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Effects of extrusion on Young's modulus and internal friction of magnesium alloys with various long period ordered structure content

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Abstract: The relevance of this work stems from the growing interest in magnesium alloys with long period ordered structure (LPSO) due to their unique mechanical properties. Investigating the effect of extrusion on Young's modulus and internal friction of such alloys provides a deeper understanding of their mechanical behaviour, which is important for the development of new materials with improved performance properties. This research explores the effect of warm extrusion on the structure, dynamic Young's modulus and internal friction of magnesium alloys containing varying amounts of LPSO phases. Alloys in the Mg–Zn–Y system with estimated LPSO phase contents of 0, 50 and 100 % vol. were analysed using the composite piezoelectric oscillator technique at 100 kHz. The results demonstrate that the Young's modulus increases with higher LPSO content, driven by the enhanced stiffness and strong interatomic bonding of the LPSO phases. Extrusion leads to a 3 % decrease in Young's modulus along the direction parallel to its axis for all samples. This effect is explained by the formation of an elongated texture and an increase in the dislocation density. Internal friction measurements revealed a rise in amplitude-independent internal friction post-extrusion, suggesting higher dislocation density, while the critical strain amplitude decreased in alloys with higher LPSO content. Additionally, Young's modulus softening was reduced after extrusion, primarily due to dislocation-induced hardening. These findings shed light on the mechanical properties of Mg–Zn–Y alloys with LPSO structures, emphasising the effects of extrusion and phase content on their dynamic behaviour.

Keywords: magnesium alloys; long-period stacking-ordered structure; LPSO; internal friction; Young's modulus; microplasticity.

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INTRODUCTION

Magnesium alloys with a long-period stacking ordered (LPSO) structure represent a unique class of materials that have attracted the attention of researchers and engineers due to their outstanding mechanical properties. Yosihito Kawamura first proposed the characteristics of the constituents and methods for preparation of LPSO magnesium ternary alloys [1]. Team of authors from the Magnesium Research Center is the most active in the development of these nanostructured materials and the publication of new

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papers to date. These alloys combine the lightness of magnesium with increased strength, creep resistance, and improved damping characteristics [1–3]. The most studied materials in this category are alloys based on Mg–Zn–Y, the LPSO structure in them is characterised by a periodic alternation of layers with different atomic stacking sequence. Mg–Zn–Y ternary compounds crystallise in different phases depending on the Zn–Y ratio and solidification methods [4]. Common types of LPSO phases in these alloys are 18R (rhombohedral structure with a period of 18 layers) and 14H (hexagonal structure with a period of 14 layers). These phases in the Mg–Zn–Y system are formed by adding a few atomic percent of zinc and yttrium (Zn/Y ratio of ~1) to magnesium alloy by rapid solidification [1]. LPSO phases significantly increase the strength of Mg–Zn–Y alloys due to an ordered structure that impedes the movement of dislocations, creates barriers at interfaces, promotes dispersion hardening and grain size reduction, and suppresses strain twinning. Of particular interest is the fracture mechanism of these alloys, which differs significantly from that of classical alloys due to the formation of so-called "kink bands" [5].

Magnesium alloys with LPSO structure are hardened after extrusion [6]. For example, in a study [7], a cast alloy with 88 % LPSO content ($Mg_{88}Zn_4Y_7$) had a low tensile strength (yield strength) of about 140 MPa at room temperature. After extrusion, the strength increased significantly. At an extrusion ratio of 10:1, the strength reached 460 MPa at room temperature, which is more than three times higher than that of the cast alloy. The strengthening occurs because the extrusion aligns the LPSO phase along the extrusion direction, preventing basal slip and increasing strength. In addition, the kink bands formed during extrusion create additional boundaries that effectively prevent dislocation movement, further contributing to the strength improvement. Although it is known that the LPSO phase increases the strength of the alloy and extrusion further enhances this strengthening, the exact mechanisms (e. g., dislocation interaction with the LPSO phase) require further clarification.

Typically, the mechanical properties of magnesium alloys with LPSO structures are studied using standard methods (tensile, fatigue and hardness tests) [8] as well as more specialised methods (acoustic emission, in situ observations) [9]. These methods provide comprehensive information on the strength, ductility, fatigue characteristics and microstructure of alloys. A relatively new avenue of research of alloys containing LPSO has been the investigation of the high-frequency damping and the effective elastic modulus [10].

The aim of our work is to investigate the effect of extrusion on the mechanical properties of magnesium alloys with LPSO structures using a composite piezoelectric oscillator method at a frequency of 100 kHz.

METHODS

Composite piezoelectric oscillator method

The main method of investigation in this work is the composite piezoelectric oscillator (CPO) technique [11]. The CPO method is based on measuring the resonant frequencies of a material sample excited by a piezoelectric transducer. The composite oscillator consists of two parts: a piezoelectric element (quartz) and the sample under study, which are mechanically connected using a cyanoacrylate adhesive. When an alternating voltage is applied to the piezoelectric element, mechanical vibrations are generated and transmitted to the sample. Analysis of the resonant frequencies and damping of oscillations (internal friction (IF), decrement) of the CPO allows for the determination of the Young's modulus (modulus of elasticity at low-amplitude vibrational deformation) and IF of the sample based on the following relationships:

$$m_{osc}\delta_{osc} = m_q\delta_q + m_s\delta_s\,;$$
$$m_{osc}f_{osc} = m_q f_q + m_s f_s\,, \quad (1)$$

where m_{osc} is mass of the entire oscillator;
m_q is mass of the quartz;
m_s is mass of the sample;
δ_{osc} is damping of oscillations of the entire oscillator;
δ_q is damping of oscillations of the quartz;
δ_s is damping of oscillations of the sample;
f_{osc} is oscillation frequency of the entire oscillator;
f_q is oscillation frequency of the quartz;
f_s is oscillation frequency of the sample.

The equation for determining the Young's modulus:

$$E = 4\rho l^2 f_s^{\,2} \quad , (2)$$

where ρ is density of the material under study;
l is length of the sample.

By measuring the change in resonant frequency, the Young's modulus and IF of the material can be determined at various temperatures and loads (strain amplitudes). The strain amplitude in the experiments varied from 2×10^{-7} to 2×10^{-4}, and the temperature ranged from 80 to 320 K with a heating/cooling rate of 2 K/min. It should be noted that IF characterises the material's ability to dissipate mechanical vibration energy. It manifests as a damping of the oscillation amplitude and is expressed through the damping decrement or logarithmic decrement. The logarithmic decrement δ (defined as $\delta=\Delta W/2W$, where ΔW is the energy dissipated per cycle; W is the maximum stored oscillation energy) served as a measure of internal friction. Young's modulus characterises the ratio of stress to strain in the approximation of uniaxial tension or compression. Studying both of these characteristics simultaneously at various strain amplitudes over a wide temperature range provides integrated information on the mechanical processes occurring in the samples.

The Young's modulus softening was measured based on the dynamic modulus at the amplitude-independent stage E_i, and modulus on strain dependency at the high amplitude stage $E(\varepsilon)$, as given by equation:

$$\frac{\Delta E}{E} = \frac{E_i - E(\varepsilon)}{E_i}\,. \quad (3)$$

The study of internal friction and dislocation modulus softening in metals by CPO method is considered in detail in the works by Lebedev et al. [12–14].

Research materials and supporting methods

The objects of study in this work were a set of polycrystalline samples of Mg–Zn–Y-based alloys, both before and after extrusion, with varying LPSO content: 0, 50, and 100 % (volume fraction). These alloys were obtained from Kumamoto University, Japan. Their nominal chemical compositions (in at. %), confirmed by inductively coupled plasma spectroscopy, are as follows: Mg99.2Zn0.2Y0.6 (LPSO-0 %), Mg93Zn2.5Y4.5 (LPSO-50 %), and Mg85Zn6Y9 (LPSO-100 %). Using a diamond saw, the samples were shaped into parallelepipeds with dimensions of $2\times3\times24.5$ mm^3. For all extruded samples, the longest direction coincided with the extrusion direction. This shape was chosen based on formula (2) to ensure resonance in the quartz-sample system.

As a complementary method, scanning electron microscopy (SEM) (MIRA III Tescan), optical microscopy (MET-5t Altami), and X-ray powder diffraction (MD-10 Radikon) were used to investigate the microstructure. SEM images and X-ray diffraction pattern were obtained from an orthogonal (smallest) cross-section of the parallelepiped sample. The sample surfaces were polished in isopropyl alcohol using sandpaper up to 1 μm grit. X-ray diffraction (XRD) data were obtained by rotating the samples around ω at a speed of 10 min^{-1}, using monochromatized by LiF(200) CrKa_α radiation.

RESULTS

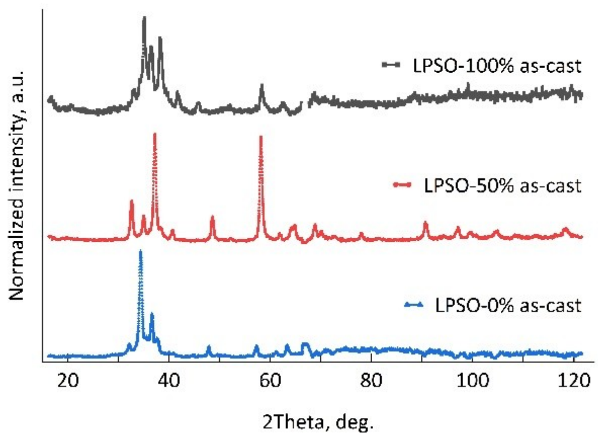
The obtained X-ray diffractograms (Fig. 1 a, 1 b) are satisfactorily described within the framework of three structures with different parameters: distorted hexagonal lattice of alpha magnesium (P63/mmc a=3.19 c=5.18), cubic W-phase and hexagonal phase. All samples are characterised by a complex diffraction pattern of the indicated structures in the region of 30–40 deg. The sample of LPSO-0 % as-cast alloy shows increased intensity in the direction 0 0 0 2 (2θ=34.59°) and the sample of LPSO-50 % as-cast alloy shows increased intensity in the direction 1 1 −2 0 (2θ=58.36°).

The main feature of extruded samples vs cast ones is the pronounced texture in the basal planes of the magnesium hexagonal lattice, which corresponds to the 1 0 −1 0 peak in Fig. 1 b. It can also be observed that the LPSO-0 % and LPSO-50 % samples, reoriented with an extinction of 0 0 0 2 and 1 1 −2 0 peaks correspondingly (Fig. 1 b).

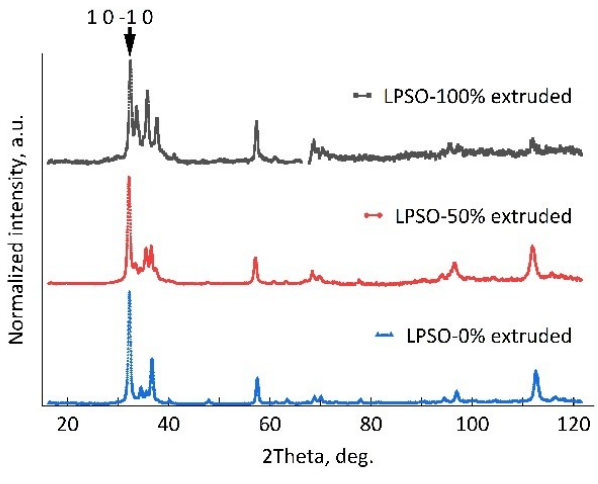
SEM micro-images of as-cast and extruded samples show the presence of blocks of different contrast in the backscattered electron mode for LPSO-50 % and LPSO-100 % (Fig. 2). The LPSO-50 % sample includes at least three different types of grains in terms of contrast.

As a result of the work, the temperature dependencies of the Young's modulus of Mg-Zn-Y alloys with varying LPSO content, both before and after extrusion, were determined (Fig. 3). It can be observed that the Young's modulus of the samples after extrusion decreases by an average of 3 % for 0 %, 50 %, and 100 % LPSO content. Despite the fact that the measurement error for Young's modulus is 0.001 % (this is true for the already fixed sample), it can reach 1 % when the sample is reglued. We tried to increase the accuracy by repeatedly repeating the experiment on different samples. The values of Young's modulus at room temperature were as follows: for 0 % LPSO before extrusion is 44.2 GPa, after is 42.6 GPa; for 50 % LPSO before extrusion is 49.8 GPa, after is 48.5 GPa; for 100 % LPSO before extrusion is 57.2 GPa, after is 55.6 GPa. Young's modulus increases with decreasing temperature.

Fig. 4 shows the amplitude dependences of internal friction for samples before and after extrusion. It is possible to see the increase of internal friction in the samples



a



b

Fig. 1. *X-ray diffraction patterns on as-cast (**a**) and extruded (**b**) LPSO samples*
Рис. 1. *Рентгеновские дифракционные картины на литых (**a**) и экструдированных (**b**) ДПС образцах*

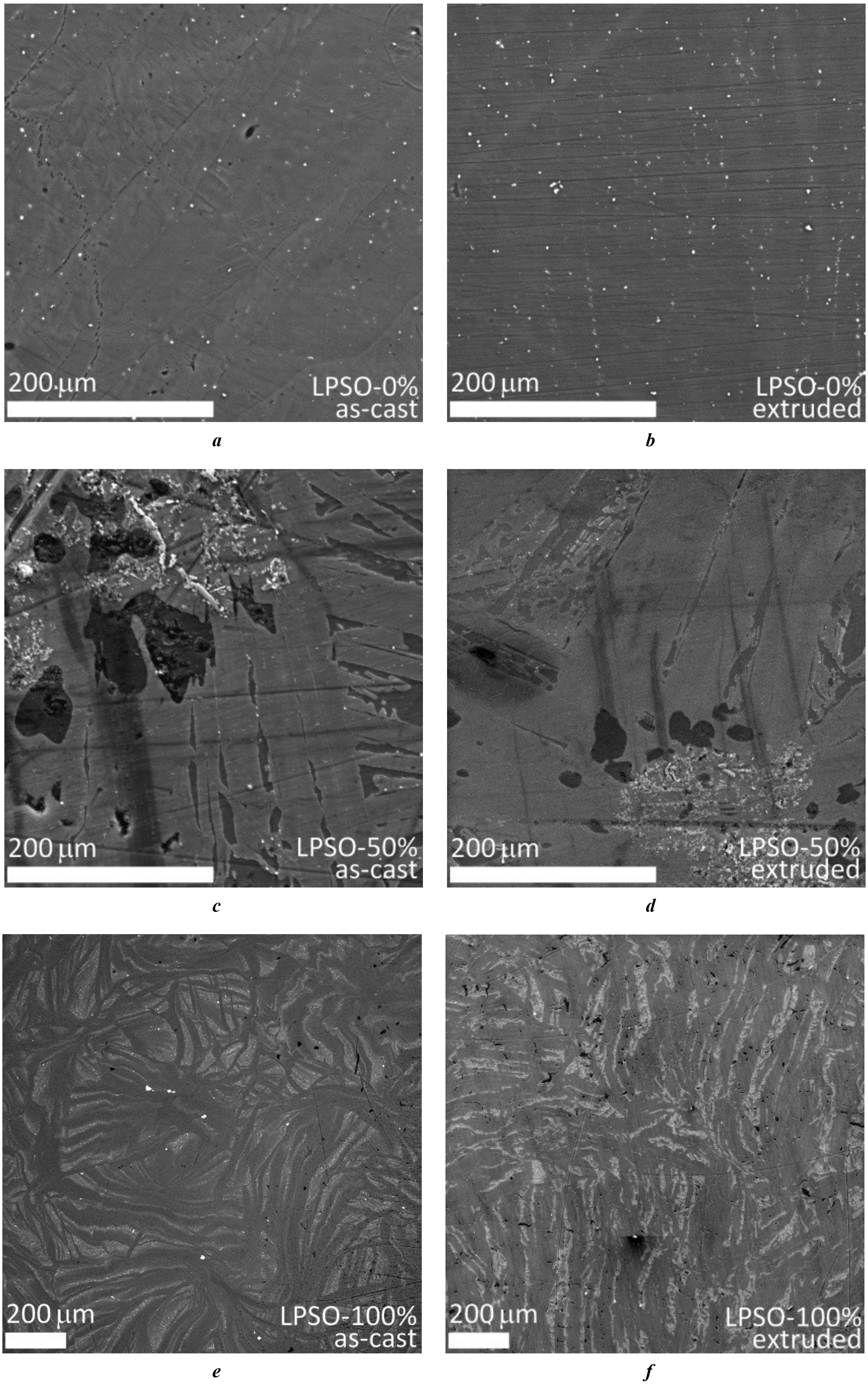


Fig. 2. *SEM microimages for as-cast (**a**, **c**, **e**) and extruded (**b**, **d**, **f**) LPSO samples obtained in backscattered electron contrast mode*

Рис. 2. *РЭМ микроизображения для литых (**a**, **c**, **e**) и экструдированных (**b**, **d**, **f**) ДПС образцов, полученные в режиме сопоставления отраженных электронов*

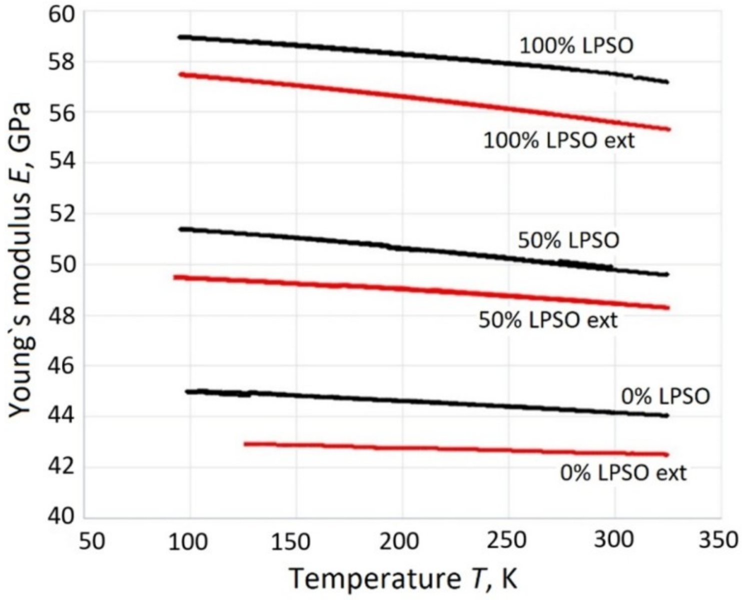


Fig. 3. *Temperature dependence of Young's modulus of magnesium alloys with different content of LPSO structures before and after extrusion*
Рис. 3. *Температурная зависимость модуля Юнга магниевых сплавов с различным содержанием ДПС структур до и после экструзии*

after extrusion, the increase of internal friction is also observed with the increase of the content of LPSO phases in the alloy. The amplitude dependent (ADIF) and amplitude independent (AIIF) part of internal friction is clearly visible in these relationships, for understanding the inset in Fig. 4 is presented. AIIF increases with increase in LPSO content but more increase occurs after extrusion. ADIF behaves differently without a pronounced pattern. In samples with LPSO content of 50 % and 100 %, a significant decrease in the critical strain amplitude can be observed, ε_c.

In Fig. 5 shows the amplitude dependences of Young's modulus softening, before and after extrusion. After extrusion, the modulus softening decreased significantly. Before extrusion, one can observe an increase in Young's modulus softening with increasing LPSO content of phases.

DISCUSSION

The X-ray diffraction peaks of the samples (Fig. 1) in the investigated range correspond to known crystal structures [4]. The texture of the samples corresponding to the phase-contrast images (Fig. 2 a, c, e) is preserved in the plane normal to the extrusion direction (Fig. 2 b, d, f). However, for all phases, the orientation of the crystal structure under shear deformation is observed (Fig. 1 b, 1 0 −1 0 peak).

The results show that the Young's modulus of magnesium alloys with LPSO structures increases with higher LPSO content (Fig. 3). Young's modulus values at a fixed strain amplitude grow with increasing volume fraction of the LPSO phase. This occurs because LPSO phases have strong interatomic bonds and increase the stiffness of the alloy. The reasons for the decrease in Young's modulus after extrusion are varied. For example, during extrusion, a texture is formed in the material where most crystallites are oriented in a specific manner (e. g., basal planes (0001) align parallel to the extrusion direction), as confirmed by X-ray results (Fig. 1). In the direction parallel to the extrusion axis, the Young's modulus may decrease due to the predominant orientation of basal planes, which have a lower *E* modulus along the c-axis [15]. In the perpendicular direction, the Young's modulus may, conversely, increase. In our case, texture may play a key role since measurements are conducted parallel to the extrusion axis.

In alloys with LPSO structures, extrusion can also lead to partial destruction or changes in the shape of LPSO phases [16]. These phases play a crucial role in strengthening the material, and their degradation can reduce the overall Young's modulus. In our experiments, the SEM results (Fig. 2) show changes in the shape of the blocks containing LPSO structures. Extrusion also induces significant plastic deformation, resulting in the accumulation of dislocations and the formation of kink band [17]. These defects reduce the elastic properties of the material as they create regions with reduced stiffness. The increase of AIIF (Fig. 4) just indirectly indicates the increase of dislocation density. Extrusion typically leads to grain refinement through dynamic recrystallisation [18]. Although grain size reduction increases strength and plasticity, it can reduce the Young's modulus due to the increased volume of grain boundaries, which have lower elasticity than the bulk crystallites [19].

The amplitude dependences of internal friction were taken at room temperature, Fig. 4. Based on classical works [20], the amplitude dependence of internal friction is related

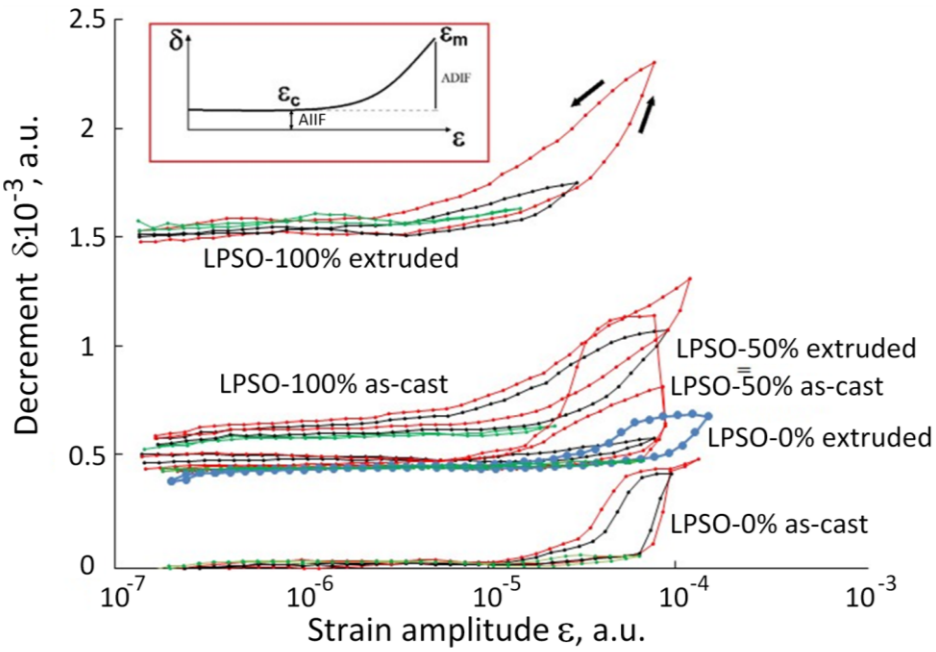


Fig. 4. *Amplitude dependence of internal friction in the Mg–Y–Zn alloy with different content of LPSO structures (LPSO – 0 %, LPSO – 50 %, LPSO – 100 %) before and after extrusion. Consequent loading cycles with increasing ε_m are marked in red, green and black, respectively. The curve for 0 % LPSO after extrusion is highlighted in blue. Inset – schematic illustration of the amplitude – independent internal friction (AIIF) and amplitude – dependent friction (ADIF). ε_c is the critical strain amplitude delineating these two regimes. The arrows indicate the direction of the forward and backward run in the global loading cycle*

Рис. 4. *Амплитудная зависимость внутреннего трения в сплаве Mg–Y–Zn с различным содержанием ДПС структур (ДПС – 0 %, ДПС – 50 %, ДПС – 100 %) до и после экструзии. Последовательные циклы нагружения с ростом ε_m обозначены красным, зеленым и черным цветом соответственно. Кривая для содержания ДПС 0 % после экструзии выделена синим цветом. Вставка – схематическое изображение амплитудно-независимого внутреннего трения (AIIF) и амплитудно-зависимого трения (ADIF). ε_c – критическая амплитуда деформации, разграничивающая эти два режима. Стрелки указывают направление прямого и обратного хода в глобальном цикле нагружения*

to the movement of dislocations in the material, where AIIF reflects the density of dislocations, ADIF reflects the movement of dislocation segments, and ε_c is related to the detachment of dislocations (their segments) from the anchoring points. It should be noted that AIIF increases with increasing LPSO content, but to a greater extent the increase occurs after extrusion, which is associated with an increase in dislocation density. ADIF behaves differently and a precise interpretation of the observed phenomenon requires a more detailed study. Also in the present work, we will only emphasize the observed phenomenon of the decrease of the critical strain amplitude in samples with high content of LPSO phases further this phenomenon should be discussed more fully.

In Fig. 5 it can be seen that the Young's modulus softening over a wide range of amplitudes is smaller for the samples after extrusion. This can be explained by several reasons, but the main one is hardening due to dislocations. During extrusion, the material undergoes significant plastic deformation, which leads to an increase in dislocation density, as evidenced by the increase in AIIF in the post-extrusion samples. The high dislocation density may make it difficult for dislocations to move during subsequent loading, which reduces the contribution of dislocation mechanisms to Young's modulus softening. The effect of LPSO phase changes on the decrease in Young's modulus softening after extrusion is not significant, which can be confirmed by the changes in Young's modulus softening in samples with 0 % LPSO phase content. In summary, the main reason for the decrease in Young's modulus softening after extrusion is the increase in dislocation density.

CONCLUSIONS

The study demonstrates that the Young's modulus of Mg–Zn–Y alloys increases with higher LPSO content due to the strong interatomic bonds of LPSO phases. However, extrusion reduces the Young's modulus by approximately 3 %, attributed to texture formation, partial degradation of LPSO phases, and increased dislocation density. Internal friction measurements reveal a rise in amplitude-independent internal friction after extrusion, indicating higher dislocation density, while the critical strain amplitude decreases in alloys with higher LPSO content. Extrusion also reduces Young's modulus softening, primarily

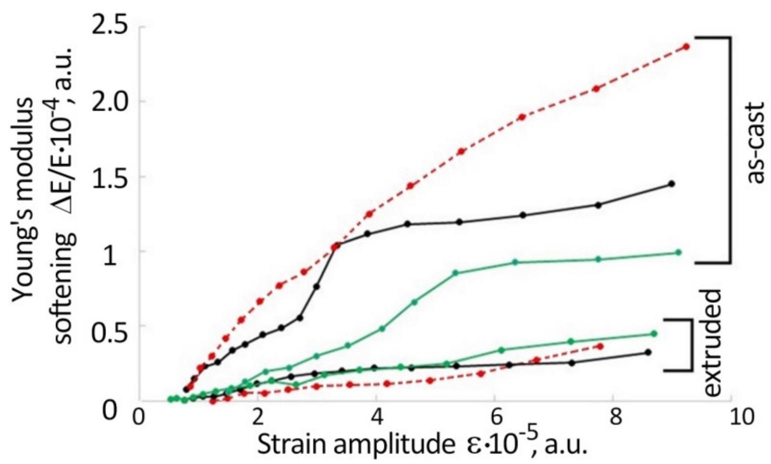


Fig. 5. *Amplitude dependence of the softening of Young’s modulus for alloys with different LPSO content before and after extrusion. Green line samples with 0 % LPSO, black line with 50 % LPSO, red-dashed line with 100 % LPSO*

Рис. 5. *Амплитудная зависимость дефекта модуля Юнга для сплавов с различным содержанием ДПС до и после экструзии. Зеленая линия – образцы с 0 % ДПС, черная линия – с 50 % ДПС, красная пунктирная линия – со 100 % ДПС*

due to dislocation-induced hardening. Microstructural changes, such as grain refinement and kink band formation, further influence the mechanical properties. These findings underscore the importance of LPSO content and extrusion processes in optimising the mechanical behaviour of magnesium alloys.

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Влияние экструзии на модуль Юнга и внутреннее трение в магниевых сплавах с различным содержанием длиннопериодной слоистой структуры

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Аннотация: Проведение исследования обусловлено растущим прикладным интересом к получению и исследованию механических свойств новых магниевых сплавов, содержащих длиннопериодную слоистую структуру (ДПС). Исследование влияния обработки теплой экструзией на модуль Юнга и внутреннее трение позволит в большей мере понять поведение данных материалов под действием различных механических напряжений, что важно для улучшения их функциональных характеристик. Представлены результаты влияния теплой экструзии на структуру, эффективный модуль Юнга и внутреннее трение в сплавах с различным содержанием фазы ДПС. Сплавы в системе Mg–Zn–Y с содержанием ДПС 0, 50 и 100 % об. были изучены с использованием пьезоэлектрического составного вибратора на частотах, близких к 100 кГц. Полученные результаты показали увеличение модуля Юнга с ростом содержания ДПС, обусловленное большей жесткостью и сильной межатомной связью в этой структуре. Экструзия вызвала уменьшение модуля Юнга на 3 % вдоль направления обработки. Этот эффект объясняется формированием удлиненной микротекстуры, преимущественной ориентацией в фазах альфа-магния и ДПС, а также возрастанием плотности подвижных дислокаций. Нелинейная часть внутреннего трения возрастала в результате экструзии благодаря увеличению плотности вовлеченных дислокаций. В то же время критическая амплитуда деформации уменьшалась с увеличением доли ДПС. Кроме того, выявлено снижение дефекта модуля Юнга после экструзии, что объясняется преимущественно дислокационным упрочнением. Полученные данные позволяют с большим пониманием взглянуть на деформационное поведение сплавов Mg–Zn–Y с ДПС, а также на влияние на него обработки теплой экструзией.

Ключевые слова: сплавы магния; длиннопериодная слоистая структура; ДПС; внутреннее трение; модуль Юнга; микропластичность.

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