Electron Microscopy of Vulcanized Rubber using Gelatine First-Stage Replicas

TWO-STAGE replication techniques using evaporated carbon as the second stage have been widely applied in the electron microscopy of solid surfaces[1]. The most popular materials for first-stage replicas, however, are soluble only in organic liquids, and since vulcanized rubber generally swells in contact with such solvents, they must be avoided if faithful reproduction of such a surface is to be obtained.

Water-soluble polymers have been employed for the replication of rubber by several workers. Harris[2] used a solution of gum arabic and glycerol, and gelatine has been used by Bromley and Downer (private communication). We have examined a number of other water-soluble materials. Ammonium polymethacrylate and polyvinyl alcohol have inferior film-forming properties, and sodium alginate films, though desirable in several ways, swell in water to such an extent that the evaporated carbon layer is severely disrupted. The present communication is concerned with the use of gelatine in the first stage of the replication process.

In the technique employed by Bromley and Downer, a 20 per cent solution of gelatine in water at 80° C. was cast on the freshly created rubber surface and allowed to dry. The gelatine wafer was stripped and a layer of carbon evaporated on to it, after which the gelatine was dissolved away by floating on warm water. While we occasionally obtained good micrographs by this method, the resolution, though passable, generally fell below the standard required in our investigation. The inconsistency was eventually traced to the persistence of a thin layer of gelatine, a few hundred angstroms thick, in contact with the carbon film. Prolonged washing in water or alkali failed to disperse the layer, even in samples which had not been exposed to the electron beam. Fig. 1 shows the edge of a replica at which the carbon film has folded back from the gelatine layer. (The dark circles are carbon-black filler particles extracted from the surface of the rubber.)

Acting upon a suggestion from Mr. A. G. Ward, enzymatic destruction of the gelatine was attempted, with complete success. The gelatine – carbon sandwich is floated in a saturated solution of trypsin in 0·1 *M* sodium chloride. The temperature, as is to be expected, is quite critical, and must be kept at 37°–39° C. Under these conditions the carbon film, in most cases wholly intact, is separated from the gelatine in 15–20 min.; this time could be even further reduced if trouble were taken to adjust the *p*H to 8, though in practice this would necessitate the use of a buffer solution. The end-point of the digestion process is indicated by disappearance of the 'streamers', visible in strong light below the dissolving gelatine wafer. The carbon film is lifted on a glass slide and refloated in distilled water, being allowed to wash there for five to ten minutes before collection on a copper grid.

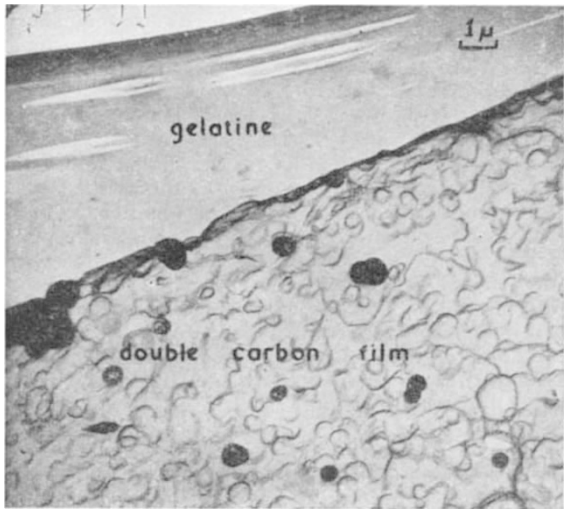

Fig. 1

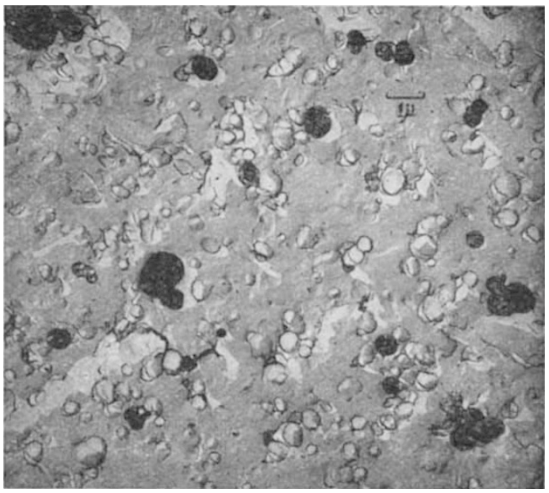

Fig. 2

An example of the results obtained is given in Fig. 2, which shows the torn surface of a vulcanized natural rubber containing 33 per cent by weight of colloidal carbon black. The replica, which has been shadowed with a small quantity of gold–palladium, probably has a resolution of better than 100 A.

This work, which will be fully reported elsewhere, forms part of a programme of research undertaken by the Board of the British Rubber Producers' Research Association, in conjunction with the Department of Applied Physics of Northampton Polytechnic.

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[1] Bradley, D. E., *J. App. Phys.*, **27**, 1399 (1956).
[2] Harris, P. H., Annual Conf. Inst. Phys. Electron Microscopy Group, July 1955.

A Method of investigating the Stress-Distribution in Granular Materials

THE problem of the direct determination of the distribution of stress in a mass of granular material, under a specified system of loads, is of considerable practical importance in such fields as powder metallurgy and soil mechanics. In the solution of such problems, however, many difficulties are encountered, and in the present communication a simple technique, believed to be original, is presented.

In the study of the stress-distribution in a granular material the technique in which the whole of the