INSTRUMENTS FOR MATERIALS ANALYSIS

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TO

Jo,

Lucy, Andrew, and Jon

PREFACE

The analytical characterization of raw materials and finished products is a prime need of those involved in research and quality control. The increasing necessity for this kind of information is documented by the growing number of institutionally-affiliated as well as private laboratories which provide analysis and characterization as a service. More often than not several instruments are required to corroborate results and to fully characterize a given material. The burden of specifying which instrumental analyses are required and of interpreting the resulting data often falls on the individual who may not be aware of the potential or the limitations of any given instrument.

At the university level courses are available which deal in depth with specific analytical instruments. Only recently is the broader approach - one which utilizes a variety of complementary instruments - being recognized as an essential component in the general body of academic knowledge. The immediate problem confronting both the teacher as well as the student is the lack of suitable textual material. To be sure, many excellent texts exist which present a detailed understanding of a specific analytical method. The acquisition of even a few of these texts involves considerable financial investment. Furthermore, such texts are by their very nature highly specialized in the presentation of detail. Such detail is of debatable significance to the student whose needs are of a more qualitative and panoramic nature. Thus there is a need for a text which deals in a comprehensible, yet not superficial, manner with a number of naturally-complementary analytical instruments frequently used in the characterization of materials. This is the background against which this book was conceived and written.

An analysis of university courses which are offered as well as the published scientific and technological literature reveals the recurrent use of the light microscope, some form of X-ray analysis, and one or more of the variants of electron beam instrumentation. These particular methods form the subject matter of this text. The attempt to describe the characterization of bulk materials using light, X rays, and electrons in a single text of reasonable size necessitates the deletion of a great amount of detail. The process of selection and deletion is by nature a subjective one.

The purpose throughout this text is to present pertinent information regarding certain analytical instruments so that the student can understand not only how they function but also what kind of information can be reasonably expected. Some thought is also given to making the reading of the text a pleasant chore. The aim, however, is to clarify. Where the text fails to do this, suggestions are welcome.

In general the "SI" system of units (Systéme International d'Unités) has been used throughout. The one exception is retention of the Ångström (Å) unit. It is my peculiar belief that there is cultural and traditional value in its retention. It would be unfortunate if it were to undergo the same sort of dehumanization as did Röntgen's rays. The conversions are as follows:

$$1Å = 10^{-10}m = 10^{-8}cm = 10^{-7}mm = 10^{-4}\mu m = 10^{-1}nm$$

Appreciation is expressed to the following:

Prof. Dr. Josef Frechen, Ordentliche Professor, and Prof. Dr. h.c. Alfred Neuhaus (†), Director, two of my teachers at the Mineralogisches Institut der Rheinische Friedrich-Wilhelms Universität, Bonn (on the beautiful banks of the Rhein) West Germany,

Prof. David R. Wilder, Chairman, and Prof. Karl A. Gschneidner, Jr., Distinguished Professor, both faculty members in the Department of Materials Science and Engineering, Iowa State University, for their supportive interest and constructive criticism,

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Elmer A. Rosauer

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I
LIGHT MICROSCOPY

1. NATURE OF LIGHT

1.1 Historical Notes

A scientific theory is considered valid when it will at any time satisfactorily explain a group of known and associated phenomena without leading to contradictory conclusions. If new facts become available which are not included in the theory or cannot be satisfactorily explained by it, then the theory must be enlarged upon, redefined, or even discarded for a newer, more comprehensive one. From a historical standpoint no other area in physics as light and light optics has gone through such a see-saw of theory formulation, modification, rejection, and reformulation.

The Greek philosopher Plato believed that particles were shot out from the eyes thereby spraying surrounding objects and making them visible. Isaac Newton (1642-1727) was satisfied in explaining rectilinear propagation of light, reflection, and refraction by means of his emission or corpuscular theory. However, some clever fellow came along and aimed a broad beam of light at a pinhole in black cardboard. According to the corpuscular theory, only the light corpuscles in line with the hole would be seen. In reality, light was seen on the back side of the cardboard from almost any angle because light passes through the pinhole in a spherical manner, best explained by diffraction and interference. So both C. Huygens (1629-1695) and A. Fresnel (1788-1827) proposed a scalar wave theory which gave satisfactory explanations for light-associated phenomena found up to that time. But in 1808, E. L. Malus appeared on the scene and embarrassed all the theorists with the new-found fact that light could be polarized. So T. Young, A. Fresnel, and D. F. J. Arago worked on the interference of polarized light and found the transversal character of light waves. About 1811 they suggested a vectorial elastic light theory, for which Fresnel can take the most credit. This theory was so good and inclusive that it explained not only known light phenomena but also successfully anticipated some new ones. Simply stated, the Fresnel theory said that any disturbance would be carried in any direction by periodic pulses through small elastic spheres in contact with one another in a hypothetical but omni-present medium called aether. Physicists today emphatically rule out the presence of aether simply because all experiments set up to establish its presence have had negative results. The final blow to the elastic light theory came with the discovery of electro-optical phenomena which suggested that there had to be a close relationship between magnetic, electrical, and optical properties. Finally the Scottish physicist James Maxwell (1831-79) presented his electromagnetic light theory which effectively pulled together and made sense out of the above phenomena. This new interpretation of light waves proposed that propagation of light in a nonabsorbing medium was identical to propagation of electromagnetic waves in an insulator. The inevitable happened once more with the discovery of the photoelectric effect which appeared to be most adequately treated by means of the corpuscular theory.

It came as a relief then in 1924 when Louis de Broglie suggested that some phenomena are best understood in terms of wave nature; others, in terms of the corpuscular theory, and finally those which take a neutral position, i.e. understood by means of both theories. At the present time it is impossible to describe all optical phenomena with a simple single theory. The field of quantum mechanics continues today to unravel the apparent contradictions.

1.2 The Electromagnetic Spectrum

The electromagnetic spectrum includes gamma rays, X rays, visible light, infrared, and radio waves. In a vacuum each of these waves travels at a common velocity (v) of 3×10^{18} Å [1] per second; but each has a different wavelength (λ) and frequency (f). Visible light, which stimulates the eye to give vision and color, occupies a very narrow frequency band in the total electromagnetic spectrum, as shown in Figure 1-1. Visible light is a form of radiant energy consisting of packets of energy called quanta. The concept of quanta neatly combines the characteristics of corpuscles and waves: light of short wavelength has more waves

1. An A with a ° above it, thusly Å, is the accepted abbreviation for the angstrom, a unit of measurement equal to 10^{-10} meter. Current efforts to convert to an all-metric nomenclature involves the use of the nanometer (nm), such that $1\ nm = 10^{-9} m = 10$ Å. Nonetheless use of the Å continues.

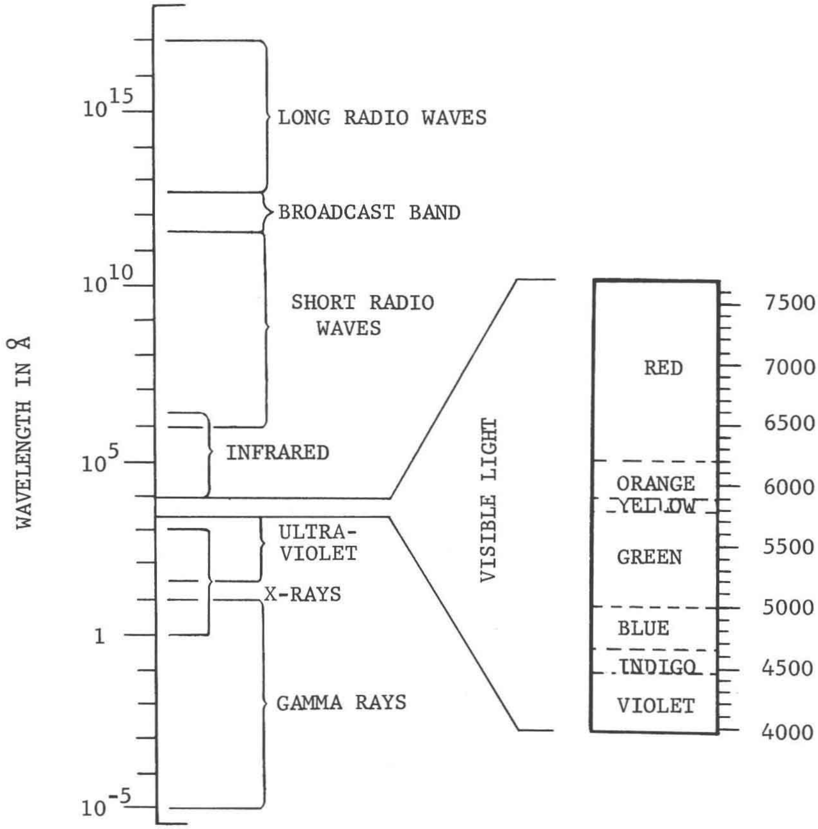

Fig. 1.1 The electromagnetic spectrum

in each bundle than light of longer wavelengths. The energy of a single quantum is a function of the frequency, or $E = hf$, where E = energy in ergs/second; h = Planck's constant, and f = frequency of the radiation.

It may be somewhat easier to conceive of the visible spectrum in terms of wavelength as below:

Wavelength, λ, Å	Color sensation
7000-6470	Red
6470-5850	Orange
5850-5750	Yellow
5750-4912	Green
4912-4240	Blue indigo
4240-4000	Violet

There are at least two memory bridges for remembering the order of visible wavelengths: "Richard of York Gained Battles in Vain" and "Roy G. Biv". An interesting sidelight is the fact that the light-adapted eye is most sensitive to the yellow-green color sensation at 5560 Å which explains the eye-catching color of jackets worn by highway workers.

The mechanism of visual perception is sufficiently complicated without asking the question of how a specific color is sensed. Yet if all visible wavelengths simultaneously strike the retina, "white" light is seen. Sky light or light from an incandescent bulb is polychromatic, i.e. it contains a range of wavelengths as contrasted to monochromatic light, such as from a sodium or mercury vapor lamp. Polychromatic light sources can be equipped with a blue filter which absorbs some of the yellow yielding a "whiter" light. But for sophisticated optical measurements a monochromatic light source, e.g. a sodium vapor lamp, is generally used.

1.3 Wave Nature of Light

If a monochromatic light source, such as a sodium vapor lamp, is placed behind a black cardboard in which a pinhole is punched, a beam of light will be seen projecting out from the pinhole. Blowing smoke across the hole helps to define the beam of light. This light can be considered as traveling along the beam by means of a wave motion (sine function) in which the vibration or oscillation of the light quanta are perpendicular to the direction of travel, i.e. the plane of oscillation is at right angles to the forward motion. A way of describing light motion is by a periodic change in the amount and direction of the light vector with time. In the left part of Figure 1-2 imagine a point which starts at $\pi/2$, moves with a constant velocity in the direction of π, over

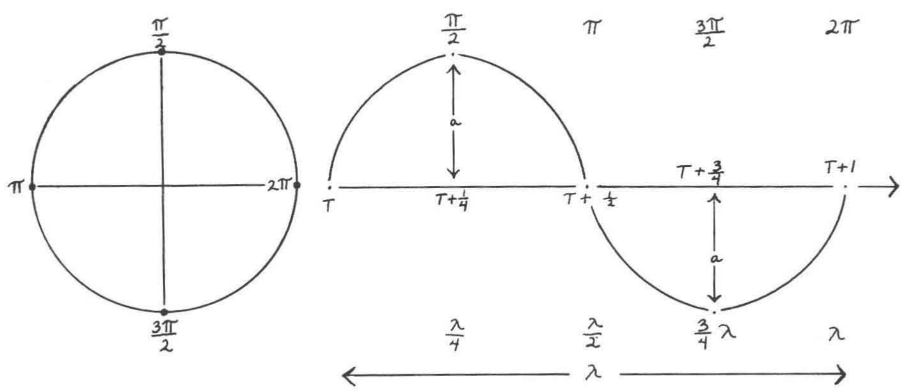

FIG. 1-2 Simple harmonic vibration or sinusoidal wave motion.