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Extracting single-crystal elastic constants from polycrystalline samples using spherical nanoindentation and orientation measurements

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Abstract

This paper describes a new approach for the extraction of single-crystal elastic stiffness parameters from polycrystalline samples using spherical nanoindentation and orientation measurements combined with finite-element (FE) simulations. The first task of this new approach involves capturing efficiently the functional dependence of the indentation modulus on the lattice orientation at the indentation site and the unknown single-crystal elastic constants. This step is accomplished by probing the function of interest using a suitably constructed FE model of spherical indentation, and establishing a compact spectral representation of the desired function using the discrete values obtained from the simulations. Note that this function needs to be established only once for a selected crystal lattice symmetry. In the second step of the approach presented here, the unknown single-crystal elastic constants for a selected phase are estimated through a regression technique that provides the best match between spherical nanoindentation measurements obtained on differently oriented grains of that phase in a polycrystalline sample (measured by orientation imaging) and the function established in the first step. The accuracy and viability of the proposed approach are demonstrated for an as-cast cubic polycrystalline Fe–3% Si sample.

Keywords: Spherical nanoindentation; Orientation imaging; Single-crystal elastic constants; Spectral representations; Finite-element models

1. Introduction

Development of robust, physics-based, multiscale materials models is significantly hampered by the lack of validated tools and protocols for characterizing reliably the local (anisotropic) properties at length scales at or below the micron scale. Although numerical techniques such as the finite-element (FE) method have been shown to be successful in simulating complex interactions between microscale constituents of a composite material system [1–14], their predictive capabilities are strongly affected by assumptions made about the constitutive laws used to describe the local response of the microscale constituents present in these systems.

It is often very expensive, and sometimes impossible, to produce sufficiently large volumes of the microscale constituents of interest in their pure form to allow the application of traditional mechanical testing methods (e.g. compression or tensile testing). One approach explored in the literature involves the fabrication of micropillars [15–17] using a focused ion-beam and testing these pillars in a scanning electron microscope. However, this approach requires access to highly sophisticated equipment and is not particularly well suited for extracting the elastic properties of the microscale constituents in composite material systems.

In this paper, we present a new approach for estimating single-crystal elastic properties from polycrystalline

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samples using indentation methods, orientation measurements (by electron backscattered diffraction) and FE models. The approach presented here is formulated to solve the following inverse problem: find the values of the fundamental single-crystal elastic stiffness parameters (for a selected material phase) that are most consistent with a set of in-grain indentation measurements obtained on a range of lattice orientations in a polycrystalline sample. This work builds on prior work [18–22] from our research group. As noted above, the challenge posed in the present work is essentially an inverse problem. In the forward direction, it is relatively easy to build a FE model [18,23,24] that predicts the indentation modulus for a selected crystal lattice orientation and a selected combination of single-crystal elastic stiffness parameters. Such FE models are not ideally suited for addressing the inverse problem stated above. In this paper, we introduce a new approach for addressing this inverse problem that is built on the compact Fourier representations used extensively in our prior work to develop a microstructure-sensitive design framework [25–27]. This new approach is developed and presented first in a general framework that is applicable to any crystal lattice symmetry. Further details of the approach and its viability are then demonstrated with a specific case study involving previously published indentation measurements on a polycrystalline sample of Fe–3% Si [20].

2. Spherical nanoindentation theory

Spherical nanoindentation data analyses procedures are largely based on Hertz's theory [28–30], which assumes frictionless, elastic contact between two isotropic quadratic surfaces. The main result of this theory can be expressed as:

$$P = \frac{4}{3} E_{eff} R_{eff}^{\frac{1}{2}} h_e^{\frac{3}{2}}, \tag{1}$$

where P is the indentation load at the elastic penetration depth, h_e. R_{eff} and E_{eff} denote the effective radius and the effective indentation modulus of the sample and the indenter system, defined as:

$$\frac{1}{E_{eff}} = \frac{1-\upsilon_s^2}{E_s} + \frac{1-\upsilon_i^2}{E_i}, \quad \frac{1}{R_{eff}} = \frac{1}{R_s} + \frac{1}{R_i}. \tag{2}$$

In Eq. (2), E and ν denote the Young's modulus and Poisson's ratio of the indenter (subscript i) and the specimen (subscript s), and R denotes the radius. In the purely elastic indentation of a perfectly flat surface, $R_{eff} = R_i$.

Although the theory for elastic indentation of isotropic materials is well established, it is not directly applicable to most in-grain nanoindentation studies on polycrystalline materials since the indentation zone size in such measurements is typically much smaller than the grain size in the sample. At this length scale, most crystalline materials exhibit significant anisotropy in their elastic response. A number of different approaches have been explored in the literature [31,32] to take the elastic anisotropy into account in the analyses of the indentation measurements. Vlassak, Nix and co-workers [33–35] have developed a rigorous analytical framework based on Hertzian theory to address the elastic indentation of anisotropic samples. Their theory indicates that the inclusion of a crystal lattice orientation-dependent parameter, β, into the definition of the effective indentation modulus will adequately capture the anisotropic elastic indentation response of cubic crystals for any arbitrary orientation of the crystal lattice in the indentation zone. More specifically, they suggest:

$$\frac{1}{E_{eff}} = \beta\left(\frac{1-\upsilon_s^2}{E_s}\right) + \left(\frac{1-\upsilon_i^2}{E_i}\right), \tag{3}$$

where E_s and ν_s denote the effective Young's modulus and Poisson's ratio, respectively [34,35], for a randomly textured polycrystalline sample.

In the present study, our goal is first to establish the main underlying features of the Vlassak–Nix theory [34,35] that are central to the indentation analyses protocols discussed in this paper. More specifically, Eq. (3) combined with Eq. (1) implies that the indentation load, P, continues to be directly proportional to $h_e^{\frac{3}{2}}$, even when the sample exhibits an anisotropic elastic response. If, indeed, this relationship holds true for any arbitrary orientation of the crystal, then it allows us to extract an orientation-dependent indentation modulus (the parameter β is expected to be orientation dependent) from the indentation measurement on any crystal in the sample. Note that this would be an extremely important step in addressing the goals of the present work.

In order to establish whether or not an indentation modulus can be defined for any crystal lattice orientation (from the expected linear relationship between P and $h_e^{\frac{3}{2}}$), we developed and employed a FE model. Note that the investigation described above cannot be conducted directly with measurements as easily, because of the difficulties in isolating the elastic portions of the indentation load–displacement curves [19]. Furthermore, numerical approaches such as FE models circumvent many of the difficulties and uncertainties faced in experimental measurements (e.g. identification of the initial point of contact, accuracy in the description of the geometry of indenter, unavoidable plasticity, friction at the contact surface), and are ideally suited for critical validations of fundamental concepts [18].

3. Finite-element model of spherical nanoindentation

The 3-D FE model to simulate elastic spherical indentation of anisotropic crystals was produced using the commercial FE code ABAQUS [36]. The FE model developed for this study is comprised of two 3-D bodies: (i) an elastically deformable sample with an initially flat surface discretized into ~57,000 eight-noded, 3-D, continuum (C3D8) elements, and (ii) a rigid hemispherical indenter of radius 13.5 μm (i.e. the same size indenter as in prior experimental studies [19,20,22]). The size of the sample was selected as 18 μm × 18 μm × 9 μm to ensure that it is much