X-Ray Diffraction

A Practical Approach

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C. Suryanarayana

*Colorado School of Mines
Golden, Colorado*

and

M. Grant Norton

*Washington State University
Pullman, Washington*

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Preface

X-ray diffraction is an extremely important technique in the field of materials characterization to obtain information on an atomic scale from both crystalline and noncrystalline (amorphous) materials. The discovery of x-ray diffraction by crystals in 1912 (by Max von Laue) and its immediate application to structure determination in 1913 (by W. L. Bragg and his father W. H. Bragg) paved the way for successful utilization of this technique to determine crystal structures of metals and alloys, minerals, inorganic compounds, polymers, and organic materials—in fact, *all* crystalline materials. Subsequently, the technique of x-ray diffraction was also applied to derive information on the fine structure of materials—crystallite size, lattice strain, chemical composition, state of ordering, etc.

Of the numerous available books on x-ray diffraction, most treat the subject on a theoretical basis. Thus, even though you may learn the physics of x-ray diffraction (if you don't get bogged down by the mathematical treatment in some cases), you may have little understanding of how to record an x-ray diffraction pattern and how to derive useful information from it. Thus, the primary aim of this book is to enable students to understand the practical aspects of the technique, analyze x-ray diffraction patterns from a variety of materials under different conditions, and to get the maximum possible information from the diffraction patterns. By doing the experiments using the procedures described herein and following the methods suggested for doing the calculations, you will develop a clear understanding of the subject matter and appreciate how the information obtained can be interpreted.

The book is divided into two parts: *Part I—Basics* and *Part II—Experimental Modules*. Part I covers the fundamental principles necessary to understand the phenomenon of x-ray diffraction. Chapter 1 presents the general background to x-ray diffraction: What are x-rays? How are they produced? How are they diffracted? Chapter 2 reviews the concepts of different types of crystal structures adopted by materials. Additionally, the phenomena of diffraction of x-rays by crystalline materials, concepts of structure factor, and selection rules for the observance (or absence) of reflections are explained. Chapter 3 presents an overview of the experimental considerations involved in obtaining useful x-ray diffraction patterns and a brief introduction to the interpretation and significance of x-ray diffraction patterns. Even though the theoretical aspects are discussed in Part I, we have adopted an approach quite different from that of other textbooks in that we lay more emphasis on the physical significance of the phenomenon and concepts rather than burden you with heavy mathematics. We have used boxed text to further explain some particular, or possibly confusing, aspects.

Part II contains eight experimental modules. Each module covers one topic. For example, the first module explains how an x-ray diffraction pattern obtained from a cubic material can be indexed. First we go through the necessary theory, using the minimum amount of mathematics. Then we do a worked example based on actual experimental data we have obtained; this is followed by an experiment for you to do. Finally we have included a few exercises based on the content of the module. These give you a chance to apply further some of the knowledge you have acquired. Each experimental module follows a similar format. We have also made each module self-contained; so you can work through them in any order, however, we suggest you do Experimental Module 1 first since this provides a lot of important background information which you may find useful when you work through some of the later modules. By working through the modules, or at least a selection of them, you will discover what information can be obtained by x-ray diffraction and, more importantly, how to interpret that information. Work tables have been provided so that you can tabulate your data and results. Further, we have taken examples from *all* categories of materials—metals, ceramics, semiconductors, and polymers—to emphasize that x-ray diffraction can be effectively and elegantly used to characterize any type of material. This is an important feature of our approach.

Another important feature of the book is that it provides x-ray diffraction patterns for all the experiments and lists the values of the Bragg angles (diffraction angles, θ). Therefore, even if you have no access to an

x-ray diffractometer, or if the unit is down, you can use these 2 θ values and perform the calculations. Alternatively, if you are able to record the x-ray diffraction patterns, the patterns provided in the book can be used as a reference; you can compare the pattern you recorded against what is given in the book.

This book is primarily intended for use by undergraduate junior or senior-level students majoring in materials science or metallurgy. However, the book can also be used very effectively by undergraduate students of geology, physics, chemistry, or any other physical science likely to use the technique of x-ray diffraction for materials characterization. Preliminary knowledge of freshman physics and simple ideas of crystallography will be useful but not essential because these have been explained in easy-to-understand terms in Part I.

The eight modules in Part II can be easily completed in a one-semester course on x-ray diffraction. If x-ray diffraction forms only a part of a broader course on materials characterization, then not all the modules need to be completed.

We realize that we have not included *all* possible applications of x-ray diffraction to materials. The book deals only with polycrystalline materials (mostly powders). We are aware that there are other important applications of x-ray diffraction to polycrystalline materials. Since this book is intended for an undergraduate course, and some special and advanced topics are not covered in most undergraduate programs, we have not discussed topics such as stress measurement and texture analysis in polycrystalline materials. X-ray diffraction can also be used to obtain structural information about single crystals and their orientation and the structure of noncrystalline (amorphous) materials. But this requires use of a slightly different experimental setup or sophisticated software which is not available in most undergraduate laboratories. For this reason we have not covered these topics.

Pullman, WA

C. Suryanarayana
M. Grant Norton

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