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Determination of the stress threshold and microstructural factors forming the nonlinear unloading effect of the ZK60 (MA14) magnesium alloy

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Abstract: Magnesium alloys are an ideal material for creating lightweight and durable modern transport systems, but their widespread use is limited due to some physical and chemical properties. This paper considers the effect of nonlinear elastic unloading of the MA14 (ZK60, Mg–5.4Zn–0.5Zr) magnesium alloy in a coarse-grained state after recrystallisation annealing. The study found that the nonlinearity of the unloading characteristic, is formed when reaching a certain threshold stress level. It is expected that the effect under the study is associated with the deformation behavior of the alloy, during which the twin structure formation according to the tensile twinning mechanism is observed. The sample material microstructure was determined, by scanning electron microscopy using electron backscattered diffraction analysis. Determination of the threshold stress, for the formation of unloading nonlinearity was carried out by two methods: 1) by the value of the loop area formed by the nonlinearity of the unloading mechanical characteristics and the repeated loading (mechanical hysteresis) characteristics, and 2) by analysing the acoustic emission recorded during failure strain. A comparison of the results obtained, allows suggesting that the unloading nonlinearity is caused by twinning in grains, in which an unfavorable configuration (low Schmidt factor), for dislocation slip is observed. Rotating the twinned crystal at an angle close to 90° does not contribute to an increase in the Schmidt factor and activation of dislocation slip systems to secure the deformed structure through the dislocation strengthening mechanism. With a subsequent decrease in the external stress, detwinning and partial restoration of the crystal lattice configuration occur.

Keywords: magnesium; magnesium alloy; ZK60 (Mg–5.4Zn–0.5Zr); nonlinear unloading; stress threshold; elasticity; twinning; detwinning; deformation behavior.

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INTRODUCTION

Lightweight magnesium alloys have a high strength-to-weight ratio, which makes them extremely attractive for application in vehicle construction. Currently, magnesium alloys are used primarily for casting into moulds or manufacturing elements using turning and milling from rolled products (plates, sheets, rods, and pipes). Elastic forging, and other methods of pressure metal treatment (PMT) of magnesium alloys are significantly limited or require the application of alloys with relatively high doping with expensive elements, which significantly reduces the attractiveness of magnesium for widespread use. The problems of pressure metal treatment of magnesium alloys, especially in the cast or coarse-grained state, are associated with the peculiarities of their deformation behaviour manifested in the form of: asymmetry of the mechanical response to the application of tensile, and compressive loads; highly limited plasticity, even at elevated shaping temperatures; nonlinear characteristics of elastic behaviour (unloading) [1–6].

The peculiarities of magnesium hardening and plasticity are associated with its hexagonal close-packed (hcp) crystal lattice, which is the cause of strong anisotropy of elastic and deformation properties. It is quite difficult to get rid of the anisotropy manifestation, so many authors are forced to study magnesium alloys, including after hardening deformation treatment, in several directions. For example, for rolling (forging), three directions are selected (normal, longitudinal, transverse), for extrusion – two (normal, transverse) [7].

When a magnesium alloy is compressed and tensioned, pronounced strain anisotropy and deformation behaviour asymmetry are observed, manifested in a significant difference in the yield strengths during tension and compression [8–10], and the initial hardening may be a reason for the material asymmetric behaviour [11]. In the above works, one can note the nonlinear behaviour of the unloading characteristics of magnesium alloys, but the authors do not specify the reasons and parameters for the formation of such an effect.

The purpose of this work is to determine the conditions and reasons for the formation of nonlinear elastic behaviour, during unloading of the MA14 magnesium alloy.

METHODS

The chemical composition of magnesium alloy samples was pre-studied according to the GOST 7728-79 method, using a Thermo Fisher Scientific ARL 4460 optical emission spectrometer. Table 1 gives the chemical composition of the studied material; the element content values correspond to the requirements for the MA14 grade according to GOST 14957-76.

The material under study has a coarse-grained structure with a grain size of 60...120 μm and a texture corresponding to homogenising recrystallisation annealing (not textured). Fig. 1 shows an image of the alloy structure obtained on a Zeiss Sigma scanning electron microscope, using electron backscattered diffraction analysis (SEM+EBSD), as well as histograms of the grain size distribution, and the crystal lattice misorientation at the grain boundaries. Determination of the mechanical characteristics of the alloy, under cyclic loading, and monotonic tensioning until failure was carried out on flat samples with blades for grippers, sample working section 4×4 mm, length 4 mm. The samples were cut using the electroerosive method on a numerically controlled machine, which makes it possible to obtain samples of identical geometry without hardening the surface layer.

The test specimens were prepared in the same initial state (after homogenising annealing), and were not subjected to initial deformation or hardening, before both compression and tensile testing, and when installing the specimens in the grips of the testing machine, the procedure for protecting the specimen from loading prior to testing was followed.

To study the reasons forming the nonlinearity of elastic behaviour and unloading, the authors performed a series of tests with a loading – unloading cycle with a maximum stress point below, near and above the yield strength at engineering stress of σ_{eng}≈50, 90, and 145 MPa, respectively, and the active grip displacement rate corresponded to a strain rate of $1\cdot10^{-4}$ s^{-1}. To measure small deformations in the elastic loading area, an HBM MX440 strain-gauge complex with strain-elements glued directly to the sample surface was used. Magnesium and its alloys are prone to creep [12] and long intervals of relaxation of elastic stresses [13], therefore, to reduce the influence of the viscous-dynamic component of plasticity, the sample was statically kept under load for a long time relative to the loading interval, and after unloading – until the activity of deformation processes decreased. The activity of deformation processes was monitored by the acoustic emission (AE) signals. Mechanical twinning, during deformation of magnesium alloys, generates discrete high-amplitude acoustic pulses [14], therefore, when the AE activity decreased to less than one signal/s, the authors considered activity of the deformation process during relaxation insignificant and proceeded to the next stage of the loading–unloading cycle. AE was recorded on the PAC PCI-2 equipment, in a wideband mode of 20 kHz – 1 MHz, with a sampling frequency of 2 MHz, gain of +60 dB, the amplitude detector threshold was 27 dB at a noise level of 25 dB. After testing, the sample material from the deformed area was re-examined using scanning electron microscopy with structure analysis.

To test the assumption that the peculiarities of the "dislocation sliding" and "twinning", deformation mechanisms may be the reasons for the nonlinear elastic behaviour formation, the same sample was tested for tension to failure at a strain rate of $1\cdot10^{-3}$ s^{-1}. During the stretching process, the AE signal was recorded in a continuous (threshold-free) mode, synchronised with the stress/strain parameters. To assess the staging, the authors used the spectral-energy parameters of AE signals [15; 16]: in this case, for the recorded signal, the change in two parameters of the spectral characteristic resulting from the applied mechanical stress was assessed: 1) power – the integral of the signal power spectral density and 2) median frequency – the median of the signal power spectral density calculated according to the method described in [17].

RESULTS

The tests of cyclic tension and compression without changing the deformation direction relative to the original direction are shown in Fig. 2, which presents three loading cycles separately for compression and tension. The first loading up to a stress of 50 MPa demonstrates elastic loading, and a linear return of the material to its original state, and the slope of the linear loading and unloading section corresponds to an elastic modulus of 42...44 GPa. In the second (90 MPa) and third (145 MPa) loading cycles, the elastic section nonlinearity is observed, which can be estimated according to the area of the hysteresis loop formed by the loading and unloading lines.

Table 1. *Chemical composition of the studied alloy*
Таблица 1. *Химический состав исследуемого сплава*

Materials	**Element weight content, %**								
	Mg	**Zn**	**Zr**	**Al**	**Fe**	**Cu**	**Ni**	**Mn**	**Si**
Studied material	Base	5.4	0.47	0.002	0.001	0.002	0.001	0.005	0.003
MA14 according to GOST 14957-76	Base	5.0–6.0	0.3–0.9	≤0.05	≤0.03	≤0.05	≤0.005	≤0.1	≤0.05

For each loop, the authors recorded the stress at the top of the loop and the energy volumetric density of the material viscoelastic behaviour – the area of the loop formed by the loading and unloading characteristics, the measurement results of which are shown in Fig. 3.

To determine the twinning activation stress, the authors processed AE recorded during monotonic tensioning of the sample; the loading characteristic is shown in Fig. 4. Calculation of the AE signal spectral parameters was carried out by post-processing, using spectral clustering algorithms [16].

The results of studying the microstructure, using scanning electron microscopy after deformation show the presence of twins, which are formed simultaneously with nonlinear elastic behaviour and, therefore, may be the cause of it. Fig. 5 shows the structure of the samples after compression and tensile testing up to a residual deformation of 1.5 %.

DISCUSSION

A quantitative comparison of the obtained elastic modulus of 42...44 GPa is fully consistent, with data from literature sources for the MA14 (ZK60) alloy – 42...45 GPa [18; 19]. When the alloy is loaded to a stress of 90 MPa, at which a microplastic deformation of 0.05 % is formed, the elastic modulus retains its value, but the material unloading occurs along a curved downward characteristic, forming a hysteresis of the “loading – unloading” characteristic. During tensile twinning, lattice rotation is observed, and the rotation directions may correspond to the maximum anisotropy of the elastic constants of the material, E(0001)=50.8 GPa, E(−1−120) (−1100)=45.5 GPa [20], but the influence of the anisotropy factor only is quite small, or insignificant, since repeated loading occurs along a curved upward trajectory.

During loading of more than 100 MPa, a transition to the mode of active plastic deformation occurs: in the experiment, this is observed after the third loading cycle, which was stopped when the stress reached 145 MPa, and the residual deformation was 0.29 and 0.22 % for compression and tension, respectively. The difference in the magnitude of plastic compressive, and tensile deformation when applying the same magnitude of stress is described in [8–11], and is a manifestation of the asymmetry in the deformation behaviour of magnesium, and some other metals and alloys.

A feature of the unloading stage after applying a stress of 145 MPa is strong nonlinearity, in which the divergence

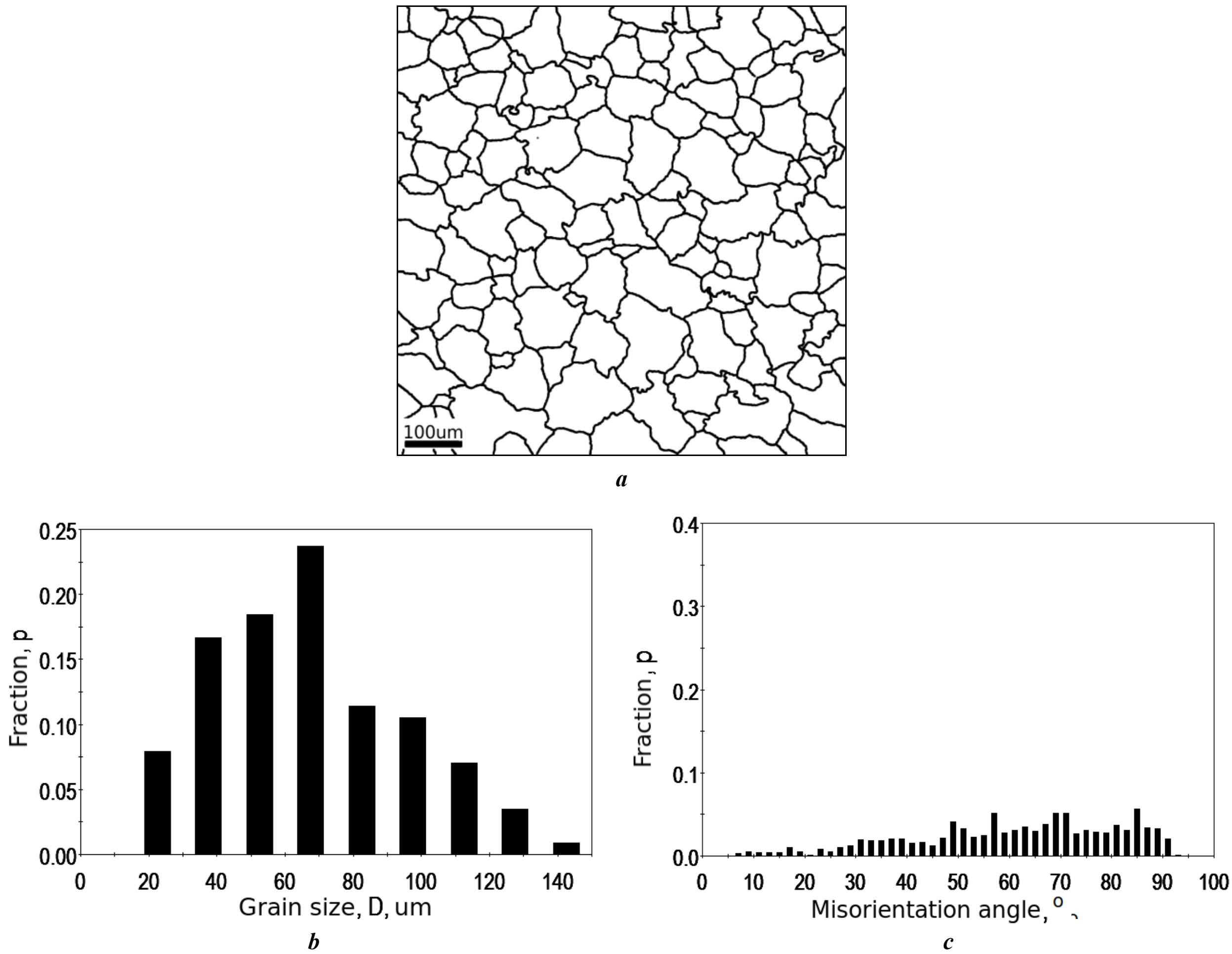

*Fig. 1. The initial structure of the MA14 alloy (**a**), histograms of the grain size distribution (**b**) and the crystal lattice misorientation at the grain boundaries (**c**)*

*Рис. 1. Исходная структура сплава МА14 (**a**), гистограммы распределений размера зерна (**b**) и углов разориентировки кристаллической решетки на границах зерен (**c**)*

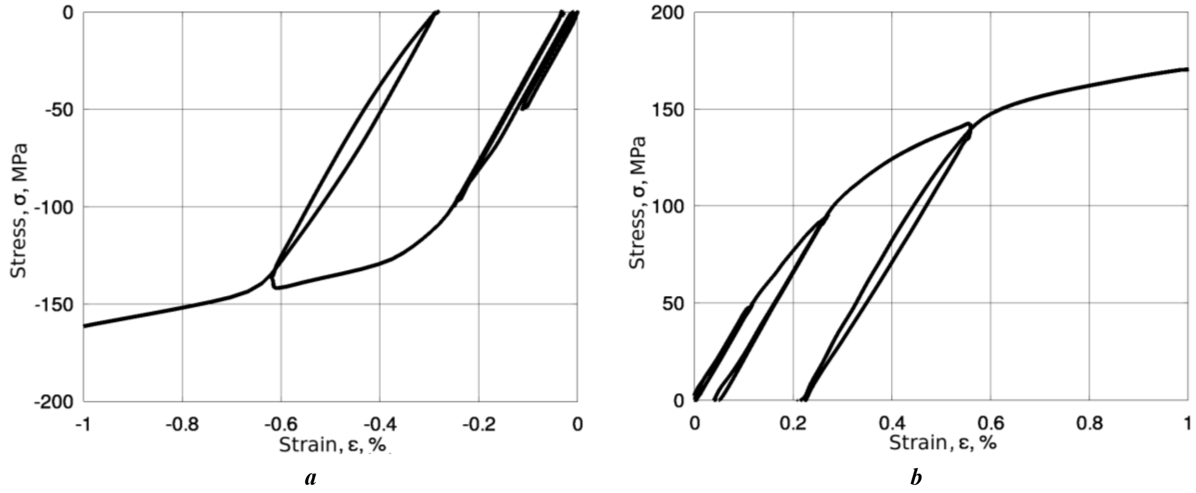

Fig. 2. *Loading – unloading diagrams of the MA14 alloy.*
*Under compression, strain and stress have a negative sign (**a**),*
*under tension – a positive sign (**b**)*
Рис. 2. *Диаграммы нагружения – разгрузки сплава МА14.*
*При сжатии деформация и напряжение имеют отрицательный знак (**a**),*
*при растяжении – положительный знак (**b**)*

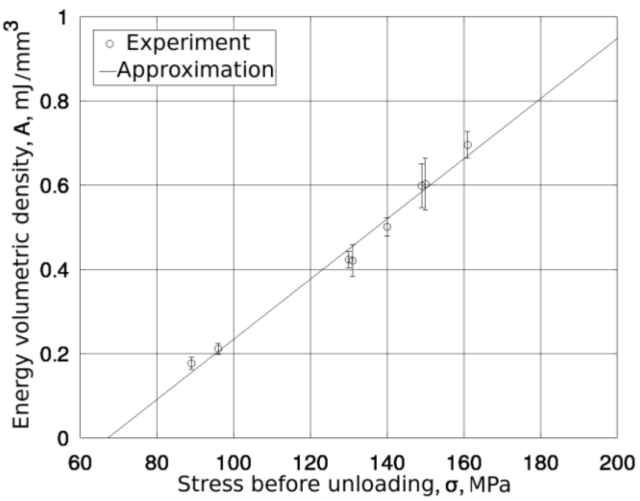

Fig. 3. *Energy volumetric density of the material viscoelastic behaviour*
Рис. 3. *Объемная плотность энергии вязкоупругого поведения материала*

of the loading and unloading trajectories increases forming a visually symmetrical mechanical hysteresis loop. Similar descriptions of the behaviour of the MA14 (ZK60) alloy are given in [1; 4; 6; 7].

Approximation of experimental values of the energy volumetric density, which forms the nonlinear elasticity behaviour, and interpolation towards the stress axis in Fig. 3 show that the nonlinear unloading effect appears, when the stress reaches a value in the range of 65...70 MPa.

It was shown in [13] and [16] that analysis of AE signal parameters and signal clustering according to the power spectral density distribution make it possible to accurately monitor twinning activity. The operation of deformation twinning systems is accompanied by high-amplitude AE pulses with a sharp leading edge and relaxation decay, and the spectral characteristic has a low median frequency. AE generated by dislocation slip is characterised by small amplitudes and a wide spectrum.

In this case, it is worth paying attention to the nature of changes in the power parameters, and median frequency of the AE signal, which are shown in Fig. 4. Acoustic emission exhibits intermittence when twinning is activated, while the measured signal parameters respond proportionally to the intensity of the process by increasing energy (amplitude), and median frequency in the mechanical stress range of 70...140 MPa, it is in this stress range that the material is most actively deformed by the twinning mechanism.

It is obvious that the stress “threshold” of 65...70 MPa corresponds to the physical yield strength of the material

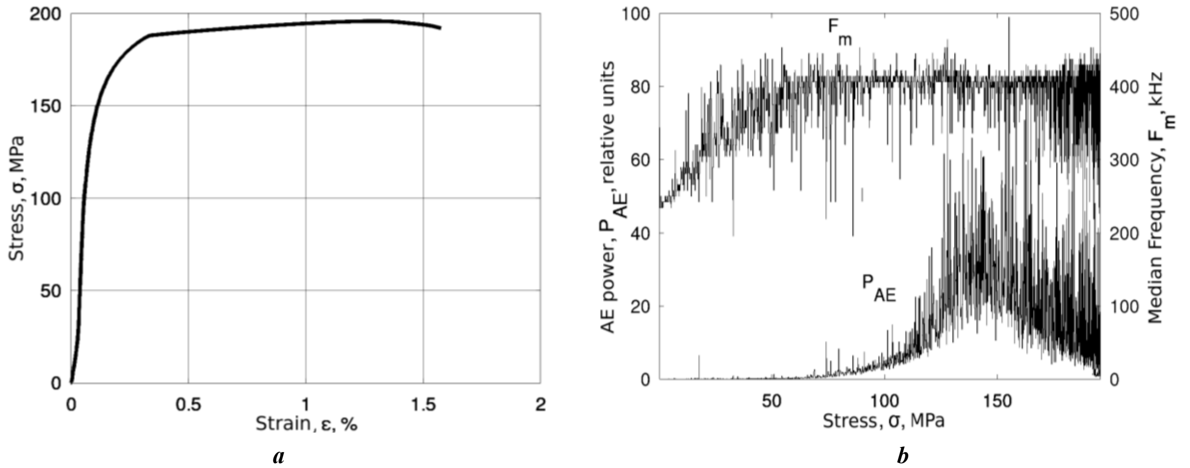

Fig. 4. *Loading diagram of the MA14 alloy sample (**a**) and acoustic emission parameters depending on the stress (**b**)*
Рис. 4. *Диаграмма нагружения образца сплава МА14 (**a**)*
*и параметры акустической эмиссии в зависимости от напряжения (**b**)*

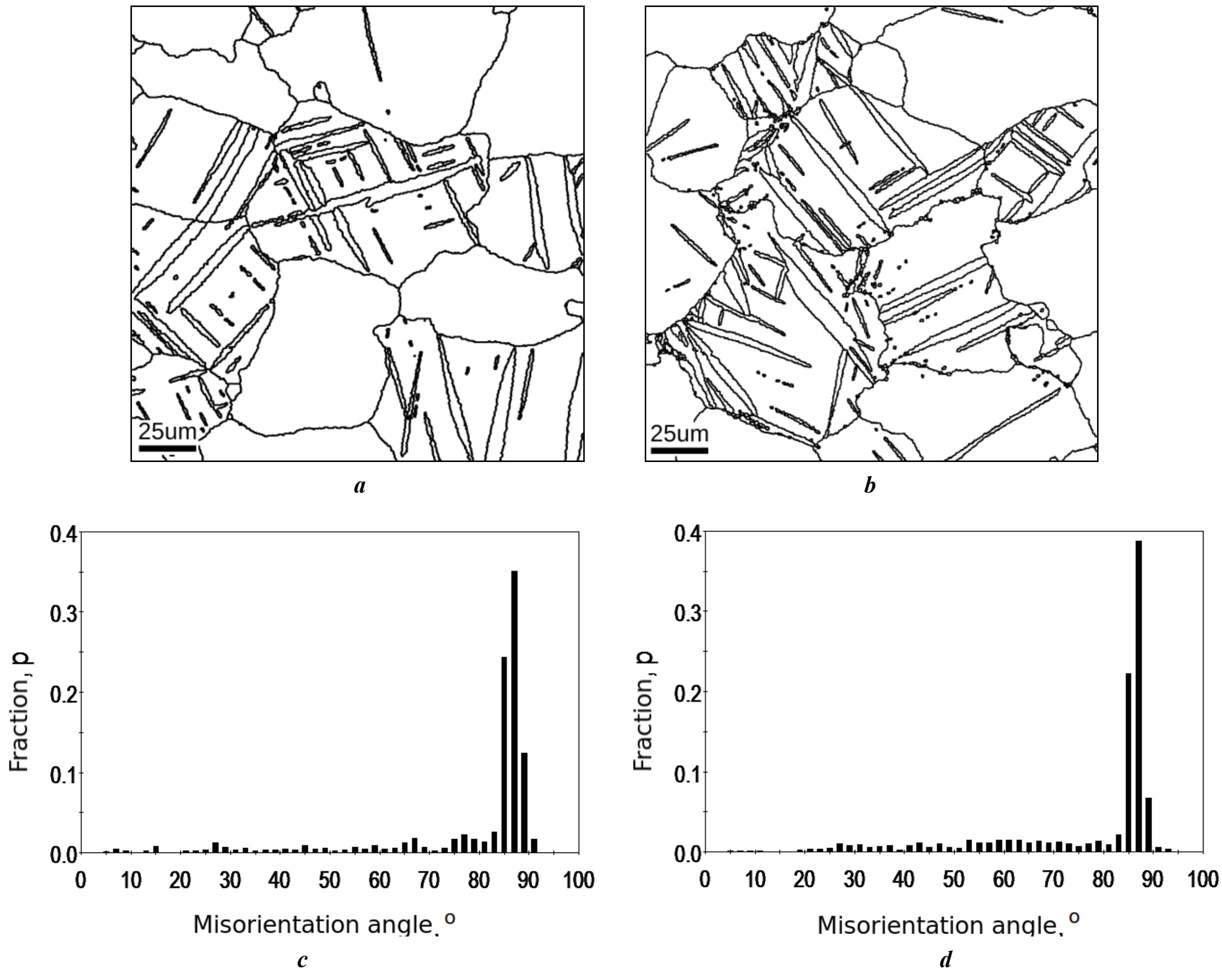

Fig. 5. *MA14 alloy structure after compression (**a**) and tensile (**b**) tests, histograms of the crystal lattice misorientation angles at the grain boundaries after compression (**c**) and tensile (**d**) tests*
Рис. 5. *Структура сплава МА14 после испытания на сжатие (**a**) и на растяжение (**b**),*
гистограммы распределений углов разориентировки кристаллической решетки на границах зерен
*после испытания на сжатие (**c**) и на растяжение (**d**)*

being tested: at this stress, strain hardening and the manifestation of the relaxation properties of the strain mechanisms, slip and, to a greater extent, twinning creating powerful AE begin.

The structural state of the samples tested in compression and tension was similar. The deformed structures contain signs of activity of dislocation slip systems forming a subgrain structure, with small grain-boundary angles (3...5°) and tensile twins with crystal lattice grain-boundary angles of about 86°. The type of active strain mechanisms in adjacent grains may be different, since the activation of a specific deformation system occurs only after a certain critical shear stress is exceeded: the basal plane slip systems have the lowest values of the critical shear stress; the prismatic slip and tensile twinning systems are the next as the critical shear stress increases [13; 21], and it is these systems that can be identified at the small deformations observed in this study. The critical shear stress is primarily achieved in crystals (grains) that are favourably oriented with respect to external stress, and is numerically determined by the Schmidt factor [22; 23]. The heterogeneity of activation of deformation systems forms a stress state that is uneven throughout the volume of the material, and elastic stress gradients can inhibit the twin propagation inside the grain, and then, when the external stress decreases under the influence of elastic forces, the reverse detwinning process can occur [24; 25].

CONCLUSIONS

The most likely mechanism forming the nonlinear unloading characteristic, and nonlinear elastic behaviour of the MA14 alloy is the twinning–detwinning mechanism in the "twinning – tension" systems. The twin formation in a grain is associated with an insufficient number of active easy slip systems in the basal and prismatic planes, which are orthogonal to each other. At the same time, "twinning – tension", with a crystal lattice rotation of 86° does not create more favorable conditions for the activation of easy slide systems. As a consequence, in magnesium, the conditions for the formation of a twin are observed in an unstrengthened lattice (with short lengths of sections of pinned dislocations), while after the twin formation, its pinning (strengthening) by active slip is not observed. Thus, apparently, unrelaxed elastic stresses are formed both at the periphery of the twin inside the grain and along the grain perimeter, and it is these stresses that lead to detwinning when removing the external stress. The mechanism of operation of an unpinned (unstrengthened "elastic" twin) is similar to the behaviour of a curved elastic beam between two supports, which has the possibility of elastic deflection and two deformation "paths" depending on the direction: increasing or decreasing stress, while the sign of loading has no effect.

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Определение порога напряжения и микроструктурных факторов, формирующих эффект нелинейной разгрузки магниевого сплава МА14 (ZK60)

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Аннотация: Магниевые сплавы – идеальный материал для создания легких и прочных современных транспортных систем, однако его широкое применение ограничено из-за некоторых физико-химических свойств. В работе рассмотрен эффект нелинейной упругой разгрузки магниевого сплава МА14 (ZK60, Mg–5,4Zn–0,5Zr) в крупнозернистом состоянии после рекристаллизационного отжига. Установлено, что нелинейность характеристики разгрузки формируется после достижения определенного порогового уровня напряжения. Предполагается, что изучаемый эффект связан с деформационным поведением сплава, при котором наблюдается формирование двойниковой структуры по механизму двойникования растяжения. Микроструктура материала образцов была определена методами растровой электронной микроскопии с применением анализа дифракции обратно рассеянных электронов. Определение порогового напряжения формирования нелинейности разгрузки было проведено двумя методами: 1) по величине площади петли, образуемой нелинейностью механической характеристики разгрузки и характеристики повторного нагружения (механический гистерезис), и 2) по анализу акустической эмиссии, зарегистрированной при растяжении до разрушения. Сопоставление полученных результатов позволяет предположить, что нелинейность разгрузки обусловлена двойникованием в зернах, в которых наблюдается невыгодная конфигурация (низкий фактор Шмидта) для дислокационного скольжения. Разворот продвойниковавшего кристалла на угол, близкий к 90°, не способствует повышению фактора Шмидта и активации систем скольжения дислокаций для закрепления деформированной структуры по механизму дислокационного упрочнения. При последующем снижении величины внешнего напряжения происходит раздвойникование и частичное восстановление конфигурации кристаллической решетки.

Ключевые слова: магний; магниевый сплав; МА14 (ZK60, Mg–5,4Zn–0,5Zr); нелинейная разгрузка; порог напряжения; упругость; двойникование; раздвойникование; деформационное поведение.

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