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DISLOCATION-LOOPS IN NATURAL GRAPHITE SINGLE CRYSTALS REACTOR-IRRADIATED AT 1 200° C

by

M. HEERSCHAP

1967

Joint Nuclear Research Center
Petten Establishment - Netherlands

Structure Department

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Brussels, March 1967

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In graphite single crystals irradiated at 1 200° C and subsequently annealed at 2 200° C separate interstitial and vacancy loops have been found. The vacancy loops are all sheared. The interstitial loops are unsheared except in some cases where they are sheared probably by the passage of a dislocation ribbon. Partial dislocations pinned by sheared interstitial loops have been observed.

In some instances the loops have grown preferentially along dislocations or grain-boundaries.

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SUMMARY

Interstitial loops of a diameter of about 1 000 Å decorated with small vacancy loops of a diameter of about 100 Å have been observed in graphite single crystals irradiated at 1 200° C and are discussed. These loops are unsheared.

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In some instances the loops have grown preferentially along dislocations or grain-boundaries.

CONTENTS

DISLOCATION-LOOPS IN NATURAL GRAPHITE SINGLE CRYSTALS REACTOR-IRRADIATED AT 1200°C.

1 - INTRODUCTION (*)

Dislocation-loops in natural graphite single crystals irradiated at 1200°C and subsequently annealed at 2000°C have been studied by Thrower [1]. Thrower found interstitial loops and smaller vacancy loops, which had grown in the strain-field of the interstitial loops.

As the determination of the nature of dislocation-loops may lead easily to controversial results it is of interest to publish our results on the study of irradiation defects in graphite single crystals. Dislocation-loops in graphite, which has been irradiated at 1200°C in HFR at Petten, the Netherlands, and which has been annealed at 2200°C, are studied and discussed.

2 - EXPERIMENTAL PROCEDURE

The graphite single crystals originated from Essex County, New York State. They were purified first by alternate washing with hydrochloric and hydrofluoric acids and then by heating in a vacuum at 2700°C. The purified crystals have been irradiated in HFR at 600, 900 and 1200°C in a Helium atmosphere at a dose of $4,5 \times 10^{20} n.cm^{-2}$. Annealing has been performed in a graphite crucible, which was heated in a high-frequency furnace at 2200°C. After cleavage the specimen have been observed at 100 kV in a Hitachi HU-11a provided with a 30° tilting-stage and the Philips EM-300 **).

(*) Manuscrpt received on January 27, 1967.

(**)I am grateful to the direction of the application laboratory of Philips, Eindhoven, for offering me the possibility to use the Philips EM-300.

3 - RESULTS

Bright-field images of dislocation-loops observed in graphite single crystals irradiated at 1200°C are presented in the figures 1a and 1b. In fig. 1a the image, which is in line contrast, is obtained in $\{11\bar{2}.2\}$ reflection, whereas the image in fig. 1b, which is in stacking-fault contrast, is obtained in $\{10\bar{1}.1\}$ reflection. In reflections however for which $\ell = 0$ the loops are out of contrast. Two distinct populations are clearly visible namely loops of a diamter of about 1000 Å and smaller loops with a diameter of about 100 Å. The smaller loops are nucleated along the circumference of the big loops.

In order to simplify the analysis of the loops the crystals have been heated for half an hour in a vacuum at 2200°C. Whether the loops in these irradiated and annealed specimen are vacancy or interstitial loops have been determined by the $(\bar{g}.\bar{b}.)s$ method as is demonstrated in the figures 2a and 2b. In these figures the bright-field images in $\{11\bar{2}.2\}$ reflection with opposite interference errors and the corresponding diffraction patterns are presented. In fig. 2a the image of the loops, indicated by the letter i, is inside, that of the loops, indicated by the letter v, outside the actual loop. The interference error "s" is positive. In fig. 2b however the image of the loops "i" is outside, that of the loops "v" inside the actual loop. The interference error "s" is negative. The tilt-axis is from the right to the left. Taking the position of the specimen with respect to the horizontal position, indicated by the expressions "up" and "down" and the direction to the operating diffraction vector into account we arrive at the conclusion that the loops "i" are interstitial and the loops "v" vacancy loops.
A dark-field determination of the nature of these loops is presented in the figures 3a and 3b. The images in these figures are obtained in a reflection for which $\ell = 3$.

In the figures 4a, 4b and 4c the images of the same loops are presented in three different reflections namely in fig. 4b in $\{11\bar{2}.2\}$ reflection, in fig. 4c in $\{10\bar{1}.1\}$ reflection and in fig. 4a in a reflection for which $\ell = 0$. All vacancy loops are visible in these reflections. Some of them are indicated by the letter v.

Interstitial loops on the other hand are invisible in reflection for which $\ell = 0$, except two, which are indicated by the letters a and b. At the sheared interstitial loop "a" a partial dislocation of a dislocation ribbon is pinned. The partial is out of contrast whereas the loop and the other partial are in contrast. In fig. 5 a dark-field image in $\{11\bar{2}.2\}$ reflection of a pinned partial and a sheared interstitial loop is presented. The loop and the pinned partial are in contrast, the other partial is out of contrast. The shear vector of the loop is perpendicular to the total Burgers vector of the dislocation ribbon.

The loops in irradiated and annealed specimen are found generally in small groups, the bigger loops being interstitial, the smaller ones being vacancy loops. The groups are randomly distributed except in some instances where they have nucleated preferentially along dislocations or grain-boundaries as is seen in fig. 6.

4 - DISCUSSIONS

Two distinct types of loops have been found in irradiated single crystals, namely loops of a diameter of about 1000 Å and of a diamter of about 100 Å. Both types are invisible in reflections for which $\ell = 0$ that will say are unsheared. That the big loops are interstitial loops may be concluded from a suggestion of Baker [3], based on his annealing experiments, that vacancy loops begin to shear at a diameter of 100 - 200 Å. Therefore the big loops having a diameter of about 1000 Å and being unsheared must be interstitial loops.

The conclusion that the big loops are interstitial and the smaller ones vacancy loops has been drawn further from the presence of interstitial and vacancy loops in irradiated and annealed graphite as has been determined by the $(\bar{g}.\bar{b}.)s$ method. These loops namely are found in small groups, the bigger loops generally being of interstitial character, the smaller ones being vacancy loops.

These results are in agreement with the findings of Thrower [1]. However Thrower's observations that interstitial and vacancy loops are present already in crystals irradiated at 600°C is not clear in our experiments.

In irradiated and annealed graphite the vacancy loops, which have a diamter of 200 - 500 Å, have been found to be all sheared. The interstitial loops however are unsheared as may be expected from Throwers calculations [2] , except in some cases where they have sheared. In some instances partial dislocations have been observed to be pinned by such a sheared interstitial loop. In such a case the shear-vector of the loop is perpendicular to the Burgers vector of the pinned partial. Thrower [2] has suggested and discussed that the shear of interstitial loops is caused by the passage of a dislocation ribbon.

Whether dislocation-loops grow consequently by preference along dislocations or grain-boundaries cannot be said with certainty as a high number of dislocations is introduced upon cleavage after irradiation and annealing.

5 - ACKNOWLEDGEMENTS

I thank Drs. R. Blackstone for irradiating the graphite and Mr. I. van Gerwen and Mrs. L. Agace for preparing the specimen.

CAPTIONS FOR FIGURES

Fig. 1 - Dislocation-loops in graphite single crystals irradiated at 1200°C.

a. Bright-field image in $\{11\bar{2}.2\}$ reflection.

b. Bright-field image in $\{10\bar{1}.1\}$ reflection.

Fig. 2 - Determination of the loop character of dislocation-loops in graphite single crystals irradiated at 1200°C and subsequently annealed at 2200°C.

a. Bright-field image in $\{11\bar{2}.2\}$ reflection and the corresponding diffraction pattern.
The image of the loops "i" is inside, that of the loops "v" outside. Interference error positive.

b. Bright-field image in $\{11\bar{2}.2\}$ reflection and the corresponding diffraction pattern.
Image of loops "i" outside, that of loops "v" inside. Interference error negative.

Tilt-axis is from the right to the left. The position of the specimen is indicated by the expressions "up" and "down".
The loops "i" are interstitial, the loops "v" vacancy loops.

Fig. 3 - Determination of the loop character of dislocation-loops in graphite single crystals irradiated at 1200°C and subsequently annealed at 2200°C by means of dark-field electron-microscopy.

a. Dark-field image in $\{11\bar{2}.3\}$ reflection and the corresponding diffraction pattern.
Image outside except that of loop "v", which is inside. Interference error positive.

b. Dark-field image in $\{11\bar{2}.3\}$ reflection and the corresponding diffraction pattern.

Image inside except that of loop "v", which is outside. Interference error negative.

The tilt-axis is from the right to the left. The position of the specimen is indicated by the expressions "up" and "down". The loops are mostly interstitial here; the loop "v" is a vacancy loop.

Fig. 4 - Dislocation-loops in graphite single crystals irradiated at 1200°C and subsequently annealed at 2200°C in three different reflections.

a. Bright-field image in a reflection for which $\ell = 0$. Vacancy loops are all in contrast; interstitial loops are invisible except the loops "a" and "b". A partial dislocation of a dislocation ribbon is pinned by the sheared interstitial loop a.

b. Bright-field image of the same loops in $\{11\bar{2}.2\}$ reflection.
The interstitial loops of which the image is inside as well as the vacancy loops of which the image is outside are all in contrast.

c. Bright-field image of the same loops in $\{10\bar{1}.1\}$ reflection. Interstitial as well as vacancy loops are all in contrast.

Fig. 5 - Partial dislocation pinned by a sheared interstitial loop. Dark-field image in $\{11\bar{2}.2\}$ reflection.

Fig. 6 - Preferential nucleation of dislocation-loops along dislocations and grain-boundaries in irradiated and annealed graphite.

R E F E R E N C E S

1 - Thrower, P.A., Brit. J. Appl. Phys., 15 (1964) 1153
AERE - R 5271 (1966)

2 - Thrower, P.A., AERE - R 5296 (1966)

3 - Baker, C., Ph.D.Thesis, University of Cambridge (1963)

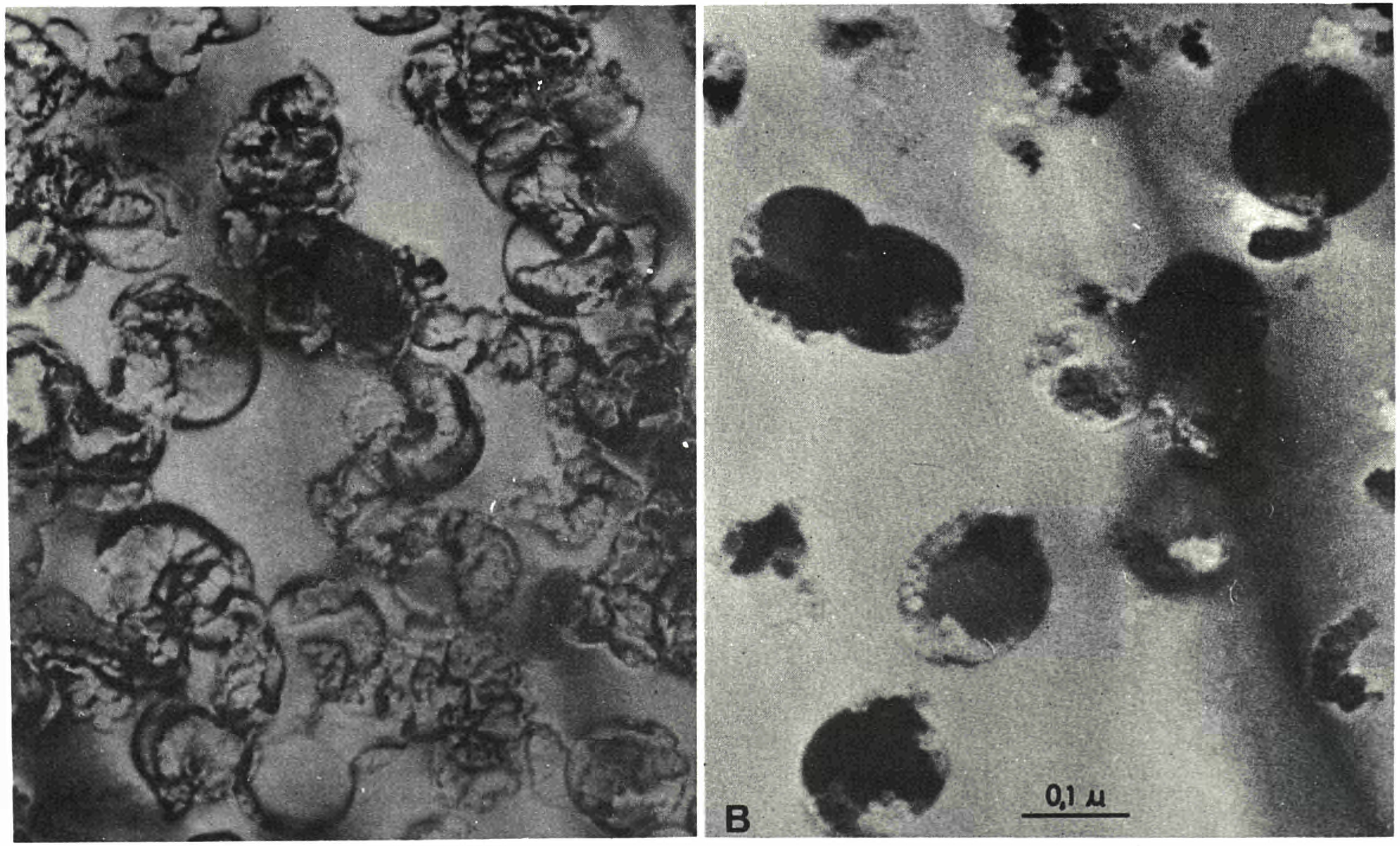


FIG 1

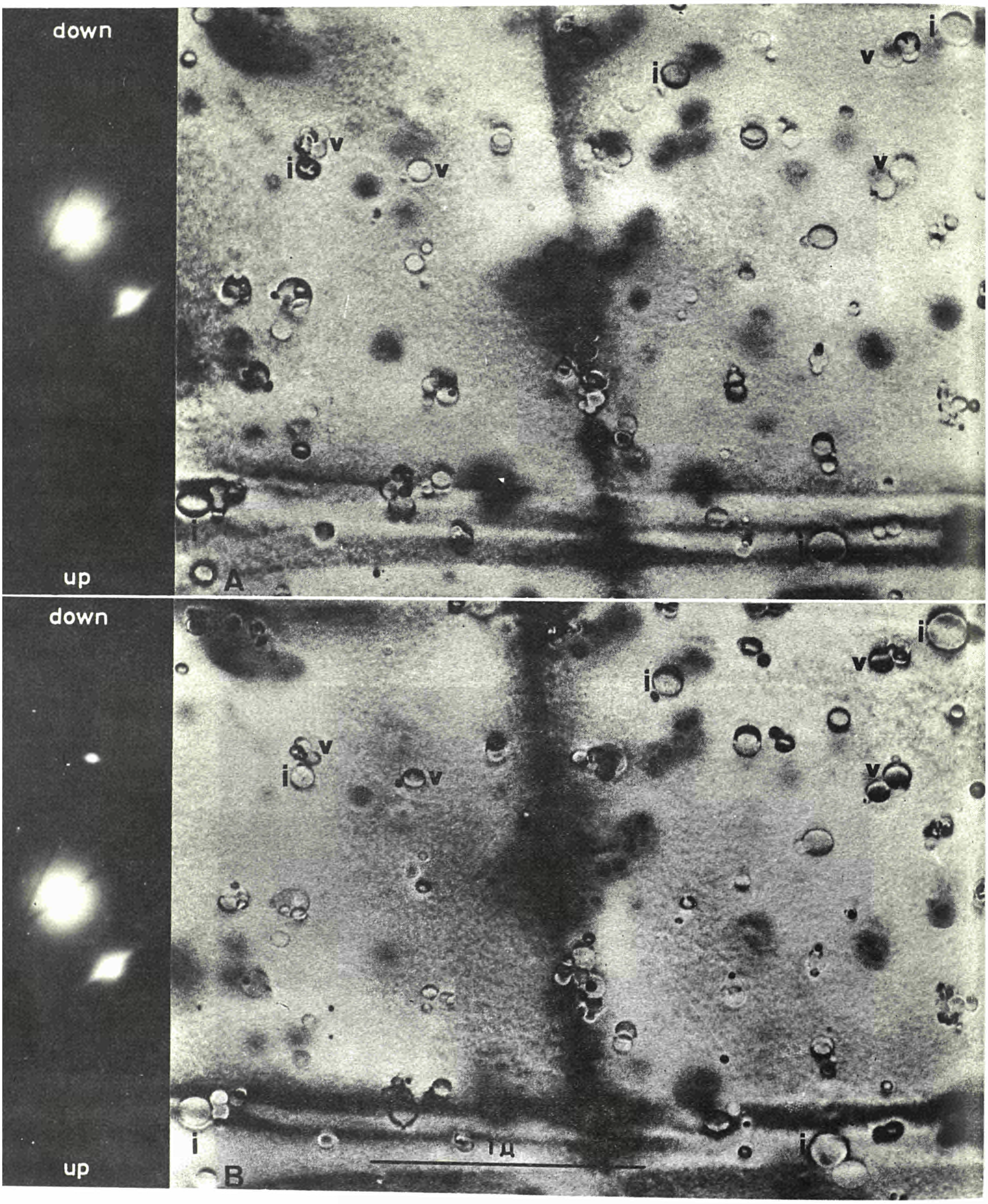


FIG 2

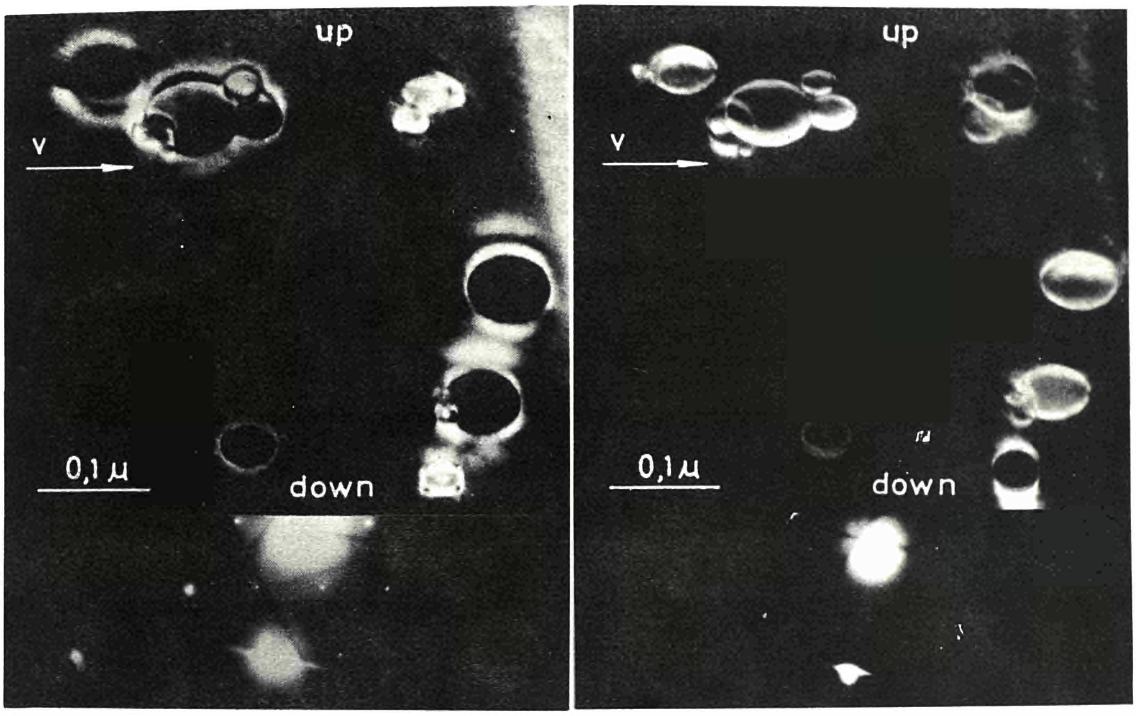


FIG 3

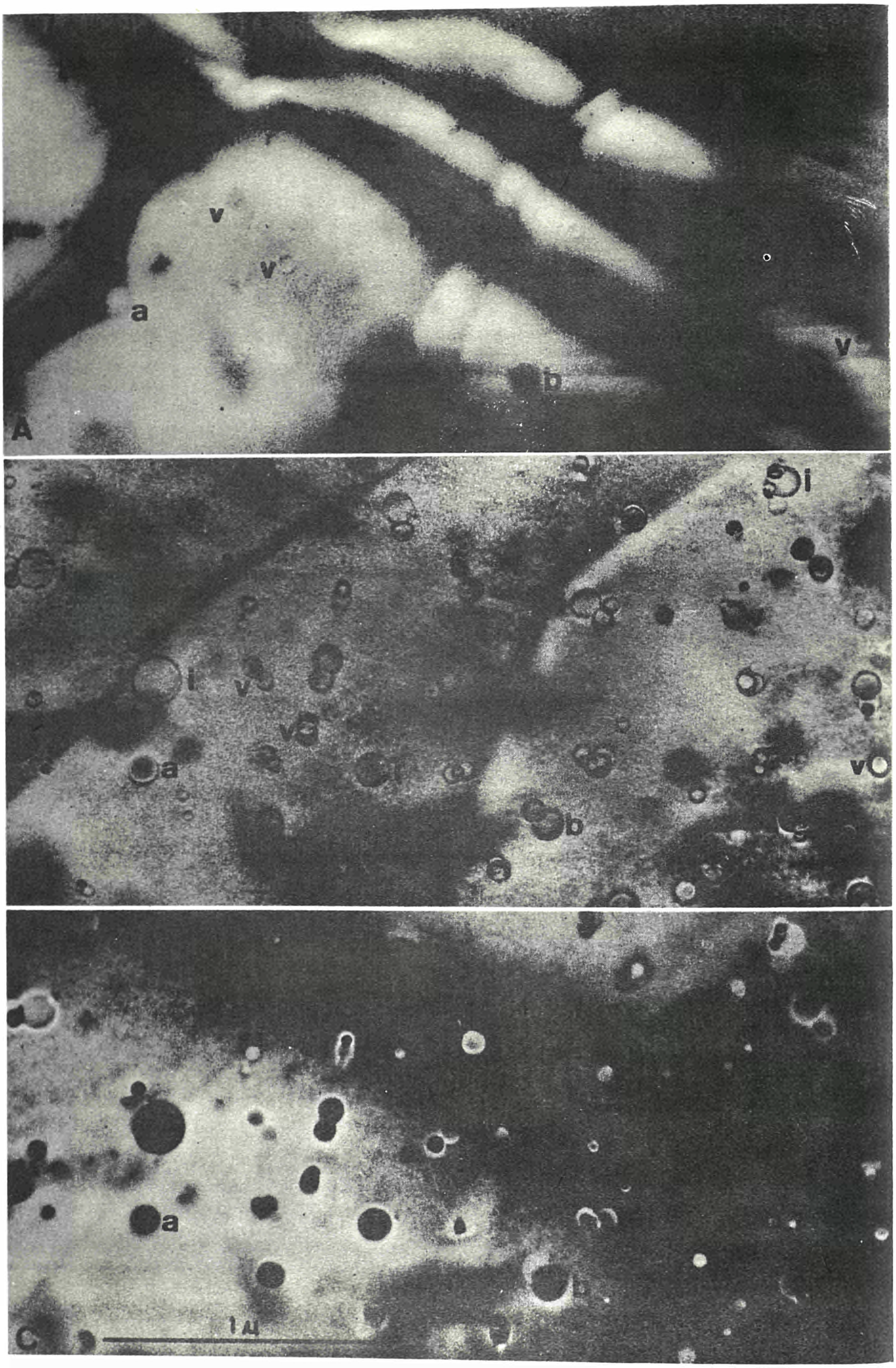
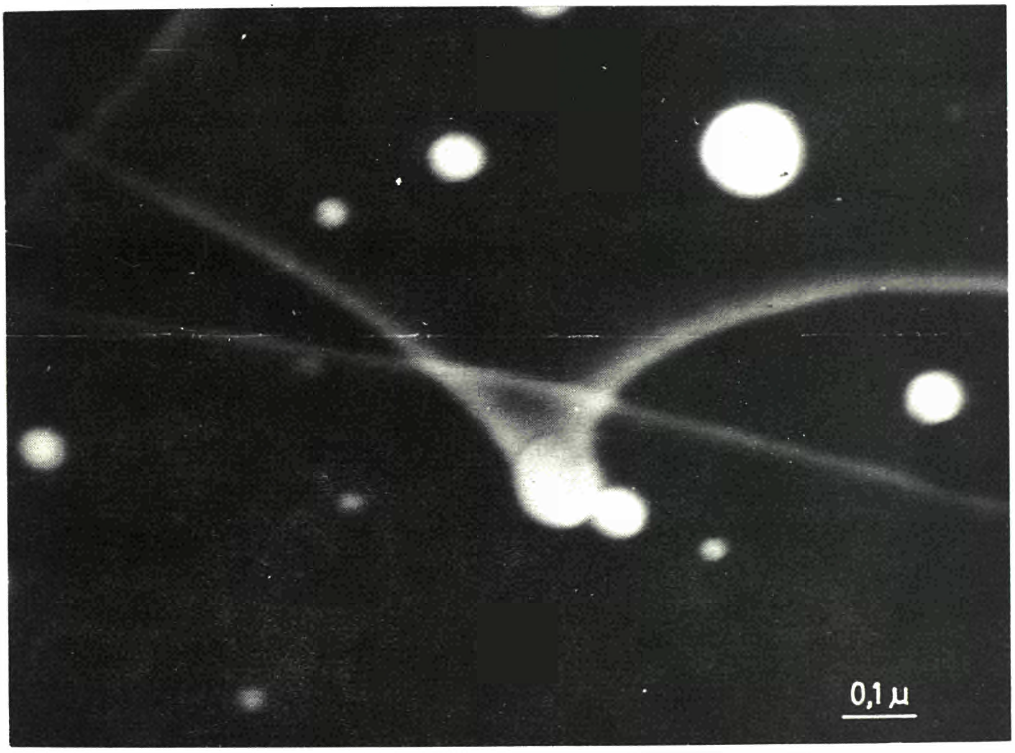


FIG 4



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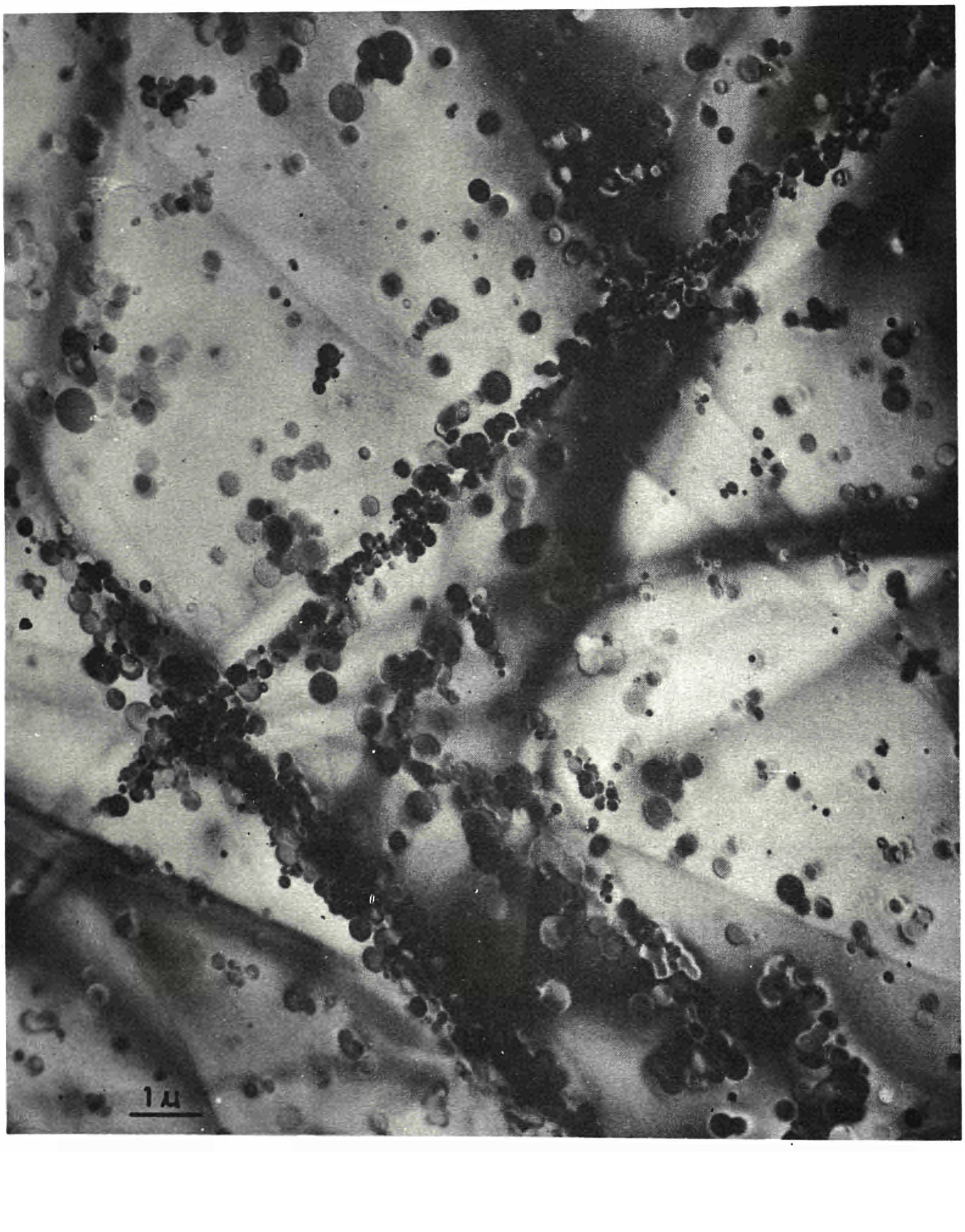

FIG 6

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To disseminate knowledge is to disseminate prosperity — I mean general prosperity and not individual riches — and with prosperity disappears the greater part of the evil which is our heritage from darker times.

Alfred Nobel

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