

FACULTY OF MATHEMATICS AND PHYSICS Charles University







International Seminar on Advanced Structural Materials

October 20 – 22, 2019 Prague, Czech Republic

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Welcome Message

Dear participants,

We are delighted to welcome you to the International Seminar on Advanced Structural Materials at Charles University in Prague, Czech Republic, October 20-22, 2019.

In 2018 a Memorandum of Understaning (MoU) was signed between Faculty of Mathematics and Physics, Charles University, and Graduate School of Science and Technology and Faculty of Advanced Science and Technology, Kumamoto University. The purpose of the Memorandum is to promote collaboration in the area of joint scientific research and education.

Thus it is timely that this meeting will enable you to discuss the new results in the research and development of next generation of structural materials, including alloy design, cast metal formation, mechanical properties, deformation behavior, corrosion, surface treatment, and recycling. Further, the seminar makes a perfect platform for networking as it brings together renowned speakers, experts and early-career scientists in materials science both from Japan and Czech Republic.

The organisers express their gratitude to all paricipants and wish to have a good time in Prague.

Sincerely Yours,

Prof. Yoshihito Kawamura and Dr. Kristián Máthis

Chair Organizers of the Seminar

Program

October 20 (Sunday)

- 10:00 12:00 Guided city tour, meeting on Hradčanské náměstí at large green lantern
- 14:00 17:00 Round-table discussion: Collaboration between Charles University and Kumamoto University

October 21 (Monday)

Faculty of Mathematics and Physics, Charles University, Ke Karlovu 3, 2nd floor (rear staircase), room M252

09:00 – 09:20 Prof. **Jan Trlifaj**, vice-dean of the Faculty of Mathematics and Physics, Charles University

Prof. **Yoshihto Kawamura**, director of Magnesium Research Center, Kumamoto University (MRC, KU)

Prof. **Robert Král**, head of Department of Physics of Materials, Faculty of Mathematics and Physics, Charles University (DPM, CU)

Welcome speech

- 09:20 09:45 Prof. **Yoshihito Kawamura** MRC, KU Introduction of LPSO-type and C36-type Mg alloys developed at KU
- 09:45 10:10 Prof. František Chmelík DPM, CU Exploring Thermal Loading of Hybrid and Ceramic Composites by the Acoustic Emission Technique

Coffee Break

10:30 - 10:55	Prof. Shinji Ando Activity of non-basal slips in magnesium all	MRC,KU loys
10:55 – 11:20	Prof. Patrik Dobroň Acoustic Emission Study of Active Deform Mg Alloys	DPM, CU nation Mechanisms in
11:20 - 11:45	Prof. Kristián Máthis Synergy of in-situ methods in study of defo of Mg alloys	DPM, CU ormation mechanisms
Lunch Break	Restaurant Legenda, Legerova 1820/39, Pra	ha 2
14:00 - 14:25	Prof. Michiaki Yamasaki Ongoing research for the LPSO-typed Mg a	MRC, KU lloys in Japan
14:25 - 14:50	Dr. Klaudia Horváth-Fekete Investigation of the effect of the LPSO-conte mechanisms in Mg-Y-Zn magnesium alloys	DPM, CU ent on the deformation by in-situ methods
14:50 - 15:15	Prof. Tsuyoshi Mayama Numerical study on development of intra-gra during compression of LPSOCrystals	MRC, KU anular misorientations
15:15 - 15:40	Dr. Takao Tsumuraya First-principles study on the origin of phase with long-period stacking ordered structure	MRC, KU stability of Mg alloys
Coffee Break		
16:20 - 18:00	Panel discussion, present and future of development	f magnesium alloys
18:00	<i>Dinner / Welcome party</i> Klášterní pivovar Strahov (Sv. Norbert), Strahovské nádvoří 301/10, 118 00 Praha 1	

October 22 (Tuesday)

Faculty of Mathematics and Physics, Charles University, Ke Karlovu 3, 2nd floor (rear staircase), room M252

10:00 - 10:25	Prof. Zuzanka Trojanová Anisotropy of Mechanical and Physical Pro Alloys and Composites Prepared by SPD M	DMP, CU operties of Magnesium lethods
10:25 - 10:50	Dr. Hiromoto Kitahara Structure and mechanical properties of Zi process	MRC, KU inc deformed by SPD
10:50 - 11:15	Dr. Tomáš Krajňák Microstructure, texture and mechanical p ECAP processed AX41 alloy	DPM, CU roperties evolution in
11:15 - 11:40	Dr. Michal Knapek Mechanical properties of novel Mg allog Plasma Sintering	DPM, CU ys prepared by Spark
Lunch Break	Restaurant Legenda, Legerova 1820/39, Pr	aha 2
13:00 - 13:25	Prof. Donald Shih Potential Usage of Advanced Mg All Methodology to accelerate the R&D result	MRC, KU oys in Aerospace - transition
13:25 - 13:50	Dr. Daria Drozdenko Optimization of mechanical properties of ra Y alloys	MRC, KU apid solidified Mg-Zn-
13:50 - 14:15	Dr. Peter Minárik From corrosion resistance measurement t magnesium-based materials	DPM, CU owards biodegradable

14:15 - 14:40	Dr. Shinichi Inoue	MRC, KU
	Improvement of flammability in L	PSO-type Mg alloys

Coffee Break

- 15:20 18:00 *Laboratory tour* Department of Physics of Materials, Faculty of Mathematics and Physics, Charles University, Ke Karlovu 5, Praha 2
 19:00 *Dinner / Sayonara party*
 - Restaurant Legenda, Legerova 1820/39, Praha 2

Abstracts

Introduction of LPSO-type and C36-type Mg Alloys Developed at KU

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Keywords: magnesium, LPSO, C36, microstructure, kinking, flammability

Mg alloys are attractive for use in aircraft components primarily because of their low density and high specific strength. We have developed two kinds of high-strength Mg alloys with a high ignition temperature: 1) Mg-Zn-Y alloys consisted of alpha Mg phase and a long period stacking ordered (LPSO) phase [1,2,3], and Mg-Al-Ca alloys consisted of alpha Mg phase and a C36-type intermetallic compound [4]. The LPSO-type $Mg_{96.75}Zn_1Y_2Al_{0.25}$ (at%) alloy, which was produced by hot extrusion of cast ingot, exhibited very high, symmetrical yield strength in both tension and compression, high heat resistance, and great flame resistance. Its corrosion resistance is the same as AZ31. The LPSO phase, which has a periodical stacking structure of the 4 atomic layers consisting of in-plane ordering of $L_{12} Zn_6 Y_8$ clusters and the 1-4 atomic layer(s) of 2H Mg, is strengthened by kinking, which was formed during the hot extrusion. This kink strengthening is a brand-new concept for strengthening mechanism of materials. On the other hand, the C36-type Mg_{84.97}Al₁₀Ca₅Mn_{0.03} alloy, which was also produced by hot extrusion of cast ingot, exhibited high, symmetrical yield strength, high corrosion resistance, and nonflammability; the ignition temperature is higher than the boiling temperature of pure magnesium. These advanced Mg alloys have passed the FAA flammability test for Mg alloys. The development of more sustainable and more affordable manufacturing technology for these next-generation Mg alloys has been conducted via an integrated and comprehensive collaboration between academia and industry. Moreover, the applications and commercialization of these advanced Mg alloys have been under serious investigation and study for automobile, aircraft, and biomedical industries.

- [1] Y. Kawamura, K. Hayashi, A. Inoue and T. Masumoto, Mater. Trans. 42 (2001) 1172-1176.
- [2] Y. Kawamura and S. Yoshimoto, *Magnesium Technology 2005* (TMS, 2005, pp499-502).
- [3] E. Abe, Y. Kawamura, K. Hayashi and A. Inoue, Acta Materialia 50 (2002) 3845-3857.

[4] Y. Kawamura and A. Inoue, Materials Science Forum 419-422 (2003) 709-714.

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Exploring Thermal Loading of Hybrid and Ceramic Composites by the Acoustic Emission Technique

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Keywords: magnesium, alumina, quartz ceramics, internal stresses, acoustic emission

Various metal/ceramic matrix composites were developed and manufactured over last decades for use as light-weight materials in a range of applications including, for example, the automotive industry or energetics. In practice, it is well established that the microstructures and the mechanical properties of these composites are strongly affected by the nature of the interfaces between the matrix and the reinforcement or between various composite constituents. The fabrication and/or standard operating conditions for most composites will generally include some form of thermal loading and, of necessity, this will introduce internal stresses because of the significant mismatch in the thermal expansion coefficients between the matrix and the reinforcement and of abrupt dimensional changes due to phase transitions that may occur within the material. It follows that even rather minor temperature changes may lead to structural changes, plastic deformation within the matrix and significant microstructural damage.

Acoustic emission (AE) is a non-destructive experimental technique in which transient elastic waves are generated within a material due to sudden and irreversible structural changes. It has been established that AE is a viable procedure for monitoring the development of microstructural changes during the thermal cycling of some composites. Specifically, since dislocation motion and microstructural damage are generally recognized to produce significant AE, there is a possibility of using AE monitoring to identify and characterize the microstructural changes occurring within the composites due to thermal loading and to correlate these measurements with the associated temperatures or other testing parameters.

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Activity of non-basal slips in magnesium alloys

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Keywords: magnesium, single crystal, dislocations, slip, twin

Magnesium and magnesium alloys are candidate materials for energy saving of transportation vehicles and devices due to their high specific strength and low density. However, the plasticity of magnesium at room temperature is poor since basal slip system, which is their main slip system, has limited number of independent slip systems available to accommodate applied plastic deformation. Therefore, activation of nonbasal slip systems is necessary to show good ductility. Recently, effect of yttrium on ductility of magnesium and discussion for activity of (c+a) pyramidal slips has been reported. In this study, to investigate effects of yttrium and other alloying elements, such as aluminium, zinc and cerium, on non-basal slips, [11-20] tensile and [0001] compression tests of magnesium alloy single crystals were carried out at 77K, 293K and 473K. Complex alloying effects on the deformation behaviour of magnesium in tension and compression were observed. In compression tests of Mg - 0.5Al, Mg - 0.5Zn, Mg -0.5Y, Mg - 1.45Al - 0.15Zn and Mg - 2.7Al - 0.4Zn, second order pyramidal slips (SPCS) were observed, and the critical resolved shear stresses (CRSS) of the SPCS sere increased by alloying. In tensile tests of Mg - 0.5Al, Mg - 1.0Al and Mg - 2.6Al - 0.3Zn, the alloys were yield due to {10-11} twin instead of SPCS. Mg - (0.6 - 0.9)Y single crystals were yielded due to first order pyramidal slip and showed high ductility than in pure magnesium. (c+a) edge dislocation immobilization by thermal activation process following double cross slip of (c+a) screw dislocation, have been proposed as deformation mechanism for SPCS in pure magnesium. Based on the mechanism, effect of alloying elements on ductility of magnesium was discussed.

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Acoustic Emission Study of Active Deformation Mechanisms in Mg Alloys

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Keywords: magnesium alloys, microstructure, mechanical properties, acoustic emission

The activity of individual deformation mechanisms during deformation tests of wrought Mg alloys was investigated using the acoustic emission (AE) technique. The investigated alloys exhibited a typical basal texture with basal planes oriented nearly parallel to the extrusion or rolling direction. Deformation tests were performed at room temperature and the obtained results are supported by microstructure evolution analysis provided by electron backscatter diffraction (EBSD). The AE signal analysis correlates the microstructure and the stress-strain curves to the active deformation mechanisms quite well. To determine the dominant deformation mechanism in a given time period, the adaptive sequential k-means (ASK) clustering was applied. X-ray diffraction was used to characterize the deformation texture before and after the test in order to obtain comprehensive data for texture characterization.

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Synergy of in-situ methods in study of deformation mechanisms of Mg alloys

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Keywords: magnesium, twinning, dislocation slip, in-situ experimental methods

Owing to their hexagonal close packed (HCP) structure and c/a ratio close to ideal value the deformation behavior of magnesium alloys differ from the other, e.g. fcc of bcc metals. The basal slip has the lowest value of critical resolved shear stress (CRSS), followed by prismatic slip and first-order pyramidal slip systems. All these slips provides deformation in $\langle a \rangle$ (i.e. $\langle 11\overline{2}0 \rangle$) direction and their combination provides only 4 independent slip systems. Therefore, the von Mises criterion requiring five independent slip systems for homogenous deformation is not fulfilled and activation of second-order pyramidal system or mechanical twinning is necessary.

The experimental study of the deformation mechanisms includes both ex-situ (e.g. optical light, scanning (SEM) or transmission electron microscopy (TEM)) and in-situ techniques (e.g. diffraction methods, acoustic emission etc.). The main drawback of microscopy methods in studying twinning and dislocations is the relatively small observed volume in the specimen. On contrary, the irradiated volume in the diffraction experiments provides statistically representative data. The X-ray line profile analysis, can be successfully used for ex-situ analysis of the temperature dependence of the dislocation structure evolution during uniaxial tensile test of magnesium alloys. In the neutron diffraction experiments, the overall twinned volume can be determined from the intensity variations of particular peaks, caused by the crystal lattice reorientation during twinning. The acoustic emission (AE) is a useful complementary experimental technique to ND during in-situ testing of the magnesium alloys. The main advantages of the method are the high time resolution and sensitivity to twin nucleation and collective dislocation motion.

In the talk the influence of various microstructural (composition, orientation of the reinforcement) and experimental (loading direction, temperature) parameters on the deformation mechanisms are elucidated using combined usage of AE, ND and SEM.

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Ongoing research for the LPSO-typed Mg alloys in Japan

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Keywords: magnesium, LPSO structure, deformation kink, multimodal microstructure

Being at the top of the list of light structural materials, Mg alloys are strong candidates for use in automotive applications. To expand their use, it is necessary to improve their strength at elevated temperatures as well as at room temperature. In the last one-and-a-half decades, we have witnessed the rapid development of the novel high-strength Mg alloys with long-period stacking ordered (LPSO) structure phase. The LPSO structures exhibit a periodic chemical modulation as well as long-period stacking, and act as alloy-strengthening component because of their unique plastic deformation behavior. This talk introduces ongoing researches of the LPSO-typed Mg alloys in Japan.

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Investigation of the effect of the LPSO-content on the deformation mechanisms in Mg-Y-Zn magnesium alloys by *insitu* methods

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<u>Keywords</u>: magnesium alloys, LPSO phase, microstructure, mechanical properties, *insitu* methods

The different amount of zinc (Zn) and yttrium (Y), as alloying elements, resulted in a various amount of the long period stacking ordered (LPSO) phase in the Mg matrix. The magnesium alloys were extruded at 350 °C with an extrusion ratio of 1:18 and an extrusion speed of 0.5 mm/s. The structure of the LPSO phase and the precipitates found in the microstructure were studied by transmission electron microscopy (TEM). The microstructure was further examined by scanning electron microscopy (SEM), using mainly backscattered electron (BSE) images and electron backscatter diffraction (EBSD) technique. The results show that as the result of the extrusion process, the LSPO phase is elongated along the extrusion direction (ED) and the magnesium matrix has a bimodal microstructure.

Mechanical properties of the alloys were investigated at various temperature between room temperature and 350 °C. The compression tests were performed with a constant strain rate of 10-3 s-1. The active deformation mechanisms were studied by the combination of in-situ acoustic emission, synchrotron diffraction, and post-mortem SEM investigation.

The results clearly indicate that both, the temperature and the LPSO phase content significantly influences the plasticity of the magnesium matrix, particularly the extension twinning and the non-basal slip mechanisms. The amount of the LPSO phase has also a high impact on the formation of the deformation kinks.

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Numerical study on development of intra-granular misorientations during compression of LPSO crystals

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Keywords: magnesium, LPSO, kinking, crystal plasticity, finite element analysis

Kink band, which is slip localization band perpendicular to slip direction [1, 2], is frequently formed in magnesium (Mg) based long-period stacking ordered (LPSO)-phase during compression where compressive axis is nearly parallel to the basal plane [3]. Notably, a contribution of kink band formation to strength was reported in the recent paper [4], which leads to further investigations for detailed understandings of kink bands formation and the effect on mechanical properties [5, 6]. In this study, to gain the understanding of formation process of kink bands in LPSO structure, full-field crystal plasticity analysis of compressive loading behavior of LPSO crystals were performed. Analysis model was generated based on experimental observation of Mg-based LPSO phase directionally solidified (DS) crystals. The effects of critical resolved shear stress (CRSS) on stress-strain behavior and development of intra-granular misorientations were studied.

[1] R. Asaro and J. Rice, J. Mech. Phys. Solids 25 (1977) 309-338.

[2] A. Marano, L. Gélébart and S. Forest, Acta Mater. 175 (2019) 265-275.

[3] K. Hagihara, N. Yokotani, Y. Umakoshi, Intermetallics 18 (2010) 267-276.

[4] K. Hagihara, Z. Li, M. Yamasaki, Y. Kawamura, and T. Nakano, *Acta Mater.* 163 (2019) 226-239.

[5] T. Inamura, Acta Mater. 173 (2019) 270-280.

[6] K. Takagi, T. Mayama, Y. Mine, Y. L. Chiu, and K. Takashima, *J. Alloys and Comp.* 806 (2019) 1384-1393.

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First-principles study on the origin of phase stability of Mg alloys with long-period stacking ordered structure

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Keywords: magnesium alloys, LPSO, first-principles DFT calculations, vacancy formation Weight reduction is one of the most important tasks for improving fuel economy and controlling CO₂ emissions of aircraft and automobiles. Therefore, it is compelling to develop light and strong materials. As a solution to this problem, the design of magnesium (Mg) based alloys has attracted much attention recently, since Mg is the lightest substance element for structural metals. Among them, a dilute Mg-based alloy with a nominal composition of $Mg_{97}Zn_1Y_2$ exhibit a remarkable high tensile yield strength and ductility. The strength of 600MPa in $Mg_{97}Zn_1Y_2$ is approximately three times larger than that of a commercial Mg alloy called AZ31B [1]. This strength is expected to be coupled with a unique atomistic structure where a concentration of solute atoms (Zn, Y) appears on the (0001) plane in a few layers of the hexagonal close-packed (hcp) Mg matrix, and Shockley's partial dislocation (stacking fault) occurs in the solute concentrated layers [2]. The stacking sequence is relatively long along the c-axis of hcp lattice. Therefore, such structure is referred to as long-period stacking ordered (LPSO) structure. Furthermore, recent structural analysis using transmission electron microscopy (TEM) revealed that L1₂ type clusters of solute elements (M_6Y_8) are embedded in the Mg matrix near the stacking fault [3,4].

In this study, to clarify the microscopic origin of the phase stabilities of LPSO structures, we calculate heats of formation and electronic structure of Mg -M-Y alloy (M = Co, Ni, Cu, Zn) with different compositions and stacking sequence using first-principle density-functional theory calculations. In this presentation, we show how the geometry of L1₂ type solute clusters affects electronic structure near the Fermi level that determine the structural stabilities of LPSO phases. Furthermore, we discuss the possibility of vacancy formation in Mg-Zn-Y alloy using convex hull on the calculated phase diagram.

[1] Y. Kawamura, K. Hayashi, A. Inoue, and T. Masumoto, Mater. Trans. 42, 1172 (2001).

[2] E. Abe, Y. Kawamura, K. Hayashi, and A. Inoue, Acta Metall. 50, 3845 (2002).

[3] D. Egusa and E. Abe, Acta Mater. 60, 166 (2012).

[4] J. Saal and C. Wolverton, Acta Mater. 68, 325 (2014).

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Anisotropy of Mechanical and Physical Properties of Magnesium Alloys and Composites Prepared by SPD Methods

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<u>Keywords</u>: magnesium, nanocomposites, severe plastic deformation, mechanical and thermal properties, internal friction

Magnesium based microcomposites are modern advanced materials which combine high specific modulus and strength with a significant damping capacity and dimension stability. Mechanical properties of materials may be improved by methods of severe plastic deformation. Magnesium alloys (AZ31, AX52) and magnesium nanocomposites (Mg+BN, ZrO₂) were submitted to hot extrusion (HE), equal channel angular pressing (ECAP), accumulative roll bonding (ARB) and rotary swaging (RSW). Mechanical and thermal properties and internal friction were estimated in various directions. Pronounced anisotropy of the tensile yield stress and ultimate tensile strength was found for samples prepared with various processing methods. Different texture developed in materials submitted to various deformation processes plays important role in controlling of mechanical and thermal properties

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Structure and mechanical properties of Zinc deformed by SPD process

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Keywords: ECAP, ARB, grain refinement, recrystallization, grain growth

Zinc is widely used for galvanizing steel products, manufacturing brass, manufacturing alloys for die casting, etc. in industries. On the other hand, zinc is an essential trace element for humans and shows ideal corrosion behavior in human bodies. Zinc is, therefore, expected to be used as biomaterials such absorbable stents. Enhancement of high mechanical properties of zinc is required for its practical uses as biomaterials, however, additions of alloying elements should be avoided from the viewpoint of reactions in human bodies. Ultrafine grained (UFG) materials – those with mean grain sizes less than 1 μ m – have been actively studied in various kinds of metallic materials since the late 1990s. Severe plastic deformation (SPD), which mechanically can induce large strain (equivalent strain: $\varepsilon_{eq.} > 4.0$) to materials, is utilized for bulk UFG material production. Various SPD process methods have been developed, including accumulative roll bonding (ARB), equal channel angler pressing (ECAP), and high pressure torsion (HPT). These processes succeed in producing bulk UFG materials with nano- or sub-micro-meter sized grains in various kinds of metallic materials, with the resulting UFG materials exhibiting high strength. In this study, ECAP deformation of both pure zinc single crystals and polycrystals, and ARB deformation of zinc polycrystals were investigated. Recrystallization occurred during ECAP and equiaxed grains were observed. The mean grain size was 12.8 µm after 8-pass of ECAP. On the other hand, the mean grain thickness was 6.9 µm after 7-cycle of ARB. ARB is the most efficient SPD for the grain refinement of zinc.

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Microstructure, texture and mechanical properties evolution in AX41 alloy processed by ECAP

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Keywords: magnesium alloy, ECAP, grain size, dislocation density, texture

The influence of three different equal channel angular pressing (ECAP) routes (A, Bc and C) and two ECAP temperatures of 220 °C and 250 °C on the microstructure evolution and mechanical properties in pre-extruded AX41 magnesium alloy was investigated. It was found that during the first passes, the rate of grain fragmentation strongly depends on the processing route. After 8 passes, despite the almost identical values of the dislocation density ($0.7 \times 10^{14} \text{ m}^{-2}$), the average grain size varied in the range of 2.0–4.5 µm for the individual ECAP routes. Macroscopic texture measurements revealed a gradual formation of very strong textures, which were significantly different for the various processing routes. Route A was found to be the most effective processing route for grain refinement. In tensile tests carried out at room temperatures, the highest strength was observed for the sample processed via route A for 8 passes, due to the highest texture hardening and the smallest grain size.

Besides the processing route, the inflence of processing temperature on the fragmentation rate of the microstructure was observed. It was found that during ECAP processing at 250 °C a smaller number of passes was enough to achieve a homogeneous microstructure than at 220 °C. However, the final grain size obtained after 8 passes at 250 °C was only 4 μ m which is larger than the value of 2.7 μ m achieved at 220 °C. Moreover, a significant influence of the temperature of ECAP processing on the dislocation density and the crystallographic texture was observed. The proof stress of the sample processed by 8 passes at 250 °C was lower than that obtained at 220 °C. It can be attributed to the lower dislocation density, the larger grain size and the texture, which facilitates the basal slip.

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Mechanical Properties of Novel Magnesium Alloys Prepared by Spark Plasma Sintering

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<u>Keywords</u>: magnesium, spark plasma sintering, microstructure, deformation, acoustic emission.

The spark plasma sintering (SPS) technique was used to prepare a bulk WN43 alloy from the gas atomized powder. Compression tests were performed to investigate the effect of different sintering regimes on the resulting mechanical properties of the material. It was shown that by increasing the sintering temperature, the ultimate compressive strength and ductility can be significantly improved. Moreover, complementary in-situ acoustic emission (AE) recording was employed to provide insights into microstructural changes during the deformation. Recent advances in the AE signal analysis in the frequency domain, together with microstructure observations, allowed us to reveal the evolution of different deformation mechanisms. It was shown that pronounced twin nucleation takes place around the yield point whereas twin growth and dislocation slip are the dominant deformation mechanisms in the later stages of deformation in this material.

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Potential Usage of Advanced Mg Alloys in Aerospace -Methodology to accelerate the R&D result transition

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Keywords: ICME, aerospace, magnesium, computation, digital data

Lightweighting of aerospace structures is an immensely impactful goal in the design and usage of aircraft development. High strength alloys based on magnesium offer an enormous opportunity to help achieve the purpose. Naturally there are a great deal of challenges in not only technology, but also in timing cycle related to these high value applications.

Integrated computational materials engineering (ICME) is a new R&D methodology that has been making important advances in materials science and engineering. Combining the theoretical models, experimental tools and digital data, it could not only make the R&D progress and results more robust, but also accelerate the critically needed transition. The two most important factors of low risk tolerance and long product cycles have a profound impact to make aerospace a most difficult industry to find benefit in ICME. This challenge is exacerbated by a large product scale and a correspondingly large component scale, which lead to substantial kinetic differences between the laboratory and production. However, considerable needs persist for cost and weight reduction, and more critically, for accelerating the introduction of materials that enable such improvements. In this presentation, I will try to deconstruct the life cycle of aircraft materials and identify where ICME can offer substantial value. Several examples of the application of computational methods to aerospace materials problems will be presented, from exploratory material design, to scale-up simulations, to the estimation of damage tolerance properties, and for polymer and metallic systems. Specific areas where ICME development is required will be highlighted. These tools will be relevant and pointed to general and specific aerospace materials and the R&D.

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Optimization of mechanical properties of rapid solidified Mg-Zn-Y alloys

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Keywords: rapid solidification, microstructure, mechanical properties, LPSO, SF

The Mg-LPSO-based alloys show enhanced mechanical properties comparing to the commercially used Mg alloys and therefore, recently they have been attracting considerable interest for engineering application and biomedical use. Moreover, it was found that alloy with small amount of Zn and Y (up to 2at.%) can exhibit promissing mechanical properties.

To investigate the influence of dilution on microstructure and mechanical properties the set of alloys with variation of Zn/Y: MgZn_{0.38}Y₁, MgZn_{0.47}Y_{1.25}, MgZn_{0.56}Y_{1.5}, MgZn_{0.75}Y₂, (in at.%) have been selected. The rapid solidified ribbons were consolidated by extrusion at 350 °C with an extrusion ratio of R10. For optimalization the preparation conditions different extrusion speeds were applied. The microstructure was analyzed by light, scanning and transmission electron microscopy, including BSE imaging and EBSD mapping.

All alloys are characterized by a microstructure containing non- and dynamically recrystallized grains with overall average grain size of 0.7-1.2 μ m. Low content of alloying element results in locally formed LPSO phase and solute segregation of stacking faults (SFs) parallel to the basal plane in α -Mg. The weak texture, owing preferentially random orientation of small dynamically recrystallized grains, promotes good ductility of the investigated alloys. Further, deformation mechanisms and resulting tensile properties were correlated to characteristics of the microstructure, particularly dispersion of SFs and LPSO phase and inhomogeneous distribution of internal strains via KAM analysis.

The investigated alloys processed by rapid solidified ribbon-consolidation have shown comparable tensile properties to the world record holders $Mg_{97}Zn_1Y_2$ alloy and $Mg_{98.5}Zn_{0.5}Y_1$ alloy prepared by powder metallurgy.

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From corrosion resistance measurement towards biodegradable magnesium-based materials

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Keywords: magnesium; corrosion; microstructure; biodegradation.

Investigation of magnesium alloys as a potential material for biodegradable implants did start at DPM UK as an unexpected continuation of research devoted to ultrafinegrained (UFG) materials. The first result which motivated deeper investigation into the difference in the corrosion resistance of UFG materials was an increase in the corrosion resistance of AZ31 magnesium alloy after ECAP. This result was rather surprising because it was believed that increase of surface grain boundary density should cause increase in the corrosion rate. From this point, investigation of underlying processes responsible for increase in corrosion resistance was studied especially on the AE21 and AE42 alloys. This research was shortly afterwards extended to magnesium alloys which were already successfully tested in vivo, particularly LAE442, WE43 and W4. Beside the investigation of new materials with unique microstructure, development of the corrosion measuring technique took place. It was shown that advanced acoustic emission analysis can be a powerful tool to explore activity of individual corrosion mechanisms during the potentiodynamic test.

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Improvement of flammability in LPSO-type Mg alloys

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Keywords: magnesium, STEM, incombustibility, high temperature oxidation, oxide film

Mg alloys have received great attention as structural materials due to their lightweight and reasonable strength. To reduce weight of vehicles, the automobile, railway, and aerospace industries expect to use Mg alloys for structural components [1]. However, the high chemical activity and easy ignition of Mg alloys at high temperatures are a serious problem. Despite this problem, the Federal Aviation Administration (FAA) of the USA has reformed regulation and established a flammability test toward the application of Mg alloys to aircraft cabin components. This regulatory reform promoted the development of nonflammable Mg alloys. Mg-Zn-Y with a long period stacking order (LPSO) phase has excellent mechanical properties [2]. However, it is necessary to improve the incombustibility of Mg-Zn-Y alloys to achieve more safety. Therefore, the ignition temperature of Mg-Zn-Y alloys is needed to be higher than the flame temperature of 1200 K of the FAA flammability test. In this study, effective elements were added to Mg-Zn-Y alloy to increase the ignition temperature and effect of effective element addition on incombustibility was investigated. To investigate effective element for incombustibility of Mg, ignition temperature measurements of $Mg_{99}X_1$ at% alloys were conducted. As results of ignition temperature measurements, the addition of Ca/Yb was dramatically effective for the increase of ignition temperature. Furthermore, Zeng et al. reported that addition of Be to rare earth containing-Mg alloy has effective for incombustibility [3]. As results of ignition temperature measurement, Be, Ca or Ybcontaining Mg-Zn-Y alloy exhibited the ignition temperature of ~1320 K. To investigate the effect of Be, Ca, or Yb addition, oxide films of Mg-Zn-Y alloys were observed. SEM observation revealed that internal oxidation and formation of crack occurred in Mg-Zn-Y ternary alloy. In the case of Be, Ca or Yb-containing Mg-Zn-Y alloys, internal oxidation and formation of crack were suppressed. Therefore, it suggests that suppression of internal oxidation and formation of cracks help to improve the incombustibility.

[1] F. H. Froes et al., Mater. Sci. Eng. A 184 (1994) 119-133.

[2] Y. Kawamura et al., Mater. Trans. 42 (2001) 1172-1176.

[3] Z. Xiaoqin et al., J. Mater. Process. Tech., 112 (2001) 17-23.

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