

Study of twinning in texture-free cast magnesium using acoustic emission technique

K. Máthis*, J. Čapek, P. Hrcuba, V. Šíma

Department of Physics of Materials, Charles University, Ke Karlovu 5, 121 16 Prague, Czech Republic

Received 9 January 2013, received in revised form 4 March 2013, accepted 27 March 2013

Abstract

The acoustic emission (AE) response of texture-free cast magnesium was studied *in-situ* during monotonic tension and compression tests at room temperature. The dependence of twinning evolution on loading mode is discussed in detail. Furthermore, it is shown that the contribution of twinning and dislocation slip to AE signal is distinguishable by means of analysis of AE parameters. The findings are corroborated by light optical microscopy and EBSD results, as well.

Key words: magnesium alloy, acoustic emission, twinning, electron backscattered diffraction

1. Introduction

Increasing demand of governments for lowering the greenhouse gas emissions forces car manufacturers to use lightweight components in their products. Magnesium alloys belong to the hot candidates, since the weight saving in certain car parts might be more than 50 %, in comparison with the commonly used aluminum alloys [1]. Nevertheless, their limited formability at ambient temperature provokes numerous technological problems, which impede their wider application. Thus, the better understanding of the plastic deformation mechanisms of magnesium alloys has been of recent interest.

The poor formability is usually rationalized in terms of limited number of equivalent slip systems in magnesium. At room temperature the dominant slip system is the basal one in $\langle a \rangle$ direction, since the critical resolved shear stresses (CRSS) of the other feasible systems (prismatic $\langle a \rangle$; pyramidal $\langle a \rangle$ and $\langle c + a \rangle$) are several orders higher than those for the basal slip [2], and therefore their activation is rather difficult. Therefore, twinning provides an additional deformation mechanism, through which the homogeneous deformation is achievable [3]. Because of its polar nature, the twinning has a different development in tension and compression, respectively [4, 5], and

usually causes the so-called tension-compression anisotropy [4]. It is obvious that detailed study of twinning plays a key role in understanding of deformation mechanisms of magnesium alloys.

The acoustic emission method detects elastic waves released during local, dynamic and irreversible changes in the (micro)structure of the material. Therefore it belongs to the most powerful *in-situ* experimental techniques for investigation of both dislocation movement and twinning [6–8]. Separation of the AE signal of twinning from dislocation signals is usually performed by means of threshold discrimination [6, 9] or statistical methods [10, 11]. In the first case it is presumed that the magnitude of twinning signals is higher than that for dislocations. Thus, two threshold levels are used: the first threshold level is set directly above the peak values of thermal noise, and the measured AE count is assumed to be the sum of the response of all deformation mechanisms detectable by AE. The burst AE count, measured at the second threshold level, is only used for gathering signals coming from strong collective effects, e.g., twinning [12]. Since the estimation of proper value of the second threshold level is rather difficult, not all twinning signals are necessarily detected. The statistical methods use very sophisticated pattern recognition technique [13, 14], which requires dedicated software. Recently,

*Corresponding author: tel.: +420-221-911-458; fax: +420-221-911-490; e-mail address: mthis@met.mff.cuni.cz

Li and Enoki [15] published a discrimination method based on frequency analysis of the recorded AE waves. They studied *wrought* magnesium alloys in compression and found that the peak frequency of twinning and dislocation movement are considerably different.

In the literature, the great majority of papers deal with twinning in *wrought* (i.e., textured) magnesium alloys, whereas articles describing *random textured* materials are less frequent [16–18]. In the current work, the AE response of *texture-free cast* magnesium was studied during monotonic tension and compression tests and at room temperature. The AE measurements revealed differences in evolution of deformation mechanisms during tension and compression, respectively. Link between the particular deformation mechanisms and AE parameters (amplitude, peak frequency) is discussed in detail. The findings are corroborated by light optical microscopy and electron back-scattered diffraction (EBSD) observations.

2. Experimental

Polycrystalline magnesium with 1 wt.% Zr content was used for this experimental study. The specimens had an average grain size of 110 μm . The testing was carried out using cylindrical specimens with a diameter of 9 mm and gauge length of 20 mm. Beyond the common tensile and compression experiments at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$, AE was monitored using a computer controlled PCI-2 device (Physical Acoustic Corporation). The facility incorporated a piezoelectric transducer with a flat response between 50 and 650 kHz and a preamplifier giving a gain of ~ 40 dB. The threshold level of detection was set as 26 dB.

The specimens for light optical microscopy were mechanically polished and etched in a solution of 10 % nitric acid. The grain orientation relations were studied by means of EBSD technique, working on a FEI Quanta 200 FEG scanning electron microscope. The specimens for EBSD investigations were first mechanically polished, than etched in AC-2 electrolyte, manufactured by Struers.

3. Results and discussion

The EBSD map of the initial state is shown in Fig. 1. The grain size was determined by linear intercept method as 110 μm . It is obvious from the pole figure (Fig. 1b) that the specimen had a random texture. The true stress-true strain curves are shown in Fig. 2. It is obvious that the loading mode has appreciable effect on the hardening. As it can be seen in Fig. 3, the compressed specimen exhibits a sigmoid shape: after the initial drop between 60 and 110 MPa, the work hardening rate increases (Fig. 3a). On the

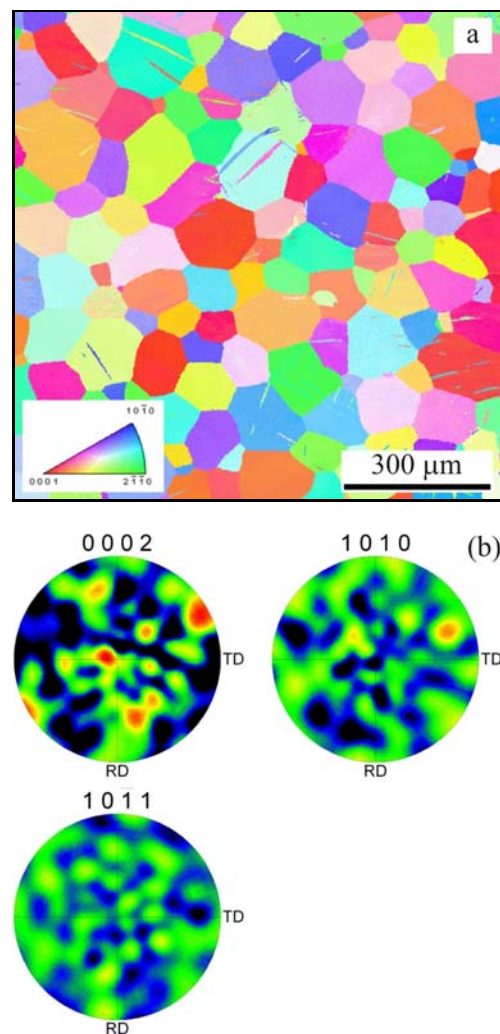


Fig. 1. a) EBSD map and b) the corresponding orientation map of the initial structure.

contrary, in tension beyond the yield point ($\sigma_{02} = 46$ MPa) the work hardening rate θ is virtually constant (Fig. 3b). The strain dependence of the AE count rate, depicted in Fig. 2, also varies with the loading modes. In compression (Fig. 2a), the count rate reaches its maximum at approx. 1 % of true strain followed by a rapid decrease to zero. In tension (Fig. 2b) the curve's fall-off is gradual; AE activity was observed practically during the whole test. In accordance with the scanning electron microscopy [19] and neutron diffraction findings [20, 21], the main contribution to the first AE peak is given by the slip of basal $\langle a \rangle$ dislocations and the $\{10\bar{1}2\} \langle 10\bar{1}1 \rangle$ twinning [12, 22]. The subsequent steep decrease of AE count rate in *compression* can be rationalized in terms of rapid growth of nucleated twins, which was observed in numerous neutron diffraction experiments [21, 23, 24]. The surface displacement caused by twin growth may be estimated as imponderable, of order 10^{-21} m, since the

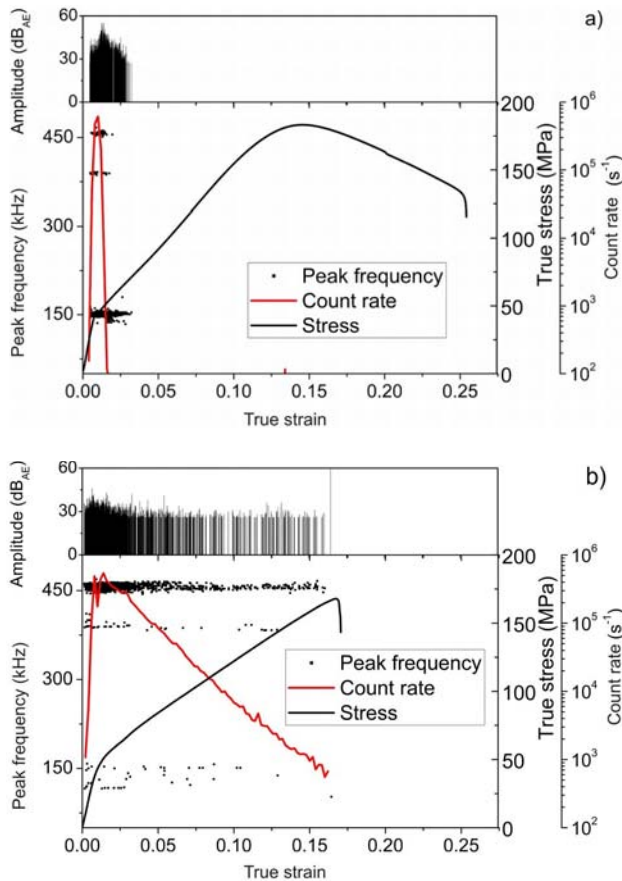


Fig. 2. Dependence of the AE count rate, AE peak frequency, AE peak amplitude and true stress on the true strain: a) in compression, b) in tension.

growth velocity of an elliptical twin is only 10^{-3} m s^{-1} [8, 25]. Consequently, the AE signal of twin growth is far below the detectable limit [26]. Furthermore, the basal dislocations pile up on the new impenetrable twin boundaries [27] and, therefore, the mean free path of dislocations is reduced. Since this parameter is directly proportional to the AE activity [7], this mechanism also contributes to the decreasing of the AE signal.

The twinning evolution in *tension* is rather different. The twin growth owing to the back stresses of neighboring grains is limited [28, 29]. Thus, persistent twin nucleation is required for the continuation of the plastic deformation that causes burst AE signal during the entire straining and broadening of the AE count rate peak.

The spectral analysis was used by several authors for partitioning of AE signals of particular deformation processes at different stages of straining [14, 15]. Figure 2 shows the peak frequency spectrum of AE signals as a function of the true strain, calculated by means of Fast Fourier Transformation. It is obvious that there are two distinct frequency domains (125–

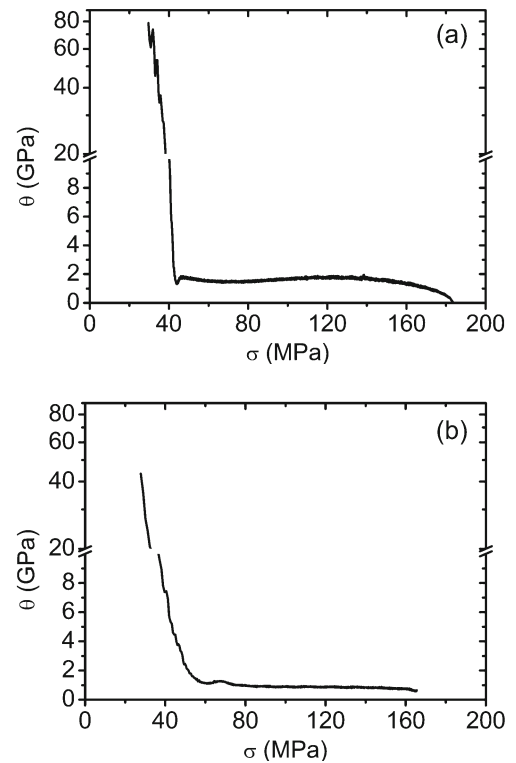


Fig. 3. Stress dependence of the work hardening curve: a) in compression, b) in tension.

160 kHz and 380–470 kHz). As it was discussed recently by Li and Enoki [15], the AE events situated in lower frequency domain are caused by dislocation motion, whereas the higher frequency domain contains signals of twinning. The distinctly different peak frequencies originate in different propagation velocities of dislocation and twin boundary movements. The atomistic simulations revealed that at low applied strains the dislocations moved at 65–70 % of transversal velocity of sound c_T [30]. On the contrary, the high speed camera records [31] indicate that the velocity of twin nucleation lies in the transonic regime ($1.3\text{--}1.6 c_T$). The light optical microscopy and EBSD observations confirmed the above mentioned findings. It is obvious from the Fig. 4, where the microstructures after 2 % straining both in tension and compression are depicted, that the twins in compressed specimens are thick (Fig. 4a), whereas in tension a large number of thin twins is visible (Fig. 4b). The EBSD maps of specimens at 10 % of strain (Fig. 5) show rather different orientation relations in tension and compression, respectively. In tension there are twins observable within the grains, but the random orientation character of the specimens remained (Fig. 5b). In compression the twin growth is not hindered, thus the twins can reorient the whole grain. Consequently, at high strain levels a strong basal texture was formed (cf. Fig. 5a). Most probably the rapid twin growth could give a reason for

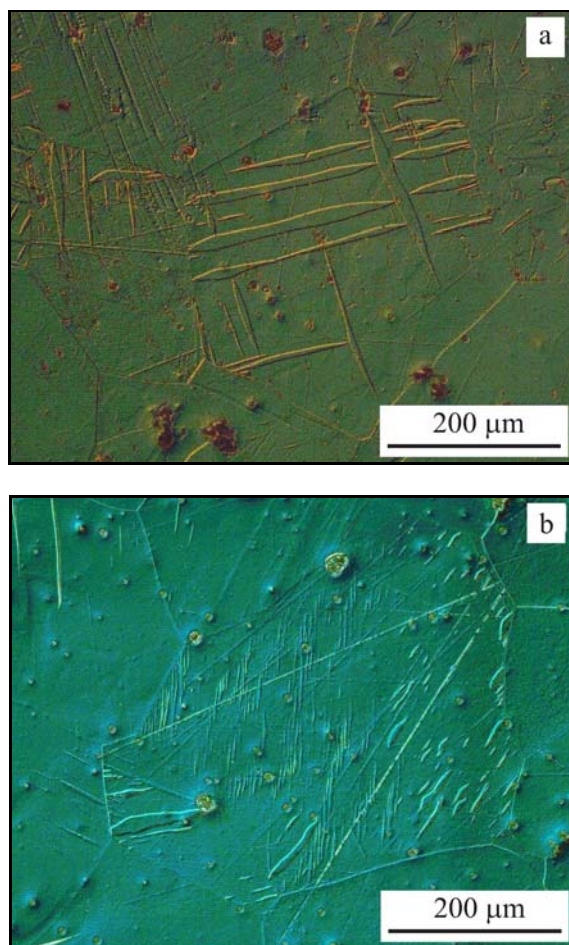


Fig. 4. The microstructures after 2 % straining: a) in compression, b) in tension.

the sigmoid shape of the work hardening curve. The twinning mechanism is exhausted, when the twins “fill out” the grains. Thus a new mechanism is required for continuation of the plastic deformation. The prismatic $\langle a \rangle$ slip and the pyramidal $\langle c + a \rangle$ slip are the most probable candidates according to the both experimental [32] and theoretical findings [4]. Since the non-basal dislocations increase the forest dislocation density [33], hardening occurs.

The amplitude distribution of the AE events is in accordance with the above mentioned findings (Fig. 2). As it is evident from Fig. 4, the size in tension observed twins is only around $20 \mu\text{m}$, while the twin length in compression is comparable with the about five times higher grain size. This corresponds also with the observed higher peak amplitudes in compression tests than in tensile test (Fig. 2).

4. Conclusions

The AE response of texture-free cast magnesium

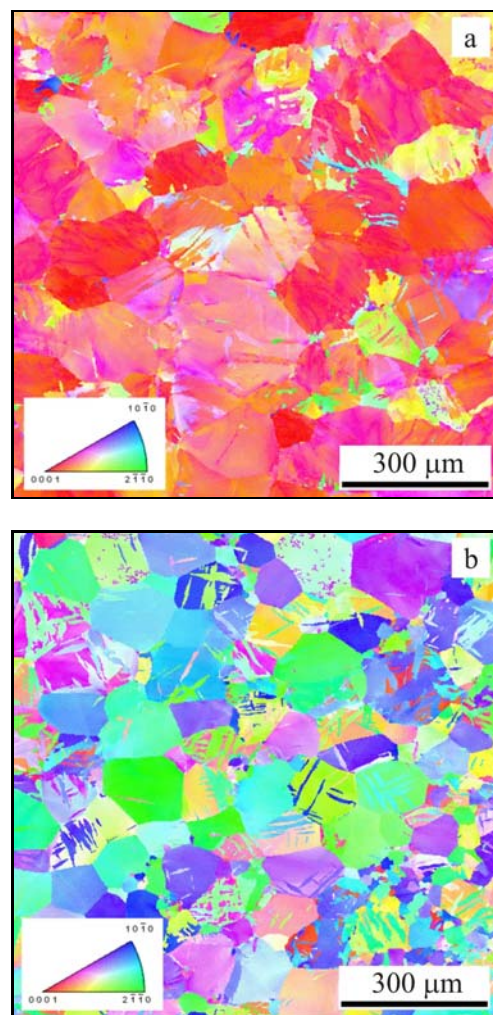


Fig. 5. EBSD maps of specimens at 10 % of strain: a) in compression, b) in tension.

was monitored during monotonic tension and compression tests at room temperature. The following conclusions may be drawn:

- The evolution of twinning $\{10\bar{1}2\} \langle 10\bar{1}1 \rangle$ differs in tension and compression, respectively. In compression, the twin nucleation takes place at the beginning of the straining followed by a rapid twin growth. On the contrary, in tension the twin thickening is limited and therefore twins nucleate during the entire test. The AE count rate corresponds to this behavior: in compression a sharp single peak is observed, whereas in tension the peak broadens towards the higher strains.

- The peak frequency analysis revealed two distinct domains related to dislocation slip ($\sim 150 \text{ kHz}$) and twinning ($\sim 450 \text{ kHz}$). In compression the “twinning frequencies” appear at shorter strain scales than in tension that corresponds to the above discussed mechanisms.

- Both the AE peak amplitude distribution and

the optical micrographs bear out that the size of the nucleated twins in compression is larger.

Acknowledgements

The authors are grateful for the financial support of the Czech Science Foundation under the contract P204/12/1360. JČ acknowledges the support from the Grant Agency of Charles University under contract Nr. 513512.

References

- [1] Mordike, B. L., Ebert, T.: *Mater. Sci. Eng. A*, 302, 2001, p. 37. [doi:10.1016/S0921-5093\(00\)01351-4](https://doi.org/10.1016/S0921-5093(00)01351-4)
- [2] Chapuis, A., Driver, J. H.: *Acta Mater.*, 59, 2011, p. 1986. [doi:10.1016/j.actamat.2010.11.064](https://doi.org/10.1016/j.actamat.2010.11.064)
- [3] Kelly, E. W., Hosford, W. F.: *Trans Met Soc AIME*, 242, 1968, p. 654.
- [4] Agnew, S. R., Duygulu, O.: *Int. J. Plast.*, 21, 2005, p. 1161. [doi:10.1016/j.ijplas.2004.05.018](https://doi.org/10.1016/j.ijplas.2004.05.018)
- [5] Máthis, K., Čapek, J., Zdražilová, Z., Trojanová, Z.: *Mater. Sci. Eng. A*, 528, 2011, p. 5904. [doi:10.1016/j.msea.2011.03.114](https://doi.org/10.1016/j.msea.2011.03.114)
- [6] Král, R., Dobroň, P., Chmelík, F., Koula, V., Rydlo, M., Janeček, M.: *Kovove Mater.*, 45, 2007, p. 159.
- [7] Scruby, C., Wadley, H., Sinclair, J. E.: *Philos. Mag. A*, 44, 1981, p. 249. [doi:10.1080/01418618108239532](https://doi.org/10.1080/01418618108239532)
- [8] Carpenter, S. H., Chen, C. M.: *J. Acoust. Em.*, 7, 1988, p. 161.
- [9] Dobroň, P., Bohlen, J., Chmelík, F., Lukáč, P., Letzig, D., Kainer, K. U.: *Kovove Mater.*, 45, 2007, p. 129.
- [10] Lu, Y., Gharghour, M., Taheri, F.: *NDT Eval*, 23, 2008, p. 211.
- [11] Kontsos, A., Loutas, T., Kostopoulos, V., Hazeli, K., Anasori, B., Barsoum, M. W.: *Acta Mater.*, 59, 2011, p. 5716. [doi:10.1016/j.actamat.2011.05.048](https://doi.org/10.1016/j.actamat.2011.05.048)
- [12] Máthis, K., Chmelík, F., Janeček, M., Hadzima, B., Trojanová, Z., Lukáč, P.: *Acta Mater.*, 54, 2006, p. 5361. [doi:10.1016/j.actamat.2006.06.033](https://doi.org/10.1016/j.actamat.2006.06.033)
- [13] Loutas, T. H., Kostopoulos, V.: *Compos. Sci. Technol.*, 69, 2009, p. 265. [doi:10.1016/j.compscitech.2008.07.020](https://doi.org/10.1016/j.compscitech.2008.07.020)
- [14] Vinogradov, A. V., Patlan, V., Hashimoto, S.: *Philos Mag. A*, 81, 2001, p. 1427. [doi:10.1080/01418610108214356](https://doi.org/10.1080/01418610108214356)
- [15] Li, Y. P., Enoki, M.: *Mater. Sci. Eng. A*, 536, 2012, p. 8. [doi:10.1016/j.msea.2011.10.010](https://doi.org/10.1016/j.msea.2011.10.010)
- [16] Nagarajan, D., Caceres, C. H., Griffiths, J. R.: *Acta Phys Pol A*, 122, 2012, p. 501.
- [17] Caceres, C. H., Blake, A. H.: *Mater. Sci. Eng. A*, 462, 2007, p. 193. [doi:10.1016/j.msea.2005.12.113](https://doi.org/10.1016/j.msea.2005.12.113)
- [18] Caceres, C. H., Lukac, P., Blake, A. H.: *Philos Mag. A*, 88, 2008, p. 991. [doi:10.1080/14786430701881211](https://doi.org/10.1080/14786430701881211)
- [19] Hazeli, K., Cuadra, J., Vanniamparambil, P. A., Kontsos, A.: *Scripta Mater.*, 68, 2013, p. 83. [doi:10.1016/j.scriptamat.2012.09.009](https://doi.org/10.1016/j.scriptamat.2012.09.009)
- [20] Máthis, K., Beran, P., Čapek, J., Lukáč, P.: *JoP: Conference Series*, 340, 2012, p. Art. No.: 012096.
- [21] Muránský, O., Carr, D. G., Šittner, P., Oliver, E. C.: *Int J Plasticity*, 25, 2009, p. 1107. [doi:10.1016/j.ijplas.2008.08.002](https://doi.org/10.1016/j.ijplas.2008.08.002)
- [22] Friesel, M., Carpenter, S. H.: *J. Acoust. Em.*, 6, 1984, p. 11.
- [23] Gharghour, M. A., Weatherly, G. C., Embury, J. D., Root, J.: *Philos Mag A*, 79, 1999, p. 1671. [doi:10.1080/01418619908210386](https://doi.org/10.1080/01418619908210386)
- [24] Máthis, K., Beran, P., Harcuba, P., Čapek, J., Lukáč, P.: In: *Proceedings 9th International Conference on Magnesium Alloys and their Applications*. Eds.: Poole, W. J., Kainer, K. U. TMS 2012, p. 84. PMID:22554253 PMCID:PMC3495408
- [25] Papiro, I. I., Karpov, E. S., Palatnik, M. I., Mileskin, M. B.: *Met. Sci. Heat Treat.*, 26, 1984, p. 887. [doi:10.1007/BF00801000](https://doi.org/10.1007/BF00801000)
- [26] Miller, R. K., Hill, E. V. K.: *Acoustic Emission Testing*. Columbus, American Society for Nondestructive Testing 2005.
- [27] Yoo, M. H.: *J. Metall. Trans. A*, 12, 1981, p. 409. [doi:10.1007/BF02648537](https://doi.org/10.1007/BF02648537)
- [28] Koike, J.: *Metall. Mater. Trans. A*, 36, 2005, p. 1689. [doi:10.1007/s11661-005-0032-4](https://doi.org/10.1007/s11661-005-0032-4)
- [29] Christian, J. W., Mahajan, S.: *Progress Mater. Sci.*, 39, 1995, p. 1. [doi:10.1016/0079-6425\(94\)00007-7](https://doi.org/10.1016/0079-6425(94)00007-7)
- [30] Gumbsch, P., Gao, H.: *Science*, 283, 1999, p. 965. PMID:9974385. [doi:10.1126/science.283.5404.965](https://doi.org/10.1126/science.283.5404.965)
- [31] Finkel, V. M., Voronov, I. N., Savelyev, A. M., Yeliseyev, A. I., Fedorov, V. A.: *Phys. Met. Metall. USSR*, 29, 1970, p. 131.
- [32] Knezevic, M., Levinson, A., Harris, R., Mishra, R. K., Doherty, R. D., Kalidindi, S. R.: *Acta Mater.*, 58, 2010, p. 6230. [doi:10.1016/j.actamat.2010.07.041](https://doi.org/10.1016/j.actamat.2010.07.041)
- [33] Lukáč, P.: *Czech. J. Phys.*, 35, 1985, p. 275. [doi:10.1007/BF01605096](https://doi.org/10.1007/BF01605096)