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Physical thermo-mechanical simulation of magnesium: An in-situ diffraction study

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ABSTRACT

Time-resolved, two-dimensional synchrotron high-energy X-ray diffraction has been utilized for the in-situ investigation of the microstructural evolution of magnesium, during heating and during plastic deformation at various temperatures. Throughout static heating of the as-extruded material, first recovery, then recrystallization and finally grain growth occurred with increasing temperature. Grain rotation was observed during grain growth of the static heated samples. Subsequent plastic deformation, through compression, at lower temperatures revealed the activated deformation systems. At room temperature, extension-twinning flips the crystallite orientations abruptly from the extrusion to the compression fiber texture. In contrast, at elevated temperatures, twinning is negligible and the texture reorientation progresses in a gradual steady-state flow regime, ending in a tilted basal texture with a tilt angle depending on the degree of deformation. The methodology described herein offers parametric studies related to microstructural and deformation processes in an unprecedented way.

1. Introduction

Magnesium-based alloys posses a hexagonal close-packed *hcp* crystal structure, making them difficult to plastically deform due to the high crystalline anisotropy. Besides twinning, very few slip systems are activated at room- and low temperatures and involve mostly basal slip. Twinning is commonly observed as a process responding to shear, and as such further enhances crystallographic texture [1]. While other slip systems become active at higher temperatures, recrystallization and grain-growth are also more likely, and grain-refinement methods through plastic deformation, such as equal-channel angular pressing, are compromised [2]. These crystallographic factors, together with dependencies on composition, microstructure, precipitates and environment, offer an infinite field of research to serve the existing and future potential for the light-weight structural engineering industry.

Deformation mechanisms of magnesium have been widely investigated and reviewed in the literature [3,4], stating that primarily basal slip and twinning [5] are activated at room and cryogenic temperature [6,7], leading to strong crystallographic texture during deformation [8]. To accommodate external load, compression-twinning is different from twinning under tension and depends on the *c*/*a* lattice parameter ratio [5]. Above 450 K prismatic slip becomes active, leading to enhanced ductility beyond 608 K. First and second order pyramidal slips activate at 623 K and 573 K, respectively, the latter occurring extensively above 673 K [9]. Dynamic recovery is reported to start at as low as 150 K [6] while dynamic recrystallization occurs at elevated temperature and is strain- and strain-rate dependent [10]. As expected, these different deformation mechanisms lead to different crystallographic orientations [11] and, as such, to a variety of mechanical properties dependent on the processing history of the material.

This wide spectrum of processing parameters and the diversity of applications of magnesium alloys require extensive on-going research, much of which is time and resources consuming.

In the past two decades, high-energy X-rays have been established at synchrotron sources [12–16]. Due to their excellent properties [17], such as penetration into the bulk of the materials, they soon found applications in the fields of materials science and engineering [18,19]. Two-dimensional detectors revolutionized the science, as large reciprocal-space coverage [20,21], orientation dependency [22] in texture [23] and stress analysis [24], and good time-resolution [25] opened unprecedented possibilities of measurement. More sophisticated data acquisition methods evolved in 3-dimensional X-ray diffraction and back-projection-calculated

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tomography [26–28], and the interpretation of finest features in individual reflection spots has been pioneered [29,30].

Conventional microstructure, phase and texture analyses occur through methods of processing, quenching and metallurgical analysis, which needs to be repeated for various process parameters, including temperature, stress, composition, orientation, and their time and spatial derivatives. In contrast, diffraction methods of penetrating high-energy X-rays [17] or neutrons [31–33] allow the direct study of the bulk of the material in-situ and in real time. In our recently established technique [34,35], a well-focused synchrotron beam reveals information from a distinguishable number of crystallites within the bulk of a sample. Reciprocal space can be analyzed both longitudinally, to observe strain and phase composition, and azimuthally for orientation analysis. Through characteristic diffraction features it is possible to distinguish slip, twinning, phase transformations and correlations therein. In addition grain rotation, subgrain formation, dynamic recovery, recrystallization and grain-growth or grain-refinement upon plastic deformation may also be distinguished. Two-dimensional diffraction images can be taken within a fraction of a second and therefore allow real-time recording in-situ, while the specimen is being processed. As diffraction patterns can be directly correlated to external processing parameters, quenching and subsequent metallurgy are obsolete, offering a novel and accelerated research technology for the future. The method has been validated and applied through tracing the evolution of single-grain orientations arising from the deformation texture in copper [36], twinning induced plasticity steel [37], hot-deformation induced dynamic recovery and recrystallization in single-phase β-zirconium [25] and multi-phase α+β and α+β+γ titanium-aluminide-based intermetallics [38,39].

Here we present the first results displaying all of the above metallurgical and crystallographic features from hot-deformation on pure magnesium, the basis of a whole family of light-weight alloys, at various temperatures, using real-time in-situ high-energy synchrotron X-ray diffraction. A profound azimuthal-angle/time (AT) data analysis is presented, building the base for future in-depth materials investigations, for different alloys and under engineering standards.

2. Experimental

Cylindrical compression specimens of pure Mg were cut to 6 mm length from an extruded rod of 5 mm diameter. The as-extruded grain size is 17 μm and the typical extrusion texture of magnesium is known to align the {002} plane perpendicular, or transverse **T**, to the cylinder axial direction **L**, while {100} and {110} have random degrees of freedom, both resulting in a broad, single pole along **L**. Because of the symmetry of the specimen and the extrusion process, such texture is axial symmetric, showing fiber textures, which has been verified by neutron scattering on similar specimens [40]. Texture components in hexagonal materials have been reviewed by Wang and Huang [8] and recently investigated experimentally on extruded magnesium by Stanford and Barnett et al. [41–43]. As the specimens undergo heating and compression testing along the cylinder axis, axial symmetry is conserved and it will be sufficient to measure the polar angle distribution only, to obtain full texture information.

In-situ synchrotron high-energy X-ray diffraction [17] has been performed at the Advanced Photon Source, beamline 1-ID-C of the Argonne National Laboratory. The beam parameters were photon energy of E=90 keV, wavelength λ=0.13 Å and wave number k=45.6 Å^{-1}. The specimen was mounted into a hydraulic stand-alone load frame MTS-858, set up for compression, combined with a halogen-lamp furnace for heating. A fine beam of X-rays, 100×100 μm^2, is transmitted through the center of the sample during the compression process. Diffraction patterns were taken

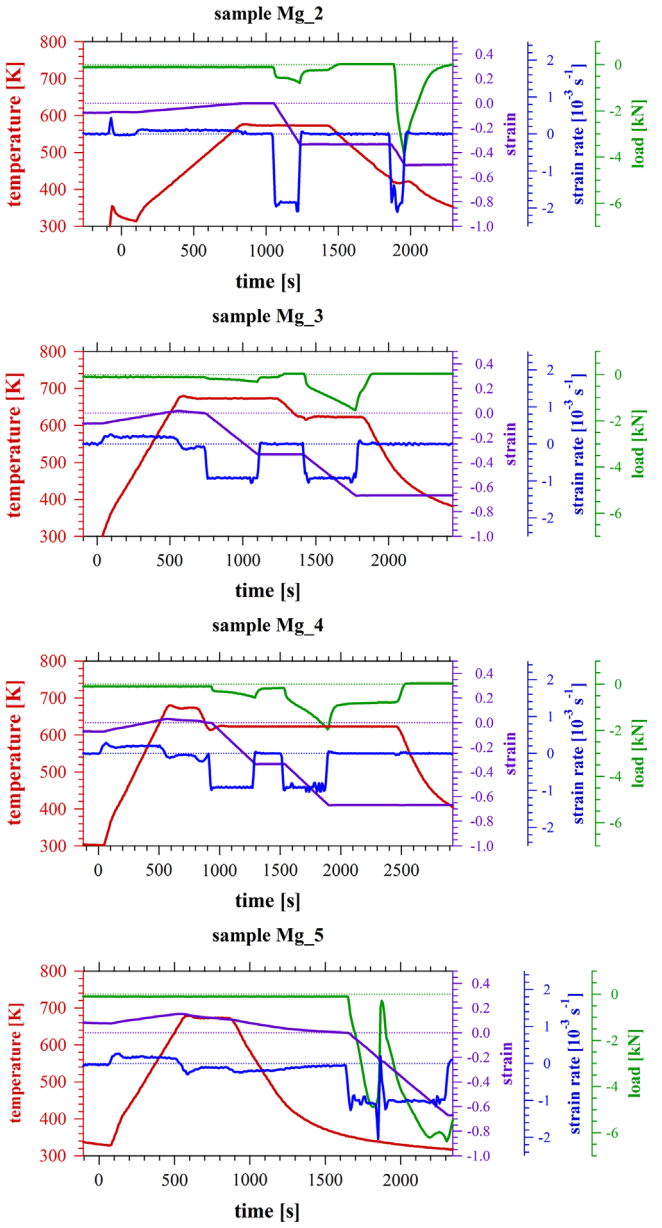

Fig. 1. Processing parameters in engineering units as a function of time for all four runs.

by a MAR-165 CCD detector every 8 s. Data reduction and treatment into azimuthal-angle/time (AT) plots were undertaken using our in-house-developed dataRring software package. A series of other metals, including bulk metallic glass, have been investigated using the same experimental arrangement, and as such, it is identical to that described by Qu [44] and Liss [45]. The comprehensive analysis technique on polycrystalline materials has been described recently by Liss and Yan [35].

Pure magnesium, which lays the basis for a large variety of light-weight alloys, has been chosen for this pioneering experiment. Four specimens, called Mg-2, Mg-3, Mg-4, and Mg-5, have been tested at different temperature and compression ramps, their recorded parameters being displayed in Fig. 1 as a function of time. Compression rates were controlled at a constant 11 μm/s for Mg-2 and 5.6 μm/s for the other three samples, resulting in initial strain rates of 1.9×10^{-3} s^{-1} and 9.6×10^{-4} s^{-1}, respectively. Thermal expansion also acted on the load frame, drifting with time, for which the strain-zero condition for strain calculations has