

SUMMARY OF PAPERS ON THE ELECTRON PROBE

BY LEROY L. WYMAN¹

One of the principal reasons for holding a symposium such as this is to evaluate progress in a particular line of endeavor and to make some attempt to foresee the future.

Metallography, as one recognizes that branch of metallurgy, is now approximately a century old. During all of this time metallographers have been increasingly aware of the need for a means of determining the composition of separate phases *in situ* in the microstructure they have been studying. In fact, a tremendous amount of effort has been expended in trying to obtain such essential data by various elaborate indirect methods.

The advent of the electron probe just a dozen years ago through the efforts of Castaing provided the metallographer with the most powerful analytical instrument since the advent of microscopes and X-ray diffraction.

In the series of papers on the electron probe presented as a part of this symposium there is reflected the entire knowledge and experience of the technical effort in the United States since Castaing's disclosure. The authors of these papers cover the entire range from those who hurriedly converted electron microscopes to probes in order to participate in this new field of analysis to those who are responsible for the design of the most sophisticated of current American probes. From these presentations one can ascertain what has

been learned through the use of the probe, what the problems are in probe application, and what improvements may be expected in the future.

In the area of the initial application of the electron probe, in metallography, Heinrich has presented an extensive array of examples wherein the analytical power of the probe has been employed in problems varying from phase and inclusion identification on through diffusion and phase diagram studies and into the areas of surface studies. In addition, some of the newer phases of probe analysis are presented wherein scanning techniques portray the distribution of elements in a structure, and supplementary instrumentation can give fine-structure information.

From Adler's discussion of the application of the probe to research on minerals one learns of a new field of application and some of the problems encountered, such as the decomposition of structures under the beam and the effects of grain size. Also Adler, as did Heinrich, devotes an appreciable amount of discussion to the necessity for careful preparation of the specimen surfaces to be examined.

Biology, as with metallography and mineralogy, has traditionally employed the microscope as the principal analytical instrument, and now through Tousimis' presentation one learns of the extensive use to which the probe has been used most successfully. Of particular interest, and in nature quite akin to the metallographers' use of the probe in diffusion

¹ Consultant to Division Chief, Metallurgy Division, National Bureau of Standards, Washington, D. C.

studies, is the outstanding result showing the segregation of copper in a thin membrane of the eye. Additionally, the ability to determine calcium and phosphorus levels of composition locally *in situ* has contributed materially to the knowledge of calcification processes and bone growth.

These papers on application brought forth excellent discussions, one of the most significant being Mr. Clark's comment that the electron probe had already proved to the metallographer that many supposedly homogeneous solid solution phases were usually quite varied in composition, and that this would necessitate a very critical re-examination of past work and concepts concerning the nature of these phases.

The second group of papers are concerned primarily with the method and instrumentation for electron probe analysis, and it is from the authors of these papers that one learns that, while this method is an X-ray method of analysis, it differs materially from the fluorescent analysis in that it is a local analysis using a very small source whereas the fluorescent analysis is a large-area sampling giving the average composition of the bulk material. As a consequence, many instrumental and correction factors are quite different for the two methods.

From Ogilvie's discussion of the X-ray optics of the probe, one learns of the different design principles which may be employed, as well as the design requirements for the spectrometers which can best employ the means of obtaining high signal-to-noise ratios. Also involved in this subject is the consideration of the monochromator crystal with respect to its perfection, dispersion, and intensity.

In another vein, Fisher calls attention to many of the factors in probe design including types of lenses, focal lengths, spherical aberration, astigmatism, and beam focusing, most of which have

optimum values of functioning and all of which should be operated near their peak for optimum probe performance.

From still another viewpoint, Birks discusses many of the significant factors that go into the techniques of probe analysis wherein the hope is held forth for the extension of probe analyses to include sodium and carbon, and that some analyses could be accomplished by substituting high for low atomic numbered atoms and analyzing for the amount of the substitute. In addition, this author cites the benefits that may be obtained through the use of thinner specimens and also by the use of extraction techniques and transfer to replicas.

In nearly all of these papers there is repeated reference to the major problem of corrections which must be applied to the raw data in order to improve the quality of the analysis. Castaing discussed this at length on his initial visit to this country in connection with probe research, and cited the great magnitude of the problem, particularly if this had to be accomplished by experimental methods. Since then, it has been approached from many angles, but in Wittry's detailed discussion of these corrections there is hope of a reasonable solution of this problem by the use of existing theories in order to predict the nature of the necessary corrections, coupled with the use of empirical results to give a measure of the magnitude of the corrections.

In summary, the papers presented have shown the expansion of probe application from metallography into mineralogy and biology; they have revealed the many factors of probe design and operation which affect optimum results, and have given some promise of improving the necessary corrections to probe data—all of these holding the promise of better probes and better results in the future.