3-D printing of crystal and polycrystal physical properties

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Abstract. The article contains a compact summary of cause-effect relationships regarding the structure-properties link in crystals and polycrystals. Neumann’s principle is addressed and visualized in tensor and longitudinal surface treatments. Properties databases and representation algorithms are presented. A software package for 3D printing of physical properties’ surfaces, as a helpful tool for anisotropy visualization, has been created. The required parameters and equipment are disclosed. The article includes examples of experimental data obtained from the MPOD database and several practical illustrations.

1. Introduction

Within the area of study and development of crystals and polycrystals, different techniques have been used for interpreting the properties of these materials. The complete characterization of physical properties is given by the tensor representation [1]. Tensors are functional but not perceptive enough. The longitudinal surface representation [2] brings an intuitive, although incomplete, representation of the anisotropy of materials’ properties. In the current work, we present the system developed in our group for the representation of crystals and polycrystals properties as tensors, computer display surfaces and 3D printings.

2. Single crystals properties and surface representations

The concept of a physical property \( K \), in this paper, refers to the linking magnitude in a linear [cause (X) – effect (Y)] [external action – material response] relationship in a given material. This relationship is represented by the following equation:

\[ Y_{i_1,i_2,...,i_m} = K_{i_1,i_2,...,i_m,j_1,j_2,...,j_m} \cdot X_{j_1,j_2,...,j_m} \]  \hspace{1cm} (1)

In (1), summation on repeated indexes is agreed upon. For practical purposes, within this paper we will frequently use the example of the elastic compliance. In general, \( X \) and \( Y \) are tensors with respective ranks \( m \) and \( n \). The property tensor rank is \( r = m + n \). For example, the elastic compliance \( S (\epsilon = S \cdot \sigma) \) is a 4th-rank tensor. \( \sigma \) is the stress \( (m = 2) \) and \( \epsilon \) is the strain \( (n = 2) \). Further examples of the presented concepts can be found in [3].

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The longitudinal surface representations show, in a vivid manner, the anisotropy of physical properties. In short, the origin-surface distance is proportional to the projection of the effect in different action orientations. Representation surfaces, as well as properties tensors, satisfy the Neumann principle: *The symmetry group of any macroscopic property contains as a subgroup the considered structure point group* [2].

Compact matrix notation is one of the most common ways of representing elasto-piezoelectric tensors. Properties matrices differ in their sizes according to the considered property. For the piezoelectricity the size of the matrix is 3 × 6 and that for elasticity it is 6 × 6. Some properties measurement methods are mentioned in [4, 5].

We use the Material Properties Open Database, MPOD (http://mpod.cimav.edu.mx). This open access catalogue has a wide variety of cases from different properties and materials, experimentally obtained. MPOD gives the user the properties’ representation as tensors and as longitudinal surfaces. A basic component of the present contribution is the systematization in MPOD of the creation of .STL files for 3D printing of longitudinal surfaces for single- and textured poly-crystals.

Equation (2) and Figures 1 – 3 show examples of tensor and surface representations of physical properties. Eq. (2) gives the elastic compliance tensor of graphite, structure point group 6/mmm. The numerical data was taken from Bosak [6]. Figure 1a shows the graphite compliance surface representation. The figure caption expresses the fulfillment of the Neumann’s Principle for this example.

\[
\begin{bmatrix}
0.0009 & -0.0001 & 0 & 0 & 0 & 0 \\
0 & 0.0009 & 0 & 0 & 0 & 0 \\
0 & 0 & 0.0258 & 0 & 0 & 0 \\
0 & 0 & 0 & 0.2 & 0 & 0 \\
0 & 0 & 0 & 0 & 0.2 & 0 \\
0 & 0 & 0 & 0 & 0 & 0.002
\end{bmatrix} \text{ GPa}^{-1} \tag{2}
\]

The values of the color bar associated with the figures represent the intensity of the cause-effect relationship. For example, in Figures 1 and 4 to 6, the compliance of graphite can be interpreted as the softness that different samples show to applied stresses. This is showing how resistant (low-softness) it is when applying a stress in the XY plane and lacks resistance (relatively high softness) if the stress is applied in the Z axis, this makes sense since graphite it is easily separated into layers. The discovery of graphene was facilitated by this fact.

Figure 1b shows details of the considered case. The longitudinal compliance \( S(0^\circ) \), which represents the value \( S_{33} = 0.0258 \text{ GPa}^{-1} \) is approximately 29 times greater than the value \( S(90^\circ) = 0.0009 \text{ GPa}^{-1} \). A graphite crystal shows less elastic resistance to a stress in the Z direction than in the XY plane. The maximum value of the compliance (minimum of resistance) is \( S(45^\circ) = 0.057 \text{ GPa}^{-1} \), about 63 times larger than in the horizontal direction.
Figure 1a. Longitudinal surface representation of the elastic compliance for a graphite single crystal. Property point group: $\infty/mmm \supset 6/mmm$. Units in the scale bar are GPa$^{-1}$.

Figure 1b. Representation of the longitudinal surface of the elasticity of a single graphite crystal with a section in the XY plane and the illustration of 3 longitudinal properties $S(\theta)$.

Figure 2. Longitudinal surface representation of the piezoelectric coefficient $d$ for an Aluminium nitride single crystal. Property symmetry $\infty/mm \supset 6mm$. Units in the scale bar are m·V$^{-1}$.

Figure 3. Longitudinal surface representation of the young modulus for an Aluminium nitride single crystal. Property point group $\infty/mmm \supset 6mm$. Units in the scale bar are GPa.

Figures 2 and 3 show, respectively, the piezoelectric coefficient $d$ and the Young modulus surfaces of AlN, structure point group 6mm. These examples further illustrate the Neumann principle.

Surface representations are of great help when analyzing the structure-properties relationship. They display specially well the properties’ anisotropy and the effect of structural symmetry on the expected performance of a given material in device design.
3. Polycrystals properties.
Consider an arbitrary tensor property of a polycrystalline macroscopic body. Let $\bar{X}$, $\bar{Y}$ and $\bar{Z}$ the average action, effect and property. These tensors are considered valid first approximations to so-called effective magnitudes of the polycrystal $\bar{K}$ is the mean property value, averaged over all the crystallites in the polycrystal volume:

$$\bar{K} = \frac{1}{V} \int_V K(r) dV$$

Crystallographic texture plays an important role in the calculation of eq (3). The texture is described by pole figures (PF), inverse pole figures (IPF) and orientation distribution functions (ODF). These descriptors, depending on the specific problem, act as modulating factors in eq (3). Model textures are represented as Gaussian distributions in Euler space or the IPF reference sphere (axially symmetric textures). This component of the work is discussed in detail by Villalobos et al [3].

Figures 4 - 6 show the longitudinal surface representations of the compliance for graphite virtual polycrystals with different orientation dispersions $\Omega$ around preferred (001). The figures show how, as $\Omega$ increases, the representation tends to be a sphere. This means as orientations tend to be random, elasticity tends to be isotropic. Calculations were performed via the Internet, by use of the MPOD polycrystal application.

**Figure 4.** Longitudinal surface representation of the compliance for a graphite polycrystal with $\Omega = 15^\circ$. Units in the scale bar are GPa$^{-1}$.

**Figure 5.** Longitudinal surface representation of the compliance for a graphite polycrystal with $\Omega = 60^\circ$. Units in the scale bar are GPa$^{-1}$.

**Figure 6.** Longitudinal surface representation of the compliance for a graphite polycrystal with $\Omega = 120^\circ$. Units in the scale bar are GPa$^{-1}$.

Scaling the properties from a single crystal to a polycrystalline sample is of utmost importance since the overwhelming majority of real-world materials are polycrystalline. Figures 4 to 6 describe the evolution of axially-textured graphite polycrystals compliance as texture changes from sharp to broad.

In the single crystal case (Figure 1), the compliance maximum occurs approximately at an angle of 45$^\circ$ from the Z axis. In the sharp texture case (Figure 4, $\Omega = 15^\circ$), a number of crystallites contribute significantly to the vertical compliance. As the texture begins to widen, as in the case of $\Omega = 60^\circ$ (Figure 5) the property starts to evolve towards isotropy and the XY plane begins to soften. As the texture resembles a random polycrystal (Figure 6, $\Omega = 120^\circ$) the compliance clearly tends to full isotropy.
4. 3D printing of surface representations.
In the 3D printing process, there are basic variables such as the printer model, the material, and the rendering software. The size, prices and technical characteristics of available 3D printers vary in a relatively wide spectrum, but parameters settings and applied software tend to be standardized.

The most used materials are ABS and PLA, each of them has advantages and disadvantages within characteristics such as hardness and printing temperature. The material used for our prints was PLA (Polylactic Acid) due to its easy handling at room temperature. It does not require a thermal insulation chamber during printing. PLA is biodegradable, i.e. environment friendly. On the other side, PLA is a sacrificial hardness compared to an ABS impression. As properties’ 3D prints are used didactically and with investigation purposes, the time of printing is not a significant variable.

The software used was Cura in version 4.6. This open-use software offers the option of modifying all kinds of parameters such as printing speed in contours, movements and filling, filling pattern, extractor temperatures, layer break, etc.

The nozzle used was a standard 4 mm nozzle which provides fine finishes and resistance in pieces with dimensions close to 10 cm. Adjusting the size of the model, as well as the main parameters mentioned above, has a great impact on the printing time. The extruder temperature was 200°C during the entire printing. The printing speed varies according to the printing region of the model, as can be seen in figure 8.

Since that the contours being finer are prone to deformations, a low speed of around 60 mm/s is recommended. The infill done by straight movements and not needing a fine finish can have a higher speed. A speed of 90 mm/s was used which is at a midpoint of our travel and contours speeds, and finally the travel movements in which no filament is extruded are those in which the speeds can increase significantly, in this case, up to 120 mm/s.

The printing process is layer by layer. Figure 7 shows one of the layers which has a thickness of 4 mm due to the nozzle used. Within this same figure, it can be seen how the infill of the piece is hexagonal. This can have a significant impact on the stiffness of the part, as well as its printing time.
Figure 9. Finished printed piece of monocrystal with a height of 8 cm and a width of 6 cm.

Figure 10. Finished printed piece of polycrystal with a height of 9 cm and a width of 3 cm.

Figures 9 and 10 show the result of the impressions, which can now be used as a reference and facilitate the understanding of the surface representation with malleable examples for the student or researcher. The piece showed no printing errors and can even be painted in such a way that it relates its colors to the values previously shown in Figure 1.

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