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Impact of grain shape on the micromechanics-based extraction of single-crystalline elastic constants from polycrystalline samples with crystallographic texture

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ABSTRACT

A micromechanics-based method ("inverse self-consistent approximation") that can extract all the independent elastic constants of single crystals from those of polycrystals with crystallographic texture was newly developed. In the developed method, all the elastic constants in an anisotropic polycrystal were measured by resonant ultrasound spectroscopy, and the crystallographic orientations and shapes of the grains were analyzed. Then, the elastic constants of a single crystal, which reproduce those of the polycrystal, were determined on the basis of Eshelby's inclusion theory and the effective-medium approximation, taking into account the elastic interaction between the grains, which reflects their shapes and orientations. The developed method determined the elastic constants of pure Cu and pure Mg single crystals from those of polycrystals quite precisely. The differences between the elastic constants obtained using our method and the values measured using single crystals were only ~ 1%. In contrast, the application of an inverse Voigt–Reuss–Hill approximation, which cannot consider the effect of the grain shape on the elastic interaction, resulted in a relatively poor evaluation for pure single-crystalline Cu which exhibits strong elastic anisotropy. This indicates that the grain shape clearly affects the elastic interaction between the grains exhibiting high elastic anisotropy and influences the extracted single-crystalline elastic constants. In terms of the actual application of the inverse self-consistent approximation, the single-crystalline elastic constants of AZ31 Mg alloy, whose single crystals cannot be prepared easily, were clarified.

1. Introduction

Elastic properties are the most fundamental mechanical properties in structural, functional, and biomedical materials [1–4]. In particular, elastic properties of "single crystals" are required not only for material design but also for numerical modeling in order to analyze the mechanical properties and microstructure development [5–11]. Also, single-crystalline elastic properties are necessary for analyzing deformation behavior on the basis of lattice-strain measurements using X-ray, neutron, and synchrotron X-ray diffraction methods [12,13].

Despite the importance of single-crystalline elastic properties, they have not been clarified for various materials because of the difficulty in the preparation of single crystals. In general, the measurement of single-crystalline elastic properties requires large single-crystalline samples several millimeters in size [14–16]. However, such large single crystals cannot be prepared in many cases. For example, the single-crystalline elastic properties of various Mg alloys have not been revealed because of problems in specimen preparation.

To overcome the difficulty in the preparation of large single crystals, methods that extract single-crystalline elastic properties using polycrystalline samples have been developed. The most well-known method is lattice-strain analysis during loading of polycrystalline samples using various diffraction methods (X-ray, neutron, and synchrotron X-ray diffraction) [17–25]. Recently, Panel et al. proposed a different approach for the determination of single-crystalline elastic constants from polycrystalline samples using spherical nanoindentation and crystallographic-orientation measurements combined with finite-element simulations [26]. These two approaches are based on the measurement of

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"microscopic strain response inside grains" under applied stress.

Recently, Tane et al. proposed a micromechanics-based method ("inverse Voigt–Reuss–Hill approximation"), which can extract all the independent elastic-stiffness components of a single crystal from those of the polycrystal with crystallographic texture [27,28]. In this method, all the elastic constants in an anisotropic polycrystal are measured by resonant ultrasound spectroscopy (RUS) [29] combined with electromagnetic acoustic resonance (EMAR) [30], which can easily determine the anisotropic elastic properties compared with the measurement of ultrasonic velocity [31], and the crystallographic orientation of grains in the polycrystal are analyzed. Then, the elastic constants of a single crystal, which reproduce those of the polycrystal, are determined, taking into account the crystallographic texture, on the basis of the Voigt–Reuss–Hill approximation [32]. In contrast to the other two methods, which require measurement of microscopic strain inside the grains, "only the macroscopic elastic properties of the polycrystal" are measured in the inverse Voigt–Reuss–Hill approximation. Thus, it can be applied to polycrystals comprising nanograins [33]. It can also easily be applied to low- and high-temperature measurements [28]. However, in the inverse Voigt–Reuss–Hill approximation, the accuracy would be worse for single crystals exhibiting strong elastic anisotropy because the elastic interaction between the grains in the polycrystals is not modeled exactly. Furthermore, the inverse Voigt–Reuss–Hill approximation has been constructed only for single crystals with hexagonal elastic symmetry, and thus it cannot be applied to crystals with other elastic symmetries. Thus, the development of a more accurate and versatile micromechanics-based method is required.

In the present study, a novel micromechanics-based method ("inverse self-consistent approximation") that can determine all the independent elastic-stiffness components of a single crystal from those of the polycrystal with crystallographic texture was newly developed. In the proposed method, the elastic interaction between the grains was modeled adequately on the basis of Eshelby's equivalent inclusion theory [34] and the effective-medium approximation [35], and the elastic constants of a single crystal, which reproduce those of the polycrystal, were determined. Thus, the developed inverse self-consistent approximation would be accurate even in single crystals which exhibit strong elastic anisotropy. To examine the accuracy of the inverse self-consistent approximation, pure Cu and pure Mg polycrystals whose single crystals exhibit strong and weak elastic anisotropy, respectively, were prepared, and the validity of the single-crystalline elastic constants determined from the polycrystals was examined. Also, the validity of inverse self-consistent approximation was examined by comparing the approximation with the Hashin-Shtrikman upper and lower bounds. Furthermore, the inverse Voigt–Reuss–Hill approximation was extended to single crystals with cubic symmetry, and the accuracy was compared to that of the inverse self-consistent approximation. Actual application of the developed method to a material whose single crystals cannot be prepared easily, a Mg–3 mass%Zn–1 mass% Al (AZ31) alloy polycrystal with crystallographic texture prepared by the extrusion process, was carried out and its single-crystalline elastic properties were clarified.

2. Experimental procedure

To prepare directionally solidified (DS)-Cu polycrystals whose crystal growth directions were oriented along the solidification direction, pure Cu ingots of 99.99% purity were melted in a carbon crucible under an Ar atmosphere. Then, the melt was cast into a mold whose bottom was cooled by a water-cooled copper chiller. Also, pure Mg (99.9 mass%) and Mg–3 mass%Zn–1 mass% Al (AZ31) ingots were extruded to cylindrical rods [36], forming the deformation texture in the polycrystals.

The surfaces of the DS-Cu polycrystal were etched using a solution containing nitric, phosphoric, and acetic acids. The microstructure after etching was observed using an optical microscope (Optiphot, Nikon Co., Ltd.). The texture formed by the directional solidification was analyzed using an X-ray pole figure (CuKα radiation; Smart Lab and Ultima IV, Rigaku). For 11 specimens used in the measurement of the elastic properties, the texture in a surface which was perpendicular to the solidification direction was analyzed. The surfaces of the extruded pure Mg and AZ31 specimens were polished using a cross-section polisher (JEOL SM-09010). The microstructure after polishing was observed using a scanning electron microscope (JEOL 7001FTY) equipped with orientation imaging microscopy (TSL solutions K.K. OIM Data Collection and Analysis Ver. 6.1.3), and the electron backscatter electron diffraction (EBSD) patterns were obtained. Also, the texture formed by the extrusion was analyzed using an X-ray pole figure in order to examine the crystallographic orientation in wide-ranging areas of the specimens; for the extruded pure Mg and AZ31 rods, the texture in a surface which was parallel to the extruded direction was analyzed.

For measurements of elastic stiffness, cubic specimens with approximate dimensions of 8 × 8 × 8 and 4 × 4 × 4 mm were cut from the DS polycrystalline ingots and the extruded polycrystals, respectively, using a spark-erosion cutting machine. All the independent components, c_{ij}, of the elastic stiffness of the prepared polycrystalline specimens were measured using RUS [29] combined with EMAR [30] at room temperature. In the RUS analysis, the elastic stiffness components, c_{ij}, were determined from the resonance vibration frequencies of the cubic specimens, where EMAR was used to identify the vibrational modes of the resonance peaks measured by RUS [37]. This mode identification was quite important for the determination of all the independent elastic-stiffness components in the polycrystalline specimens with high accuracy.

3. Experimental results

3.1. Microstructures of directionally solidified and extruded polycrystals

Fig. 1 shows the microstructures of a DS-Cu polycrystal in cross sections (a) perpendicular and (b) parallel to the solidification direction. In Fig. 1 (a) and (b), the grains were isotropic and elongated along the solidification direction, respectively. This indicates that the rod-shaped grains elongated along the solidification direction were formed during the directional solidification. When the shape of the grains was approximated as a spheroid, the aspect ratio a_3/a_1 was regarded as ∞, because the length of most grains along the solidification direction was larger than that of the side of a cubic specimen.

Fig. 2(a) shows the inverse pole figure map derived from the EBSD pattern in a cross section perpendicular to the extrusion direction (ED) of an extruded (EXTR) Mg polycrystal. The $\langle 10\bar{1}0\rangle$ and $\langle 11\bar{2}0\rangle$ directions were mainly observed, which means that the two directions were oriented along the extrusion direction. Correspondingly, the [0001] directions were frequently observed in the cross section parallel to the extrusion direction, as shown in Fig. 2(b). In both cross sections, the shapes of the grains were isotropic, indicating that equiaxed grains were formed by the extrusion. In contrast, the grains elongated along the extrusion direction were formed in an extruded AZ31 polycrystal, as shown in cross sections (c) perpendicular and (d) parallel to the extrusion direction in Fig. 2. In the EXTR-AZ31 polycrystal, the $\langle 10\bar{1}0\rangle$ directions were mainly oriented along the extrusion direction, and the [0001] directions were correspondingly oriented perpendicular