

Goniometry of Direct Lattice Vectors Supporting Students' Comprehension of Crystallographic Core Concepts and Demonstrating Image-Based Nanocrystallography

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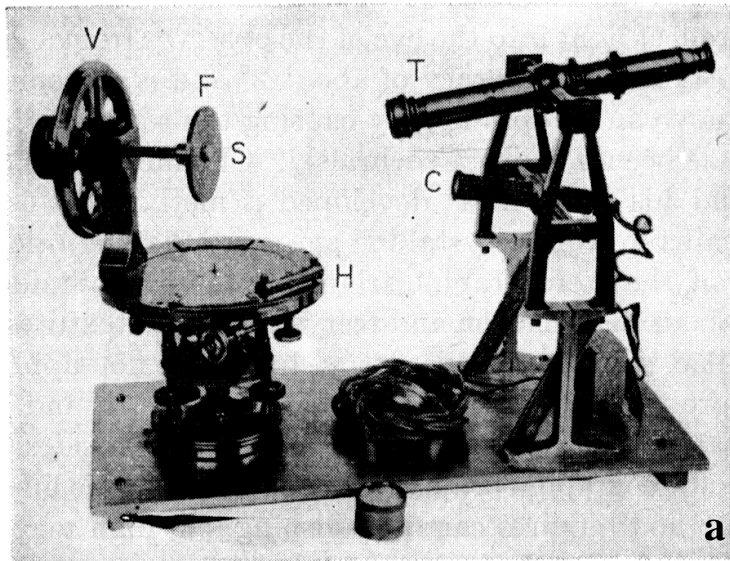
ABSTRACT

We are of the opinion that students of an introductory materials science and engineering course should gain a thorough understanding of crystallographic core concepts by applying them quasi-experimentally in computer simulation sessions that run parallel to the lectures. Software simulations of goniometry of direct lattice vectors in a transmission electron microscope (TEM) will serve two purposes at once: to introduce students to practical aspects of electron microscopy and support their comprehension of crystallographic core concepts. We use the programming software Matlab and Java (Jmol applets) on a PC platform for the creation of software simulations that demonstrate this methodology and complement already existing software simulations. The newly created software is used in classroom demonstrations of an introductory materials science and engineering course at Portland State University and will become freely accessible over the internet. This software will also support and promote image-based nanocrystallography in TEM.

INTRODUCTION

Like many other materials science and engineering educators, e.g. [1,2], we are of the opinion that students of an introductory materials science and engineering course should gain a thorough understanding of crystallographic core concepts. In the European materials science and engineering education tradition, hands-on crystallography by means of optical goniometry supports the students' acquisition and comprehension of crystallographic core concepts. Although a simple apparatus that is perfectly capable of demonstrating the optical crystallography method can be assembled from spare part of an optical microscopy laboratory, see, e.g., Fig. 1a - taken from the first edition of Charles S. Barrett's classical text *Structure of Metals, Crystallographic Methods, Principles, and Data* [3] - and Fig. 1b, we think computer simulations of the actions of different kinds of goniometers are more appropriate for teaching large (i.e. more than about 50 students) classes in the 21st century.

Realizing that it is quite impossible to give a large number of students access to a transmission electron microscope (TEM), we think computer simulations may to some extent also substitute for TEMs in the education of prospective materials scientists and engineers. Software simulations of goniometry of direct lattice vectors in a TEM will, thus, serve two purposes at once: to introduce students to practical aspects of TEM and support their comprehension of crystallographic core concepts. This paper will give a very brief overview of what has already been achieved in developing software for curiosity-driven explorations of the nanoworld [4,5] and mention our plans to develop complementary goniometry software that will support students' comprehension of crystallographic core concepts. The research tool version of this latter software will be used to support and promote image-based nanocrystallography, as described below. First, however, we should like to clarify the position we take in the ongoing debate on the merits of the two basic approaches to teaching introductory materials science and engineering courses in this country.



A two-circle goniometer. *V*, vertical circle; *H*, horizontal circle; *F*, specimen mount; *S*, specimen; *C*, collimator; *T*, telescope.

This simple two-axis goniometer head could complement the apparatus to the left when the specimen (*S*) is fixed to this device and the loaded goniometer head is attached to the specimen mount (*F*).



Figure 1. (a) A simple two-circle goniometer built from spare parts, bits, and pieces before 1943 [3]. (b) A simple goniometer head with two degrees of freedom to adjust a zone axis of a specimen crystal parallel to the vertical circle (*V*) in (a) [6].

THE TWO BASIC APPROACHES TO TEACHING INTRODUCTORY MATERIALS SCIENCE AND ENGINEERING COURSES

In the US, there are two basic approaches to teaching an introductory course in materials science and engineering. The first one is frequently called the *metals first* approach in which structures, mechanical and other physical properties, synthesis and processing techniques, and performances in different engineering applications are first and exclusively discussed for metallic systems. Subsequently the same topics are discussed for ceramics (including semiconductors) and polymers.

Conversely, in the *integrated* (or non-traditional, alternative) approach, the abstract (crystallographic) aspects of structures of the different classes of materials (metals, ceramics - including semiconductors, and polymers) are introduced first and the general properties of the different classes of materials are derived - as far as this is possible in an introductory course - directly from their (ideal and real) crystalline, semi-crystalline, or amorphous structures. Once this general and abstract framework has been laid as the foundation on which the students can organize their thoughts in a systematic manner, specific structures and properties, synthesis and processing techniques, and performances in different engineering applications of each type of material are discussed. In other words, the characteristic of the *integrated* approach is that the general and abstract crystallographic and crystal physics concepts are first taught in a general and abstract way. Only later on, the specifics of chemical compositions, physical properties, synthesis, processing, and performance in different technologically relevant environments of specific materials are added. One, thus, develops a coherent and general theory first and then applies this theory to specific examples, i.e. goes from the more general to the more specific.

The *metals first* approach, on the other hand, does not really develop a coherent and general theory. The multitude of facts that are associated with contemporary materials science and engineering are taught more or less isolated from each other. Examples are the primary teaching aid to support the acquisition and comprehension of crystallographic core concepts. These examples are primarily chosen from metals and alloys.

Evidently, more and more materials science and engineering educators are using the *integrated* approach to teaching introductory materials science and engineering courses [7]. We strongly believe that the *integrated* approach is the more appropriate approach to teaching and learning materials science and engineering in the 21st century. Although this does not seem to be the majority position at present, we feel that we are in good company with this belief.

Robert W. Cahn, FRS, stated for example in his 2002 David Turnbull lecture: “... *the most worrying trend is precisely the reactionary pressure to separate the students of metals and alloys from all the rest.*” [8]. This statement may be understood as an address to introductory materials science and engineering educators to either abandon or not to return to the *metals first* approach. This position is probably no surprise to anybody who has read Robert W. Cahn’s *The Coming of Materials Science* [9].

A few recent undergraduate text books follow the *integrated* approach, e.g., *The Science and Design of Engineering Materials* by James P. Schaffer et al. [10]. This is the text mainly used in Portland State University’s introductory materials science and engineering course, which is taught by the first author of this paper. During this course, the Schaffer et al. text is supplemented by excerpts from Samuel M. Allen and Edwin L. Thomas’s text *The Structure of Materials* [2], which is crystallographically more rigorous.

These supplementations are made because we strongly believe that S. M. Allan and E. L. Edwin are spot on with their remarks: “... *there is a common set of principles governing the structure and properties of many different types of materials ... an understanding of these principles forms the foundation of a modern education in the field of materials science and engineering ... Facility with crystallography is the primary skill for communication in materials science and engineering*” [2]. Following up on the last quoted sentence and arguing with Bernhardt Wuensch [1] that materials science is primarily about the “*relation between the structure of matter and its properties*” and materials engineering is primarily about the “*modification of properties and performance during and after processing, and with manufacture*”, one may define the materials science and engineering super-discipline loosely as being about communications between materials scientist and materials engineers. Surely a “language” is needed for this communication to happen. As far as the crystalline state is concerned, this “language” is in our opinion crystallography and its “words” are the crystallographic core concepts.

As ardent supporters of the *integrated* approach to teaching introductory materials science and engineering courses, we consider the crystallographic simulation programs we are developing as our contribution to promoting this approach.

ALREADY EXISTING SOFTWARE FOR FOSTERING EXPLORATIONS OF BOTH THE NANOWORLD AND THE IMAGE-BASED NANOCRYSTALLOGRAPHY METHOD

Different versions of a simulation program for the exploration of the nanoworld by means of a virtual microscope that combines aspects of TEM and scanning electron microscopy have already been made available by Phil Fraundorf over his web pages [4]. Snapshots from one of these programs are shown in Figs. 2a-c and 3a,b. Exploring this web site as either a homework or a course work assignment shall be fun for students of many introductory science and engineering classes and a rubric for grading such assignments has also been provided [5]. The incorporation of the actual positions of the Si atoms at the thinnest margin of the specimen disk and the unlimited tilt range of the simulated specimen goniometer also allow for the subsequent adjustment of distinctly different two-dimensional projections of the Si atoms. Phil Fraundorf’s program can, thus, also be employed to demonstrate the mechanics of image-based nanocrystallography [11-14], see Figs. 3a,b.

While Fig. 3a was captured at the goniometer setting: tilt = 66.8° and rotation = 97.5° and clearly shows the Si structure in a <110> zone axis projection, Fig. 3b represents the same structure in a <001> zone axis projection, and was captured at the goniometer settings: tilt = 25.7° and rotation = -111°. From these two projections of the atomic arrangement of the specimen at the given goniometer settings (accurate to about 0.1°) of a generic tilt-rotation goniometer alone and a few simple crystallographic calculations, we can conclude that this specimen represents a cubic crystal of the highest Laue group [15].

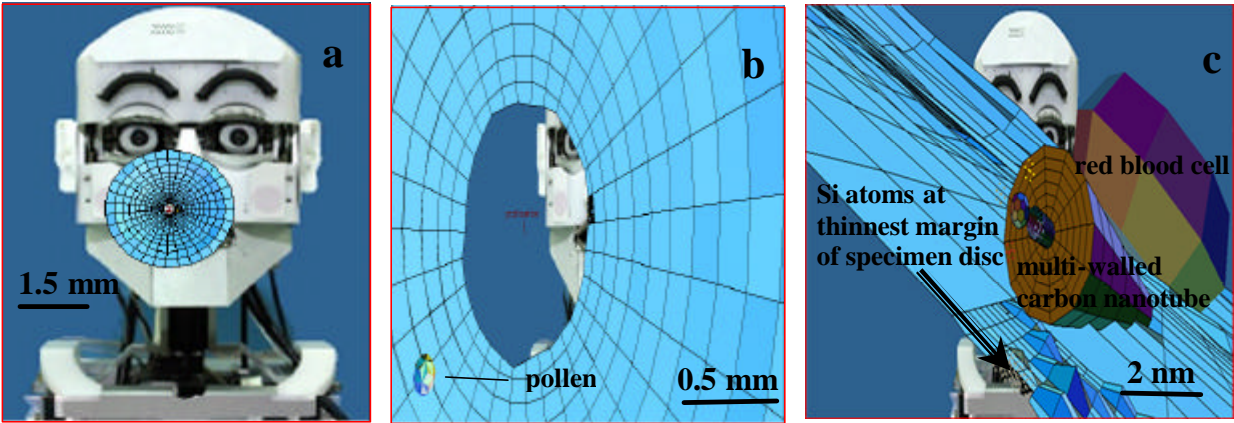


Figure 2. Snapshots from Phil Fraundorf's [4] interactive software simulation program that takes the viewer on a voyage into the nanoworld through zooming and tilting of a generic tilt-rotation specimen goniometer. (Never mind the robot in the background, it just stands for an ever patient observer). The specimen disc is Si, prepared by cutting a three mm diameter disc (a) from a wafer, thinning via abrasion to about 100 micrometer thickness, dimpling from one side and finally perforating by argon-ion milling. As one continues to zoom in with the virtual microscope, one finds a grain of pollen (b), a few red blood cells, ..., a multi-walled carbon nanotube, a so called "buckyball" (the soccer-ball shaped C_{60} molecule with a diameter of 0.688 nm), - just some hypothetical contaminations from the environment for the sake of a comparison of sizes -, and finally the Si atoms themselves (c).

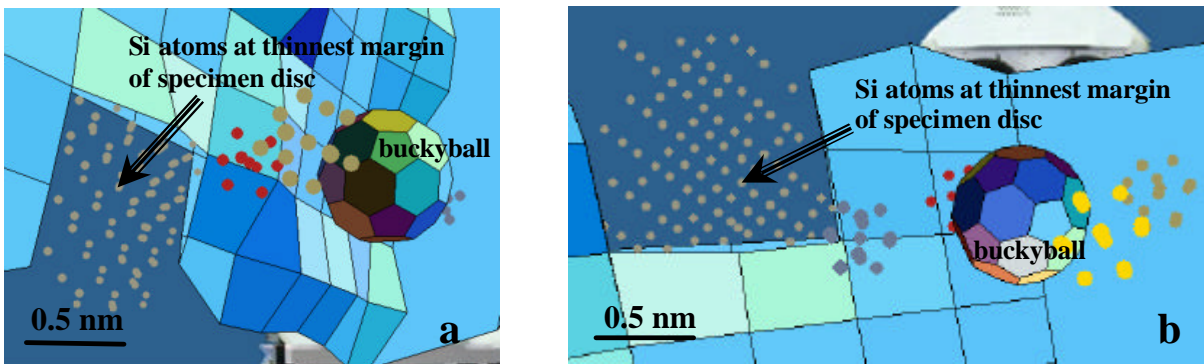


Figure 3. Higher magnification snapshots from Phil Fraundorf's [4] interactive software simulation program. Two characteristic arrangements of the Si atomic columns are shown at the thinnest margin of the specimen disc. An experienced transmission electron microscopists will immediately recognize these two projections as being those of $\langle 110 \rangle$ (a) and $\langle 001 \rangle$ (b) zone axes. The acquisition of this matching pair of images, the recording of their corresponding goniometer settings, and the crystallographic interpretation of these data can be considered as a popular science demonstration of the image-based nanocrystallography method [11-14].

IMAGE-BASED NANOCRYSTALLOGRAPHY

Two complementary versions of imaged-based nanocrystallography have been proposed independently by two of the authors of this paper. While Phil Fraundorf's method [11] can be considered as constituting goniometry of reciprocal lattice vectors, Peter Moeck's method [14] constitutes goniometry of direct lattice vectors. On the practical level, goniometry of direct lattice vectors of any crystalline material involves three parts, (i) the recording of high-resolution TEM images at a set of certain specific orientations, (ii) the simultaneous recording of the respective goniometer readings, and (iii) the crystallographic interpretation of the whole investigation on the basis of a 3 by 3 matrix and/or the stereographic projection of the crystal under investigation. Note here that the 3 by 3 matrix and/or the stereographic projection, i.e. the basis of the crystallographic analysis, are derived exclusively from the

information gathered in steps (i) and (ii). The two-dimensional projections in steps (i) and (ii) may be chosen according to certain tilt protocols [12] and are typically those that correspond to easily recognizable zone axes, e.g. Figs. 3a,b, which are revealed by the crossings of low indexed lattice fringes.

The experimentally derived 3 by 3 matrix is simply the transformation matrix between the coordinate system of the crystal that is to be analyzed by a generalized crystallography procedure and a Cartesian coordinate system that is conveniently fixed to the electron microscope (or an optical goniometer, Figs. 1a,b, or any other kind of goniometry system, as the case may be). Regardless of how the employed goniometer is constructed, e.g. Fig. 1a,b, there will always be a set of simple trigonometric relations that lead from the curvilinear coordinates (setting readings) of the goniometer to the Cartesian coordinates of the system, which the goniometer is a part of. Now, this 3 by 3 transformation matrix is also frequently referred to as the crystal matrix of the direct lattice. This matrix lends itself perfectly to all kinds of crystallographic analyses and calculations. For example the metric tensor is simply obtained by multiplying this crystal matrix with its transposed matrix from the left. From the components of the metric tensor, the lattice parameters can be easily calculated.

Since we made deliberately the connection to classical crystallography by means of optical goniometry in the introduction to this paper, it is straightforward to see that all of the crystallographic analysis techniques that have been developed on the basis of optical goniometry and the stereographic projection for more than a hundred years [16] can be used in modified forms for image-based nanocrystallography in TEM. This is because the information contained in a stereographic projection of a crystal that sits in any kind of goniometer can simply be regarded as a chart that represents the relationships between three coordinate systems. Note that the above description was completely general. Any symmetry the crystal under investigation may possess is not taken advantage of in the calculations. The described procedure is, therefore, applicable to any type of crystal regardless of the symmetry of its atomic arrangement.

It will be noticed here only briefly that image-based nanocrystallography in TEM will become much more viable with the increased availability of aberration-corrected electron optics. This is because with significantly improved point-to-point resolutions down to approximately the size of the Bohr radius of the hydrogen atom, crystal tilts to large angles will no longer be necessary as there is an approximately exponential (or at least strongly super-linear) increase in the visibility of crossed lattice fringes with improved TEM resolution [13]. One can even make the case that one will have so many different two-dimensional projection that reveal crossed lattice fringes that one actually needs a computer simulation of the tilt procedure for a set of model crystals in order to foster the speedy conclusion of an experimental investigation at a future aberration-corrected TEM. The research support version of a former goniometry of direct lattice vectors teaching aid for students' acquisition and comprehension of crystallographic core concepts will then become a research tool in its own right.

FUTURE SOFTWARE DEVELOPMENTS AND STUDENT/EXERCISE EVALUATION

We shall use the programming software Matlab and Java on an IBM compatible PC platform for the creation of software simulations that demonstrate this methodology and complement Phil Fraundorf's nanoworld explorer software [4]. Rubrics for assessing student contributions [5] and conceptual pre/post tests for assessing normalized learning gain [17] are presently under development. Different versions of these programs are going to be teaching aids and research support tools. Our computer program developments will complement existing crystal structure visualization and description programs and data bases that were developed at the Materials Science and Technology Division of the Naval Research Laboratory [18] and will also take advantage of freely available Java-based Jmol applets [19]. The newly created software will be used for classroom demonstrations of crystallographic core concepts in an introductory materials science and engineering course at Portland State University and will become freely accessible over the internet [20].

SUMMARY

As ardent supporters of the *integrated* approach to teaching introductory materials science and engineering courses, we consider the crystallographic simulation programs we are developing as our contribution to promoting this approach. Phil Fraundorf's nanoworld exploration program can also serve as a tool to explore the mechanics of image-based nanocrystallography. We will use the programming software Matlab and Java on a PC platform for the creation of software simulations that demonstrate this methodology and complement already existing crystallographic software simulations. The newly created software will be used for classroom demonstrations in an introductory materials science and engineering course at Portland State University and will become freely accessible over the internet. We also took the opportunity to make a case for the *integrated* approach to teaching introductory materials science and engineering courses by quoting from the writings of leading educators and scientists in the field of materials science and engineering.

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