New Methods for Preparing Surface Replicas For Microscopic Observation

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RECENTLY a method was described[1] for studying surfaces in the electron microscope by the direct replica method. The technique, briefly, consists in flowing over the surface a dilute solution of a resin dissolved in a suitable solvent. With the evaporation of the solvent, a very thin film coats the specimen. The coated specimen is then immersed in water, the replica film peeled from it, brought to the water surface, and mounted in the usual way for study in the microscope.

In a number of instances, especially with certain types of metallographic specimens which form corrosion products upon contact with water, it is desirable to remove the surface replica without immersion in water. Three new methods for removing replicas have recently been devised in this laboratory which overcome these corrosion problems and simplify the stripping process.

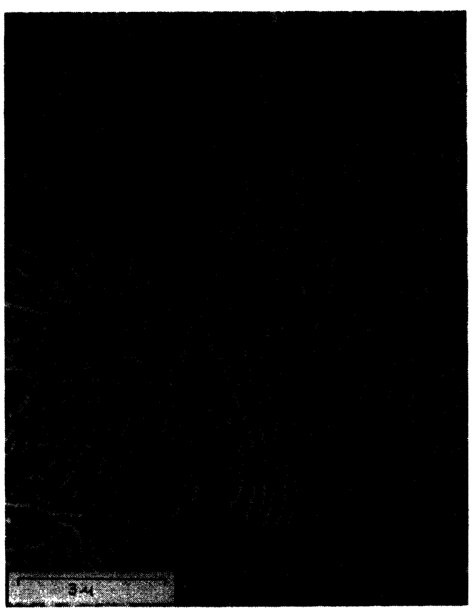

FIG. 1. Electron micrograph of replica of oil quenched 1.1 carbon steel surface.

One of these termed the "warm method" consists of the following procedure. After the thin replica film is formed on the specimen a 20 percent solution of gelatin dissolved in warm water is applied to the surface (about 0.5 drops per cm^2). The water is evaporated from the gelatin by a stream of warm dry air. Upon drying, the gelatin film often cracks off the surface spontaneously or may be split off by inserting a razor edge under one side. As the gelatin leaves the surface it takes with it the replica film. This

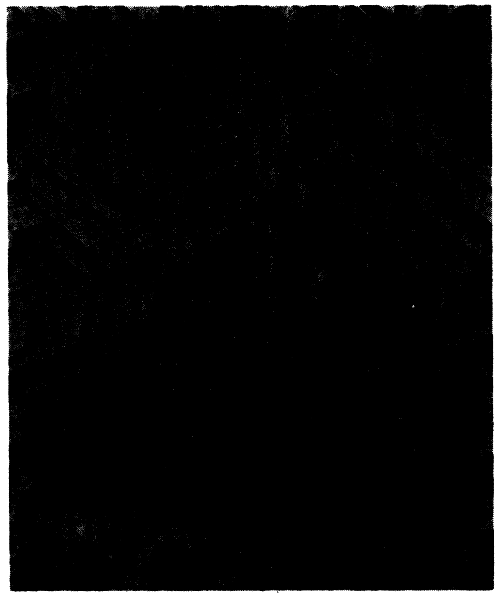

FIG. 2. Photomicrograph of replica of etched quartz surface at crystal boundary.

double film is then placed gelatin side down in a shallow tray, washed with several changes of warm water until the gelatin dissolves, leaving the replica floating on the surface ready for mounting.

Figure 1 is an electron micrograph of a polyvinyl formal replica removed by this method from a Nital etched specimen of oil quenched 1.1 percent carbon steel.

A second procedure termed the "cold method" may be preferred for some specimens. Again with polyvinyl formal resin as replica material the dry film is covered with a dilute 0.5 percent solution of gelatin (or other surface active protein) dissolved in distilled water. A lens of protein solution several mm thick should be applied and the specimen placed on the freezing coils of a refrigerator. As soon as the lens is frozen the lower part of the specimen is placed in a shallow tray containing liquid air and left there until the frozen protein solution in contact with the replica cracks off at the resin-metal interface due to the unequal contraction of the cooling materials. The lens, now having the replica film as part of it, is lifted from the specimen, placed on a piece of filter paper, and warmed to room temperature. The replica is then floated on a water surface and mounted in the usual manner.

A third procedure is mentioned here because it has been found to be very useful for preparing high contrast photomicrographs of the structure of surfaces by the replica method in which specimens ranging all the way from etched quartz crystals to scored metal surfaces, soft leaf mining worms to flower petals are used. The best results with this third method are obtained with thicker replica films since the distance between the high and low spots of the type of specimens described is often a micron or more. After the replica film is formed by flowing a one to ten percent solution of a suitable resin over the specimen the solvent is evaporated. A strip of transparent scotch tape is pressed into optical contact with the coated specimen and then pulled away from it. When this is done the replica adheres to the scotch tape. This may be fastened to a sheet of glass for study under the light microscope, the Cellophane surface serving as a cover glass. Figure 2 is a photomicrograph of such a replica obtained from an etched quartz crystal showing the contact between a primary and a penetration crystal.

Acknowledgments are due to Miss Ethel Thompson, who prepared many of the replicas and the electron micrograph shown in Fig. 1 and to Miss Margaret Murphy, who prepared the metallographic specimens used in developing the warm and cold methods described.

[1] V. J. Schaefer and D. Harker, J. App. Phys. 13, 427 (1942).

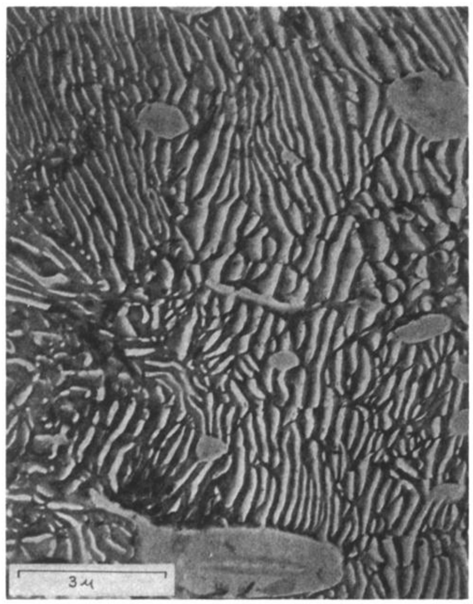

FIG. 1. Electron micrograph of replica of oil quenched 1.1 carbon steel surface.

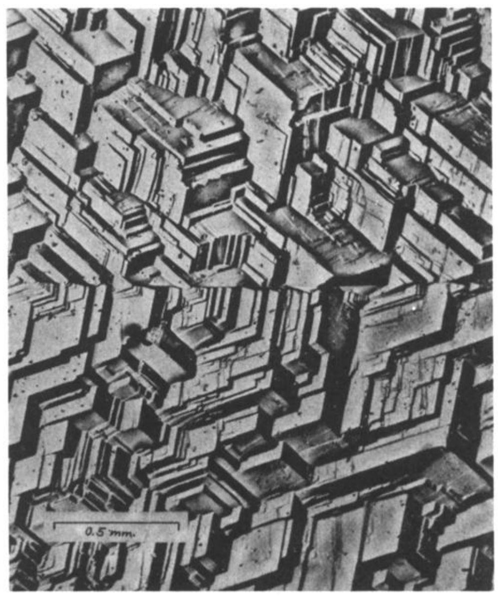

FIG. 2. Photomicrograph of replica of etched quartz surface at crystal boundary.