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Advances and Achievements in In Situ Analysis of Corrosion and Structure–Property Relationship in Mg Alloys

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Every year, the TMS Magnesium Committee carefully selects a special topic in research and development related to magnesium (Mg) representing subjects of high interest from both academic and industrial perspectives, as well as demonstrating major achievements within this area. Following last year's topic on Mg microallying,[1] this year's focus is on in situ methods and associated techniques in their broad definition spanning from laboratory- to large-scale facilities to process monitoring. The applications of in situ techniques have a wide spectrum from the analysis of melts and liquid–solid transitions, to solid-state phenomena during thermomechanical processing and heat treatments, to surface interactions with various environments. Therefore, such works are of significant interest to scientists working in the area of Mg alloy development as well as to a much broader audience from both academia and industry.

This interest is primarily caused by challenges in the analysis of a structure–property relationship in Mg alloys, and even a cursory glance of literature reveals a sharp increase of publications relevant to this topic recently. For instance, very high reactivity of Mg as well as its well-known propensity to alter the structure substantially during unloading in mechanical testing makes it difficult to understand and, thus, to simulate a correlation between microstructures observed in postmortem analysis and physical processes during testing or fabrication. Nevertheless, recent advances in in situ analysis based on large-scale research facilities such as neutron scattering and synchrotron radiation sources, as well as microscopy-based, acoustic emission, and other more traditional techniques allowed for significant achievements. Apart from the apparent development of relevant experimental techniques, a significant part of this success should also be attributed to increasing accessibility of the facilities and simplification of their use from a user perspective.

This special topic is not intended to be a comprehensive review covering all aspects of in situ methods available for metallic materials development. Instead, this selection is intended to present several representative studies on Mg alloys and Mg-based composites with the use of in situ techniques. In addition, it overviews two techniques, acoustic emission (AE) and ambient-pressure x-ray photoelectron spectroscopy (AP-XPS), that are novel to the Mg community. These studies are intended to give readers representative examples of the techniques' potential and to help in navigating the spectrum of modern state-of-the-art analytical methods facilitating the development of Mg alloys. These articles are organized corresponding to a typical Mg alloy lifecycle, from material preparation and solidification to thermo-mechanical processing and product fabrication to degradation.

The first article, by Wim Sillekens et al., is titled "The Use of In Situ x-ray Imaging Methods in the Research and Development of Magnesium-Based Grain-Refined and Nanocomposite Materials." It presents three case studies of how in situ imaging comprising laboratory-scale micro-focus x-ray radiography and synchrotron x-ray tomography are used by practitioners in visualizing and quantifying phenomena during Mg-based grain-refined and nanocomposite materials fabrication. The findings from the first study reveal crystal nucleation and growth in a Mg-Nd-Gd alloy depending on the addition of zirconium and cooling rate. The aim of the second study is to investigate the influence of SiC particle addition on microstructural development of a Mg-Zn-Al alloy during solidification. The

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focus of the third study concerns fabrication of nanocomposites based on Mg alloy Elektron21 with the addition of AlN or Y_2O_3 particles.

The second article, by Domonkos Tolnai et al., is titled "The Role of Zn on the Elevated Temperature Compression Behaviour of Mg5Nd: An In Situ Synchrotron Radiation Diffraction Study." With use of in situ synchrotron radiation diffraction and *post mortem* microscopy, the authors investigate the effect of Zn content in as-cast Mg5Nd-based alloys on hot deformation mechanisms and the evolution of microstructure during compression at 350°C, which is typical for industrial production technologies.

The third article, by Alexei Vinogradov and Kristian Máthis, is titled "Acoustic Emission as a Tool for Exploring Deformation Mechanisms in Magnesium and Its Alloys In Situ." The authors of this work review the capacities of an advanced acoustic emission technique in understanding the interplay between two primary deformation mechanisms in Mg alloys vis. dislocation slip and twinning. This technique alone as well as in conjunction with neutron diffraction allows for monitoring of acting deformation mechanisms during thermo-mechanical processing *in operando*. The authors use the combination of techniques for the analysis of mechanical response in a pure Mg and Mg-Al alloy during tensile loading.

The fourth article, by Andrij Milenin et al., is titled "Analysis of Microstructure and Damage Evolution in Ultra-Thin Wires of the Magnesium Alloy MgCa0.8 at Multipass Drawing." In this article, the authors use in situ tensile tests of an MgCa0.8 wire in a scanning electron microscope to parametrize a numerical finite-element model. The model is then used for the design and analysis of a deformation process for a combined hot and cold 40-pass drawing for manufacturing ultra-thin MgCa0.8 alloy wires having a final diameter of 0.05 mm.

The fifth article, by Ashley R. Head and Joachim Schnadt, is titled "UHV and Ambient Pressure XPS: Potentials for Mg, MgO, and $Mg(OH)_2$ Surface Analysis." The authors of this article review x-ray photoelectron spectroscopy (XPS) and its ambient pressure variation (AP-XPS) in the context of potential applications for the analysis of Mg alloys degradation. This is a surface-sensitive technique that allows for analysis of chemical and electronic structures on solid sample surfaces ranging from model systems to industrial materials. XPS is typically carried out at "model" ultra-high vacuum conditions, whereas AP-XPS allows for much closer to real-life pressures. Rather than reporting on a specific case study, the authors illustrate the information XPS can provide and discuss the possibilities of AP-XPS for the analysis of Mg oxidation as well as discuss the properties of MgO, and $Mg(OH)_2$ systems in both fundamental and applied studies.

The following articles being published under the topic Advances and Achievements in In Situ Analysis of Corrosion and Structure–Property Relationship in Mg and Mg Alloys provide excellent details and research on the subject. To download any of the articles, follow the URL http://link.springer.com/journal/11837/68/11/page/1 to the table of contents page for the December 2016 issue (vol. 68, no. 12).

- "The Use of In Situ x-ray Imaging Methods in the Research and Development of Magnesium-Based Grain-Refined and Nanocomposite Materials" by W.H. Sillekens, D. Casari, W.U. Mirihanage, S. Terzi, R.H. Mathiesen, L. Salvo, R. Daudin, P. Lhuissier, E. Guo, and P.D. Lee
- "The Role of Zn on the Elevated Temperature Compression Behaviour of Mg5Nd: An In Situ Synchrotron Radiation Diffraction Study" by D. Tolnai, R.H. Buzolin, F. D'Elia, T. Subroto, S. Gavras, A. Stark, N. Schell, K.U. Kainer, and N. Hort
- "Acoustic Emission as a Tool for Exploring Deformation Mechanisms in Magnesium and Magnesium Alloys In Situ" by Alexei Vinogradov and Kristian Máthis
- "Analysis of Microstructure and Damage Evolution in Ultra-Thin Wires of the Magnesium Alloy MgCa0.8 at Multipass Drawing" by Andrij Milenin, Piotr Kustra, Dorota Byrska-Wójcik, Oleксandr Grydin, Mirko Schaper, Thorben Mentlein, Gregory Gerstein, and Florian Nürnberger
- "UHV and Ambient Pressure XPS: Potentials for Mg, MgO, and $Mg(OH)_2$ Surface Analysis" by Ashley R. Head and Joachim Schnadt.

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