

From the Editor



100 Years of Powder XRD

In 1916 Peter Debye and Paul Scherrer invented the powder method of X-ray diffraction at the University of Göttingen. This method uses a monochromatic X-ray beam to irradiate a fine powder of an unknown solid. Cones of X rays are diffracted from the sample at specific angles for which Bragg's Law ($\lambda = 2d_{hkl} \sin\theta$) is satisfied. The positions and intensities of the diffraction peaks form a specific signature for each crystalline substance.

Indeed, in 1919 A. W. Hull, who independently discovered the method in the United States, gave a paper titled "A New Method of Chemical Analysis" in which he stated that "every crystalline substance gives a pattern; the same substance always gives the same pattern; and in a mixture of substances each produces its pattern independently of the others." Powder X-ray diffraction (XRD) allowed the identification of crystalline substances from diffraction peaks on films exposed in the Debye-Scherrer powder camera. Even different compositions of solid solutions could be detected since changes in crystal lattice parameters cause shifts in high-angle peak positions.

This story has an important connection with light microscopy of metals (metallography). In 1900 the first equilibrium phase diagram was plotted showing the phases and phase mixtures present as various compositions of two elements (Fe and C) were held at different temperatures. This work was accomplished by examining the phases present using reflected light microscopy since high-temperature microstructures could be retained by quenching specimens into water. But there was a problem. While these phases could be seen to appear at specific compositions and temperatures, the identity of the phases was unknown. Enter powder XRD. Here for the first time these phases could be identified. This led to a particularly active period of phase diagram determination between 1920 and 1930.

Incidentally, the ASTM Committee E4 on metallography also was organized in 1916. In 1924 a subcommittee was formed on X-ray methods, and in 1940 efforts were begun to harness powder XRD as a general method of chemical analysis. Within a year nearly 4,000 powder patterns of crystalline substances were collected into a file catalog. This led to the formation of the Joint Committee on Powder Diffraction Standards (JCPDS), an effort that survives today as the International Centre for Diffraction Standards (ICDD). The latter organization, markets a database known as the Powder Diffraction File (PDF-4+). An unknown substance can be identified by entering a few d-values from a Debye-Scherrer camera, or its successor the X-ray diffractometer, into this searchable database containing the powder diffraction patterns of nearly 400,000 inorganic materials.

Charles Lyman
Editor-in-Chief

Publication Objective: to provide information of interest to microscopists.

Microscopy Today is a controlled-circulation trade magazine owned by the Microscopy Society of America that is published six times a year in the odd months. Editorial coverage spans all microscopy techniques including light microscopy, scanning probe microscopy, electron microscopy, ion-beam techniques, and the wide range of microanalytical methods. Readers and authors come from both the life sciences and the physical sciences. The typical length of an article is about 2,000 words plus figures and tables; feature articles are longer. Interested authors should consult "Instructions for Contributors" on the *Microscopy Today* website: www.microscopy-today.com.

ISSN 1551-9295

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Magazine website:

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Free subscriptions are available

Publisher

Cambridge University Press
32 Avenue of the Americas
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(212) 337-5000

Circulation: 18,000

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