14 Scanning Electron Microscopy: A Powerful Tool for Imaging Freeze-Dried Material

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ABSTRACT

Observation of freeze-dried material is a real challenge for both scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The samples are usually very brittle, electrically insulating, highly hygroscopic, and very difficult to embed in resins.

Sample preparation methods are described: cutting, sticking on the SEM stubs, conductive coating with carbon and/or metals like gold or platinum or embedding, and cutting with a microtome.

Examples of structural observations are given including digital image processing.

Over and above SEM observation is the possibility of qualitative elemental analysis by energy dispersive X-ray spectroscopy (EDX) of foreign bodies, which may contaminate the freeze-dried material.

INTRODUCTION

Visualization of microstructures is of the highest importance in material sciences. At present, the tendency for many scientists is to trust in spreadsheets and digital information, thus forgetting the necessary connection to the visible reality of nature. Numeric interpretation of images, so called image analysis is, of course, useful and yields highly important information of structures, but the basis of analysis is the morphological picture of the material, be it at visual size or at very high magnification. The choice of the observational instrument is made according to the desired magnification.

- Macrophotography, magnification 1× to 10×.
- Light optical microscopy, magnification 10× to 1000×.
- Scanning electron microscopy (SEM):
  - Conventional SEM with a tungsten thermoelectronic gun, magnification 1× to 50,000×.
  - Field-emission scanning electron microscopy (FE-SEM) with a field-emission "cold" electron gun, magnification 1× to 500,000×.
- Transmission electron microscopy (TEM), magnification 100× to 10^6×.

In this chapter, only the contribution of modern SEM to the global subject of this book will be exposed and discussed.

ANALYTICAL SCANNING ELECTRON MICROSCOPY

The SEM is an electron optical device designed to observe and analyse the surfaces of material samples of all nature. An electron beam, focused by electromagnetic lenses, scans the specimen's surface. The interaction of the "primary