J. Indian Inst. Sci. 63 (A), May 1981, Pp. 123–130

Preparation of conical pinholes suitable for X-ray projection microscopic techniques

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Abstract

Attempts were made to fabricate a conical pinhole for studying biological specimens using the techniques of X-ray projection microscopy. Mechanical, photolithographic, electroforming, and jet electropolishing methods were employed for this purpose. It was found that whereas the electroforming methods yield a better conical shape, better polishing is obtained with the last method.

Key words: Conical pinhole, X-ray projection method.

1. Introduction

In recent years, there has been interest in the fabrication of pinholes for application in the fields of optics, projection microscopy, electron microscopy, and in the study of extremely slow leakage of gases. The world's smallest circular hole (approx. 25 Angstroms) appears to be the one made in a nickel plate by an American group of workers[1]. We have been interested in fabricating a divergent micropinhole (microvent) for the purposes of examination of biological samples by projection microscopy using soft X-rays[2]. In this technique, the role of pinhole is analogous to that of aperture in a pinhole camera—to permit the entry of a small amount of radiation and block the rest of it. The following is a brief account of these investigations.

2. Experimental

As mentioned above, in addition to working as an aperture the substrate (plate with the pinhole) also has to act as a shield against the radiation. Since soft X-rays were

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being used in the studies, a 1·5 mm thick plate of copper or any heaveir metal like Ni was considered adequate.

The following techniques were tried to achieve such a divergent pinhole : (1) Mechanical ; (*b*) Photolithography ; (*c*) Electroforming and (*d*) Jet electropolishng.

2.1. *Mechanical methods*

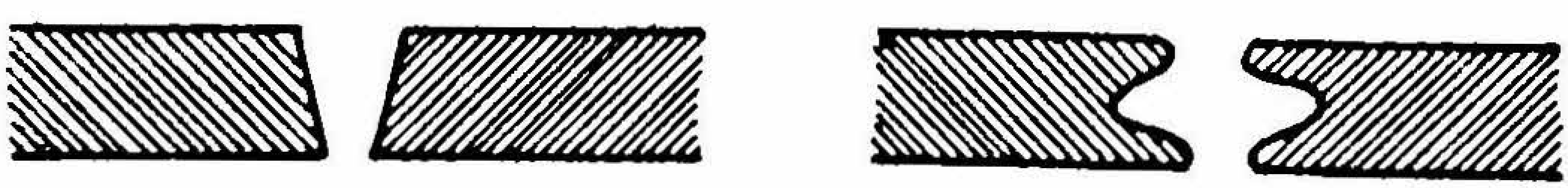
A fine hole was drilled in a flat lead sheet of approx. 1·5 mm thickness in such a manner that the tip of the drill-bit just appeared on the other side of the sheet. The hole was further narrowed down by gently beating the sheet with a wooden hammer. This technique did not yield satisfactory results as the contours were found to be highly uneven and the beating could not be refined further. Moreover, lead being a soft and malleable material, it was difficult to retain the shape of the hole.

2.2. *Photolithography*

The standard technique for making printed-circuit boards (pcb) was tried. We attempted to work on the assumption that if a surface is etched chemically in a suitable bath, the etching proceeds in a conical form, provided the time and speed of the process are controlled. In other words, the part of the surface of the substrate directly in contact with the chemical will be affected the most whereas the inner sections of the substrate will be affected correspondingly less ; the etching would proceed in gradation, so that a conical hole is formed.

To make the photo-mask, a black circle with 10 times bigger diameter than the base of the required cone was made on a translucent plastic sheet. The etching was continued for about an hour until a hole appeared on the other side of the substrate.

Contrary to our expectation (Fig. 1*a*) the etching proceeded in the manner shown schematically in fig. 1*b*. There was a considerable amount of under-cutting as the etching proceeded and this led to cones of unacceptable configurations. It seems as though the photo resist has a tendency to peel off the surface of the substrate after a few minutes.



a *b*

FIG. 1. Photolithographic method : (*a*) Expected configuration ; (*b*) Observed configuration.

2.3. *Electroforming*

This has proved to be the most promising technique so far. Here, a perspex rod, 1 cm in diameter, was machined very smoothly to obtain an approx. 110° tapering with

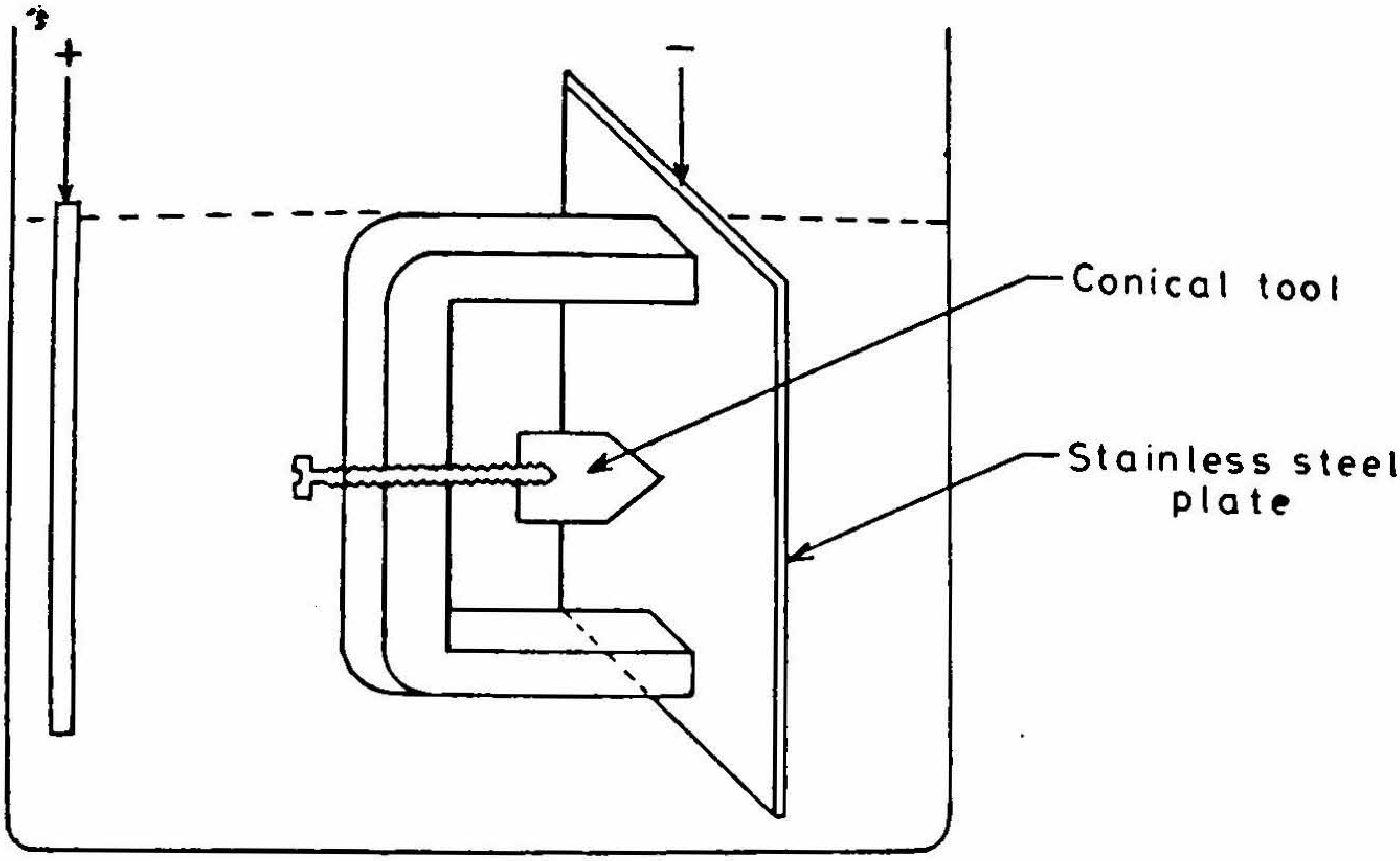


FIG. 2. Schematic diagram of the sample-holder used in the electroforming method.

the finest possible tip on the vertex (The cone angle is not critical.) This was held against a smooth stainless steel plate (fig. 2). This assembly formed the cathode of an electrolysis cell. After the deposition was completed, the copper plate was peeled off the substrate. The copper plate so formed had a conical pit in it. In some cases, a hole had already formed. In those cases where a hole was not formed, the following two techniques were employed to open a hole ;

(i) Laser pulses were bombarded at the valley of the pit. After a total of 64 pulses at 100 J, a hole was punched which is shown schematically in fig. 3*b*. The size of the hole is ~ 40 microns. It is seen that the hole is not uniform, and is cylindrical in geometry. In addition, some molten copper was observed projecting inside the hole.

In this process, we encounter two major difficulties: (*a*) it is extremely difficult to align the axis of the cone with the axis of the laser beam ; moreover, the accuracy with which the laser strikes the same spot repeatedly is also limited ; (*b*) it is almost impossible to remove the molten material from the substrate.

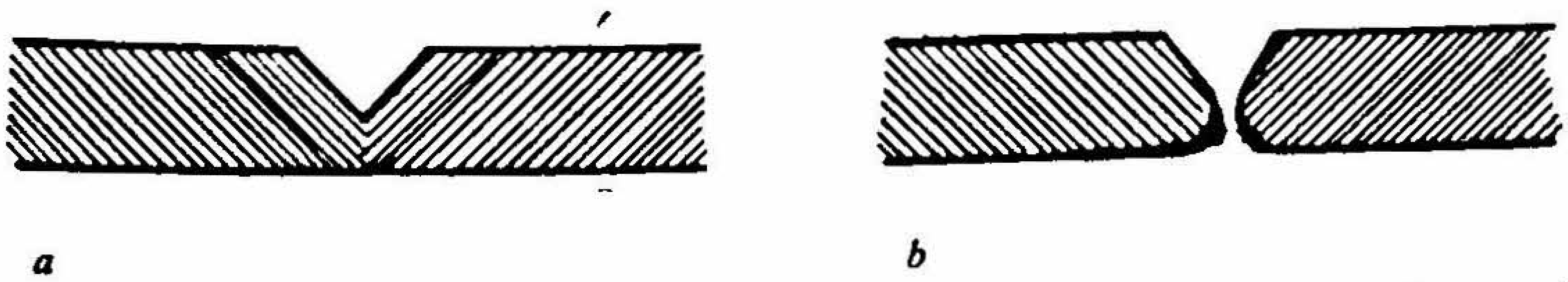
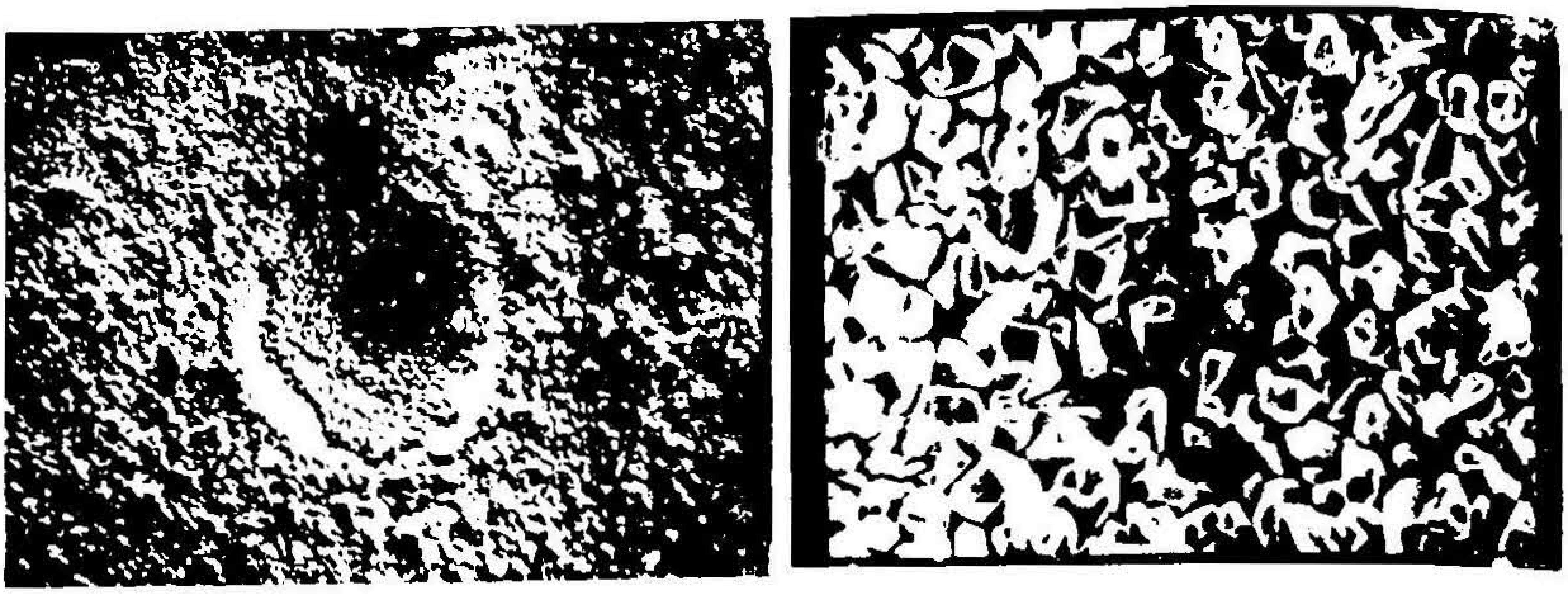


FIG. 3. Results of the laser punching method : (*a*) before punching ; (*b*) after punching.



a *b*

FIG. 4. Results obtained with the electroforming technique : Material—copper.

In another technique, attempts were made to open a hole by slowly etching out material from the closed end of the substrate. In this case, the copper plate formed the anode of the electrolysis cell. The hole so formed was subjected to a process of periodic etching and polishing.

Several attempts were made using the technique mentioned above. The Scanning Electron Microscope (SEM) pictures of some of the samples are shown in figs. 4-6. Each set of figures (4*a* and *b*, for example) corresponds to a different trial. A thin layer of gold (approx. 1 micron thick) was evaporated on the sample corresponding

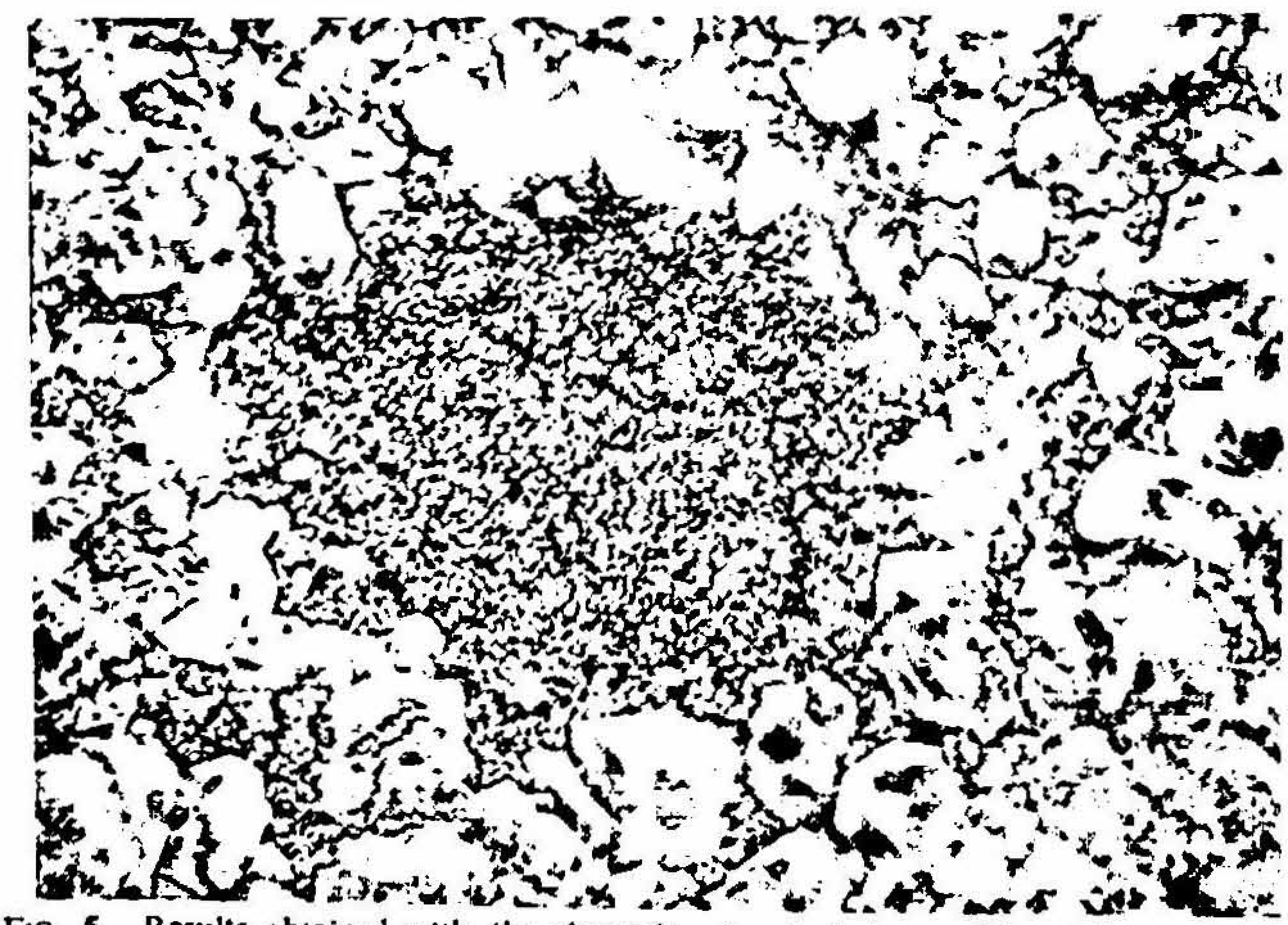
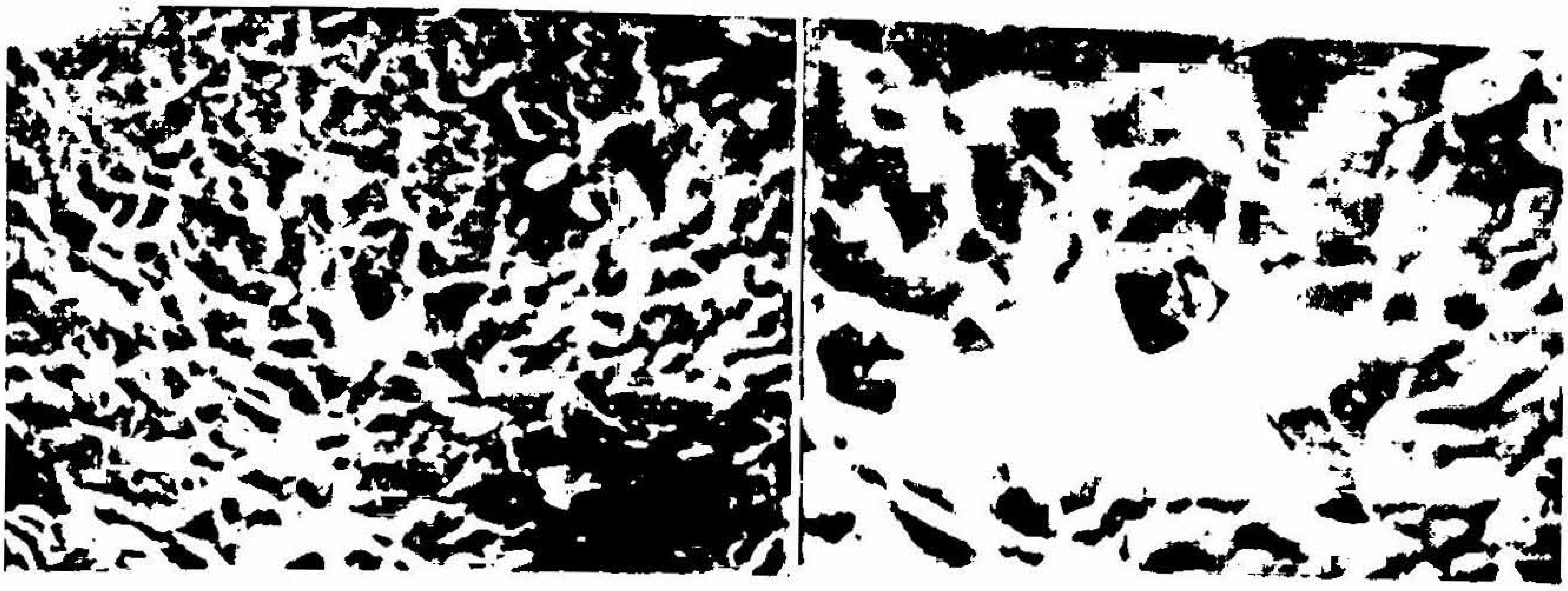


FIG. 5. Results obtained with the electroforming technique : Material—copper.



a *b*

FIG. 6. Results obtained with the electroforming technique using a gold-plated sample.

to fig. 6. A continuous tapering is clearly seen in all the samples. A closer examination of the photographs reveals some interesting features. For example, in fig. 4 (*a* and *b*), copper crystals are seen lying in a random manner, and consequently, no clear-cut hole is formed. In sample 2 (fig. 5) the surface appears to be smoother as compared to that in sample (1) (fig. 4). However, there is no hole in this case either. We have not been able to account for the deposition of extremely fine particles in the valley.

In the case of sample 3 (fig. 6), a hole was identified under an optical microscope. It was narrowed down by evaporating a thin film of gold (approx. 1 micron thick) over the sample. As is clear from the pictures in fig. 6, the surface conditions are improved considerably by gold evaporation. There is a very clear and smooth rectangular hole, except for some kind of a cyst formation on one side. Unfortunately there is no way of checking the sample at each stage of development and exercising precise control. Perhaps with a very large number of trials, it may be possible to obtain one hole with acceptable characteristics.

Our experience is that copper is not a good material for making pinholes with these techniques. It is extremely difficult to obtain a smooth surface and a clean hole with this material. Attempts were, therefore, made on nickel. But, unfortunately, electroforming of nickel was found to be very sensitive to the surface conditions of the substrate and physical conditions of the electrolyte. A further technique, described below, was, therefore, tried to make a pinhole in a stainless steel substrate.

2.4. *Jet electropolishing technique*

This technique is discussed in detail by Goodhew[3], and Nandedkar[4]. The arrangement is shown schematically in fig. 7. Briefly, a small aquarium pump compresses

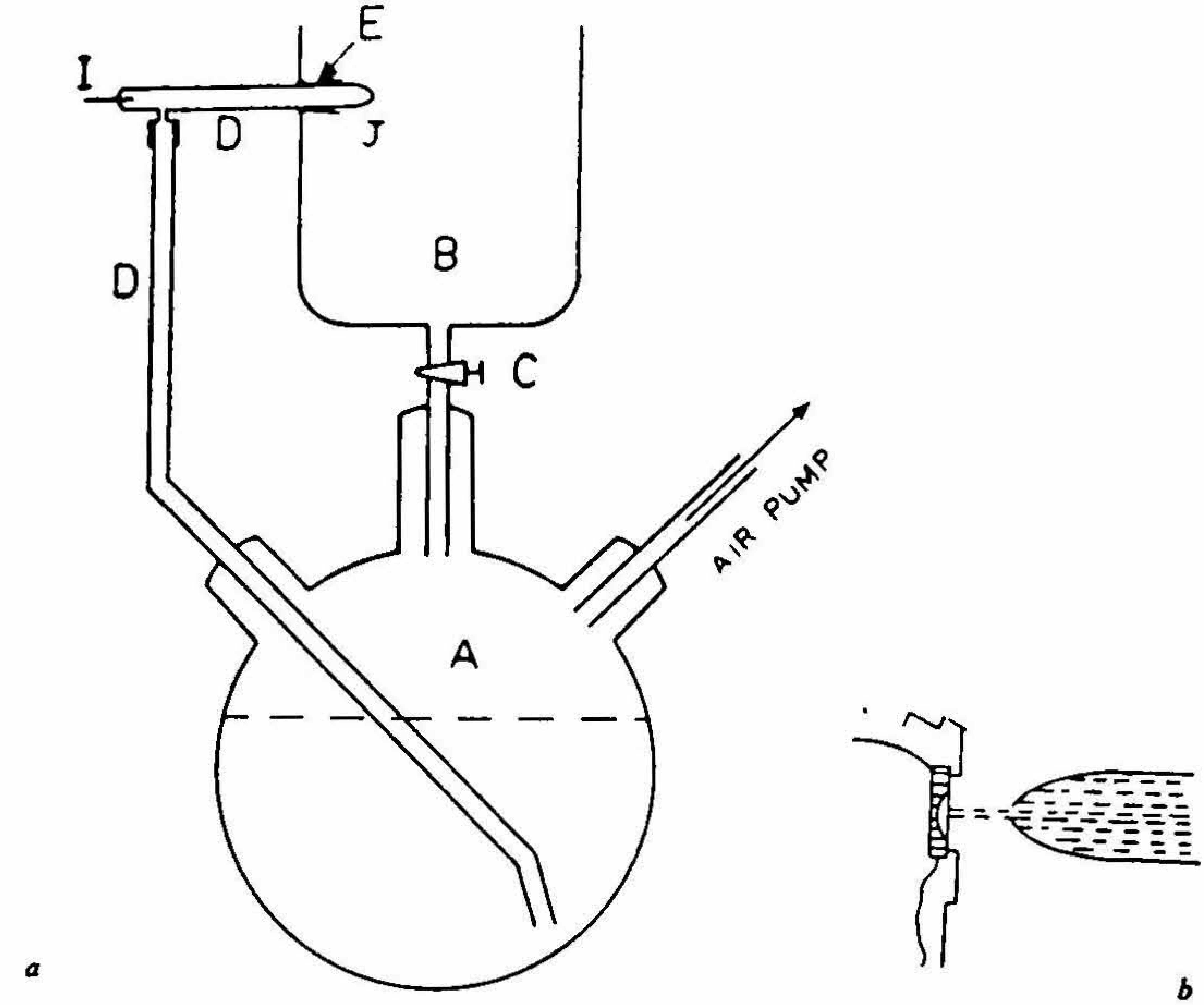


FIG. 7. (*a*) Schematic diagram of the single Jet polishing unit ; A—Round bottom flask ; B—Sink for collecting the electrolyte ; C—Stop-cock ; D—Glass tube ; E—Standard joint ; I—Platinum wire which acts as a cathode ; J—Jet. (*b*) Schematic representation of single jet operation.

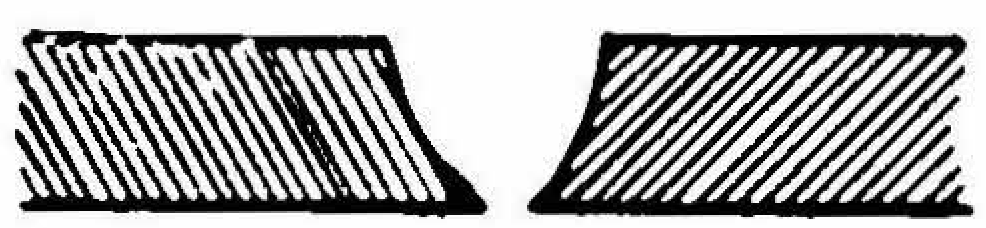


FIG. 8. Schematic representation of the results obtained with the jet electropolishing technique.

air in a glass flask half-filled with electrolyte. This electrolyte passes through a tube and, eventually, falls on the sample in the form of a jet. The temperature of the electrolyte, voltage across the electrodes, and diameter of the jet can be selected depending upon the requirements.

Several attempts were made on stainless steel substrates varying in thickness from 1 to 1·5 mm. The electrolyte was always maintained at a temperature range of 60° C to 65° C and 55 v to 65 v was applied between the electrodes. Jets of diameter 1·5, 1·0 and 0·5 mm were used. A typical result is shown schematically in fig. 8. It is seen from the figure that the tapering does not continue up to the tip of the pinhole.

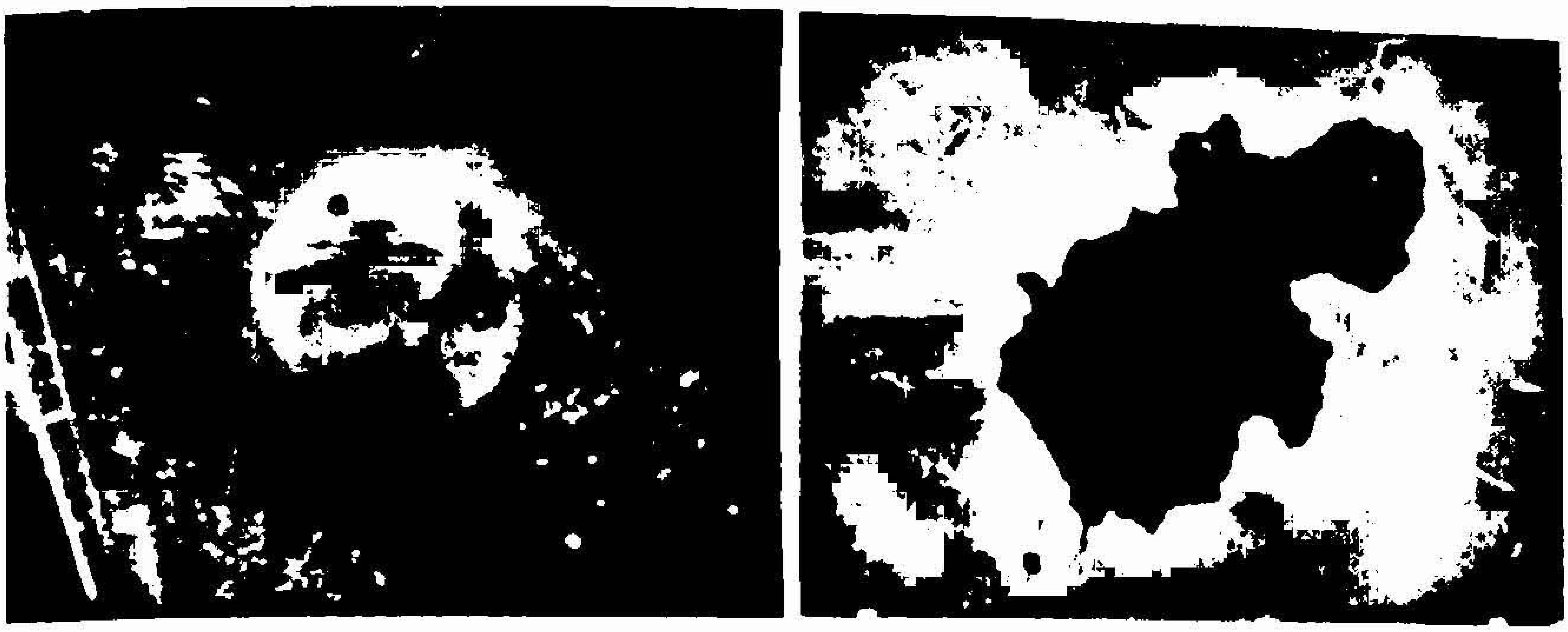


FIG. 9. A sample of the result obtained with the jet electroplishing technique.

However, jets of smaller diameter have been found to yield better results. An exceptionally smooth surface is a remarkable feature of this process (fig. 9). It is seen in this photograph that in addition to the desired hole (shown by an arrow mark) of approx. 4 microns, a big hole of ~ 110 microns is also formed. This has resulted from the washing away of about four grains of the substrate.

In its simple form, this technique did not yield consistent results. One of the crucial requirements is that the power supply should be switched off exactly at the moment the hole starts forming. Depedence on the visual perception, as was done in the present case, is not reliable. Instead, a photo-detector or a small evacuated chamber in conjunction with a senistive current meter fixed behind the sample holder should improve the consistency considerably.

3. Conclusion

We believe that with more trials and more precise controls, techniques (3) and (4) should yield holes with the desired characteristics.

Acknowledgements

We wish to acknowledge the help of Dr. G. Venkataraman and Mr. R. V. Nandedkar of the Materials Science Laboratory, Reactor Research Centre, Kalpakkam, for providing laboratory facilities for part of this work. This work was supported by grants to HSC and MAV from the Department of Science and Technology, Government of India. One of us (SKJ) thanks the same agency for the award of a Research Associateship.

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