BOOK REVIEW

Spectroscopic Methods in Mineralogy and Materials Sciences (RIMG vol.78), Grant S. Henderson, Daniel R. Neuville and Robert T. Downs, editors. (2014) Reviews in Mineralogy and Geochemistry, i–xvii + 569 pages. ISBN 0-939950-84-7; ISBN13 978-0-939950-84-3. http://www.minsocam.org/ MSA/RIM/rim78.html

Spectroscopic techniques for mineralogical analysis are expanding and improving at an extremely rapid pace. Spectroscopic Methods in Mineralogy and Materials Sciences builds on the foundation presented by its predecessor, Reviews in Mineralogy Vol. 18, Spectroscopic Methods in Mineralogy and Geology, presenting both new techniques and updates to some of those presented in the earlier volume. As a whole, the volume is well organized and related chapters by different authors crossreference one another, providing complementary information without duplicating background information. Most of the chapters provide an overview of the fundamental physics exploited by their respective techniques, the benefits and limitations, and examples of geological problems to which the techniques are applicable. Thus, this book should be helpful both as a general introduction to the array of spectroscopic methods available to mineralogists and materials scientists and as a more rigorous reference volume describing different techniques for those seeking to address a specific scientific problem. Like many of the other RIMG volumes, Spectroscopic Methods in Mineralogy and Materials Sciences is a valuable resource for graduate students and experienced researchers alike.

The volume begins with a series of chapters on X-ray techniques. Chapter 1 introduces X-ray diffraction theory, including the characteristics and advantages of different types of X-ray sources available, from lower energy sources used in local laboratories through high-energy synchrotron sources. This general information provides a foundation for both the X-ray diffraction techniques described in this chapter, as well as for later chapters that focus on different X-ray techniques. It goes on to explain the different methods available for refining crystal structure using different diffraction methods as well as their pros and cons. Chapter 2 introduces the physics of X-ray absorption fine-structure (XAFS). In particular, chapter 2 focuses on the extended XAFS rather than the prominent X-ray absorption near-edge structure (XANES) peak detailed in the following chapter. It begins with background information, describing X-ray absorption and fluorescence. The diagrams included in this section should be particularly helpful to those who have not used X-ray techniques, but seek to better understand the physical principles behind the numbers and data. The theoretical background is followed by details on making and modeling XAFS measurements, including estimation of uncertainty. Chapter 3 is devoted to XANES spectroscopy. Though some background is provided, chapter 3 is specific to XANES and does not repeat the general XAFS information from the previous chapter. Instead, it dives quickly into analysis of X-ray absorption edges for metals and ligands. After a general overview, analytical methods for specific elements are detailed. Although this level of detail might be beyond the interests and needs of a casual reader, who is





SPECTROSCOPIC METHODS

in Mineralogy and Materials Sciences

EDITORS: Grant S. Henderson, Daniel R. Neuville, Robert T. Down



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interested in the technique, it is a welcome reference for those seeking to use XANES or better understand XANES analyses of specific elements. The chapter concludes with a few short examples of studies using XANES. Chapter 4, the final chapter on X-ray techniques, describes the use of X-ray Raman (XRS) scattering to probe bonding at high pressure. In this technique, hard X-rays are tuned to the binding energy of electrons in the elements of interest. Hard X-rays penetrate high-pressure sample capsules, enabling the study of high-pressure phases in-situ. Some of the drawbacks of XRS are explained, which includes the long collection times required to produce acceptable signal to background ratios. Examples of XRS variations as a function of pressure are presented, as are several examples of applications.

The next several chapters cover various unrelated techniques, but each of these chapters has been structured similarly. These chapters begin with an explanation of the physics of the technique, followed by a description of instrumentation and analysis, inherent limitations, and specific scientific applications. Chapter 5 details luminescence spectroscopy, describing the properties of crystals that can be deduced from luminescence spectroscopy including site occupancy and partitioning of elements among crystallographic sites. Applications for several minerals are included. Chapter 6 delves into analytical transmission electron microscopy (AEM). A brief introduction to diffraction patterns in crystalline and amorphous material is presented, though the chapter focuses more heavily on energy-dispersive X-ray (EDX) and electron energy-loss spectroscopy (EELS).

Chapters 7 and 8 cover techniques used primarily for exam-

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ining the physics and chemistry of mineral surfaces. Chapter 7 focuses on core- and valence-level X-ray photoelectron spectroscopy (XPS). The physics of this technique are presented in great detail, specifically detailing XPS of silicates. Chapter 8 describes atomic force microscopy (AFM). This technique uses a tip attached to a cantilever beam that is deflected by interactions with the surface and measured with an optical beam. Unlike scanning tunneling microscopy, this technique allows measurement of the surfaces of both conductors and insulators. After explaining the various experimental methods and details of sample preparation, a number of applications are described, including studies of dissolution reaction rates, hydration forces, and surface structures.

The next several chapters detail an assortment of optical methods, which exploit the reflection and scattering of ultraviolet (UV) through infrared (IR) light. Chapter 9 provides a concise but rigorous introduction to the types of absorptions in the near-UV through the mid-IR. Though quantitative measurements like site occupancy and transition metal concentration can be derived from optical spectra, the advance of smaller spatial scale and higher elemental precision techniques has resulted in optical spectra being less commonly selected for laboratory analyses. However, optical spectroscopy is a powerful tool for studying planets from orbit or telescopically. Chapter 10 introduces the topic of spectroscopy from space, providing some background on surface scattering issues and frozen volatile signatures. An overview of mineral and volatile detections through the solar system is included, but this review is heavily weighted towards discoveries on Mars and on the solid bodies in the outer solar system and provides only limited references to other similar reviews in the literature. Chapter 11 details Fourier transform infrared (FTIR) microscopy and imaging, outlining the technicalities of narrowing the spatial resolution of IR analyses, instrumentation, and examples of applications. Chapter 12 provides an excellent summary of the hidden errors that are commonly overlooked in UV through IR spectra of minerals, which should be a helpful reminder to those who work with remotely sensed optical spectra.

Chapters 13 and 14 focus on techniques that measure scattered

optical radiation. In Chapter 13, the authors describe Raman spectroscopy, summarizing instrumentation and data reduction and applications to crystalline and amorphous materials. Chapter 14 focuses on Brillouin scattering, a powerful technique to determine the acoustic velocities and characterize elasticity in materials by observing inelastic scattering of light. The chapter details physical principles, instrumentation, and applications for addressing geophysical questions by probing samples in-situ using Brillouin scattering at high pressures and temperatures.

Chapter 15 describes nuclear magnetic resonance (NMR) spectroscopy of solid and molten inorganic materials. Spin states of nuclei depend on structure and neighboring nuclei, so pulsed manipulation of nuclear spin states can be used to probe mineral site occupancy. NMR can be used in-situ on molten materials, making it particularly useful for studying short-range order in silicate melts and phase transitions. Chapter 16 focuses on electron paramagnetic resonance (EPR), presenting principles of EPR and information about how experiments are conducted and analyzed. This is followed by examples of applications, including probing point defects in minerals and geochronology.

The final three chapters focus not on specific spectroscopic techniques, but rather on broadly applicable advances in spectroscopic analysis. Chapter 17 describes how, due to major advances in computing power, many types of spectra can be computed from first principles. Chapter 18 focuses on the integration of synchrotron-based spectroscopy with high-pressure apparatuses such as diamond-anvil cells, and Chapter 19 details in situ high-temperature experiments. Taken together, these capabilities provide an opportunity to theoretically and empirically explore material properties and phase transitions deep within the Earth and other planets.

RACHEL L. KLIMA
Applied Physics Laboratory
Johns Hopkins University
11100 Johns Hopkins Road
Laurel, Maryland 20723-6099, U.S.A.

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