LPSO/MFS2022

The 5th International Symposium on Long-Period Stacking/Order Structure and Mille-feuille Structure

11-14 December, 2022
Prince Hotel SHINAGAWA, JAPAN

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科研費 KAKENHI

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Preface

We warmly invite you to the 5th International Symposium on Long-Period Stacking/Order and Mille-feuille Structures “LPSO/MFS 2022” to be held in Tokyo on 11 – 14 December 2022.

The LPSO/MFS 2022 serves to share rapid developments of layer-structured LPSO/MFS Mg alloys, which reveal remarkable strength through unique kink-type deformations. The symposium primary aims to provide a platform for those working in these materials to present their most recent research findings. Furthermore, the symposium will provide an opportunity to spark frontier ideas, which encourage extending the LPSO/MFS concept to various materials, including light alloys (Al, Ti, etc.), ceramics and even polymer materials.

The scientific scope of LPSO/MFS 2022 will cover all important aspects of a layer-structure design and/or kink-type deformations in high-strength structural materials, both for experiments and theory in the field of materials science, structural science, mechanics, computer simulations and mathematical modeling.

We heartily welcome you to Tokyo to participate in the Symposium along with your students, postdocs, colleagues, and others. Please mark the conference dates in your calendar NOW!

Eiji Abe and Yoshihito Kawamura
Chairpersons
LPSO/MFS 2022

Committee

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Tomotaka Miyazawa, Tokyo Institute of Technology

Daisuke Egusa, University of Tokyo

Invited speakers

Keynote speakers

Prof. Eiji Abe University of Tokyo

Prof. Yoshihito Kawamura MRC, Kumamoto University

Prof. Michel W. Barsoum Drexel University

Prof. Jian-Feng Nie Monash University

Prof. Alexey Romanov ITMO University

Prof. Sean R. Agnew University of Virginia

Invited speakers

Prof. Koji Hagihara Nagoya Institute of Technology

Dr. Hidetoshi Somekawa National Institute for Materials Science

Dr. Koji Kimura Nagoya Institute of Technology

Prof. Ken-ichi IKEDA Hokkaido University

Prof. Hajime Kimizuka Nagoya University

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Prof. Michiaki Yamasaki Kumamoto University

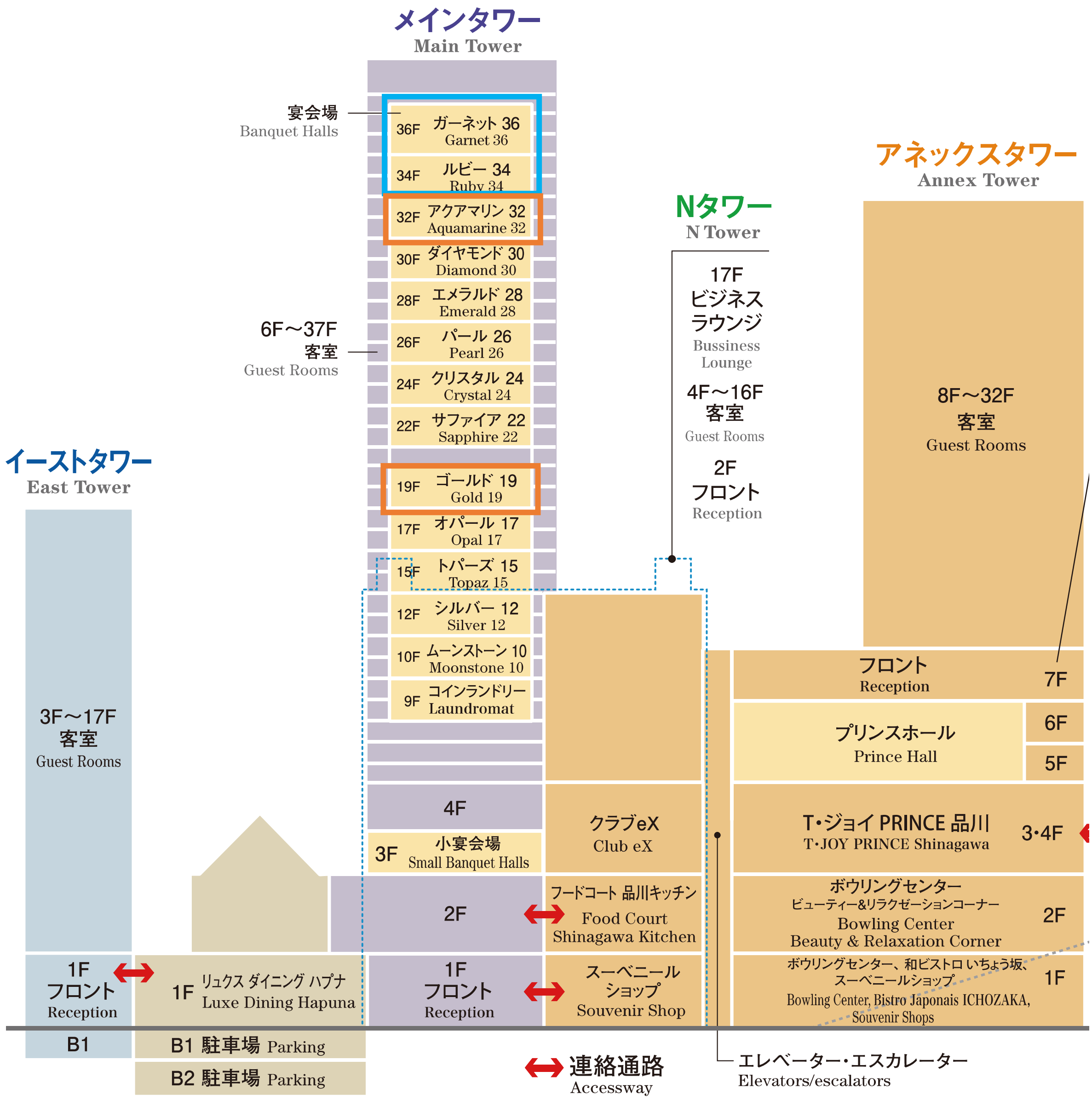
Prof. Xiaohong Shao Chinese Academy of Sciences

Dr. Toko Tokunaga Nagoya Institute of Technology

LPSO/MFS2022 Schedule

12/11(Sun)	12/12(Mon)	12/13(Tue)	12/14(Wed)
	9:00~12:00 1st session (Main Tower 34F Ruby)	8:00~13:00 Bus Tour Excursion around Imperial Palace & Sennsoji Temple(Asakusa)	8:30~10:10 6th session (Main Tower 34F Ruby)
			10:10~10:40 Coffee break (Main Tower 34F Ruby)
			10:40~12:30 7th session (Main Tower 34F Ruby)
	12:00~13:30 Lunch (Main Tower 34F Ruby)		12:30~14:00 Lunch (Main Tower 34F Ruby)
	13:30~15:35 2nd session (Main Tower 34F Ruby)	13:30~15:55 4th session (Main Tower 34F Ruby)	14:00~ Poster session (Main Tower 36F Garnet)
15:00~ Registration (Main Tower 19F Gold)	15:35~16:05 Coffee break (Main Tower 34F Ruby)	15:55~16:25 Coffee break (Main Tower 34F Ruby)	
17:00~ Welcome Reception (Main Tower 19F Gold)	16:05~18:10 3rd session (Main Tower 34F Ruby)	16:25~18:30 5th session (Main Tower 34F Ruby)	~17:00 Closing Remarks (Main Tower 36F Garnet)
		19:00~21:00 Banquet (Main Tower 32F Aquamarine)	

SHINAGAWA PRINCE HOTEL Floor map



The 5th International Symposium on

Long-Period Stacking/Order Structure and Mille-feuille Structure

Program
December 12 (Mon.), 2022
1st session: 9:00 ~ 12:00 Chair: Prof. M. W. Barsoum & Prof. T. Fujii

9:00~9:30 Keynote
"Mille-Feuille Structured Alloys/Ceramics/Polymers: Present Status and Future Perspectives"
Prof. E. Abe University of Tokyo

9:30~10:00 Keynote
"Production and Kink Strengthening of MFS-type Mg-Zn-Y Alloy with a-Mg Single Phase"
Prof. Y. Kawamura MRC, Kumamoto University

10:00~10:25 Invite
"Kink-band formation in mille-feuille structured metallic materials prepared by directional solidification"
Prof. K. Hagihara Nagoya Institute of Technology

10:25~10:50 Invite
"Kink boundary formation and morphology in wrought-processed Mg-Y-Zn alloys"
Dr. H. Somekawa National Institute for Materials Science

10:50~11:15 Invite
"Self-Tuned Structure of Solute-Clusters in Dilute Mg-Zn-Y Alloy Revealed by X-ray Fluorescence Holography"
Dr. K. Kimura Nagoya Institute of Technology

11:15~11:30 Oral
"Direct observation of pseudo-gap electronic structure in the Mg-Zn-Y alloys studied by hard X-ray photoelectron spectroscopy"
Prof. H. Miyazaki Nagoya Institute of Technology

11:30~11:45 Oral
"Temperature and strain dependence of plastic deformation behavior of long-period stacking-ordered magnesium alloys"
Dr. T. Miyazawa Tokyo Institute of Technology

11:45~12:00 Oral
"Microstructure and hardness in Mg-Y-Zn alloys with long-periodic stacking ordered phase processed by equal-channel-angular extrusion"
Prof. M. Yuasa Doshisha University

Lunch: 12:00 ~ 13:30

2nd session: 13:30 ~ 15:35 Chair: Prof. K. Mathis & Prof. K. Hagihara
13:30~14:00 Keynote
"Ripplocations: A Universal Deformation Mechanism in Millefeuile Solids"
Prof. M. W. Barsoum Drexel University

14:00~14:25 Invite

“Effects of Kink Boundaries on High Temperature Deformation Behavior of Textured Ti_3SiC_2 MAX Phase Sintered Body”

Prof. K. Ikeda — Hokkaido University

14:25~14:50 Invite

“Atomistic analysis of microscopic mechanisms of kink formation in metallic and ceramic mille-feuille structures”

Prof. H. Kimizuka — Nagoya University

14:50~15:05 Oral

“Kink Strengthening in TiNi-Nb and TiCo-Nb Alloys with Eutectic Mille-feuille Structure”

Prof. K. Ishikawa — Kanazawa University

15:05~15:20 Oral

“Plasticity enhanced in kinking orientations of cubic-structured ceramics micropillars”

Dr. H. Masuda — University of Tokyo

15:20~15:35 Oral

“Nanoindentation Examination of Kink Boundary Hardness in Ti_3SiC_2 MAX Phase”

Dr. K. Morita — National Institute for Materials Science

Coffee break

3rd session: 16:05 ~ 18:10 Chair: Dr. D. Drozdenko & Prof. M. Yamasaki

16:05~16:35 Keynote

“Microstructure, Deformation and Property of Wrought Magnesium Alloys”

Prof. J. F. Nie — Monash University

16:35~17:00 Invite

“Tensile and compressive mechanical behaviour in fully-lamellar extruded $MgGd_2Zn_1$ alloy”

Prof. G. Garces — CENIM-CSIC

17:00~17:25 Invite

“Hot deformation of Mg-Y-Zn alloy with different content of the LPSO phase studied by in-situ synchrotron radiation diffraction”

Prof. K. Mathis — Charles University

17:25~17:40 Oral

“Monitoring of strengthening of LPSO in αMg/LPSO dual phase alloy by hot-extrusion using in situ neutron diffraction during deformation”

Dr. S. Harjo — Japan Atomic Energy Agency

17:40~17:55 Oral

“Kink strengthening of LPSO Mg-Zn-Y alloys after processing by high-pressure sliding (HPS)”

Dr. Y. Tang — Kyushu Institute of Technology

17:55~18:10 Oral

“Texture evolution and mechanical properties of Mg-9Y-6Zn alloy via vortex extrusion”

Prof. D. Ando — Tohoku University

December 13 (Tue.), 2022

Excursion 12/13 8:00 ~ 13:00
Tour around Imperial Palace & Sennsoji Temple (Asakusa)

4th session: 13:30 ~ 15:55 Chair: Prof. H. Ito & Prof. H. Saito
13:30~13:55 Invite (Online)
"Stretch-induced structural evolution of semicrystalline polymers: the effects of strain rate"
Prof. L. Li University of Science and Technology of China

13:55~14:20 Invite
"Deformation Behavior of High-strength Mille-feuille Structured PVDF"
Prof. H. Saito Tokyo University of Agriculture and Technology

14:20~14:45 Invite
"Effect of Smectic Layer Deformation on Mechanical Properties of Glassy Liquid Crystal Polymer Fibers"
Prof. M. Tokita Tokyo Institute of Technology

14:45~15:10 Invite
"Preparation and Mechanical Properties Evaluation of Multilayer Film having Mille-feuille and Kink Structures"
Prof. H. Ito Yamagata University

15:10~15:25 Oral
"Large-scale MD simulations of reinforced polyethylene crystals by pre-elongation"
Prof. K. Hagita National Defense Academy

15:25~15:40 Oral
"Introduction of millefeuille-like α/β layered structure and investigation of its kink deformation behavior in β titanium alloys"
Dr. S. Emura National Institute for Materials Science

15:40~15:55 Oral
"Kink deformation and strengthening in an Al-30wt%Ag alloy with mille-feuille structure fabricated by aging precipitation and subsequent cold-rolling"
Prof. D. Terada Chiba Institute of Technology

Coffee break

5th session: 16:25 ~ 18:30 Chair: Prof. J. F. Nie & Prof. T. Inamura
16:25~16:55 Keynote (Online)
"Disclinations: from Theory to Practice"
Prof. A. Romanov ITMO University

16:55~17:20 Invite
"Direct observations of kink microstructure in mille-feuille structured Mg alloys"
Dr. D. Egusa University of Tokyo

17:20~17:45 Invite
"Thermal stability of the microstructure of dilute Mg alloys prepared by rapid solidified ribbon-consolidation technique"
Dr. D. Drozdenko Charles University

17:45~18:00 Oral

"Analysis of Kink Formation Behavior in Al/Al_2Cu Mille-feuille Structured Alloys"

Dr. T. Shiraiwa University of Tokyo

18:00~18:15 Oral

"Understanding of kink-band formation by means of a rate-independent model obtained by homogenization of mille-feuille structure"

Prof. K. Svadlenka Kyoto University

18:15~18:30 Oral

"Modeling and Numerical Analysis of Kink Deformation based on Geometrical Elasto-Plasticity Theory"

Mr. S. H. Pranoto Osaka University

Banquet: 19:00 ~ 21:00 @ Shinagawa Prince Hotel

December 14 (Wed.), 2022

6th session: 8:30 ~ 10:10 Chair: Prof. G. Garces & Prof. H. Kimizuka

8:30~9:00 Keynote (Online)

"Crystal Plasticity-Based Prediction of Forming Limit Diagrams: Application to Magnesium Alloys"

Prof. S. R. Agnew University of Virginia

9:00~9:25 Invite

"Influence of electrochemical and geometrical heterogeneities on corrosion behaviour of LPSO phase-containing Mg-Y-Zn alloys"

Prof. M. Yamasaki Kumamoto University

9:25~9:40 Oral

"Effects of Additional Deformation and Heat Treatment on Kink Bands on the surface of LPSO-type $Mg_{85}Y_9Zn_6$ Directionally Solidified Alloy Thin Plates"

Prof. M. Suzuki Toyama Prefectural University

9:40~9:55 Oral

"Microstructure and Deformation Behavior of BCC-V/MAX two-phase alloys"

Prof. S. Miura Hokkaido University

9:55~10:10 Oral

"Observation of as-cast and extruded $Mg_{97}Zn_1Y_2$ alloys by neutron diffraction and thermal dilatometer measurement"

Dr. K. Aizawa Japan Atomic Energy Agency

Coffee break

7th session: 10:40 ~ 12:30 Chair: Dr. H. Somekawa & Prof. S. Miura

10:40~11:05 Invite (Online)

"Synergetic kinking and twinning in an Mg-Zn-Y-Zr alloy with intragranular LPSO structures"

Prof. X. H. Shao Chinese Academy of Sciences

11:05~11:30 Invite

"Kink-band formation in Mg/LPSO two-phase alloys"

Dr. T. Tokunaga Nagoya Institute of Technology

11:30~11:45 Oral

"Crystal plasticity analysis of kink band formation in Mille-feuille structure"

Prof. T. Mayama Kumamoto University

11:45~12:00 Oral

"On Scale-Free Nature of Kink Formation/Strengthening based on FTMP"

Prof. T. Hasebe Kobe University

12:00~12:15 Oral

"Mesoscale modeling of system of planar wedge disclinations and edge dislocations via the Airy Stress Function method"

Prof. P. Cesana Kyushu University

12:15~12:30 Oral

"Effect of Kink Band Spacing on Kink Strengthening in LPSO-type Magnesium Alloy"

Prof. Y. Tadano Saga University

Lunch: 12:30 ~ 14:00

Poster session: 14:00 ~

37 posters (23 from students and 14 from researchers)

Poster Awards: 16:45 ~

Closing remarks: **Prof. E. Abe**
~17:00

Poster session
Students

S-01: Toughening of the LPSO-type Mg-Zn-Y-Al RS P/M alloys
Mr. S. Nishimoto Kumamoto University

S-02: Recrystallization Suppression and Kink Strengthening of MFS-type Mg-0.4Zn-1.0Y alloys
Mr. M. Hayashida Kumamoto University

S-03: Investigation of fracture toughness of extruded Mg-Zn-Y alloys with multimodal microstructure
Mr. T. Yasuda Kumamoto University

S-04: Mechanical properties and microstructure of dilute Mg-Y-Zn alloys prepared by combination of low cooling rate solidification and extrusion techniques
Mr. S. Ishizaki Kumamoto University

S-05: The Effect of Hot Rolling on Kink Formation in the MFS-type Mg-0.4Zn-1.0Y Alloy with α-Mg Single Phase
Ms. A. Yoshida Kumamoto University

S-06: Work-hardening Behavior of Mg-Y-Zn Alloy with Long-periodic Stacking Ordered Phase in High-pressure Torsion Process
Mr. H. Ohkoshi Toyohashi University of Technology

S-07: Texture evolution and mechanical properties of Mg-2Y-1Zn alloy via vortex extrusion
Mr. T. Tsuji Tohoku University

S-08: Numerical Investigation of the Microstructural Features Favoring Kink Band in Layered α/β Ti-9Cr Alloy
Mr. J. Zhu University of Tokyo

S-09: Deformation mechanism of LPSO-type Mg alloy after kink deformation
Mr. K. Gonome University of Tokyo

S-10: Deformed microstructure in LPSO-type magnesium alloys exhibiting PLC effect
Mr. N. Amemiya University of Tokyo

S-11: Dislocation analysis of solute-enriched stacking faults in kink boundaries in $Mg_{97}Zn_1Y_2$ alloys
Mr. Y. Zhao Kyushu University

S-12: Deformation anisotropy in hcp-Mg structure based on generalized stacking fault energy
Mr. Y. Ito University of Tokyo

S-13: Elastic energy of disclination multipole formed at intersection of kink band and shear band
Mr. R. Matsumura Tokyo Institute of Technology

S-14: Stable Shape of Curved Ridge-Kink Consisting of a Sequence of Fine Kink-bands
Mr. X. Zhang Tokyo Institute of Technology

S-15: Experimental Verifications of Kink-Band Strengthening in Long-Period Stacking Ordered Mg–Zn–Y Alloy
Mr. T. Tokuzumi Kyushu University

S-16: Deformation Behavior of Nb_2Co_7 as Crystal-Structure-Based Mille-feuille Structured Material
Ms. K. Yamada Hokkaido University of Science

S-17: Orientation dependence of various properties of textured MAX phase ceramics fabricated by slip casting under strong magnetic field
Mr. J. Muraoka Hokkaido University

S-18: Mille-feuille Structures in Al-RE Eutectic Alloys and Its Kink Formation
Mr. K. Hakita Hokkaido University

S-19: Fabrication of Al_2O_3-GAP eutectic ceramics with fine anisotropic microstructure by flash event and deformation behavior
Mr. Y. Aoki University of Tokyo

S-20: Effect of a high-pressure press on the strength and the morphorogy of PP
Ms. Y. Ito Yamagata University

S-21: Effect of Compression Molding of Co-Extruded PS/PBT Multilayered Film on Crystal Structure and Mechanical Properties
Mr. A. Iwamura University of Yamagata

S-22: Changes in the Elastomeric Mechanical Properties of Lamellae-Forming Block Copolymer Specimens by Introduction of Kink
Ms. Y. Kokuryo Kyoto Institute of Technology

S-23: Strengthening mechanism of crystalline polymer by heat elongation
Mr. M. Endo University of Tokyo

Researchers

R-01: Development of hcp/bcc layered structure in Mg-Sc alloy and kink formation by compression
Dr. Y. Ogawa National Institute for Materials Science

R-02: Effect of Additional Element on Nonflammability of LPSO type Mg-Zn-Y Alloy
Dr. S. Inoue Kumamoto University

R-03: Creep characterization of a long period stacking ordered $Mg_{85}Zn_6Y_9$ alloy
Prof. H. Takagi Nihon University

R-04: Observation of Kink Deformation in an LPSO $Mg_{85}Zn_6Y_9$ Alloy using Two-directional Micro-Laue Diffraction Mapping under Compression
Dr. S. Kimura JASRI

R-05: Phase transformation behavior of LPSO structure during annealing investigated by in-situ neutron diffraction
Dr. W. Gong Japan Atomic Energy Agency

R-06: Development of 4D in-situ observation system for the investigation of kink development during compression of Mg-based LPSO alloys
Dr. M. Uesugi JASRI/SPring-8

R-07: Electronic structure of Mg-Zn-Y cluster in dilute Mg alloys studied by STEM-EELS
Prof. Y. Sato IMRAM, Tohoku University

R-08: DFT Calculation of Kink Boundary Migration in LPSO Structure
Dr. M. Itakura Japan Atomic Energy Agency

R-09: First-Principles Study on the Electronic Origin of Phase Stability in Mg-Zn-Y alloys with a Long-Period Stacking Order
Dr. T. Tsumuraya Kumamoto University

R-10: Experimental Investigation on Kink Strengthening of Biotite Single Crystals: Rank-1 Connection and Surface Dislocation
Prof. H. Nagahama Tohoku University

R-11: Shape of Local Buckling in Cu/SBS Stacking Films Subject to Compressive Deformation
Prof. Y. Kaneko Osaka Metropolitan University

R-12: Elastic properties in a Ti_3SiC_2 MAX phase with a nanolayered crystal structure
Prof. M. Tane Osaka Metropolitan University

R-13: Thermodynamic Evaluation and Experimental Investigation of Suzuki Effect in the Al–X Binary Systems
Prof. T. Tokunaga Kyushu Institute of Technology

R-14: Kinking in Compression of Pearlitic Steel and Resultant Plastic Anisotropy
Dr. R. Ueji National Institute for Materials Science

December 12 (Mon.)

1st ~ 3rd session

Mille-Feuille Structured Alloys/Ceramics/Polymers: Present Status and Future Perspectives

Eiji Abe

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In general, in structural materials design the von Mises condition has been regarded as a fundamental necessary rule, which states that '*there must be five independent slip systems with the relevant crystal structure*' for continuous and stable plastic deformation of polycrystalline materials. In the "Materials Science of Mille-feuille Structure (MFS)" project, we have been aiming at a new concept of material strengthening by creating a layered structure with microscopic hard and soft layers, in which the easy-slip system is significantly restricted (non von Mises condition) and hence kink deformation is effectively induced.

Long-period stacking-ordered (LPSO)-type Mg alloys exhibit high strength only when kink deformation zones are introduced at high density by hot extrusion (kink strengthening phenomenon). For example, in dilute LPSO-type Mg alloys a mille-feuille structure is formed with solute-enriched stacking faults (SESF), which are the LPSO structural units, are randomly and sparsely arranged in the hcp-Mg matrix, and high strength is indeed realized through kink deformation. In MFS materials science, the possibility of kink strengthening has been investigated not only for metallic materials other than Mg alloys - steel, Ti alloys and Al alloys - but also extended to the three major materials including ceramics and polymers. In the presentation, I will summarize the results of MFS materials science to date in terms of "elucidation of the mechanism of kink strengthening" and "extending kink strengthening to the three major materials".

Production and Kink Strengthening of MFS-type Mg-Zn-Y Alloy with α-Mg Single Phase

Yoshihito Kawamura
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We have succeeded in producing the MFS-type α-Mg alloys with cluster arranged nanoplates (CANaPs) in the Mg-0.4Zn-1.0Y (at%) alloy. The solute contents of this alloy are much lower - ~50% lower in both Zn and Y - than those in other LPSO-type Mg-Zn-Y alloys [1]. The cast Mg-0.4Zn-1.0Y alloy always has residual segregation which cannot be entirely eliminated by further heat treatment. However, when rapidly solidified by melt spinning and subsequently consolidated by hot pressing, the single-phase solid solution could be obtained by a solution heat treatment. During aging, CANaPs evolve in a well-controlled manner in the α-Mg matrix of the Mg-0.4Zn-1.0Y alloy, as shown in Fig. 1. Thus, it paves the way for a quantitative understanding of the evolution of the CANaPs and their relations with the mechanical properties.

The four atomic layers in an fcc-type stacking sequence (ABCA) of the cluster arranged layer (CAL) initially grow sideways until they encounter the α-Mg matrix grain boundaries. We've then quantitatively studied the effect of thickness and number of the CANaPs on the strength of this MFS material. The CANaPs thicken by growing or coalescing newer layers. The CALs in the CANaPs, unlike LPSO, are separated by a random number of Mg layers with four or fewer atomic layers and initially grow with time. However, at a longer aging time, the dispersion of CANaPs decreases.

The formation of kink bands during extrusion across the CANaPs was observed. The dispersion of these kink bands is directly related to the dispersion of the CANaPs. The yield strength is linearly related to the dispersion of the kink band. Effectively, the presence of the CANaPs facilitates a kink band strengthening of the alloy. The MFS samples having dispersed CANaPs show only a limited improvement of the alloy's tensile strength, where the tensile yield strength and the elongation were 115 MPa and 8.3 %, respectively. However, the strength improves significantly when the MFS samples were extruded at 623 K, as shown in Fig. 2. The extruded MFS-type Mg-0.4Zn-1.0Y alloy has a tensile yield strength of 368 MPa and a tensile elongation of 10.5 %. It's comparable to the 375 MPa tensile yield strength for the extruded LPSO-type Mg-1.0Zn-2.0Y alloy with a 26 % volume fraction of LPSO phase [1]. The MFS-type Mg-0.4Zn-1.0Y alloy having half of the concentrations of both Y and Zn yields similar strength as the LPSO-type Mg-1.0Zn-2.0Y alloy. This result clearly points out that the dispersion of CALs is more effective in strengthening the materials than the localization of CALs in LPSO phase.

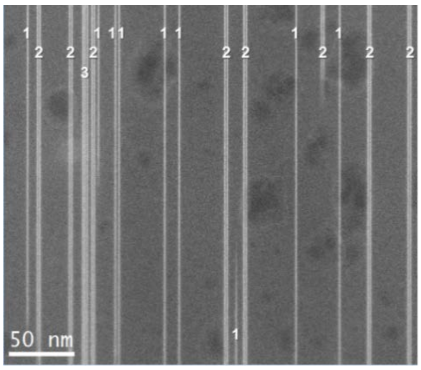

Fig. 1 HAADF-STEM micrograph of the CANaPs composed of 1 to 3 CALs in MFS-type Mg-0.4-1.0Y alloy.

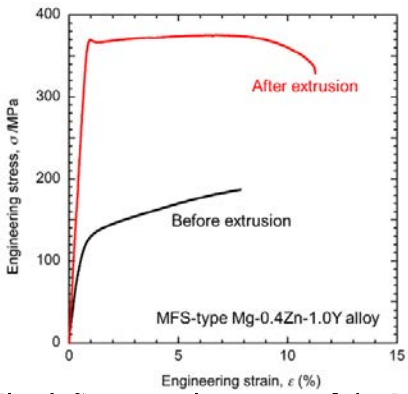

Fig. 2 Stress-strain curves of the MFS-type Mg-0.4Zn-1.0Y alloy before and after hot-extrusion.

References

[1] Y. Kawamura and S. Yoshimoto, *Magnesium Technology 2005*, (TMS, Warrendale, PA, 2005) 499-502.

Kink-band formation in mille-feuille structured metallic materials prepared by directional solidification

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Mg-based long-period stacking ordered (LPSO) phase with an approximate composition of $Mg_{12}ZnY$ is known to contribute to increase in both of strength and ductility of Mg alloys [1]. As a deformation mode in it, kink-band formation is recently focused [2]. A kink band is a deformation band formed by the explosive generation of dislocations in a localized region and their subsequent alignment along a direction normal to the slip plane. Recently, the kink band was found to contribute to the strengthening of the alloys prepared by the thermomechanical processes [3]. However, details of the formation criteria of deformation kink band have not yet been clarified. According to the study on the LPSO phase, its unique crystal structure which is constructed by the alternative stacking of soft and hard layers, called mille-feuille structure, are supposed as plausible factors to govern the formation of deformation kink bands [2].

To confirm these assumptions, we have examined the deformation behavior of several directionally solidified Mg-based and Al-based two-phase eutectic alloys with lamellar microstructure as a model mille-feuille structured material, such as $Mg/Mg_{17}Al_{12}$ [4], Mg/Mg_2Yb [5] and Al/Al_2Cu [6,7]. Consequently, the formation of kink-bands was confirmed in all the mille-feuille structured alloys, as expected (Fig. 1(b)). The details of the deformation microstructure and accompanying deformation behavior will be discussed in the presentation.

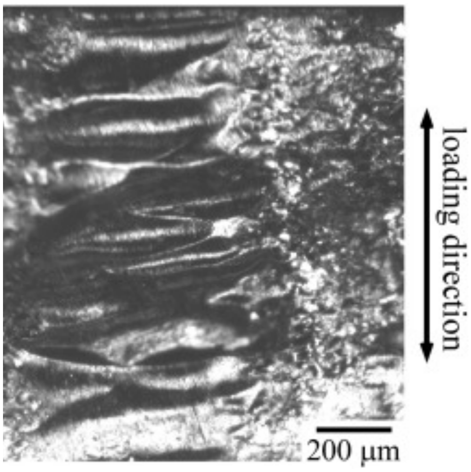

Fig. 1 typical deformation microstructures observed in Al-16.3 at% Cu [6]

References
[1] Y. Kawamura et al., *Mater. Trans.* **42** (2001) 1172-1176.
[2] K. Hagihara et al., *Intermetallics* **18** (2010) 267-276.
[3] K. Hagihara et al., *Acta Mater.* **15** (2019) 226-239.
[4] K. Hagihara et al., *Mater. Sci. Eng. A* **798** (2020) 140087.
[5] K. Hagihara et al., *Jour. Magne Alloys* **10** (2022) 492-500.
[6] K. Hagihara et al., *Mater. Sci. Eng. A* **825** (2021) 141849.
[7] K. Hagihara et al., *Int. Jour. Plast.* **158** (2022) 103419

Kink boundary formation and morphology in wrought-processed Mg-Y-Zn alloys

Hidetoshi Somekawa[1*], Yoshikazu Todaka[2], Daisuke Ando[3] and Motohiro Yuasa[4]

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Kink boundary, which is introduced *via* wrought-processing at elevated temperatures, has taken an attention as a new strengthening mechanism for metallic materials [1,2]. In general, as well as process conditions such as temperature and strain rate, the processing method affects microstructure of thermomechanical processed metallic materials, owing to the shear direction along the billet. This suggests that the selection of wrought-processing method leads to change the mechanical properties and microstructures including kink boundary; however, to the best of our knowledges, there are not any reports about this point. In this study, we investigated the effect of wrought-processing method on the mechanical property and microstructure. We also quantitatively evaluated the kink boundary fraction through experiments, and obtained strain distribution by numerical method. Finally, the correlation between the quantitative value and the influential factor related to strain was considered to determine the suitable conditions for introducing kink boundaries.

Cast Mg-9at.%Y-6at.%Zn alloy was subjected by several types of wrought-processes, i.e., forging, rolling, extrusion, ECAE, caliber rolling and HPT, at the temperature of 623 K. The simple compression test was also performed using the unique shape to consider the influential processing factors. Microstructures of each alloy were observed by optical and laser microscopy. The fraction of kink boundary was measured by the point-counting method [3]. The mechanical property of each wrought-processed and unique compressive alloys was evaluated by micro-Vickers hardness tests. As for the numerical study, strain distributions during compressive deformation at 623 K were obtained by finite element simulation using the DEFORM-3D software. The plastic deformation response obtained at various temperatures and strain rates using the same cast alloy [4] was used to this calculation.

The micro-Vickers tests shows that all of wrought-processed alloys exhibit higher hardness than that of the cast alloy, and this value increases with the magnitude of equivalent strain, irrespective of wrought-processing. For instance, ECAE-ed alloys have superior hardness to that in the extruded alloys. This is mainly due to the difference in kink boundary formation; where an alloy with denser kink boundaries has a possibility to exhibit higher hardness. In interesting, our previous study has pointed out, even in fabricating at the same number of rolling passes, that the area fraction of deformation kink boundaries and mechanical properties are influenced by the billet insert direction during caliber rolling [5]. While the tandem- and reverse-rolled alloys have the same number of rolling passes (indicating the same magnitude of applied equivalent plastic strain), the reverse-rolled alloy has higher hardness and a larger kink boundary fraction as compared to those in the tandem-rolled alloy. The billets in the case of caliber rolling are repeatably applied by "shear" with the same direction. These results propose that rather than the equivalent plastic strain, shear strain influences the kink band formation and mechanical property. As comparison with kink boundary fraction measured by the point-counting method, there is some scattering of the data; whereas, shear strain has a close relationship with area fraction of kink boundaries.

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Self-Tuned Structure of Solute-Clusters in Dilute Mg-Zn-Y Alloy Revealed by X-ray Fluorescence Holography

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Mg-Zn-Y alloys have received much attention as next-generation light-weight structural materials [1]. In these alloys, formation of $L1_2$-type Zn_6Y_8 clusters was found using transmission electron microscopy (TEM) [2], which has been discussed in relation to the superior mechanical properties. Recently, Hagihara et al. [3] reported that such mechanical properties can be achieved even with very small concentrations of solute-elements. However, the arrangements of these dilute solute-elements have not been clarified well, while aggregation of solute-elements at the stacking fault was observed by TEM [3]. X-ray fluorescence holography (XFH) can provide three-dimensional atomic configurations around a specific element, and has been used for characterizing dopant clusters in various materials [4]. Thus, XFH is a promising tool to determine the arrangement of dilute solute-elements in Mg matrix. In this study, XFH was applied to $Mg_{99.2}Zn_{0.2}Y_{0.6}$ alloy in order to elucidate the Zn/Y arrangements.

XFH measurements were performed at BL39XU in SPring-8, Japan. The incident X-ray energies were set from 10.0 to 14.5 keV in steps of 0.25 keV and totally 18 Zn-Kα holograms were obtained.

Figure 1(a) shows the reconstructed atomic image around Zn. Here, the Y atomic images are observed indicating that the Zn and Y are located almost on the same plane, although these Y images were not present in the conventional $Mg_{75}Zn_{10}Y_{15}$ alloy including Zn_6Y_8 cluster [5]. From this result, we derived a symmetry-breaking $Zn_3Mg_3Y_8$ cluster as shown in Fig. 2(b) using first-principles calculations. Here, the position of Y atoms deviates only by 0.2 Å in the *c*-axis direction from the Zn atoms, which is significantly smaller than the corresponding deviation of 0.7 Å in the high-symmetric Zn_6Y_8 cluster. In the talk, the electronic states of this system are also discussed, which reveals that the arrangement of solute-elements are self-tuned to adopt the $Zn_3Mg_3Y_8$ cluster structure to enhance the phase stability

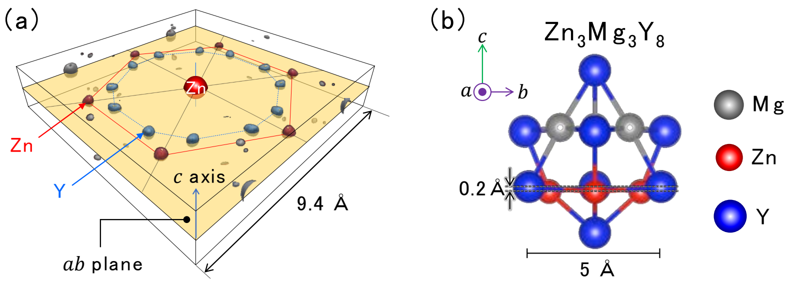

Fig.1 (a) Atomic image around Zn in $Mg_{99.2}Zn_{0.2}Y_{0.6}$ alloy obtained by XFH experiments. (b) $Zn_3Mg_3Y_8$ cluster derived from XFH results and first-principles calculations.

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Direct observation of pseudo-gap electronic structure in the Mg-Zn-Y alloys studied by hard X-ray photoelectron spectroscopy

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Mg alloys doped with transition metal and rare earth element having a long-period stacking structure are attracting attention as next-generation lightweight structural materials due to their excellent properties of ultra-high strength, flame retardance, and light weight. The excellent functionality and stability are believed to be due to the dispersion of the added elements in the Mg matrix to form local clusters. In the $Mg_{99.2}Zn_{0.2}Y_{0.6}$ alloy [1], it has been experimentally determined by X-ray fluorescence holography method by K. Kimura that local cluster structures different from those in previous Mg-Zn-Y alloys are formed. However, it is still unclear how the existence of these local clusters contributes to the stabilization of the electronic structure in Mg-Zn-Y alloys. In this study, we have investigated the electronic structure of $Mg_{99.2}Zn_{0.2}Y_{0.6}$ alloy by hard X-ray photoelectron spectroscopy (HAXPES), which can directly observe the electronic structure near the Fermi level that contributes to phase stabilization, to gain insight into the mechanism of phase stabilization in Mg-Zn-Y alloy systems.

$Mg_{99.2}Zn_{0.2}Y_{0.6}$ single-crystal sample was prepared by the Bridgman method [1]. Mg was also measured as a reference sample. HAXPES measurements were performed at the BL09XU beamline of SPring-8. All photoemission spectra were recorded at 30 K. Clean surfaces of the materials for HAXPES measurement was obtained by ex-situ scraping with a diamond file and immediately installing the samples in the HAXPES chamber. Fermi level and total energy resolution were determined by the Fermi edge of evaporated gold films. The total energy resolution of the HAX-PES measurement was set to 180 meV at the excitation photon energies of 7942 eV.

Figure 1 shows the valence band photoemission spectra of $Mg_{99.2}Zn_{0.2}Y_{0.6}$ and Mg near the Fermi level. A peak associated with Fermi surface Brillouin zone interaction around 0.3 eV was observed in both $Mg_{99.2}Zn_{0.2}Y_{0.6}$ and Mg alloys, and a pseudogap-like electronic structure was observed near the Fermi level. The pseudogap structure near the Fermi level is shifted to the higher binding energy side in the $Mg_{99.2}Zn_{0.2}Y_{0.6}$ alloy compared to Mg alloy, suggesting that the pseudogap is deeper than in Mg alloy. This result suggests that the energy gain of the electronic system in Mg-Zn-Y alloys contributes to the phase stability in this alloy.

In the presentation, the phase stability of Mg-Zn-Y alloys will be discussed in detail together with the results of first-principles calculations.

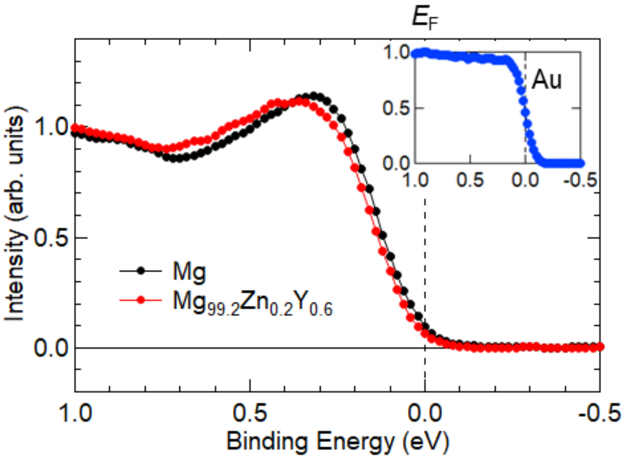

Fig. 1 Valence band photoemission spectra of $Mg_{99.2}Zn_{0.2}Y_{0.6}$ and Mg alloys with $h\nu$ = 7942 eV. Inset shows the photoelectron spectra of metallic gold film.

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Temperature and strain dependence of plastic deformation behavior of long-period stacking-ordered magnesium alloys

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Plastic deformation behavior in long-period stacking ordered (LPSO) magnesium alloys, including the formation of a kink-band, has been investigated by many researchers recently. Hagihara et al. reported the temperature dependence of the strain rate sensitivity factors of directional solidified (DS) Mg–Zn–Y alloys with 14H, 18R, and 10H LPSO structures [1]. In the temperature range of 293–573 K, the strain rate sensitivity (*m*) factors were negative, and the Portevin–Le Chatelier (PLC) effect was observed in all the alloys. These results indicated that the dynamic segregation of Y and Zn occurred at the boundaries of the kink-bands and impedes their migration. Conversely, low temperatures (< 250 K) hider the diffusion of solute atoms, causing the PLC effect to disappear and the strain rate sensitivity to change from negative to positive. However, there have been no studies on the *m* values at temperatures below room temperature. Therefore, in this study, to discuss the plastic deformation behavior of the LPSO Mg alloys, the investigation of the strain rate sensitivity *m* was conducted with strain rate jump tests at low temperatures.

The DS Mg–6at%Zn–9at%Y alloy composed of the 18R LPSO phase was used. The rectangular compression test specimens were cut to the required dimensions (2 mm × 2 mm × 5 mm) by an electrical discharge machine. The compression axis is parallel to the growth direction. The strain rate jump tests were carried out between the strain rates of 1.67×10^{-4} s^{-1} and 1.67×10^{-3} s^{-1} at temperatures ranging from 89 to 296 K under the atmospheric pressure. The *m* value was calculated from the change in the flow stress with the jump in strain rate.

Fig. 1 shows true stress–true plastic strain curves at each temperature. The flow stress curve exhibited serration, with a drop occurring after yielding caused by the formation of kink-bands. In the middle and later stages, linear work hardening was observed at all temperatures. Fig. 2 shows the strain dependence of the *m* value. The *m* values decreased with increasing the true plastic strain in this alloy. At 296 K, although the *m* values were positive in the early stage of the plastic deformation, they became negative and the PLC effect appeared as true plastic strain exceeded 10%. In contrast, at 89 K, the *m* values remained positive and the PLC effect disappeared. The temperature and strain dependence of the *m* value observed in this study are the same as for a Au-Cu alloy [2]. That means the plastic deformation behavior of the 18R LPSO Mg alloy including the kink-band formation, is similar to that of a solid solution alloy.

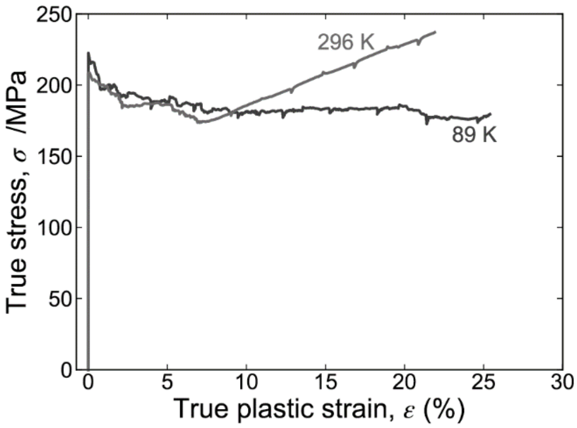

Fig. 1 True stress–true plastic strain curves.

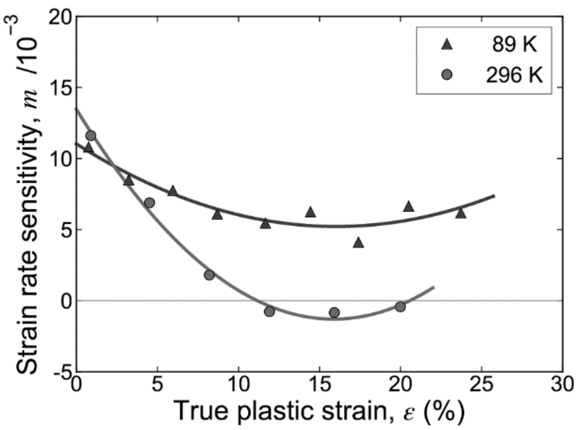

Fig. 2 True plastic strain-dependence of *m* value.

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Microstructure and hardness in Mg-Y-Zn alloys with long-periodic stacking ordered phase processed by equal-channel-angular extrusion

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Recently, magnesium alloys containing the long-period stacking order (LPSO) phase have been attracting attention because of their strength exceeding the high-strength aluminum alloys [1]. It has been reported that this high strength is due to the formation of deformation kink bands by wrought-processing methods [2]. Our previous study has reported the effects of wrought-processing methods on the formation of the kink bands and their mechanical properties [3]. The study has suggested that the strain introduced into the specimens by the wrought-processing methods affects the formation of kink bands and mechanical properties. In this study, we focused on the equal-channel-angular extrusion (ECAE) process, in which strong shear is applied to specimens. The objective of this study was to evaluate the effect of the strong shear during the ECAE process on the formation of kink bands and their mechanical properties.

The cast Mg-9at%Y-6at%Zn alloys were processed by ECAE at 623 K with a ram speed of 1 mm/min. The ECAE was carried out up to three passes using route Bc. The microstructure was observed using a laser microscope, and the area fraction of deformation kink bands was measured by the point-counting method. The hardness of the specimens was measured by micro-Vickers hardness tests under the indentation load of 300 gf and 10 gf. Using local hardness with the indentation load of 10 gf, the magnitude of kink bands strengthening was estimated by subtracting the hardness in the area where kink bands were observed with the laser microscopy from that in the area where kink bands were not observed [4].

Figure 1 shows the microstructure of ECAEed specimens. The area fraction of the deformation kink bands tended to increase with the number of ECAE passes. The result of micro-Vickers hardness tests shows that the hardness increased with the area fraction of the kink bands. However, the magnitude of increase in local hardness related to kink band strengthening was comparable to the forged alloys [4] despite the strong shear deformation induced by ECAE. This suggests that the shear strain induced by ECAE is effective for increasing the area fraction of kink bands in the entire specimens, but not effective for a higher density of kink boundaries in the local region.

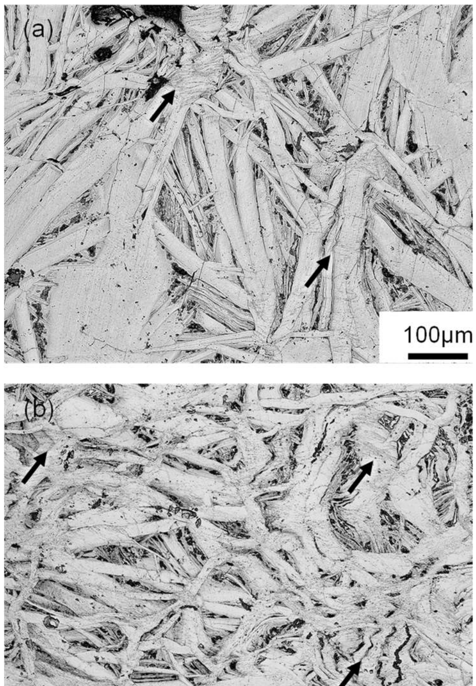

Fig. 1. Deformation microstructure in the ECAEed alloys: (a)1pass and (b) 3pass. The black arrows indicate the kink bands.

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Ripplocations: A Universal Deformation Mechanism in Millefeuile Solids

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Geologic formations and layered materials/solids - such as laminated composites, card decks, wood, the MAX phases, mica, graphite, etc. will, when compressively loaded edge-on, fail by buckling, that can result in kink bands. Recently we have shown that ripplocations and not basal dislocations, BD, are responsible for kink band formation. Ripplocations, at least in graphite, are highly mobile, fully reversible, dissipative waves that form as a result of confined buckling. When standing, the crests and troughs of the waves define ripplocation boundaries – also by definition fully reversible - that, beyond a critical load, naturally transform into ripplocation boundaries, that share some similarities to kink boundaries are fundamentally different. By confining and indenting decks of cards, thin steel or Al sheets edge-on with a cylindrical indenter we nucleated and propagated ripplocations. Molecular dynamics, MD, simulations in graphite at 10 K and Ti_3AlC_2 nucleated and propagated ripplocations that, but for scale, were identical to those nucleated in our macroscopic experiments. In this talk, a first attempt to model the mechanics of ripplocation nucleation and propagation is presented. We also show that the observed energies dissipated per cycle can be accounted for by Coulombic friction between the layers. As importantly direct compelling evidence for ripplocations in high resolution TEM images of the MAX phases, graphite and mica are presented. The latter being quite similar to those modelled by MD. Lastly, we predict the presence of a new type of basal dislocations pairs in the MAX phases whose core energies and mobilities are relatively low [2]. The implications of these discoveries for our understanding of the deformation of layered solids is profound and will be touched upon.

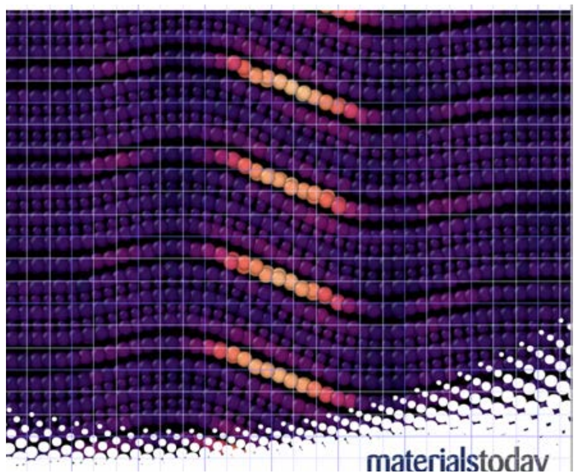

Fig. 1. Molecular dynamics of ripplocations in Ti_3AlC_2. [1]

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Effects of Kink Boundaries on High Temperature Deformation Behavior of Textured Ti_3SiC_2 MAX Phase Sintered Body

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The ternary MAX phase ceramics have the general formula $M_{n+1}AX_n$, where "*M*" is an early transition metal; "*A*" is group A element; "*X*" is C and / or N, and n = 1, 2, 3. The MAX phase ceramics are classified as mille-feuille materials because of their hard-soft two-layered structure consisting of MX and A layers due to the difference in bonding strength. It is also known to be a novel material that combines ceramic and metallic properties due to the difference in its bonding strength. Focusing on plastic deformation, it has been reported that kink deformation occurs in many MAX phase ceramics during deformation and fracture [1]. We have successfully fabricated textured dense and porous Ti_3SiC_2, a kind of MAX phase ceramics, sintered bodies, and investigated various mechanical properties and deformation structures [2-4]. The objective of this study is to clarify the effect of kink boundaries on high temperature deformation behavior using these textured sintered bodies.

In this study, commercial Ti_3SiC_2 powder was used. Textured dense and porous Ti_3SiC_2 sintered bodies were prepared by slip casting in a strong magnetic field (12T) followed by spark plasma sintering (SPS). Specimens of textured dense sintered body with the compression axis perpendicular to the c-axis direction were prepared, and kinks were introduced by compressive deformation at 1200°C up to 6.8% using SPS machine. Compression specimens were prepared from textured dense with kink, textured dense, and textured porous sintered bodies. The direction of the compressive axis was 45° to the c-axis direction. High temperature compression tests were conducted at 1200°C in vacuum and at a strain rate of 3×10^{-4} s^{-1}. SEM-BSE observation, EBSD analysis, and TEM/STEM observation were carried out to observe the microstructures of the specimens before and after the compression tests.

Compression tests at 1200°C showed plastic deformation in all cases, and the 0.2% proof stress was the highest for the oriented dense sintered specimens. It was suggested that the pores introduced in advance and the delamination induced during the kink deformation process affected the 0.2% proof stress. On the other hand, a comparison of the work hardening exponent n values showed that the kinked specimens tended to be larger than the other specimens. Microstructural observations also revealed a unique structure in the deformed microstructure in the vicinity of the introduced kink boundary. From these results, it is considered that the introduction of kink in MAX phase ceramics causes a kink strengthening that affects the work-hardening behavior.

Acknowledgments
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Atomistic analysis of microscopic mechanisms of kink formation in metallic and ceramic mille-feuille structures

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The reinforcing phase of Mg alloys with a mille-feuille (MF) structure or LPSO structure is characterized by a layered structure, in which hard layers enriched with solute elements and soft layers composed of Mg are arranged on the nanometer order. A kink formation accompanied by crystal rotation during deformation results in high strength. Kink bands induced by stress loading have been observed in a wide range of materials, not limited to Mg-based MF structures. To elucidate the mechanisms of changes in mechanical properties and strengthening due to the introduction of kinks, it is useful to understand the similarities and differences between various MF structures in which kinks are frequently observed. In this study, we investigated the microscopic mechanisms of kink formation in different types of MF structures and clarified their characteristics by capturing the microscopic elementary processes during plastic deformation of metallic and ceramic MF structures using atomic-level simulation methods.

A Cu-Nb nanolamellar structure was taken as an example of a mettalic MF structure to investigate the microscopic elementary processes during plastic deformation. Molecular dynamics simulations were performed to analyze the behavior of dislocations emitted from the lamellar interfaces, as well as the atomic-level microstructures observed in the deformation process of the lamella structure. The results showed that the interface acts as both a source and sink of dislocations during compressive deformation parallel to the lamellar interface, and that large plastic deformation occurs without interlayer delamination. Furthermore, it was confirmed that a sharp kink is formed due to elastic buckling when the layer thickness is small, while a blunt kink is gradually formed by a combination of complex plastic deformation processes as the layer thickness increases.

A $Ti_3(Si,Al)C_2$ MAX phase was taken as an example of a ceramic MF structure. The $Ti_3(Si,Al)C_2$ crystal consists of hard Ti_6C octahedral layers and soft (Si,Al) layers alternately stacked in the *c*-axis direction, and the difference in slip between the two layers causes significant anisotropy in deformation. When compressed at 45° to the basal plane, slip deformation was dominant due to the activity of dislocations in the basal planes, whereas when compressed at an orientation close to parallel to the basal plane, kink formation was observed due to the cooperation of dislocation activity and interlayer delamination along the basal planes. In addition, the generation and activity of ripplocations played an important role in this process. The basal dislocation, which is a component of the kink interface, showed significant asymmetry in mobility depending on the dislocation component and the position of the slip plane [1, 2].

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Kink Strengthening in TiNi-Nb and TiCo-Nb Alloys with Eutectic Mille-feuille Structure

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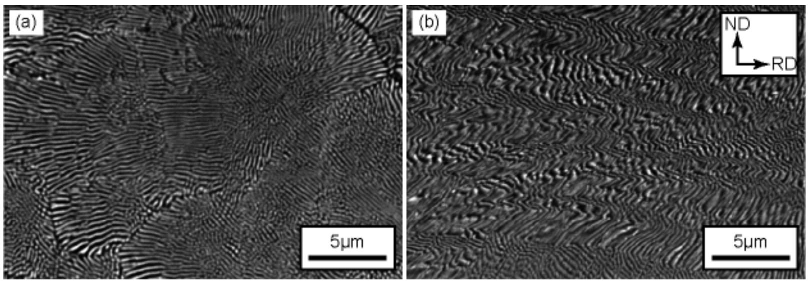
The TiNi-Nb and TiCo-Nb alloys are strong candidates for next-generation hydrogen separation and purification materials to replace Pd-based alloys. Since Nb shows severe hydrogen embrittlement, TiNi or TiCo phase is added to Nb to achieve both hydrogen permeability and resistance to hydrogen embrittlement. The TiNi (TiCo)-Nb alloy has an eutectic Mille-feuille structure consisting of B2-TiNi (TiCo) and bcc-Nb phases [1, 2]. Since hydrogen flux passing through the alloy is inversely proportion to the alloy thickness, thin alloy membrane is produced by rolling industrially. Thus, its mechanical properties are also important. In this research, kink formation, thermal stability of microstructure and mechanical properties of TiNi (TiCo)-Nb alloys were investigated, and kink strengthening effects in these alloys are estimated quantitatively on the basis of the experimental data.

The TiNi (TiCo)-Nb alloys were prepared by arc melting. The samples, cut from the ingots using an electrical discharge machine, were rolled at room temperature, and annealed at different temperatures in an electric furnace. Microstructural observations were carried out using scanning electron microscopy (SEM). The Vickers hardness of the samples were measured by a micro-Vickers testing machine. Tensile tests were carried out using a tensile testing machine with an initial strain speed of 0.83 s^{-1}.

SEM micrographs of (a) as-cast and (b) 30% rolled TiNi-Nb ($Ti_{40}Ni_{41}Nb_{19}$) alloys are shown in Figs. 1. The eutectic type mille-feuille structure consisting TiNi (gray) and Nb (white) phases was clearly observed in the (a) as-cast alloy. After rolling, layer bending occurred without peeling of TiNi/Nb phase, as shown in Fig. 1(b). The Vickers hardness increased from 321 for the as-cast alloy to 366 for rolled one, which indicates that the TiNi-Nb alloy was strengthened by introduction both dislocations and kinks. After annealing at 923 K for 1 h, the hardness recovered to the same value for the as-cast alloy. Furthermore, kinks remained in this sample after annealing. Therefore, an alloy having a mille-feuille structure including kinks but no dislocations can be obtained. The yield stresses of as-cast, 30% rolled and annealed (at 923 K) alloys were obtained as 529, 783 and 577 MPa by tensile tests, respectively. Here, yield stress of rolled alloy can be expressed by the following equation:

$$\sigma_{\mathrm{y}}^{\mathrm{rolled}} = \sigma_{\mathrm{y}}^{\mathrm{as\text{-}cast}} + \Delta\sigma_{\mathrm{d}} + \Delta\sigma_{\mathrm{k}} \qquad (1)$$

where $\sigma_{\mathrm{y}}^{\mathrm{as\text{-}cast}}$ is the yield stress of the as-cast alloy, and strengthening effects by dislocations and kinks are defined as $\Delta\sigma_{\mathrm{d}}$ and $\Delta\sigma_{\mathrm{k}}$, respectively. Comparing the yield stresses of as-cast, rolled and annealed alloys, the extent of the strengthening effects of dislocations and kinks can be estimated to be about 210 and 50 MPa, respectively. In the case of TiCo-Nb alloys, larger kink strengthening and smaller dislocation strengthening effects were estimated than those of TiNi-Nb alloys. In conclusion, we can demonstrate that the TiNi (TiCo)-Nb alloys having a mille-feuille structure consisting B2 and bcc phases are strengthening by kinks.



Figs. 1 SEM micrographs of (a) as-cast and (b) rolled TiNi-Nb ($Ti_{40}Ni_{41}Nb_{19}$) alloys.

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Plasticity enhanced in kinking orientations of cubic-structured ceramics micropillars

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Ceramics materials exhibit superior mechanical strength but with low ductility and toughness. As a potential strategy to overcome the brittleness, we have focused on the feasibility of kink toughening in ceramics, where kink formation contributes to small-scale plasticity and enhances ductility and/or toughness. Our recent studies found that kink formation is associated with enhanced energy absorption densities during compressions in near-[111] orientations of Y_2O_3-stabilized ZrO_2 (YSZ) ceramics [1, 2].

In this study, orientation-dependent plasticity of several cubic-structured oxide ceramics, such as 10 mol% Y_2O_3-stabilized ZrO_2 (10YSZ; fluorite), non-doped Y_2O_3 (bixbyite) and non-doped $SrTiO_3$ (perovskite) was investigated through single-crystal micropillar compression experiments from various orientations. Cylindrical micropillars with diameters of 0.9–1.1 μm and heights of 2.5–4.0 μm were fabricated via a focused ion beam technique and compressed at room temperature with an initial strain rate of 1×10^{-3} s^{-1} using a nanoindentation facility equipped with a flat-ended diamond tip. Surface slip trace analysis and cross-sectional dislocation characterization were performed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM), respectively. Furthermore, orientation dependence of strain and dislocation structure developments inside 10YSZ micropillars were numerically simulated using a crystal plasticity finite element method (CPFEM).

Nominal stress–strain curves of 10YSZ micropillars compressed at room temperature along different orientations are demonstrated in Fig. 1, indicating a significant orientation dependence in the plastic deformability. The [001] and [101] compressions exhibit plastic yielding but were associated with earlier fracture at strains below 10%. In contrast, extensive deformability is recognized in the compression along the [111] direction (i.e., the kinking orientation), where the pillar exhibited no fracture up to the nearly 40% strain. This enhanced plasticity could be associated with dislocation activities on multiple {001}<110> slip systems, as revealed by the slip trace analysis and dislocation characterization through SEM and TEM observations. Such plastic anisotropies were also observed in Y_2O_3 and $SrTiO_3$ micropillars, where considerable plasticity was achieved with multiple slip activations.

The CPFEM simulation demonstrated that the deformation homogeneity was enhanced with increase in secondary slip activities. This suggests that the enhanced deformability at kinking orientations of 10YSZ (and possibly other cubic-structured ceramics) is attributed to the enhanced deformation homogeneity and suppression of slip localization as the trigger of brittle fracture.

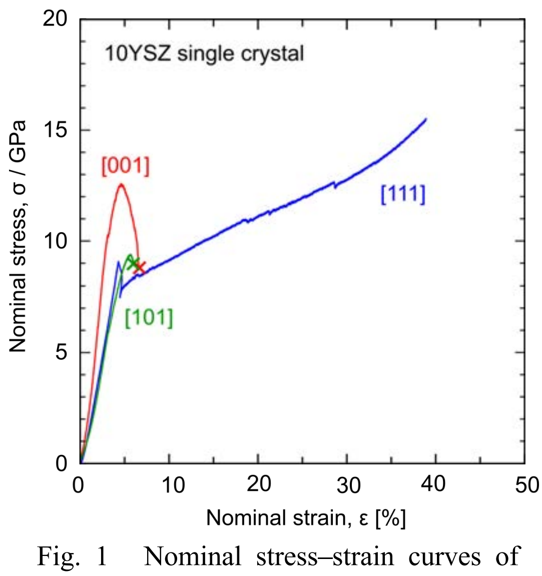

Fig. 1 Nominal stress–strain curves of 10YSZ micropillars compressed at room temperature along different orientations.

Acknowledgement: This study was supported by JSPS KAKENHI for Scientific Research on Innovative Areas "MFS Materials" (21H00091).

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Nanoindentation Examination of Kink Boundary Hardness in Ti_3SiC_2 MAX Phase

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The MAX phase ceramics, which can be expressed by the formula of $M_{n+1}AX_n$ (n = 1~3), have attracted much attention as multi-functional materials because those can simultaneously possess ceramic- and metallic-like properties due to its unique bonding nature [1-3]. Since the MAX phase ceramics with hexagonal structure have frequently reported kink formation through deformation, it is expected to exhibit king strengthening similar to that of LPSO-type Mg alloys [4,5]. This study, therefore, was carried out to examine the possibility of kink strengthening in the MAX phases using the Ti_3SiC_2 polycrystal as a reference material, which is known as the most representative MAX phases. In order to examine the kink strengthening in the Ti_3SiC_2-MAX phase, kink boundaries were formed by high temperature compressive tests, and then, nanoindentation tests were performed around the kink boundaries with several characters formed through the compressive creep test [6].

First, polycrystalline Ti_3SiC_2 MAX phase was prepared by a reaction sintering process using a spark-plasma-sintering (SPS) technique. In order to examine mechanical properties, nanoindentation tests were conducted around the formed kink bands. The kink boundary showed that the nanohardness linearly changed with the distance from the kink bands and showed higher values around the kink bands. Since the kink bands blocked the slip line caused by the nanoindentation, those become obstacles against the dislocation motion caused by the indentation deformation. This suggests that the kink bands would contribute to improving the mechanical properties of the Ti_3SiC_2 MAX phase.

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Microstructure, Deformation and Property of Wrought Magnesium Alloys

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Pure magnesium develops a strong basal texture after conventional processing of hot rolling or extrusion. Consequently, it exhibits anisotropic mechanical properties and is difficult to form at room temperature. Adding appropriate alloying elements can weaken the basal texture or even change it, but the improvement in formability and mechanical properties is still far from expectations. Over the past 20 years, considerable efforts have been made and significant progress has been made on wrought Mg alloys at the fundamental and technological levels. At the fundamental level, textures formed in sheets and extrusions of different alloy compositions and produced under different strain paths or thermomechanical processing conditions are relatively well established, with the assistance of the advanced characterization technique of electron backscatter diffraction. At the technological level, room temperature formability of sheet has been significantly improved, and tension-compression yield asymmetry of extrusion is also remarkably reduced or eliminated. This talk starts with a review of microstructure (texture and grain size) and deformation of polycrystalline pure Mg with different textures, grain sizes, and loading conditions. With this information as a base, texture, grain size and deformation of polycrystalline Mg-alloy sheets and extrusions produced under different processing conditions, are examined and compared. Remaining and emerging scientific and technology issues are then highlighted and discussed in the context of texture and grain size. The need for better-resolution diffraction and spectroscopy techniques will also be discussed in the relationship between texture change and grain boundary solute segregation.

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Tensile and compressive mechanical behaviour in fully-lamellar extruded $MgGd_2Zn_1$ alloy.

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Abstract

The tension and compression behaviour in the extruded ECO-$MgGd_2Zn_1$ (%at.) alloy with fully lamellar structure has been evaluated following the evolution of the internal strains during in-situ tension and compression tests. Before extrusion, a thermal treatment is carried out in the alloy to promote the formation a lamellar structure within magnesium grains. After extrusion at 350 ºC, the microstructure is characterised by a 14H-LPSO fibres elongated along extrusion direction, dynamic recrystallised grains of around 1 μm and coarse highly-oriented non-dynamic recrystallised grains. The 14H-LPSO fibers behave as a reinforcement. After yielding, internal strains of grain orientations favourable oriented for yielding exhibits a strong relaxation. Under compression, the activation of twinning is observed but lamellae within grain delay twinning propagations. The activation of <a> dislocation slip along the basal plane controls the beginning of the plastic deformation.

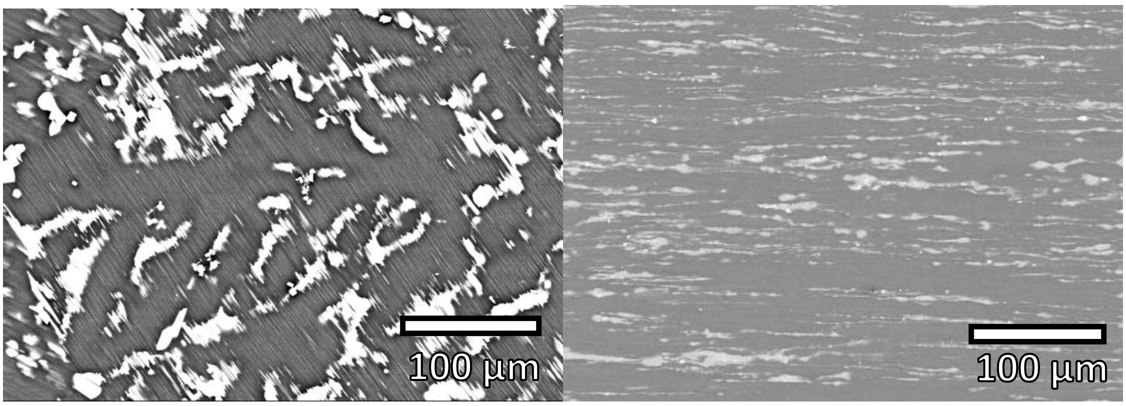

Figure 1. a) Microstructure of the ECO-$MgGd_2Zn_1$ (%at.) alloy after lamellae-formation thermal treatment and a) extruded at 350 ºC.

Hot deformation of Mg-Y-Zn alloy with different content of the LPSO phase studied by in-situ synchrotron radiation diffraction

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The compressive deformation behavior of the extruded Mg-Y-Zn magnesium alloys containing a low (~10%) and high (~85%) amount of long-period stacking ordered (LPSO) phase was studied by in-situ synchrotron radiation diffraction technique [1,2]. Tests were conducted at temperatures between room temperature and 350 °C. Detailed microstructure investigation was provided by scanning electron microscopy, particularly the backscattered electron imaging and electron backscatter diffraction technique. The as-extruded microstructures exhibit a recrystallized α-Mg phase with nearly random texture. The LPSO phase, identified as the 18R polytype, is represented by wavy lamellae elongated along the extrusion direction and has an intensive basal texture.

For the low LPSO content alloy, the results show that twinning lost its dominance and kinking of the LPSO phase became more pronounced with increasing deformation temperature. Neither cracks of the LPSO phase nor debonding at the interface between the LPSO phase and the Mg matrix were observed at temperatures above 200 °C. At 350 °C, the LPSO phase lost its strengthening effect and the dynamic recrystallization of the Mg matrix is dominant [1]. The alloy with high volume fraction of the LPSO phase exhibit a superior yield strength of 480 MPa, if it is compressed along the extrusion direction at room temperature [2]. With increasing deformation temperature, the yield strength is reduced by 15% at 200 °C and by 46% at 300 °C, respectively. At all tested temperatures, the basal slip is activated in the α-Mg matrix far below the yield strength. The macroscopic yielding of the alloy is controlled by the activation of deformation kinking in the LPSO phase. The synchrotron radiation diffraction data indicate the stress localization at kinks.

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Monitoring of strengthening of LPSO in αMg/LPSO dual phase alloy by hot-extrusion using *in situ* neutron diffraction during deformation

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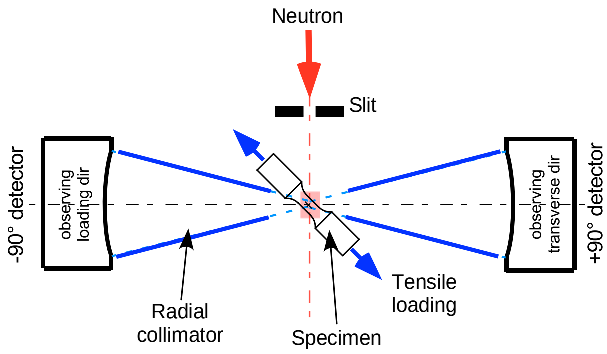
The strength of Mg-Zn-Y alloys containing LPSO was found to be enhanced by post mechanical processes such as hot-extrusion [1] or hot-rolling [2]. The increase in strength is considered due to the introduction of kink-bands in LPSO by the post mechanical processes.[1] The yield stress of $Mg_{89}Zn_4Y_7$ containing 86-vol% LPSO increased monotonically by hot-extrusion and with increasing the extrusion ratio (R), which was explained by the increased amount of kink bands and the texture development in LPSO [3]. The yield stress of $Mg_{97}Zn_1Y_2$ containing 25-vol% LPSO increased by hot-extrusion with the small R value, but it decreased as the R value increased due to the occurrence of dynamic recrystallization in αMg during hot-extrusion with the high R value.[4] These reports show that the effects of hot-extrusion to the microstructures and strengthening of αMg and LPSO were different, and the understanding of strengthening mechanism that was evaluated mainly from the macroscopic yielding behavior may be not enough without knowing the individual phase stresses.

Thus, in this study, *in situ* neutron diffraction during deformation was performed using three kinds of samples of $Mg_{97}Zn_1Y_2$ alloy: an as-casted sample (As-Cast), hot extruded samples with the R values of 5.0 (R=5.0) and 12.5 (R=12.5). The reason for varying the R value is to extend the kink amount in LPSO and change the microstructure of αMg in the starting materials before deformation. Fig. 1 shows the geometrical illustration of experimental setup for *in situ* neutron diffraction experiment during deformation and the tensile true stress-true strain curves of the three samples. Crystallographic evolutions of αMg and LPSO by hot-extrusion and deformation behavior of the individual phases were investigated, and the unique deformation mechanism by hot-extrusion in this alloy will be discussed based on the obtained results.



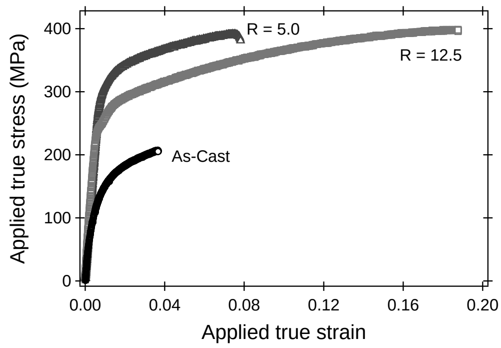


Fig. 1. Geometrical illustration of experimental setup for *in situ* neutron diffraction experiment during deformation and tensile true stress-true strain curves of the three samples..

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Kink strengthening of LPSO Mg-Zn-Y alloys after processing by high-pressure sliding (HPS)

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Formation of kink bands, which are induced in the long-periodic stacking ordered (LPSO) phase, have been attracted attention, because these kink bands play a role in preventing dislocation motion and result in enhancing the strength [1,2]. It was shown that kink bands formed in the LPSO phase during hot extrusion processing [3,4] and several other processings such as hot compression, high-pressure torsion (HPT) and caliber rolling [5,6]. However, it is an issue how to induce a high density of kink bands in the LPSO phase.

High-pressure sliding (HPS) is one of the severe plastic deformation (SPD) techniques and it is gaining attention since it is operated under high pressure to reduce fracture and cracking in less ductile materials [7]. Furthermore, it was found that the strain generated by the HPS process is proportional to the hardness in a rod sample [8]. Consequently, it can be possible to establish a relationship between the imposed strain and the strengthening effect.

In this study, the HPS process is applied for processing a Mg-Zn-Y alloy to induce kink bands in the LPSO phase. The formation of kink bands was quantitatively examined with respect to the equivalent strain and evaluated the strengthening effect via the measurement of microhardness.

The equivalent strain and hardness variations on the longitudinal cross-section were shown in Fig. 1 (c) and (d), respectively. It appears that the strain is more accumulated around the mid-height part on the cross-section and reaches higher values than those on the top and bottom sides of the cross-section. Accordingly, the hardness variation is similar to the strain distribution. Formation of kink bands is observed in the mid-height part of the HPS processed sample.

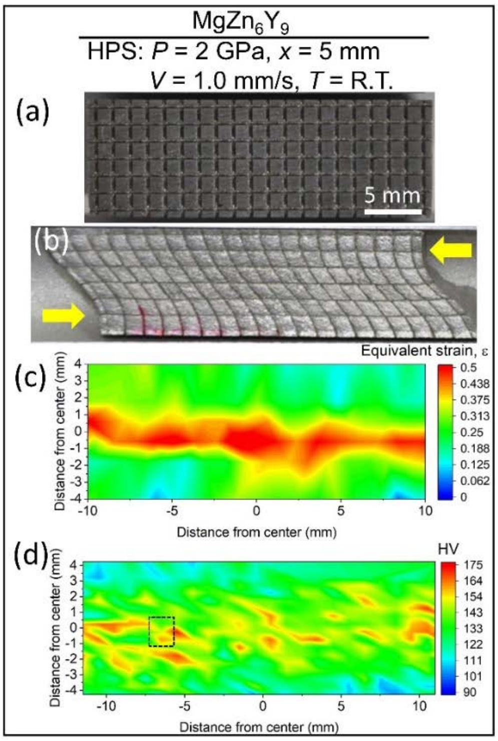

Fig. 1. Appearances of grids (a) before and (b) after sliding process, (c) distribution of equivalent strain and (d) hardness variation throughout longitudinal cross-section.

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Texture evolution and mechanical properties of Mg-9Y-6Zn alloy via vortex extrusion

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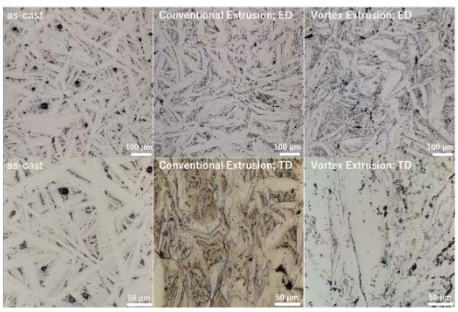
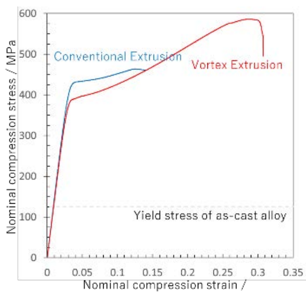
The authors systematically investigated the microstructure and mechanical properties of alloys containing LPSO phase by applying various wrought-processing to Mg-9Y-6Zn(at.%) alloy castings, which are almost a single phase of 18R-type LPSO phase[1]. As a result, kinked deformation could be introduced into the LPSO phase by all wrought-processing, and the hardness increased with increasing the introduced equivalent plastic strain. On the other hand, the amount of strengthening varied depending on the plastic working process when compared at the same amount of equivalent plastic strain, and it was found that introduction of shear strain from multiple directions was effective. These results indicate that combined plastic forming by extrusion and torsion is an effective means for strengthening mechanism by kink deformation.

In this work, the effect of vortex extrusion (VE) on the texture evolution and mechanical properties of Mg-9Y-6Zn alloy was investigated. The VE is a severe plastic deformation method that imposes high values of strain with simultaneous torsion and extrusion of a workpiece. While most of the kink bands in conventional extrusion (CE) are formed as a jig-zag into the LPSO phase whose a-axis is perpendicular to the ED direction, the kink bands in VE can be also formed as a wavy bent into the LPSO phase whose a-axis is nearly parallel in the extrusion direction. The mechanical properties of VEed sample is shown in Fig.2. The yield stress of VEed sample is lower than that of CEed one. On the other hand, the fracture elongation is superior. In my presentation on the day, I would like to discuss the relationships these mechanical properties and texture evolution via VE process.





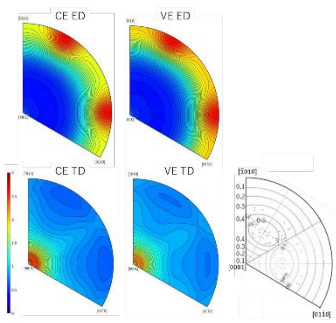


Fig. 1 Microstructures of as-cast, conventional and vortex extrusion from ED and TD direction (left)
Fig. 2 Nominal compression stress-strain curves of conventional and vortex extrusion (middle)
Fig. 3 Invers pole figures of conventional and vortex extrusion from ED and TD direction and the Schmid factor of basal slip form TD direction (right)

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~MEMO~

December 13 (Tue.)

4^{th} ~ 5^{th} session

Stretch-induced structural evolution of semicrystalline polymers: the effects of strain rate

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ABSTRACT

Strain rate is an important factor affecting the structural evolution of polymeric materials in both processing and service conditions. In this talk, we present our recent studies on deformation-induced structural evolution of semicrystalline polymers, including polyethylene, polypropylene, poly(1-butene) and PDMS rubber, under wide range of strain rates. Under high strain rate, hexagonal phase of polyethylene emerges, which normally appears under high pressure. The crystal transition from form II to form I of poly(1-butene) under quiescent and low strain rate conditions is changed when high strain rate is loaded, during which melting or disordering of the thermodynamic stable phase form I is observed. The non-equilibrium phase diagram of stretch-induced crystallization and structural transitions of PDMS rubber is also strongly depended on strain rate. It seems a general rule that high strain rate results in conformational defects in polymer crystals and consequently brings new phases or different kinetic pathways of structural transitions, which is consistent with the far from equilibrium nature under high strain condition.

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Deformation Behavior of High-strength Mille-feuille Structured PVDF

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We found that strengthening occurred in the mille-feuille structured crystalline polymers such as high density polyethylene (HDPE) and poly(vinylidene fluoride) (PVDF) consisting of hard crystalline lamellar layer and soft amorphous one by heat elongation [1]. In this report, to understand the strengthening mechanism of the mille-feuille structured crystalline polymers, we investigated the deformation behavior of PVDF by small-angle X-ray scattering (SAXS).

Anisotropic layer shaped SAXS pattern was observed at the meridian direction in the heat elongated PVDF (Figure 1). The results suggest the presence of mutually stacked layer structure of crystalline lamellar layer and amorphous one with periodic distance in which the longitudinal direction of lamellae is perpendicular to the elongated one. Thus, mille-feuille-like layer structure was obtained by heat elongation of the PVDF film.

Figure 2 shows the stress-strain curves of the unelongated and heat elongated PVDF. Here the stretching direction was parallel to the elongated direction and was perpendicular to the longitudinal direction of lamellae. The stress increased steeply with strain after the initial increase in the linear region without fall by yielding. After the linear increase of the stress up to 200 MPa, the increase of the stress became smaller. Owing to the continuous increase of the stress without fall, the stress at break of the elongated PVDF obtained by heat elongation at λ=300 % was about ten times larger than that of the unelongated one.

Long period of the mille-feuille structure was estimated by the intensity profile of Fig. 1. In Figure 3 is shown the change of long period of the elongated PVDF (λ=300%) with tensile strain during stretching. Here the long period of the stretched specimen was recovered to the unstretched one after releasing in the elastic region while the change remained in the plastic region. It was found that long period increased steeply with tensile strain in the elastic region and then the increase of the long period became smaller when the plastic deformation started to occur. Since the change of the SAXS pattern was slight, the result suggests that ripplocation occurs in the plastic region. The strengthening of the mille-feuille structured PVDF might be attributed to the toughness to cause the continuous increase of the stress during stretching by preventing the fracture of lamellae due to the chain orientation and ripplocation.

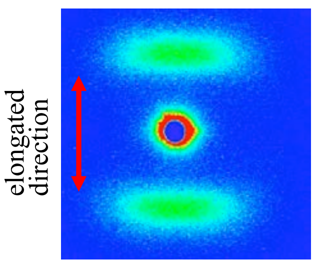

Fig. 1. SAXS pattern of the heat elongated PVDF (λ=300%).

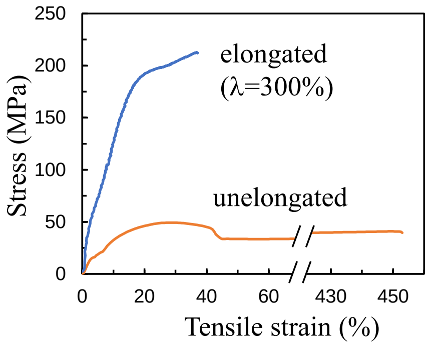

Fig. 2. Stress-strain curves of the unelongated and heat elongated PVDF.

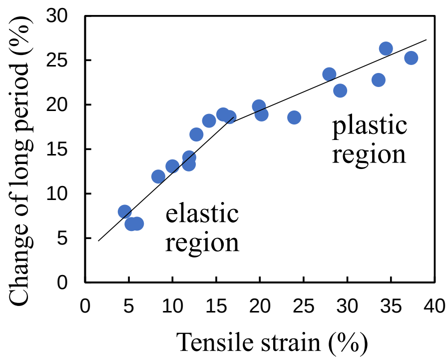

Fig. 3. Change of long period of the heat elongated PVDF (λ=300%) during stretching.

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Effect of Smectic Layer Deformation on Mechanical Properties of Glassy Liquid Crystal Polymer Fibers

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We determined that the stress response of a glassy smectic liquid crystal (LC) polymer fiber on tensile deformation was largely affected by the type of pre-deformations of the layers. The LC polymer used herein was a main-chain type BB-5(3-Me) polyester (Fig. 1), which forms a smectic layer structure at a spacing of d = 1.6 nm with alkyl chains segregated from biphenyl mesogens [1]. This polyester yielded three types of fibers different in smectic layer deformations. Fiber A with smectic layers stacked along the fiber axis was prepared by pulling the isotropic liquid. Stretching this fiber in the LC state at a rate of 5% min^{-1} up to a strain of 80% folded the smectic layers, producing fiber B with chevron-shaped layers. Further stretching fiber A up to a strain of 270% yielded fiber C with smectic layers divided.

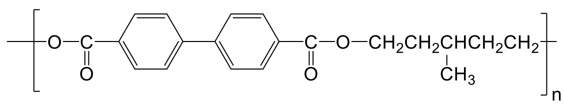

Fig. 1. Chemical structure of BB-5(3-Me) polyester.

These three types of fibers were stretched at a strain rate of 5% min^{-1} in the glassy state at 25 °C, and the stress (σ)–strain (ε) curves and X-ray diffraction patterns were simultaneously measured (Fig. 2). Fiber A exhibited an initial elastic modulus E = 0.33 GPa and the stress yielded at ε = 27% increasing σ up to 33 MPa. As the σ increased, the smectic layers increased the smectic layer spacing (d). Fiber B was similar to fiber A in the value of E, however, this fiber could be elongated up to ε more than 300%, maintaining σ = 23 MPa at ε in the range of 100–200% and increased σ again up to 25 MPa with further increasing ε to 300%. The smectic layers increased the inclined angles and decreased the d, involving the polymer chain glided along the chain axis. Fiber C had a greater E = 0.97 GPa and the σ yielded at ε = 32% with a maximum σ = 82 MPa. This yield stress was greater than that of fiber A. Fiber C increased the d at a similar rate to fiber A as the ε increased, however, fiber C markedly promoted layer dividing, differing from fiber A. This suggests that dividing smectic layers effectively enhances σ to improve the fiber strength.

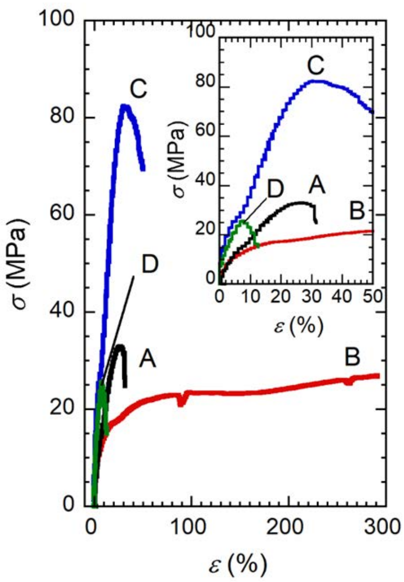

Fig. 2. σ–ε curves measured at 25 °C for fiber A (non-deformed smectic layers), B (folded layers), C (divided layers), and D samples. Fiber D samples were prepared by annealing fiber C samples at 110 °C for 12 h. The part at smaller ε is enlarged and shown in the inset.

The mechanical enhancement of the glassy smectic LC can be associated with layer division. Fiber D which was prepared by annealing fiber C in the LC state, had long smectic layers, like fiber A, and exhibited σ = 25 MPa and ε = 9% at the yield point. These yield stress and strain were more than three times smaller than that of fiber C. Similar mechanical strength enhancements by dividing lamellae have been found for the lamellae of semi-crystalline polyethylene [2] and a liquid crystal block copolymer [3].

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Preparation and Mechanical Properties Evaluation of Multilayer Film having Mille-feuille and Kink Structures

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A new material strengthening method has been discovered in which kinks are introduced into Long-Period-Stacking/Ordered (LPSO) structures with hard and soft layer by layer laminated structures such as mille-feuille to improve mechanical strength in the field of magnesium (Mg) alloys. Although the mechanism of kink structure in enhancing material properties has not been elucidated, the strengthening conditions as mille-feuille in the metals were expressed based on the experience. For enhancement, a continuous periodic structure of hard/soft layered structure is required, and kink introduction is essential for this structure. To establish the kink strengthening of this hard/soft layered structure (mille-feuille structure), in this study, we investigated the relationship between the morphological structure of kink and its mechanical behavior in polymers. To expand the kink strengthening method into the polymers field, we fabricated a polymer-based mille-feuille material with kink formation and kink strengthening by plastic deformation processing on polymers mille-feuille structure, referring to the experience-based strengthening.

In this study, polystyrene (PS, PS Japan, modulus of elasticity (GPa): 3.3) was used as the hard layer and styrene-ethylene-butylene-styrene copolymer (SEBS, modulus of elasticity (GPa): <1) as the soft layer. PS/SEBS (32 alternating layers of two types) multilayer films were produced using a multilayer co-extrusion machine (Fig. 1). In addition, super multilayer PS/SEBS film (320 alternating layers of two types) was produced by laminating 10 sheets of 32-layer film and applying heat press processing using a heat press machine. The TEM observation results of the produced mille-feuille structure are shown in Fig. 2. These images show that the increase in the number of layers (multilayering) has resulted in a decrease in the layer thickness per layer. In addition, the drawing process reduced the difference in layer thickness in each layer compared to that before drawing. The mille-feuille structure with a layer thickness so thin that the PS spheres in the microphase-separated structure are regularly aligned (about 300 nm per layer) was fabricated by the stretching process. Various kink formation processes were applied to these mille-feuille structures.

By heat treatment and cooling after stretching, a kink structure was formed in both the stretch (MD) and width (TD) directions in the thin PS layer. However, in PS layers with thicker layers (1 μm or more per layer), cracks were formed that broke without bending. This image suggests that a layer thickness of about 500 nm per layer is required for kink formation. However, these kink formations were limited to a portion of the film. Therefore, we attempted to control the shrinkage of the layer structure by applying heat treatment to the stretched sample and tried to form kinks in the entire film. The TEM image after heat treatment shows that shrinking the layer structure above the Tg of PS.

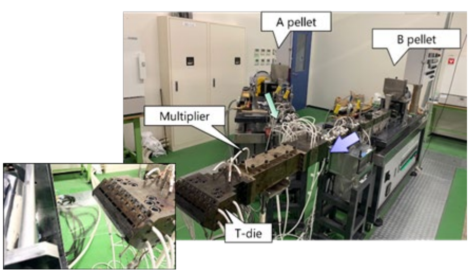

Figure 1 Multi-layer extrusion machine. Preparation of multilayer films of PS(A: hard)/SEBS(B: soft)

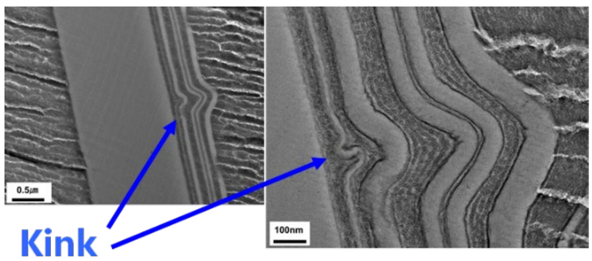

Figure 2 TEM observation after uniaxial stretching and relaxation for PS/SEBS

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Large-scale MD simulations of reinforced polyethylene crystals by pre-elongation

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A new strategy, "nanomosaification," for toughening semicrystalline polymers is proposed. Reinforcement of polyethylene (PE) via uniaxial pre-elongation before isothermal crystallization was reported [1]. Although such a reinforcement was also observed in deformation with rolling with side constraints and compression [2], we developed uniaxial deformation systems for comparison between experiments and molecular dynamics (MD) simulations [3]. We confirmed nano-crystal-domain downsizing in the reinforced PE through transmission electron microscopy, scattering experiments, and ultra-large-scale MD simulations as shown in Fig. 1 (upper). The MD simulations revealed the contributions of tie-chains to downsize nano-crystal-domains, suppress slippage among the nano-crystal-domains, and toughen the specimen. The deformation-enhanced crystallization of the crowded PE chains was different from the Lauritzen–Hoffman situation and was modeled using the orientation-controlled interconnected nodule model [3]. Figure 1 (lower) shows results of primitive path extraction [4,5] of amorphous chains (tie-chains). The MD simulations provided direct atomic-level insights and confirmed that the formation and working behavior of amorphous and crystal subchains depend on the magnitude of pre-elongation. Based on our virtual experiments with cutting tie-chains in MD simulations, contribution upon reinforcement by tie-chains was confirmed to be much larger than that only by the nano-crystal-domain downsizing [3]. Upon "nanomosaification," the process–structure–property relationships enable the tailoring of monomaterials and recyclability enhancement.

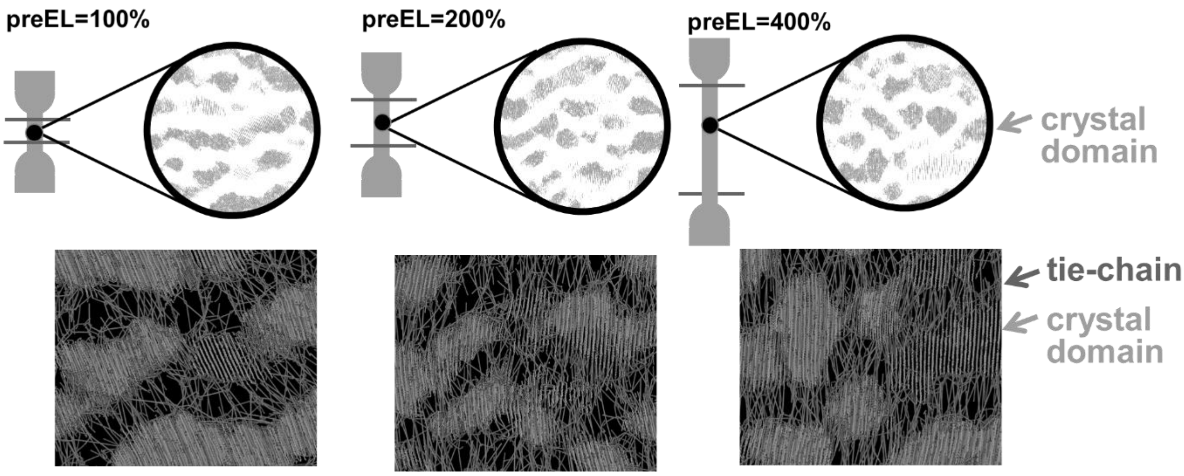

Fig. 1. Cartoon of "nanomosaificaion" in pre-elongate PE crystals (upper) and primitive path presentation of tie-chains (lower).

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Introduction of millefeuille-like α/β layered structure and investigation of its kink deformation behavior in β titanium alloys

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Recently, new type of strengthening mechanism, called 'kink strengthening', was found in Mg alloys with long-period stacking ordered (LPSO) structure [1]. This 'kink strengthening' was also found in other materials with layered structure (millefeuille-like structure), such as MAX phase ceramics [2]. In this study, to pursue possibility of kink strengthening in titanium alloys, millefeuille-like α/β layered structure was introduced in β titanium alloys [3], and compression tests were performed to form kink deformation in this layered structure.

β titanium alloy ingots with composition of Ti-12 mass%Mo and Ti-9Cr were prepared through cold crucible levitation melting (CCLM). CCLM ingots were forged and hot rolled to 6-mm-thickness plates. Coupons of around 10-mm width and 50-mm length were cut from the hot rolled plates, and then heat treated at 1173 K for 18 ks in Ar atmosphere followed by water quenching to obtain β single phase samples with large β grains. Heat treated coupons were cold rolled with the reduction of 5 %. After cold rolling, plate-like {332}<113> deformation twins, typical deformation twins in β titanium alloys, were introduced in the coupons. Cold rolled coupons were finally aged in α/β two phase region (973 K for Ti-12Mo and 923 K for Ti-9Cr). After aging, plate-like α phases were precipitated on the twin boundaries, and alternately stacked millefeuille-like α/β layered structure was obtained.

To introduce kink deformation, compression tests were performed at the temperature range of 473 K to 673 K. Specimens for compression tests were cut from aged coupons. Compression direction was parallel to the TD direction of cold rolling. The microstructure after compression tests was observed optical microscope and scanning electron microscope (SEM). Figure 1 shows the SEM microstructure of 50% compressed Ti-12Mo samples at 573 K. As can be seen in Fig.1, kink deformation of α/β layered structure was observed.

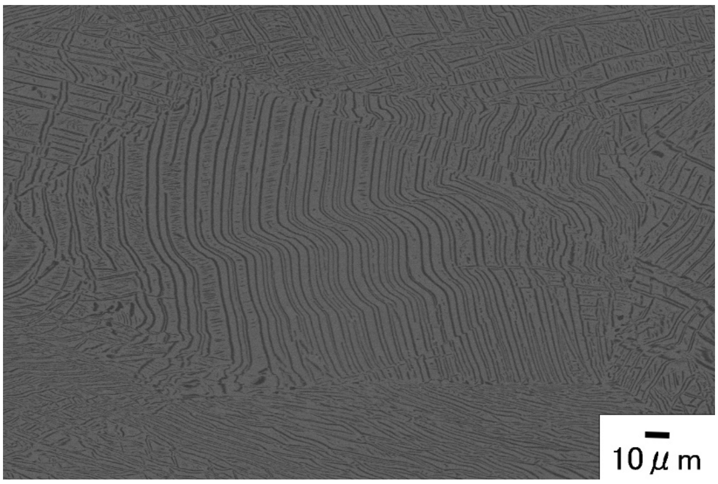

Fig. 1. SEM image of kink deformation in Ti-12Mo alloy after 50% compression at 573 K.

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Kink deformation and strengthening in an Al-30wt%Ag alloy with mille-feuille structure fabricated by aging precipitation and subsequent cold-rolling

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Materials having "mille-feuille structure" consisting of soft layers and hard layers is expected to exhibit kink strengthening. In this study, an Al-Ag alloy having late-like precipitates of Ag_2Al were focused to product the mille-feuille structure.

Al (purity of 99.99%) and Ag (purity of 99.9%) were melted and mixed, and Al-30wt%Ag alloy was obtained by casting. The casted ingots were homogenized at 500 °C for 100 h and deformed by cold roll with 10% of reduction. The deformed alloy was aged at 400°C for 100 h for precipitation of p late-like Ag_2Al. Subsequently, the aged alloy was deformed by cold rolling with 80% of reduction. Mille-feuille structure consisting of Al matrix and deformed Ag_2Al was observed in the cold-rolled sample. The deformed Ag_2Al precipitates were parallel to rolling direction (RD). Specimens for compression test cut from the obtained alloy. Compression test was carried out at ambient temperature. Compression direction was parallel to rolling direction. A stress-strain curve of the alloy having mille-feuille structure was shown in Fig.1. In the figure, photos of the specimen at various compress strains during the test were also shown. It was found that two kink bands were introduced during the compression test. At first a narrow kink band formed (arrow A) and width of the kink band increased. Then, the other kink band introduced from left bottom (arrow B) and crossed the first kink band. Compression stress dropped when a first kink band was introduced, subsequently increased gradually with growing in width of the kink band. Stress increased when second kink band formed and cross the first kink band. The increment of stress was 9.5MPa, which was regard as the amount of kink strengthening.

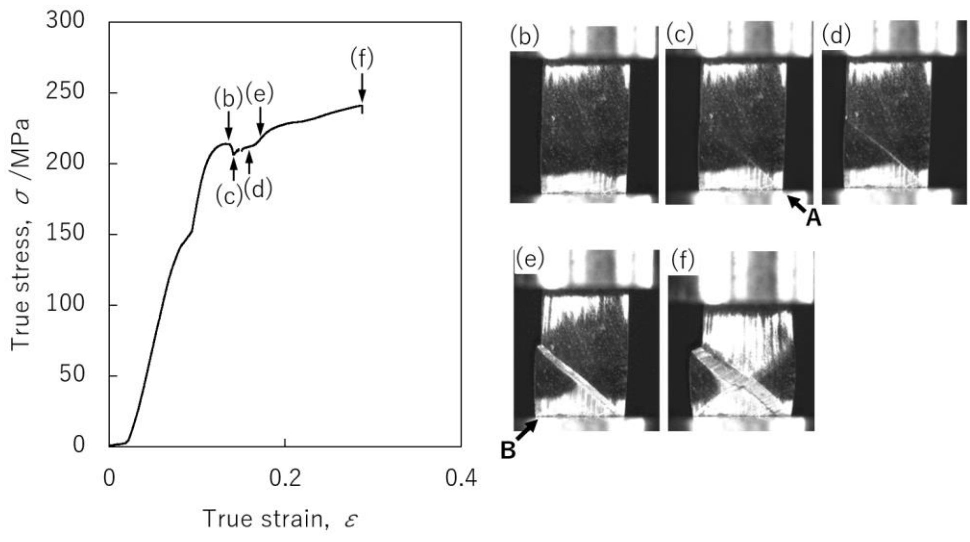

Fig. 1. (a) The stress-strain curve of the Al-30wt%Ag alloy with mille-feuille structure, and (b)-(f) the photos of specimen at various compression strains.

~MEMO~

Disclinations: from Theory to Practice

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We present up-to-date information on the properties of disclinations – defects of rotational type, in solids [1]. First, we discuss geometry and nomenclature for straight and circular disclinations including their main characteristic – Frank vector [2]. Second, we analyze analytical solutions of isotropic elasticity boundary-value problems for straight wedge disclinations [3]. The considered plane elasticity problems include those for wedge disclinations in uniform or two-phase cylinders, at a free surface of a half-space, and in a plate of finite thickness [3]. Three-dimensional problems under analysis deal with wedge disclinations in a bulk sphere or spherical layer or with the defects with the lines being normal to a free surface of a half-space or to surfaces of the plate [3].

Possible applications of the elasticity solutions for wedge disclinations are discussed [1-3]. We demonstrate that the disclination properties become a controlling factor when considering rotational plasticity in solids [2], grain boundaries and their junctions in conventional polycrystals and nanostructured materials [4], crack nucleation and initiation of ductile fracture [2], pentagonal rods and icosahedral micro- and nanoparticles [5], amorphous solids and glasses [2], domains, and twins in ferroelastic films adjusted to a bulk substrate [6], and defects in graphene and pseudo-graphenes [7,8].

Special attention is paid to the associated with disclinations development of kink bands in layered structures including LPSO alloys [9]. In this last case, the geometry and energetics of kinking is discussed in detail.

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Direct observations of kink microstructure in mille-feuille structured Mg alloys

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Materials with a mille-feuille structure (MFS), which is represented by alternative staking of soft/hard layers, are strengthened by introducing kink deformations. It is important to clarify the mechanism of kink deformation for designing materials that effectively exhibit kink strengthening, but the details have not yet been clarified. We have investigated the microstructures of kink-deformed MFS-type Mg alloys, including long-period stacking ordered (LPSO) phases, and confirmed that the crystal rotation at the kink boundary (KB) does not always satisfy the lattice correspondence and that the KB is steep at the atomic scale. Phenomenological models of kink deformation are understood as collective rearrangement of dislocations [1] or internal generation of dislocation pairs [2], but it is difficult to understand the formation process only by dislocation motion due to the microscopic characteristics of the KB. In this study, we investigated the kink-deformed microstructure of Mg alloys using electron microscopy to clarify their formation process and features that contribute to the strengthening of the materials.

Nominal compositions of the alloys used in this study were LPSO-type: $Mg_{97}Zn_1Y_2$ and MFS-type: $Mg_{97}Zn_1Gd_2$ (at. %), respectively. The alloys were hot extruded to introduce kink deformation with following conditions; extrusion temperature at 623 K, extrusion ratio of 10, and ram speed of 2.5 mm/s. Samples for TEM/STEM observations were prepared by mechanical polishing and ion milling.

Fig. 1 show HAADF-STEM images and schematic illustrations of defect structures on KBs formed in the LPSO phase and MFS, respectively. In both cases, KBs with crystal rotation of a few degrees consist of basal dislocations in the close-packed structure. In the case of LPSO phase, dislocations selectively locate in solute-enriched fcc layers, as shown in Fig. 1 (b), which leads to changing the stacking sequence from fcc to hcp as well as decreasing solute concentrations. As a result, nanoscale hcp-Mg regions are developed on the KB. In the case of MFS, dislocations form local fcc regions with solute enrichments (Fig. 1 (e)) that correspond to nanoscale solute-enriched staking faults (SESF). The observed structures suggest that kink formation involves structural changes including thermal activation processes (solute diffusion and dislocations climb), which contribute to the stability of KB formed at the high temperature processing.

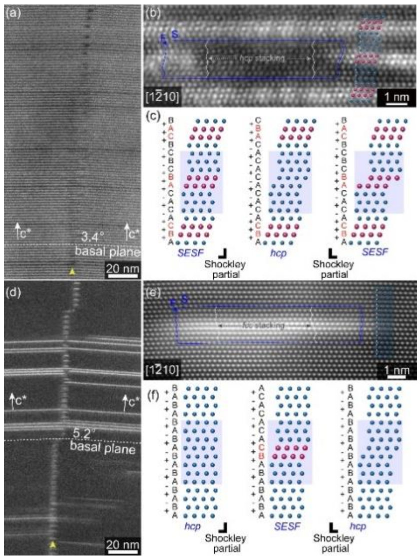

Fig. 1 STEM images of KB formed in Mg alloys (a-c) LPSO phase、(d-f) MFS

Fig. 2 shows a three-dimensional KB structure reconstructed by STEM tomography observations. In the case of MFS, basal dislocations on KB are always accompanied by solute segregations, so the bright plate-like contrasts in Fig. 2 (a) correspond to SESFs. Looking carefully, SESFs are separated along the [0001] direction inside of the sample, as shown in Fig. 2 (b), indicating formation of terminated dislocations on KB. In general, terminations of dislocations cause discontinuities of deformation, which should be compensated by defects such as disclinations [3]. The observed defect structure suggests activations of a structural relaxation different from the one by dislocation motions. In the presentation, we will discuss the details of the defect structure examined by STEM and the first-principles calculations.

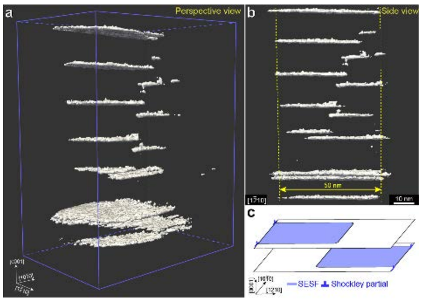

Fig. 2 (a-c) 3D reconstructed KB structure formed in MFS-type Mg alloy.

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Thermal stability of the microstructure of dilute Mg alloys prepared by rapid solidified ribbon-consolidation technique

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The thermal stability of the microstructure of dilute Mg alloys prepared by the rapidly solidified ribbon-consolidation (RSRC) technique has been investigated with respect to alloy compositions. In the as solidified state, the RSRC material has a very fine grain structure with an average grain size about 1 μm. The random orientation of fine grains results in a weak basal texture with a more pronounced intensity at the (10-10) pole. Moreover, the Mg-Zn-RE (rare earth) alloys are characterized by dispersive Zn- and RE-rich stacking faults formed in basal planes. These microstructure features lead to enhanced mechanical properties, particularly, a superior yield strength of 360 MPa and moderate elongation of 18%, even in Mg-Zn-RE alloys with low amount of alloying elements (up to 2 at.%).

In order to reveal the thermal stability of such microstructure, isothermal annealing in a range of 300 °C - 500 °C was applied. Scanning electron microscopy (SEM), including backscatter electron images (BSE) and electron backscatter diffraction (EBSD) techniques, as well as transmission electron microscopy (TEM) have been used to analyze the microstructure changes. Furthermore, X-ray measurements were performed for reliable texture analysis. The microstructure was found to be stable with increasing annealing temperature up to 400 °C. With further temperature increase, the growth of the grain size and changes in the texture of the alloy, particularly, redistribution of the intensity at the (10-10) pole, has been observed, which can be related to the recrystallization process. The order of dispersion of the solute-segregated stacking faults was found to be rather independent of the thermal treatment. In addition, the microstructure development was correlated with the results of the microhardness measurements.

Analysis of Kink Formation Behavior in Al/Al_2Cu Mille-feuille Structured Alloys

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Directionally solidified Al/Al_2Cu eutectic alloys have a lamellar structure consisting of a soft Al phase and a hard Al_2Cu phases. It has recently been reported that the introduction of micro kink-bands in the Al/Al_2Cu eutectic alloys improves their strength [1]. It has been suggested that the strengthening mechanism is affected by the size and spatial distribution of the introduced kink-bands. The introduction of multiple micro kink-bands is necessary to increase the strength, but the mechanism for the formation of such micro kink-bands is not clear. In this study, in-situ surface observations of Al/Al_2Cu eutectic alloys during compression tests were conducted to understand the mechanism of kink band formation. To quantitatively analyze the microstructural changes before and after the formation of kink bands, image analysis using the Radon transform was performed on the optical micrographs. This technique allowed us to quatitatively evaluate the microstructural changes over a large area (several mm^2) with submicron spatial resolution and sub-degree rotational resolution without SEM-EBSD analysis. The results showed that multiple micro kink-bands occurred simultaneously in a single lamellar colony and that they were aligned in the same orientation. The observed kink-bands are shown in Fig 1 (a). In order to formulate the relationship between microstructure and kink band formation, we proposed a model to predict the simultaneous formation behavior of kink bands (number of kink bands or kink bands spacing), as shown in Fig. 1 (b). It is based on a ripplocation model [2]. Comparison with experimental results of kink-bands spacing showed the validity of the proposed model.

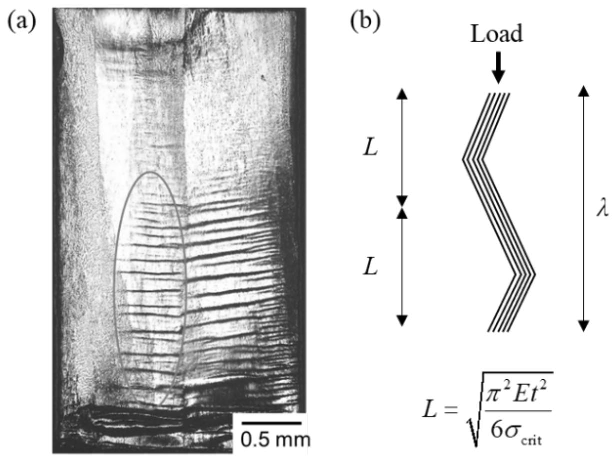

Fig. 1. (a) In-situ observation of micro kink-bands in Al/Al_2Cu eutectic alloy, (b) a model of multiple kink-bands formation based on ripplocation theory.

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Understanding of kink-band formation by means of a rate-independent model obtained by homogenization of mille-feuille structure

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Structured materials, such as metallic alloys with atomic-scale layers, show peculiar deformation patterns, which may have significant implications on material properties. In this presentation, we discuss one possible approach to modeling and understanding of this kind of pattern formation through the so-called rate-independent evolution in the variational setting of finite-strain elasto-plasticity [3].

We start with a mathematical analysis of the macroscopic behavior of mille-feuille structured materials, which is composed by alternating rigid and soft layers. This is done within the mathematical framework of homogenization via Gamma-convergence [1]. Next, we build a mathematical model of an evolutionary rate-independent system, which reflects the theoretically obtained insights, and study its mathematical properties. Finally, we perform numerical simulations of this mathematical model and show that it yields results that are in a good agreement with experimental measurements [2].

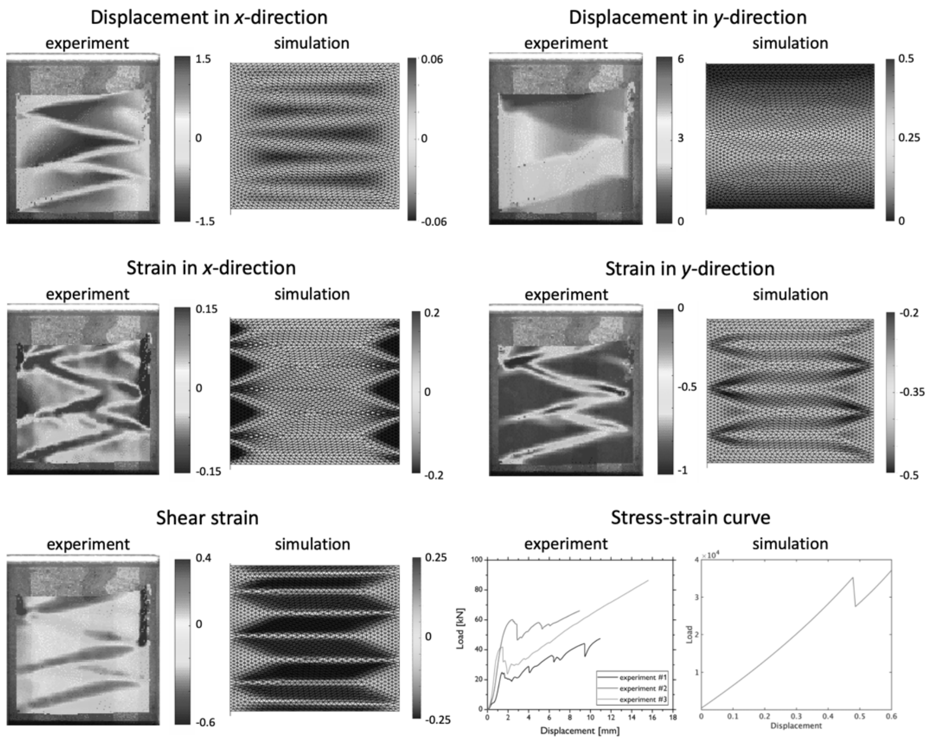

Fig. 1. Comparison of numerical simulations and experiment.

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Modeling and Numerical Analysis of Kink Deformation based on Geometrical Elasto-Plasticity Theory

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The long-period stacking ordered (LPSO) has attracted attention due to its excellent mechanical properties originating from the kink deformation. This study aims to conduct modeling and numerical analysis of kink deformation on a three-dimensional continuous medium based on the theory of geometric elasto-plasticity [1-3]. Here, we construct the ortho kink and ridge kink models with several dislocation configurations. We also construct these two models under the influence of shear deformation. Referring to the geometrical framework, the kinematics are represented as the reference $\mathcal{R}$, intermediate $\mathcal{B}$, and current $\mathcal{S}$ configurations. The total deformation is expressed by the multiplicative decomposition of the deformation gradient; $F = F_e \cdot F_p$. For a given dislocation density, we calculate the plastic deformation gradient F_p by solving the weak form of the Cartan first structure equation so that the intermediate configuration is obtained. The elastic deformation gradient F_e is obtained by minimizing the strain energy functional. Then, we solve the variational problem numerically using the finite element method. The ortho kink is modeled by a pair of kink interfaces containing an array of edge dislocations and the ridge kink is modeled by a combination of vertical and inclined kink interfaces.

The present analysis showed that the deformation agrees qualitatively with the experimental results. In the ortho kink model, the deformation exhibits a sharp inflection at the kink interface where the edge dislocation array is placed. While the ridge kink model shows sharp extrusions on the top and bottom surfaces of the domain where the ridge kink structure is placed. Note that, the deformation is highly localized around the kink structure. We also investigated the internal stress distribution for these two models. In the ortho kink model, when the kink interface penetrates and reaches the free surface, stress concentration could not be confirmed. However, when the kink interface does not penetrate the stress distribution appears at the tip of the kink interface. This is believed due to the presence of a disclination. On the other hand, the ridge kink model shows a low-stress field because at the tip of the ridge kink structure the disclination cancels each other out. The last thing we need to address is strain energy. We reported that the strain energy is higher after the shear deformation is applied. Here, we would like to clarify the effect of the kink interface length for these two models on the deformation and the stress field. Then, we will discuss the strengthening mechanism due to the kink structure.

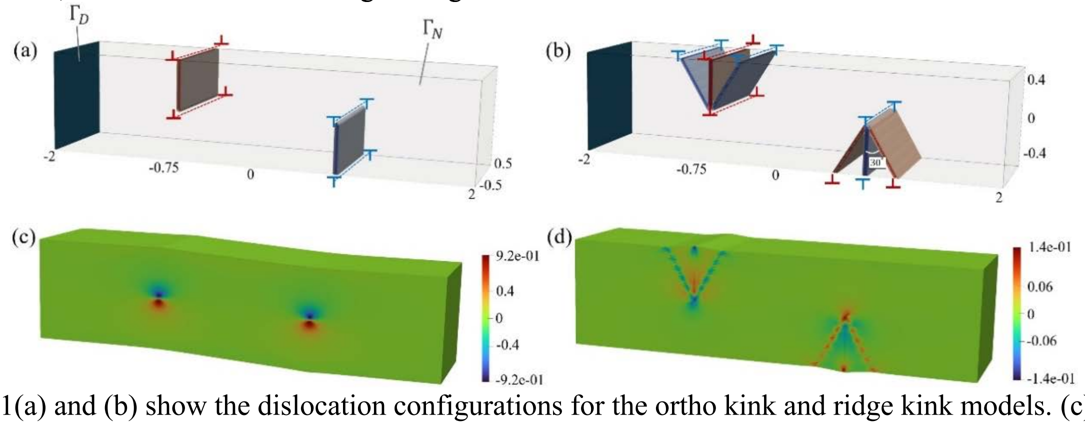

Fig. 1(a) and (b) show the dislocation configurations for the ortho kink and ridge kink models. (c) and (d) show stress components σ_{23} for the ortho kink and ridge kink models, respectively.

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December 14 (Wed.)

6th ~ 7th session

Crystal Plasticity-Based Prediction of Forming Limit Diagrams: Application to Magnesium Alloys

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There has been much study focused on developing an improved understanding of the limited cold formability and, conversely, excellent warm formability of Mg alloys. Countless attempts have been made to improve the cold ductility of Mg via alloying, texture engineering, and microstructure control (e.g., grain size reduction). Many of these efforts have been accompanied by polycrystal plasticity modeling to provide mechanistic understanding, and this has often highlighted the potentially critical role of particular dislocation slip or deformation twinning mechanisms.

In the present research, the potentially important role of diffusion-controlled mechanisms, namely ***dislocation climb*** is reconsidered. The present authors recently reported that a new crystal plasticity model (VPSC-CLIMB), which explicitly accounts for the kinematics of dislocation climb, enables a more accurate description of strain anisotropy and texture evolution within the strain rate and temperature regime in which the ductility of Mg alloys can be excellent. [1] Perhaps more surprisingly, the results suggest that the climb of basal dislocations with <a>-type Burgers vectors accommodates a significant fraction of the strain under these conditions. The constitutive parameters employed in the modeling were obtained by a genetic algorithm (GA) based optimization of an objective function which involved minimization of the residual difference between measured and predicted crystallographic textures.

In the present research, this VPSC-CLIMB model is integrated with the Marciniak–Kuczynski (M–K) approach for forming limit curve (FLC) prediction. The approach allows for the incorporation of crystallographic texture-induced anisotropy and the evolution of the same during the deformation. Previous attempts at such polycrystal plasticity-informed FLC prediction occurred at a time when few experimental assessments had been made. [2] Since that time, the FLCs of numerous Mg alloys with various textures and microstructures have been assessed. [e.g., 3] These assessments include measurements of the crystallographic texture prior to and after forming along drawing-, plane strain-, and stretching-type strain paths. These new data provide a more critical validation of the polycrystal model-based FLC predictions and further highlight the importance of dislocation-climb accommodated plasticity under conditions when Mg alloys are considered formable. Finally, the modeling effort provides possible explanations for some of the rather strange behaviors exhibited by some Mg alloys, such as lower formability along the stretching direction as compared to that observed along the plane strain (FLC0) condition.

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Influence of electrochemical and geometrical heterogeneities on corrosion behaviour of LPSO phase-containing Mg-Y-Zn alloys

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Magnesium alloys have attracted considerable attention as promising for light-weighting applications. In addition, Mg alloys are suitable for metallic implants such as stents and bone screws that require bio-compatibility and bio-absorbability. However, commercially available Mg alloys manufactured by conventional ingot metallurgy have strength, ductility, and corrosion resistance inferior to commercial Al alloys. Thus, the applications of Mg alloys can be further extended by the development of Mg alloys with better mechanical properties and better corrosion resistance.

Excellent mechanical properties have been provided by the development of the high-strength Mg-Zn-Y alloys with long-period stacking ordered (LPSO) phases[1-3]. These LPSO-typed Mg-Y-Zn alloys are promising, novel, high-strength wrought alloys. The multimodal microstructure of the extruded LPSO-type Mg-Y-Zn alloys comprises three distinct regions: a dynamically recrystallized (DRXed) α-Mg grain region with random orientation; a worked α-Mg grain region with a strong fiber-texture of <$10\bar{1}0$>//extrusion direction (ED), and a worked LPSO phase grain region with the strong fiber texture. The fine DRXed α-Mg grains contribute toward the improvement in the ductility of the alloy, whereas the coarse worked α-Mg and LPSO grains contribute toward the mechanical strength of the alloy[4]. The LPSO phase provides strengthening because of its unique anisotropic plastic deformation behavior which originates from its unique long-period/chemical ordered structure.

The corrosion performance of these LPSO-typed Mg alloys is mainly determined by two factors. The LPSO phase increases the corrosion rate of the alloy, by micro-galvanic acceleration for the corrosion of the α-Mg matrix, because the 18*R*-structured LPSO phase contains high concentrations of Y and Zn. In addition, corrosion is induced by the variations in crystal orientation. In this study, the influence of the dispersed LPSO phase and the multimodal microstructure on the corrosion behavior of the α-Mg/LPSO two-phase wrought alloys has been clarified.

The extruded Mg-Y-Zn alloy showed orientation dependent corrosion behavior, the longitudinal section exhibited a higher corrosion rate than the transverse section[5]. The orientation dependence was attributed to the varying in-plane atomic densities of the crystallographic planes on the longitudinal and transverse cross sections. The LPSO phase was the cathode and galvanic corrosion occurred in the adjacent α-Mg matrix. In this study, we have also attempt to improve corrosion resistance of the LPSO-typed Mg alloys. The Al alloying into the Mg-Y-Zn alloy facilitated the formation of a protective surface film, which decreased the corrosion rate in spite of geometrical and electrochemical heterogeneities.

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Effects of Additional Deformation and Heat Treatment on Kink Bands on the surface of LPSO-type $Mg_{85}Y_9Zn_6$ Directionally Solidified Alloy Thin Plates

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Mg-TM-RE (TM: transition metals, RE: rare earth element) based alloys containing the long period stacking ordered (LPSO) phase, a type of mille-feuille structure, have been investigated by many researchers for their high mechanical properties [1] and unique crystalline structures. LPSO-type magnesium alloys significantly increase their strength by plastic deformation and this phenomenon is explained by the formation of kink bands, which act as the strong obstacles to the primary slip of dislocations (basal ***a***-dislocation slip) [2]. On the other hand, the response of kink bands introduced into a material to subsequent plastic deformation remains unresolved and requires experimental investigation. Furthermore, effects of heat treatments on kink strengthening are not well understood. In this study, effects of additional plastic strain on the character of kink band and heat-treatment on the kink strengthening were investigated in LPSO-$Mg_{85}Y_9Zn_6$ directionally solidified (DS) alloy.

The material used in this investigation is a DS LPSO type $Mg_{85}Y_9Zn_6$ alloy. Thin specimens were cut from parallel to the solidification direction and adjusted to a thickness of approximately 100~250 μm. The thin specimen was then placed between the indium plate and the round bar and bending strain was applied by pressing the thin specimen against the round bar with the indium plate. Many wedge-type kink bands with undulations were observed on the surface of compression-side after the bending test. After observing these kink bands, the bending direction was reversed (2nd bending) for the same sample and the observation was carried out again. For a part of specimens, heat treatment was applied before the 2nd bending test. Microstructures were observed by using transmission electron microscope (TEM) and analyzed by electron backscatter diffraction pattern (EBSD) measurements in a field-emission type scanning electron microscope (FE-SEM).

From optical microscopic observations, most of kink bands of several tens of microns or more did not expand/shrink after additional bending deformation at room temperature. On the other hand, the degree of surface undulation caused by the wedge-type kink bands tended to decrease after the 2nd bending test. EBSD analysis revealed that the rotation angle of a kink boundary decreased compared to that after the 1st bending test. Furthermore, when further plastic deformation was applied after kink band formation, the strain tended to concentrate near the kink band. Investigation of the Vickers hardness of the specimens after bending deformation showed higher values around the kink band than in the matrix. The Vickers hardness around the kink band increased significantly after heat treatment at 500-650 K. However, after 2nd bending test, the hardness around kink bands increased only slightly and/or decreased. It was suggested that Vickers hardness around the kink band tends to depend on the rotation angle of its boundary.

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Acknowledgments

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Microstructure and Deformation Behavior of BCC-V/MAX two-phase alloys

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Introduction

Max phases have attracted attentions because it shows excellent properties such as high strength, high melting point and low density [1]. The present authors have conducted a study to obtain a new class of mille-feuille materials by attaining lamellar structure composed of the BCC-V based materials and Max phase V_2AlC. It was already found that BCC-V matrix alleviate the delamination tendency of MAX phase during plastic deformation even at relatively lower temperatures. In this study effect of various quaternary elements on the stabilization of lamellar structures is investigated, and also the deformation behavior is studied.

Materials and Methods

Based on the V-Al-C ternary phase diagram [2], alloy composition with and without quaternary additives such as Cr, Ti, Mo and W were determined and ingots were prepared using an Ar-arc melting machine. A part of ingots sealed in evacuated silica tubes was subjected to a heat-treatment at 1473 K for 1 week. Microstructure observation using FE-SEM and WDS analysis for determining the composition of each phase was conducted, and the crystallographic orientation relationship between BCC-V and MAX phase was investigated using EBSD. Compression tests are also conducted to understand its deformation behavior and kink formation tendency.

Results and Discussion

It was confirmed that the alloys with and without the heat-treatment have two-phase microstructure, however, plate-like V_2AlC-MAX phase in as-cast ternary and some quaternary alloys spheroidized during the heat-treatment. The MAX phase in alloys with W keeps its plate-like shape even after the heat-treatment, resulting in fine mille-feuille microstructure. The tendency can be explained in terms of the solubility (partitioning) ratio of quaternary elements in BCC V and Max phases, which control the lattice mismatch through the effect of quaternary elements on lattice constant ratio between BCC and MAX phases. Kink formation was also found in the sample during the compression tests.

Acknowledgement

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Observation of as-cast and extruded $Mg_{97}Zn_1Y_2$ alloys by neutron diffraction and thermal dilatometer measurement

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From the viewpoint of acquiring the basic properties of the two-phase Mg-based LPSO alloy, typically $Mg_{97}Zn_1Y_2$ [1], the thermal response of the as-cast alloy, and the extruded alloy with an extrusion ratio of 10, which is introduced kink structure, was analyzed by using neutron diffraction and thermal dilatometer in order to investigate the effect of kink structure on the thermal properties. In thermal expansion measurements to observe macroscopic changes of the alloy, the Mg-phase of the extruded alloy is less than that of pure-Mg reported previously. This behavior of the Mg phase in the extruded alloy seems to be due to the introduction of kinks of 18R LPSO phase. Fig. 1(a) shows the temperature dependence of $(10\bar{1}0)$ and (0002) in the atomistic level of α-Mg phase in the as cast and extruded $Mg_{97}Zn_1Y_2$ alloys with downward convex quadratic fitting functions. From the figure, the change of the extruded alloy is more moderate. This is consistent with macroscopic thermal dilatometer result. $Mg_{97}Zn_1Y_2$ alloy consists of volume fractions of 75% α-Mg phase and 25% 18R LPSO phase. Because of this less volume fraction of LPSO phase, the detectable LPSO diffraction peaks are quite limited. We used the $(4\bar{2}\bar{2}10)$ and $(4\bar{2}\bar{2}8)$ peaks of 18R LPSO phase [2] for the analysis in this study. Fig.1(b) shows the temperature dependence of d-spacing of $(4\bar{2}\bar{2}10)$ plane of 18R LPSO phase in the $Mg_{97}Zn_1Y_2$ alloy. In the case of extruded alloy, the temperature dependence of the $(4\bar{2}\bar{2}10)$ plane of the LPSO phase in the $Mg_{97}Zn_1Y_2$ alloy is suppressed at higher temperature.

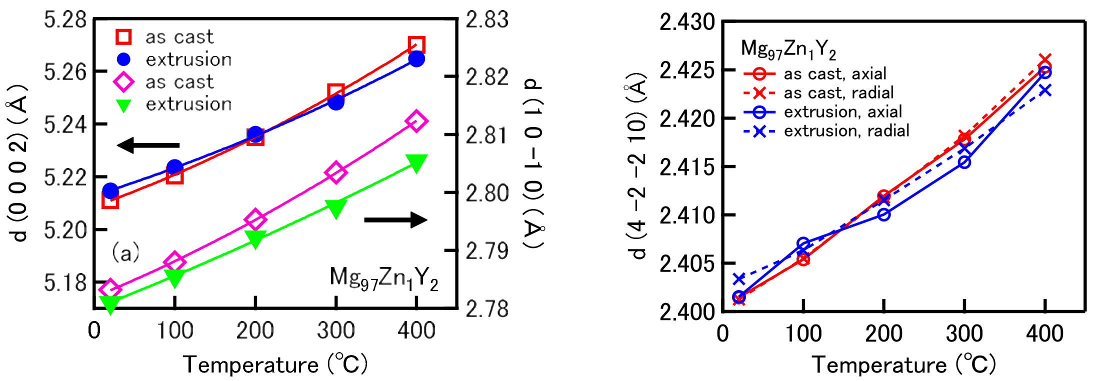

Fig. 1. (a) the temperature dependence of d-spacing of $(10\bar{1}0)$ and (0002) planes in the atomistic level of α-Mg phase in the $Mg_{97}Zn_1Y_2$ alloy with quadratic fitting functions. (b) the temperature dependence of d-spacing of $(4\bar{2}\bar{2}10)$plane of LPSO phase in the $Mg_{97}Zn_1Y_2$ alloy.

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~MEMO~

Synergetic kinking and twinning in an Mg-Zn-Y-Zr alloy with intragranular LPSO structures

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Deformation kink is one of the important strengthening mechanisms for the magnesium (Mg) alloys containing long-period stacking ordered (LPSO) phase [1], while deformation twin is generally suppressed by the thick LPSO structure. The synergistic effect between the deformation kink and the twin needs further understanding.

To optimize the mechanical properties of the Mg alloy with LPSO structures, we simultaneously introduced kink and twin in the Mg-Zn-Y-Zr alloy featuring the intragranular LPSO phase and free grain boundary LPSO phase by homogenization [2]. We unraveled the corresponding strengthening and toughening mechanisms through transmission electron microscopy characterization and theoretical analysis. The high strength and good plasticity of the homogenized alloy benefit from the synergistic deformation mechanism of multiple kinking and twining in the grains. And the activation of kinking and twinning depends on the thickness of LPSO lamellae and their relative spacing. These results may shed light on optimizing the design of Mg alloys regulating the microstructure of LPSO phases.

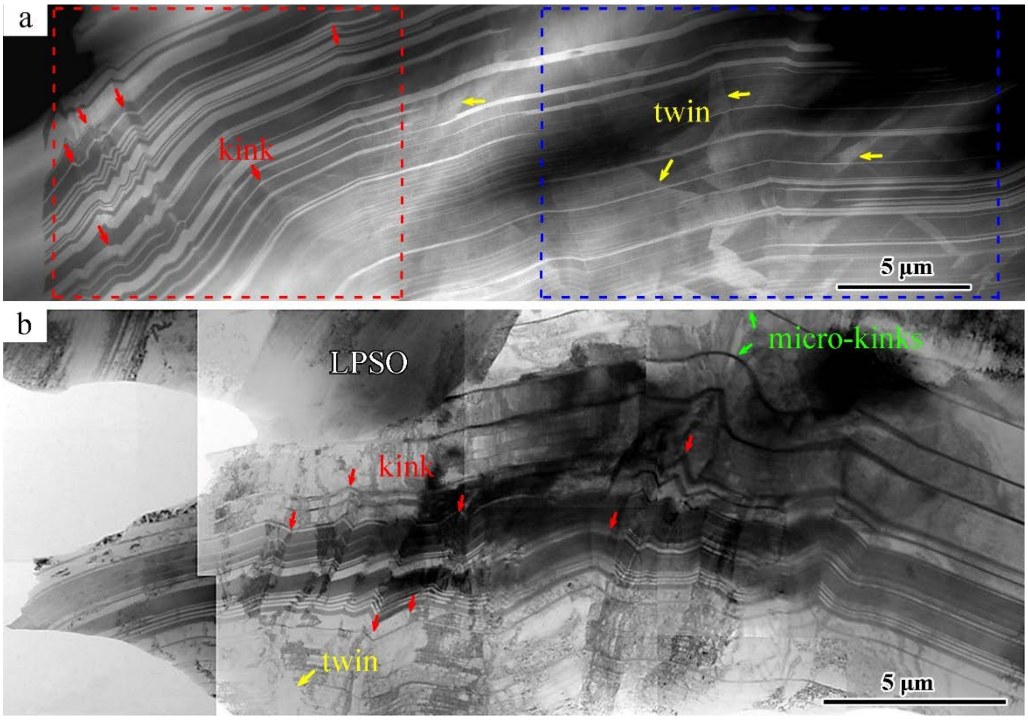

Fig. 1. (a) STEM and (b) TEM images of two grains in the homogenized sample after compressive deformation. One can find that kinks, micro-kinks, and twins, as shown by the red, green, and yellow arrows, respectively, coexist in the grains in the homogenized alloys.

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Kink-band formation in Mg/LPSO two-phase alloys

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The development of lightweight high-strength alloys has been gaining attentions because of increasing awareness of reduction in CO_2 emissions to mitigate global warming problems. Mg alloys have the lowest density among all structural materials, and they have been expected to solve the environmental issues. However, the strength of the Mg alloys must be improved for practical applications [1, 2]. Recently, it was reported that the superior mechanical properties were achieved in the Mg-Zn-Y ternary alloys with long-period stacking ordered (LPSO) phase. Although the strengthening mechanism of the alloys is considered to be related to the strong plastic anisotropy of the LPSO phase, the detailed mechanism has not fully clarified yet. In the present study, to investigate the controlling factors of the mechanical properties of Mg/LPSO two-phase alloys from a basic point of view, the influence of the volume fraction of the LPSO phase on the mechanical properties of Mg/LPSO two phase alloys was examined with directionally solidified (DS) alloys. Mg/LPSO single-phase and two-phase alloys of $Mg_{85}Zn_6Y_9$, $Mg_{89}Zn_4Y_7$, $Mg_{92}Zn_3Y_5$, $Mg_{94}Zn_2Y_4$, and $Mg_{97}Zn_1Y_2$ (at%), were prepared by induction melting followed by the directional solidification with Bridgman technique in an Ar gas atmosphere at a growth rate of 10 mm/h. The volume fractions of the alloys were measured to be 100, 86, 61, 39, 26 vol.%, respectively. The microstructures were observed by optical microscope and scanning electron microscopy (SEM), and compression tests were conducted at an initial strain rate of 1.67×10^{-4} s^{-1} and at room temperature, 200, 300 and 400 ^{o}C. In the compression tests, the loading axes were set at the direction parallel to the growth direction, and the direction inclined at 45^{o} to the growth direction in the DS process.

From the microstructure observation, in both the LPSO single-phase alloy and the Mg/LPSO two-phase alloys, it was found that the platelet LPSO phase grains and the basal plane of the LPSO phase were well-aligned parallel to the growth direction. Although the LPSO phase is basically considered as an effective strengthening phase in Mg alloys when stress is applied parallel to the growth direction, the alloys exhibited a nonmonotonic increase with an increase in the volume fraction of the LPSO phase. The highest strength was obtained in the Mg/LPSO two-phase alloys containing 61-86 vol.% of the LPSO phase. From the observation results of the deformation microstructure, it was clarified that this anomalous variation in the yield stress was due to the change in the formation stress of kink bands, which varied with the thickness of the LPSO-phase grains. Furthermore, the coexistence of Mg phase in the two-phase alloys induced the homogeneous formation of kink bands, whose boundaries can act as effective obstacles against further motion of the basal dislocation. This is referred to as the "kink-band strengthening" [3]. Activation of the kink-band strengthening was evaluated by specially-designed compression tests, i.e., "double compression tests". The double compression tests are composed of two compression tests; specimens were firstly compressed at 0^{o} to the growth direction to induce kink bands, and secondly, the specimens were compressed at 45^{o}.The yield stress of all the single-phase and two-phase alloys increased from the conventional compression tests loaded at 45^{o} to the growth direction. The results demonstrated that microstructural control is significant in the Mg/LPSO two-phase alloys to optimize mechanical properties.

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Crystal plasticity analysis of kink band formation in Mille-feuille structure

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Kink banding is a type of localized deformation modes where large crystal rotation within the band region is accompanied [1]. In several metallic materials, possible contributions of formation of kink bands to strengthening is suggested [2-4]. Magnesium (Mg) alloys containing Long-Period Stacking Order (LPSO) phase show significant improvement of mechanical properties owing to metal forming processes where a number of kink bands are formed [2]. Al-Cu eutectic alloys with fine kink bands also show high strength at room temperature strength [3, 4]. Especially, the latter case indicates possible alloy design where frequency of kink band formation and strength are deliberately controlled. To achieve such alloy design, it is necessary to understand underlying mechanism of kink band formation in eutectic alloys consisted of hard and soft lamellar phases. In this study, we investigate active deformation mode in Al-Cu eutectic alloys during kink banding under compressive loading via numerical calculations by crystal plasticity finite element method [5, 6]. In order to clarify the effective parameters on kink band formation, systematic parameter study was performed by changing critical resolved shear stresses in each phase, volume fraction of each phase, and distribution of defects. Figure 1 shows a typical calculated result where kink band is formed during compressive loading. The distribution of equivalent strain indicates that larger plastic strain is accumulated in softer Al phase while both phases show significant bending regardless of much smaller amount of equivalent strain in Al_2Cu phase compared with Al phase.

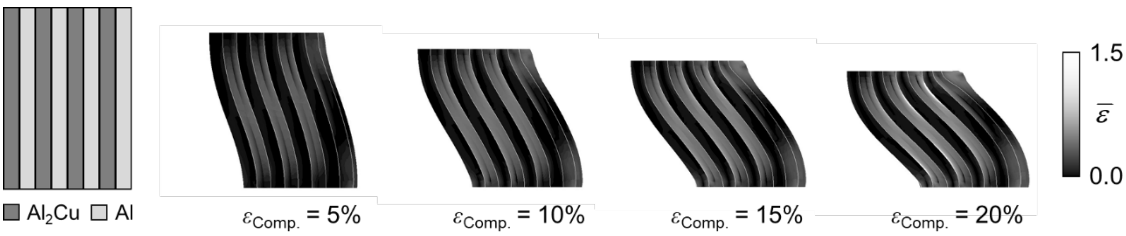

Fig. 1. Calculated result of kink band formation in Al-Cu eutectic alloy model.

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On Scale-Free Nature of Kink Formation/Strengthening based on FTMP

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Scale-free relationships, together with the associated specific return maps, have been experimentally reported for AE characteristics of deforming LPSO-Mg DS sample both before and after kink formation stages [1], although their roles and significance have not been disclosed hitherto. FTMP (Field Theory of Multiscale Plasticity) [2,3], on the other hand has been successfully applied to reproduce the kink formation processes when combined with crystal plasticity-based finite element (CP-FE) simulation [4,5]. This study makes a primary series of attempts to clarify the above by means of FTMP-based CP-FE simulations assuming *a priori* the experimentally measured AE characteristics, as shown in Fig.1(left), where the interaction field formalism [6] and the flow-evolutionary law in FTMP are utilized. Thus enriched simulation results are also compared in Fig.1(right) with those in the previous counterparts. Demonstrated preliminarily are either changes in the kink morphologies or an improvement of the energy return map characteristics. Starting with this, detailed contributions of the enrichment are further investigated.

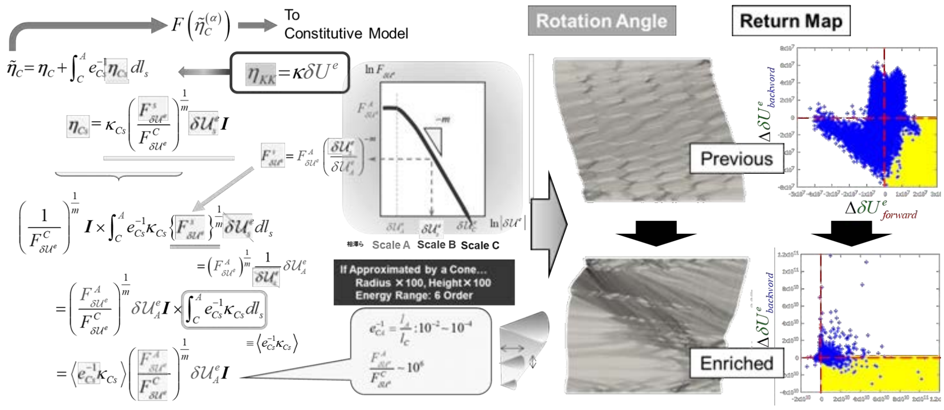

Fig. 1. Overview of the enrichment process, together with preliminary simulation results (rotation angle contour and the associated return map), comparing with the previous results.

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Mesoscale modeling of system of planar wedge disclinations and edge dislocations via the Airy Stress Function method

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I will describe recent results on the modeling and analysis of finite systems of planar wedge disclinations and edge dislocation via a new energy minimization principle for the elastic energy of the system.

Planar wedge disclinations are angular mismatches appearing at the nano-scale in a crystal lattice entailing a violation of rotational symmetry [1,4]. Together with dislocations, disclinations are observed in crystal plasticity [2] and in Shape-Memory Alloys undergoing the austenite-to-martensite phase-transition [3]. We introduce a novel mathematical formulation for the elastic energy of finite, isotropic systems of planar wedge disclinations and edge dislocations [6]. We operate in the regime of planar mechanical strains and linearized kinematics. In our theory disclinations and dislocations emerge as the solutions to minimization problems for isotropic mechanical energies under the constraint of kinematic incompatibility. As it is customary when working with 2-dimensional geometries in linearized elasticity, we write the mechanical equilibrium problem by introducing the Airy potential of the system, for which we construct a new variational formulation in the context of incompatible elasticity and for a non-simply connected domain. Our main result is the exact characterization of the energetic equivalence of a disclination dipole and an edge dislocation for various configurations identified by suitable rescaling of the energy of the system. By adopting the core-radius regularization approach [7] we show that, as the core radius vanishes, the (renormalized, rescaled) energy of a disclination dipole converges to the (renormalized, rescaled) energy of an edge dislocations.
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Effect of Kink Band Spacing on Kink Strengthening in LPSO-type Magnesium Alloy

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Magnesium alloys with the long period stacking order (LPSO) structure show the superior strength and are expected as the next generation structural material. In the LPSO-type magnesium alloy, a peculiar deformation called kink plays an important role and induces a drastic strengthening of materials. Therefore, understanding the kink strengthening phenomena is essential. Kink takes various morphologies, and it may affect kink strengthening. Additionally, multiple kink bands sometimes exist in proximity. Therefore, the spacing of kink bands may also be important. Kink band causes the strain gradient around the band. The higher-order gradient crystal plasticity [1] is an efficient way to represent the strain gradient effect in the crystalline scale. In this model, an additional governing equation expressing the dislocation density filed is introduced, and both the displacement and dislocation density fields can be solved simultaneously.

In this study, a higher-order gradient crystal plasticity analysis is conducted to evaluate the stress field around kink band to understand the strengthening mechanism due to kink. The finite element method sometimes provides an improper solution in the higher-order gradient crystal plasticity analysis [2]; therefore, the reproducing kernel particle method (RKPM) [3,4], which is a kind of meshfree method, is introduced into the higher-order gradient crystal plasticity analysis. A numerical analysis of a specimen with two kind bands is demonstrated, and the effect of kink spacing is quantitatively investigated. The present result suggests that there are three mechanisms in kink strengthening, that is texture, defect and neighboring strengthening shown in Fig. 1. The amplitude of neighboring strengthening depends on the kink spacing, and closer kink bands give a higher kink strengthening.

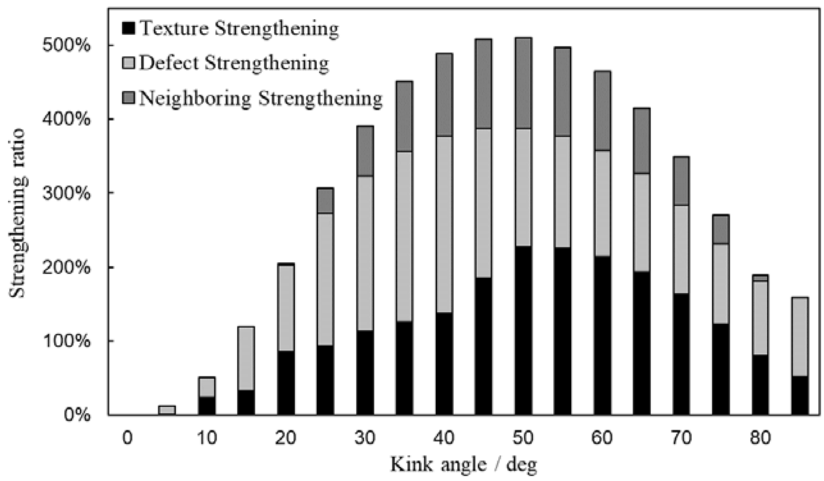

Fig. 1. Contributions of three strengthening mechanisms to kink strengthening with respect to kink angle.

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December 14 (Wed.)

Poster session

Toughening of the LPSO-type Mg-Zn-Y-Al RS P/M alloys

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Mg alloys are attractive structural materials for use in transportation vehicles owing to their low densities. However, current use is limited due to low strength of Mg alloys. Recently, LPSO-type Mg-Zn-Y alloy having excellent mechanical property was developed [1-3]. Especially, the Mg-Zn-Y-Al rapidly solidified (RS) P/M alloys through non-equilibrium processing exhibit high strength, excellent fatigue properties, and high corrosion resistance [4-6]. Therefore, the Mg-Zn-Y-Al RS P/M alloys could be excellent material choice for aerostructures. In structural design, plane strain fracture toughness is an important property requirement. In our previous study, we found out a pre-extrusion heat-treatment is effective in improving the fracture toughness of the Mg-Zn-Y-Al RS P/M alloys due to the formation of micrometer-sized α-Mg DRX grains and block-shaped LPSO phase grains [7,8]. However, effect of other manufacturing parameters and alloy composition on the fracture toughness and microstructure have not been clarified yet. In this study, therefore, we systematically investigated the effect of the heat-treatment condition, extrusion condition, cooling rate during melt spinning, and alloy composition on the fracture toughness and microstructure of the Mg-Zn-Y-Al RS P/M alloys. Furthermore, we tried to clarify the toughening mechanisms of the Mg-Zn-Y-Al RS P/M alloys.

A master alloy ingot with a nominal composition of $Mg_{96.75}Zn_{0.85}Y_{2.05}$, $Mg_{96.95}Zn_{0.85}Y_{2.05}Al_{0.15}$, $Mg_{96.75}Zn_{0.85}Y_{2.05}Al_{0.35}$ (at.%), were prepared by high frequently induction melting. RS ribbons were prepared by a single-roller melt spinning method with a circumferential speed of 10 $m\cdot s^{-1}$ and 42 $m\cdot s^{-1}$. Then, the RS ribbons were compacted into copper billets and degassed at 523 K for 15 min. The RS ribbon-compacted billets were heat-treated at 673 K and 738 K for 24 h. To consolidate the RS ribbons, extrusions at an extrusion ratio of 10, temperatures of 608 K and 623 K, and ram speeds of 0.5 $mm\cdot s^{-1}$, 1 $mm\cdot s^{-1}$, 2 $mm\cdot s^{-1}$, 2.5 $mm\cdot s^{-1}$, and 3 $mm\cdot s^{-1}$ were performed. Mechanical properties were measured through a tensile test and plane-strain fracture toughness test. Tensile tests were performed with an initial strain rate of 5×10^{-4} s^{-1}. Fracture toughness was tested following ASTM standards E399-12 with a test speed of 0.55 MPa $m^{1/2}$ s^{-1}. Microstructure of the RS ribbon-consolidated specimen was investigated using SEM, TEM, and HAADF-STEM. Crystal orientation analysis was performed by electron backscatter diffraction (EBSD) technique.

The alloy composition and manufacturing process, such as pre-extrusion heat-treatment conditions and cooling rate during melt spinning were optimized for improving the fracture toughness of the LPSO-type Mg-Zn-Y-Al RS P/M alloys. As a result, high-strength and high-toughness alloys with the yield stress of 400~460 MPa and plane-strain fracture toughness of 18~24 MPa $m^{1/2}$ were developed. For the formation of the block-shaped LPSO phase, its dispersion and volume fraction are important in enhancing fracture toughness of the Mg-Zn-Y-Al alloys, due to the frequent formation of microcracks and the promotion of crack deflection. Moreover, the higher level of work hardenability induced by the fine DRX grains with low dislocation density can improve their fracture toughness.

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Recrystallization Suppression and Kink Strengthening of MFS-type Mg-0.4Zn-1.0Y alloys

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Mg-Zn-Y alloys with a Long-Period Stacking Ordered (LPSO) structural phase have been reported to form kink bands by plastic forming, resulting in a significant increase in tensile yield strength maintaining ductility [1-3]. The LPSO phase has a long-period stacking structure of soft α-Mg layers and hard cluster-arranged layers (CAL) which is enriched with solute elements [4, 5]. Such a layered structure of soft and hard layers is called "mille-feuille structure (MFS)". Material strengthening in MFS-type Mg alloys by forming kink bands have been attracted attention [6, 7]. The MFS-type Mg-0.4Zn-1.0Y alloys used in this study form an MFS microstructure with cluster-arranged nanoplates (CANaPs) by aging treatment after solution treatment. The non-recrystallized region of the MFS-type Mg-0.4Zn-1.0Y alloys forms kink bands by hot extrusion. These regions can improve the yield strength of the alloys by kink strengthening. However, the effect of kink formation on mechanical properties has not been quantitatively evaluated. To evaluate kink strengthening, other strengthening factors such as grain refinement strengthening and work hardening must be eliminated. In this study, we established extrusion conditions to suppress dynamic recrystallization (DRX) and annealing conditions to suppress static recrystallization (SRX). Moreover, we investigated the microstructure and mechanical properties of the alloys to evaluate kink strengthening.

The nominal composition of the alloy used in this study was Mg-0.4Zn-1.0Y (at.%). RS ribbon was prepared by a single-roller melt spinning method with a cooling rate of 1.4 x 10^5 K/s. RS ribbons were pressed into copper billets and degassed at 523 K for 15 min. To consolidate the RS ribbons, hot-pressing was performed at 623 K. The hot-pressed alloy was solution heat-treated at 813 K for 72 h in Ar gas atmosphere. After the solution heat-treatment, MFS material with precipitated CANaPs was prepared by aging at 623 K for 12 h. The MFS material was extruded at an extrusion temperature of 623 K, an extrusion ratio of *R*5, and an extrusion ram speed of 1.5 mm/s in which DRX suppressed. After extrusion processing, annealing treatment was applied. Mechanical properties were evaluated using tensile tests with an initial strain rate of 5.0 x 10^{-4} s^{-1}. Microstructure was investigated by confocally optical microscope (OM), X-ray diffraction (XRD), and scanning electron microscope (SEM). Crystal orientation analysis was also performed by electron backscatter diffraction (EBSD) technique.

We revealed extrusion conditions that can suppress DRX. The extruded alloys without DRX exhibited a tensile yield strength of 386 MPa and an elongation of 5.9%. On the other hand, the extruded alloys with DRX showed relatively lower tensile yield strength and higher elongation in comparison with the extruded alloys without DRX. Next, we revealed annealing conditions of the extruded alloys to suppress SRX. The mechanical properties of the annealed alloys without SRX were not changed by annealing. On the other hand, the tensile yield strength of the annealed alloys with SRX decreased and their elongation increased with increasing the area fraction of SRXed region. Therefore, the superior mechanical properties of the annealed alloys without DRX and SRX may be due to kink strengthening.

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Investigation of fracture toughness of extruded Mg-Zn-Y alloys with multimodal microstructure

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Mg alloys are expected to be used as structural materials for transportation equipment because they are one of the light structural materials. However, the application of this alloys as structural materials is limited because the strength of them is low. Recently, Mg-Zn-Y alloys consisting of α-Mg matrix and long-period stacking ordered (LPSO) structure phase were developed[1]. The alloys show excellent mechanical properties after thermo-mechanical treatments such as extrusion and rolling. It is because the microstructure of the extruded alloys are characterized by multimodal microstructure that consists of: fine dynamically recrystallized (DRXed) α-Mg grains with a random crystallographic orientation, coarse-worked α-Mg grains with strong fiber texture in which the $<10\bar{1}0>$ of the grains are parallel to the extrusion direction (ED), and LPSO phase grains also have a $<10\bar{1}0>$ fiber texture[2]. The DRXed α-Mg grains contribute to an improvement in ductility. The worked α-Mg grains and the LPSO phase grains act as effective components for increasing the strength. In recent years, various studies have been conducted to commercialize the alloys. From a safety point of view, investigation of fracture toughness is important. Nishimoto *et al.* reported the fracture toughness of the Mg-Zn-Y-Al RS ribbon-consolidated alloy[3]. However, there are few studies on the fracture toughness of alloys with multimodal microstructure. Therefore, in this study, the fracture toughness was investigated by using several Mg-Zn-Y alloys with different volume ratios of three regions in multimodal microstructure.

The materials were $Mg_{97}Zn_1Y_2$ (at. %) alloys and $Mg_{99.2}Zn_{0.2}Y_{0.6}$ alloys. The billets were extruded with an extrusion ratio of 10, extrusion temperatures of 573 K, 623 K, 673 K, 723 K, and an extrusion ram speed of 0.9 mm s^{-1}. The mechanical properties were evaluated by tensile tests and fracture toughness tests. Tensile tests were carried out using an Instron testing machine at room temperature with an initial strain rate of 5 x 10^{-4} s^{-1}. The tensile axis was held parallel to the ED. The fracture toughness tests were conducted by using 1/4 compact tension specimens as per the ASTM E399-20 standard. A testing speed was 0.55 MPa $m^{1/2}$ s^{-1}. The loading directions were vertical to the ED (T-L specimen) and parallel to the ED (L-T specimen). The microstructure was observed by optical microscopy and scanning electron microscopy. The textures of the alloys were analyzed by an electron backscatter diffraction analysis.

Regardless of alloy compositions, volume fraction of DRXed grains increased with increasing extrusion temperature. Conversely, volume fraction of worked grains decreases with increasing extrusion temperature. Volume fraction of LPSO phase of $M_{97}Zn_1Y_2$ and $M_{99.2}Zn_{0.2}Y_{0.6}$ alloys were ~23% and~2%, respectively. Ultimate tensile strength (UTS) and yield strength (YS) of the extruded two alloys decreased with increasing extrusion temperature. UTS and YS of $M_{97}Zn_1Y_2$ alloys were higher than those of $M_{99.2}Zn_{0.2}Y_{0.6}$ alloys. The fracture toughness of $M_{97}Zn_1Y_2$ alloys also showed higher than that of $M_{99.2}Zn_{0.2}Y_{0.6}$ alloys at any extrusion temperature. In the T-L specimens of $M_{97}Zn_1Y_2$ alloy, fracture toughness increased with increasing extrusion temperature. It was observed that the worked grains elongated along ED suppressed the deflection of cracks. On the other hand, fracture toughness of the L-T specimens decreased with increasing extrusion temperature. This is due to texture orientation of the worked alpha-Mg and LPSO grains.

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Mechanical properties and microstructure of dilute Mg-Y-Zn alloys prepared by combination of low cooling rate solidification and extrusion techniques

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The wrought Mg-Y-Zn alloys containing long-period stacking ordered (LPSO) phase show excellent mechanical properties[1]. Typical LPSO phase in Mg-Zn-RE alloys is found to have 18*R* and 14*H* structures. These structures have chemical modulation around stacking fault, in which solute elements are enriched in four atomic layers ($L1_2$-type Zn_6RE_8 cluster-arranged layers: CALs) of closely packed planes at six- and seven-period intervals, respectively. The unique crystallographic structure of LPSO phase limits the activation of slip system to only the basal <*a*>slip at room temperature and suppresses deformation twins. The strong constraint of the slip system activates kinks as a accommodative deformation mechanism for plastic deformation[2]. It is known that introduction of deformation kink band into the LPSO phase induces strength in extruded alloys. More recently, Hagihara *et al*. reported that a deformation kink occurred not only in the LPSO single-phase crystal but also in dilute $Mg_{99.2}Y_{0.6}Zn_{0.2}$ single crystal with aperiodic dispersion of the CALs [3, 4]. The kink-deformed $Mg_{99.2}Y_{0.6}Zn_{0.2}$ single crystal showed excellent mechanical properties. Therefore, in this study, we have attempted to develop high strength polycrystalline dilute Mg-Y-Zn alloys with high density CALs by combination of low cooling rate solidification and extrusion techniques. The cooling rate and alloy compositional dependence on mechanical properties of the alloys has been investigated.

$Mg_{99.2}Y_{0.6}Zn_{0.2}$ (at.%), $Mg_{98.4}Y_{1.2}Zn_{0.4}$, and $Mg_{97.6}Y_{1.8}Zn_{0.6}$ alloys were melted by high-frequency induction heating and then solidified in a carbon crucible at cooling rates of 1.6 x 10^{-2}, 3.4 x 10^{-2}, 6.7 x 10^{-2}, and 15 x 10^{-2}, 10 Ks^{-1}. These alloys were gravity-cast at the cooling rate of 10 Ks^{-1}, and then heat-treated (solution-treated at 793 K for 5 h, and then aged at 573 K for 5 h). The extrusion was performed at an extrusion temperature of 573 K and an extrusion ratio of *R*10. Mechanical properties were evaluated by Instron-type tensile test. Microstructure was observed by OM, SEM/EBSD, and TEM.

It was confirmed that the as-solidified $Mg_{99.2}Y_{0.6}Zn_{0.2}$ alloys that were produced via furnace cooling possessed large CALs-dispersed regions. The area fraction of the CALs-dispersed region tends to increase with decreasing cooling rate. In other words, the two-region structure that consists of the CALs-dispersed region and solute-lean alpha-Mg matrix region could be formed by cooling rate control technique.

The area fraction of the CALs-dispersed region in the solidified alloys affects the multimodal microstructure evolution and mechanical properties of the extruded ones. The area fraction of the worked grain region in the extruded alloys and the kink dispersion in the worked grain region increased with increasing the CALs-dispersed region. Developing the kink-deformed worked grain region strengthen the alloy. It was also revealed that decrease in the density of CALs in the CALs-dispersed region promoted dynamic recrystallization during extrusion. Increase in the dynamic recrystallized grain region improve ductility of the alloy. In addition to the area fraction of the CALs-dispersed region, the density of CALs in the CALs-dispersed region in as-solidified alloys definitely affects multimodal microstructure evolution.

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The Effect of Hot Rolling on Kink Formation in the MFS-type Mg-0.4Zn-1.0Y Alloy with α-Mg Single Phase.

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Magnesium alloys containing a long period stacking ordered (LPSO) phase have attracted a great deal of attention because of its excellent mechanical properties [1]. The property enhancement was mainly caused by kink deformation and strengthening in LPSO phase. The LPSO phase consists of hard- and soft-layers, in which the hard-layer is a cluster arranged layers (CAL) enriched with solute elements and the soft-layer is the α-Mg layers. Such layered structure is called "mille-feuille structure (MFS)". Recently, it was found that kink deformation occurs not only in the LPSO structure, but also in a non-periodic MFS with cluster arranged nanoplates (CANaPs).

In the previous studies, the non-recrystallized region of MFS-type Mg-0.4Zn-1.0Y (at%) alloys with α-Mg single phase form kink bands by hot extrusion. This region can improve the yield strength of the alloys by kink strengthening. However, kink strengthening should be investigated under conditions of suppressed recrystallisation. Rolling process is an effective and more quantitative method of investigating kink deformation while suppressing recrystallisation, as it can introduce low amounts of strain multiple times. Therefore, this study is aimed to study the effect of deformation strain on the kink formation and mechanical property of the MFS-type Mg-0.4Zn-1.0Y alloy.

The master ingot with a nominal composition of Mg-0.4Zn-1.0Y (at%) was prepared by high frequency induction melting and casting. Rapidly solidified (RS) melt-spun ribbons were compacted into copper billet and first degassed at 523K for 15 min. The billets were then hot pressed at 623K for 10 min. After hot pressing, the bulk alloys were solution treated at 813K for 72h and then aged at 623K for 12h. After aging treatment, hot rolling was carried out at 573K, at a peripheral speed of 8.0×10^{-3} m/s, and a thickness reduction ratio of 5% per pass. The final total deformation strains were set at 0.5 and 1.0. Tensile test were conducted at room temperature with an initial strain rate of 5×10^{-4} /s. Microstructure was characterized by using SEM and SEM/EBSD.

The tensile yield strength of the hot-rolled alloy with total strain of 1.0 was two times as high as as-aged alloy. Figure 1 shows the SEM microstructures of as-aged and as-rolled specimens. Many chevron-shaped kink bands were observed on both the RD (rolling direction) and the TD (transverse direction) samples, Fig. (c) and (d). But only few kink bands were observed on the ND (normal direction) sample, Fig. (b). The dispersion of the kink bands and their bending angle observed on the RD and TD planes increase with increasing total deformation strain. The result shows that the kink bands form and kink strengthening takes place in the hot-rolled MFS-type Mg-0.4Zn-1.0Y alloy.

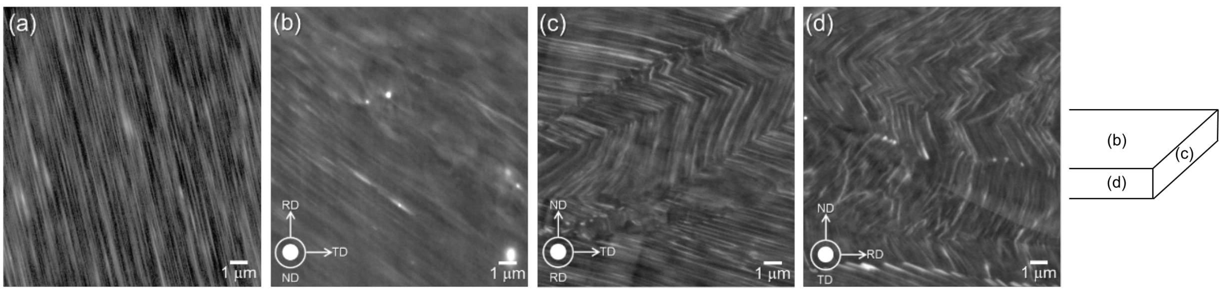

Fig. 1 SEM microstructures of MFS-type Mg-0.4Zn-1.0Y alloys: (a) as-aged, (b) $\Sigma\bar{\varepsilon} = 1.0$ for ND, (c) $\Sigma\bar{\varepsilon} = 1.0$ for RD, (d) $\Sigma\bar{\varepsilon} = 1.0$ for TD.

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Work-hardening Behavior of Mg-Y-Zn Alloy with Long-periodic Stacking Ordered Phase in High-pressure Torsion Process

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Our previous study has showed the effect of wrought-processing on kink boundary formation and mechanical properties using the Mg - 9at.%Y - 6at.%Zn alloy consisting of the long-periodic stacking ordered (LPSO) single phase (Mg-Y-Zn alloy). [1] Hardness closely related to the magnitude of equivalent plastic strain introduced by the wrought-processing. Among the various wrought-processes, the Mg-Y-Zn alloy after high-pressure torsion (HPT) processing, in which torsion is applied under high compressive stress, exhibited excellent work-hardening behavior. The aim of this study is to investigate the effects of initial microstructure in the Mg-Y-Zn alloy on kink boundary formation and work-hardening behavior in the HPT process. The effect of processing temperature was also investigated.

The Mg-Y-Zn alloys produced by casting (Cast) and directional solidification (DS) were deformed by HPT processing. Figure 1 shows the Vickers hardness of the Mg-Y-Zn alloys after HPT processing. The DS_0° specimen which was cut perpendicular to the <1 1 -2 0> growth direction showed higher value after compressive deformation (Number of turns $N = 0$) in the HPT process than the DS_90° specimen cut parallel to the growth direction. This reason is that the formation of the kink boundaries facilitated through compressive deformation because the (0 0 0 1) basal planes in the DS_0° specimen were parallel to the compression axis. However, the subsequent torsional deformation resulted in higher work-hardening in the DS_90° specimen. The Cast specimen showed the similar result to the DS_90° specimen. In the DS_90° and Cast specimens, numerous finer kink bands were formed compared to the DS_0° specimen, as shown in Fig.2, indicating that the formation of such microstructures results in the higher work-hardening. It was also found that more effective work-hardening (kink band formation and microstructure refinement) could be achieved by controlling the processing temperature in the HPT process to accelerate plastic deformation.

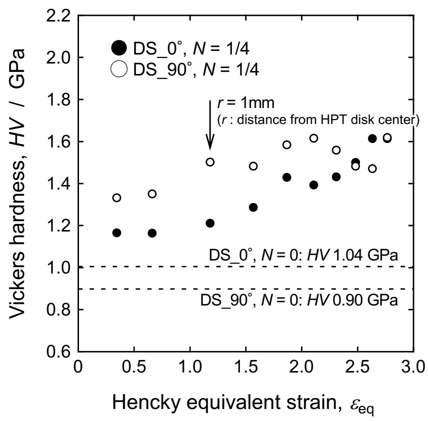

Fig.1 Vickers hardness in DS specimens after HPT process.

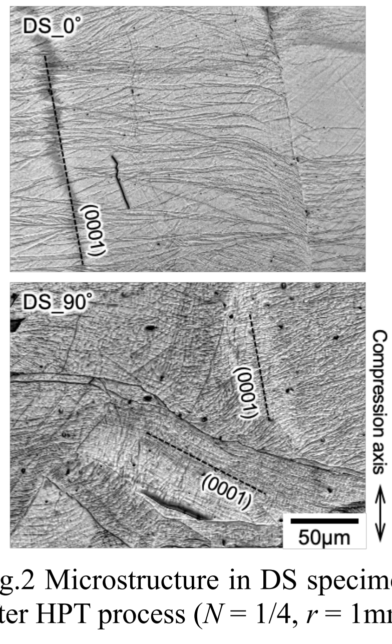

Fig.2 Microstructure in DS specimens after HPT process ($N = 1/4$, $r = 1$mm).

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Texture evolution and mechanical properties of Mg-2Y-1Zn alloy via vortex extrusion

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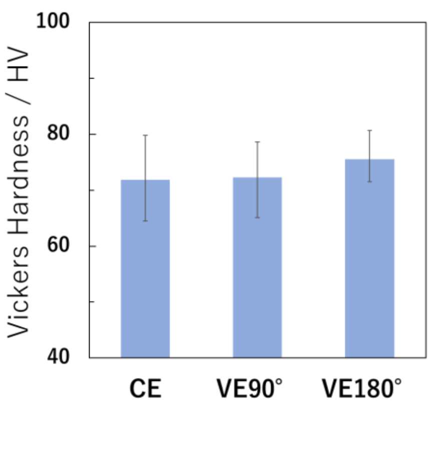
LPSO-type magnesium alloys have a two-phase alloy consisting of an α-Mg phase and an LPSO phase; in Mg-2Y-1Zn alloys, the volume fraction of the LPSO phase is about 25%. This alloy exhibits ordinary mechanical properties in the cast state, but it is known that the mechanical properties can be significantly enhanced by warm extrusion. The extrusion ratio required for this strengthening is 3-10. The equivalent plastic strain is about 1.0-2.3. This work ratio is relatively smaller than that required to fabricate a conventional wrought Mg alloy. If the extrusion ratio is further increased, the alloy tended to soften due to dynamic recrystallization caused by processing heat. On the other hand, some researchers have reported that in Mg-9Y-6Zn alloys exhibiting a single phase of LPSO, the more equivalent strain applied, the more kink-strengthened this alloy turns out to be. The reason is thought to be that the kink deformation of the LPSO phase without recrystallization increases the plastic deformation resistance of the basal slip. From another perspective, some other researchers have reported that in Mg-Gd-Y-Zn alloys with LPSO phases, using the severe plastic deformation method results in strengthening of the alloy by refining the microstructure. In this study, we focus on vortex extrusion, in which a large shear strain can be added to the normal extrusion process by carving a groove in the die that rotates the work piece in warm extrusion, to investigate the microstructural evolution of the Mg-2Y-1Zn alloy and its mechanical properties when the amount of strain applied, in this case the material rotation angle, is changed. The results showed that the Vickers hardness increased as the rotation angle was increased. Observation of the microstructure showed that the kink deformation changed from a ridge-type kink, in which the LPSO phase folds in a zigzag pattern, to an ortho-type kink, in which the LPSO phase curves with a gradual change in angle. In the day's presentation, we would also show the results of XRD and SEM/EBSD investigating the texture evolution and the percentage of dynamic recrystallization.



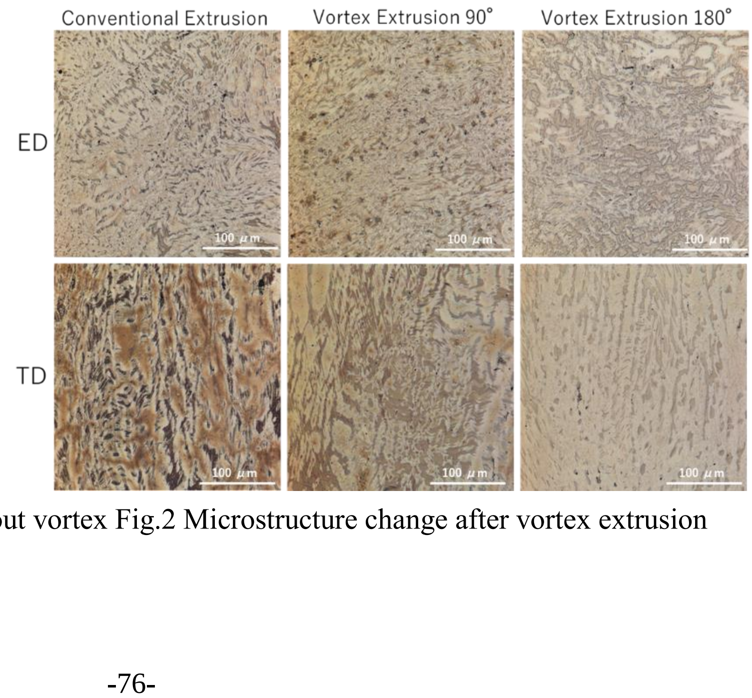


Fig. 1. Vickers Hardness with or without vortex Fig.2 Microstructure change after vortex extrusion

Numerical Investigation of the Microstructural Features Favoring Kink Band in Layered α/β Ti-9Cr Alloy

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Deformation kink bands were found in a layered α/β Ti-9Cr alloy after uniaxial compression tests. The metastable Ti-9Cr alloy was developed with a controlled thermomechanical process to decrease the dislocation mean free path in the β phase and obstruct the softer deformation mechanisms [1]. In this study, the effect of alternative stacking α/β layers on the kink deformation mechanism was simulated with Crystal Plasticity Finite Element Method (CPFEM).

The mechanical properties of the Ti-9Cr alloys were investigated with uniaxial compression tests. The microstructures of the deformed surface were then characterized by SEM, TEM observation, and EBSD-EDS measurements to identify the crystal orientation and operative deformation mechanism. Nanoindentation tests were carried out on the undeformed specimen. The CP parameters were calibrated according to the response of the nanoindentation tests. Phenomenological crystal plasticity models with different sets of dimensions and tilt angles were developed. 25% of plastic strain was applied to the models with corresponding boundary conditions to simulate the mechanical response of the layered structure in the CPFEM software Abaqus. By inspecting the favorable microstructure for the deformation kinking to take place, the mechanism of kink band formation was evaluated.

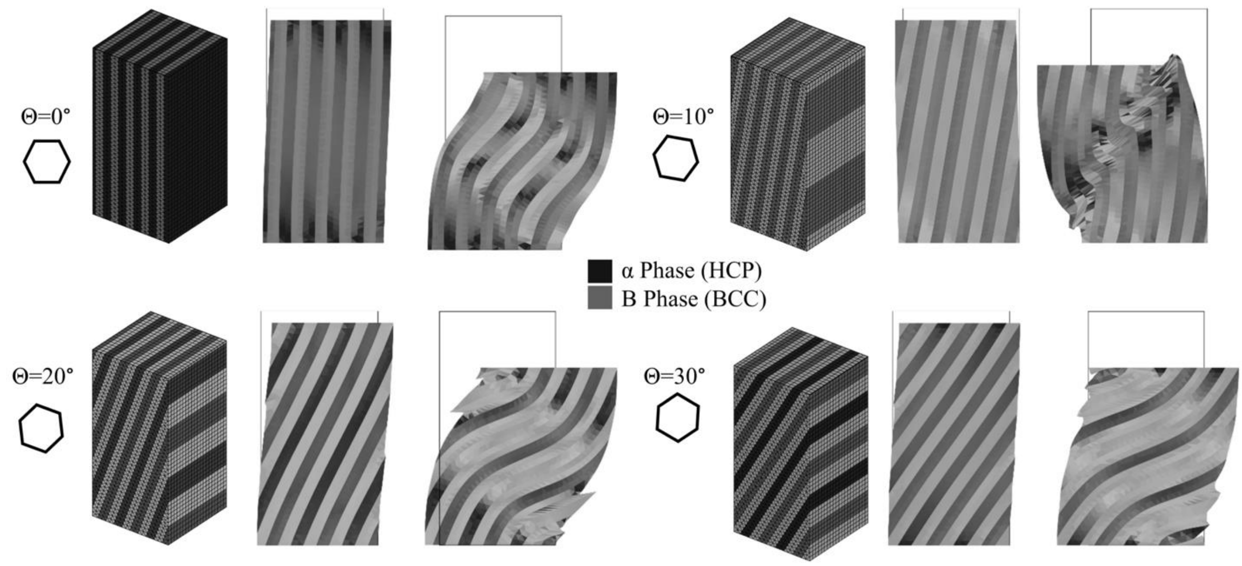

Fig. 1. Simulations of uniaxial compression tests with 25% plastic strain with tilt degree of 0°, 10°, 20°, and 30°

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Deformation mechanism of LPSO-type Mg alloy after kink deformation

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Mg alloys containing long period stacking/order (LPSO) phase appear high strength by adapting high-temperature processing. The strengthening of LPSO-type Mg alloys is believed to derive from kink deformation which has been experimentally demonstrated [1]; however, details of kink deformation mechanisms have not yet been elucidated. The main deformation modes in the LPSO phase are limited to basal slips and kink deformations, due to the high anisotropy of the LPSO structure. Kink deformation is thought to dominate their yielding, especially in situations where basal slip is suppressed, but it is not clear what deformation mechanisms are active when further additional deformation is applied after kink deformation. In this study, we observed deformation microstructures of LPSO-type Mg alloys compressed at room temperature to beyond the yielding to clarify the deformation mechanisms that develop in the kink deformed microstructure.

We prepared directionally solidified $Mg_{85}Zn_6Y_9$ (at.%) alloy that is compressed at room temperature up to the nominal strain of 10 %. Microstructures were investigated with scanning electron microscopy (SEM), transmission electron microscopy (TEM) and aberration-corrected scanning transmission electron microscopy (STEM) operating at 200kV.

Fig. 1 shows a TEM bright field image obtained from the compressed $Mg_{85}Zn_6Y_9$ alloy. Kink boundaries (KBs) were often observed, as indicated by the white arrowheads, and rotation angles at the KBs were generally greater than 10 degrees. Focusing on the near-KB, several boundaries extended from the KB, which are inclined from the KB. According to Rank-1 theory [2], boundaries inclined to the KB do not necessarily satisfy the continuity of deformation, suggesting that the boundaries introduced by a deformation mechanism different from kink deformation. Fig. 2 shows a LAADF-STEM image, strongly enhance strain contrast, obtained around the KB. The boundaries (denoted as SB: secondary boundary, hereafter) can be confirmed as bright contrasts which were aligned with an average interval of about 200 nm and terminated within the grain. Angles between KB and SB are constant at almost 58 ± 1.5 degrees, indicating that SB exhibits features similar to shear bands. If the shear bands are terminated within the grain, additional deformation is necessary to compensate for the deformation discontinuities. Fig. 3 (a) shows an atomic resolution STEM image around the intersection between KB and SB. At the SB, the basal plane is rotated by a few degrees, suggesting that crystal rotation occurred to resolve the deformation discontinuity. Fig. 3 (b, c) shows a schematic illustration of the SB introduction on KB. When the KB moves due to additional deformation, there is a volume discontinuity between the left and right sides of the boundary. Introductions of SB will compensate the discontinuity, which can be represented by a disclination dipole. In the presentation, we will discuss details on structural features of SB and its role on deformation after yielding of the alloy.

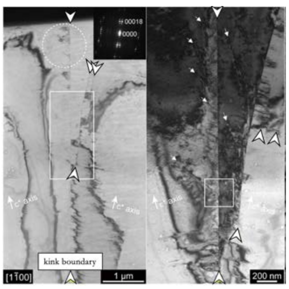

Fig.1 Bright-field TEM image of a LPSO grain in the $Mg_{85}Zn_6Y_9$ alloy

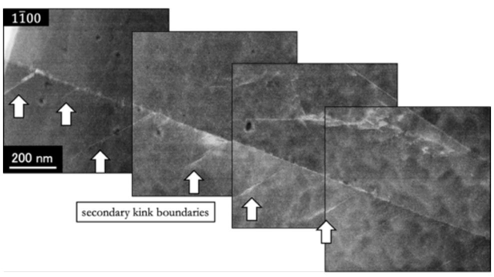

Fig. 2 [$1\bar{1}00$] projected LAADF-STEM image of a KB and SBs

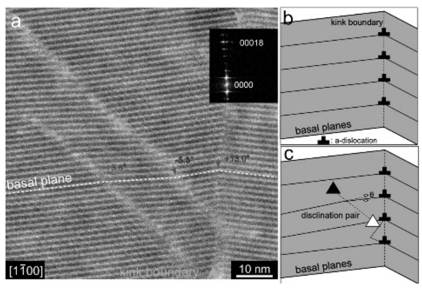

Fig. 3 Atomic resolution STEM image around the intersection between KB and SB

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Deformed microstructure in LPSO-type magnesium alloys exhibiting PLC effect

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Magnesium alloys containing a few atomic percent of Zn/RE (RE denotes rare earth) form a phase with a unique structure called long period stacking/order (LPSO) structure. In this structure, stacking faults are periodically introduced into α-Mg which has an hcp structure, and solute is concentrated in the fcc structure formed by the stacking faults[1)]. The superior strength of this alloy is believed to be due to the kink boundary formed in the LPSO phase. Hagihara et al[2)]. evaluated kink strengthening and reported that the kink boundary acts as a resistance to basal slip. He also reported that kink strengthening can be expected to provide further strengthening in addition to the conventional strengthening mechanism. Recently, the Portevin-Le Chatelier (PLC) effect has been found to occur in room temperature deformation of LPSO phases. The PLC effect is understood as a phenomenon caused by the interaction between dislocations and solute atoms, which results in negative strain rate sensitivity [3)]. Although the PLC effect is generally observed in high-temperature processing, in the case of the LPSO phase, it is observed in room-temperature compression. The purpose of this study is to find the microstructural features responsible for the PLC effect in deformed Mg-Zn-Y alloys.

Directional solidified $Mg_{85}Zn_6Y_9$ (at. %) alloys were compressed in the direction parallel to the solidification direction at room temperature. To evaluate strain rate sensitivity (m-value), the specimen was compressed at two different strain rates (1.67×10^{-3}/s, 1.67×10^{-4}/s) until the m-value became negative and the PLC effect was clearly observed (nominal strain 15%). After compression, the specimens were mechanically polished, then ion-milled into thin foils for STEM observation.

Fig. 1 (a) shows a HAADF-STEM image of the kink boundary. The image intensity increases on the kink boundary indicated by arrows, suggesting that solute atoms segregate at the boundary even though the specimen was deformed at room temperature. The wavy shape of the kink boundary and discrepancy in the stacking of the LPSO phases on either side of the boundary suggests that the kink boundary moved with further deformation after it was formed. In addition, solute segregation can be observed in the rectangular region near the kink boundary in Fig.1(a). Fig. 1 (b) shows a magnified HAADF-STEM image of the rectangular region in Fig. 1 (a). Burgers circuit analysis in Fig. 1 (b) indicates that there is a defect corresponding to the Frank partial dislocation (b = 1/6[2023] in hcp) in the solute segregated region. Fig. 1 (c) shows the intensity profiles obtained in regions ① and ②in Fig. 1 (b). Based on the intensity profiles, we found that the existence of the <a+c> dislocation changed the stacking structure from intrinsic-II type stacking fault to extrinsic type stacking fault.

The above results suggest that the migration of kink boundary with solute segregation and the formation of defects including <c> contribute to the PLC effect observed in this alloy. Details of microstructure will be discussed in the presentations.

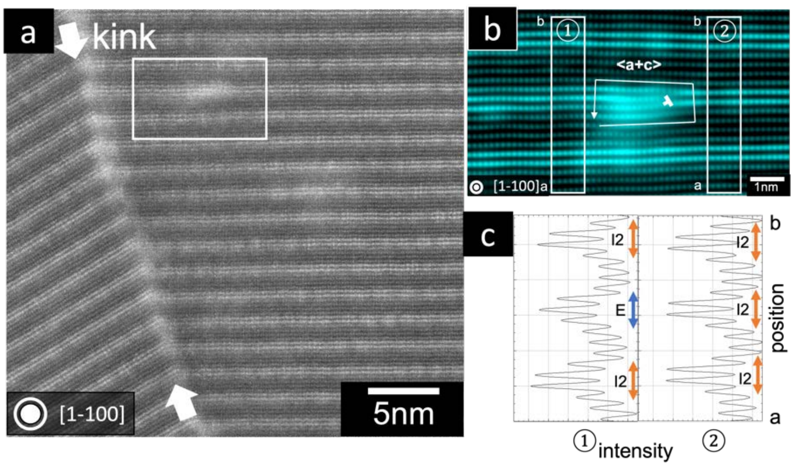

Fig. 1 (a)HAADF-STEM image of kink boundary in $Mg_{85}Zn_6Y_9$ alloy, (b)HAADF-STEM image around the <a+c> dislocation in (a), (c) intensity profile of the area ①,② in (b)

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Dislocation analysis of solute-enriched stacking faults in kink boundaries in $Mg_{97}Zn_1Y_2$ alloys

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Mg-Zn-*RE* (rare earth elements) alloys have received great attention in the field of light metal research because of their superior mechanical properties. This fact is due to a unique alloy strengthening of kink deformation promoted by long-period stacking order (LPSO) structure. The microstructure formed by kink deformation is typically characterized as kink boundaries (KBs) accompanied by crystal rotation. In LPSO structures, KBs are often manifested as micrometer-scale flat boundaries accompanied by geometrically necessary (GN) dislocations arrays. These flat boundaries are essentially enriched with the solute Zn/*RE* atoms (solute-enriched stacking faults, hereafter referred to as SESFs) [1]. Recent reports indicate that SESFs in the α-Mg matrix can also cause kink deformation and promote kink strengthening in some Mg-Zn-*RE* alloys [2]. In this study, we observed microstructures around KBs to characterize the configuration of SESFs and dislocations.

The KBs in the annealed $Mg_{97}Zn_1Y_2$ specimen were observed using a scanning transmission electron microscope (STEM), Titan Cubed G2, operated at an acceleration voltage of 300 kV under a parallel-beam STEM mode with a convergence semi-angle of 1.2 mrad. The specimen was set in a two-beam excitation condition for g = 0006 during specimen tilt. The bright-field (BF), dark-field (DF) and high-angle annular dark-field (HAADF) images were obtained by detecting the direct beam (0 ~ 5 mrad), diffracted beams in the range of 7–49 mrad, and diffracted beams in 52–200 mrad, respectively.

Figure 1 shows a BF-STEM image obtained under the two-beam condition, $g = 0006$. In this condition, c'-dislocations, which have c<000l> components in their Burgers vectors, are visible. By measuring the rotation angles at KBs from the solute Zn/Y atom enrichment layers arranged along Mg(0001), indicated by white dotted lines, it was noted that the crystal was rotated about 4.2° and 1.4°at KB1 and KB2, indicated by black dotted lines, respectively. The KB is shown here as arrays of stacking-fault layers with short linear dark contrast that are composed of the SESFs and GN dislocations. Both KB1 and KB2 are segmented at inflection points to form step shapes, which are common features in annealed specimens [2]. Within the KBs, the c'-dislocations only appear at the position of segmentation indicated by black arrows. This can be explained that the crystal rotation becomes discontinuous at the terminated edges of the segmented KBs, and this discontinuity is compensated by the introduction of an array of non-basal dislocations to avoid the appearance of micro-voids [2]. Outside the KBs, the c'-dislocations appears inside the linear solute Zn/Y atom enrichment area indicated by white arrows. This can be explained that the solute-atom enrichment area can be separated into the nanosized segments by c'-dislocations, and these nanosized segments can form the tilt boundary to significantly reduce the boundary energy [3]. Based on the observation results described above, the formation process of the microstructure at the KBs will be discussed using a dislocation-disclination model.

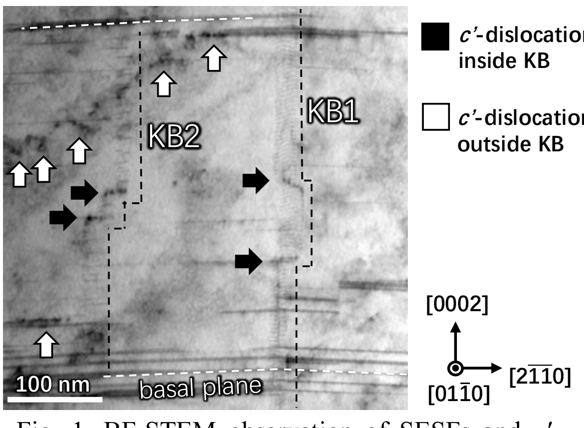

Fig. 1. BF-STEM observation of SESFs and c'-dislocations in KBs in $Mg_{97}Zn_1Y_2$ specimen.

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Deformation anisotropy in hcp-Mg structure based on generalized stacking fault energy

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Mg alloys containing long-period stacking/order structure (LPSO) phases exhibit high strength that is believed to derive from kink deformation due to the high structural anisotropy of the LPSO phase. Kink deformation is represented by the collective motion of dislocations in limited slip systems, which forms sharp kink boundaries with crystal rotation. However, detailed mechanisms of kink deformation and their strengthening mechanism are still unclear. In this study, we have investigated an anisotropy of dislocation motion under uniaxial compressive stress on the hcp-Mg structure, based on the generalized stacking fault energy (GSFE) calculated by first-principles calculations to deepen our understanding on the kink deformation mechanism.

For calculations of GSFE, we set a superlattice of the hcp-Mg structure as a model structure. Structural optimizations were performed by the VASP code. Fig. 1(a) shows the [0001] projection of the model cell with a dimension of 1×1×3 to the hcp-Mg. The maximum restoring stress τ_{max} was calculated along the path with the minimum GSFE gradient, and the Peierls stress σ_p was evaluated using the Joos and Duesbery equation [1]. We also calculated GSFE and Peierls stresses with the model structures under uniaxial compression of 5% along the $[11\bar{2}0]$ and $[1\bar{1}00]$ directions, respectively.

Fig. 1(b) shows a calculated GSFE surface with the optimized hcp-Mg cell. The path with the smallest GSFE gradient is depicted by the arrows which correspond to the partial dislocations of the basal slip in the hcp-Mg. Fig. 1 (c) shows a profile of GSFE along the path. The highest gradient of GSFE (τ_{max}) was 1.6 GPa that represents the Peierls stress of σ_p=3.6 MPa. The corresponding GSFE curves with uniaxial compressions are also plotted in Fig. 1 (c). When the model compressed along the $[1\bar{1}00]$ direction, the first peak of GSFE apparently decreased, resulting in a decrease of the Peierls stress of corresponding partial dislocations to $\sigma_p^{1\bar{1}00}$=0.023 MPa, that is about 1% of the one in the original hcp-Mg. On the other hand, the Peierls stress in the $[10\bar{1}0]$ direction increased to $\sigma_p^{10\bar{1}0}$=11 MPa, indicating the dislocation motion show high anisotropy by the compressive stress. In the case of compression along the $[11\bar{2}0]$ direction resulted in $\sigma_p^{1\bar{1}00}$=4.2 MPa and $\sigma_p^{10\bar{1}0}$=3.2 MPa. The above results indicate that compressive stress may provide anisotropy in the equivalent slip system, leading to activating different deformation modes.

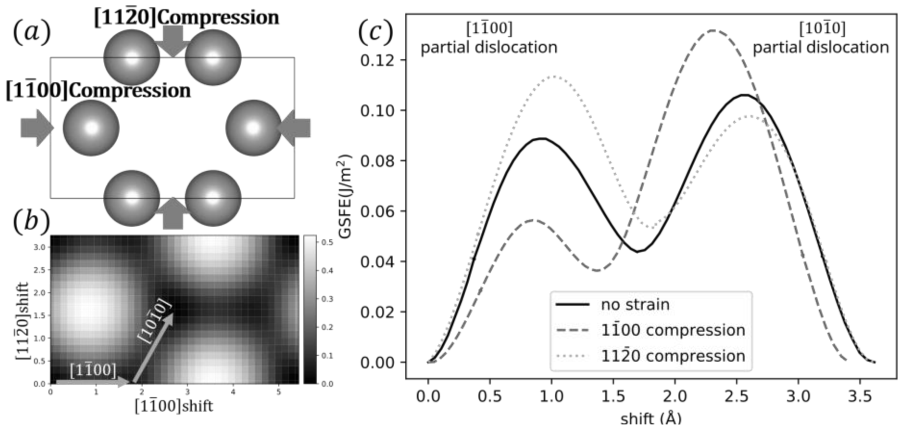

Fig. 1 (a) [0001] projection of Slab cell. (b) GSFE surface for each atomic displacement. (c) GSFE curves of the models with each stress state.

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Elastic energy of disclination multipole formed at intersection of kink band and shear band

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In order to construct a theory of kink strengthening found in LPSO-Mg alloys [1], we have performed kinematic analysis of kink band, ridge kink, and ortho kink sheared by basal slip using the rank-1 connection which is a condition for the continuity of deformation. We have shown that disclination multipole is formed at the intersection of shear band and kink and they should act as energy barrier for deformation[2,3]. In this study, we investigate the effect of disclination multipole formed in these three types of kinks on the deformation behavior in terms of elastic energy of the disclination multipole (E_m).

We define a shear band as a region of uniform shear deformation parallel to the basal plane with a finite thickness, d. Kink band is characterized by the basal shear and rigid body rotation as described by the model by Inamura[4]. The sign of the shear direction is important to consider ridge and ortho kinks. Connecting two kink bands which were formed by the basal slips with opposite sign form ridge kink. Ortho kink is defined as aggregate of kink bands with the same sign of the basal shear. When the three types of kinks, namely kink band, ridge kink and ortho kink, are sheared by the shear band with keeping the continuity of deformation, additional rigid body rotation is required at the intersection of the kink and the shear band. This rotation generates disclination multipole at the intersection[2]. The configuration and the strength of disclination multipole were obtained by analyzing Rank-1 connection. E_m was evaluated using the elastic energy of disclination multipole formula derived by Romanov et al[5].

Figure 1 and 2 schematically show disclination multipole formed in sheared kink band and sheared ridge kink, respectively. In these figures, the magnitude of shear to form kink band is s_1=0.3 in Fig.1, s_2=0.3 and s_3=−0.3 for ridge kink in Fig.2 with the thickness of the shear band d = 10^{-6}m, kink width l= 10^{-5}m, the magnitude of shear in the shear band s_m = 0.002. Figure 3 shows E_m of disclination multipole with the integral radius R from the center of the multipole as a variable. In this example, we found E_m for ridge kink is higher than that of kink band. E_m is evaluated for three types of kinks and its effects on the deformation behavior will be discussed.

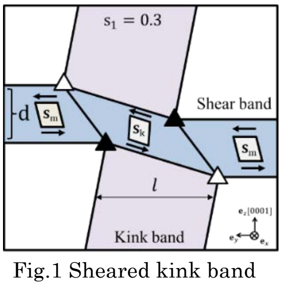

Fig.1 Sheared kink band

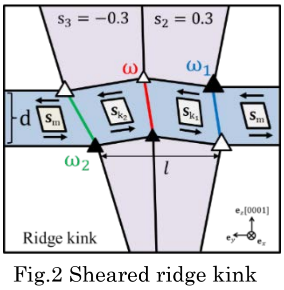

Fig.2 Sheared ridge kink

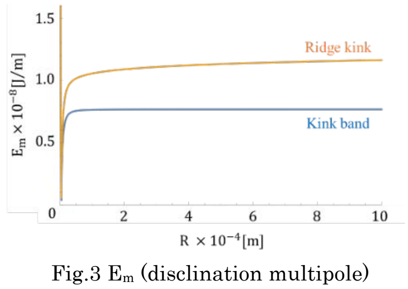

Fig.3 E_m (disclination multipole)

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Stable Shape of Curved Ridge-Kink Consisting of a Sequence of Fine Kink-bands

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A geometric model that can quantitatively describe kink deformation is required to elucidate the mechanism of "kink strengthening" which has been discovered in LPSO-Mg alloys. Based on the rank-1 connection, which is the condition for continuity of deformation, Inamura [1] proposed a geometric model of kink deformation that is consistent with experimental results and proved that the lattice defects called disclinations inevitably exist in connecting kink-bands. Such disclinations should have significant impacts on the formation of kink-microstructure and the mechanical properties of the material. However, Inamura's model cannot quantitatively describe various kink structures observed in experiments [2], especially kinks with curved boundaries. In this study, we formulate the geometry of a curved kink microstructure by extending Inamura's model and clarify the stable shape of a curved kink by elastic energy minimization.

We modeled a curved kink by introducing a sequence of ortho-kinks parametrized by a vector $\mathbf{s}=(s_1,\ldots,i_s,\ldots s_{on})$ (called "shear vector", hereafter) into a ridge kink with uniform shear s_0, where i_s represents the uniform shear of the i-th internal ortho kink. And the inequality $s_0>s_1>s_2>\ldots>s_{on}>0$ is set to express the experimentally observed feature of ridge-kink such that a curved kink boundary is convex towards the exterior of the kink. For a given shear sequence **s**, we can analytically obtain the corresponding shape of the kink and the accompanying disclinations using Inamura's model. The elastic energy, E_{tot}, due to the accompanying disclinations is computed using Romanov's model in the framework of linear elasticity [3]. We define the elastic-energy parameterized by the shear vector **s** as $E_{tot}= E(\mathbf{s})$. Then, according to the principle of minimum potential energy, the stable shape of the kink, $\hat{\mathbf{s}}$, is obtained by the minimization of $E(\mathbf{s})$ as $\hat{\mathbf{s}}=\text{argmin } E(\mathbf{s})$.

As a result, it was found that the minimum value of $E(\mathbf{s})$, $\hat{E}$, decreases rapidly and then gradually converges to a certain constant as the number of the internal ortho-kinks, *n*, increases. In other words, from an elastic point of view, the curved kink with more internal ortho-kinks is preferred in the material. It is, therefore, deduced that the curved kink observed experimentally is formed as the relaxation of the disclinations. We also analyzed the geometry of the curved kink when $n\to\infty$, and it was in good agreement with the morphology of the curved kink in experiments, as shown in Fig. 1 (b).

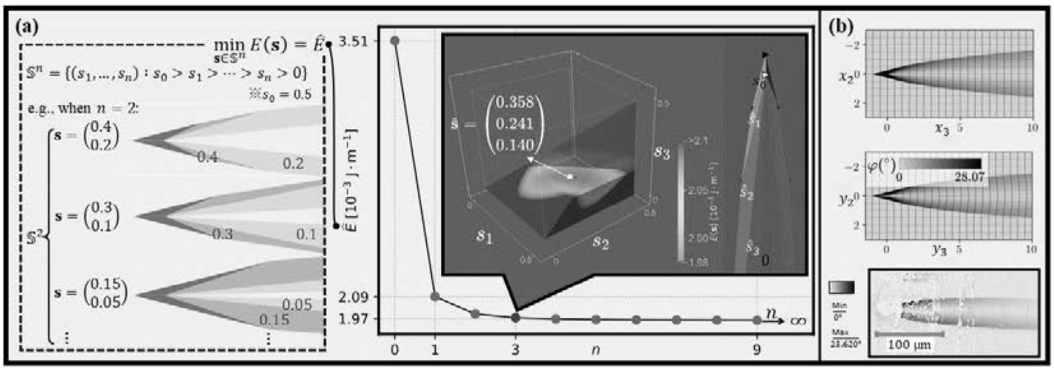

Fig. 1. (a) The plot of the number of the internal ortho kinks (*n*) and the minimum elastic energy $\hat{E}$. (b) Morphology of the curved kink ($n\to\infty$) with a microscopy image (bottom).

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Experimental Verifications of Kink-Band Strengthening in Long-Period Stacking Ordered Mg–Zn–Y Alloy

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Mg–Zn–Y alloys that contain a Mg-based long-period stacking ordered (LPSO) phase exhibit excellent mechanical properties because of the kink bands formed by plastic deformation. These properties have attracted significant attention as the kink-band strengthening [1]. Herein, we conducted deformation tests and electron microscopy to investigate the kink-band strengthening mechanism.

The master ingot was a LPSO single-phase directionally solidified $Mg_{85}Zn_6Y_9$ alloy. In the alloy, the *c*-axis and $<11\bar{2}0>$ directions of the LPSO phase were respectively almost perpendicular and parallel to the growth direction of the DS specimens. The plate-like specimen was approximately 3 mm × 4 mm × 1 mm ($W \times L \times T$), with the longer side of the specimen parallel to the direction of growth, that is, the 0° orientation. Kink bands were formed on the specimens by manual compression tests using a vice at room temperature; the loading axis was parallel to the 0° orientation. The surface relief of the kink bands was flattened by mirror polishing using a diamond suspension. Then, we performed double deformation tests with a second loading in the 45° orientation after a first compression in the 0° orientation. Simple compression (i.e., double compression) or tensile via bending was applied to the loading in the 45° orientation. To prove the existence of disclinations around the kink bands, the magnitude of the Frank vector $\boldsymbol{\omega}$ was estimated using high-angular-resolution electron backscatter diffraction (EBSD) measurements by scanning electron microscopy (SEM). The SEM image observations were conducted. These SEM images were used for digital image correlation (DIC) analyses to quantify the amount of plastic strain.

An EBSD analysis was performed to detect misorientations between the matrices across the kink bands. The existence of disinclinations was supported by the misorientations, that are the Frank vectors of the disclinations around the kink, and closely matched estimation results from the geometric analysis [2]. Moreover, the Frank vector increased by shear deformation (second loading in the 45° orientation) of the kink bands. The introduction of new disclinations around the kink may have resulted from shear deformation. The SEM observation demonstrated that the kink clearly obstructed basal <*a*> slips. This is definitive experimental evidence that kink bands behave as an obstacle to subsequent deformations, that is, we confirmed the kink-band strengthening phenomenon. When the shear deformation via tensile was applied to the kink, the DIC analyses indicated that the shear strains were concentrated in the kink bands. The shear strains on the left and right bands had different signs from each other. Moreover, when the shear deformation via compression was applied to the kink, the direction of shear strain applied was reversed from that of the tensile. The introduction of shear strains is considered the cause of the change in the Frank vectors, as described above.

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Deformation Behavior of Nb_2Co_7 as Crystal-Structure-Based Mille-feuille Structured Material

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The binary intermetallics, Nb_2Co_7, with layered mC18 crystal structure shows excellent compressive deformability and high strength [1,2] and is expected to be a new crystal-structure-based mille-feuille structured (MFS) material like the LPSO phase found in Mg alloys. However, deformation mechanism of Nb_2Co_7 still remains poorly understood. In the present study, deformation behavior and microstructural evolution of Nb_2Co_7 are investigated mainly focusing on the kink formation and kink strengthening phenomenon in Nb_2Co_7.

The Co-22.2at.%Nb alloy was vacuum induction melted, and was cut into 1.2×1.2×2.5 or 2×2×4mm^3 rectangular parallelepiped specimen. The samples were heat-treated at 1000°C for 50h to attain single-phase Nb_2Co_7. Uniaxial compression tests at room temperature were carried out with a strain rate of either 3.75×10^{-4} or 1.0×10^{-4} s^{-1}. Microstructure and crystallographic orientation relationship were investigated by SEM and EBSD before and after compression tests.

The compressive stress-strain curve of Nb_2Co_7 is shown in Fig. 1. Nb_2Co_7 shows plastic deformability and work hardening. Shown in Fig. 2. is the microstructure of the compressed sample with 0.2% plastic strain observed by SEM. The kink-like structure without delamination was observed on the side surface of the sample. The detailed EBSD analyses clarify that such kink-like structure forms along the basal (001) plane with the rotation axes of (hk0) and that the rotation angles are varied, which is important characteristics of kink. The excellent compressive deformability of Nb_2Co_7 could be achieved by operation of basal (001) slip, twinning and kink formation. It is thus concluded that Nb_2Co_7 is a new crystal-structure-based MFS material.

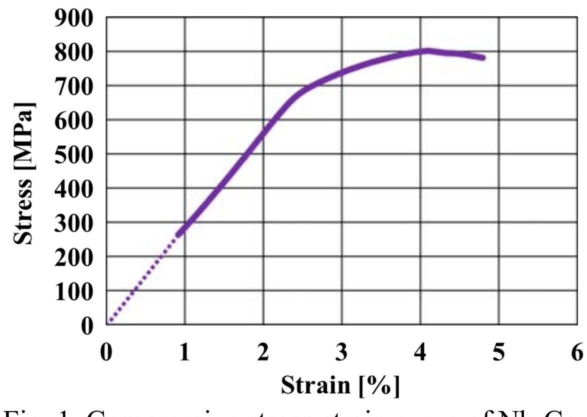

Fig. 1. Compressive stress-strain curve of Nb_2Co_7.

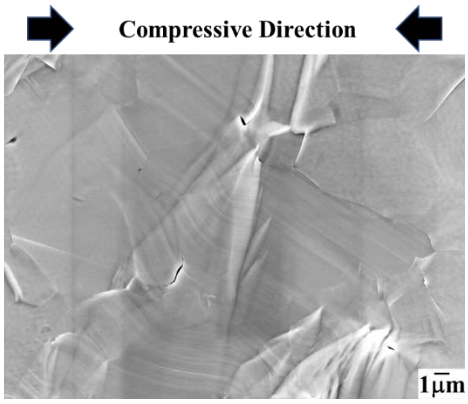

Fig. 2. SEM microstructure of Nb_2Co_7 after the compression of 0.2% plastic strain.

Acknowledgement

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Orientation dependence of various properties of textured MAX phase ceramics fabricated by slip casting under strong magnetic field

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MAX phase ceramics—ternary carbides or nitrides expressed by the general formula $M_{n+1}AX_n$ (M: transition metal, A: group A element, X: C and/or N, n=1,2,3) have unique properties that combine metallic (thermal and electrical conductivity, plastic deformability) and ceramic (thermal shock and oxidation resistance, low density). Since the properties of the MAX phases are anisotropic because of its hexagonal and layered structure, texturing the MAX phases can improve properties and add anisotropy[1]. In addition, the layered materials including the MAX phases can form a buckling structure called as kink band, which enables material strengthening.

In this study, therefore, we fabricated textured Ti_2AlC and Ti_3SiC_2 MAX phase ceramics using slip casting in strong magnetic field alignment followed by spark plasma sintering (SPS) [2,3]. Vickers hardness test, high-temperature compression, and high-temperature oxidation tests were carried out to evaluate the orientation dependence of room temperature deformation behavior, kink band formation, and oxidation behavior.

The slurry was made by mixing commercial MAX phase powder (Ti_2AlC or Ti_3SiC_2), ethanol as a solvent, and Polyethyleneimine as a dispersant. Green bodies were obtained by slip casting of the slurry under a rotating magnetic field for Ti_3SiC_2 and a static magnetic field for Ti_2AlC (Fig.1). A magnetic field B of 12T was applied. The green bodies were sintered with a SPS machine at 1150°C for Ti_2AlC and 1300°C for Ti_3SiC_2. For Vickers hardness tests, the sintered bodies were cut to an angle of 0~90° with c-axis. The orientation dependence of Vickers hardness was evaluated from the SEM images of the indent. For introducing the kink bands, each sample was compressed perpendicular to the c-axis at high temperatures, and the amounts of kink bands were evaluated using SEM-EBSD technique. High-temperature oxidation tests were conducted at 1100°C for 4 and 24 h in air.

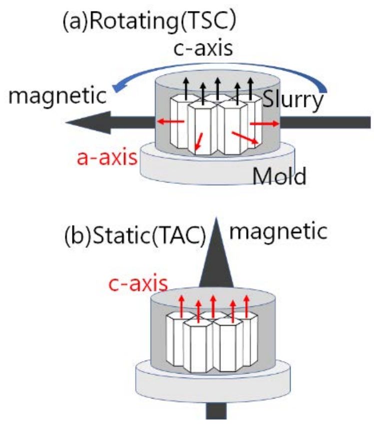

Fig. 1. Schematic of slip casting under(a) rotating and (b) static magnetic fields.

Fig. 2 shows the optical micrographs of Vickers indent tested in Ti_2AlC. Vickers indent shows anisotropic shape and hardness changed depending on the loading angle against the c-axis. Kink band formation was confirmed in the sample tested at high-temperature compression. The oxide scale growth rates were different between Ti_2AlC and Ti_3SiC_2, and its anisotropy was observed depending on the angle with the c-axis.

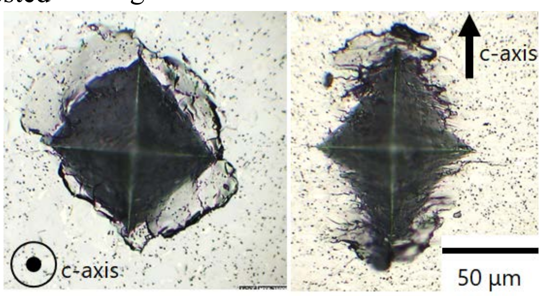

Fig. 2. Optical micrographs of Vickers indents in Ti_2AlC by loading 49 N parallel to *c*-axis (left) and perpendicular to *c*-axis (right).

Acknowledgments

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Mille-feuille Structures in Al-RE Eutectic Alloys and Its Kink Formation

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Introduction

In order to verify the occurrence of kink strengthening in the materials composed of two-phase lamellar structure, eutectic Al alloys with rare earth elements are prepared in the present study. FCC-Al forms eutectic lamellar structure with tri-aluminide phase Al_3RE such as Al_3Y and Al_3Er. By substituting Y in Al_3Y with Eu, the crystal structure of Al_3RE changes from $BaPb_3$ type to Ni_3Ti type to Al_3Ho type. Stacking sequence of each of them are denoted by hhc, hc and hchcc: it means Al_3(Y, Er) has partly HCP type stacking [1]. Because of the similarity of the crystal structure among fcc Al and these tri-aluminide phases, it was expected that the close-packed planes of Al and Al_3RE are the phase boundary of Al/Al_3RE lamellar structure. It may satisfy one of the fundamental ideas, i.e., the "Mille-feuille conditions": dislocations in the soft layer moves along the soft layer-hard layer boundary, because these tri-aluminide phases include partially hcp stacking which stop the motion of the dislocation on the plane not-parallel to the phase boundary of Al/Al_3RE lamellar structure. This crystallographic relationship between Al and tri-aluminide phases strongly restricts the dislocation motion in Al along only the phase boundary of Al/Al_3RE lamellar structure, which is similar with the dislocation motion in Zn only on the basal slip, resulting in the formation of kink in the lamellar structure.

Experimental procedures

In the present study several Al/Al_3RE two-phase alloys are prepared by Ar-arc melting machine from high purity elements. Alloy composition are Al-3 at.%(Y, Er) and the composition of the tri-aluminides are designed to be $Al_3Y_{1-x}Er_x$(x=0, 0.1, 0.5, 0.67, 1). Samples are cut, mounted and polished, then subjected to optical microscopic observation, SEM observation and composition analysis of phases by EDS, confirmation of crystal structure of phases by XRD, and investigation of crystallographic orientation relationship between Al and tri-aluminides using EBSD. Compression tests using the rectangular parallelopiped samples with the size of 2 x 2 x 4 mm are conducted at room temperature with a strain rate of 2.8×10^{-3} s^{-1}. The sample side surfaces are investigated before and after the compression tests using SEM to confirm the kink formation during the plastic deformation process.

Results and Discussion

A well-developed fine lamellar structure is confirmed to be formed in all of the alloys as the result of eutectic solidifications. The composition of primary phase together with the average composition of lamellar area enables us to establish the partial ternary phase diagram near the Al corner. EBSD analysis revealed that the close-packed planes of Al and beta Al_3Y are the phase boundary of Al/ beta Al_3Y lamellar structure, and kink formation during plastic deformations are confirmed in alloys.

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Fabrication of Al_2O_3-GAP eutectic ceramics with fine anisotropic microstructure by flash event and deformation behavior

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Remarkable mechanical strength has been observed in LPSO-Mg alloy by introducing kink band in the mille-feuille structure [1]. Aiming to apply this strengthening technique to structural ceramic materials, we attempted to fabricate multiphase ceramics with a pseudo-mille-feuille structure by using eutectic reaction. In the present study, we focused on Al_2O_3-$GdAlO_3$(GAP) eutectic ceramics, which are composed of a hard Al_2O_3 phase and a relatively soft GAP phase. We have successfully fabricated fine anisotropic Al_2O_3-GAP eutectic microstructure by using a flash sintering technique; flash sintering enables the almost immediate densification of green compact of oxide ceramics at a relatively low furnace temperature under an electric field above a critical strength [2]. Increased electric conductivity in oxide ceramics is another characteristic of the "flash sintering" events. The increased conductivity causes significant Joule heating and abrupt raising of specimen temperature. The plastic deformation behavior of the present eutectic sample was evaluated by micropillar compression tests.

Commercially available high-purity Al_2O_3 and Gd_2O_3 raw powders were mixed by ball-milling at the eutectic composition of Al_2O_3-23 mol% Gd_2O_3. The powder mixture was cold-isostatically pressed and calcined at 1450°C × 2 h in air. The dimension of the calcined sample was 1.4 mm × 3.8 mm in cross-section and 23 mm in length. The calcined sample was heated up to 1400°C in air, and a sinusoidal voltage of 1000 V/cm at a frequency of 1 kHz was applied from an AC power supply with the current limit of 110 mA for 40s, and then the external power supply was turned off. As a result, we successfully obtained anisotropic fine eutectic microstructure. From the eutectic region of the sample, micropillar with a diameter of 1 μm and a height of approximately 2.5 μm was fabricated by focused ion beam (FIB). For comparison, micropillar composed of Al_2O_3 single crystal was also produced from coarse Al_2O_3 single crystal grain in the same specimen with the [0001]Al_2O_3 axis 20° declined from the compression direction. Micropillar compression test was performed at room temperature at a constant strain rate (1×10^{-3} s^{-1}) using a nanoindenter equipped with a flat-end diamond tip. The microstructure was observed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

The flash event resulted in partial melting of the calcined sample through the current path, wherein an anisotropic eutectic microstructure was formed with rod-like GAP phases aligned in Al_2O_3 single crystals. The average interphase spacing was approximately 170 nm in the finest area, which was comparable to the finest spacing reported in the literature [4]. The TEM observation demonstrated that the orientation relationship of both phases in this area were [0001]Al_2O_3 // [010]GAP for the growth direction.

In the micropillar compression tests, the pillar of the Al_2O_3 single crystal showed brittle fracture at the compressive stress of 20 GPa, while the pillar composed of the Al_2O_3-GAP eutectic specimen showed plastic deformation of about 3 % with strain hardening after yielding at about 8 GPa. This result suggests that microscopic plastic deformation can occur in the high-strength ceramics by introduction of fine multiphase microstructure. Interphase delamination did not occur even after the plastic deformation.

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Effect of a high-pressure press on the strength and the morphorogy of PP

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【Introduction】

Polypropylene (PP) is an excellent crystalline polymer with ductility, good processability, and so on. However, the strength of PP is not enough. In order to be more widely used, the high strength of PP is needed. In development of high strength PP, there are many study by using the rolling. But, in the case of rolling, the stress at break is low in perpendicular to the rolling direction. In other words it is affected by the processing direction and becomes an anisotropic sample. In this study, we tried to increase the isotropic high strength of PP by using the high pressure press below the melting point. In addition, We studied the effect of high pressure press on the morphology of PP.

【Experiment】

The polymer used in this study was a EA9(iPP) made by Japan Polypropylene Corporation. The density is 0.90 g/cm^3. The melting point is 167 °C. Melt flow rate MFR=0.5 g/10min. A 2 mm thick PP sheet was produced for pelletized PP using a manual hydraulic heating press. The sheet compressed by 100 t lab machine. The compression temperature was 120 °C, the compression load was 30-1000 kN(43-1400 MPa)。 Mechanics test of sheet was tensile test. Tensile speed was 10 mm/min. We used SAXS, WAXS, OM, and TEM to analyze the hierarchical structure of the higher-order crystal structure.

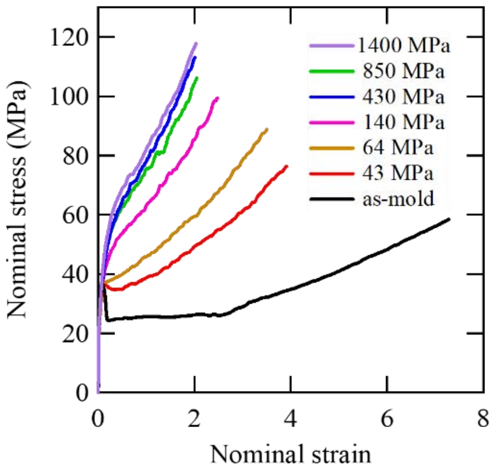

Figure.1. Tensile SS curves of as mold PP and PP pressed by pressures

【Result and discussion】

Figure. 1. shows tensile stress-strain curves of as mold PP and compressed PP by various pressure. The stress at break was more than 3 times higher than that of as mold samples. Also, the strength increases as the pressure increased.

Figure. 2. shows long period, lamella thickness and crystallinity obtained from SAXS and WAXS as a function of pressures. As mold PP is shown at pressure of 0 MPa. The long period increase but lamella thickness and crystallinity doesn't change by press. In addition, these parameters doesn't change as pressure increases. On the other hand, crystallinity of the compressed PP didn't change from as mold PP. In other words, the molecular chains of the amorphous phase affects the strength of PP.

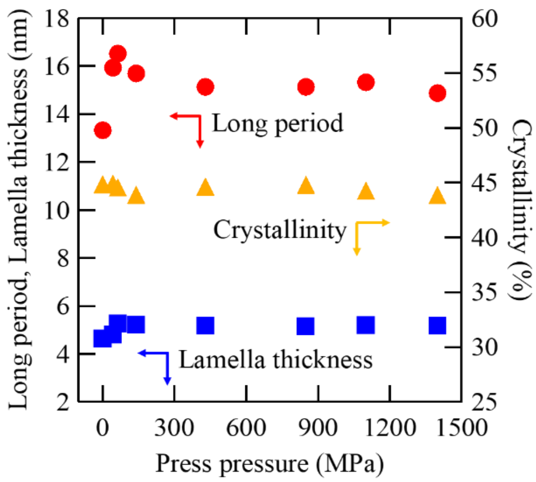

Figure.2. Long period, Lamella thickness and crystallinity obtained from SAXS and WAXS as a function of pressures

【Conclusion】

In this study, succeeded in increasing strength PP by compression. In addition, we understand, the reason for the strength of PP is that the molecular chains of the amorphous phase have an effect. The details of the relationship between high strength and higher-order crystal structure will be introduced on the day.

Effect of Compression Molding of Co-Extruded PS/PBT Multilayered Film on Crystal Structure and Mechanical Properties

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Kink strengthening phenomenon has been firstly discovered in a long-period stacking-order (LPSO) structured Mg alloy that revealed unusually high strength beyond theoretical predictions. The LPSO structure can be generally observed as a mille-feuille structure, which is formed by alternate stacking of microscopic hard and soft layers. The Kink strengthening phenomenon has been recently expected to strengthen polymer materials. This research aimed to develop a mille-feuille structure based on reinforced polymer materials and to investigate the effect of compression molding on the mechanical and structural properties of the mille-feuille polymer.

Polystyrene (PS) and polybutylene terephthalate (PBT) were utilized as a hard and soft layer, respectively. The PS containing oxazoline groups was used to improve the interfacial adhesion between PS and PBT. A 32-layered PS/PBT film and a 16-layered PBT film were prepared using a multilayer co-extruder. Each of ten-layered film was adhered using a heat press to produce a 320-layered PS/PBT sheet and a 160-layered PBT sheet. Finally, the fabricated sheets were pressed under various high pressures at 60 °C. Small-angle X-ray scattering (SAXS) and transmission electron microscope (TEM) were performed for structural observation. Differential Scanning Calorimetry (DSC) and Wide-Angle X-ray Diffraction (WAXD) were performed for structural analysis. Three-point bending test was performed to evaluate toughness properties.

Fig. 1 shows 2D SAXS patterns of the edge direction of the samples and TEM images in PBT layer. From the 2D SAXS patterns, a typical pattern was observed from the virgin sheet, meanwhile, four characteristic patterns were observed after the compression molding process. This could be assumed that folding structures were formed in the lamella structure within the PBT. From the TEM images in PBT layers, it was confirmed that the lamellae in the PBT layer was folded, and kink structures were formed in both sheets. It was also confirmed that the angle of folding was different between the 320-layered PS/PBT sheet and the PBT 160-layered sheet. **Fig. 2** shows the relationship between toughness obtained from a 3-point bending test. Although the mechanical properties of PS/PBT were affected by the load, no direct correlation of the SAXS patterns and TEM images were observed.

These results indicated that compression molding formed folding structures in the lamellae of PBT, and the angles of folding structures of PS/PBT and PBT were different. Furthermore, compression molding affects the mechanical properties and crystal structure of mille-feuille polymer sheets.

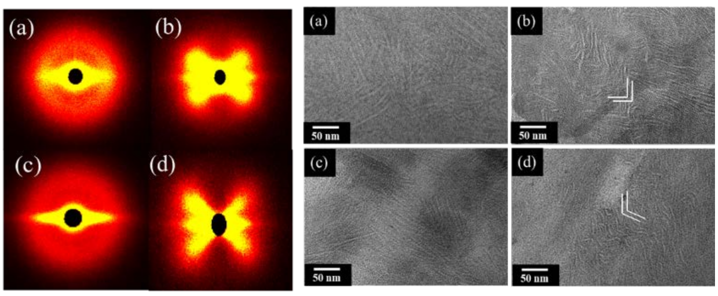

Fig. 1 2D SAXS patterns and TEM images in PBT layer,
(a) PS/PBT before compression, (b) PS/PBT after compression
(c) PBT before compression, (d) PBT after compression

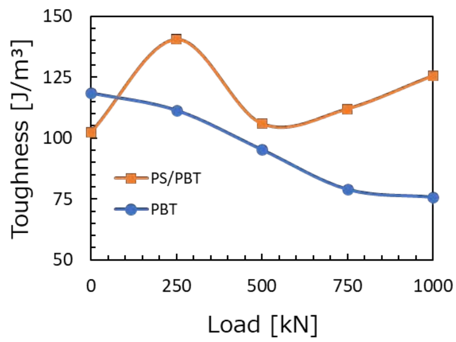

Fig. 2 relationship between pressing load and toughness

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Changes in the Elastomeric Mechanical Properties of Lamellae-Forming Block Copolymer Specimens by Introduction of Kink

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[**Introduction**] In recent years, "kink strengthening phenomenon" has been discovered in the field of the metallic alloy. Kink is a region where crystals are sharply bent. We consider the possibility of applying this concept to polymeric materials and are focusing on block copolymers that form lamellar microdomains consisting of hard and soft components. Previous investigations in our laboratory have used non-oriented lamellar microdomains, and the kink was introduced by uniaxial stretching, a method that destroys the internal structure. To date, material strengthening through introduction of kinks has not been achieved. In this study, we focused on the changes in the elastomeric mechanical properties of the block copolymer by the introduction of kink and examined whether the kink strengthening is realized.

[**Experimental**] The specimen used for this study is a polystyrene-*block*-polybutadiene-*block*-polystyrene triblock copolymer. The S/B volume ratio of this sample is 56/48. As for the molecular weight, Mn = 6.31 x 10^4 and Mw/Mn = 1.15. The specimens were cast from solutions using the selective solvents (mixtures of *n*-heptane and dichloromethane, which are selectively good solvents for PB (polybutadiene) and for PS (polystyrene), respectively). Note that the mixtures of *n*-heptane and dichloromethane were used for more precise control of the degree of solvent selectivity to PS and PB. To analyze the nano structures in the as-cast and annealed specimens along with the uniaxial stretching, two-dimensional small-angle X-ray scattering (2d-SAXS) measurements and tensile test were simultaneously conducted at room temperature. The measurements were performed mainly at BL-15A2 of Photon Factory at High Energy Accelerator Research Organization (KEK). The X-ray wavelength was 0.118 nm and the camera length was 3200 mm. A PILATUS2M was used as the 2-dimensional detector. The tensile test was performed with an initial chuck distance of 10 mm. Tensile speed was 6 mm/min.

[**Results and Discussion**] The stress-strain curves shown in Fig. 1 were obtained, based on the five specimens results of the off-line SS measurements in our laboratory. Fig. 1 indicates that the kinked material (the curve(a)) was not clearly strengthened. Although it shows the highest stress of the early stage of the strain among all specimens, the specimen was not elongated well so that the elongation at break is not high enough and either the stress at break was not high as compared to the curve (c). Note here that the kink formation was detected by the simultaneous SAXS measurements for the case of the curve (a) during the period where the stress remained constant due to the macroscopic necking of the specimen. The stress level at this stage may be related to the fracture of the hard (glassy) PS lamellae. It is interesting to find that the final strain of the necking depends on the individual specimens. As for the D0 specimen (the curve (a)), the stage of the constant stress level (necking) was shortest among all specimens. To understand what determines this length, the results of the simultaneous SAXS measurements were helpful. As a result, the manner of the fracture of the PS domains differs depending on the microdomain orientation. The details will be explained on site. It is also noteworthy that the slope of the SS curve at the later stage (successive to the stage of the constant stress level) is almost similar to each other for all specimens examined in this study. Although it may be considered that the rubbery PB chains mainly contributes to the stress as such rubbery polymer chains are elongated with the aid of the fragmented PS microdomains which play a role of the physical crosslinking points, the SS curve in the reversing process (by releasing the load from the specimen) was found to be not coincident with the forwarding process. Actually, the reversing process exhibited much lower stress. This clearly reminds the more proceeding of the fracture or crazing of the fragmented PS domains in the microscopic level. The correlation between these mechanical properties and nanostructural changes will be reported also on-site.

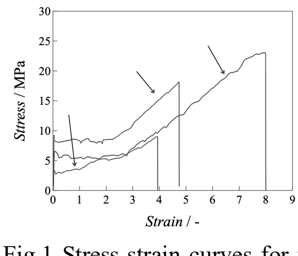

Fig.1 Stress-strain curves for the as-cast specimens. The curve (a) is for the specimen prepared by the solution cast using dichloromethane. The curve (b) is for the specimen prepared by the solution cast using solvent mixture of *n*-heptane / dichloromethane (25:75) and (c) is for the case of the mixture of (75:25).

Strengthening mechanism of crystalline polymer by heat elongation

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Crystalline polymers have hard lamellae crystalline layers and soft amorphous layers, and they are alternately stacked to form a layered microstructure termed as the lamellae stack. The material properties are expected to be improved by controlling the lamellae stack structure.

In recent years, it has been reported that some crystalline polymers become strengthened by heat elongation. In the case of high-density polyethylene (HDPE) [1], the heat elongation process significantly increases yield strengths of the HDPE whose degree depends on the heat elongation ratio. X-ray diffraction analysis using HDPE suggests that this strengthening originates from the alignment of lamellae stacks orientations and shortening of lamellae crystal length [1], but details of the microstructure and the strengthening mechanisms are still unclear.

In this study, we investigated the microstructure of HDPE based on transmission electron microscopy (TEM). We prepared the extruded HDPE films by blown film extrusion process, and the elongated HDPE films were obtained by heat elongation of extruded films at 373 K up to various elongation ratio. The samples were thinned by the microtome method, stained with ruthenium tetroxide solution at room temperature, and subjected to TEM observation.

Fig. 1 shows TEM images acquired from the extruded HDPE film, and inverse Fourier transformed image reproduced from a specific frequency domain in the power spectrum. Since staining with ruthenium tetroxide preferentially introduces heavy metals into amorphous materials, we identified that bright contrasts in the TEM image correspond to crystalline layers in the lamellae structure. Inverse Fourier transformed image with contrast enhancement corresponding to lamellae stack confirms that lamellae stacks form an oriented microstructure in the extrusion direction of the extruded HDPE. After headt elongation with a ratio of 400%, the lamellae crystal length decreased while the orientation of the lamellae stacks increased (Fig. 2).

The above results are consistent with the structure predicted from small-angle X-ray scattering reported previously [1]. We will discuss details on the deformation mechanisms of lamellae stack before and after heat elongation in the presentation.

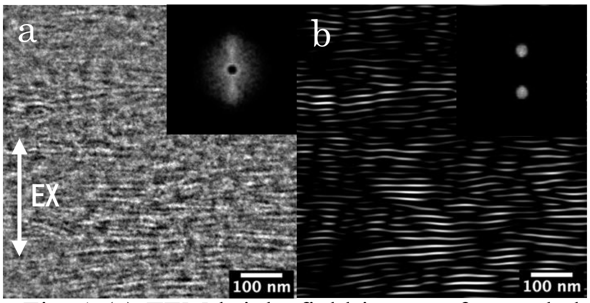

Fig. 1 (a) TEM bright field image of extruded HDPE and its FFT pattern. EX corresponds to the extruded direction. (b) Inverse FFT image of the specific frequency domain.

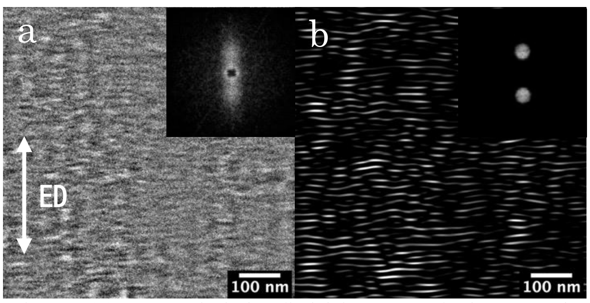

Fig. 2 (a) TEM bright field image of elongated HDPE (elongation ratio: 400 %) and its FFT pattern. ED corresponds to the elongation direction. (b) Inverse FFT image of the specific frequency domain.

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~MEMO~

Development of hcp/bcc layered structure in Mg-Sc alloy and kink formation by compression

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Recently, high strength Mg alloys with Long-Period Stacking Order structure (LPSO-Mg) were developed and their high strength was reported to be due to kinking at the LPSO phase which consists of multilayer of hard layers and soft layers [1-3], called mille-feuille structure. Therefore, to obtain kink strengthening in other alloy, the investigation for how to develop mille-feuille structures and how to form kink in the obtained mille-feuille structures is required.

According to Mg-Sc binary phase diagram, bcc phase is stable in high temperature region and hcp phase is stable in low temperature region at the Sc content of over around 18 at%. In fact, the microstructure of Mg-Sc alloy is known to be controlled by heat treatment through phase transformation [4]. Layered hcp/bcc structure is reported to be obtain in Ti alloy which shows hcp/bcc phase transformation as with Mg-Sc. That hcp/bcc layered structure in Ti alloy is formed though cold rolling and subsequent heat treatment for the process of hcp precipitation [5]. Based on these backgrounds, we investigated the microstructure of Mg-Sc developed by cold rolling followed by heat treatment and kink formation in the developed hcp/bcc layered structure in Mg-Sc.

In this study, the Mg-Sc ingot with the nominal composition of Mg-30 wt%Sc was used. The semi-circle shaped plate with the thickness of about 25 mm was cut from the ingot and that plate was hot rolled at 873 K to obtain 5 mm thick plate. Then, the hot rolled plate was annealed at 963 K for 0.5 h followed by water quenching to obtain bcc single phase. The bcc single phase plate was cold rolled and then heat treated for the hcp precipitation. Compression test was carried out for the kink formation with a strain rate of 10^{-2} s^{-1}. The microstructure was observed by optical microscope and SEM.

After cold rolling with a thickness reduction of 5~10% followed by annealing at 893 K for 10 min, the hcp/bcc layered-like structure in which the precipitated hcp phases glowed particular direction in each bcc grain was obtained (Fig.1). The precipitated hcp and bcc had a Burgers orientation relationship and the hcp was found to preferentially precipitate at the slip lines which formed in bcc during cold rolling.

By 20% compression at room temperature, kinking was observed at a part of region of hcp/bcc layered sample.

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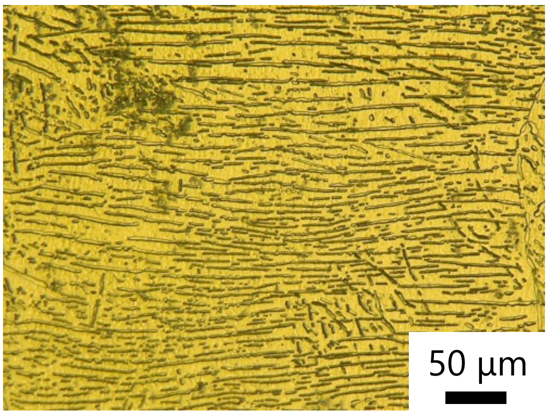

Fig. 1. Optical microscope image of Mg-Sc 5% cold rolled followed by annealing at 893 K for 10 min.

Effect of Additional Element on Nonflammability of LPSO type Mg-Zn-Y Alloy

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Mg alloys have been attracting keen attention as promising lightweight materials for aerospace, automobile, and railway applications. On the other hand, it is often pointed out that Mg alloys have poor oxidation resistance and burn easily. This flammability of Mg alloys is a problem to be solved when we consider using Mg alloys as structural materials of mass transportation vessels. In fact, the Federal Aviation Administration (FAA) in the USA banned the use of Mg alloys for aircraft cabins. However, from point of view of reducing aircraft weight, FAA decided to lift the ban on using the Mg alloy in an aircraft cabin and set up a flammability test for Mg alloys. As a part of development of nonflammable Mg alloy, some metal elements have been added into Mg alloys. It has long been known that the addition of rare earth elements can improve incombustibility of the surface of the oxide film on Mg alloy [1-4]. Among the RE-containing Mg alloys, Mg-Zn-Y alloy with a long period stacking order (LPSO) phase has excellent mechanical properties and is expected to be used in aircraft components. LPSO-type Mg-Zn-Y alloy produced by rapidly solidified powder metallurgy has an extremely high yield strength of ~600 MPa. LPSO-type Mg-Zn-Y alloy produced by ingot metallurgy and extrusion has a multimodal microstructure, and high yield strength of ~400 MPa [5, 6]. In addition, the $Mg_{97}Zn_1Y_2$ alloy exhibits an ignition temperature of 1150 K [7, 8]. However, this ignition temperature is lower than the FAA oil burner temperature, so further improvement in ignition temperature is required to safely use LPSO-type Mg-Zn-Y alloys. It has been reported that the addition of some elements such as Be, Yb, Ca, and Sr improved the ignition temperature of Mg alloys [8-10]. In this study, we attempted to improve the ignition temperature of LPSO-type Mg-Zn-Y alloys by addition of Be, Yb, Ca, and Sr.

Mg-Zn-Y-X alloys (X = Ca, Yb, Be, and Sr) were prepared using high-frequency induction melting in Ar atmosphere. Ignition temperature measurement was performed using TG/DTA. The heating rate was 50 K/s. Specimens were heated at 973 K in a muffle furnace in air. For investigating the structure of oxide films, XRD measurement, SEM, and TEM observation were conducted on a cross-section of the film formed on the Mg-Zn-Y alloys.

With the addition of Ca, Yb, Be, or Sr, the Mg-Zn-Y alloys exhibited an ignition temperature of approximately 1300 K. XRD measurement and SEM observation revealed that the surface film of the alloy was mainly composed of Y_2O_3. In the case of Ca or Yb-containing alloys, outer CaO and Yb_2O_3 layer formed on the Y_2O_3 film. Furthermore, Be and Sr were concentrated in the Y_2O_3 film [8]. Thin and uniform Y_2O_3 film without internal oxidation of Y was observed in Ca, Yb, Be, or Sr-containing Mg-Zn-Y alloys from SEM observation. This suggests that addition of Ca, Yb, Be, or Sr, helps to form high protectivity Y_2O_3 film on the surface of Mg-Zn-Y alloy by suppressing internal oxidation and abnormal growth of Y_2O_3 film.

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Creep characterization of a long period stacking ordered $Mg_{85}Zn_6Y_9$ alloy

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[Introduction] A new type of Mg alloy, with a mille-feuille structure, is stronger than the conventional alloys due to the laminated structure of the soft and hard layers, and the introduction of a deformation band called a kink band (KB). Hagihara et al. showed that a KB introduced during deformation provided high resistance for dislocation motion in the temperature range from room temperature to approximately 300 °C. [1)] In addition, nanoindentation tests at room temperature were conducted on KBs, which determined that a KB contributed to a higher hardness level [2)] or KB played the role of relaxation sites for dislocations [3)]. In this study, we evaluated the creep properties of the $Mg_{85}Zn_6Y_9$ alloy via an indentation technique at 250 °C and 300 °C and investigated the role of KBs in creep deformation.

[Experimental Methods] A rectangular piece of the unidirectional solidified (DS) $Mg_{85}Zn_6Y_9$ alloy was cut and shaped to approximately 3 × 8 × 2 mm via emery polishing. KBs were introduced by rolling the alloy in the 2 mm-thick DS direction at a reduction ratio of 0.2. The indentation creep tests were conducted at the test surface (2 × 8 mm) after the rolled alloy was polished with emery paper. Subsequently, the alloy was annealed at 320 °C for 24 h and then polished with colloidal silica to remove the surface oxide layer. The indentation creep tests were conducted at 250 °C and 300 °C at the indentation creep rates from 10^{-3} to 10^{-6} s^{-1} using a microindenter (Advance Riko, Inc.)

[Results] Figure 1 shows the stress dependence of the indentation creep rate at 250 °C and 300 °C. For the high creep rates at 250 °C, the stress exponent, *n*, which was the slope of the data, was equal to 9. When the creep rate was low, the stress dependence of the indentation creep rate was weak. This result indicated that there was limited stress for deformation such as threshold stress. For 300 °C, the *n*-value from 1 x 10^{-3} to 4 x 10^{-5} s^{-1} is similar to that of the low creep rates at 250 °C. However, in the low creep rates at 300 °C, the stress dependence of the indentation creep rate was more pronounced and the *n*-value was approximately 3.5. According to the conventional creep theory, the *n*-value of 3.5 demonstrated that the dislocation motion was the creep-rate controlling mechanism; therefore, the KB had a different effect on the dislocation motion at 250 °C compared to 300 °C in this experiment.

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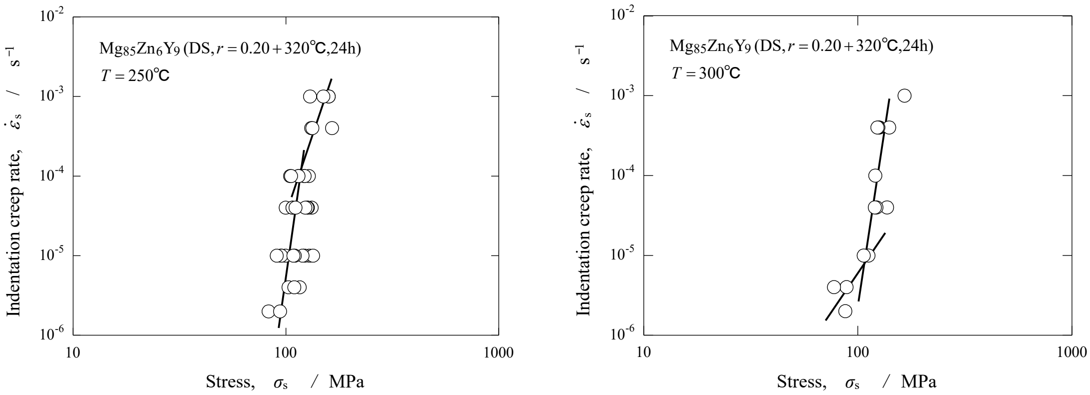

Fig. 1. Stress dependence of the indentation creep rate

Observation of Kink Deformation in an LPSO $Mg_{85}Zn_6Y_9$ Alloy using Two-directional Micro-Laue Diffraction Mapping under Compression

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Ternary magnesium (Mg)–transition metal (TM)–rare earth (RE) alloys with long-period stacking-ordered (LPSO) structures, which are formed by synchronizing structural modulation and chemical enrichment of TM and RE atoms, are paid much attention as lightweight structural materials because of their extraordinary mechanical properties such as high strength and high ductility [1-6]. These excellent mechanical properties are believed to be due to the unique deformation behavior—referred to as “kink deformation”—of the LPSO phase [5].

Synchrotron-radiation (SR) micro-Laue-diffraction (MLD) mapping is a unique technique for visualizing grain boundaries with 10-μm-order spatial resolution [7,8]. MLD mapping can reveal internal grain boundaries, which cannot be observed by electron backscatter diffraction (EBSD). It is thus a key means of studying deformation of those boundaries.

To investigate the deformation behavior of the LPSO phase of Mg polycrystal, we developed a compact compression-test stage for SR-MLD measurements, which can compress a small sample with a size of 0.3 mm × 0.3 mm× 1.0 mm. The compression-test stage was used for SR-MLD mapping experiments at the white x-ray diffraction beamline (BL28B2) of SPring-8. In the experiments, change in the grain boundary was successfully visualized by changing the compression load [9]. However, depth information could not be obtained because projection images could be obtained from only one direction.

Aiming to obtain that depth information, in the present work, we therefore modified the compact compression-test stage so that grain-boundary images could be taken from two orthogonal directions. As a result of this structural modification, it became possible to obtain depth information concerning the kink-formation position. Also, change in surface morphology of the sample could be observed simultaneously by using an optical microscope to compare bulk and surface images.

Comparing the grain-boundary images with the surface-morphology photographs revealed kink deformation in the grain-boundary images but no change in the surface-morphology photographs. This observation demonstrates the advantage of SR-MLD measurements compared to the surface sensitive measurements such as optical microscopy, scanning electron microscopy, and EBSD.

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Phase transformation behavior of LPSO structure during annealing investigated by *in-situ* neutron diffraction

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Recently, a new class of dispersion strengthened magnesium alloys has attracted high attention due to their excellent mechanical properties [1]. A long period stacking ordered (LPSO) phase in these alloys contributing to the enhanced strength and ductility has been clarified. The LPSO phase includes polytypic structures such as 14H, 18R, 24R, and 10H [2]. These polytypic LPSO structures can coexist with other precipitations in the Mg–RE–TM system depending on chemical composition and temperature. In order to clarify the stability and the structure evolution of LPSO phase at high temperature, an as-cast $Mg_{85}Zn_6Y_9$ alloy with duplex LPSO structure of 18R and 10H was investigated by *in-situ* neutron diffraction during annealing. *In-situ* neutron diffraction experiments were performed by BL19 'TAKUMI' of Japan Proton Accelerator Research Complex (J-PARC). Fig.1(a) shows the geometrical arrangement of the *in-situ* heating test. A cylindrical specimen with an 8-mm diameter and 16-mm length was set up on the high temperature frame horizontally, with its length direction oriented at +45 to the incident neutron beam. Two detector banks with 5-mm-wide radial collimators positioned at +90° and −90° relative to the incident beam were used to collect the neutron diffraction patterns, which correspond to the axial and radial directions of the specimen, respectively. Fig.1(b) shows the heating history, that the specimen was heated to 300 °C at a rate of 5 °C /min and then up to 550 °C at a rate of 2 °C /min for 2 hours isothermal holding followed by furnace cooling. Neutron diffraction profiles during the whole process were continuously collected in an event recording mode by two detector banks simultaneously. The temperature was measured by a thermocouple attached to the specimen. A 10H-LPSO to 18R-LPSO structure transformation was observed during heating. The evolutions of phase fraction and lattice parameters were estimated quantitatively from the diffraction profiles, and these results will be discussed to reveal the phase transformation behavior in the as-cast $Mg_{85}Zn_6Y_9$.

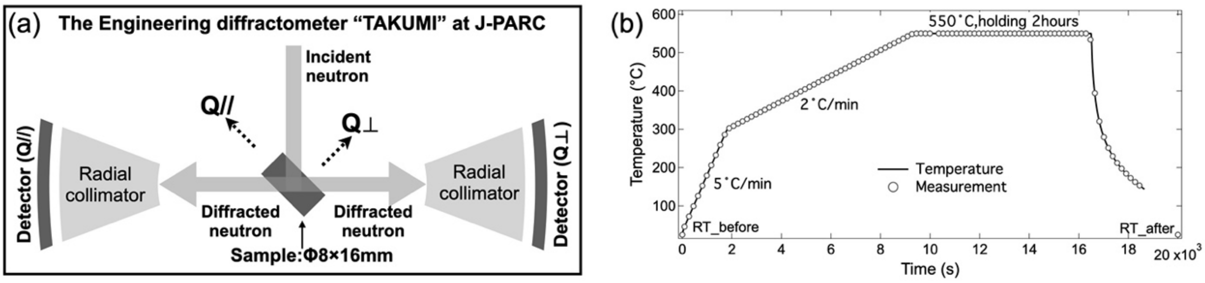

Fig. 1 (a) Geometrical arrangement of *in-situ* neutron diffraction experiment at TAKUMI, J-PARC. (b) Temperature history of the heating test.

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Development of 4D in-situ observation system for the investigation of kink development during compression of Mg-based LPSO alloys

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A new setup was developed for 3D observation of kink development in Mg-based LPSO alloys utilizing sample load apparatus and grain boundary imaging with synchrotron radiation X-ray CT, to investigate the relationship between formation of kink structure and strain and stress. Using this setup, $Mg_{85}Zn_6Y_9$ material was compressed, and kink development was observed as a time series of CT data (4D, 3D + time series).

Figure 1 shows the images of the sample load apparatus. Rotating stages above and below the specimen rotate synchronously. The specimen was compressed by the Z-stages which also located above and below the specimen. This configuration allows the specimen to be rotated for CT observation with compressed. Experiments were carried out using 40 keV X-rays generated by a multilayer mirror at BL20B2@SPring-8. The X-ray beam monitor includes a CMOS camera (ORCA Lightning, Hamamatsu Photonics) with 4608x4608 pixels. The number of CT projections was 3600, and the CBP method was used for image reconstruction. The specimen was a piece of $Mg_{85}Zn_6Y_9$, cut into 0.2 mm thick and 1.5 mm x 2 mm. The experiment was performed in step by step flow. The specimen was observed by CT, then compressed along with observation with transmitted X-ray image. Then the CT observation was performed again. The specimen was compressed 3 steps with 10 μm for each step.

Figure 2 shows the result of the 30 μm compression. Formation of a kink on the surface of the specimen (righthand-side of the figure) could be observed. The kink structure was also observed inside the specimen, through the bending of grain boundaries. The direction of the grain boundary bending indicates that this structure was formed by the movement of the material to the left in the figure. In contrast, another kink was observed on the back side of the specimen surface but same position, which formed through the movement of the material to the right. This is opposite direction to that of front side. The results indicate that the kink structures in this specimen occurred by pushing materials outward from the sample surface by compression. Because the material was extruded in opposite directions from both sides of the thin specimen, kinks on the front and back surfaces show opposite direction. Thus, multiple kink structures were formed in correlation.

This experiment demonstrated that the new 4D in-situ observation system has sufficient capability to observe kink development. In future improvement, a load cell will be introduced to enable more quantitative analysis. In addition, we plan to apply the digital volume correlation method (DVC) to automatic recognition of kinked structure from CT data to enable statistical discussions.

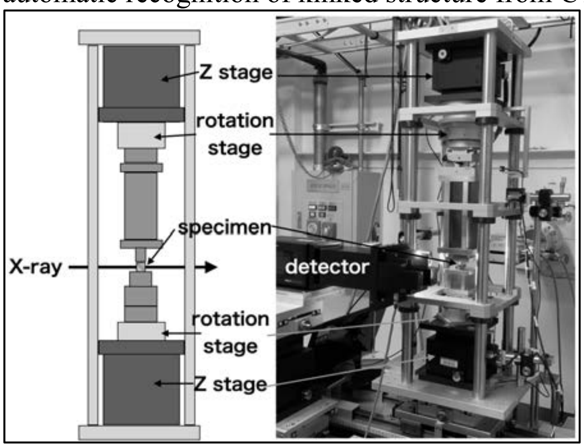

Fig. 1 Schematic illustration and an image of sample load apparatus.

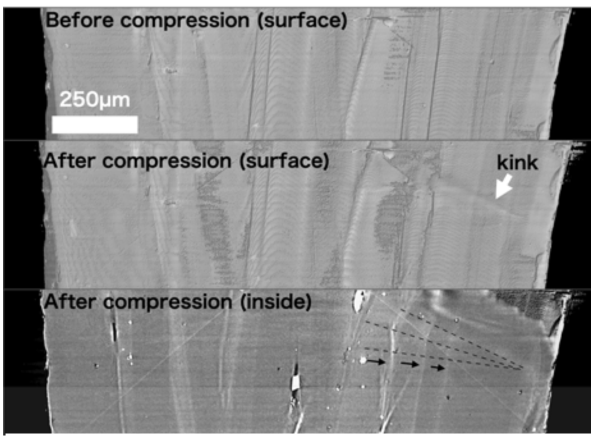

Fig. 2 CT images of specimen. Dashed lines show shape of kink structure and black arrows show bended grain boundaries.

Electronic structure of Mg-Zn-Y cluster in dilute Mg alloys studied by STEM-EELS

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In solute enriched stacking faults (SESF) in Mg-Zn-Y alloys with a long-period stacking ordered (LPSO) structure, the formation of Zn_6Y_8 clusters was confirmed [1]. Recently, synthesis of a single-crystal Mg alloy with dilute solute of Zn and Y has been reported, where SESFs are sparsely distributed in the hcp-Mg matrix [2]. Although clusters composed of Zn and Y atoms appear to exist in the dilute Mg-Zn-Y alloy, the cluster structure has not been determined using usual structural analysis methods because of random arrangement of clusters due to the low concentration in SESF. Recently, structural analysis using X-ray fluorescence holography (XFH) has been carried out and the presence of $Mg_3Zn_3Y_8$ cluster structure in dilute Mg-Zn-Y alloy has been proposed. On the other hand, the electronic structures of the clusters in the dilute and the LPSO Mg-Zn-Y alloys has not been investigated. In this study, electron energy-loss spectroscopy (EELS) combined with scanning transmission electron microscopy (STEM) was applied to the dilute ($Mg_{99.2}Zn_{0.2}Y_{0.6}$) and LPSO ($Mg_{75}Zn_{10}Y_{15}$) Mg alloys to clarify the electronic structures of the clusters in SESFs for both Mg alloys. In particular, the momentum transfer (**q**) resolved EELS measurements have been carried out to investigate the anisotropic electronic structure in dilute alloys.

Fig. 1A shows the Y-L_3 absorption spectra obtained from SESF of $Mg_{99.2}Zn_{0.2}Y_{0.6}$. The spectra were shown for $\boldsymbol{q}$ parallel to the *ab*-plane ($\boldsymbol{q}//\boldsymbol{ab}$) and *c*-axis ($\boldsymbol{q}//\boldsymbol{c}$) directions of the hcp structure. The spectrum of $\boldsymbol{q}//\boldsymbol{ab}$ has an additional structure around 2080 eV, which was not observed for $\boldsymbol{q}//\boldsymbol{c}$, indicating the anisotropic feature of the Y-L_3 spectra. The spectral simulations using the cluster structure model ($Mg_3Zn_3Y_8$) proposed by STEM and XFH analysis also showed anisotropy feature. On the other hand, the Y-L_3 spectra (Fig. 1B) obtained from $Mg_{75}Zn_{10}Y_{15}$ were almost isotropic. These results suggest that the electronic structure of the cluster of the dilute Mg alloy is different from that of the Zn_6Y_8 cluster. Furthermore, the difference of electronic structures between two Mg alloys indicates that the cluster structure of the dilute Mg alloy should be different from the Zn_6Y_8 cluster in the LPSO Mg alloy. The fact that the ELNES simulation in Fig. 1A agrees with the experiment indicates that the cluster model ($Mg_3Zn_3Y_8$) is a valid structure.

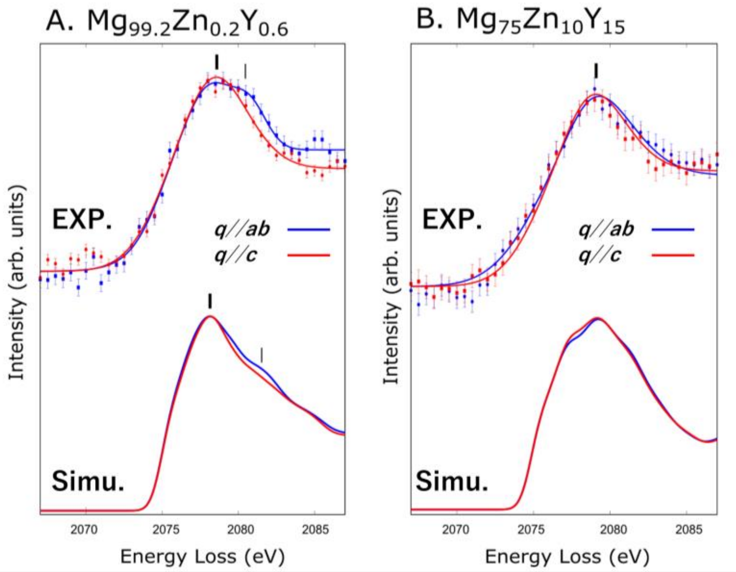

Fig.1A: Anisotropic Y-L_3 spectra of $Mg_{99.2}Zn_{0.2}Y_{0.6}$. Momentum transfer $\boldsymbol{q}$ were directed along *ab* in-plane direction ($\boldsymbol{q}//\boldsymbol{ab}$) and along *c*-axis direction ($\boldsymbol{q}//\boldsymbol{c}$). Fig 1B: Isotropic Y-$L_3$ spectra of $Mg_{75}Zn_{10}Y_{15}$.

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DFT Calculation of Kink Boundary Migration in LPSO Structure

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Kink boundaries formed in Mg-based long period stacking order (LPSO) alloys play a key role in strengthening of these materials. As the kink structure grows, many high-angle kink boundaries are eventually formed which has inclination angle close to 34 degrees. We show that this peculiar structure is a result of irreversible structural transformation and is energetically stable. We also calculate segregation energies of alloying elements Y and Zn to this boundary. Segregation energy is greater for Zn case and the maximum value is about 0.7eV. Finally, the critical resolved shear stress for the migration of kink boundary is estimated for a pure-Mg kink and that with saturated with segregation. We show that segregated kink boundary requires very high shear stress about 700 MPa for migration.

Figure 1(a) shows high-angle kink boundary saturated with Zn and Y atoms, which occupy segregation sites estimated by DFT calculation. The color of atoms indicates stacking order A, B and C in the 18R structure as shown in Fig.1(b), and apparent asymmetry with respect to the boundary can be seen. This asymmetry is induced by irreversible relaxation of the boundary structure. Fig.1(c) shows stress-strain relation for the applied shear stress acting on the kink boundary. Without solute atoms, the critical resolved shear stress (CRSS) for boundary migration is about 400 MPa, whereas saturation of solute atoms increases the CRSS to about 700 MPa. In the actual LPSO structure where hard solute clusters prevent local shuffling of atoms, the CRSS for boundary migration should be much higher and expected to be irrelevant to plastic deformation.

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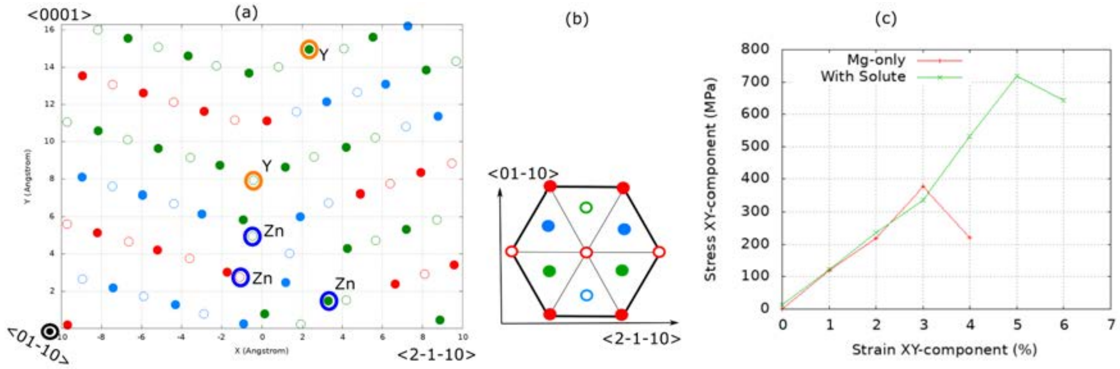

Fig.1 (a) Atomistic configuration of 34-degree kink boundary in 18-R LPSO structure. Colors of atoms indicate their coordinate in the direction vertical to the figure, as shown in (b). (c)Stress-strain relation for the applied shear stress acting on the kink boundary with and without solute atoms. The drops in stress correspond to migration of kink boundary.

First-Principles Study on the Electronic Origin of Phase Stability in Mg-Zn-Y alloys with a Long-Period Stacking Order

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A dilute Mg-1Zn-2Y (at%) alloy exhibits a tensile yield strength of ~600 MPa [1]. This strength is coupled with the appearance of a unique crystal structure called the long-period stacking order (LPSO), where solute atoms of Zn and Y are enriched in a few layers of the (0001) plane of the hexagonal close-packed Mg matrix, and Shockley partial dislocation occurs in the solute atoms enriched layer. The stacking faults order periodically, and their stacking sequence is relatively long along the [001] direction of the hexagonal lattice [2]. Furthermore, structural analysis using transmission electron microscopy (TEM) revealed that $L1_2$-type clusters of solute elements are embedded in the Mg matrix near the stacking fault [3]. This study examines the origin of the phase stability of the 18*R* LPSO structure using first-principles density-functional theory calculations. Generally, the density of states (DOS) of metals with an hcp structure is characterized by a deep valley (pseudo gap) around the Fermi energy [4,5]. The depth of the valley in DOS represents the magnitude of splitting of the peaks in the occupied and unoccupied states, which is an energy separation between the bonding and antibonding orbitals [4]. By comparing the electronic structures between hcp Be and hcp Mg, Inoue and Yamashita suggested that the degree of energy splitting between the two main peaks (the width of the DOS valley) appears as a difference in the enthalpy of formation between the metals [5]. In this study, the heat of formation as a function of the number of Zn vacancies is calculated to evaluate the role of Zn atoms. The calculated convex hull indicates that the Zn atoms in LPSO are stable even at about half of the Y atoms. The partial density of states of Mg atoms nearest to the Zn atoms forms a valley structure due to the bonding state with Zn atoms, leading to the stability of the LPSO structure [6].

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Experimental Investigation on Kink Strengthening of Biotite Single Crystals: Rank-1 Connection and Surface Dislocation

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Materials science has pointed out that kink formation in Mille-feuille structures can lead to kink strengthening, which strengthens beyond the intrinsic strength of the material [1, 2]. A previous study [3] reported that shear occurs in finite slip planes in long-period stacking-ordered (LPSO) structures, and deformations that satisfy rank-1 connections (Hadamard's compatibility) form kink bands. An analogue experiment of kink formation in layered rocks [4] showed that strain hardening occurs with kink formation. However, it is not clear whether similar kink strengthening occurs in real rocks. It is also unclear whether rock kink formation is induced by shear at restricted slip planes [5], similar to the LPSO structure. In this study, we performed deformation experiments on biotite single crystals, a layered mineral that plays an important role in strengthening the crust, and investigated kink formation and kink strengthening by observing the stress-strain behavior and microstructure, and discuss the relationship between rank-1 connections and surface dislocations [6].

A high-pressure and high-temperature gas apparatus installed at Tohoku University was used. Experimental conditions were selected in the order of temperature (°C), confining pressure (MPa), and crystal orientation as follows: 1) 300°C, 10MPa, [010], 2) 300°C, 100MPa, [010], 3) 600°C, 100MPa, [100], 4) 600°C, 100MPa, 45° to [010], and 5) 300°C, 185MPa, [010]. Note that all samples were compressed parallel to the above directions. In addition, scanning electron microscopy (SEM) was used to observe the microstructures of the deformed samples under the conditions of 300 °C, 10 MPa, [010] and 300 °C, 185 MPa, [010]. From the stress-strain curves of each experiment, we observed that strain hardening occurred immediately after yielding. As the confining pressure increases from 10 MPa to 100 MPa and 185 MPa, both the yield stress and the ultimate stress increase while the stress decreases with temperature. Microstructural investigation revealed the formation of kink bands in both samples deformed under 300 °C, 10 MPa, [010] and 300 °C, 185 MPa, [010] conditions. Moreover, comparing the kink band orientation and the stress field according to a previous study [2], it was observed that the matrix rotates due to kink formation. In this experiment, axial compression was performed parallel to the basal plane of biotite, so the matrix phase should be parallel to the maximum compressive stress after kink band formation [2]. The kink band boundary has a symmetric tilt boundary. This indicates that the mechanism by which kink bands are formed in the LPSO structures by satisfying rank 1 connections [3], equivalent to the surface dislocations [6], may also apply to biotite. A previous study [2] reported that yielding and kink formation are likely to occur in the part of the foliated structure with a slight initial gradient of the slip plane, so the result indicates that the initial gradient of the slip plane may cause shear and kink band formation occur by a mechanism similar to that of the LPSO structure. In future investigations, we will perform microstructural analysis of the samples before deformation to observe how the slight initial gradient of the slip surface contributes to kink formation. In addition, we need to understand how kink strengthening, known in materials science, affects the strength of the lithosphere.

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Shape of Local Buckling in Cu/SBS Stacking Films Subject to Compressive Deformation

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To investigate buckling behavior of metal/polymer stacking structures, we conducted compressive test on thin films consisting of alternate stack of copper and styrene-butadiene-styrene (SBS) block copolymer layers fabricated on annealed copper substrates. The copper layers were deposited by electroless plating and the SBS layers were painted by dip coating technique. By alternating the electroless plating and the dip coating, we obtained the several films of Cu/SBS staking structure consisting of six layers. The thickness of the copper layer varied from 0.2 μm to 1.5 μm, while the SBS layer thickness was set at the constant value of 0.25 μm. The copper substrates with the Cu/SBS film were shaped to square pillar samples ($3\times3\times6$ mm^3) for compressive test. The Cu/SBS film was coated on one of four longitudinal substrate surfaces. Compressive strain of 10% was imposed along the longitudinal axis in air at room temperature.

Figure 1a shows a typical surface of the Cu/SBS film after the compressive test. Surface relieves extending perpendicular to the compressive axis are visible. A cross section machined by FIB milling is shown in Fig.1b. It is clear that each surface relief is owing to a local buckling of the stacking film. The buckled film was detached from the copper substrate. This should be because fracture strength of the bottom SBS layer is poor.

To investigate the effect of the copper-layer thickness on occurrence frequency and the buckling shape, we measured the average spacing between adjoining bucklings, the height and width values of the bucklings. The average spacing increased linearly with increasing copper-layer thickness. This agree well with the theoretical wavelength of sinusoidal deformation of a single metallic film that is deposited on a soft polymer substrate [1]. Both height and width of the bucklings increased with increasing copper-layer thickness. However, the observed bucklings revealed wide variation in both height and width. This scattering of the buckled shapes is attributed to different growths of the respective bucklings. To eliminate the buckling growth affection, we paid attention to the minimum buckling width at each copper-layer thickness: the minimum buckling width probably corresponds to the buckling width at nucleation. The minimum buckling width was linearly proportional to the copper-layer thickness. This change in the minimum buckling width can be explained by the elastic buckling of component copper layers.

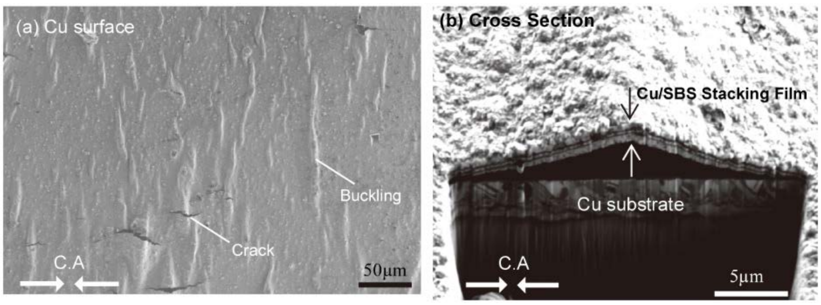

Fig. 1. SEM images showing (a) the surface and (b) the cross section of a compressed Cu/SBS stacking film.

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Elastic properties in a Ti_3SiC_2 MAX phase with a nanolayered crystal structure

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A Ti_3SiC_2 phase is an important MAX phase, exhibiting excellent plastic deformability [1,2] and high Young's modulus (~340 GPa). The Ti_3SiC_2 MAX phase has a nanolayered crystal structure, in which $Ti_{(2)}$-C, C-$Ti_{(1)}$, and $Ti_{(1)}$-Si bonding layers are stacked along the *c*-axis ([0001]). Notably, atomic bonding is inhomogeneous between the layers. The bonding in C-$Ti_{(1)}$ and $Ti_{(2)}$-C is strong owing to the covalent bonding nature combined with metallic and ionic contributions, whereas the $Ti_{(1)}$-Si bonding is weak, especially in shear deformation. Reflecting the nanolayered crystal structure and inhomogeneous atomic bonding, the Ti_3SiC_2 MAX phase exhibits strong anisotropy in plastic deformation and fracture. However, the effects of nanolayered crystal structure and heterogeneous atomic bonding on the elastic properties of the Ti_3SiC_2 MAX phase have not been clarified yet despite their importance for understanding the mechanical properties and atomic bonding nature in the MAX phase. One of this reason is the difficulty in growing large single crystals required for the measurement of single-crystalline elastic properties.

In the present work, the elastic properties in a Ti_3SiC_2 MAX phase single crystal were studied, focusing on the effects of the anisotropic nanolayered crystal structure and heterogeneous interlayer atomic bonding. First, we prepared polycrystalline Ti_3SiC_2 samples with crystallographic texture using a slip casting technique combined with a spark plasma sintering (SPS) process. For the prepared polycrystals, the crystallographic texture was analyzed using the X-ray pole figure, and the all the independent components of elastic stiffness in the polycrystals with the crystallographic texture were measured using resonant ultrasound spectroscopy (RUS) combined with electromagnetic acoustic resonance (EMAR). Next, by analyzing the polycrystalline elastic stiffness reflecting the elastic properties of constituent single crystals and crystallographic texture on the basis of an inverse Voigt-Reuss-Hill (iVRH) approximation, the elastic properties of the Ti_3SiC_2 single crystal were determined. This revealed that the single-crystalline elastic properties were almost isotropic despite its highly anisotropic layered structure. Furthermore, for the Ti_3SiC_2, Ti_3AlC_2, and Ti_3AlC_2 single crystals, their elastic properties were analyzed using first-principles calculations based on density functional theory (DFT). The analyses revealed that $Ti_{(1)}$-Si bonding is relatively stable owing to the hybridization of the *d* electron in Ti and the *p* electron in Si, as well as the localized electrons around the Si atom, which contribute to the isotropic elastic properties and high elastic constants in the Ti_3SiC_2 MAX phase [4].

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Thermodynamic Evaluation and Experimental Investigation of Suzuki Effect in the Al–X Binary Systems

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As mille-feuille structures (MFS) with alternating layers of hard and soft layers, it has been proposed that they can be classified into the crystal structure-based MFS (single phase with two layers), which consists of stacking at the atomic level as seen in Mg alloys with the synchronized long period stacking ordered (LPSO) structure, the microstructure-based MFS (two-phase with two layers) based on two-phase equilibrium, , and their hybrid type MFS [1]. Recently, the segregation behavior of solute elements to stacking faults in $Mg_{97}Y_2Zn_1$ (at.%) alloy containing LPSO structures has been thermodynamically evaluated by Umebayashi *et al.* [2] applying the Hillert's parallel tangent law [3]. Fundamental information on the formation of stacking faults in the matrix phase and the segregation behavior of solute elements to the stacking fault regions (Suzuki effect [4]) is indispensable for the design of novel alloys with crystal structure-based MFS.

In this study, the effects of alloying elements on the Suzuki effect and the stacking fault energy in the fcc matrix phase for Al–X (X: Ag, Cu, Zn) binary alloys were thermodynamically evaluated using the first-principles calculations and the CALPHAD method. In the application of Hillert's parallel tangent law, the hcp phase was regarded as stacking faults in the fcc matrix phase. The stacking fault energy was evaluated based on the treatment proposed by Olson and Cohen [5]. According to the calculated results, it was predicted that the Al–Ag and Al–Cu alloy had a high segregation tendency of solute elements to the stacking fault region, in contrast, the Al–Zn alloys showed no segregation tendency of a solute element. In addition, the stacking fault energy in the Al–Ag alloy would be significantly reduced with increasing Ag content. Detailed of the results of microstructural observation using TEM for Al–Ag alloys will be presented.

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Kinking in Compression of Pearlitic Steel and Resultant Plastic Anisotropy

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Pearlitic structure consisting of lamellar ferrite and cementite is one of the fundamental microstructure in steel and it has some preferable properties such as preferable resistance against hydrogen embrittlement [1]. It has been clarified that the kinking occurs during the compression of pearlitic steels [2], indicating the potential of advanced microstructural control to achieve preferable mechanical properties. Pearlitic structure provides the anisotropy of strength due to its morphology and it is unclear whether the kinking changes this anisotropy. The aim of this work is to clarify the effect of the deformation microstructures including kinked lamella on the plastic isotropy of pearlitic steel.

A spring steel (SAE9254; Fe - 0.55C - 0.7Mn - 0.76Cr - 1.48Si [wt%]) was studied. The pearlitic structure was prepared by the heat treatment with the austenitization at 950 °C followed by isothermal holding at 700 °C. Double compression test [3] was conducted to clarify the plastic anisotropy which means, in this work, the dependence of yield stress on the loading direction. The 1st compression with a cylindrical test piece (8 mm in diameter and 12 mm in height) was applied so as to evolve deformation microstructure such as kinking. The loading directions of the second compressions were set parallel or perpendicular to that of the first compression. The perpendicular loading was realized by the cutting of the first compressed sample to rectangle shape. In both loading directions, the flow stress at the 2nd compression increased with increasing of the strain applied by 1st compression. When compared at similar strain by the 1st compression, the 2nd flow stress at the perpendicular loading was smaller than that at the parallel loading. This difference keeps even after the 1st compression (~30%) enough to obtain the kinking [2], indicating the kinking appears keep the plastic isotropy.

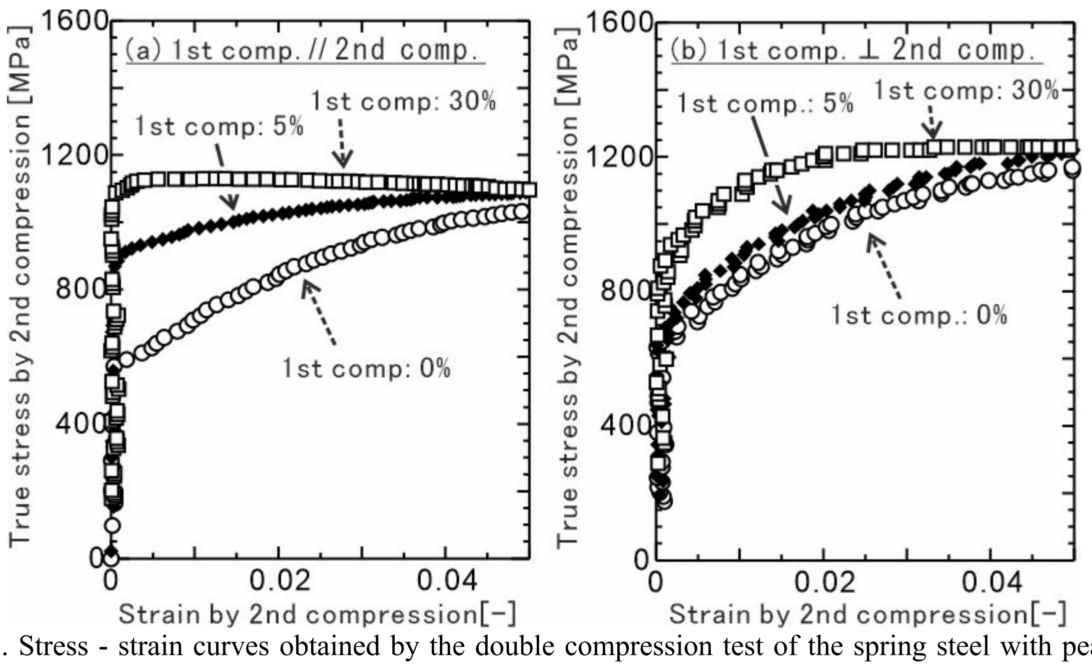

Fig. 1. Stress - strain curves obtained by the double compression test of the spring steel with pearlitic structure. The loading direction of the 2nd compression was parallel (a) and perpendicular (b) to that of the 1st compression.

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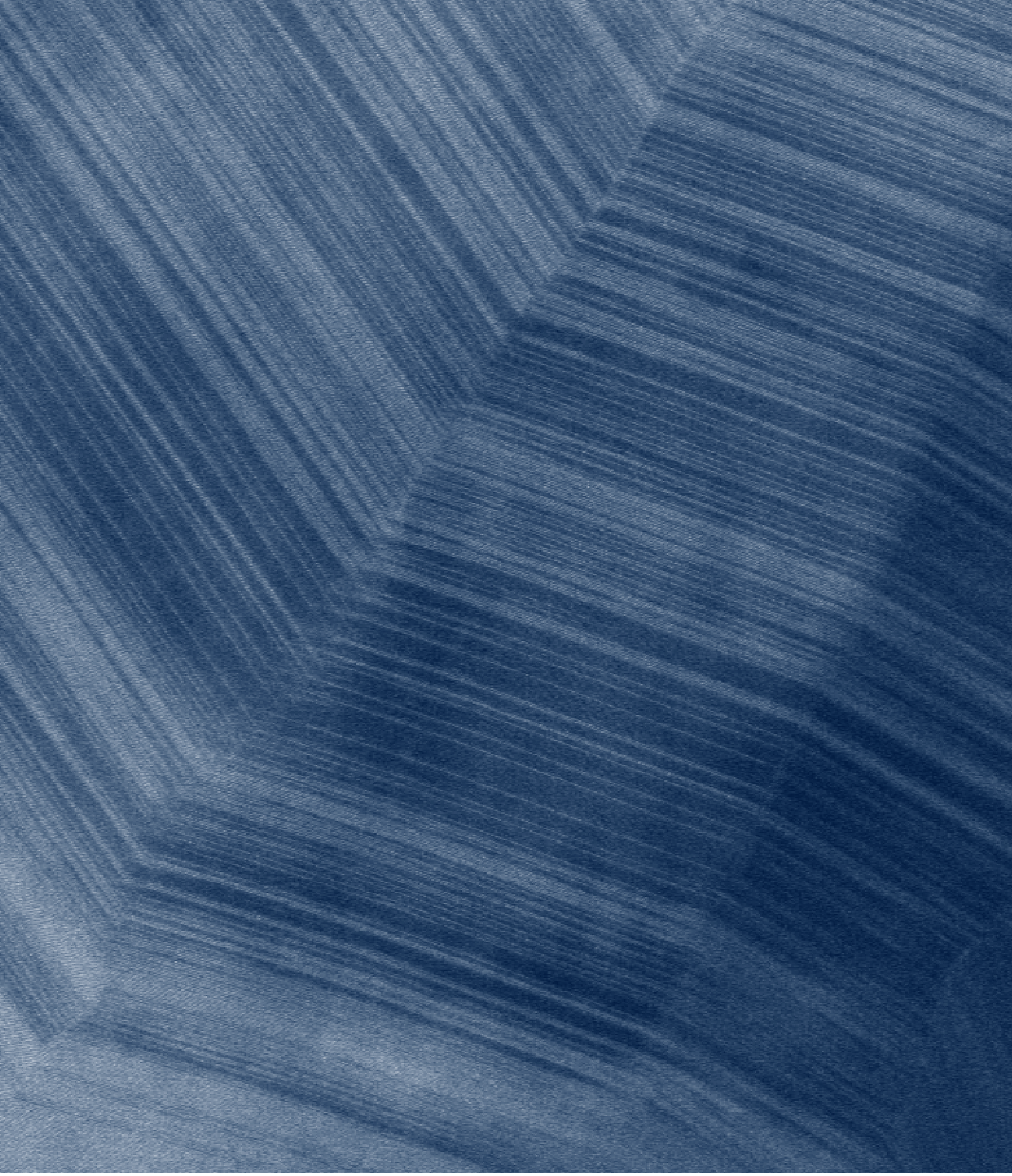

MFS
MATERIALS