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Magnesium Technology

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Editors

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Preface

In recent decades, Magnesium (Mg) and its alloys have emerged as a sustainable structural material owing to their high strength-to-weight ratio, excellent vibration damping, low toxicity, and controllable corrosion rates in dissolvable applications. The possibility to traverse novel alloy designs that offer a distinctive combination of these properties as well as an improvement in ductility has ignited research and development throughout the world. And as many nations seek to reduce their carbon footprint, it is evident that Mg will play a crucial role in these efforts by forwarding the development of sustainable technology. In applications where light weighting is important, Mg has the potential to replace heavier conventional materials such as steel and aluminum leading to a reduction in greenhouse gas emissions.

Coalitions of researchers, scientists, and engineers from academic institutions, industry, and government laboratories have had tremendous success in addressing these challenges through innovative alloy designs and methods. These collaborations have and continue to develop roadmaps for next generation technologies that strengthen Mg as a premier structural material. The TMS Magnesium Committee has been actively involved in providing a platform for these institutions to disseminate the latest information, developments, and cutting-edge research and development, and to present the latest research and development trends related to magnesium and its alloys through the Magnesium Technology Symposium held each year at the TMS Annual Meeting & Exhibition.

The twenty-fifth volume in the series, *Magnesium Technology 2024*, is the proceedings of the Magnesium Technology Symposium held during the 153rd TMS Annual Meeting & Exhibition in Orlando, Florida, March 3–7, 2024. The volume captures full-length manuscripts and extended abstracts from 14 different countries. The papers have been categorized based on topics pertaining to alloy design, fundamentals of plastic deformation, primary production, recycling and ecological issues, characterization, joining, machining, forming, degradation and biomedical applications, corrosion and surface protection, and computational materials engineering.

The symposium began with keynote sessions that featured several distinguished invited-speakers from industry, government organizations, and academia, who provided their perspectives on the state of the art, goals, and opportunities in magnesium alloy research and development. Petra Maier from the University of Applied Sciences Stralsund discussed the role corrosion plays in performance of Mg alloys in biomedical applications. Alexander Grant, CEO of Magrathea Metals addressed the development of next generation electrolytic technology for making Mg metal. Ashley Bucsek of the University of Michigan spoke about the role 3D diffraction microscopy has in uncovering crystallographic texture development in Mg alloys. Jian-Feng Nie from Monash University discussed the progress made in the development of magnesium wheels. Maria Teresa Perez Prado, IMDEA Institute, spoke about the role alloy segregation has in suppressing deformation twinning during mechanical loading.

In conclusion, the 2023–2024 Magnesium Committee would like to thank and express its deep appreciation to all authors who contributed to the success of the symposium; our panel of distinguished keynote speakers for sharing the newest developments and valuable thoughts on the future of magnesium technology; all the reviewers for their best efforts in reviewing the manuscripts; and the session chairs, judges, TMS staff members, and other volunteers for their excellent support, which allowed us to develop a successful, high-quality symposium and proceedings volume.

Aeriel Leonard
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Neale R. Neelameggham
Victoria M. Miller
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Contents

Part I Corrosion and Coatings

Part II Microstructural Evolution and Phase Transformations

Part III Deformation Mechanisms

Part IV Advanced Processing

Part V Primary Production, Recycling, and Modeling

Part VI Biomedical Applications

Part VII Poster Session

About the Editors

Aeriel Leonard is an Assistant Professor in the Materials Science and Engineering Department at The Ohio State University. She was awarded the National Science Foundation CAREER Award in 2024, Department of Energy Early Career Award in 2022, and the Office of Naval Research Young Investigator Award in 2021. She earned her Bachelor's Degree in Metallurgical and Materials Engineering from the University of Alabama in 2012. In 2013, she began her Ph.D. journey at the University of Michigan in Materials Science and Engineering where she earned her Ph.D. in 2018. Dr. Leonard's Ph.D. work investigated real-time microstructural and deformation evolution in magnesium alloys using advanced characterization techniques such as high energy diffraction microscopy and electron back scatter diffraction. During her time at the University of Michigan she led and worked on many teams aimed at increasing the number of underrepresented minorities in engineering including developing and implementing a leadership camp for female engineering students in Monrovia, Liberia. Dr. Leonard was awarded an NRC Postdoctoral Fellowship at the US Naval Research Laboratory in Washington, DC where she worked for two years. During this time, she used advanced characterization techniques such as x-ray computed tomography and high energy diffraction microscopy to understand damage and texture evolution during in-situ loading in additive manufactured materials. She also runs a lifestyle blog titled Aeriel-Views aimed at young graduate and professional students.

Steven Barela is from Pueblo, Colorado, home to the Colorado Fuel & Iron Steel Mill (now Evraz). Driven by the need to resolve welding problems encountered when fabricating race cars, he attended the University of S. Colorado (now CSU-Pueblo) earning an A.A.S. in Metallurgical Engineering Technology. This led to a position at Rocky Flats/DOE nuclear assembly facility in the Non-Nuclear Joining R&D group as an Intern Engineer while simultaneously earning a B.S. in Metallurgical & Materials Engineering at the Colorado School of Mines (CSM) where he specialized in joining and was involved in the CSM Joining Research Center. Mr. Barela then went on to work at Martin Marietta Astronautics Group in the Advanced Manufacturing Technology Group which oversaw procedures and the production of the Titan family of launch vehicles, specifically all joining operations (braze, TIG, MIG, Variable Polarity Plasma) on aluminum, stainless steel, titanium, and metal matrix composites materials. He also participated in the development of Weldalite, a program to produce the Al-Li external tank for the NASA space shuttle program. In the late 1990s, Mr. Barela transitioned his career from welding metallurgy technology to marketing, product development, and sales of welding, forging, and fabricated products as a Technical Sales Engineer at Timminco Extruded Magnesium Products. He then worked for 10 years for Solikamsk Magnesium Works (Russia) by running the U.S. subsidiary Magnesium. com, Inc. During his tenure, Mr. Barela championed forged Magnesium (Mg) wheel projects, generating, and overseeing sales of various Mg products worldwide. Currently, Mr. Barela is with Terves Inc./Magnesium-USA overseeing marketing, product development, and technical sales for extruded and forged products. Over the 20+ years as a member of the TMS Magnesium Committee, Mr. Barela has brought practical industrial end-use knowledge, experience, and insight to the proceedings.

Neale R. Neelameggham, IND LLC, is involved in international technology and management licensing for metals and chemicals, thiometallurgy, energy technologies, Agricoal, lithium-ion battery, energy efficient low cost OrangeH2, Net-zero sooner with Maroon gas and Pink hydrogen, rare earth oxides, etc. He has more than 38 years of expertise in magnesium production and was involved in the process development of its startup company NL Magnesium to the present US Magnesium LLC, UT when he was instrumental in process development from thesolar ponds to magnesium metal foundry. His expertise includes competitive magnesium processes worldwide. In 2016, Dr. Neelameggham and Brian Davis authored the ICE-JNME award winning paper “Twenty-First Century Global Anthropogenic Warming Convective Model.” He is working on Agricoal® to greening arid soils, and at present energy efficient Orange hydrogen, and turbine generator electric car with hydrocarbons and steam. He authored *The Return of ManmadeCO2 to Earth: Ecochemistry*.

Dr. Neelameggham holds 16 patents and applications and has published several technical papers. He has served in the Magnesium Committee of the TMS Light Metals Division (LMD) since its inception in 2000, chaired in 2005, and since 2007 has been a permanent advisor for the Magnesium Technology Symposium. He has been a member of the Reactive Metals Committee, Recycling Committee, Titanium Committee, and Program Committee for LMD and LMD council. Dr. Neelameggham was the Inaugural Chair, when in 2008, LMD and the TMS Extraction and Processing Division (EPD) created the Energy Committee and has been a Co-Editor of the Energy Technology Symposium through the present. He received the LMD Distinguished Service Award in 2010. As Chair of the Hydrometallurgy and Electrometallurgy Committee, he initiated the Rare Metal Technology Symposium in 2014 and has been a co-organizer to the present. He organized the 2018 TMS Symposium on Stored Renewable Energy in Coal and initiated Light Elements Technology in 2023.

Victoria M. Miller is an Assistant Professor in the Department of Materials Science and Engineering at the University of Florida, a position she started in September 2019. She was previously an assistant professor at North Carolina State University from 2017 to 2019. Originally from Michigan, she received her B.S.E. in Materials Science and Engineering from the University of Michigan in 2011 and completed her Ph.D. in Materials at the University of California Santa Barbara in 2016. After graduate school, she worked for a year at UES, Inc. onsite in the Materials and Manufacturing Directorate of the Air Force Research Laboratory in Dayton, Ohio. She also previously worked at Ford Motor Company, Toyota Engineering and Manufacturing, and Lockheed Martin Aeronautics. Her primary research interest is microstructural evolution during thermomechanical processing of metals and alloys, particularly for those with low symmetry crystal structures. She has been researching Mg alloys since the age of 16. Professionally, Dr. Miller has served on many committees within TMS, is Associate Editor for *JOM*, and is a Key Reader for *Metallurgical and Materials Transactions A*. She was a recipient of the 2017 TMS Young Leaders Professional Development Award, the 2020 ASM Bronze Medal Award, and the 2022 TMS-JIMM Young Leaders International Scholar Award.

Domonkos Tolnai is a scientist with the Department of Functional Magnesium Materials at Helmholtz-Zentrum Hereon in Geesthacht, Germany. He earned his master's degree in engineering physics from the Eötvös University in Budapest, Hungary, where he investigated the fatigue behavior of Al based particle reinforced MMCs with synchrotron tomography. In 2007 he began his Ph.D. studies at the Vienna University of Technology in Vienna, Austria on investigating the solidification of Al alloys by in situ synchrotron tomography. After defending his thesis in 2011, he moved to Geesthacht, where he started as a post-doctoral fellow and then continued later as a scientist. His research focuses on the microstructural response of Mg based materials to thermo-mechanical and degradation load, and on the development of advanced in situ characterization environments based on synchrotron radiation.

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Magnesium Technology

Corrosion and Coatings

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Microstructural Evolution and Phase Transformations

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Steven Johnson, Central Connecticut University

Deformation Mechanisms

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Victoria Miller, University of Florida

Advanced Processing

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Part I

Corrosion and Coatings

Different Analytical Methods to Determine the Influence of Pitting on the Residual Performance of Mg Alloys as Implant Materials

Petra Maier

Abstract

Mg alloys are prone to pitting due to their non-uniform protective corrosion layers, which can lead to an increase in stress intensity based on the notch effect, pit-to-crack transition, and thus premature failure. A small set of analytical methods to determine the extent of pitting and its effect on the resulting residual strength is presented. Micrographs, 3D microscopy, or 3D analysis using CT are used to determine the amount and geometry of pitting—each with advantages and disadvantages. The influence of the corrosion pits on the mechanical properties is tested by static, quasi-static, and cyclic test methods: by tensile, flexural, or fatigue testing—either after corrosion or overlapping. Knowledge about the critical pit is of general interest. Stress corrosion is discussed by applying static tests like C-ring testing, which also plays a role in slow strain rate tensile tests and stress corrosion cracking is more or less influenced by corrosion pits.

Keywords

Mg–RE alloys • Pitting corrosion • 3D-µCT analysis • Residual tensile strength • Corrosion fatigue

Extended Abstract

Mg alloys are susceptible to pitting due to their non-uniform protective corrosion layers, which can lead to an increase in stress intensity based on the notch effect, a transition from pitting to cracking, and thus premature failure. To quantify the influence of corrosion pits on residual strength, the corrosion pit should be described as completely as possible, see an example with a Mg–3Y–3RE alloy in Fig. 1. A pit with a depth of about 300 µm was found to have a residual force was found to be 90% [1]. A small set of analytical methods for determining the extent of pitting and its effect on the resulting residual strength is presented in this keynote presentation at the Magnesium Technology symposium at the TMS 2024. The Standard Guide for Examination and Evaluation of Pitting Corrosion provides a chart to describe the shape of pits [2]: critical are pits with a narrow and deep shape and undercutting, less harmful are elliptical pits that are wide and shallow. The pitting factor [3, 4] is calculated by dividing the deepest pit by the average penetration depth, which is usually determined by the corrosion rate (CR) based on weight loss. The deepest pit can be determined by 3D microscopy, see Fig. 2 for a 3D confocal image from a study on corrosion properties of extruded Mg10Gd modified with Nd and La [5]. The study in [5] and a similar study on Mg–Y–Nd–Gd–Dy alloys [6] show that large corrosion pits lead to a high PF when the CR is low and the protective corrosion layer is only very locally discontinuous. 3D laser confocal scanning [7] or 3D laser profilometer measurements [8] can also be used to determine the depth of the pits. However, the shape cannot be determined with these methods. Undercutting pits, of course, cannot be visualized with 3D microscopy in top view. Atomic force microscope analysis can also be used to determine the corrosion morphology and depth of corrosion pits, but the area and shape, according to undercutting appearance, are limited [9].

Micrographs, see Fig. 3a, provide 2D determination of shape and size, but only a 2D view—the selected cross-section need not present the most critical shape of a single pit. SEM, see image in Fig. 3c, offers imaging with a great depth of field, but has its limitations in terms of fully quantifying the size and shape. Cross-sectional micrographs, however, provide the ability to determine the average

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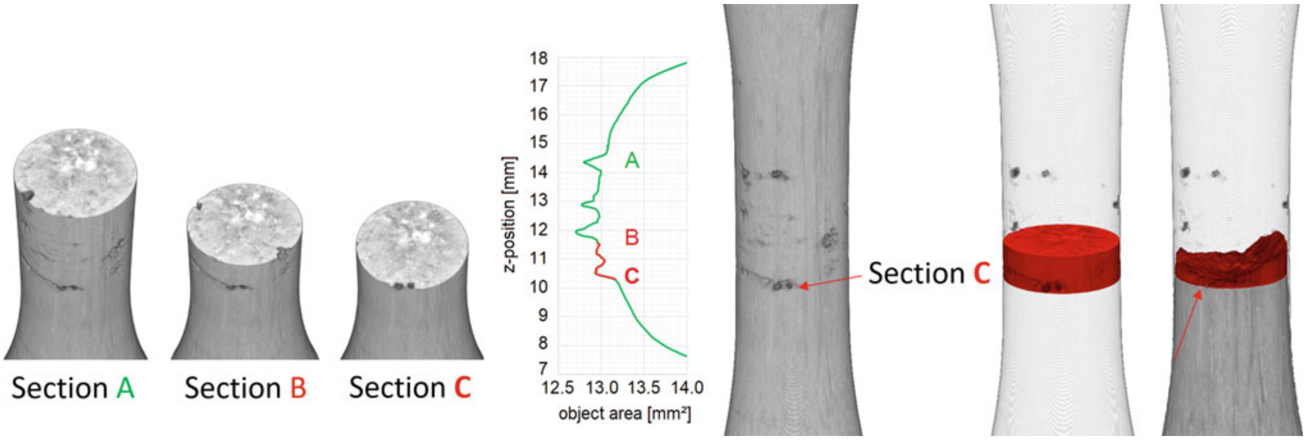

Fig. 1 Remaining cross-sectional area after corrosion of a Mg–3Y–3RE tensile sample after corrosion time of 24 h in Ringer solution at 37 °C (left: µCT cross-sections before tensile test, right: µCT before and after tensile test), red part of green curve (sample cross-sectional area) indicates the fractured area, based on [1]

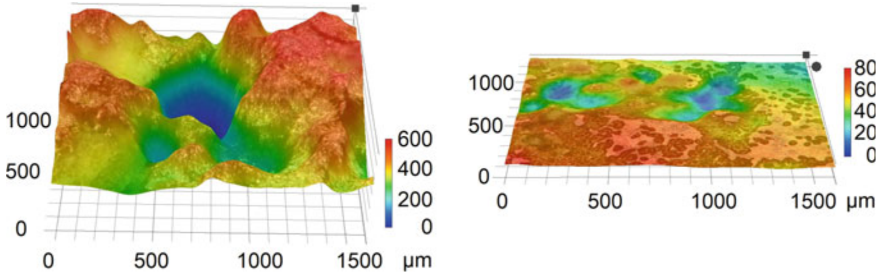

Fig. 2 3D height map by a confocal 3D microscope, showing a corrosion pit of significant size (left) and material with higher pitting resistivity (right)

penetration through the corroded area of individual slices independent of the volume (weight) loss, as described in [10]. The average penetration from corrosion rate based on weight loss will differ from the average penetration of cross-sections when the corrosion rate is not uniform—and severe pitting is obviously the clearest manifestation of non-uniform corrosion. In this case, the weight loss is a result of only a few local spots and distorts the evaluation.

Returning to the corrosion pits in Fig. 1, the larger of the two in Section C on the left has an elliptical shape in this cross-section. The longitudinal cross-section, on the other hand, shows a more pointed undercutting shape, resulting in a higher stress intensity increase. Only in complete 3D analysis using CT it is possible to assess the shape and size of a corrosion pit.

The influence of the corrosion pits on the mechanical properties is of interest under static, quasi-static, and especially under cyclic loading. Stress corrosion, slow strain rate tensile tests, flexural, and fatigue testing can be either applied after corrosion or simultaneously. The micrographs in Fig. 3a show corrosion pits on a Mg–RE alloy forming under stress corrosion [11]. It can be seen that these large

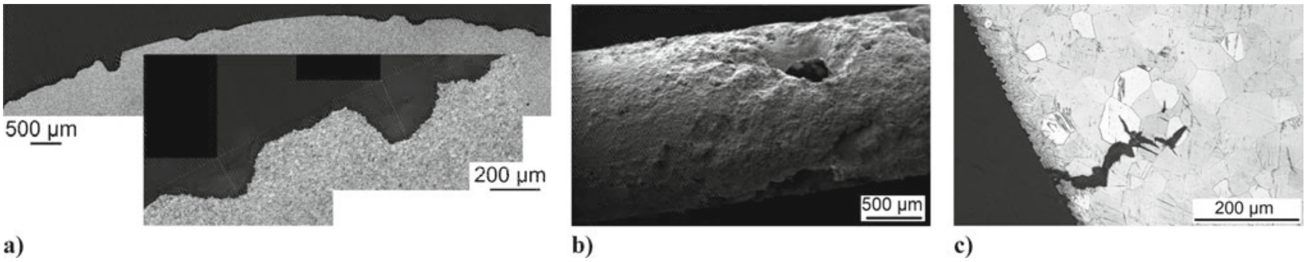

Fig. 3 Corrosion pits presented by **a** cross-sectional micrographs (based on [11]) and **b** SEM imaging [10] and **c** initiating stress cracks in stress corrosion (study presented in [12])

pits, which are elliptically formed and have a wide opening do not cause a crack initiation. Another is found when testing the wires shown in Fig. 3b in three-point bending after corrosion, the pits acting as crack initiation [10]. Under stress corrosion even small pits transits into cracks, see Fig. 3c [12]. In this study, it could be seen that the near-surface material of an Mg–Dy alloy is heavily twinned and, in combination with corrosion pits and tensile loading, cracks form. Twin boundaries strongly influence the crack propagation direction [12, 13]. The effect of surface roughness can reduce the fatigue strength to a high degree and corrosion fatigue cracks originate mainly from the corrosion pits [14, 15]. Knowledge about the critical pit is of general interest.

More and more effort is undertaken in automated detection of pitting corrosion and its effect on the mechanical integrity [16]. The identification and description of surface-based corrosion features are in main focus. CT analyses also offer to calculate the CR by volume loss; however, its segmentation is challenging. Machine learning is applied to define residual material, degradation/corrosion layers, bone/tissue, and background [17].

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Effect of Heat Treatment on the Microstructure and Corrosion Properties of Mg–15Dy–1.5Zn Alloy with LPSO Phase

Genzhi Jiang, Yuanding Huang, Sarkis Gavras, Hui Shi,
and Norbert Hort

Abstract

The influence of the amount and type of long-period stacking ordered (LPSO) phase on the corrosion behavior of both the as-cast and heat-treated Mg–15Dy–1.5Zn alloys in 0.9% NaCl solution was investigated. It was found that the network structure 18R-LPSO phase is an effective barrier to further corrosion of the as-cast sample. After T4 treatment for 24 h, the dendrites disappeared and part of 18R-LPSO dissolved in the matrix, which weakened the corrosion protection. Meanwhile, such LPSO phase acts as a cathodic to accelerate the corrosion of the matrix because of its potential difference from the magnesium matrix. After T4 treatment for a longer time, 18R-LPSO phase could transform into 14H-LPSO phase which has a different effect on corrosion. The galvanic corrosion also occurs between the 14H-LPSO phase and the matrix. Its uniform and dense distribution results in the formation of continuous corrosion products on the surface, which is beneficial for corrosion resistance.

Keywords

Mg–Dy–Zn alloy • Heat treatment • Corrosion rate • LPSO phase • Microstructure

Introduction

Magnesium (Mg) alloys have long been an interesting research topic in the field of biomedical applications due to their low density, high specific strength, and good biocompatibility [1]. Nevertheless, the critical obstacle to their extensive application is how to balance their integral strength and degradation rate. Precipitation strength is one of the most popular methods to improve mechanical properties. Nevertheless, previous studies showed that the intermetallic phases can act as either a continuous network barrier to retard corrosion propagation, or as a galvanic cathode to accelerate the corrosion of the Mg matrix, or as a micro-anode to dissolve preferentially at the initial corrosion stage [2].

It was reported that optimizing the size, distribution, and morphology of long-period stacking ordered (LPSO) phases can change the corrosion behavior of Mg-RE alloys from pitting corrosion to uniform corrosion and reduce the corrosion rate to some extent [3, 4]. For example, the heat treatment of Mg alloy with LPSO phase influences the corrosion rate due to the phase transformation from the bulk reticular LPSO phase to the lamellar 14H-LPSO phase. The corrosion rate increased significantly after such heat treatment since the high volume fraction of 14H-LPSO phase changes the corrosion propagation paths and provides more galvanic corrosion points [5]. Magnesium alloys with the co-existence of the 18R and 14H-LPSO structures exhibited worse corrosion resistance than those with a single LPSO structure (either 18R or 14H), which could be attributed to the accumulation of stacking faults as well as the enrichment of solute atoms in the phase transition zone [6]. In this work, the effects of different LPSO phases on the corrosion behavior of Mg–15Dy–1.5Zn alloys have been investigated.

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Experiments

The Mg–15Dy–1.5Zn alloy was prepared using permanent mould by direct chill casting [7]. High-purity Mg (Magnesium electron, Manchester, UK, 99.94 wt.%) was melted in a mild steel crucible under a protective atmosphere (Ar + 2% SF$_6$). Pure zinc (Zn) and pure dysprosium (Dy) were then added to the melt at 750 °C. The melt was stirred for 30 min at 200 rpm and then poured into a mold preheated at 680 °C and covered with a release agent (boron nitride) [2]. Then the filled crucible was held at 680 °C for 15 min with gas protection (Ar + 2% SF$_6$). Finally, the melt was solidified by lowering the crucible into cooling water at a rate of 10 mm/s. When the melt was fully immersed in the water, the solidification finished. Then, the as-cast Mg–15Dy–1.5Zn alloy was heat treated at 500 °C for 24, 48, and 264 h, followed by immediate quenching in water. These treated alloys are thereafter termed AC, 24HT, 48HT, and 264HT

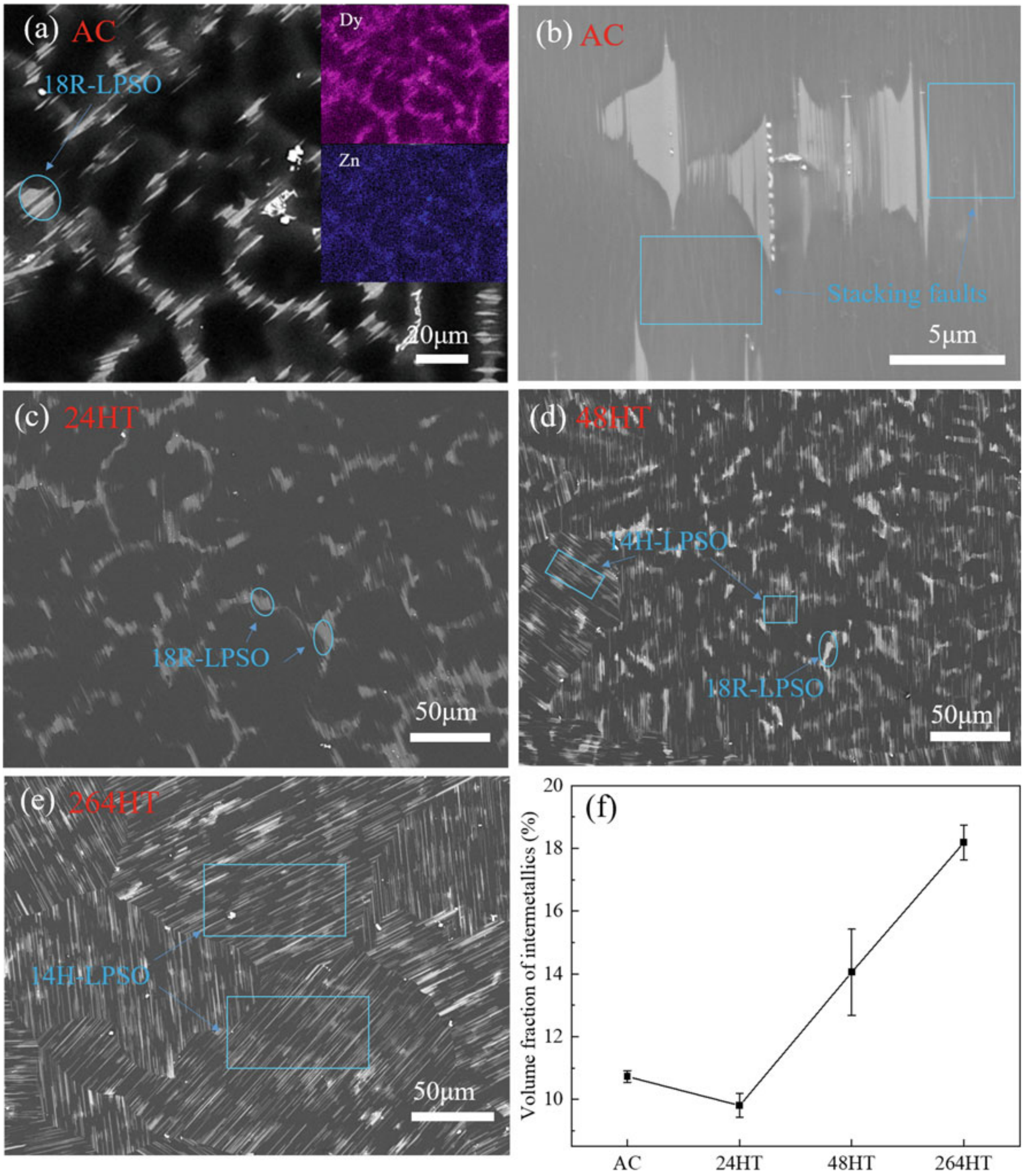

Fig. 1 BSE images of Mg–15Dy–1.5Zn alloys with different T4 treatment time at 500°C: **a** AC, **b** local magnified image of AC, **c** 24HT, **d** 48HT, **e** 264HT alloys, together with **f** their corresponding measured volume fraction of intermetallics