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Nanomechanical analysis of AZ31 magnesium alloy and pure magnesium correlated with crystallographic orientation

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ABSTRACT

The anisotropic nanomechanical properties of AZ31 magnesium alloy and pure Mg were measured in situ via nanoindentation of individual grains with simultaneous observations using a scanning electron microscope. Values of the nanohardness, indentation size effect, elastic modulus, and yield strength were correlated with the crystallographic orientation provided by electron backscattering diffraction and were further used to investigate the relationships between the nanomechanical properties of the materials and the work of nanoindentation.

The nanohardness of AZ31 was found to be generally above that of pure Mg due to solid solution strengthening. The nanohardness of AZ31 first considerably decreased and then marginally increased, whereas the nanohardness of pure Mg steadily decreased as the angle between the hexagonal lattice *c*-axis of both materials and the indentation direction increased. The indentation size effect was stronger for AZ31 than for pure Mg, and its magnitude decreased as the angle between the lattice *c*-axis and the indentation direction increased. The AZ31 modulus remained nearly constant throughout the range of investigated orientations; the modulus of pure Mg followed a theoretical angular dependence but was generally lower than expected. The yield strength behaved in a similar manner to the nanohardness in both materials. Plots of the ratio of the nanohardness to the yield strength revealed that both materials underwent significant work hardening shortly after nanoindentation began. It was also shown that the amount of plastic deformation increased for Mg and increased or remained nearly constant for AZ31 as the angle increased. The observed orientation dependencies were interpreted as a consequence of the anisotropic activities of the dominant slip systems and extension twinning.

1. Introduction

The AZ31 magnesium alloy is the most common wrought magnesium alloy used for lightweight components in the aerospace and automotive industries, portable electronic devices and sports equipment due to its convenient combination of mechanical and physical properties [1,2]. A relatively low content of alloying elements usually makes AZ31 a single-phase alloy strengthened by solid solution. The hexagonal close-packed (hcp) structure of Mg, however, produces its well-known anisotropic mechanical properties [2], which represent one of the primary obstacles to its usability. The same anisotropy also occurs during a small-scale deformation of individual grains in pure Mg [3–6]. In this respect, nanoindentation is a valuable and generally accepted technique for evaluation of the nanomechanical properties of a material [7].

Studies correlating the crystallographic orientation with the nanoindentation may be divided into two groups. The first group includes uniaxial compression of micropillars by a flat-ended indenter [8–10], and the second group comprises triaxial compression by a spherical [3] or tapered indenter [4–6]. Due to a different degree of strain complexity, the presumptions and interpretation of results are different for each group. Therefore, only the latter is of the utmost importance for this study. Catoor et al. [3] investigated pop-ins (i.e., a sudden displacement excursion on an otherwise smooth load-displacement curve) generated during nanoindentation in single-crystalline Mg on three low-index crystallographic planes (0001), (10–12) and (10–10). Based on the results from transmission electron microscopy (TEM), they concluded that the dominant dislocation mechanisms always involved ⟨a⟩ slip and that the most common {10–12}⟨10–11⟩ extension twins occurred during nanoindentation of the (10–12) and (10–10)

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planes. Shin et al. [4] studied the role of the extension twins during the nanoindentation of single-crystal Mg on the basal (0001) and second-order prismatic {11–20} planes. They identified zones underneath the indenter with favorable extension twinning and proved that the mechanism of extension twinning is as important as that of dislocations. Sánchez-Martín et al. [5] attempted to interpret the nanohardness of Mg and an MN11 magnesium alloy via nanoindentation with a Berkovich indenter in terms of active slip systems in Mg. A material response to the nanoindentation was simulated using a crystal plasticity finite element (CPFE) model. Concentrating on the presented Mg results, their theoretical predictions roughly approximated the experimental data; however, only a few values of the Mg nanohardness were determined for the CPFE model calibration, which could have led to the omission of some unexpected phenomena. Selvarajou et al. [6] conducted a similar measurement with a cono-spherical indenter and TEM observations and improved the CPFE model by considering basic twinning mechanisms [6]. Two extreme cases of the indentation of the basal and prismatic planes were investigated, and a contribution of the individual deformation mechanisms was discussed in detail.

Although the deformation mechanisms induced by triaxial stresses during the nanoindentation of Mg have been investigated in several detailed experimental studies, only a few crystallographic orientations have been investigated. However, an in-depth nanomechanical analysis covering a number of grains with various orientations with respect to the indentation direction is no less important for the fundamental understanding of processes developed under strictly localized loading conditions. Additionally, neither of the above-mentioned studies was performed using nanoindentation with simultaneous observations using a scanning electron microscope (SEM). Such an approach provides full control over the indentation process, the precise positioning of indents and the immediate avoidance of regions containing impurities, scratches or grain boundaries, all of which can devalue the measurement. Nanoindentation in an evacuated SEM chamber further protects the indented surfaces of sensitive Mg against oxidation and air humidity, which may have a detrimental effect on the nanoindentation results, particularly for shallow indents. There is also the potential for a direct observation of twinning/detwinning phenomena on the sample surface during loading/unloading.

In this study, we thoroughly investigate the nanomechanical properties of the AZ31 magnesium alloy determined by the SEM-supported nanoindentation of individual AZ31 grains with known crystallographic orientations. To infer the influence of the alloying elements, the results obtained for AZ31 are compared with those for pure Mg, which are determined using the same experimental procedure. In addition to the nanohardness, the elastic modulus, the indentation size effect (ISE), and the yield strength are correlated with the crystallographic orientation, and the work of nanoindentation is also analyzed.

2. Materials and experimental methods

2.1. Preparation of samples

An AZ31 sample was prepared from an as-rolled alloy with a nominal composition of 3 wt% Al, 1 wt% Zn, 0.3 wt% Mn (balance Mg) and a mean grain size of 17 μm. The starting material for an Mg sample was a coarse-grained as-cast 99.95% Mg with millimeter grains, which was processed in one pass via equal channel angular pressing (ECAP) and subsequent annealing at 300 °C for 30 min. A relatively strain-free microstructure contained grains with a mean size of 15 μm. With respect to the ranges of translation of the nanoindenter stage [11], such samples provided a variety of crystallographic orientations, whose range was determined by the material texture that was examinable within a reasonable time. The grains were also sufficiently large to allow the placement of a number of indents far from each other and from the surrounding grain boundaries, which could eventually affect the results of the nanoindentation.

For the ISE measurement, the AZ31 and pure Mg samples were additionally thermally treated to create bigger grains. AZ31 was annealed at 500 °C for 17 h, and pure Mg was annealed at 350 °C for 20 min. An average cooling rate of 2 °C per minute was applied, and the mean grain size was found to have increased to approximately 50 μm for both materials.

AZ31 and pure Mg were then carefully cut with a low-speed saw and prepared based on a standard metallographic procedure. Polishing included 3 μm and 1 μm diamond suspensions and a 40 nm colloidal silica suspension (Struers). As the final step, the investigated surfaces of the finished samples were gently etched with 3 keV Ar^+ ions for several minutes (Gatan 682 PECS) to improve the signal quality of electron backscattering diffraction (EBSD) and to reduce the magnitude of residual surface stresses induced during the mechanical preparation of the materials.

2.2. Surface characterization

Individual samples were first examined by an FEI Quanta 3D Dual-Beam SEM/FIB equipped with an EDAX Hikari EBSD detector. Several clean and scratch-free areas were circumscribed with thin shallow frames milled by a focused ion beam (FIB) of 30 keV Ga^+ ions. These frames were clearly visible via SEM and provided unambiguous identification and navigation during the EBSD and nanoindentation. The crystallographic orientation data of grains within the frame interior was collected using an EDAX/TSL 5.31 system (20 keV, 23 nA, 1 μm step) and analyzed with OIM 5.31 software. The samples were consequently removed from the SEM sample holder and attached to the nanoindenter stage; this nanoindenter setup was then installed in the SEM chamber.

2.3. Nanoindentation measurement

In situ nanoindentation was performed using a Hysitron PI 85 PicoIndenter controlled by TriboScan 9.3 software. Grains of the desired crystallographic orientation and size were indented by a Berkovich indenter (see Fig. 1a). Nanoindentation proceeded in a load-controlled mode [11]. A trapezoidal loading function used to determine the nanomechanical properties of materials consisted of 5 s of loading, a 2 s hold with a peak load of 500 μN for AZ31 and 350 μN for pure Mg, and 5 s of unloading (see inset in Fig. 1b). The maximum indentation depth h_{max} in both materials ranged from 95 nm to 125 nm. The trapezoidal loading function for the ISE measurement consisted of the loading and unloading segments with a constant loading rate of 100 μN per second and a 2 s hold interval. The peak loads ranged from 100 μN to 2 mN, which corresponded to h_{max} of 25 nm to 300 nm for AZ31 and 40 nm to 370 nm for pure Mg. A variation in the indentation depth and loading strain rate due to the crystallographic orientation was up to 20% in all measurements. A rate sensitivity influence was expected to be of a minor importance for this study. The experimental error of the correlation between crystallography and nanoindentation was within 3° in accordance with [12].

The number of indents per grain varied from 10 to 20 during the measurement of the nanomechanical properties and from 5 to 10 during the ISE measurement depending on the grain size and surface quality. The influence of the grain boundaries was minimized by indenting the interiors of grains (i.e., more than 1.5 μm from grain boundaries) [13]. The center-to-center distance of the individual indents was approximately 3–5 times the average