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A RAPID AND RELIABLE METALLOGRAPHIC
TECHNIQUE FOR SILVER

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Arthur E. Calabra

Abstract. A reliable and rapid metallographic preparation procedure for silver is described. The metallographic sample need only be ground through 600-grit (soft) silicon carbide paper, followed by an electrolytic lap-polish to remove the flowed metal. A chemical etch is then used to resolve the grain structure in fine detail. There are no artifacts or evidence of prior preparation steps, and the true grain structure is achieved in a minimum of time.

Emphasis is also made on illustrating the effects that improper metallographic preparation can have on the microstructure.

INTRODUCTION

High purity silver, fabricated by Rocky Flats or purchased from commercial manufacturers, must be examined for quality acceptance or rejection. Because of the numerous samples submitted, the urgency for results, and the large laboratory work load, a reliable and rapid evaluation must be provided by the Metallography Laboratory. Therefore, there existed a need for a simplified method of rapidly

determining the grain structure characteristics of silver. This report describes such a technique.

The general metallographic technique for the preparation of soft metals can be slow, laden with pitfalls, not always reliable, and requires considerable finesse by even the most experienced technician.^{1,2} The procedure described herein significantly reduces preparation time, and does not require a highly skilled technician to achieve a well-defined microstructure.

DISCUSSION

The electrolytic lap-polish system has not been exploited extensively; however, it does provide an effective method of preparing metallographic specimens. The apparatus used in this investigation is the Struers Relapol, which incorporates the lap-polish system described by G. Reinacher.^{3,4} The use of the Struers lap-polish unit for the metallographic preparation of silver is briefly described in Reference 5.

Figures 1 (a) and (b) are photomicrographs of the grain structure of silver prepared by the procedure described

Figures 1(a) and (b) Are Typical of the Silver Grain Structure Revealed by the Described Procedure. Note the fine structural detail showing no evidence of preparation artifacts.

(a) Silver 100X.

(b) Silver 50X.

herein. The specimen was lap-polished and chemically etched and shows excellent structural detail with no evidence of preparation artifacts. Preparation time, after specimen mounting, was approximately 15 minutes.

This report will also survey some of the causes of preparation defects, illustrate their appearance under the microscope, and recommend precautions which will avoid defects.

PROCEDURE

Mounting

The silver specimen may be cast into any available laboratory potting material for ease of handling. Large specimens need not be mounted. Electrical contact for the mounted sample is made by drilling a hole through the rear of the mount to the metal. A brass screw through the back of a Plexiglas specimen holder is screwed into the back of the mount to contact with the silver, Figures 2 (a) and (b). The brass screw head is attached to the anode lead from the power unit.

Grinding

The silver sample need only be ground through 600-grit (soft) silicon carbide paper. The grinding procedure is

Figures 2(a) and (b). Specimen Mount Holder. The Brass Screw Tapped through the Back of the Plexiglas Mount Holder is Tightened to Contact with the Silver Metal. The Anode (+) Lead from the Power Source is Attached to the Brass Screw.

(a) Plexiglas Mount Holder.

tabulated in Table 1. Grinding procedure precautions are also listed. These precautions should be a part of the technicians normal metallographic technique, and will generally apply to all materials. However, it is always good to review and remind.

Table 1. Mechanical Grinding Parameters.

Grinding Steps	Grit Size Sequence	Lubricant and Coolant
Coarse ^{a,b,c}	60 and 120	water
Intermediate	180, 240, 320, 400, and 600	water
Final ^d	600 grit (soft)	water

^a The coarse grit papers are only used if a great amount of material must be removed to reveal the plane of examination.

^b Grinding papers must always be fresh; never use a worn paper.

^c Clean the specimen thoroughly between steps. *Avoid the use of the ultrasonic cleaner.*

^d Buehler Microcut Paper; Adolph I. Buehler, Inc., Evanston, Illinois.

An important precaution is the use of fresh grinding papers at all steps. A worn or over-used paper will smear the surface and introduce an abnormally deep worked layer. Figure 3 shows the surface of a silver sample as it would appear after grinding on a fresh 600-grit silicon carbide grinding paper. The furrows cut by the carbide particles are uniform and well defined. Figure 4 is the same surface after prolonged grinding on a worn 600-grit paper. Note the general appearance of a "polished" surface. Rather

(b) Holder with anode lead attached and sample.

Figure 3. Silver 200X. Normal Appearance of the Silver Surface from a Fresh 600-Grit Silicon Carbide Grinding Paper. Note the well defined furrows made by the 600-grit Particles.

Figure 4. Silver 200X. Prolonged Grinding on a Worn 600-Grit Silicon Carbide Grinding Paper. The surface appears "polished." The depth of the worked surface is abnormal.

than metal removal by the carbide particles, the surface has been smeared by the bare paper resulting in the "polished" appearance and an abnormally deep worked layer. A surface such as this, which may occur during any step of grinding, is difficult to correct during the subsequent grinding steps. It is even more difficult to effectively remove this worked layer during mechanical polishing. Chemical etchants have a marked tendency to pit an abnormally smeared or worked surface. The structure revealed will also have distorted grain boundaries. Figures 5 and 6 are photomicrographs of silver purposely prepared by a poor technique to illustrate pitting and the distorted grain boundaries. Figure 6 also illustrates remnants of smeared metal not removed by the electrolytic lap-polish and chemical etch.

Figure 5. Silver 200X. Chemical Etching Pits on a Heavily Worked Surface as a Result of Improper Specimen Grinding.

Smearing of a metal surface can fold over furrows cut by the grit particles, leaving an underlying worked area. If subsequent grinding and polishing does not remove this worked condition, etching can reveal remnants of the disturbed metal as shown in Figure 7. These artifacts appear as an in-line series of parallel twins.

Figure 6. Silver 200X. A Photomicrograph of a Chemically Etched Surface which Had an Abnormally Deep Worked Surface. There are, as a result, uneven grain boundaries as shown by (a) as well as flowed metal remaining within the grains as shown by (b).

Figure 7. Silver 200X. Photomicrograph Showing the Effects of Improper Grinding. Furrows cut into the surface by coarse grit particles were folded over and hidden by the use of a worn paper. Subsequent electrolytic lap-polishing was terminated before complete removal of the worked metal to illustrate the resulting artifact. After chemical etching, the remnants of improper coarse grinding are revealed as a series of in-line parallel twins.

Another danger of prolonged "grinding" on worn papers, is the embedding of silicon carbide particles dislodged from the paper. These can be misinterpreted as an inclusion or porosity if it is dislodged. See Figure 8.

Figure 8. Silver 400X. Silicon Carbide Grit Particle Embedded into the Surface of the Metallographic Sample by Extended Use of Grinding Papers.

A distinct advantage of the electrolytic lap-polish procedure to be described is that sufficient material can be removed in a short time to eliminate artifacts brought about by improper or careless grinding.

Item C in Table 1 cautions against cleaning the silver sample in the ultrasonic cleaner. If necessary, it should be done for only a very short period of time — 30 seconds or less. This applies for all soft metals. Prolonged exposure to the cavitation will effect a physical change in the grain structure of soft metals such as silver. A mottled appearance on the surface of the metallographic specimen is an indication of excessive exposure to the cavitation action of the ultrasonic cleaner. Figures 9(a) and (b) are of an etched silver sample intentionally "over-cleaned" in the ultrasonic cleaner.

Experience at Rocky Flats has shown that silver items machined to a specific tolerance were out of specification, over-size, after cleaning in an ultrasonic cleaner. From this it was apparent that structural damage occurs within the bulk of the material. Elimination of this type of cleaning decreased the "out-of-specification" losses.

(a) 20X.

(b) 200X.

Figure 9. The Surface Topography Affected by Excessive Ultrasonic Cleaning. Note the disturbed grains due to the cavitation in the ultrasonic cleaner. This effect is shown on, what was, a satisfactorily chemically etched surface.

Electrolytic Lap-Polish Apparatus

The lap-polish equipment used is the Struers Relapol, which consists of a rotatable, cloth-covered, stainless steel disk in a PVC corrosion resistant bowl. A baffle plate in the bowl

returns the electrolyte slurry to the center of the bowl from the perimeter, where it will collect due to centrifugal force. Figure 10 is a sketch of the principle of the Reinacher polishing system. Figure 11 is that of the laboratory set-up.

Figure 10. Sketch of the Struers Relapol Lap-Polishing Unit. (The Reinacher polishing system.) See Figure 2 for detail of mount holder.

Figure 11. The Struers Relapol Lap-Polish Unit and Laboratory Power Source in Operation.

Electrolytic Lap-Polish Procedure

Table 2 shows the composition of the electrolyte slurry. Table 3 tabulates the parameters for the electrolytic lap-polishing. Sufficient quantity of the electrolyte slurry is poured into the PVC bowl to cover the cloth-covered stainless steel cathode [Table 3 – Item (a)]. The sample, attached to the Plexiglas holder and in contact with the

brass screw, is then connected to the power source anode, as shown in Figure 2 (b). It is held gently against the rotating cathode with the power on. See Figure 11. The actual voltage of 25 volts (Table 3) will result in a rapid and uniform removal of metal, producing a polished surface below the layer of worked metal. The suggested voltage is 25 volts, however, satisfactory results have been obtained with a voltage as low as 17 volts and as high as 35 volts. Polishing time is longer using the lower voltage.

If reasonable care has been taken during mechanical polishing, a surface ready for chemical etching can be obtained in approximately 3 minutes using 25 volts. If an excessively deep deformation is suspected, additional time can be used. If after chemical etching there is evidence of preparation defects, the sample may be lap-polished again without going back to mechanical grinding.

Lap-polishing forms a light tan-colored stain on the surface of the silver specimen as shown in Figure 12. This stain does not impede the electrolytic lap-polishing action and is quickly removed during chemical etching.

Figure 12. Silver 100X. Surface of the Polished Sample after Lap-Polishing on the Struers Relapol. A light-tan discoloration forms during polishing; however, it does not impede the electrolytic lap-polish and is immediately removed during chemical etching.

Table 2. Composition of Electrolyte.

Composition*
Ethylene Glycol – 195 ml
Nitric Acid (HNO_3) – 3 ml
Hydrochloric Acid (HCl) – 2 ml
Gamma Alumina, (Al_2O_3 – 0.06 μ) – 30 g

*This quantity is normally sufficient for the Struers Relapol electrolyte bowl.

Table 3. Parameters for Electrolytic Lap-Polishing.

Electrolyte	See Table 2
Anode (+)	Specimen
Cathode (–) ^a	Cloth covered stainless steel disc
Voltage ^b	25 volts – actual
Time ^c	3 minutes

^aStruers type RC-1 and RC-3 polishing cloth; Struers Scientific Instruments.

^bThis voltage is optimum; however, voltages as low as 17 volts and as high as 35 volts have been effective.

^cThe 3 minutes lap-polish time, at 25 volts, will effectively remove the 600-grit (soft) scratches. If the flowed surface is abnormally deep, giving a poorly-etched structure, additional time can be used.

Chemical Etch

Following the electrolytic lap-polish the silver metallographic specimen is chemically etched to reveal its grain structure. Table 4 shows the composition of the etchant that is used in the Rocky Flats Metallography Laboratory. As previously stated the discoloration is quickly and effectively removed by this etchant. If the metallographic sample has been prepared to below the worked metal surface, the grain structure will be quickly resolved to a fine detail as shown in Figures 1 (a) and (b).

Table 4. Silver Chemical Etchant.*

<u>Composition</u>	
Stock Solution:	
Potassium dichromate ($K_2Cr_2O_7$) – saturated solution	100 ml
Sodium chloride (NaCl) – saturated solution	2 ml
Sulfuric acid (H_2SO_4) – concentrated	10 ml
Working Solution:	
Dilute 1 part stock solution with 9 parts distilled water.	
Swab etch time – 5 to 10 seconds; or until the desired resolution is obtained.	

* "Metals Handbook – 1948 Edition," American Society for Metals.

CONCLUSIONS

The electrolytic lap-polishing procedure for silver metallographic samples has proven extremely effective and

efficient for the removal of flowed metal to a surface ready for chemical etching.

The true grain structure can be obtained in a short period of time making this procedure ideal for large volume turn out.

Sample preparation defects, as described in this paper, are quickly and easily corrected. The system is ideally suited for the most inexperienced laboratory technician.

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