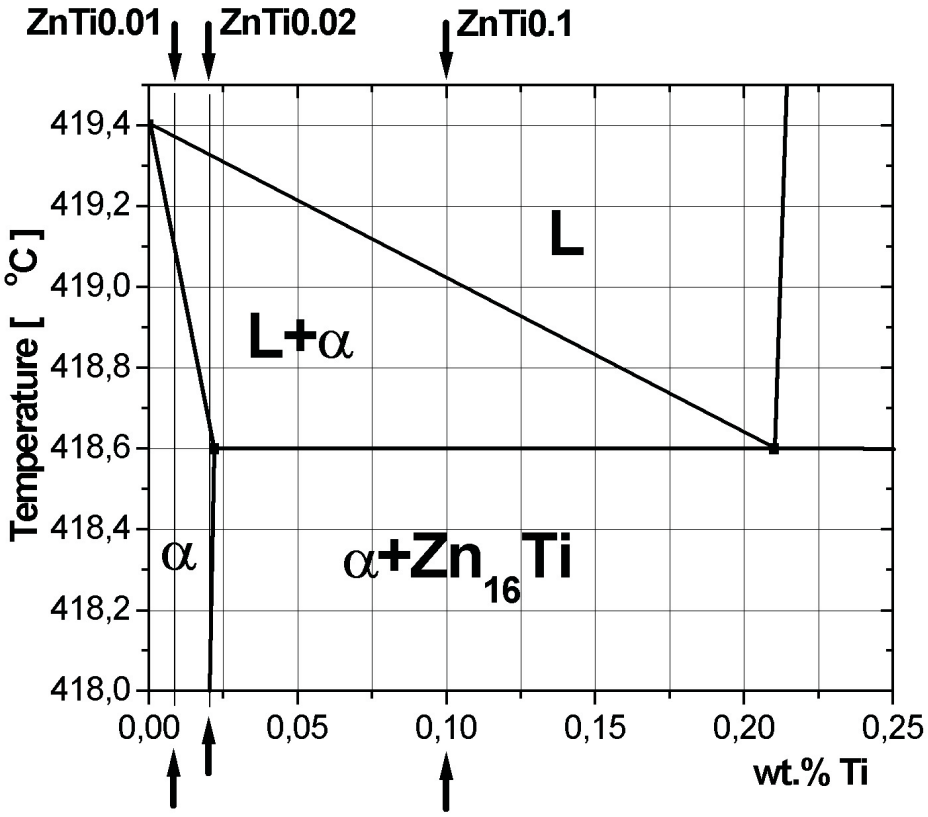


Fig. 1. Chemical compositions of the investigated single crystals marked on the Zn-Ti diagram [15,16]

The single crystal microstructures on the lateral faces of the deformed samples (SEM) are presented in Fig.2a-c.

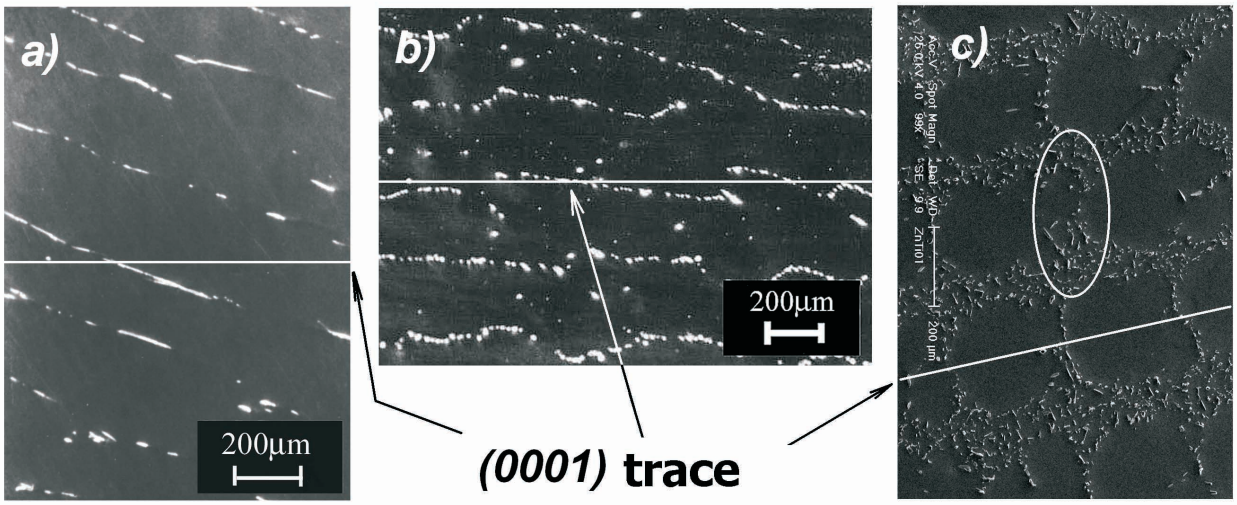


Fig. 2. Microstructures of the single crystals; (a) ZnTi0.01, (b) ZnTi0.02, (c) ZnTi0.10. (SEM microphotography)

A common feature of all of the examined microstructures is the occurrence of a needle-like phase, identified as $Zn_{16}Ti$ [17]. The precipitates of this phase create an ordered layer arrangement in the matrix. For individual single crystals the layer thickness and the distance between them are constant and constitute their characteristic parameter (Table 1).

TABLE

Structural parameters of the investigated single crystals ZnTi0.01, ZnTi0.02 and ZnTi0.10

| Single crystals | Total content titanium [wt.%] | Titanium content in matrix [wt.%] | Mean thickness of a layer containing $Zn_{16}Ti$ phase [μm] | Quantity of layers on 10 mm of sample height | Mean distance between following layers [μm] | % of filling of sample volume by layers of precipitates |
|---|---|---|---|---|---|---|
| ZnTi0.01 | 0.0094 | 0.012 | 6 | 58.2 | 165.7 | 3.49 |
| ZnTi0.02 | 0.0230 | 0.022 | 10 | 81.2 | 120.4 | 8.12 |
| ZnTi0.10 | 0.1001 | 0.022 | 56.6 | 48.5 | 127.3 | 27.4(34)* |
| (34)* – value with respect egsisting “the bridges” between following layers of the precipitates. | | | | | | |

The structural differences between Zn-Ti single crystals, which contain different titanium contents, consist in the amount of the $Zn_{16}Ti$ phase in the matrix. In ZnTi0.01 and ZnTi0.02 single crystals the layers rich in $Zn_{16}Ti$ needle-like precipitates are separated by precipitates free layers of the matrix, whereas, in case of ZnTi0.10 single crystals the precipitates also occur between the layers, in the form of characteristic “bridges” (Fig.2c).

Individual Zn-Ti single crystals differ in that the titanium contents in the matrix vary. The ZnTi0.01 matrix contains 0.010 ±0.002 wt.% of Ti, whereas, the titanium contents in the ZnTi0.02 and ZnTi0.10 matrix amounts up to 0.022 ±0.002 wt.% of Ti. The quantitative description of the microstructure of individual Zn-Ti single crystals is given in Table 1.

The precipitates show an anisotropy of their position in relation to the matrix orientation. The direction of the arrangement of individual needles of the $Zn_{16}Ti$ phase is parallel to the $(0001)\langle 11\bar{2}0\rangle$ direction in the single crystal matrix.

The crystallographic relation between the lattice of the $Zn_{16}Ti$ phase and that of the matrix was determined. TEM diffraction results are presented in Fig.3.

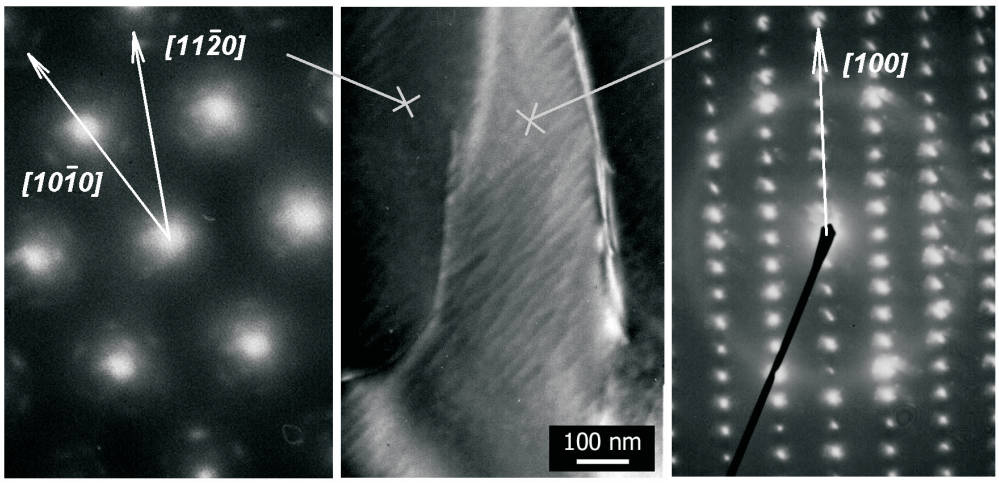


Fig. 3. Diffraction images of the hexagonal matrix plane (0001) (left) and the $Zn_{16}Ti$ tetragonal phase (right) (TEM)

Also a study by the EBSD technique was performed. It was found that the <100> directions in the $Zn_{16}Ti$ phase are parallel to the (0001)<11$\bar{2}$0> direction in the matrix. Fig. 4 - 7 present the changes in the CRSS and $\Theta_A$ value for individual Zn-Ti single crystals as a function of the temperature. For ZnTi0.01 and ZnTi0.02 single crystals the CRSS and $\Theta_A$ values are similar; this is obtained to the existence of matrix areas without precipitates separating layers rich in the $Zn_{16}Ti$ phase. In this case the prevailing hardening mechanism is that of solid solution hardening, a consequence of the titanium occurrence in the matrix [18,19].

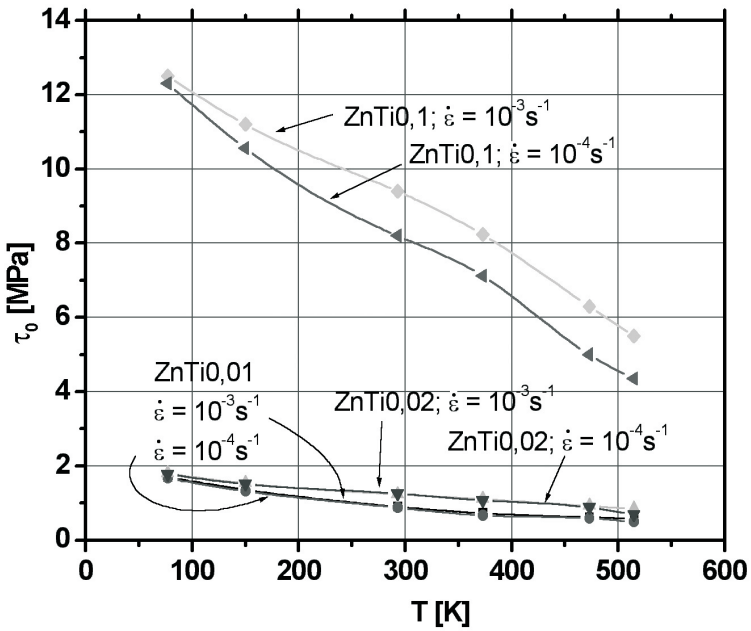


Fig. 4. Dependence between CRSS and temperature

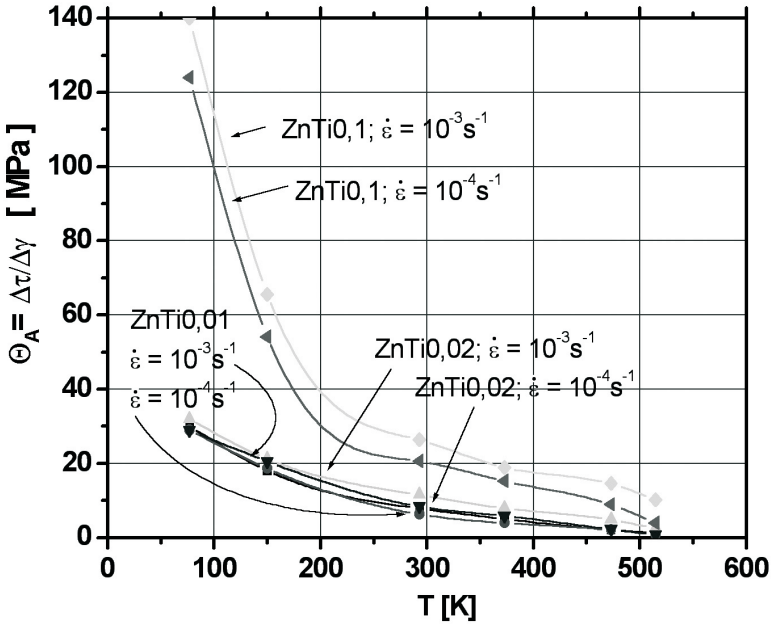


Fig. 5. Dependence between $\Theta_A$ and temperature. The hardening coefficient $\Theta_A$ was measured at a shear strain $\gamma$=0.2

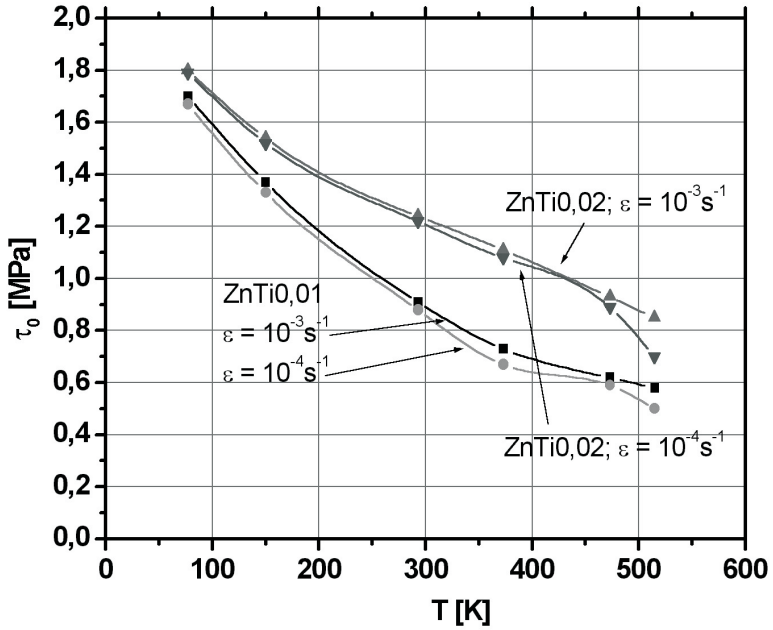


Fig. 6. Dependence between CRSS and temperature. Scale $\tau_0$ was optimized for the ZnTi0.01 and ZnTi0.02 single crystals

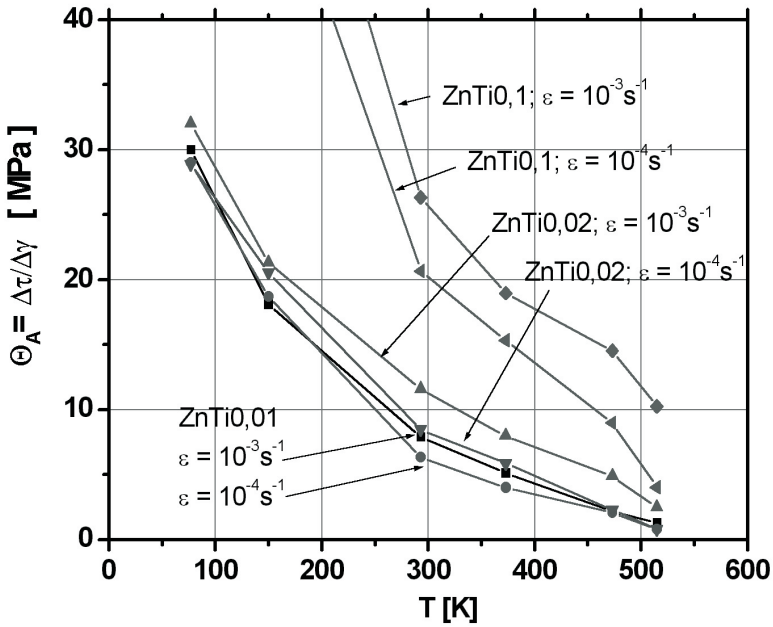


Fig. 7. Dependence between $\Theta_A$ and temperature. The hardening coefficient $\Theta_A$ was measured at a shear strain $\gamma$=0.2. Scale $\tau_0$ was optimized for the ZnTi0.01 and ZnTi0.02 single crystals

The ZnTi0.10 single crystal mechanical properties are much higher than that of ZnTi0.01 and ZnTi0.02. This is due to a much greater density of $Zn_{16}Ti$ needles in the layers and also to the existence of "bridges" (Fig.2c) which create skeleton construction within the matrix. Small distances between the precipitates cause the stress fields of individual needles to interact. Consequently a stress field of a differentiated potential is generated within the microstructure [20]. The slip dislocations being a distortion carrier, their stress fields interact with the stress fields of the precipitates [21]. This leads then a hardening which is the sum of components the solid solution hardening and the influence of second phase particles.

## 4. Summary

The Zn-Ti single crystal microstructure depends on the titanium content. In all examined single crystals, i.e. ZnTi0.01, ZnTi0.02 and ZnTi0.10, there are intermetallic Zn16Ti phase precipitates. The quantity of these precipitates depends on the titanium addition and their orientation is strongly related to the matrix crystallographic orientation. These precipitates create a layer arrangement. The Zn-Ti single crystal matrix is solution hardened by a 0.010...0.022 wt. % addition of Ti. In the case of ZnTi0.01 and ZnTi0.02 single crystals containing only a small amount of second phase, a prevailing mechanism is solid solution hardening, while ZnTi0.10 single crystals are hardened in addition by the stress fields of the densely arranged intermetallic $Zn_{16}Ti$ phase needles.
For all the examined examples it has been established that the influence of strain rate from $\dot{\varepsilon}_1 = 10^{-4}s^{-1}$ to $\dot{\varepsilon}_2 = 10^{-3}s^{-1}$ upon the hardening characteristics of Zn-Ti single crystals is not essential. This results from a weak influence of the strain rate on the slip dislocation movement in zinc [22,23] The temperature, however, influences the Zn-Ti hardening curve shape strongly. At 373 K a bend in the CRSS = f (T) curve can be observed, which results from the activation of dislocation climb, which is characteristic for zinc deformed at elevated temperature [11].

**Acknowledgements**

The autors acknowledge the support of the Polish Committee for Scientific Research, Grant No. N N507 4617 33.

REFERENCES

[1] E. Orowan, Fracture and Stress of Solids, Rep.Progr.Phys. **12**, 185-232 (1948/49).
[2] M. F. Ashby, Oxide Dispersion Strengthening, (ed. G.S.Ansell et al.), Gordon and Breach, New York 1958.
[3] R. Ebeling, M. F. Ashby, Phil.Mag. **13** 805-834 (1996).
[4] D. Dew-Huges, W. D. Robertson, Act.Metall. **8** 147-155 (1960).
[5] J. G. Byrne, M. E. Fine, A. Kelly, Phil.Mag 1119-1145 (1961).
[6] A. Kelly, M. E. Fine, Acta Metall. **5** 365-367 (1957).

[7] N. Z a r u b o v a, B. S e s t a k, Phys.Stat.Sol. **30** 365-374 (1975).
[8] B. M i k u ł o w s k i, G. B o c z k a l, Arch.of Metall.and Mat. **49** 37-42 (2004).
[9] G. B o c z k a l, B. M i k u l o w s k i, Journ. of Alloys and Comp. **378** 135-139 (2004).
[10] R. L. B e l l, R. W. C a h n, Proc.Roy.Soc. **A239** 494-521 (1957).
[11] B. M i k u l o w s k i, Strain Hardening of Zinc Monocrystals with Additions of Silver or
[12] Gallium, Metallurgy and Foundry Practice, Scientific Bulletin of Univ. of Mining and Metallurgy **96**, Cracow 1982.
[13] G. B o c z k a l, B. M i k u l o w s k i, Arch.of Metall. **48** 11-19 (2003).
[14] P. L u k a c, Z.Metallkde.Bd.57, H.7 559-562 (1966).
[15] W. D. L a w s o n, S. N i e l s e n, Preparation of Single Crystals, (Butterworths Scientific Publications), London 1958.
[16] V o n W. H e i n e, U. Z w i c k e r, Z.Metallkde. Bd. 53, H.6 380-385 (1962).
[17] J. L. M u r r a y, Phase Diagrams of Binary Titanium Alloys, ed.ASM International, Metals Park, OH, 336-339 (1987).
[18] M. S a i l l a r d, G. D e v e l e y, C. B e c l e, J. M. M o r e a u, D. P a c c a r d, Act.Cryst. Section B **37B** 224-226 (1981).
[19] P. K r a t o c h v i l, E. N e r a d o v a, Czech.J.Phys. **B21** 1273-1278 (1971).
[20] P. L u k a c, Mechanicke Vlasnosti Pevnych Latek, Ed. SPN Praha 1982.
[21] N. F. M o t t, F. R. N. N a b a r r o, Proc.Phys.Soc. **52** 86-89 (1940).
[22] T. G l a d m a n, Mat.Sci.and Tech. **15** 30-36 (1999).
[23] P. L u k a c, Czech.J.Phys. **B35** 275-285 (1985).
[24] P. L u k a c, Czech.J.Phys. **B31** 135-141 (1981).

*Received: 10 April 2008.*