SYMPOSIUM ON METHODS OF METALLOGRAPHIC SPECIMEN PREPARATION

INTRODUCTION

By Mary R. Norton[1]

The phrase "metallographic specimen preparation" used in the present symposium refers to the surfacing, prior to etching, of metal specimens to be studied with the microscope.

Pioneer metallographers (1–8)[2] first described their methods of specimen preparation throughout the last half of the nineteenth century. In the decades which have followed a considerable literature on the subject has accumulated. With the exception of the completely different processes involved in chemical and electrolytic polishing, however, the basic mechanism of specimen preparation has undergone no change. It consists of removing successive layers of the specimen through the mechanical action of increasingly finer abrasives.

A surface suitable for microstructural studies must possess qualities that have been pointed out repeatedly in texts and technical publications for nearly a century. The following quotations are illustrative:

Sorby (1) (1868): ". . . (the final) polish must not be one which merely gives bright reflection but one which may show all the irregularities of the material and is as far removed as possible from a burnished surface."

Bayles (3) (1882): ". . . final polish must be delicate enough to leave the most minute particles of metal undisturbed and free from polished grooves or scratches."

Goerens (9) (1908): "Metallographic specimens should be flat and free from irregularities such as scratches."

Fay (10) (1917): ". . . (the) polish should give a perfectly smooth finish free of scratches."

Hoyt (11) (1920): ". . . (a microsection is) a plane surface polished free of scratches."

Epstein and Buckley (12) (1929): ". . . (in metallographic polishing) the essential thing is the quality of the finished surface which must be undisturbed, with all the constituents left in place and not covered or removed."

Chamot and Mason (13) (1931): "Polishing in the sense of producing a mirror surface is less desirable for microscopic preparation than is cutting by very fine abrasive particles which leave the surface covered with minute furrows but which do not obscure its inner structure by a film of flowed metal."

Vilella (14) (1937): ". . . the ideally prepared metallographic surface must possess at least the following qualities: . . .

It must be sufficiently flat from edge to center to permit examination at all magnifications, . . .

It must be free from scratches, stains . . .

It must contain all nonmetallic inclusions . . .

It must be free from all traces of disturbed metal."

Kehl (15) (1943): ". . . these (polishing) operations ultimately produce a flat, scratch-free mirrorlike surface."

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Thompson (16) (1953): "... a plane or flat surface entirely free from marks or irregularities with a high degree of polish must be obtained."

Samuels (17) (1956): "Polishing operations are defined as those which are intended to produce a specularly reflecting surface in which the scratches are so fine that they are not readily detectable by optical methods of microscopy."

Two requirements are evident among those just quoted and they are applicable whether the preparation procedure be mechanical or chemical, namely, (1) the surface should be smooth (polished), and (2) the surface structure must typify the unaltered structure of the bulk specimen.

Although the objectives of specimen preparation appear to be clear-cut, the choice of techniques and materials to achieve these objectives is indeed a most complex matter. The early workers employed a number of different abrasives, for example, emery, rouge, tin oxide, and alumina; many kinds of laps such as wood, cast iron, lead, copper, glass, wax, and pitch; and a variety of coverings for laps including wool, cotton, and silk fabrics, and leather. Each worker favored particular speeds, pressures on the specimen, and lubricants. Some, like Martens, Stead, Osmond, and Boyer, attributed their success to the careful preparation or grading of their abrasive papers and powders. Patience and cleanliness, too, were emphasized. Most workers recommended small specimen size. In the face of so many variables it is small wonder that good results depended largely on experience and judgment, and that metallographic specimen preparation came to be considered an art.

That the wide range of materials and techniques employed by the originators of metallography stemmed from existing arts is easily deduced. In 1905 Osmond and Cartaud (18) reminded us that: "Polishing is one of the earliest arts of humanity. Its introduction serves to differentiate the two phases of the Stone Age, the Paleolithic and the Neolithic. As polishing could scarcely have rendered the early stone implements more useful, we are forced to conclude that its object was to render them more beautiful. From the time of Homer to the present day, poets and prose writers have alike extolled the brilliancy of arms, which rivals that of jewels; and modern methods of polishing adapted to modern machinery are but the improved methods handed down to us by these medieval craftsmen, the armourers, goldsmiths, and jewellers, still imbued with some of that mystic empiricism which attaches to all those arts not based upon scientific considerations."

If the modern metallographer still doubts the source of some of his methods, he should be convinced by a volume entitled, "The Handbook for the Artisan, Mechanic, and Engineer," compiled by Oliver Byrne (19) in 1853. Mr. Byrne presents in great detail the materials and processes employed in the grinding and sharpening of cutting tools, the configuration of metal and glass by abrasion, lapidary work (including mineralogical specimens), the engraving of gems and glass, and so on. Here one finds specific reference to every abrasive and every lap material (either lap base or lap covering) which metallographers have employed well into the twentieth century.

PROGRESS IN METALLOGRAPHIC SPECIMEN PREPARATION DURING THE TWENTIETH CENTURY

Progress will be covered in part by the topics of the present symposium. Three general investigative categories have motivated advances in procedures:

1. The correlation of microstructure

with the mechanical and physical properties of metals in the range where microstructural details are of the order of the resolving power of the instruments employed. Surfaces to be used for such studies require the utmost in careful handling.

2. The production control of a single structural characteristic and hence the need of preparing and examining quantities of specimens in a short time.

3. The remote metallography of irradiated specimens.

Abrasives:

With the advent of electric furnaces, the modern metallographer was given the advantage of man-made abrasives—silicon carbide, boron carbide, and fused alumina.

The use of magnesium oxide as a desirable final abrasive was recommended by Desch and by Lucas (20), and more recently by Samuels (21). Lucas, whose original results with high-power microscopy demanded exceptionally thorough specimen preparation, claimed that magnesia gave a good polish and left finer scratches than he obtained with other abrasives.

Diamond, in the form of bort and mixed with olive oil, had long been employed in the gem cutting arts. A need to study the microstructure of the extremely hard cemented tungsten carbides prompted Schroeter (22) to apply the same mixture in his metallographic surfacing. He is credited with the first publication (1928) of results with cemented tungsten carbide structures. While working with similar specimens Hoyt (23), in 1930, achieved excellent final polishing results with a diamond dust prepared by personnel of the General Electric Co. and graded to a particle size of 0.5 μ and less.

In the early 1940's, work done at the National Bureau of Standards (24) on the grading of diamond powder led ultimately to the writing of a standard for the sizing and grading of diamond powders for commercial use. Subsequently, the metallographer was able to procure, at will, carefully sized diamond particles dispersed in a paste. There have been some published results (21,25–27). The subject of diamond as an abrasive in specimen preparation will be treated at length in this symposium (28).

Mechanical Polishing:

Except for improvements in abrasives and the availability of synthetic polishing cloths, the metallographer who employs manual procedures for applying the cutting action of abrasives to his specimen preparation has found his methods remaining unchanged. His work falls into the first category listed previously. His high resolving microscopes will soon tell him if his surfaces are poorly prepared. His research projects demand the best possible specimen preparation, and he remains, truly, an artisan. Provided the specimen material is amenable to this form of preparation, delicate hand work remains the sole choice of experienced research metallographers. The subject will be discussed in the current symposium (29).

Automatic Polishing:

Automatic polishing is a procedure in which the final stages of the specimen preparation are carried out by machine rather than manually. Several such machines were described in the 1930's (12,30–32). One of the latter (32) was inspired by Vanderwilt's (33) success in the polishing of ore specimens. Procedures for automatic polishing will be described in a later paper during this symposium (34). Such operations facilitate work in the second and third categories.

Vibratory Polishing:

A novel form of automatic polishing—vibratory polishing—is a product of the

1950's (35,36) which should find applications for work in the second and third categories. Complete coverage of the subject will be made in two papers of the current symposium (37,38).

Polishing for the Retention of Inclusions:

Preparation of specimens for the study of inclusions became a subject of great interest to metallographers during the 1920's and 1930's (39-44). The problem is particularly difficult because it demands fast answers for production control and at the same time requires well-prepared surfaces. Current interest in the possible effect inclusions have on fatigue characteristics of steel has shown the need to evaluate particles of smaller and smaller size. One paper in particular (45) may be expected to provide some highly valuable information on this subject.

Electrochemical and Chemical Polishing:

The smoothing of metal surfaces by electrochemical or chemical methods instead of by mechanical abrasion is the first real change in specimen preparation procedures. Electrochemical procedures have been described frequently since Jacquet first published his results on copper specimens in 1936 (46). Chemical procedures came into vogue in the 1950's. Both electrochemical and chemical methods originated as smoothing techniques for industrial applications. There is an abundance of literature on both, including a treatise by Tegart (47). Both methods are now solving the problems encountered with materials which cannot be surfaced satisfactorily by mechanical means without too much deformation of structure.

Electrolytic and chemical procedures are often used in conjunction with mechanical abrasion procedures. The metallographer should not expect removal of deformed structures by chemical polishing if his preliminary mechanical steps have introduced deformation of too great a depth; neither should he regard chemical procedures as universally applicable to all specimens.

A more recent use of electrolytic and chemical procedures has been made in obtaining thin foils for electron microscopy. Kelly and Nutting (48) have reviewed these techniques for electrolytic thinning.

The entire subject could well constitute the topic of a separate symposium. A few examples of the application of chemical polishing to nuclear reactor materials are cited in one of the contributions (49) to the present symposium.

Microtoming:

Although no discussion of the microtoming of metals is included in the current program, it might be well to note that metallographers have used a microtome for special cases and that there is some revival of the procedure. Lucas (50) sliced sections 2 μ thick from soft metals and, after etching the remaining bulk specimen surface, was able to reveal structures more clearly than had been the case after conventional specimen preparation. Phillips (51) sliced a variety of metals with a diamond knife and obtained specimens for electron microscopy investigations. One interesting result is that he appears to verify and extend the general principles of Ernst and Merchants relative to machining theories.

Effect of Mechanical Polishing on Structure:

Hook, Newton and, later, a few nineteenth-century physicists theorized about the scientific aspects of polishing hard materials, particularly glass. In 1921 Beilby propounded his theory that an amorphous layer is formed on the surface of a metallographically polished metal specimen. Since the 1930's when electron diffraction became available as a tool, physicists have investigated the Beilby layer, building up a considerable literature both pro and con. In many of these studies the method of producing a polished metal surface was not typical of metallographic procedure.

The metallographer should rightfully be concerned about the possible existence of a noncrystalline surface on his finished specimen. Fortunately the depth of layer which was the object of study is considered to have a magnitude consistent with the low penetrating power of the electrons used to reveal it. If a specimen has received careful surfacing, normal etching attack should remove the suspect material.

Not long ago Samuels (52), who has for some time expressed the belief that metallographic polishing occurs by cutting rather than by flowing, carried out an electron diffraction study of carefully prepared surfaces. He concluded that his results justify the claim for cutting action.

An important thought is voiced for the metallographer by Samuels. It is a fact which the conscientious worker probably suspects intuitively—if the deeply deformed zone caused in the earliest stage of specimen preparation is removed by continuing the cutting action of the next course for a sufficiently long time and if each successively finer step is similarly carried out, the residual deformation becomes less in depth the smaller the particle size of the cutting abrasive.

Thus far only the microscopist has concentrated on and worried about the condition of his specimen surface. It is becoming increasingly evident, however, that the X-ray metallographer and physicist using solid specimens, especially single crystals instead of the usual powder sample, need to be concerned with sample preparation. In some cases they desire to eliminate as much extinction as possible. The obtaining of a surface which will give such an effect is still very much the concern of the man who prepares specimens.

The above fact indicates that whereas we have come very far with regard to techniques we have not come proportionately far with regard to an intimate knowledge of the conditions existing in the surface of a metal on an atomic scale at various stages of polishing.

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