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Characterization of Materials by X-Ray Microanalysis and Other Techniques

J. I. Goldstein

Mechanical and Industrial Engineering, University of Massachusetts, Amherst, MA, USA

Henry Clifton Sorby is known as the father of metallography, as he developed the preparation of metals, meteorites and then steels (Fig. 1) so that they could be viewed by reflected light (in1863,150 years ago). Although the optical microscope is a powerful and widely used instrument, it provides limited information on the chemical composition of the observed specimen. Its spatial resolution is limited to about 1 micron and it has a short depth of field limiting examination of rough surfaces.

The measurement of chemical composition at the micron level started with the development of the electron probe microanalyzer (EPMA) by Castaing in the early 1950s [1]. A focused electron beam of ≤1micron excites x-rays at a point on a specimen. Using wave length x-ray spectrometers, element standards, and correction factors for atomic number, absorption, and fluorescence (ZAF), one can obtain quantitative chemical analysis of a ~1micron region (Fig. 2) [2]. The analysis is highly dependent on specimen preparation since a flat surface is necessary for accurate microanalysis.

The EPMA was improved by scanning the electron beam [3] to produce a picture of x-ray intensity distribution of the various elements present in a sample, (Fig. 3), a picture of backscatter electrons which are a function of atomic number, and a picture of surface features (the modern SEM). Another improvement was the development of the energy dispersive spectrometer, allowing for rapid qualitative analysis [4]. Even in the early days of the EPMA, Peter Duncumb began improving spatial resolution for microanalysis. He analyzed thin samples in the high voltage transmission electron microscope (TEM) which improved the spatial resolution of x-ray microanalysis. The development of quantitative thin film analysis by Cliff and Lorimer [5], enabled an x-ray microanalysis instrument (AEM) with spatial resolution ≤ 100nm. Fig. 4 shows an early AEM x-ray quantitative scan. Since spatial resolution improves as the specimen thickness decreases, the development of specimen preparation instrumentation, such as the focused ion beam (FIB) dual beam (ion and electron) instrument, was critical to preparing thin specimens. Equally important is the capability of the FIB to make thin specimens at specific analysis regions of a specimen. With the use of wide angle acceptance angle EDS detectors, it is now possible to obtain useful x-ray signal with spatial resolutions, pixel sizes of 1-2nm (Fig. 5). The development of computer analysis procedures to handle very large quantities of data at each pixel in a scanning picture [6] has led to chemical analysis pixel by pixel in large data arrays. High spatial resolution with pixel sizes ≤1nm, has been achieved in SEM instruments using field emission guns and improved electron optics and detailed surface analysis has improved with low voltage analysis.

Characterization of materials has improved greatly since Sorby's initial improvement of light microscopy. The developments in the last 50 years have been revolutionary. Predictions of the future are hard to make but it is clear that x-ray measurements will be improved by better detectors, and sample preparation will continue to be limiting. Instruments with multiple energetic beams will allow for improved characterization of materials and computers will increasingly handle ever larger amounts of data. Finally, because of cost, it will be difficult for most laboratories to own the new multifunctional instruments and therefore investigators will be increasingly dependent on large multi-user facilities [7].

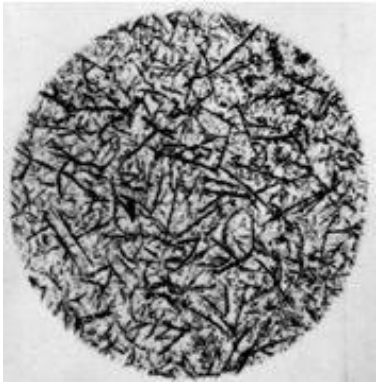

Fig 1a High C iron, Sorby 1863

Fig 1b Bristol iron meteorite, Berha Etch, 20x, 500 μm bar (George vander Voort)

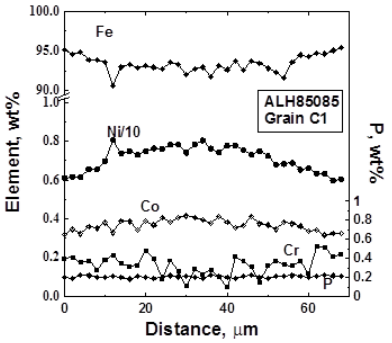

Fig 2 Composition vs distance profiles in CH chondrite ALH 85085 Grain C1 [2]

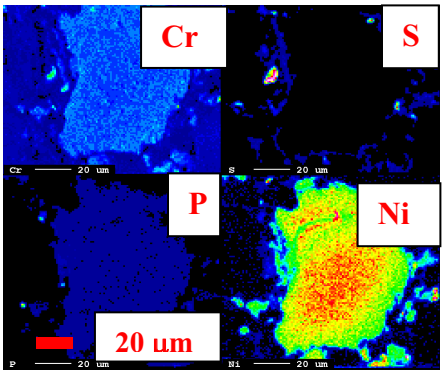

Fig 3 EPMA X-ray scanning of Grain C1 in CH chondrite ALH 85085 [2]

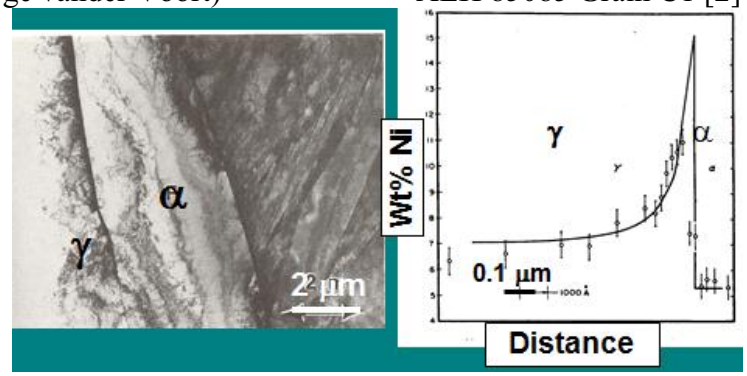

Fig 4 AEM Ni x-ray scan across α and γ phases in a Fe-6.9Ni-0.5P alloy, cooled 5^0C/day.

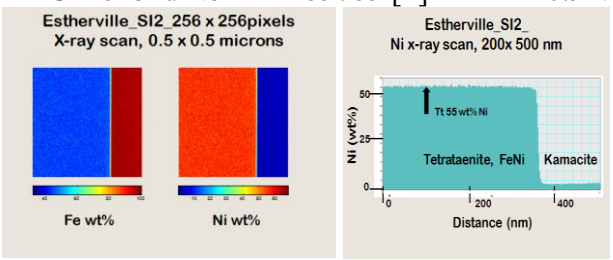

Fig 5 X-ray scanning and composition profiles across α/γ interface in the Estherville meteorite. Resolution 2nm per pixel

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