ORNL-2494
Metallurgy and Ceramics

PREPARATION OF METALLOGRAPHIC SPECIMENS THROUGH VIBRATORY POLISHING

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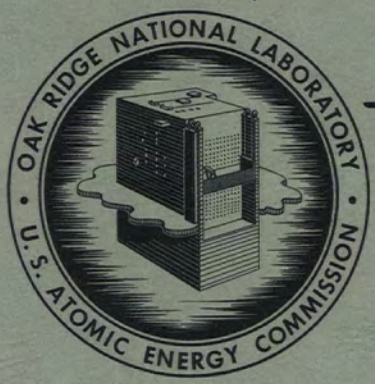

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Metallurgy and Ceramics

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PREPARATION OF METALLOGRAPHIC SPECIMENS THROUGH VIBRATORY POLISHING[1]

E. L. Long, Jr., and R. J. Gray

ABSTRACT

In principle, the art of preparing metallographic specimens with abrasives has not altered for more than 50 years. In 1956, vibratory polishing, which was then in its infancy, was introduced to the field of metallography. Since that time much effort has been expended by the Metallography Department of the Oak Ridge National Laboratory toward the development of this new mode of polishing. Vibratory polishing has proved its worth in the Metallography Laboratory and has many inherent advantages that could be a real asset to remote metallographic facilities.

INTRODUCTION

Vibratory polishing was first introduced[2] to the field of metallography in July 1956 and, from the principle of operation, seemed to have many inherent advantages that outweighed the disadvantages. Since the Metallography Department of the Oak Ridge National Laboratory's Metallurgy Division handles thousands of specimens annually, the Department is in constant search for better, more efficient, and more economical methods of processing metallographic specimens. At the time that information was received on vibratory polishing, the only work that had been done was on a small scale and had been confined to the final polishing of aluminum alloys. There was no reason to assume that this new method of polishing could not be extended to other nonferrous alloys as well as ferrous alloys with the proper abrasive and cloth combinations.

RECENT DEVELOPMENTS

In November 1956 a small vibratory lap-polishing machine[3] was purchased for an investigation of the potentialities of this method in preparing metallographic specimens in a conventional laboratory as well as in remote metallographic facilities. The small 5-in.-dia polisher is shown in Fig. 1. Over a period of two months various abrasives, cloths, speeds, weights, and samples were tried. The results were very encouraging; however, two difficulties had to be eliminated in order to make this mode of polishing economical: the polishing speed was too slow, and the capacity of the polishing bowl was too small. Therefore three larger vibratory-polishing machines[4] equipped with 12-in.-dia bowls were ordered and were received in March 1957. As received from the supplier, the bowls were secured to the polishing unit. Adaptor plates for quick-change bowls were fabricated to facilitate washing out spent abrasive, collected contaminants, etc.; this also allowed a supply of bowls containing various abrasives to be kept on hand. Figure 2 shows the larger vibratory polishers with the adaptors for quick-change bowls. The larger polishing machines increased the capacity of the bowl to 24 specimens compared with a maximum capacity of four specimens in the 5-in.-dia bowl. The larger vibratory polishers have the capacity of increasing the polishing speed to approximately six times that of the maximum of the small vibratory-polishing machine.

EQUIPMENT DESCRIPTION

The schematic sketch shown in Fig. 3 gives the various parts of the vibratory polisher, as well as a simplified vectorial diagram. In

[1] Paper presented at the 12th AEC Metallography Committee Meeting, Mallinckrodt Chemical Works, Weldon Springs, Mo., Feb. 27–28, 1958.

[2] F. M. Krill, "Vibratory Polishing of Metallographic Specimens," *Metal Progress* 70(1), 81–82 (1956).

[3] Purchased from the Syntron Company, Homer City, Pennsylvania, Model LP.00.

[4] *Ibid.*, Model LP.01.

Fig. 1. The Small 5-in. Vibratory Polisher Used in the Initial Investigation.

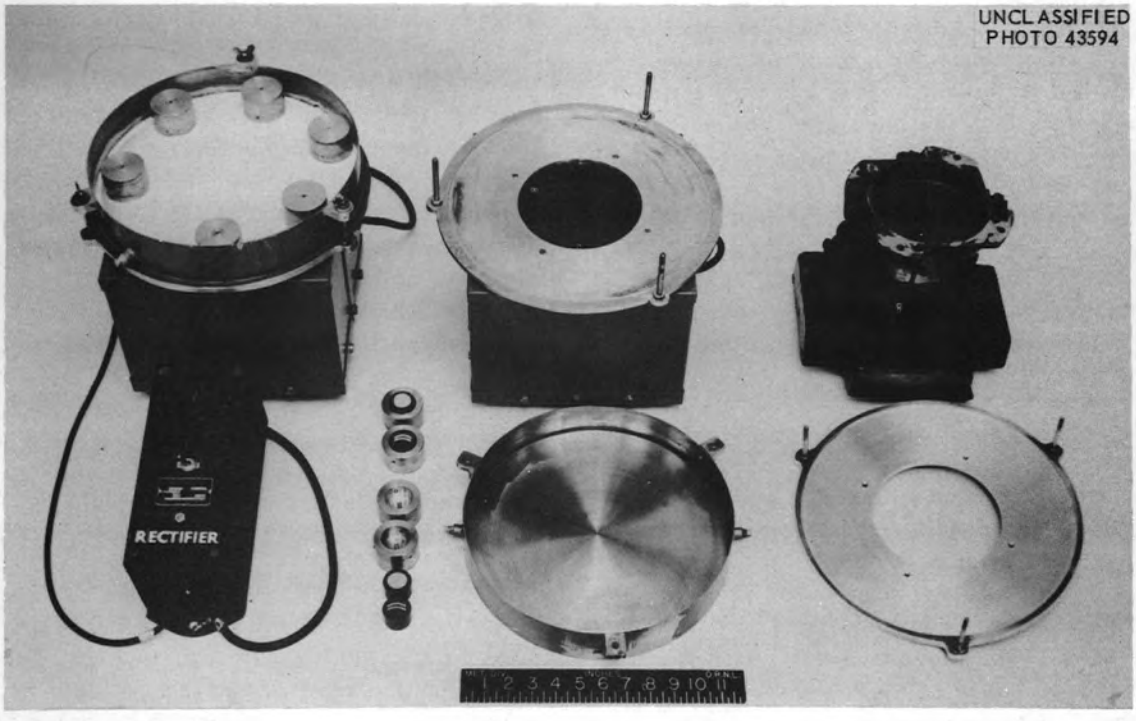

Fig. 2. Vibratory Polishers with the Larger 12-in.-dia Bowls Presently Employed.

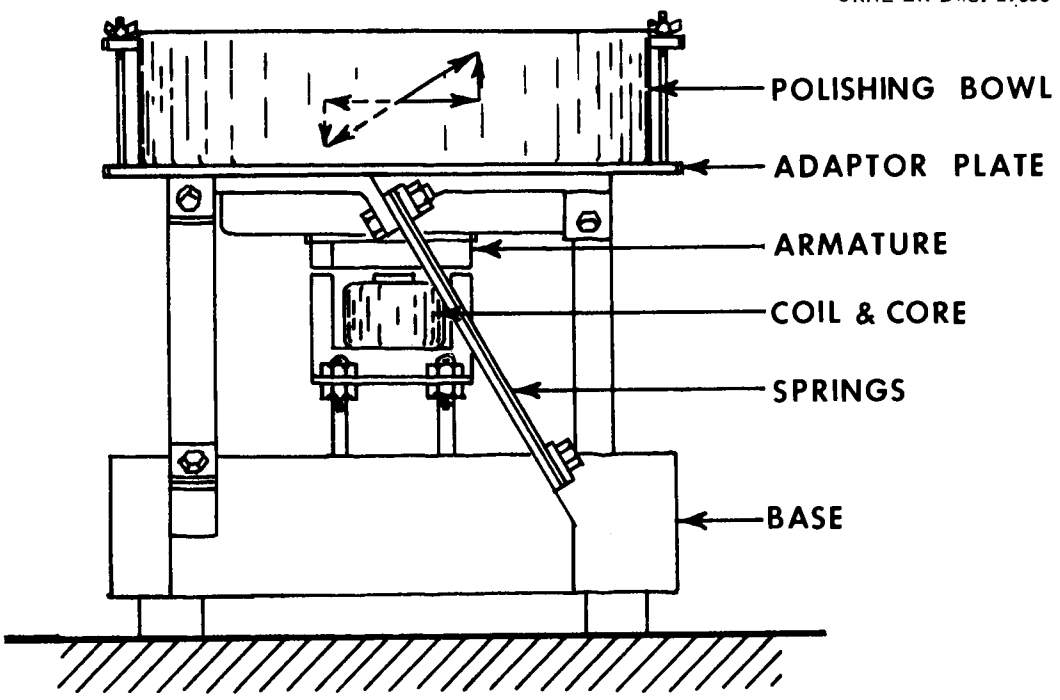

Fig. 3. Schematic Sketch Showing the Various Parts of the Vibratory Polisher Along with a Simplified Vectorial Diagram.

essence, the vibratory polisher consists of a base-power unit, which houses the spring and magnet assembly, and a flat-bottom bowl containing the selected polishing cloth or abrasive paper. Attached to the armature of the electromagnet is the bowl-mounting plate. Directly under and facing the armature is the coil and core assembly, which is anchored to a large, heavy-base casting. The bowl-mounting plate in the 12-in. model is supported by four-leaf springs spaced 90 deg apart and at an angle of 65 deg to the base casting.

The principle of polishing can be illustrated by the example of a single specimen resting on the polishing cloth. When the coil is energized, the armature, bowl-mounting plate, and bowl are sharply pulled down and away from the specimen. Because of the angular leaf-spring arrangement a slight radial twist is imparted on the bowl with the downward movement, thus distorting the springs. The inertia of the specimen keeps it from moving with the bowl, and the specimen then drops vertically to a new spot on the polishing cloth slightly forward of the initial position. When the coil is de-energized, the springs return the bowl to its original position, thus advancing the specimen. However, in order for any polishing action to be obtained, the specimen should be heavy enough for its inertia to cause slippage between the specimen and the cloth during the spring-back cycle of the bowl when the coil is de-energized. An optimum weight must be used so that a maximum amount of polishing occurs with a minimum amount of directional polishing. Directional polishing is prevented by allowing the specimen to travel enough to permit rotation during polishing. The key to vibratory polishers lies in a system properly designed to assure a tuned-spring system. Specifically, the tuned system should be three to five cycles higher than the frequency of the supply current. For example, a 60-cycle polisher should have a period of 63 to 65 cycles. The selection of such a range eliminates the possibility of harmonic motion and ensures stability of the polisher.

The vibratory polishers are powered by an alternating current. The current is initially passed through a half-wave selenium rectifier, which is housed in the base-unit covers, or in a separate control box with a rheostat and an operating switch. The function of the rectifier is to cut out half the sine wave; this results in the current flowing in

one direction only. Thus, the coil of the electromagnet is energized once per cycle, which develops a vibration frequency of 3600 vibrations per minute on 60-cycle current. The movement of the specimen is approximately 0.030 in. per cycle, but at this high frequency the displacement cannot be seen. Instead, the specimens appear to be flowing smoothly around the periphery of the polishing bowl. While the frequency of the unit is constant, being dependent upon the frequency of the input current, the amplitude of the vibration can be varied by control of the power input, thereby varying the polishing speed.

GENERAL PROCEDURE

Specific instructions for successful vibratory polishing cannot be given; each metallographic laboratory will generally perfect its own techniques for the various types of samples and the accompanying problems. However, the procedures listed below have worked on samples ranging in hardness from gold to B_4C and in size from wires 0.019 in. in diameter to specimens 2 in. in diameter.

Two types of cloths are employed primarily: a good grade of silk cloth and a stiff deep-nap cloth such as Micracloth. The nap cloth is secured to the bowls with a rubber-base adhesive. The life of the cloths is dependent upon the usage, but they can be expected to last at least one week with severe usage. The silk cloth obviously cannot be glued in place, but it can be secured in the polishing bowl by means of an expanding spring-steel band, as shown in Fig. 4. The silk will last for only one day.

Fig. 4. Method Used for Securing Silk Cloth in Polishing Bowl with Expanding Spring-Steel Band.

The majority of the specimens processed at this laboratory are mounted in the $1\frac{1}{4}$-in.-dia mounts. It has proved uneconomical to lap the specimens on the vibratory polishers because of the time involved and the number of polishers that would be required to include this operation. Therefore the specimens are carried through the 600-grit or 3/0 abrasive papers by hand, depending upon the metal or alloy. Some specimens are still carried through the lead laps which are charged with 5-μ emery. However, usage of the lead has been reduced considerably since the introduction of the vibratory polishers to the laboratory. As was pointed out in the discussion on the principle of vibratory polishing, an additional weight must be placed on the specimen for polishing. From experience the optimum weight seems to be approximately 350 g. The weights were machined from 2-in. stainless steel bar stock to the shape of a cap. The specimen is held in place with three Allen screws around the sides of the cap; the height of the specimen is adjusted by means of a screw in the top of the cap. The specimens are then ready for placing in the polishers.

The arrangement of the three polishers is shown in Fig. 5. The Lucite cover serves as a dust protector, and is hinged to permit access. From right to left, the first bowl contains the silk with a Linde A abrasive slurry, the middle bowl contains the nap cloth with a Linde A abrasive

Fig. 5. The Arrangement of the Three Large Vibratory-Polishing Machines. Note that controls for the two polishers on the right are located outside and above the Lucite dust protector. The polisher on the left is the deep nap, Linde B bowl, the middle polisher is equipped with deep nap, Linde A, and the polisher on the right is equipped with silk, Linde A bowl.

slurry, and the third bowl contains a nap cloth also, but with a Linde B slurry. The specimens are shown in position for polishing. Two control boxes can be seen located above the polishers. The controls for the polisher on the left are located on the base unit covers. Although the latter polisher is a more compact unit, the other two show how the polishers could be operated in a remote facility.

The rheostat setting is rather critical; if set too low, slow polishing along with directional polishing will result. However, if the speed is too great, the specimens will bounce and little or no polishing will result. There are two variables in vibratory polishing which allow control of the polishing speed: the weight placed on the specimen and the amplitude of vibration which is controlled with the rheostat.

It has been found that greater ease of control is established when a constant weight is used on the specimen, which leaves only the amplitude as a variable. At the start of the polishing operation it is better to begin with the rheostat setting too high and gradually reduce the speed of the polishers. When the correct polishing speed has been attained, the specimens will not bounce but will flow smoothly around the periphery of the bowl counterclockwise while turning slowly with a clockwise motion. Typical polishing times for the average specimen would be as follows: 15–30 min in the silk bowl, 15–20 min in the deep-nap cloth with the Linde A slurry, 20–25 min in the deep-nap cloth with Linde B slurry.

The 12-in.-dia bowl will hold up to 24 specimens at one time. Thus, theoretically, if 24 specimens were carried through the entire series of polishers, the total polishing time would average slightly over 3 min per specimen.

RESULTS ON SPECIFIC APPLICATIONS

Resultant finishes on specimens, typical of those submitted to this department, are as follows. The microstructure of one specimen is shown in Fig. 6. The base metal is an Ni-Mo alloy,

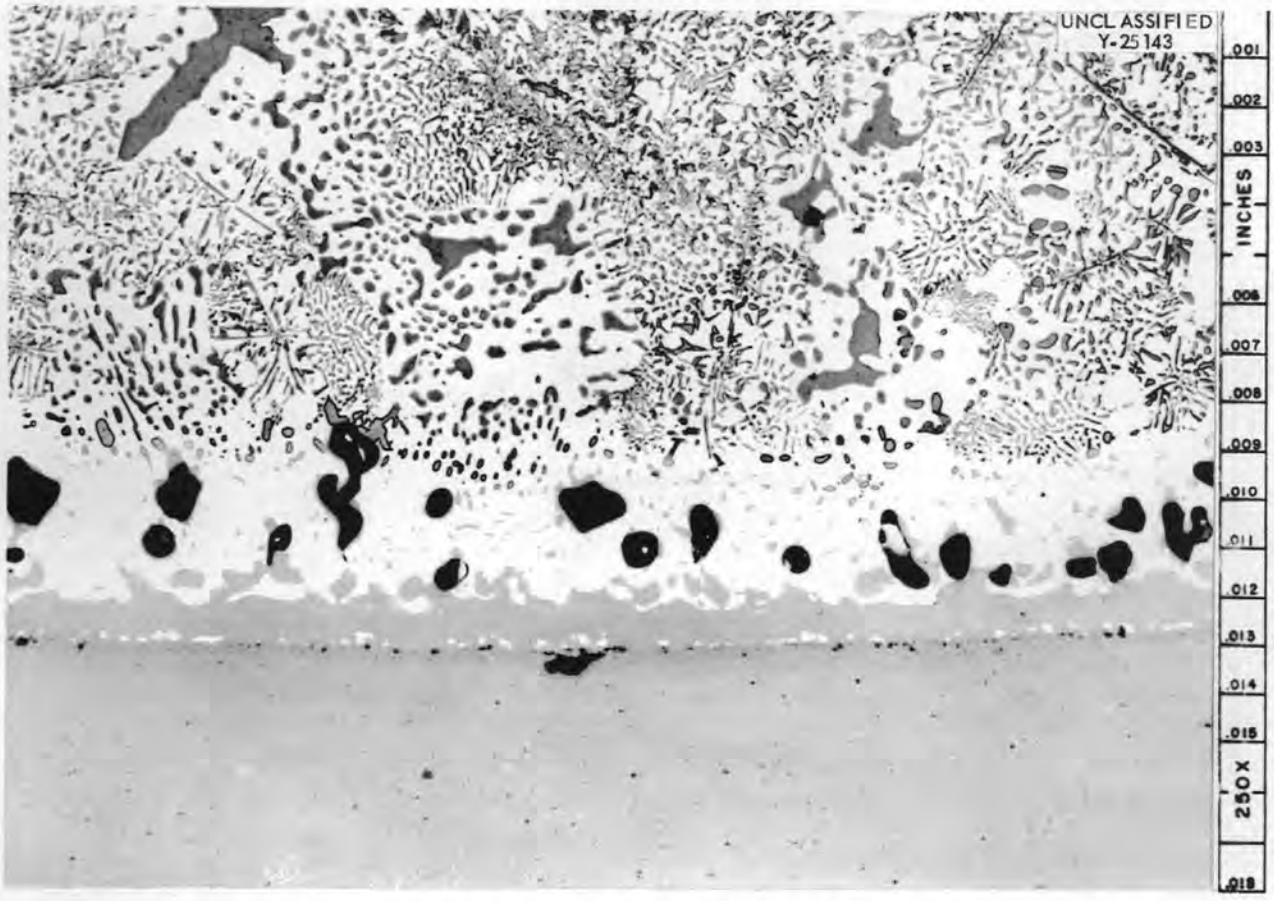

Fig. 6. Ni-Mo Alloy Which Has Been Nickel-Plated, Silver-Plated, and Brazed with the Cu-Ag Eutectic. Note that no relief polishing has occurred and that only slight rounding of the void edges is visible. Etchant: 1 part 3% H_2O_2, 1 part H_2O, 1 part NH_4OH. 250X.

which was electroplated with nickel and then electroplated with silver. The specimen was then brazed with a commercial Ag-Cu alloy of the eutectic composition. The investigator was interested mainly in the reaction at the silver-plate-braze interface. The specimen has been polished to a scratch-free condition; yet there is no detectable relief-polishing between any of the constituents, even though there are considerable differences in hardness; nor is there any excessive rounding of the void edges.

In order to acid-test vibratory polishing for directional polishing and/or smearing, a plain carbon steel sample was selected. This particular specimen had undergone an isothermal transformation from the austenite region to 591°C for 1.6 sec, and was then quenched. This treatment resulted in small pearlite colonies of extremely fine lamellar spacings. Because the pearlite was much too fine to resolve under the light microscope, an examination was made on the electron microscope. A palladium-preshadowed carbon replica was made of the fine pearlite. It was then examined for defects in the structure which could have occurred during polishing. A typical area is shown in Fig. 7; no polishing defects were observed except for an occasional small scratch.

With materials that tend to corrode after relatively long exposures to water, an oil or some other medium can be substituted for the water in the polishing bowls. For example, uranium can be successfully polished with an alcohol–oxalic acid solution as the vehicle.

Experimental fabrication of fuel plates and control rods results in a type of sample which is very time-consuming to prepare for examination. The object of the examination is to determine whether any reaction occurred at the surface of the clad particles, to permit a particle size determination, and to provide a check on the distribution of the particles. Such a sample is shown in Fig. 8. The matrix is aluminum and the inclusions are U_3O_8 particles. A sharp interface between the matrix and the particles has been retained. There are but a few scattered points where the U_3O_8 has fallen out during polishing. Another example of this nature is shown in Fig. 9. The inclusions are rare-earth oxides; the matrix is a stainless steel. Again good retention of the particles and sharp interfaces were obtained.

Extreme care in polishing was required on another occasion. Electron microscopy was essential to determine whether any reaction had occurred at the interface of the europium oxide particle, surrounded by a stainless steel. The sample, shown in Fig. 10, was in an as-polished condition. The reaction layer is clearly seen completely surrounding the europium oxide particle. Note the absence of smearing or scratches. After further investigation, it was shown that the silicon in the stainless steel was responsible for the reaction layer.

Vibratory polishing is paying off handsomely in another instance. Periodically several dozen platinum-gage strips are submitted for polishing. A high polish is not required, since determination of microstructure is not the object. However, the specimens must be apparently scratch-free at a magnification of 20X. One technician working for 8 hr can prepare approximately 12 strips. When the vibratory polishers became available, 12 strips were cemented to a weight plate, placed in a bowl containing a Linde A abrasive slurry, and polished for 1 hr. This setup is shown in Fig. 11. The results were quite satisfactory, as shown in Fig. 12. Note the flat surface of the finished strip as compared with the unfinished strip. As a consequence, the requester was charged for only approximately 20 min of the technician's time rather than for the usual 8 hr.

One of the more difficult metals to polish to a scratch-free condition is niobium. An example of a vibratory-polished niobium specimen is shown in Fig. 13. Although this sample was a little over 0.04 in. across, its unprotected edges were only slightly rounded as a result of vibratory polishing. Had it been hand-polished to a scratch-free condition, the unprotected edges would have been excessively rounded.

CONCLUSIONS AND RECOMMENDATIONS

As a result of a 14-month test period, during which time samples of practically every description were tried on the vibratory-polishing machines, many advantages have become quite apparent when compared with conventional hand-polishing techniques and many of the automatic polishing machines. No attempt is made to list all the advantages, but only the major points.

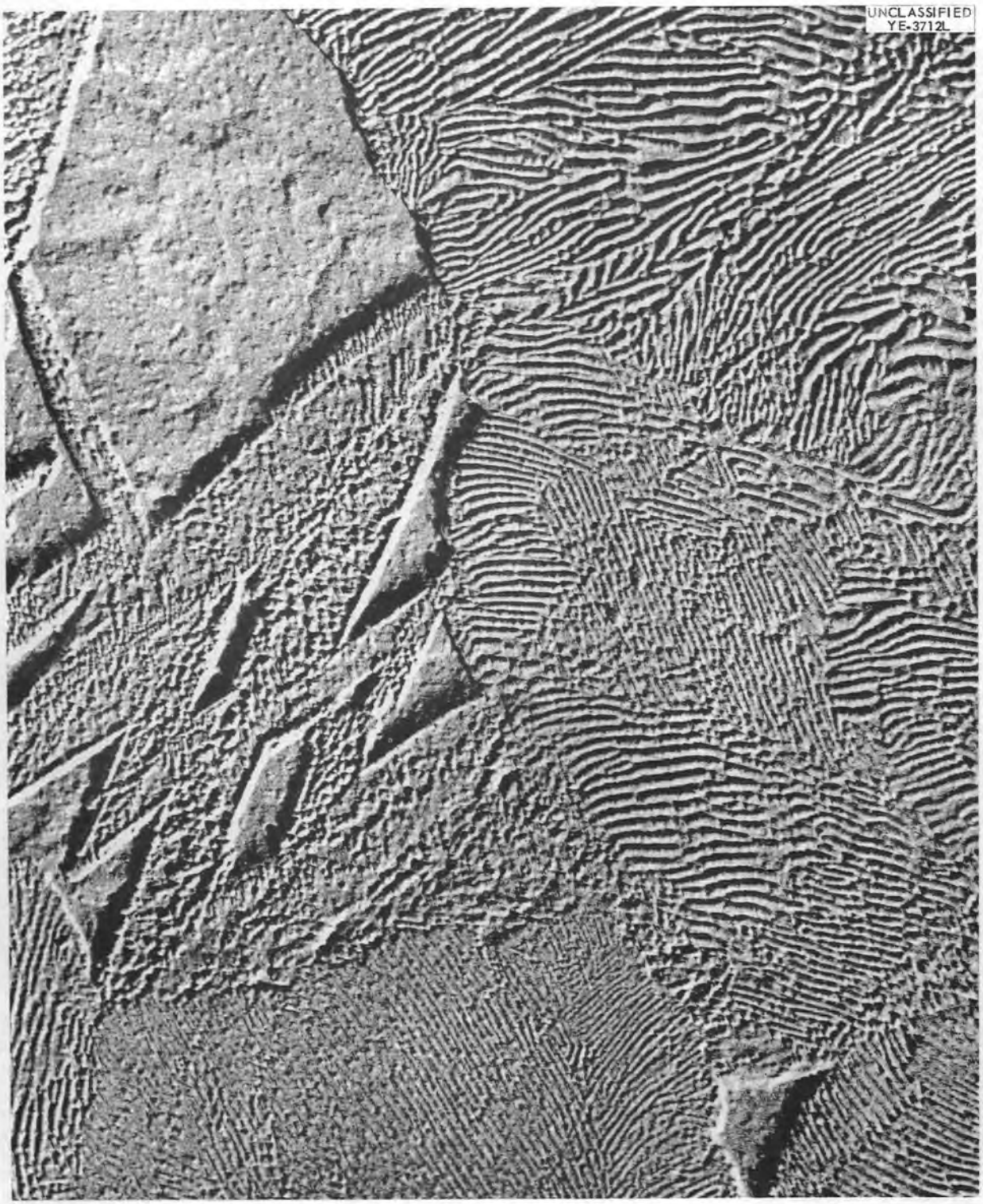

Fig. 7. Plain Carbon Steel Isothermally Transformed at 591°C for 1.6 sec. Note the fine lamellar spacing of the pearlite. No polishing defects, such as smearing, are present. Etchant: a mixture of HCl gas dissolved in absolute ethyl alcohol and 5% trichloroacetic acid. Carbon replica, preshadowed with palladium. 26,000X.

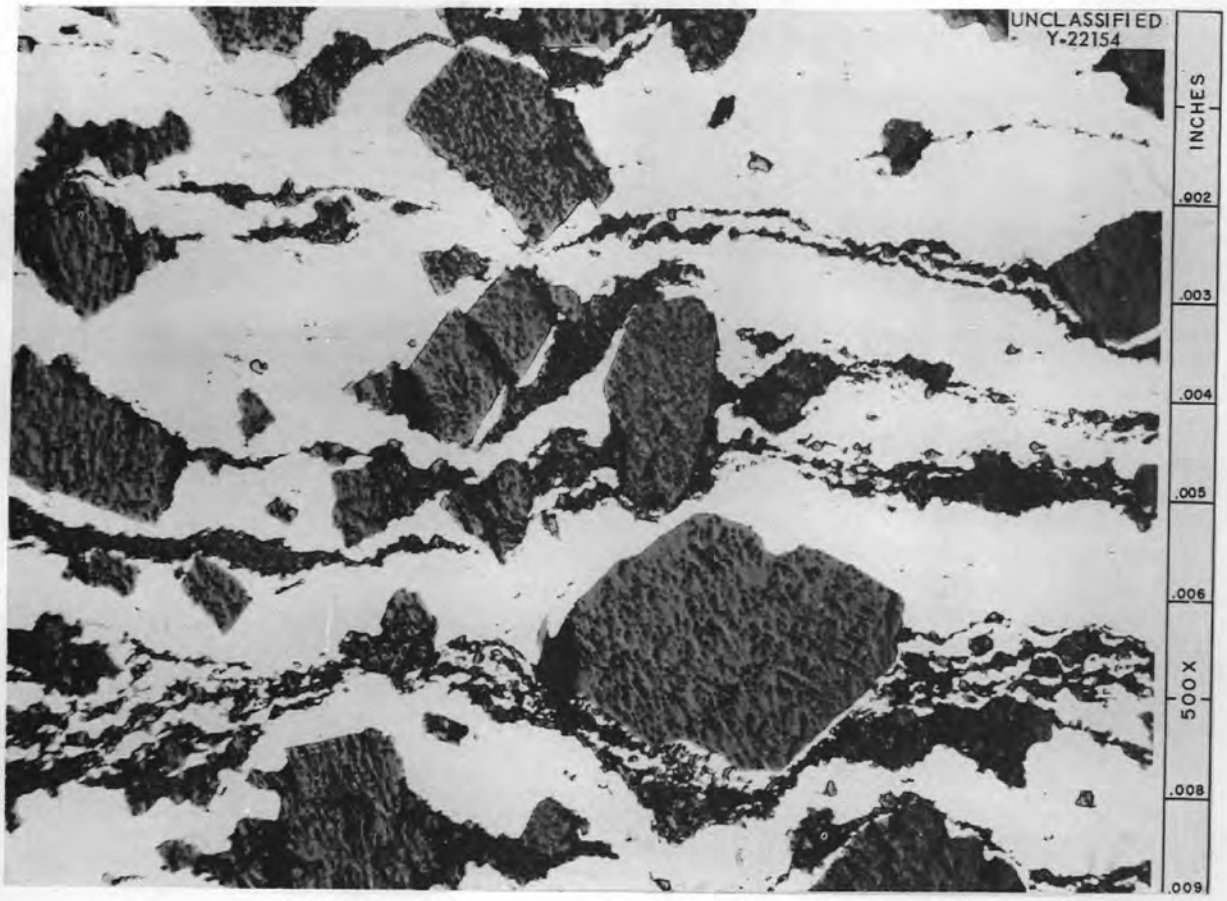

Fig. 8. U_3O_8 Particles in an Aluminum Matrix, Vibratory-Polished. Note that sharp interfaces and very small amount of "pull-out" have occurred. 500X.

In contrast to the commercially available automatic polishers, vibratory polishing is a continuous process – not a batch process. Frequently in the automatic polishing operations, one or two of the samples will require additional polishing either at one of the successive stages of polishing or at the end of the sequence. With a continuous process, such as vibratory polishing, there is no minimum number of specimens required – as many as 24 specimens can be polished simultaneously. For a check on the progress of a polishing operation, one specimen can be removed from the bowl and examined without interrupting the remainder of the specimens.

With conventional hand-polishing techniques, the metallographer or technician devotes all his time to the polishing operations. However, with vibratory polishing, the metallographer is free to attend to many other duties while the specimen is being polished. One of the inherent properties in this new mode of polishing is that over-polishing is less likely to occur; so, if a specimen should remain in the polishing bowl for an additional 5 or 10 min, it is unlikely that repolishing would be required.

It has been the experience of this department that it takes less time to train personnel in the art of metallographic preparation through vibratory polishing. This is probably due to several reasons. This type of polishing does not tax the operator's patience as much as does hand-polishing; he is able to polish various types of samples simultaneously and observe the action of various cloths and abrasives; and he has more free time for instructions, along with learning his new duties and responsibilities.

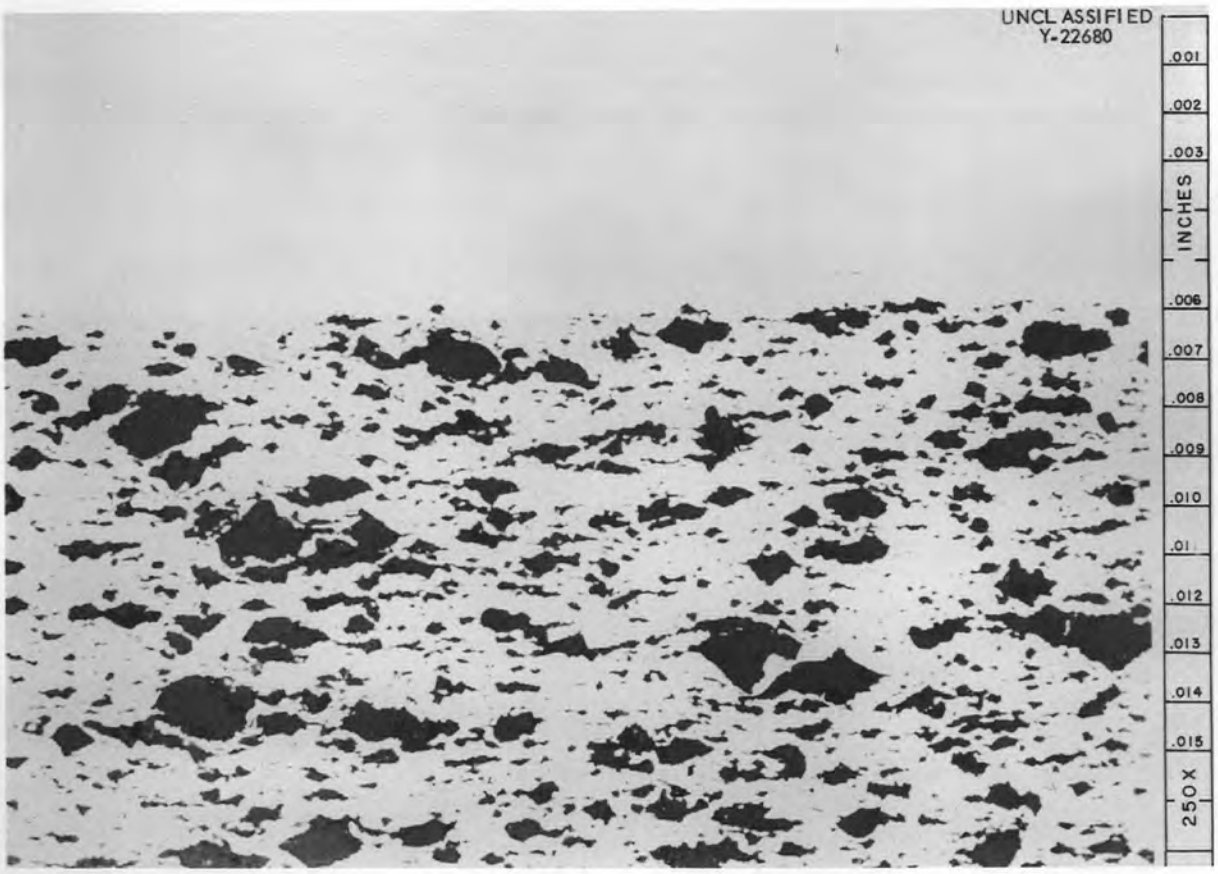

Fig. 9. Rare-Earth Oxide Particles in a Stainless Steel Matrix, Vibratory-Polished. Sharp interfaces and good retention of particles were obtained. 250X.

There are no apparent reasons why vibratory polishing could not be extended to remote metallographic facilities advantageously. The machines are rugged pieces of equipment. The three polishers in this department have required no maintenance, after the original adjustments, in the 14 months of practically continuous daytime operation save for the changing of polishing cloths and other normal operational procedures of any metallographic laboratory. To repeat, it is a continuous process and not a batch process. The polishers do not require mounted specimens of close tolerances, as do most remote facilities, and with simple precautions, such as substituting silicone oils or petroleum oils for water in the abrasive slurries, radioactive dust contamination should offer no additional problem. The controls for the polishers can be located outside the hot cells, as was indicated earlier.

Not only can finishes comparable (and in many cases superior) to those of hand-polishing be attained with vibratory polishing, but also this method of specimen preparation has been accepted by the technicians. In conclusion, it is the concensus of this department that vibratory polishing is fulfilling a definite need in the metallographic laboratory.

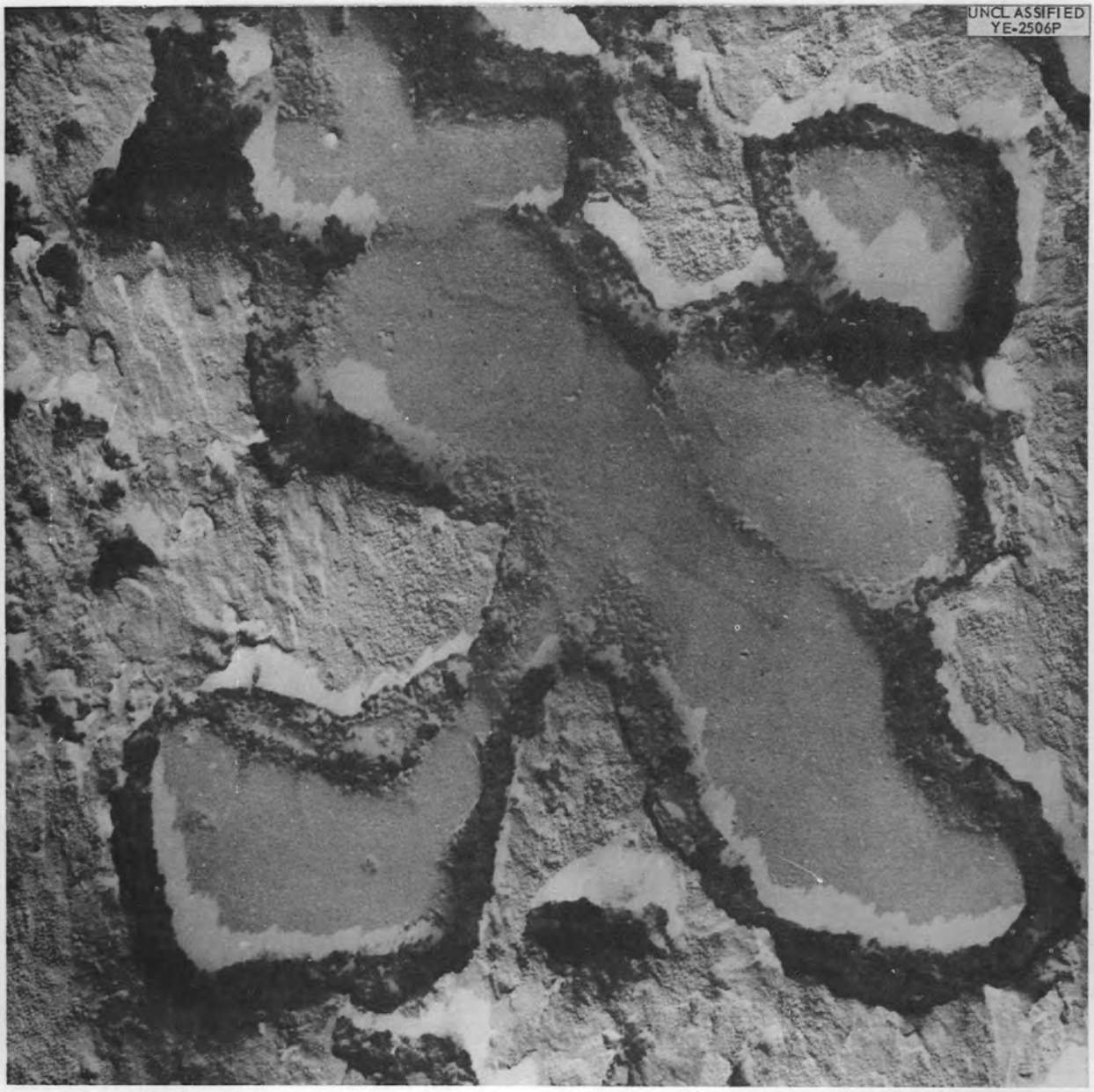

Fig. 10. Electron Micrograph of a Europium Oxide Particle in a Stainless Steel Matrix, Vibratory-Polished. Note scratch-free surface and absence of any smearing of the reaction layer around the oxide particle. Palladium-preshadowed, carbon replica. 22,600X.

Fig. 11. Platinum-Gage Strips Cemented to a Weight Plate Prior to Being Placed in Vibratory Polisher.

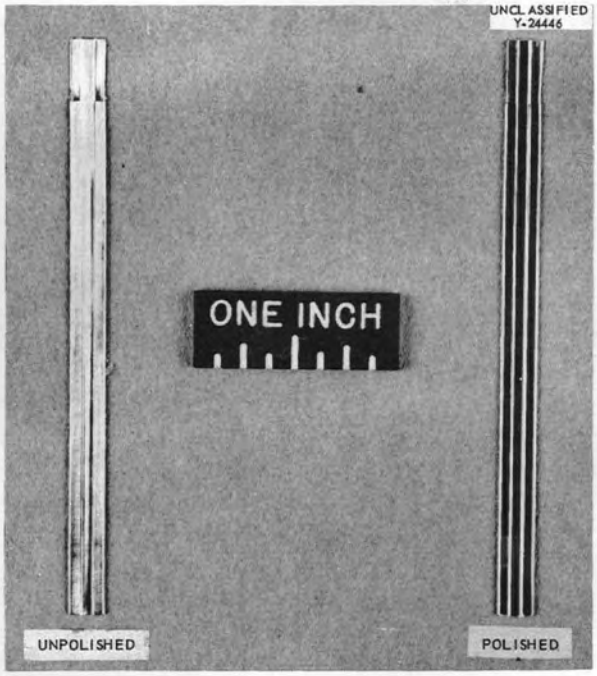

Fig. 12. Unpolished and Polished Platinum-Gage Strips. Note the flat surface of the polished strip compared with the unfinished strip.

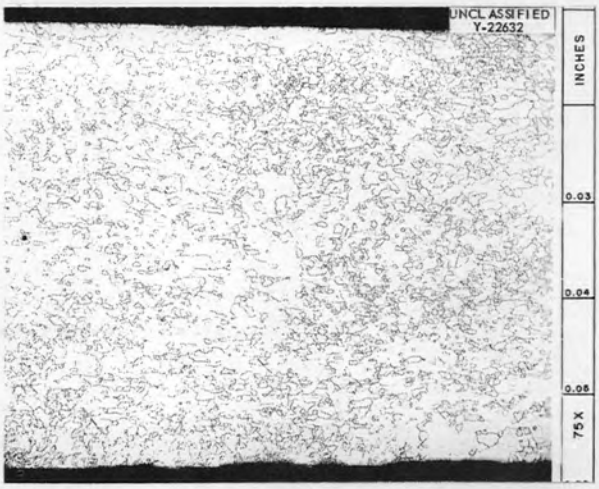

Fig. 13. Niobium Specimen Vibratory-Polished. Note sharp delineation of grain boundaries and only slight rounding of unprotected edges. Etchant: 50 parts H_2O, 25 parts HF, 10 parts H_2SO_4 (conc), 10 parts HNO_3 (conc). 75X. Reduced 32%..

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