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Orientation dependent deformation by slip and twinning in magnesium during single crystal indentation

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Abstract—We present the orientation dependent indentation response of pure magnesium during single grain indentation. A conical indenter and maximum loads between 50 mN and 900 mN were employed. Indent topographies were acquired by confocal microscopy. The indents were also characterized by electron backscatter orientation microscopy for their microstructures. Pronounced activation of specific twinning systems was observed around the impressions. The resulting data were compiled into the inverse pole figure presentation of indent microstructures and topographies after Zambaldi and Raabe, Acta Mater. (2010). Three-dimensional crystal plasticity finite element simulation of the indentation deformation supports the interpretation of the orientation dependent slip and twinning patterns around the indents. The match between the activation of observed and simulated twinning variants is discussed with respect to the conditions for nucleation and growth of extension twins. Furthermore, the compatibility of the twinning strains with the imposed deformation is discussed based on the expanding cavity model of indentation. The orientation dependent response of magnesium during indentation is compared to the literature data for indentation of alpha-titanium and beryllium. Recommendations are given on how to exploit the characteristic nature of the observed indentation patterns to rapidly assess the relative activity of deformation mechanisms and their critical shear stresses during alloy development.

Keywords: Indentation; Twinning; Nucleation; EBSD; Anisotropy

1. Introduction

1.1. General introduction and state of micromechanical models for magnesium

Magnesium alloys are an attractive class of structural materials [1–4]. Their widespread application is currently hampered by the low ductility of many available alloys at room temperature. The goal to increase the use of lightweight materials in structural applications has lead to an increased interest in magnesium wrought alloys in the recent years.

The addition of small amounts of rare earth elements or lithium improves the forming potential of these alloys [5–8]. The understanding of this ductilizing effect advanced considerably in the recent years [9–13]. A method to efficiently quantify the strength of this effect, specifically regarding the change in the relative activation of different types of slip and twinning systems for variations in rare earth contents, could support and even guide the alloy design process.

Due to the pronounced twinning which is commonly observed during the plastic deformation of magnesium, new interest was also triggered for an improved understanding of this deformation mechanism. Furthermore, micromechanical models have progressed and started to incorporate physically-based twinning models [14]. However, these formulations need accurate and physically meaningful parameters to calibrate their internal twin nucleation and growth models. Even if some of the relevant parameters can be predicted by ab initio or molecular dynamics studies [15,12,16], the resulting predictions need to be validated by targeted experiments.

Understanding the mechanisms of twin formation in hexagonal metals is a long-standing problem. Despite the increased amount of research, several key issues have not been resolved yet and a better understanding and quantification of the competition between the predominant deformation mechanisms is necessary to develop improved alloying concepts. Based on current characterization techniques a number of studies have been carried out to clarify the micromechanical processes [17,18] as well as statistical variations [19,20] of the twinning process in hexagonal close packed crystals.

One of the major challenges in the study of twin formation is that the location where a twin will form is generally not known beforehand. Also the growth of twins is fast once a viable nucleus has been formed [21]. Therefore, the initial stages of twinning are very difficult to investigate by experiments. Recently a combination of molecular dynamics simulation and electron backscatter diffraction

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(EBSD) crystal orientation mapping was used to study twin nucleation at grain boundaries in magnesium [19].

The often observed, extended and wedge-like morphology of deformation twins in magnesium cannot readily be incorporated into finite element based simulations. Current research of Glüge et al. [22], Zhang and Joshi [23] and Abdolvand and Daymond [24] shows some promising results on this problem. Most of the literature, however, uses a homogenized formulation for the twinned volume [25–28].

Phase field models have been adapted most recently to represent the kinetics of twin nucleation and growth in a spatially resolved manner [29–32]. The resulting models were applied to indentation-induced twinning of calcite and sapphire [30] and NiAl [32], as well as to crack-induced twinning in magnesium [31].

We develop in this study one possible prerequisite to the problem of calibrating such emerging micromechanical models. Based on the reproducible generation of twins by single-crystal indentation, the resulting microstructures can be easily compared to simulation results. Correspondingly, the models could be validated against these experiments.

1.2. Motivation: limitations of the existing tools to determine relative activation of deformation systems

The most pragmatic way to study micromechanical processes in magnesium is to analyze the microstructure of a polycrystal after deformation. The main advantage of this method is the immediate relevance of the findings to the deformation of wrought alloys. The main drawback of this approach is the multitude of mechanisms that are operating at the same time which makes it difficult to isolate their exact nature. Also, the strong flow anisotropy of individual crystals in magnesium results in a rather heterogeneous local stress field even during homogeneous external loading. Often a complex three-dimensional stress state will be present inside the microstructures. Under these conditions each grain's plastic deformation is of unique character and depends on its immediate surroundings. Owing to these non-homogeneous stress and strain fields, any experimental work in polycrystalline material that does not quantify the deformation mechanisms with good statistics will fail to extract general knowledge. Similarly, any characterization method for polycrystalline materials that does not take into account the third dimension will not be able to explain the observed deformation structures at the surface because of the effects from the material beneath it [24].

Further, the simulation of deformation textures has been applied to the problem of indirectly identifying the predominant slip systems in magnesium through the asymmetric portions of the deformation gradient, causing crystal rotation [6]. While this approach has the advantage to be based on the technologically relevant case of polycrystalline deformation, its drawback consists in the lack of appropriate models to describe the complex deformation of (hcp) metals especially when twinning deformation is present. Also, crystal rotations, i.e. texture evolution, does not always relate to the activated shear systems in a unique fashion. Hence, despite large efforts, no fully satisfying crystal plasticity model is available for magnesium because of the difficulty to link the length scales of microscopic twinning and polycrystal deformation as modeled by the crystal plasticity finite element method (CPFEM).

The relative activity of deformation systems has also been studied by X-ray diffraction experiments [33]. Further volume characterization of deformation processes in magnesium and other hexagonal metals was carried out by 3D X-ray diffraction [34,35] and 3D-EBSD [36].

To isolate the contributions from different deformation mechanisms to the overall strain and the changes in the microstructure, it is desirable to perform single crystal deformation experiments in addition to the mechanical testing of polycrystals. Besides the indentation of single crystals that will be presented here, other kinds of micromechanical experiments have been performed in the past [37–43] such as the deformation by channel die plane strain compression [39–41] or micropillar compression experiments [42,43]. They are discussed and compared in more detail in Section 5.11.

1.3. Crystal plasticity of magnesium

Pure magnesium deforms mainly by basal slip along the $\langle a \rangle$-directions and extension twinning of type $\{10\bar{1}2\}\langle\bar{1}100\rangle$ [37,44,39,45]. It is not clear, however, whether these two types of deformation systems can accommodate arbitrary three-dimensional strain [46,47]. Occasionally the activation of additional slip systems is energetically more favorable and dislocation glide on prismatic and pyramidal systems as well as compression twinning [40,41] have been observed.

Hutchinson and Barnett [45] compiled the literature on experiments conducted on single crystals of pure magnesium and Mg-based alloys and the critical resolved shear stress (CRSS) values derived from them. In magnesium the high ratios between the CRSS values for hard and soft deformation modes largely prevent the harder deformation mechanisms from operating.

Stohr and Poirier [48] have investigated the activation of a second order pyramidal slip in Magnesium. They have reported a minimum of its CRSS around room temperature. Jones and Hutchinson [49] discussed the primary slip systems and dislocation mechanisms of many hexagonal metals.

1.3.1. Twin nucleation

General summaries of the nature of twinning and twin nucleation were given by Christian and Mahajan [50] and Yoo [47]. Aydiner et al. [51] have studied the nucleation of twin variants in an AZ31 alloy during compression of a polycrystal by in situ 3D-XRD. Barrett et al. [52] have studied the Schmid-factor dependency of twin nucleation by molecular dynamics simulation. Abdolvand and Daymond [24] investigated the spatial distribution of twinning in a magnesium polycrystal by crystal plasticity simulations. Yu et al. [53] have reported a size effect on deformation twinning for compression of microcolumns with square cross-sections. This phenomenon has also been discussed by El Kadiri et al. [54].

1.3.2. Twin variant selection

The formation of specific twin variants has been shown to significantly deviate from predictions based on a macroscopic Schmid-factor in several cases [55–59]. Some authors