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Introduction

"METALLOGRAPHY" or "MATERIALOGRAPHY"? IN MODERN TECHNOL-ogy and Materials Science we are examining the microstructure of *all* solid materials; therefore, materialography seems to be the correct word instead of the traditional metallography. In 1968, Crowther and Spanholtz[1] suggested this and it now seems appropriate to use the word "materialography" to cover the examination of the infinite number of existing and future materials. Also, the term "metallographer" should be changed to "materialographer." Changes of this kind, however, take time, and therefore the terms "metallography" and "metallographer" are used in this book, except in contexts where materials other than metals are discussed.

G. Petzow[2] defines Materialography (metallography) as "an investigative method of materials science. It encompasses the optical examination of microstructures, and its goal is a qualitative and quantitative description of the microstructure."

The term materialography includes ceramography (ceramics), metallography (metals), plastography (polymers), and mineralogy (minerals), in this way covering the microstructural examination of most materials.

Metallography/materialography includes a wide field in material investigation; it bridges the gap between science in new and existing materials and engineering using the materials in modern technology. Figure 1.1[3] shows how materialography covers the examination of parts from the centimetre and metre (in and ft) range to atomic dimensions in the nm and sub nm range.

The microstructure is characterized through size, shape, arrangement, amount, type, and orientation of the phases and the defects of these phases, as schematically

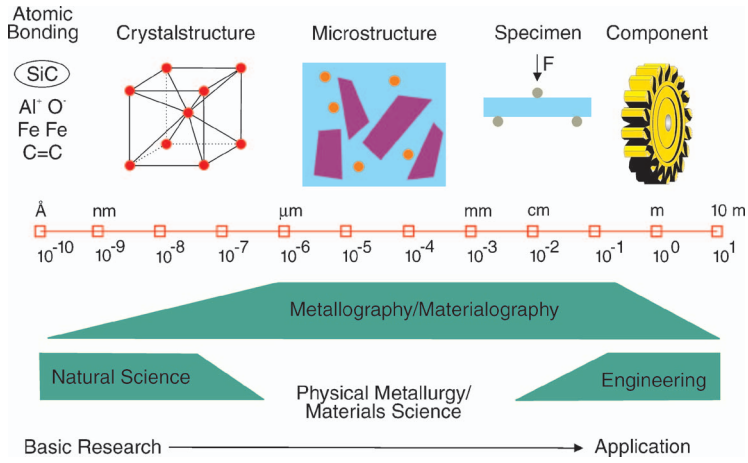


Fig. 1.1—Metallography/materialography can be described as a bridge between engineering and science, covering the examination of the part in cm and m to the examination of the single atom in Å.

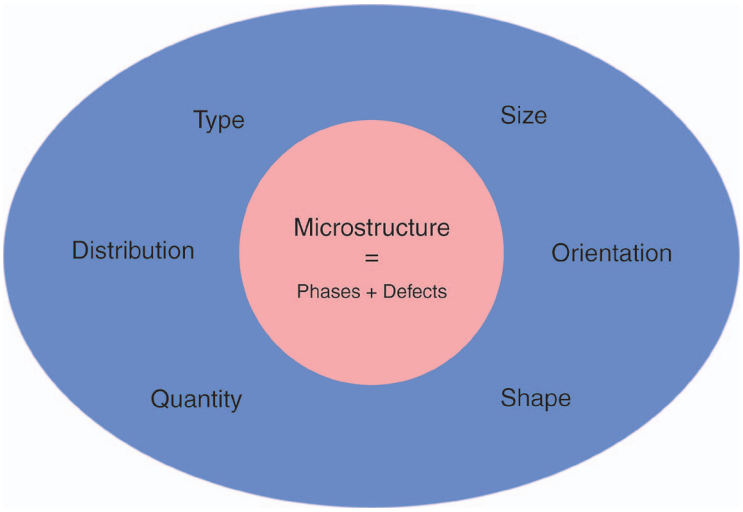


Fig. 1.2—The constituents of a microstructure and the factors affecting them.

shown in Fig. 1.2[3]. Each material contains many millions of microstructural features per cubic centimetre and these features strongly influence many of the properties of the material. As seen in Fig. 1.1, the microstructural features can exist in sizes of at least ten orders of magnitude. There are many instruments today that visualize nearly all of the features across this range.

The image we see in the typical microscope is two-dimensional, but we should not lose sight of the fact that the constituents in a material are three dimensionally arranged.

A photomontage shows the prepared surface of a silicon nitride alloy superimposed on a pile of silicon nitride crystals (see Fig. 1.3).[3] It shows that the true size of the crystals cannot be deduced directly from the microstructure. A statistical extrapolation of the two-dimensional surface shows that approximately 80 % of the crystals are relatively short and have an equiaxial shape. Stereological calculations, however, show a much higher variation in crystal length. The average crystal length is larger, corresponding to the three-dimensional characteristics shown in Fig. 1.3.

It can be concluded that the analysis of the microstructure plays an important role in modern materials science and engineering, and consequently, the metallographic/materialographic preparation. It is important to secure the true microstructure because without this the best examinations and inspired interpretations will be of no avail.

As stated in the Preface, this book concentrates on metallographic/materialographic preparation and the most commonly used examination methods. For a comprehensive, in-depth coverage of metallurgy and microstructures, including interpretation of the microstructures, ASM Handbook, Volume 9, Metallography and Microstructures,[4] is recommended.

This part of the book concentrates on the preparation of the specimen surface for examination in the reflected-light optical microscope. This preparation can also be used frequently for the scanning electron microscope (SEM). The mechanical removal

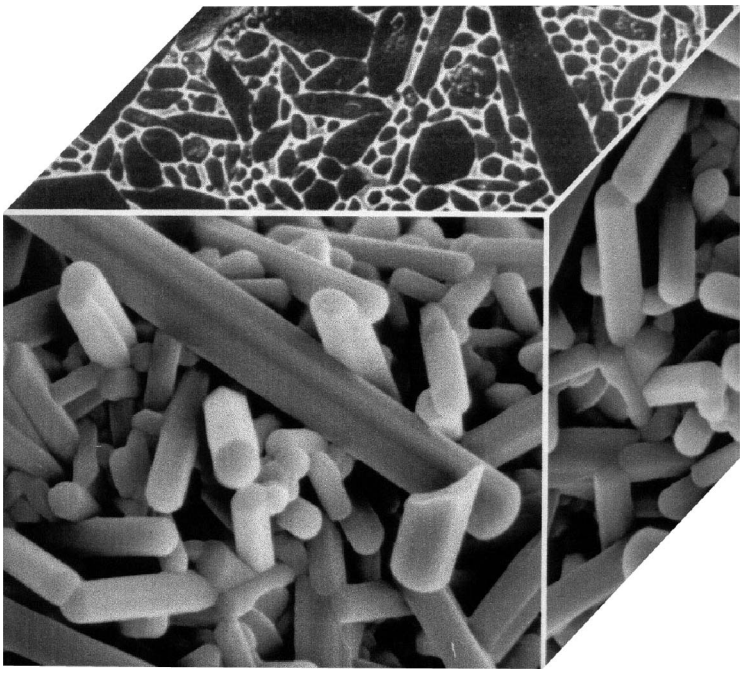


Fig. 1.3—Photomontage of a microsection of silicon nitride alloy superimposed upon a pile of silicon nitride crystallites.

of material will be described and discussed rather intensively because it is the central process in abrasive cutting, sawing, plane/fine grinding, and polishing, as will the problems involved in obtaining the true microstructure. The machines and consumables available will also be described and discussed.

Etching, often performed after the specimen preparation process to obtain a contrast to highlight or clearly reveal certain features, will be described in theory and practice.

1.1 Metallographic/Materialographic Preparation—The True Microstructure

The goal of the metallographic/materialographic preparation is to obtain the true microstructure or "The True Structure," meaning an undisturbed material surface, which can be analyzed in an optical (light) microscope or an SEM.

The basic problem for a metallographer preparing a specimen is that the preparation process itself modifies the specimen surface and, theoretically, a "true structure" completely without artifacts can never be obtained. Consequently, a preparation process should be used that creates the smallest amount of artifacts, making it possible, in practice, to analyze a microstructure in a satisfactory way.

1.1.1 Henry Clifton Sorby (1826–1908)

In the 1860s, because he understood that to obtain a "true structure" he had to remove the irregularities of the material surface, H. C. Sorby was able to produce what is con-

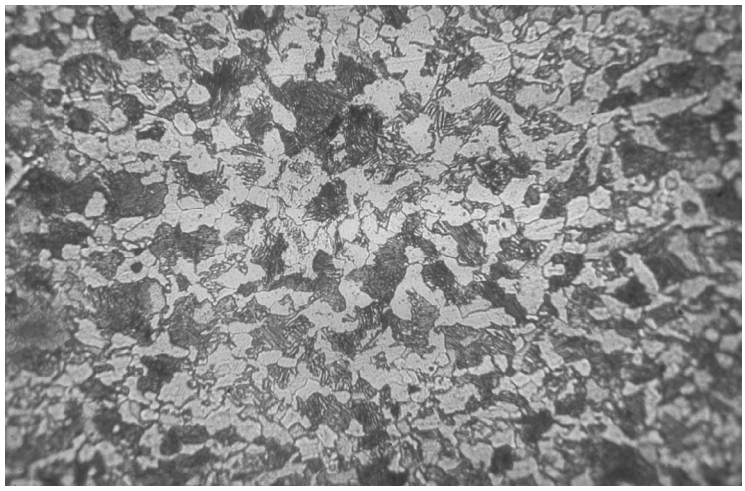


Fig. 1.4—Original specimen prepared by H. C. Sorby, 1863, Bessemer steel 0.2 % carbon. BF, 450:1. Preparation Method—Rough grinding: Emery paper from coarse to fine. Fine grinding: "Fine grained" water-of-Ayr stone. Rough polishing: "Finest grained" crocus (Fe_2O_3 used for industrial polishing). Polishing: "Very best and finest washed" rouge (Fe_2O_3, jeweler's rouge).

sidered the first true microstructure. In 1863 he prepared a specimen of Bessemer steel by using a preparation method with several steps, a method similar to the mechanical preparation used today. Figure 1.4[5] shows the microstructure, which was prepared in several steps, a rough polishing step and a fine polishing step.

1.2 The True Microstructure

Based on studies by Vilella and Samuels,[6–8] the true structure can be defined as:

No deformation—The plastically deformed layer created by the preparation should be removed or be negligible.

No scratches—Scratches normally indicate a surface that is not yet sufficiently prepared, but small scratches might be allowed if they do not disturb the examination.

No pull-outs—Especially in brittle materials, particles can be pulled out of the surface leaving cavities that can be taken for porosity.

No introduction of foreign elements—During the preparation process, abrasive grains can be embedded in the surface.

No smearing—With certain materials, the matrix or one of the phases might smear (flow), resulting in a false structure or covering of structure details, or both.

No relief or rounding of edges—Relief can develop between different constituents of the surface, caused by different hardness or other condition. Edge retention is important if the edge has to be examined.

1.3 Selection of Preparation Method

The preparation process will always influence the prepared surface, creating artifacts. Artifacts are defined as false structural details introduced during the preparation.

The choice of preparation is usually between using mechanical or electrolytic polishing, but chemical and chemical-mechanical polishing are also used.

1.3.1 Artifacts

A number of artifacts are already stated above under the true structure, but a few more can be added. Microcracks, comet tails, pitting, contamination, and lapping tracks are all caused by the preparation process. Artifacts can also be introduced during chemical etching of the surface. Most of these artifacts can be readily observed under the microscope. In some cases, artifacts can be accepted and the metallographer can decide whether, for example, a scratch is acceptable as it does not disturb the structural analysis, or whether the specimen surface should be reprepared.

In some cases it can be very difficult to establish the true structure, e.g., a smeared layer can cover pores. It is important that the metallographer pay attention to this possibility when analyzing a structure (see Section 13.5).

Artifacts of Mechanical Polishing

With mechanical polishing, it is possible to obtain an approximate true structure when the correct procedures are followed, even with very heterogeneous materials. Figure 1.5 shows the following most common artifacts: relief between phases caused by difference in hardness; embedded abrasive grain; inclusion protruding (it could also be missing); pull-out looking like a pore; rounding of the edge; and deformation of the matrix.

Artifacts of Electrolytic Polishing

With electrolytic polishing, the electrolysis might create problems if more than one phase is present in the structure. Figure 1.6 shows the most common artifacts. Relief between phases caused by a difference in electrochemical potential: in some cases one phase will be removed much faster than another phase, in other cases a phase might not be electrically conductive and, as such, will not take part in the polishing process. Inclusions might react in the same way; they will often be *dug out* during the process. Pitting might develop if the electrolytic process is not controlled correctly. Also, a pronounced rounding of the edge will take place because the current density is always stronger at the edge.

1.3.2 Preparation Methods

Because most materials are heterogeneous (or even nonconductive), the conclusion must be that mechanical polishing is by far the most commonly used method. For certain materials, however, electrolytic polishing gives very good results.

Alternatives to the above-mentioned methods are chemical polishing and chemical-mechanical polishing. Chemical polishing is not used much, although recipes for polishing of a number of materials are developed. Chemical mechanical polishing or attack polishing can be seen as an extension of mechanical polishing and, when relevant, recipes will be stated in connection with the specific material.

For recipes on chemical and chemical mechanical polishing, see Refs. 2, 4, and 9.

1.4 The Metallographic/Materialographic Specimen

In practice, the total work piece normally cannot be prepared and examined. For this reason, a small part of the work piece, the sample (specimen) must be extracted. For

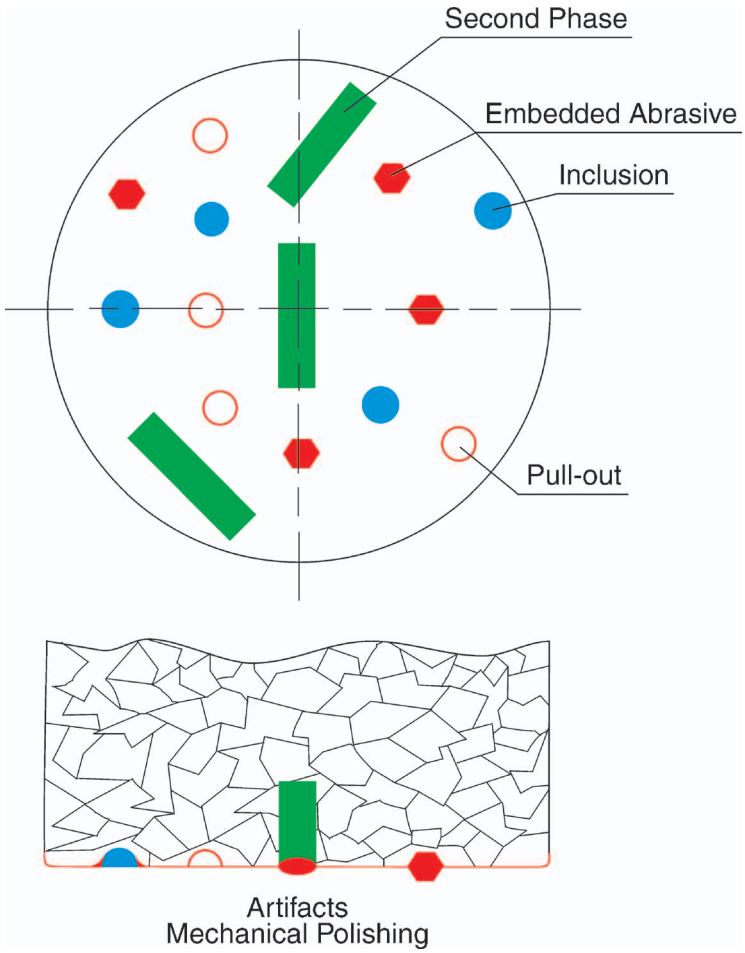


Fig. 1.5—Mechanical polishing: the most common artifacts shown schematically.

both specimen preparation and examination, using an optical microscope or an SEM, the ideal specimen size is 12–40 mm (0.5–1.5 in) square or cylindrical, with a height of 12–30 mm (0.5–1.2 in). There are, of course, exceptions like welds, where larger specimens have to be prepared.

With the specimen being only a small part of the material to be examined, if the interpretation is to be valuable, it is very important that the specimen be representative of the material to be studied. This usually happens by cutting out the specimen from a correct location and in the correct direction (see Section 2.1). Most ASTM standards covering examination of a metallographic/materialographic specimen offer guidance in selection and sectioning of specimens (see Section 12.4). The preparation can be performed once the specimen is established.

1.4.1 "Specimen" or "Sample"

The two words are often used indiscriminately, describing the object prepared and examined. The "sample" can be defined as the piece of material in its "raw" state, as taken

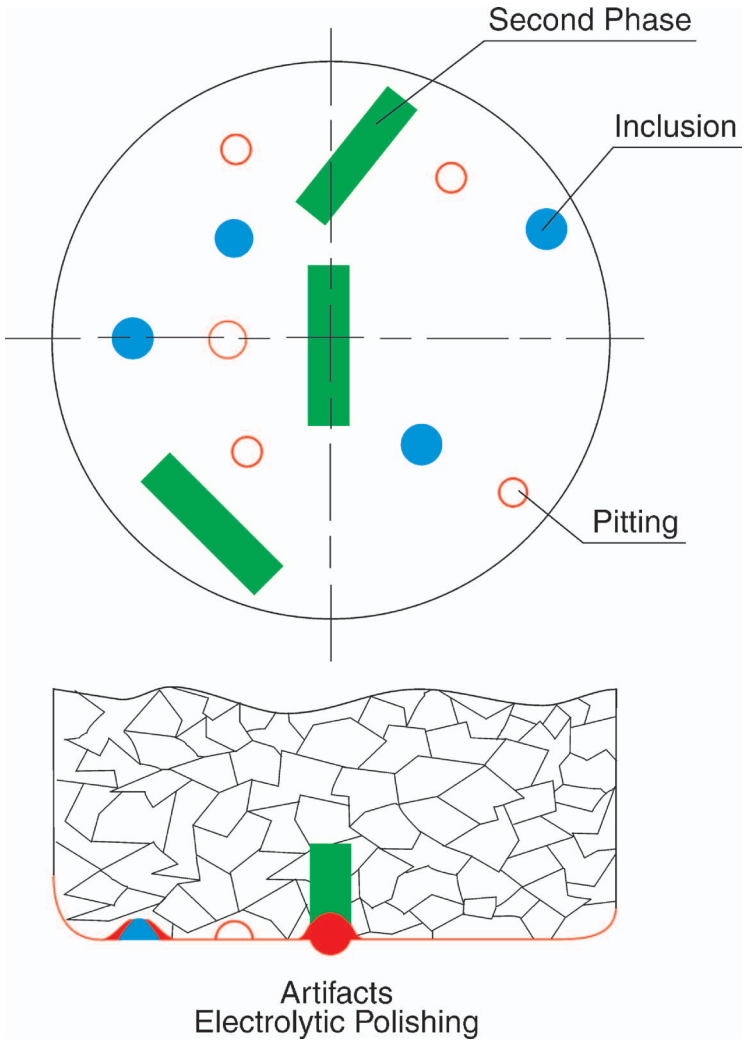


Fig. 1.6—Electrolytic polishing: the most common artifacts shown schematically.

from the original material (work piece). As soon as the "sample" is treated (prepared) and described, it turns into a "specimen," and for this reason only the word "specimen" is used in this book, except in a few cases where "sample" is the correct description.

1.5 The Preparation Process

As mentioned above, several polishing methods are available, but in the diagram, Fig. 1.7, only the two methods used for almost all preparation, mechanical and electrolytic, are shown. The diagram gives an overview of the total process, of which each step will be discussed further in this part of the book.

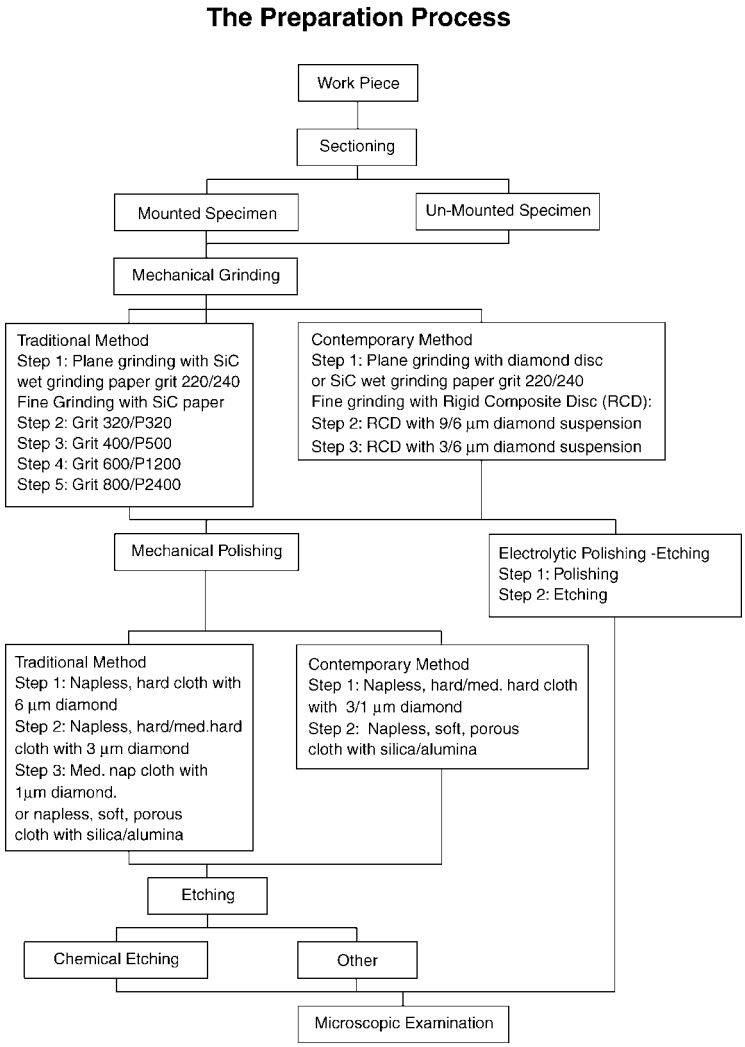


Fig. 1.7—Diagram showing the total preparation process based on mechanical and electrolytic preparation.

1.5.1 Sectioning

To obtain a specimen, some kind of sectioning from the basic material (work piece) is necessary. If this sectioning could take place without disturbing the specimen surface, the specimen could be examined without further work, but unfortunately all the known sectioning methods will leave some kind of irregularities on the surface. Abrasive wet cutting using a precision cut-off machine is considered as a sectioning method giving a low deformation of the specimen surface. Figure 1.8 shows a surface from a specimen cut on a precision cutter and measured with an atomic force microscope (AFM), and the irregularities of the surface are evident.

Abrasive wet cutting is the most frequently used sectioning method, but other

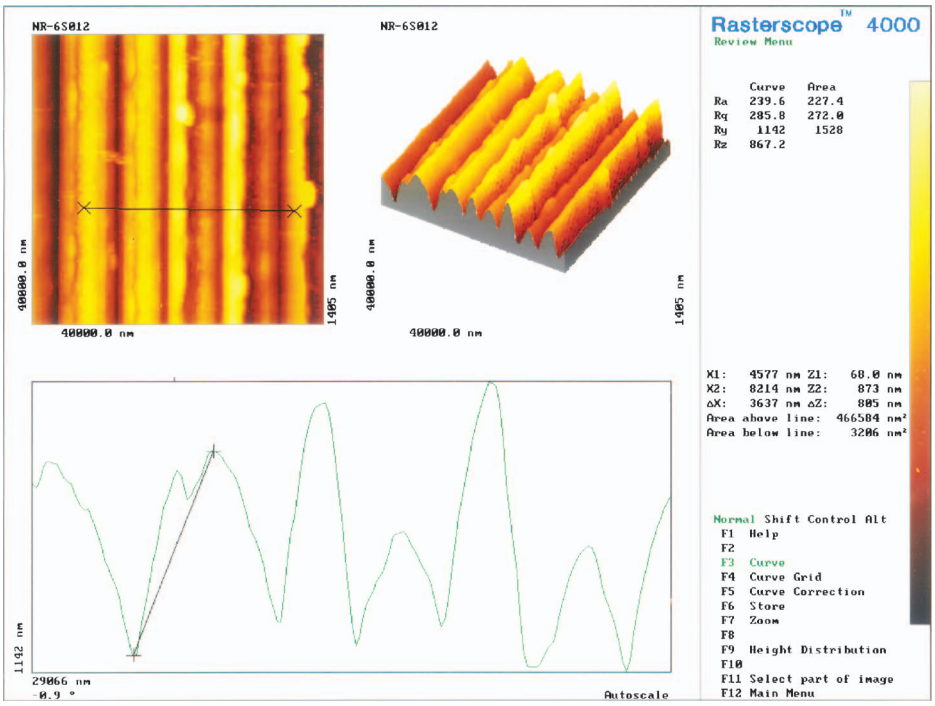


Fig. 1.8—Surface cut with a precision cut-off machine in a very careful way to avoid irregularities in the cut surface. Measurements with an atomic force microscope (AFM) give the peak-to-valley value of irregularities: higher than 1000 nm (1 μm). This shows that even with the most gentle sectioning technique, the cut surface will have deformations which have to be removed in the following preparation steps.

methods, such as shearing, sawing, and punching are used as well (see Section 2.7).

1.5.2 Mounting

In some cases, the sample taken from the base material can be handled and treated directly as a specimen, but often a mount must be made to secure the handling and a satisfactory preparation. The mounting can be made by clamping the specimen between two pieces of a material compatible to the specimen material. This way of mounting has a number of drawbacks (see Section 3.2.1); therefore mounting mainly takes place as hot compression or cold (castable) mounting in a mounting plastic (resin). Figure 1.9(a) shows three mounts made with hot mounting, giving mounts with very precise dimensions. Figure 1.9(b) shows three mounts made with cold mounting; these mounts, made in molds, are less exact than the hot mounts.

1.5.3 Preparation of the Surface

The goal of the preparation is to obtain the true microstructure or at least a microstructure in a condition that makes a satisfactory examination possible. This means that the number of irregularities (artifacts) in the surface must be kept at a minimum.

The preparation is done through a number of steps, either mechanical or electrolytical (see Fig. 1.7).

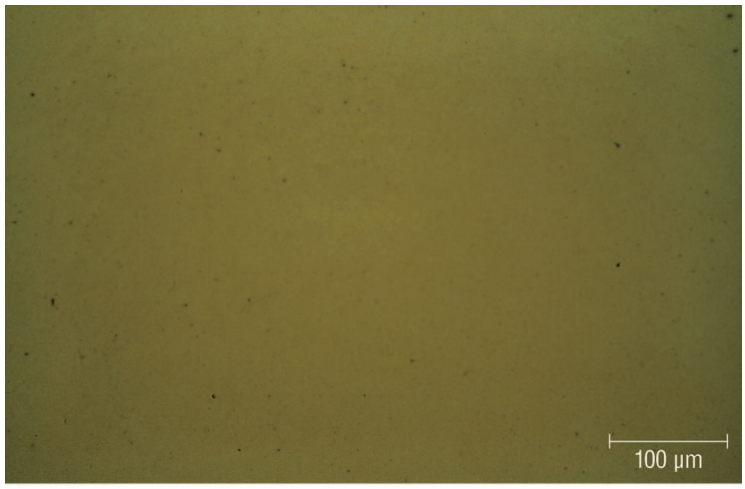
(a)

(b)

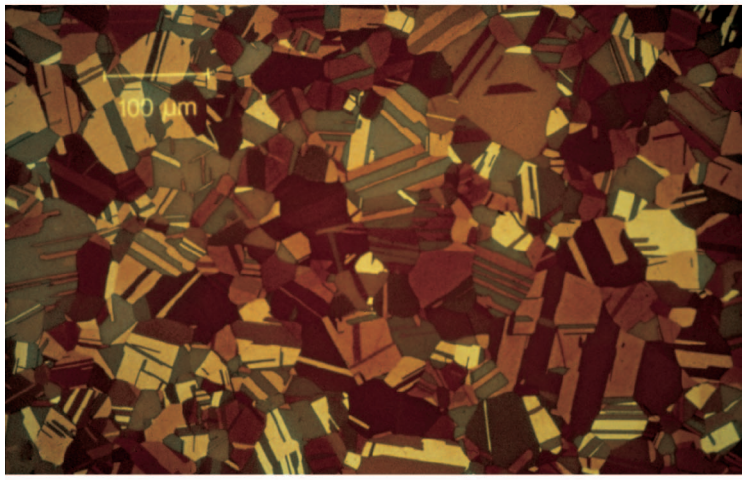
Fig. 1.9—Mounts made with hot compression mounting (a) and cold (castable) mounting (b).

A mechanical preparation method will normally contain a plane grinding step, one or more fine grinding steps, and one or more polishing steps.

Electrolytic polishing usually takes place as one electrolytic step, performed on a mechanically ground or polished surface.



(a)



(b)

Fig. 1.10—Copper unetched (a) showing a bright, reflecting surface and color etched with Klemm III[45] (b), revealing the microstructure.

1.5.4 Etching

The prepared surface often reacts as a mirror when examined in the microscope, not showing all phases of the microstructure. For this purpose, the surface can be etched chemically or electrolytically or treated in other ways to discriminate between phases, grains, grain boundaries, and other details. Figure 1.10 shows a copper specimen (a) in an unetched condition, giving very little information; and (b) one that is etched, showing the microstructure.