MASTER

PREPARATION OF SCHOTTKY DIODES FOR EBIC INVESTIGATION OF GRAIN BOUNDARY PASSIVATION IN Si RIBBONS.

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ABSTRACT:

The preparation of Schottky Diodes for repeated EBIC investigations of the same area is described. Application of the technique to the study of grain-boundary passivation with atomic hydrogen shows that the degree of passivation varies strongly from boundary to boundary.

1) Introduction

The electrical activity of defects in Si is easily studied in the scanning electron microscope (SEM) with the aid of electron beam induced current microscopy (EBIC). Minority carriers generated by the incident electron beam are collected by a p-n or by a Schottky junction. The collected current, after amplification by a fast current amplifier, is used to modulate the video signal of the SEM. The resulting image represents the collecting efficiency for minority carriers under electron illumination. The resolution is about equal to the size of the generation volume, which depends on the incident energy.[1] In the usual operating range $10<V<40$ KeV, the resolution is in the μm range.

In this note, we address the reproducibility with which Schottky diodes can be prepared on p-type EFG silicon. Reproducibility becomes important if one wants to change the electrical activity of defects such as grain boundaries by treatments such as preferential diffusion along grain boundaries, passivation with atomic hydrogen[2,3] etc. In general, such treatments require the removal of the Schottky diodes in order to gain access to the semi-conductor surface. After the treatment, a new Schottky diode must be prepared. In order to correlate changes in the EBIC images "before and after" unambiguously with the treatment, the properties of this new diode must be identical to that of the original diode, and the removal and re-deposition must not alter the electrical activity of the defects under study. One way to check for this, is to take a Si sample containing electically active defects and to deposit and remove Schottky diodes without any in between treatment. If the deposition and removal process are without influence, identical EBIC images will result. In this report we describe a suitable preparation method and give an example of this technique to study selective grain boundary passivation by atomic hydrogen.

2) Properties of Schottky Diodes

The measured barrier heights on p type Si, as listed by Hovel[4] are:

Metal	Barrier Height ϕ_b [volts]
Au	0.35
Ag	0.55
Al	0.58
Cu	0.46
Ni	0.51
Pb	0.56

indicating that of the metals listed, Al has the highest barrier height. In addition Al is easy to deposit, relatively transparent to electrons* (and indeed used as Lenard windows material), and stable over long periods of time. Most of our investigations were therefore carried out on Al Schottky diodes. In addition, we fabricated Schottky diodes with Au, Pt, Pb and Ti as barrier material using both evaporation and sputtering as deposition methods. In our experience, sputtering does not generate better diodes (for EBIC purposes). Since it is a more energetic process and therefore more likely to change the structure of the surface we now use evaporation exclusively with either Al or Ti as a barrier material. In our experience Ti gives marginally better EBIC pictures (presumably because of its good adherence to Si) but is more difficult to evaporate in a controlled manner than Al. Consequently, it is more difficult to achieve a consistent thickness of the metal layer which of course, is one of the requirements for good reproducibility. Ti is also less transparent to electrons than Al, where one can tolerate somewhat thicker layers, which are easier reproduced. For this reason, we prefer Al for repeated preparation of Schottky diodes on one and the same sample.

*Some investigators use Al films as thick as 5000 Å with an SEM operating between lo and 25 KV (see ref.5).

We find that our Al Schottky diodes are stable and do not change properties with time. Our experience is similar to that of Bell and Freedman[6] on somewhat differently prepared evaporated Al Schottky diodes used for spectral response curve measurements. These authors comment on excellent electronic stability, high signal levels and faithful data reproducibility of their devices, some of which were used as calibration standards for over one year.

3) Preparation of Al Schottky Diodes

The p type EFG Si ribbons prepared by Mobil Tyco have a slightly wavy surface and are usually covered with a thin layer of SiC. As a first step, the ribbons were mechanically polished until flat. This polish was followed by a Syton polish in order to eliminate the damage introduced by the mechanical polishing.

Next, the specimen went through a cleaning cycle consisting of:

a) Boil in HNO_3 for 10 minutes. Rinse with deionized water.

b) Immerse in H_2O: NH_4OH: H_2O_2 (7:2:1) for 5 min.

c) Rinse with deionized water

After cleaning, the specimen were ready for evaporation. Immediately before evaporation, i.e. before loading into the evaporator, the specimen were given a final 30 sec etch in 48% Hf and rinsed with methanol. Then, 200 Å of Al were evaporated onto the Si.

Contacts to the diode were made as follows: The back surface of the wafer was sanded with 400 grid SiC paper and glued with carbon paint onto the Al SEM holder. Contact to the Al top electrode was made with a fine spring steel wire bent to a smooth curve (see Fig. 1.) This arrangement was then loaded into the SEM. The signal from the diode was amplified via a fast Keithly 427 current amplifier and fed into the video circuit of the SEM.

After EBIC, the Si is easily detached from the SEM stub by ultrasonic cleaning in acetone. The cleaning cycle, which also removes the Al film, is then

The above preparation method is similar to that of Bell and Freedman who describe their proceedure as follows : "The as-grown Si samples are cleaned in organic solvents, washed in deionized water, and given a light Silicon acid etch($HF:HNO_3::1:4$). Quenched in deionized water, the samples are once again rinsed thoroughly, and dried. An evaporated Al film of 500 Å thickness is applied to one side of the sample. This is sintered at 575°C for 15 minutes in a forming gas ($H_2:N_2::5\%:95\%$) ambient to create an ohmic contact. A transparent (125 Å) Al Schottky barrier is next vacuum deposited on the opposite surface. Finally, a strip of ultrasonically-soldered indium is applied to the ohmic (sintered) contact to disperse any oxides of Al, thus enhancing contact of the sample to the sample holder."

The two proceedures differ mainly in the fabrication of the back side contact, which is less permanent (and therefore more easily removed) in our procedure. Ease of removal was not a consideration in B & F process.

4) Results.

The reproducibility of the technique was tested by removing and applying Schottky diodes (without any treatment to change the electrical activity) on EFG ribbon material fabricated by Mobil Tyco. Up to five cycles were investigated. No noticeable changes in the EBIC images were observed. Fig. 2 shows an EBIC image after the first deposition. Fig. 3 shows an enlarged section demonstrating the good images that can be obtained with Al metallization. Fig. 4 shows the same are as Fig. 2, but in this case the EBIC image was obtained with the 3rd diode evaporated. A comparison of Fig. 2 and 4 shows that no noticeable changes have occured in the EBIC images. A quantitative measurement of the ratio of injected to collected current (in relative defect free areas) collaborates this impression.

Fig. 5 and 6 give an example of the application of this technique to the selective passivation of planar defects. The two figures show the same area before and after treatment with atomic hydrogen of 550°C. Notice that the line contrast indicated by an arrow in Fig. 5 disappears to a large degree after annealing in atomic hydrogen, and becomes comparable to the faint EBIC contrast of neighboring planar faults. Other strong contrasts appear unchanged. The reason for the selective passivation are presently not known. A TEM investigation of the structure of "passivatable" and "non-passivatable" defects is currently under way in order to clarify the relation between structure and the degree of hydrogen passivation that can be achieved.

Another observation after hydrogen passivation is that the ratio of injected to collected current (in relative defect free areas) tends to increase. Quantitative measurements of this and other effects are being carried out as a function of annealing parameters to obtain a better understanding of the hydrogen passivation effect.

5. Summary

A process to deposit and remove Schottky diodes reproducibly, and with no changes in the EBIC images of electrically active defects has been developed. Application of the technique to the study of hydrogen passivation of planar defects in EFG solar Si ribbons shows that the degree of passivation varies strongly from boundary to boundary.

References

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4) H.J. Hovel, in "SEMICONDUCTORS AND SEMIMETALS, VOLUME II, SOLAR CELLS", Academic Press (1975) New York, San Francisco, London; p. 121.

5) S. Kawado, Y. Hayafuji a. T. Adachi, "Observation of Lattice Defects in Silicon by Scanning Electron Beam Microscopy Utilizing Beam Induced Current Generated in Schottky Barriers", Japan J. Appl. Phys. 14 (1975) p. 407.

6) R.O. Bell and G.M. Freedman, "Minority Carrier Diffusion Length From Spectral Response Measurements" 13th IEEE Photovoltaic Specialists Conference - 1978, June 5-8, Washington, D.C., (78CH1319-3), p. 89.

Figure Captions

Fig. 1 Schematic view of the EBIC holder.

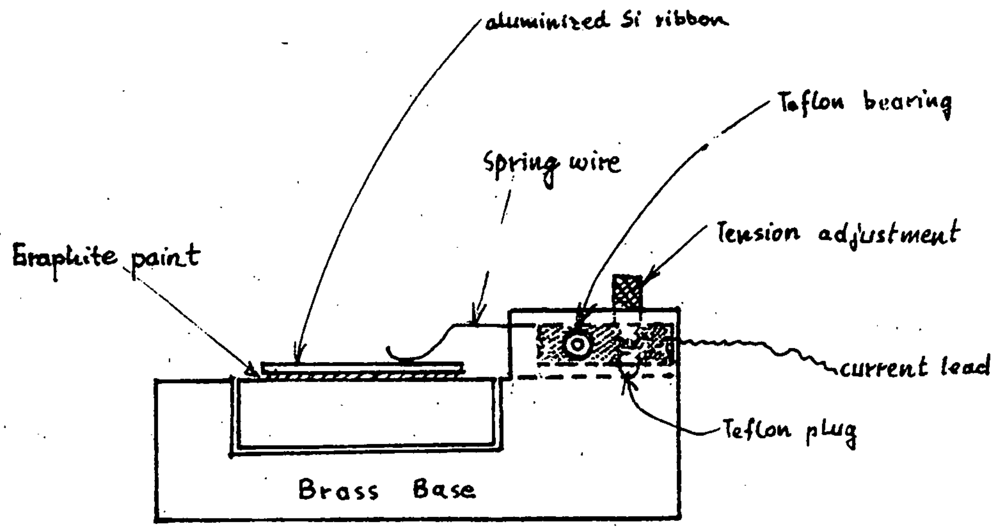
Fig. 2 EBIC image of EFG Si, Magnification x250, taken after the first metallization with Al. Part of dashed area is shown enlarged in Fig. 3.

Fig. 3 Section of Fig. 2, x 1000.

Fig. 4 EBIC image of the same area as Fig. 2, taken after the third metallization with Al.

Fig. 5 EBIC image of EFG Si, Magnification x 100, taken after the first metallization. Note relatively strong contrast of linear defect indicated by arrow.

Fig. 6 EBIC image of the same area as Fig. 5, taken after hydrogen passivation in a glow discharge at 550°. The arrow indicates the linear defect of Fig. 5. Note that the contrast is reduced.


aluminized Si ribbon
Teflon bearing
Spring wire
Tension adjustment
Graphite paint
current lead
Teflon plug
Brass Base

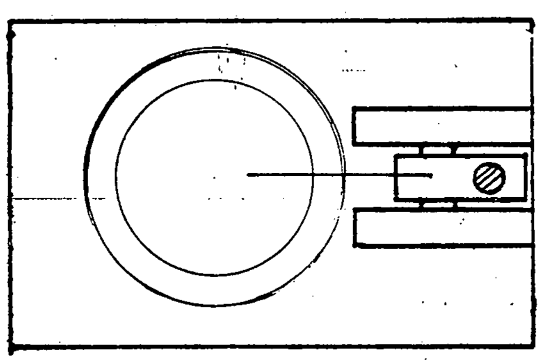
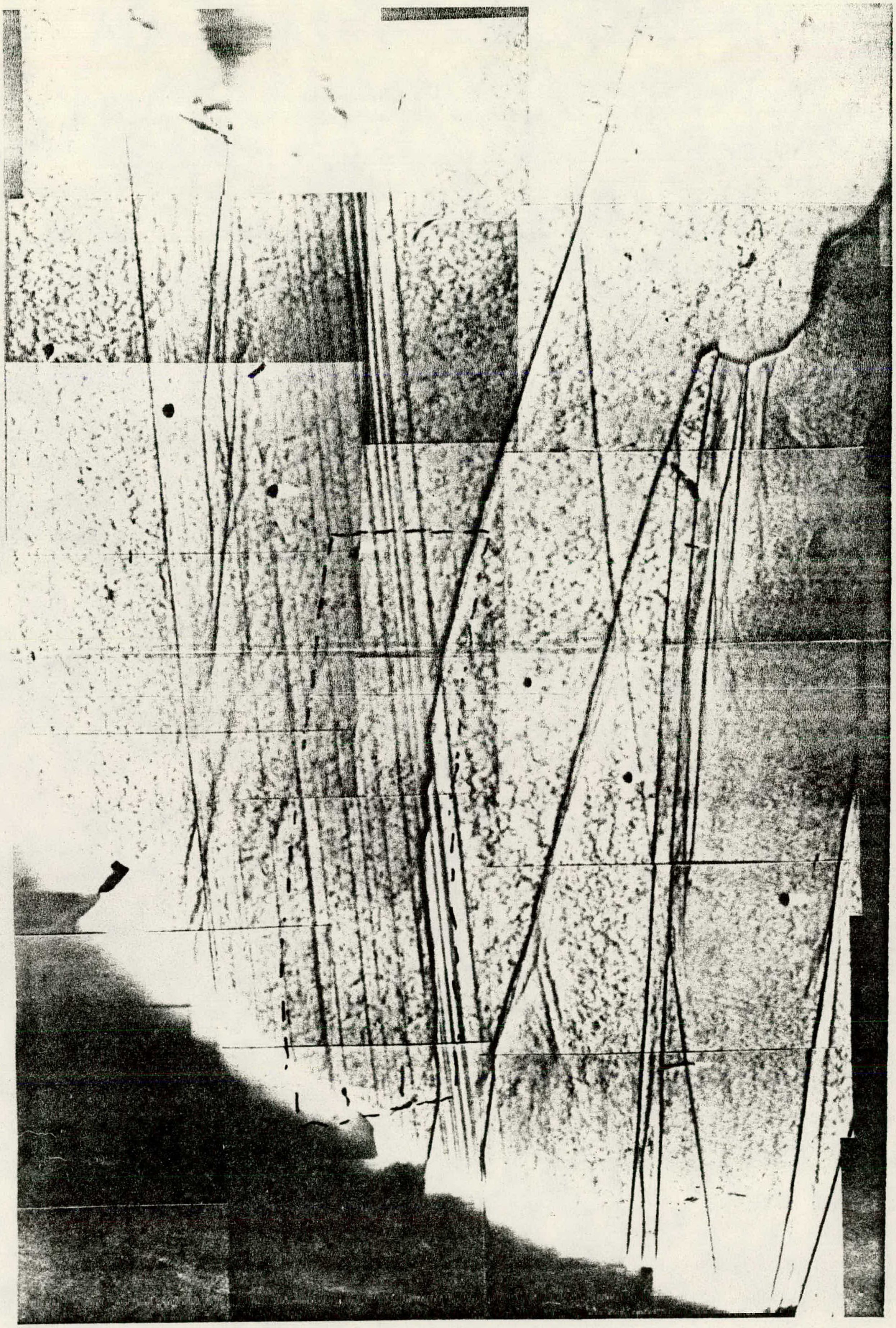


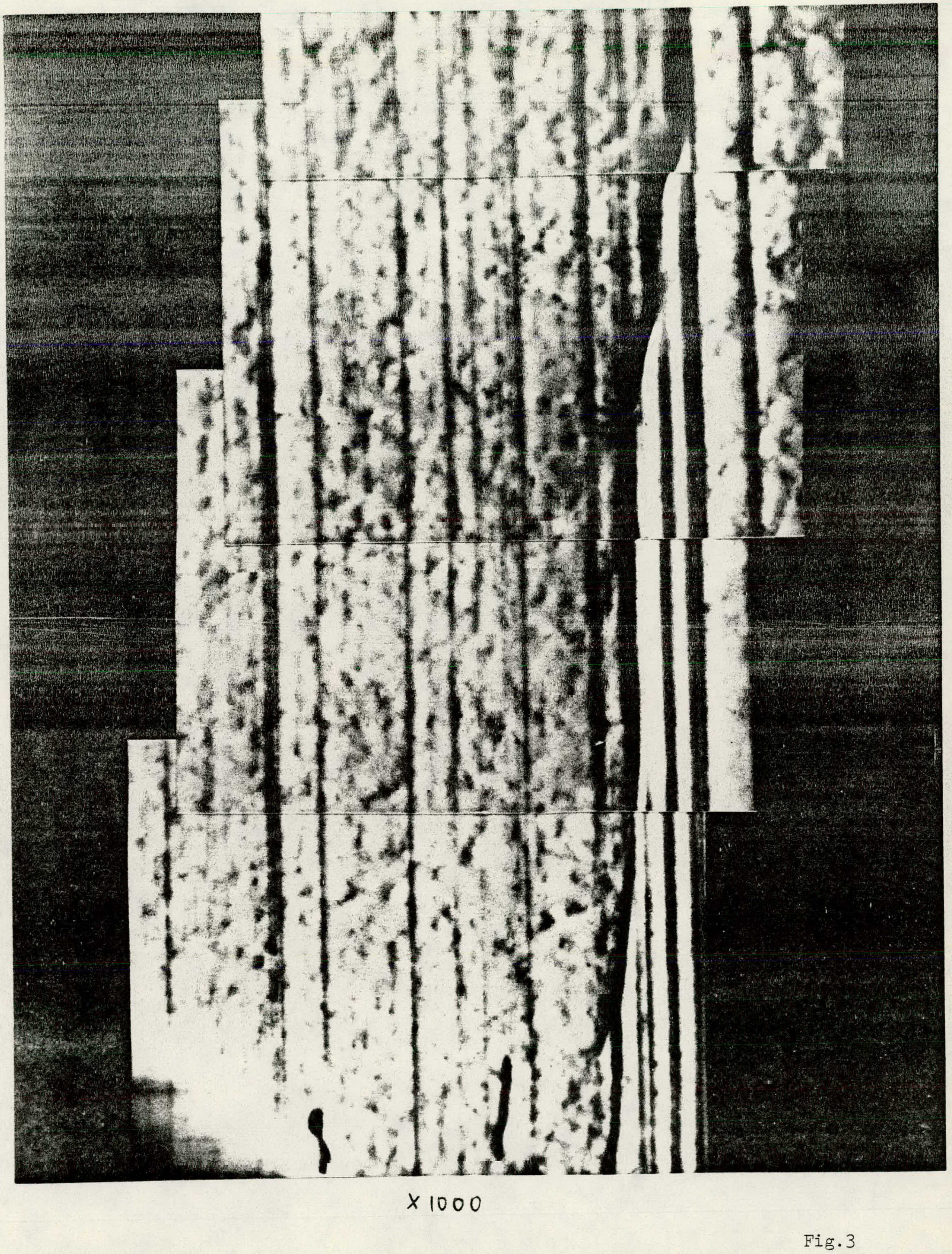
Fig. 1



x 250

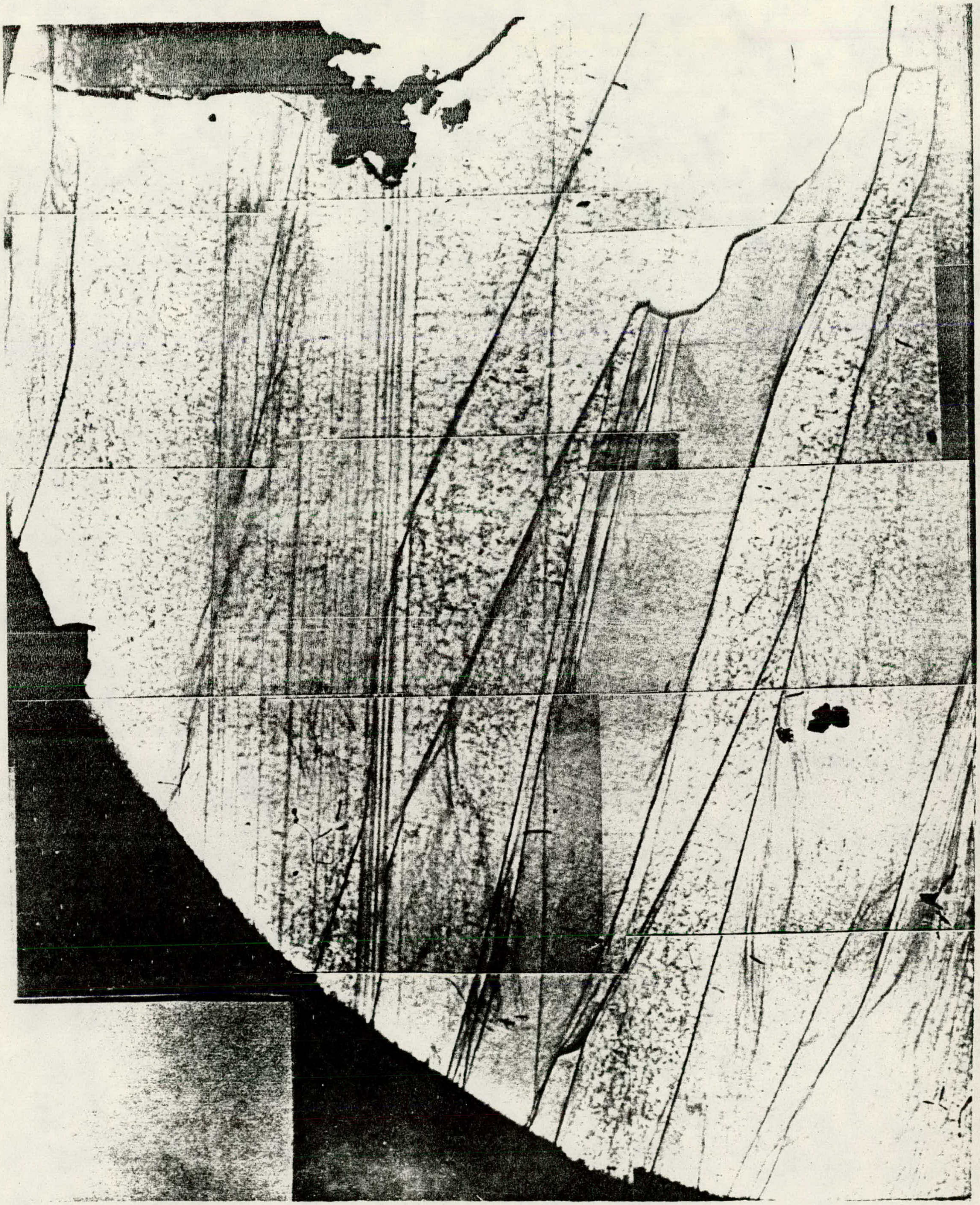
2

Fig. 2



X 1000

Fig.3



x 250

Fig. 4

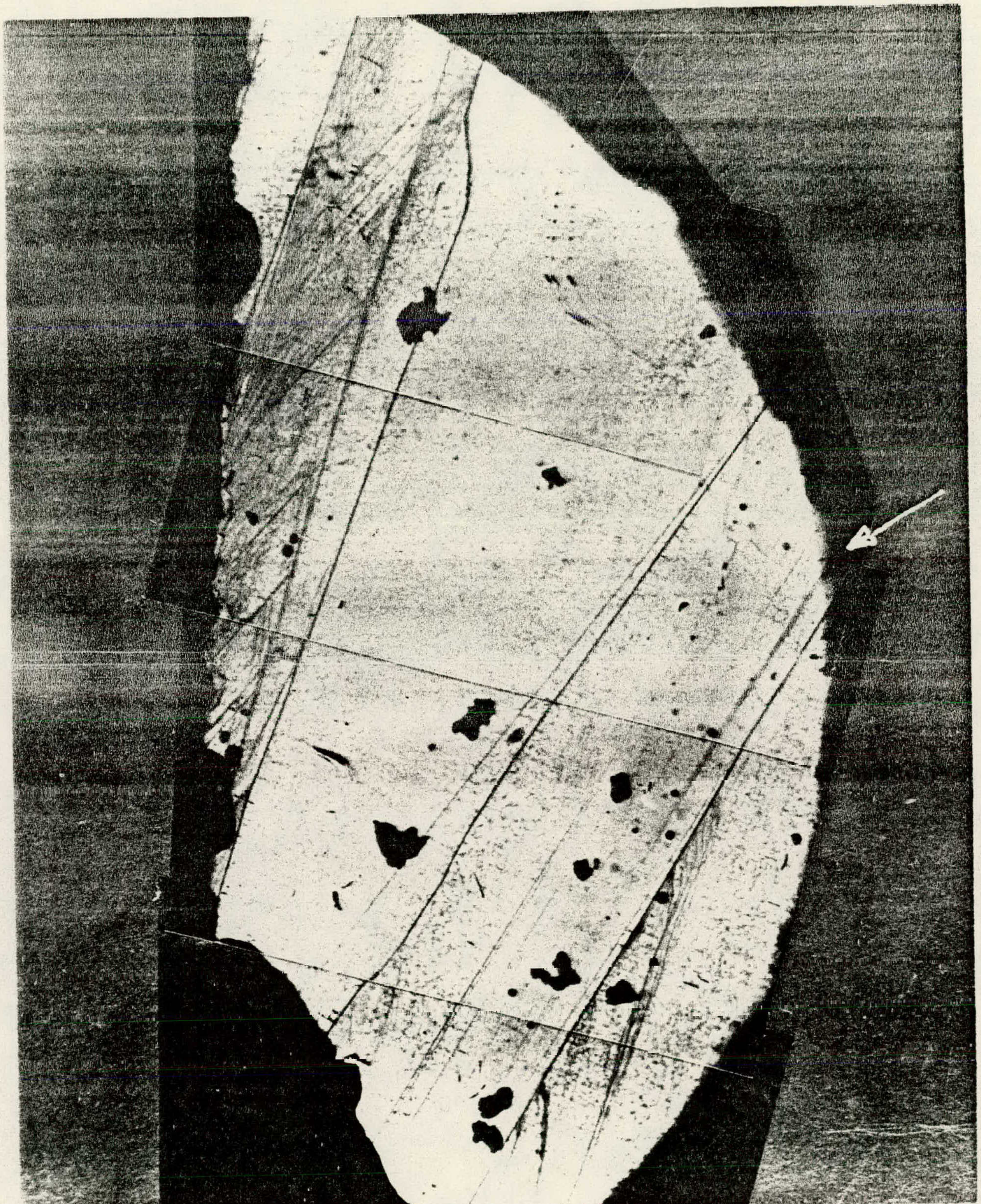


Fig.5

x 100

Fig 6

Fig.6