METALLOGRAPHY
PRINCIPLES AND PRACTICE

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Principles and Practice

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PREFACE

Metallography has proved to be an exceptionally useful metallurgical tool for both production and research work. Since the initial work of Sorby nearly 120 years ago, a multitude of techniques have been developed and applied to nearly every conceivable material. The vast scope of material available on this subject presents a formidable challenge to the student and to the practicing metallographer or metallurgist. This book brings together much of the existing knowledge pertaining to metallographic techniques and their application to the study of metals, ceramics, minerals, and polymers, although primary attention is given to metals.

This book concentrates on techniques relevant to visual and light microscopy—techniques fundamental to the study of macrostructure and microstructure. A similar treatment of techniques relevant to electron metallography is beyond the scope of this book, although some of the information presented is directly applicable. The historical development of metallographic techniques and the underlying scientific principles are discussed. Emphasis, however, has been placed on the practical problems associated with the use of these methods in order to facilitate their implementation. Metallography is both an art and a science, and both of these areas have been covered in detail. A complete list of recipes for polishing and etching solutions has been included plus comments regarding their safe and successful application. There are also extensive reference lists of key work at the end of each chapter to permit the reader to obtain additional information when needed. An extensive collection of macrographs and micrographs has also been included to illustrate the various methods discussed and to provide examples of their application to various materials.

This book should be useful to both undergraduate and graduate students in courses devoted to microscopy and physical metallurgy but should also prove useful to those studying ceramics, minerals, polymers, and carbonaceous materials. Engineers and technicians should find the book to be a valuable source of reference for use on the job. Although metallography is a relatively mature field, there has been substantial progress made in recent years in automation of sample preparation and in quantification of microstructural measurements, subjects that are thoroughly covered in this book.

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CONTENTS

CHAPTER

ONE

MACROSTRUCTURE

1-1 INTRODUCTION

Macroscopic examination techniques are frequently employed in routine quality control, in failure analysis, and in research studies. These techniques are generally a prelude to microscopic examination; however, in quality control, they are often used alone as a criterion for acceptance or rejection. A great variety of destructive and nondestructive procedures are available. The most basic procedure involves simple visual examination for surface features such as seams, laps, or scale.

This chapter describes only destructive test procedures; nondestructive methods are not covered. These destructive methods include the following procedures:

- Macroetching
- Contact printing
- Fracturing
- Lead exudation

Proper implementation of these methods is fundamental to the manufacture of materials. In quality control, the manufacturing routine is usually established according to set practices, and the macroscopic methods are used to detect deviations from the norm. In failure studies, one often does not know specific details of the manufacturing process and practices, and the engineer uses these tests to judge quality, to locate problem areas for further study, and, in some cases, to determine how the component was produced. In research studies, the processing steps are often varied, and the macroexamination is designed to show differences due to changes in manufacturing practices. Thus for each type of study, the specific details of the macroscopic examination will vary somewhat, and the practitioner must have a thorough understanding of the test method, its application, and the interpretation of test data.

Interpretation of the data from these tests requires an understanding of the manufacturing process, since the macrostructure is dependent on the solidification and hot- or cold-working procedures used. There can be pronounced differences in macrostructure because factors such as casting method, ingot size and shape, and chemical properties will significantly alter the solidification pattern. In addition, the use of manufacturing techniques other than traditional ingot casting, such as continuous casting, centrifugal casting, electroslag remelting, or hot-isostatic pressing, produce noticeably different as-cast patterns. Also, there is a wide variety of metalworking processes that can be applied to material made by any of the above processes, and each exerts a different effect upon the material. All these factors influence the interpretation of the test results.

No material can be said to be entirely homogeneous either macroscopically or microscopically. The degree of heterogeneity can vary widely depending on the nature of the material, the method of manufacture, and the cost required to produce the material. Fortunately, the usual degree of heterogeneity is not a serious problem in the use of commercial materials as long as these variances are held within certain prescribed limits. Certain problems, such as pipe and hydrogen flakes, are in general, quite harmful. The effect of other features, such as porosity, segregation, and inclusions, can be quite difficult to evaluate, and one must consider the extent of these features, the amount of subsequent metalworking, and the nature of the application of the material.

Of the metallographic procedures listed, the macroetch test is probably the most informative, and it is widely used for quality control, failure analysis, and research studies. Classification of the features observed with the macroetch test is often confusing because of the use of "jargon" created since the introduction of this test procedure. The macroetch test is covered in considerable detail in this chapter, and numerous examples of its application to a variety of materials are presented.

1-2 VISUALIZATION AND EVALUATION OF MACROSTRUCURE BY ETCHING

All quality evaluations should begin on the macroscale using tests designed to survey the overall field in a simple and reliable manner. After the macrostructure of a material has been evaluated, specific features can then be examined microscopically. Abnormalities observed on the etch disc can be studied by fracturing the disc or by preparing metallographic polished samples. Macroetching of transverse or longitudinally oriented samples, i.e., oriented with respect to the hot-working axis, enables the mill metallurgist to evaluate the quality of a relatively large area quickly and efficiently. Thus, macroetching is an extremely powerful tool and is a cornerstore of the overall quality program.

The earliest macroetchants were rather weak solutions used at room temperature. Reaumur (1683–1757) used macroetchants to distinguish between different types of steel and sketched the appearance of macroetched pieces of steel in his work. Rinmann promoted this technique in his book *On the Etching of Iron and Steel,* written in the late 1700s. Sorby, in his classic work published in 1887 "On the Microscopical Structure of Iron and Steel," showed "nature prints," which were inked contact prints of steel etched in moderately strong aqueous nitric acid solutions [1]. The early etching solutions have been reviewed in the classic text by Berglund [2].

1-2.1 Macroetching with Acid Solutions

The first "deep"-etching procedure for steel was developed by Waring and Hofamman using nine parts hydrochloric acid, three parts sulfuric acid, and one part water. Considerable adverse comment about the use of strong acids to evaluate highly stressed components was generated by this paper. Overall, the initial response to deep-acid etching was negative; however, numerous subsequent studies revealed the great value of such etchants.

After the initial work by Waring and Hofamman, considerable attention was devoted to the study of strong acids for deep etching steels. The most widely used deep etch consists of a 1:1 solution of reagent-grade† hydrochloric acid and water heated to 160 to 180°F for 15 to 45 min. Etching can be conducted on a saw-cut face, but better resolution is obtained with ground faces. Gill and Johnstin found that this etch was more selective in its attack than similar solutions involving nitric acid and water or sulfuric acid and water [3]. An important feature of this etchant is that evaporation does not significantly vary its composition during use.

The following items should be considered in the development of a macroetchant:

- The etchant should produce good all-around results, should be applicable to the majority of materials, and should reveal a great variety of structural characteristics and irregularities.
- The etchant should be simple in composition, inexpensive, and easy to prepare.
- The etchant should be stable during use or storage.
- The etchant must be safe to use and should not produce noxious odors.

The widespread popularity of the 1:1 hydrochloric acid and water etch is due to the fact that it satisfies these requirements better than other etchants. Appendix A lists macroetchants for iron and steel as well as for other metals.

The 1:1 hydrochloric acid and water etch attacks manganese sulfides readily but does not attack aluminum oxides. Steels high in aluminum content, such as the nitriding alloys, are etched best with an aqueous solution containing 10% hydro-

†The reagent grade contains 36.5 to 38% HCl, whereas the technical grade contains 28% HCl.

chloric acid and 2% nitric acid, developed by V. T. Malcolm. Etching is conducted at 180°F for 15 to 60 min.

As the alloy content increases, so does the degree of segregation and its associated problems. Etching is pronounced at the segregate-matrix interface, and segregate or matrix areas may etch out, leaving pits. Sulfides or carbides may also etch out, leaving pits. Before the investigator can distinguish between pits due to nonmetallic inclusions or segregates and carbides, the disc must be hardened and reetched. If the pits were due to nonmetallics, they will be present to the same degree in both the annealed and the hardened discs.

Watertown Arsenal [4] developed a variant of the standard etch that consists of 38 parts of hydrochloric acid, 12 parts sulfuric acid, and 50 parts water.† This reagent often produces a sharper definition of features than the standard etch, and like the standard etch, its acid concentration does not change markedly during use.

Macroetching provides an overall view of the degree of uniformity of metals and alloys by revealing:

- Structural detail resulting from solidification or working
- Chemical uniformity in qualitative terms
- Physical discontinuities due to solidification, working, etc.
- Weldment structure or heat-affected zones from burning operations
- Hardness patterns in non-through-hardened steels or patterns due to quenching irregularities
- Grinding damage
- Thermal effects due to service abuse

The first three features are best revealed by hot-acid etching, and the remaining four are best revealed by room temperature etchants. Macroetching is usually performed on ground surfaces, although in some cases, especially with cold etchants, better results are obtained when the surface is polished. Chemical segregation can be shown by certain cold etchants. The information obtained can be recorded by photographing the samples or, where possible, by contact printing.

In order to observe these features, one must sample the material properly and use the macroetch test procedure correctly. Fortunately, these test procedures are straightforward and simple to use as long as a few precautions are followed. In practice, one must consider the following test variables:

- Selection of representation samples
- Choice of surface orientation
- Proper preparation of sample surface
- Selection of the best etch composition
- Control of etchant temperature and etch time
- Documentation of test results

†Add the sulfuric acid *slowly* to the water and allow it to cool; then add the hydrochloric acid.