

## SYMPOSIUM ON ADVANCES IN ELECTRON METALLOGRAPHY

## INTRODUCTION

BY N. A. NIELSEN<sup>1</sup>

The present volume is the second ASTM Special Technical Publication presenting techniques developed for electron metallography—techniques which are advancing and broadening the usefulness and range of application of the electron microscope in metallurgical studies.

The techniques described in the earlier publication<sup>2</sup> relate almost entirely to surface replication and extraction procedures for studying metallurgical structure. These procedures still remain in standard use, although metal surface replication by films of evaporated carbon was developed in the intervening period and is now the preferred replication technique in many electron metallography laboratories. Examples of carbon replicas are shown in several papers in the present symposium volume, especially in the article by Bridges and Long who studied several metal and oxide surfaces by direct carbon replication, stripping the replicas with an evaporated wetting agent.

Examination of the contents of this symposium publication quickly reveals that the papers cover a broad range of electron metallography and are not concerned with electron microscopy alone. Four areas of investigation are included: (1) new improved techniques in specimen

preparation and replication; (2) studies of the microstructure of age-hardenable and heat-resistant alloys; (3) electron probe microanalysis; and (4) direct transmission electron microscopy of metals. Electron metallography no longer includes only the areas of electron microscopy and diffraction but encompasses broadly all techniques which employ electron beam radiation to provide information on the nature of metals. This symposium publication, therefore, will serve as a review of progress made in electron metallography over the five-year period.

While each paper in the symposium contains information on techniques, the First Progress Report of the Non-Ferrous Task Group of Subcommittee XI can be referred to for techniques successfully employed in studying a titanium – 8 per cent manganese alloy. This report demonstrates the application of electron microscopy to a non-ferrous material and indicates, as have past reports on the electron metallography of steel, that the superior resolution of the electron microscope, as compared with ordinary light microscopy, can provide new microstructural information.

An abstract of a paper by Long and Gray is included to indicate the usefulness of vibratory polishing of specimens for subsequent examination by electron microscopy.

A comprehensive discussion of etchants developed for the electron micro-

<sup>1</sup> Engineering Research Laboratory, Engineering Department, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.

<sup>2</sup> Symposium on Techniques for Electron Metallography, Am. Soc. Testing Mats. (1953). (Issued as separate publication *ASTM STP No. 155*.)

scope study of magnesium alloys is provided in the paper by Moe. Both silica and carbon replicas have been used successfully in examining etched magnesium surfaces.

An increasing amount of attention is being given to the electron metallography of commercial high-temperature alloys. Such alloys, because of their high alloy content and age-hardenability, possess complex microstructures containing both intermetallic compounds and nonmetallic precipitates of carbides and nitrides. The two papers by Bigelow and co-workers at the University of Michigan and the paper by Mihalisin and Carroll illustrate how electron microscopy and electron diffraction are being applied in the identification of these phases to provide information on the precipitation-hardening process and understanding of the role of alloy heat treatment. In these studies, palladium-shadowed collodion replicas and Parlodion replicas shadowed with germanium were used to identify and study the distribution of the minor phases present. (A task group of Subcommittee XI on Electron Microstructure of Metals, of ASTM Committee E-4 on Metallography, is concluding a study of high-temperature alloys; a report on this work will be published later.)

The new techniques being developed in electron probe microanalysis provide a new research tool complementary to electron microscopy. Now, in addition to observation of the microstructure of a metal by electron microscopy, it is possible also to determine the chemical composition of the microarea under study. Brooks and Birks, in their paper on "Electron Probe Analysis of Segregation in Inconel," demonstrate nicely how this

new technique complements standard electron microscopy to furnish a more complete metallographic interpretation of a high-temperature alloy. (Electron probe microanalysis has also become a special area of attention by a newly organized task group of Subcommittee XI. Progress reports on the status of this ASTM activity will be made in future publications.)

Still another new area in electron metallography has been opened for exploration with the development of procedures for examining metals by direct transmission microscopy. Boswell and Smith describe how these techniques can be used for several different metals. The procedure avoids the necessity of surface replication by using metal foils thin enough to serve as transmission specimens themselves. While it appears possible to study a wide variety of metallurgical phenomena by the use of thinned specimens, dislocation movement within the foils has received the most attention. The paper by Wilsdorf discusses dislocation phenomena observed in aluminum while subjected to tensile loading in a modified specimen holder for the electron microscope.

Direct transmission electron microscopy of metals and alloys has attracted the attention of metallographers in laboratories all over the world. Along with electron probe microanalysis, the main effort of Subcommittee XI is now directed toward development of this attractive field of metallography. Development of a technique for thinning various metals and high-temperature alloys is under way and progress will be reported at a future ASTM symposium.