# Applications of Polystyrene Particles in Conjunction with Shadow Casting to the Study of Polished Metallic Surfaces

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This is a report of a study of polished metallic surfaces with the electron microscope. Negative collodion replicas were used in this experiment. The determination of width and depth of scratches by means of polystyrene particles and shadow casting is briefly discussed. Experimental results are summarized as follows: The effect of polishing pressure applied is that greater pressure gives wider and deeper scratches and also causes severer flow and distortion of metal near the scratch. In compaiison with unidirectional polishing, multidirectional polishing gives better surface finish with fewer scratches: these scratches run in all directions and have a wide range of width and depth; mostly they are shallower and narrower than would be the case with unidirectional polishing. The average widths and depths of scratches on surfaces polished with various conditions are also given here. Harder abrasives give a higher depth/width ratio.

## INTRODUCTION

SURFACE finish is receiving increasing attention at the present time because of its close connection with friction, wearing, and scoring phenomena. Recent development of the shadow-casting technique and the amazing discovery of the remarkable uniformity of polystyrene latex sizes make such a study of highly polished surfaces much easier than before. In this experiment, the effect of polishing pressure, multidirectional polishing manipulation, and hardness of abrasives are studied, and the average width and depth of scratches are determined for various cases.

## METHODS OF MAKING SURFACE REPLICAS

The bulk materials of surface finish specimens are opaque to an electron beam. In dealing with "electronically opaque" specimens, a thin, structureless, surface replica, suitable for use in the transmission-type electron microscope, is made to reproduce the original surface finish. Many methods of surface reproduction have been developed with great success. One of the principal requirements for a satisfactory surface replica is that it be structureless. Methods of surface reproduction can be classified into two types, according to the principle of obtaining the structureless property. One of them is to change a thin surface layer of the original preparation into a structureless one, as first described by H. Mahl.[1] Another method is to take the advantage of structureless materials, such as resins, silica, etc.

In methods of the first type, the thin, structureless surface replica, usually oxide film, is first formed on the surface of the original sample, and then removed from the bulk material by appropriate chemical means. The original preparation is thus destroyed in the process of obtaining the surface film. This places a serious limitation upon the application of methods of this type. Furthermore, it is not always possible to find some way to change the original surface layer into a structureless one, which is another limitation.

Methods of the second type have no such disadvantages, and hence are in greatest current application. They can further be divided into two kinds, negative and positive replicas.

In making a negative replica, a thin structureless coating is produced on the surface of the original specimen by flowing a suitable solution of a structureless material over the surface and evaporating the solvent. The thin film is usually separated from the original surface by mechanical stripping. After mounting the stripped negative surface replica on a supporting screen, it is ready for use. It is obvious from the name, "negative replica," that this process yields a negative reproduction of the original surface finish; that is, scratches on the original surface will appear as peaks on the negative reproduction, and vice versa.

Now, if we repeat this process by making a second surface replica from a negative reproduction, we will get a positive reproduction of the original surface. In this positive replica process, the first negative reproduction is usually obtained by evaporating a thick layer of metals onto the original surface or by making an impression of the original surface in a plastic.

Negative replicas are most suitable for the purpose of studying the surface finish of a polished metallic surface when the shadow-casting technique is employed. This is because the depth of a scratch on the original surface can be easily obtained from the length of the shadow cast by its corresponding peak on the negative reproduction.

## SHADOW CASTING—A VALUABLE TECHNIQUE IN STUDYING SURFACE FINISH

Shadow casting is a technique of depositing an extremely thin layer of heavy atoms obliquely onto the surface of a replica. Its greatest advantage is to enhance the contrast. As pointed out by Williams and

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[1] H. Mahl, Z. tech. Physik **21**, 17 (1940); Metallwirtshaft **19**, 1082 (1940); Z. tech. Physik **22**, 23 (1941).

Wyckoff,[2] the diffuseness of electron micrographs obtained from collodion or Formvar replicas is due to insufficient contrast, not the lack of sharpness of replica reproductions themselves. For example, as reported by Schaefer and Harker,[3] an unshadowed replica of a surface, carefully polished with MgO, has a variation of thickness not greater than 200A,which gives such poor contrast that little, if any, surface detail can be observed under an electron microscope. Since shadow casting greatly enhances the contrast, it was used by Williams and Wyckoff to bring out the fact that such replicas are faithful reproductions of the original surface, and the diffuseness is due to insufficient contrast. Thus, the necessity of using shadow casting technique to bring out the surface detail of highly polished surface is obvious.

This technique is now widely in use. It not only enhances the contrast but also gives the following advantages:

1. It enables us to estimate the height of surface elevations from the length of their shadows. This method of determination of the height of elevation is much simpler and easier than many other methods, e.g., stereoscopy.
2. It gives a direct impression of projections and depressions and hence a 3-dimensional feeling of the surface contours.
3. It enables us to detect the existence of extremely small objects beyond the resolution limit of electron microscope.
4. It makes the thickness of the replicas not so critical as in the case of unshadowed replicas.

From the discussion above, it is obvious that shadow casting is a valuable technique for studying surface finish.

The material used for shadow casting is usually metal. For satisfactory result, the metal ought to have the following properties: (1) it should be structureless, (2) it should not migrate after deposition on the replica, and (3) it should possess great scattering power. The second property is necessary for yielding sharp shadows. The third property allows the use of a very thin coating to minimize the alteration of the contours of replica surfaces. Chromium, gold, and uranium are most commonly used. All of them yield a structureless coating and sharp shadows. Among them uranium is the best because of its inherently great scattering power, and is used in this experiment.

## APPLICATION OF POLYSTYRENE PARTICLES

Spherical polystyrene particles were found by Williams and Backus[4] to be exceptionally uniform in size. The average size has been determined[5] to be 2593±40A. Owing to this inherent remarkable uniformity of size, they can be used for magnification calibration. As mentioned before, a shadowed replica yields an electron micrograph which gives a 3-dimensional impression. The shadows of these little spheres, like shadows of tall telephone poles on the street, further assist in giving observers a 3-dimensional feeling. This is demonstrated very nicely by a couple of spherical particles in the center of Fig. 1.

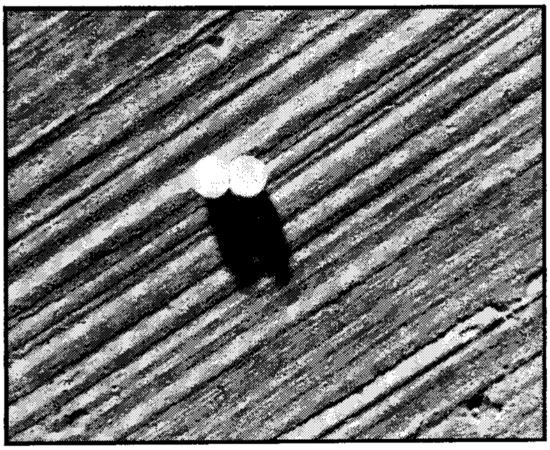

Fig. 1. Brass surface polished with 600F Alundum, heavy pressure, and unidirectional polishing technique.

Considerable inaccuracy in the estimation of surface elevation from their shadow and the angle of shadow casting are usually introduced by the following sources of errors:

1. The surface replica on its supporting screen is often not horizontal. This makes the computed angle of shadow casting deviate from the actual angle.
2. The local angle of shadow casting is frequently different from point to point.

The application of polystyrene particles has the advantage that the actual angle of shadow casting at any point can easily be obtained. It has also the advantage of assisting in focussing the image.

Precautions in handling polystyrene latex are important. As summarized by Gerould,[5] they include freezing or evaporation of water from undiluted latex, chemical or bacterial contamination, change of *p*H, overheating or contamination under electron bombardment, and fusing due to insufficient dilution.

## PREPARATION OF SURFACES

The material used in this experiment was large-grain 70–30 brass in its anealed condition. Flat surfaces of brass samples were ground successively with metallographic emery papers of increasing fineness from 1/0 to 4/0. After being ground with 4/0 emery paper, the surfaces were polished with 600F Alundum, which gives a surface with fine scratches when examined under an optical microscope at 100×. A number of samples were further polished with 0–$\frac{1}{2}$ micron diamond dust, which gave a scratch-free appearance at 100×. On each step

[2] R. C. Williams and R. W. G. Wyckoff, J. Appl. Phys. **17**, 22–23 (1946).
[3] V. J. Schaefer and D. Harker, J. Appl. Phys. **13**, 427–33 (1942).
[4] R. C. Williams and R. C. Backus, J. Appl. Phys. **20**, 224–5 (1949).
[5] C. H. Gerould, J. Appl. Phys. **21**, 183–4 (1950).

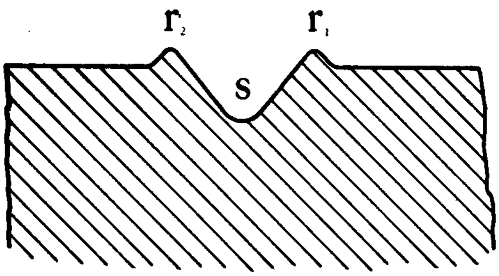


FIG. 2A. Diagram showing a scratch on the surface bounded by ridges on both sides.

of passing from a coarser to a finer abrasive, the specimens were cleaned to avoid carrying the coarser grits used in the previous step over to the next step. The surfaces were cleaned with benzene after the last step of polishing, and collodion negative replicas were made very shortly after they had been cleaned.

## RESULTS

### 1. Effect of Pressure

In order to study the effect of normal pressure applied during polishing, two surfaces were polished with same

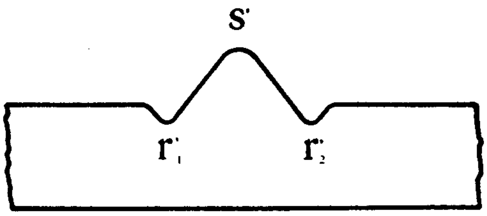


FIG. 2B. Sketch of the negative reproduction of the scratch.

abrasives, 600F Alundum, but with different pressures. A unidirectional polishing technique was used. Figure 1 is an electron micrograph which represents the average condition of a surface polished with heavy pressure. The scratches are running parallel. The width of the scratches, which can easily be obtained by comparison with the two polystyrene particles (2593±40A) in the center, is *ca* 0.3 micron. The length of the shadow of the scratches shows that the depth of scratches is *ca* 0.04 micron. A point of great interest is that the heavy pres-

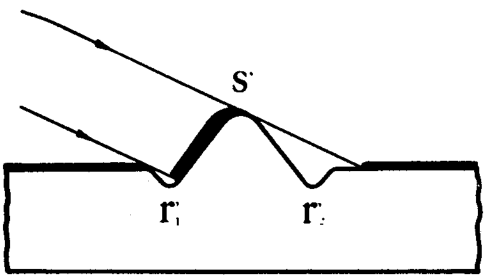


FIG. 2C. Diagram showing the deposition of uranium during shadowcasting.

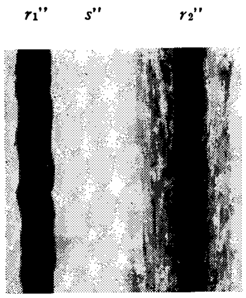


FIG. 2D. The sketch of an electron micrograph resulting from such a shadowed replica as shown in Fig. 2C.

sure causes a noticeable flow of metal near the scratch. This is clearly indicated in Fig. 1 by the dark lines which are interpreted as ridges on the original surfaces, bounding both sides of almost every scratch. This interpretation is further illustrated by a number of sketches as follows: Fig. 2A shows a scratch on the original surface, bounded by ridges $r_1$ and $r_2$. The negative reproduction has the form shown in Fig. 2B. The scratch $s$ appears as a peak $s'$, while ridges $r_1$ and $r_2$, as valleys $r_1'$ and $r_2'$, on the negative reproduction. During the shadow-casting process, the valleys $r_1'$ and $r_2'$ do not receive any uranium. Figure 2C shows

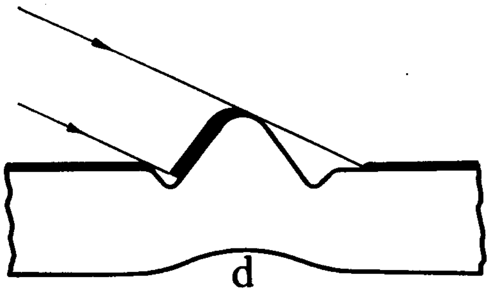


FIG. 2E. Same as Fig. 2C but with a depression due to surface tension.

the situation after shadowcasting. Figure 2D gives the corresponding sketch of an electron micrograph (with dark shadow) resulting from such a shadowed replica. Two very dark lines, $r_1''$ and $r_2''$, bounding the scratch are due to valleys, $r_1'$ and $r_2'$, on the negative replica, where, in addition to the absence of uranium, the thickness of the collodion film is relatively thinner than on the surrounding area.

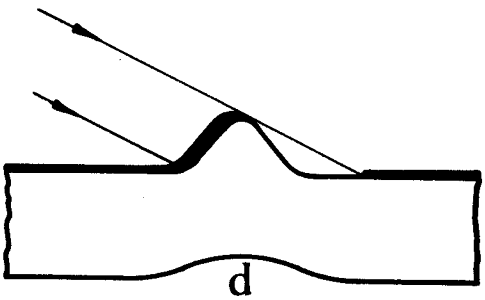


FIG. 2F. Same as Fig. 2E but without valley.

Deacon, Ellis, Cross, and Sennett[6] showed that the free surface of the replica has a depression, $d$, near the scratch, as shown in Figs. 2E and 2F, because of the action of surface tension. Nothing conclusive regarding the presence of such depression can be obtained from Fig. 1. We can have three possible cases: (1) only the depression due to surface tension is present on the negative replica as shown in Fig. 2F, (2) only the valleys $r_1'$ and $r_2'$ are present as shown in Figs. 2B and 2C, and (3) both are present as shown in Fig. 2E. The presence of only the depression due to surface tension causes the collodion film to be thinner than its surroundings on both sides of scratch, but will not cause two dark lines to appear on both sides of the peak in an electron micrograph. It is possible for only one to appear on the shady side, the side away from the source of shadow casting, because on the sunny side of the peak the effect of the uranium coating upon the appearance of the electron micrograph overcomes the effect due to variation of the film thickness. Furthermore, in the case of the presence of the depression only, the boundary of the dark line could not be very sharp.

Figure 1 shows that the presence of only the depression is not the case. There is no way to determine whether (2) or (3) is the case. Nevertheless, there is no doubt about the presence of ridges bounding both sides of scratches as the result of a flow of metal under heavy polishing pressure.

Figure 3 gives the appearance of a second surface which was prepared the same as the first surface, except that this second surface was polished with light pressure, while the first one was polished with heavy pressure. As the result of unidirectional polishing, scratches are also running parallel. Comparing with the calibration spheres, the width of scratches is *ca* 0.2 micron, which is narrower than that on the first surface polished with heavy pressure. The scratches are also shallower. Their depth is *ca* 0.03 micron, which is obtained by comparing the length of their shadow with that of the spheres. No noticeable ridge on either side of the scratch is observed. This indicates that the flow and distortion of the metal near the scratch is much less in this case than in the case where a heavy pressure was used.

Thus far, we can see that heavier pressure gives wider and deeper scratches and also causes a severer flow and distortion as indicated by the ridges bounding both sides of the scratches.

## 2. Unidirectional *vs* Multidirectional Polishing

It is a common manipulation to rotate the specimen counterwise to the rotation of the polishing wheel during the polishing operation. A specimen was polished this way with 600F Alundum abrasives and light pressure. Figure 4 shows the multidirectional nature of the scratches on a surface thus prepared, because such a manipulation gives a continuous change of direction of polishing. This continuous change of direction of polishing causes the deep, long, continuous scratch which is formed at a certain instant to become shallower at later stage, then to change into discontinuous segments, and finally to be washed out. This is fully demonstrated by Fig. 4. Scratches are running in all directions; the number of scratches appear to be fewer due to the washing-out action; the width and depth of scratches vary over a wide range; there are comparatively fewer wider and deeper scratches, most of the scratches are narrower and shallower in comparison with that shown in Fig. 3; some scratches show the early stage of breaking down into discontinuous segments, and some scratches have reached the disappear-

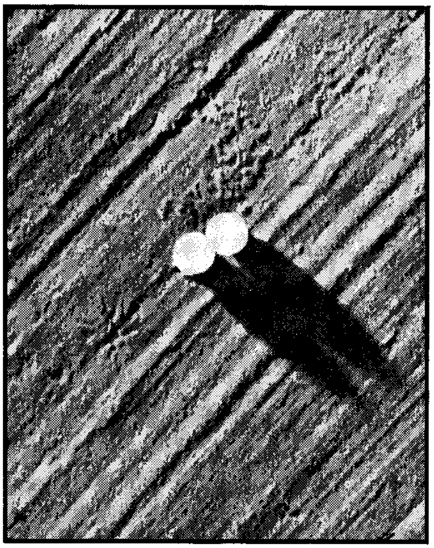

FIG. 3. Brass surface polished with 600F Alundum, light pressure, and unidirectional polishing technique.

FIG. 4. Brass surface polished with 600F Alundum, light pressure, and multidirectional polishing technique.

[6] Deacon, Ellis, Cross, and Sennett, J. Appl. Phys. 19, 704–12 (1948).

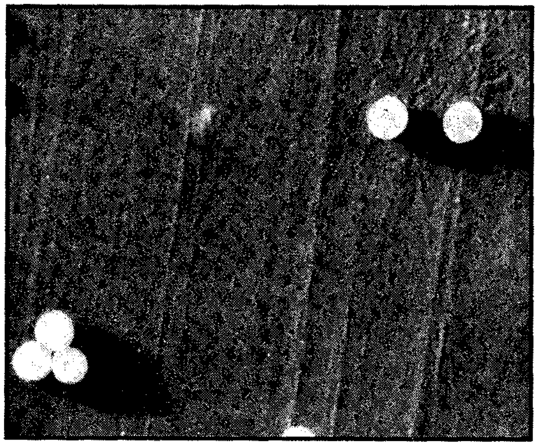

FIG. 5. Brass surface polished with 0–½ micron diamond dust, light pressure, and unidirectional polishing technique.

ing stage so that only faint traces of small broken segments are left.

Another pair of specimens was further polished with 0–½ micron diamond dust. The pressure applied was light. The average width and depth of scratches obtained are *ca* 0.05 and 0.01, respectively. Figure 5 shows the electron micrograph of a surface finished by unidirectional polishing and Fig. 6 that of another surface finished by the multidirectional polishing manipulation. They show the same improvement of the surface finish as a result of the application of multidirectional polishing manipulation.

### 3. Effect of Hardness of Abrasives

It is also interesting to notice that harder abrasives gives a higher depth/width ratio. Diamond dust (10 on Mohs scale) gives a ratio of *ca* $\frac{1}{5}$, while Alundum (8–9 on Mohs scale), a ratio of *ca* $\frac{1}{10}$.

FIG. 6. Brass surface polished with 0–½ micron diamond dust, light pressure, and multidirectional polishing technique.

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