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HIGH VOLTAGE ELECTRON MICROSCOPY AND ELECTRON DIFFRACTION OF PYROXENES IN TYPE B LUNAR SAMPLES FROM APOLLO 11.

H. Fernández-Morán and M. Ohtsuki

ELECTRON MICROSCOPY LABORATORY
DEPARTMENT OF BIOPHYSICS
PRITZKER SCHOOL OF MEDICINE

and S. Hafner and D. Virgo

DEPARTMENT OF THE GEOPHYSICAL SCIENCES

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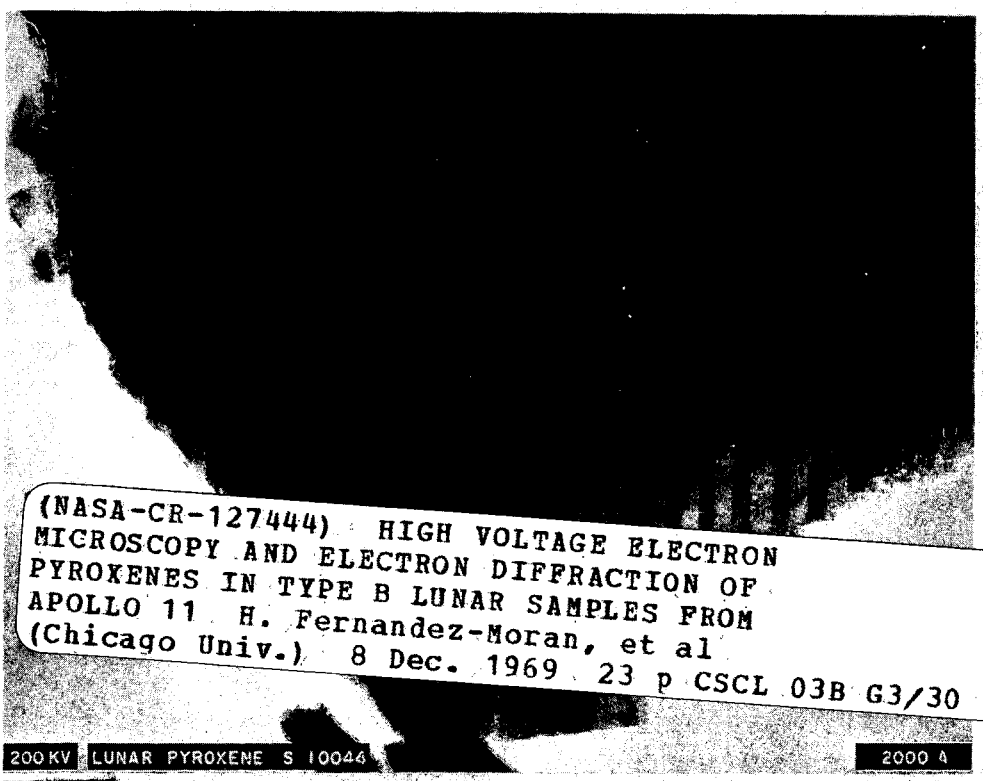

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HIGH VOLTAGE ELECTRON MICROSCOPY AND ELECTRON DIFFRACTION OF PYROXENES IN TYPE B LUNAR SAMPLES FROM APOLLO 11

Humberto Fernández-Morán and Mitsuo Ohtsuki
Electron Microscope Laboratory
Department of Biophysics
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Department of the Geophysical Sciences

The University of Chicago
Chicago, Illinois 60637

HIGH VOLTAGE ELECTRON MICROSCOPY AND ELECTRON DIFFRACTION OF PYROXENES IN TYPE B LUNAR SAMPLES FROM APOLLO 11

By: Humberto Fernández-Morán, Mitsuo Ohtsuki
Electron Microscope Laboratory, Department of Biophysics and
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ABSTRACT:

Lunar pyroxene 10044 specimens cleaved and sectioned by diamond knife ultramicrotomy were examined by standard (75 to 100 kV) and high voltage (200 kV) electron microscopy and diffraction.

Salient findings based on evaluation of 2000 plates show uniform 300 to 600Å-wide bands, probably corresponding to single crystal domains, with lattice spacings of 2.5Å. These dense bands, found predominantly in lunar pyroxene, are absent in terrestrial pyroxene XYZ. Lattice spacings of 6.5Å in lunar pyroxene and 18.2Å in pyroxene XYZ were directly visualized. High resolution bright and dark field images of iron-rich and magnesium-rich crystals were compared with corresponding electron diffraction patterns. Possible relations of observed structures to magnetic domains were considered.

Prepared and submitted: December 8, 1969.

Electron Microscopy of Lunar Pyroxenes

In view of the unusual variations of chemical composition within each crystal of lunar pyroxene (1) and of the well known distinct phases of exsolution phenomena observed in terrestrial and meteoritic pyroxenes (2,3,4), study of the fine structure of lunar pyroxenes as revealed by electron microscopy is essential.

Separated lunar pyroxene 10044 crystals cleaved and sectioned by diamond knife ultramicrotomy (5) and mounted directly on thin film specimen grids (without water or solvent contamination) were examined by both standard (75 to 100 kV) (6) and high voltage (200 kV) electron microscopy (fig. 1) and selected area electron diffraction techniques under conditions of higher penetration power, reduced radiation damage and negligible contamination (7) in a cryogenic vacuum (figs. 2,3,12).

Based on the examination of numerous representative samples and on the quantitative evaluation of 2000 plates, we can state the following observed characteristics:

1. Exceptionally regular, periodically spaced dense bands with uniform widths of 300 to 600Å (graphs). These straight-edged bands exhibit electron-optical phenomenon corresponding to single crystal domains (figs. 4,5,6,7,8,9,11), and they appear to be oriented with their long axis in the plane of the crystalline layers (approximately normal to crystallographic c).

By combined high resolution dark field electron microscopy and selected area electron diffraction (fig. 6), intrinsic lattice spacings of 2.5Å can be detected within the bands, arranged parallel to their long axis (i.e. normal to c).

Detailed studies show that these bands resemble electron-optical images of magnetic domain walls as seen in thin layers of ferromagnetic materials (8).

They are predominantly seen in iron-rich lunar pyroxene crystals, and the 2.5Å spacings could correspond to a dense population of the iron atoms at the M positions within the bands.

The single crystal band domains are absent in both terrestrial pyroxene XYZ (figs. 4,10) and in magnesium-rich lunar pyroxene 10044 specimens. The latter show instead irregular striations along the planes of the cleaved lamellae. Dense granules (ca. 100 to 1000Å in diameter) are also found in iron-rich pyroxene crystals (fig. 5).

2. Lattice spacings of 6.5Å in lunar pyroxene and 18.2Å in terrestrial pyroxene XYZ were directly visualized in high resolution bright and dark field images which could be compared with the corresponding

electron diffraction patterns (fig. 4).

The 18.2Å spacings may tentatively be correlated with the a axis of pyroxene XYZ, and the 6.5Å spacings may correspond to the M-M interatomic distances in the cleavage planes.

These results, which are being further analyzed, are of particular importance in determining the cationic order-disorder phenomena in these silicates (9). However, more work must be carried out to establish the precise correlation with the unit cell dimensions of lunar pyroxene crystals.

The significance of present results indicates the potential contribution of correlated electron-optical and crystallographical studies to a better understanding of the intrinsic atomic organization of pyroxenes and their possible bearing on the nature and evolution of the moon.

December 8, 1969
Condensed version to be submitted for publication in Science.

Electron Microscopy of Lunar Pyroxenes

Correlation with new observations made by S. Hafner and D. Virgo on the magnetic behavior of pyroxenes in type B lunar samples as revealed by nuclear gamma ray resonance (NGR) studies.

Dr. Stefan Hafner and Dr. David Virgo (1) have independently made Mössbauer resonant absorption studies of ^{57}Fe in the same lunar pyroxene type B specimen 10044 and have demonstrated that the crystal structure in these specimens is ferrimagnetically ordered.

The lunar pyroxene crystals exhibit a sharp Curie point in the range of 10° to $20^{\circ}K$. The spin orientations in this lunar pyroxene are assumed to be ferrimagnetic.

This result is unusual. Chain silicate crystal structures are generally not magnetically ordered, even at very low temperatures (i.e. $1.7^{\circ}K$), particularly when the amount of diamagnetic cations (Mg, Ca, etc.) substituting for Fe is larger than 25 per cent as is the case in lunar augite. (G.K. Shenoy, G.M. Kalvius and S.S. Hafner, J. Appl. Phys. 40, p. 1314, 1969.)

We believe that the unusual ferrimagnetic ordering in lunar augite ($Fe_{0.34}Mg_{0.30}Ca_{0.36}SiO_3$) is due to iron-iron clustering in the ca. 300Å wide single crystal domain bands depicted in the electron micrographs of the present report.

December 13, 1969

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8. J. Silcox, Philos. Mag., 8, 7 (1963).
9. S. S. Hafner and D. Virgo, Science 165, 285 (1969)

Electron Microscopy of Lunar Pyroxenes

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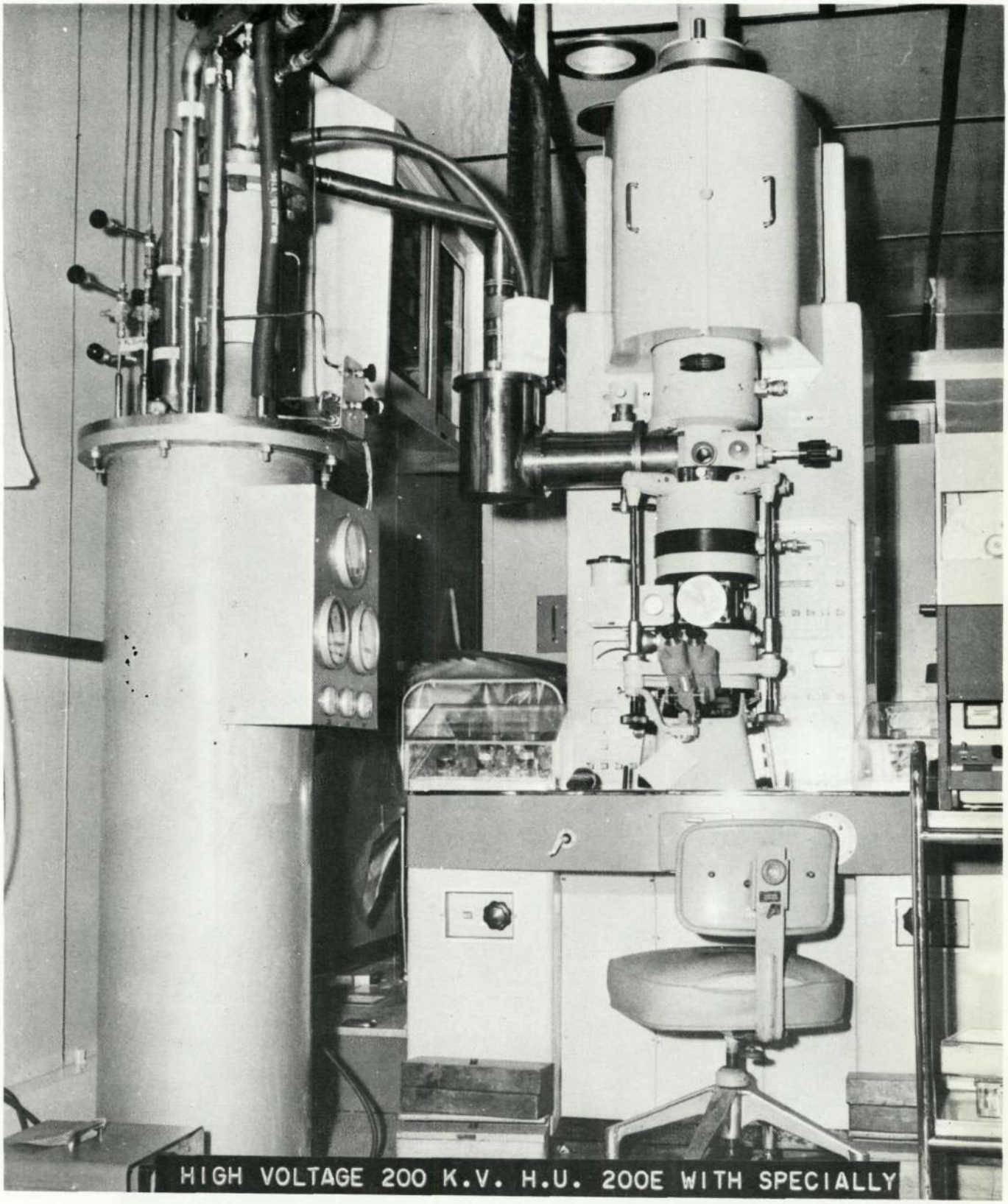
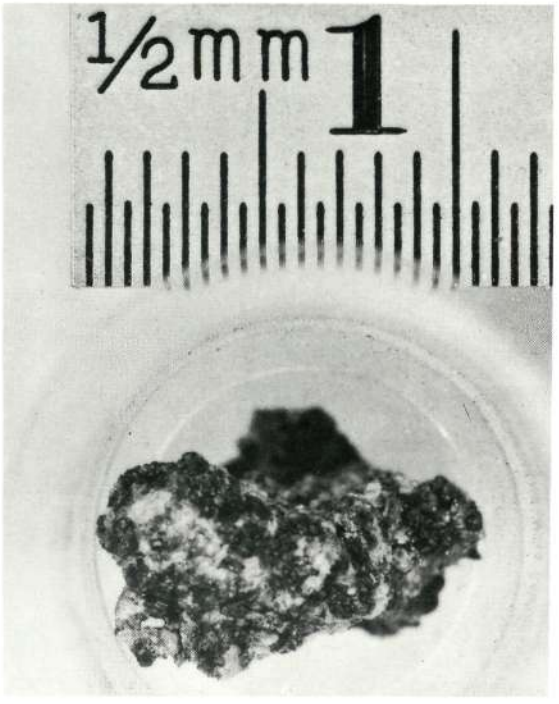
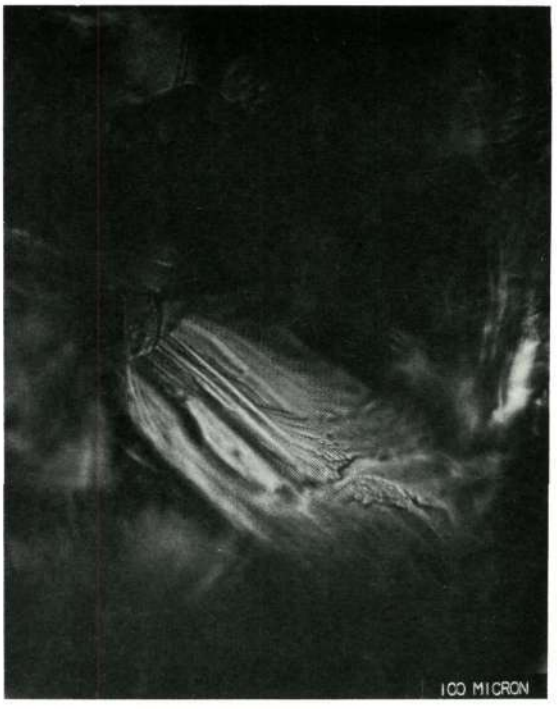


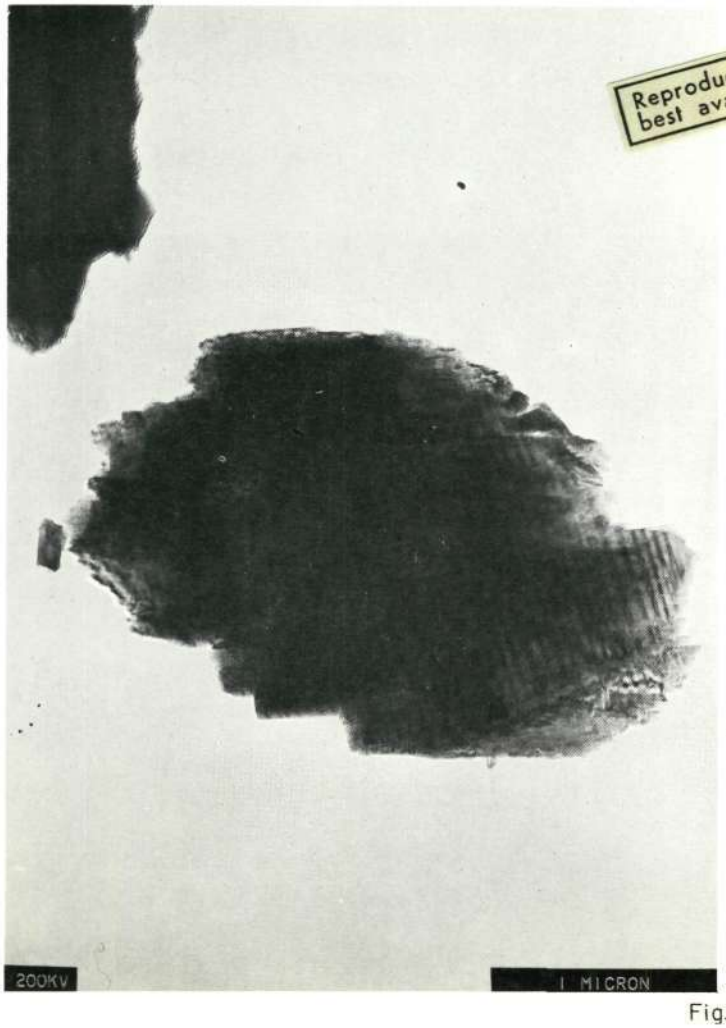
HIGH VOLTAGE 200 K.V. H.U. 200E WITH SPECIALLY DESIGNED LIQUID HELIUM SPECIMEN STAGE ATTACHED TO CLOSED CYCLE SUPERFLUID LIQUID HELIUM REFRIGERATOR WITH COLLINS/ADL HEAT EXCHANGER AND ACCESSORIES FOR SUPERCONDUCTING HIGH VOLTAGE ELECTRON MICROSCOPE 1969

Fig.1





CORRELATED LIGHT MICROSCOPY AND HIGH VOLTAGE ELECTRON MICROSCOPY STUDIES OF LUNAR SAMPLES RETURNED BY APOLLO 11



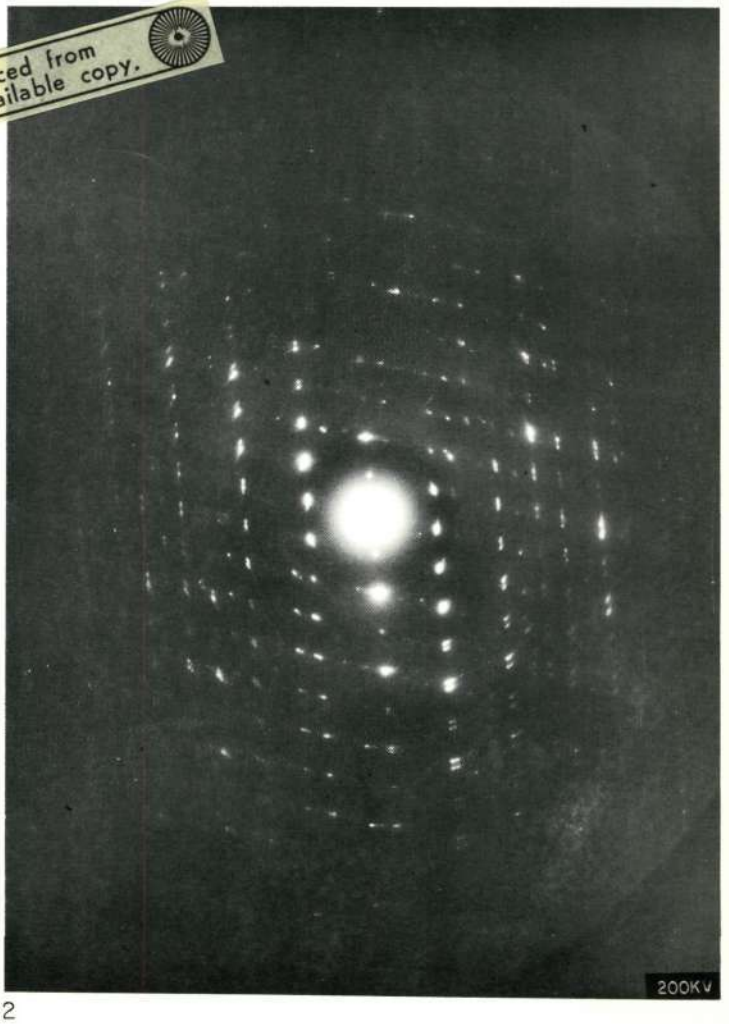
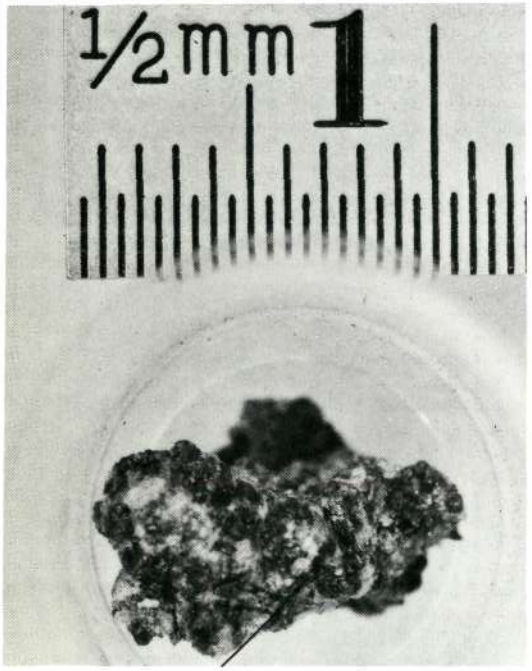
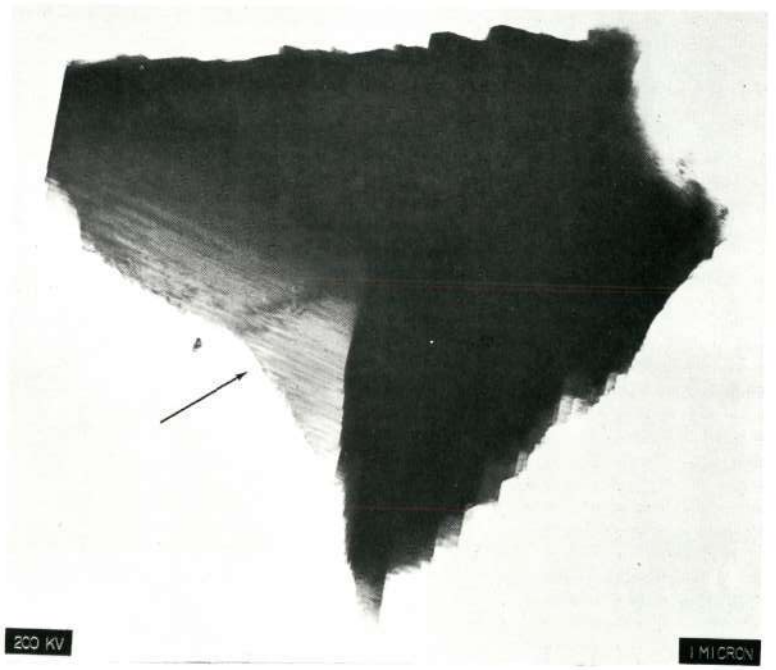


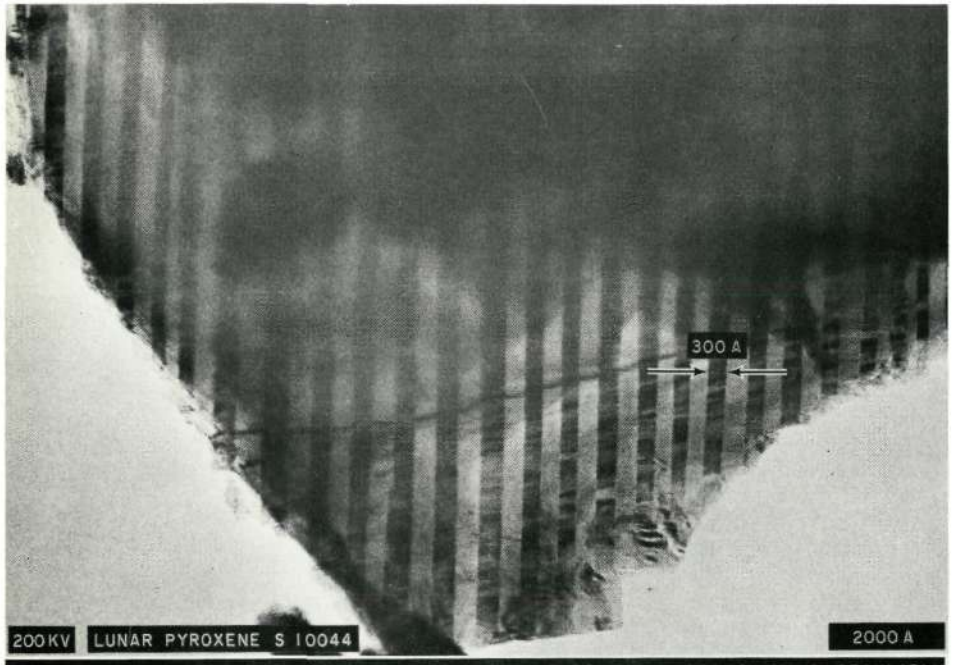
Fig. 2



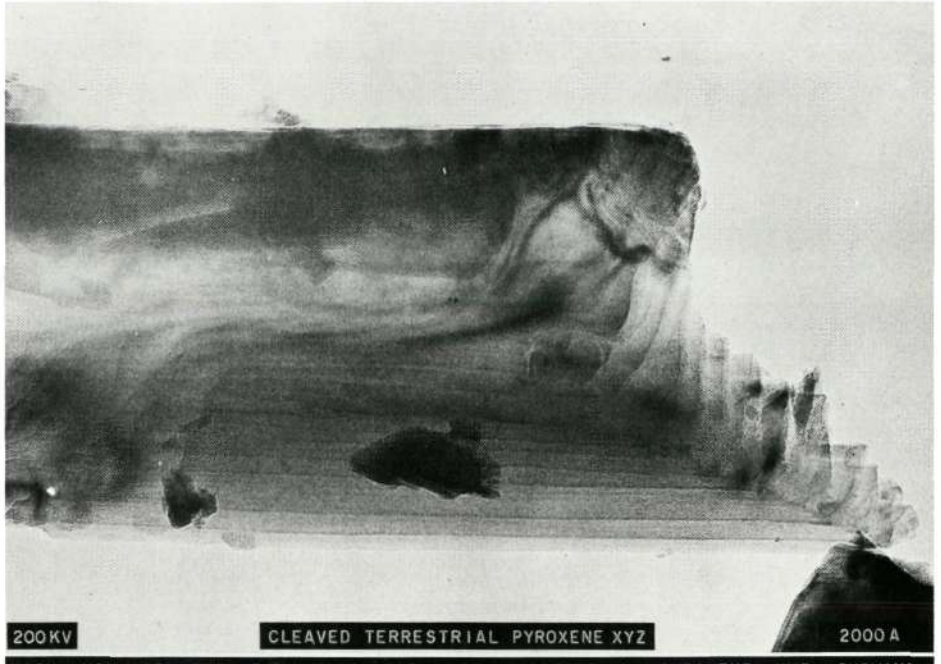


CORRELATED LIGHT MICROSCOPY AND HIGH VOLTAGE ELECTRON MICROSCOPY STUDIES OF LUNAR SAMPLES RETURNED BY APOLLO 11

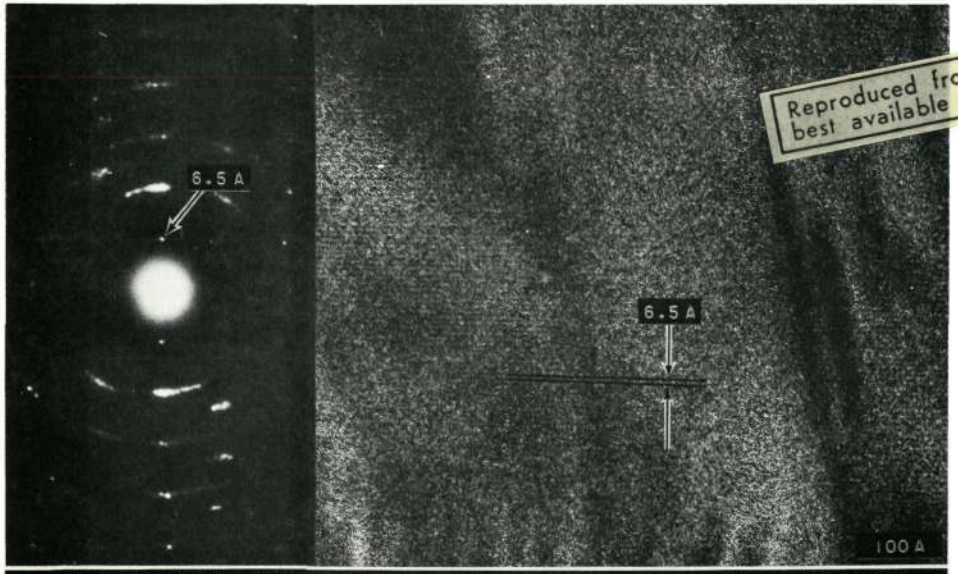
Fig. 3



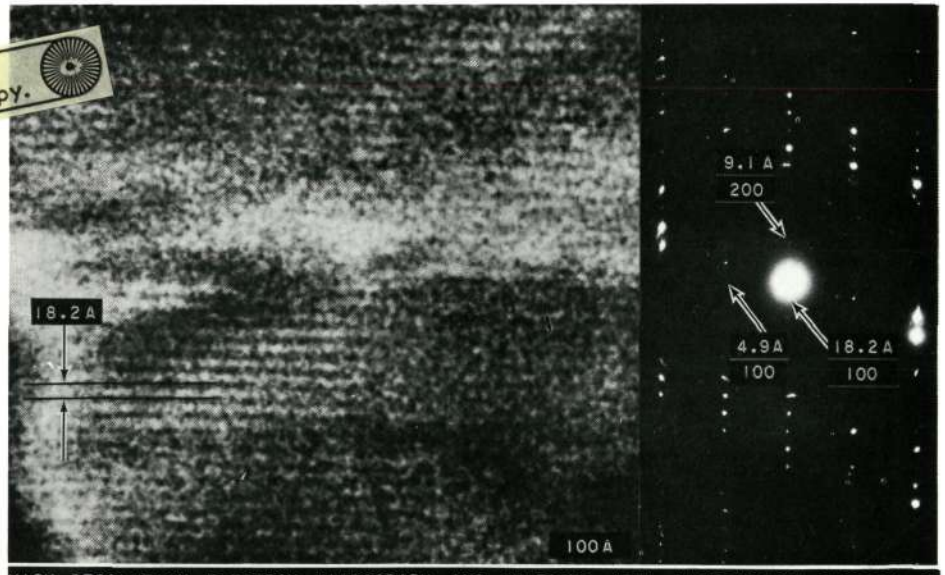
HIGH VOLTAGE ELECTRON MICROGRAPH OF LUNAR PYROXENE 10044 SHOWING REGULARLY SPACED UNIFORM DENSE BANDS



HIGH VOLTAGE ELECTRON MICROGRAPH OF TERRESTRIAL PYROXENE XYZ SHOWING CRYSTALLINE LAMELLAE

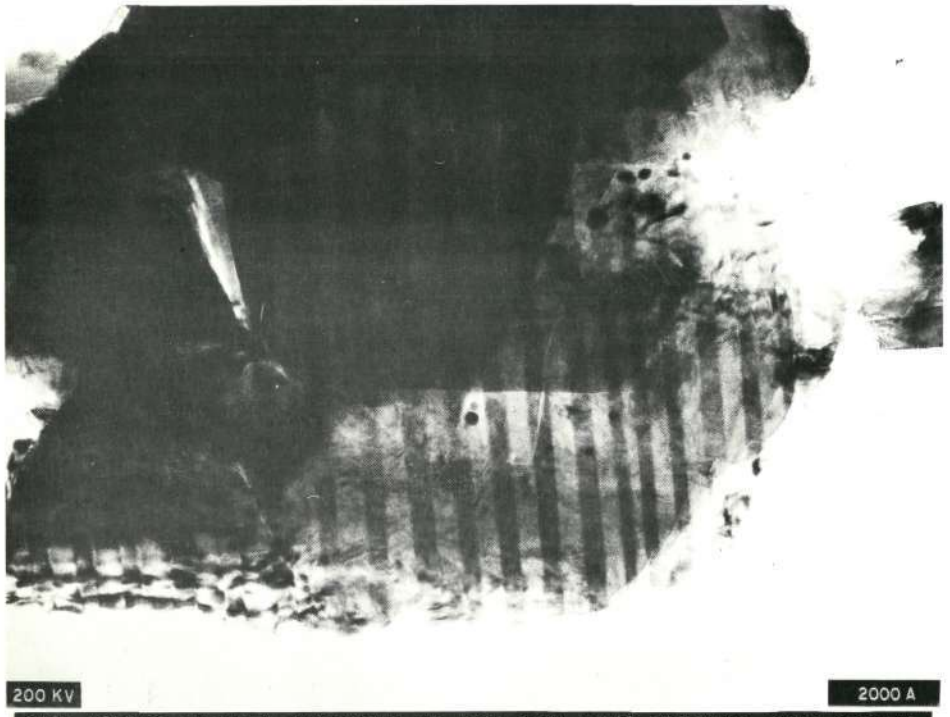


HIGH RESOLUTION ELECTRON MICROGRAPH AND SELECTED AREA DIFFRACTION PATTERN OF LUNAR PYROXENE 10044 SHOWING 6.5 A LATTICE SPACING

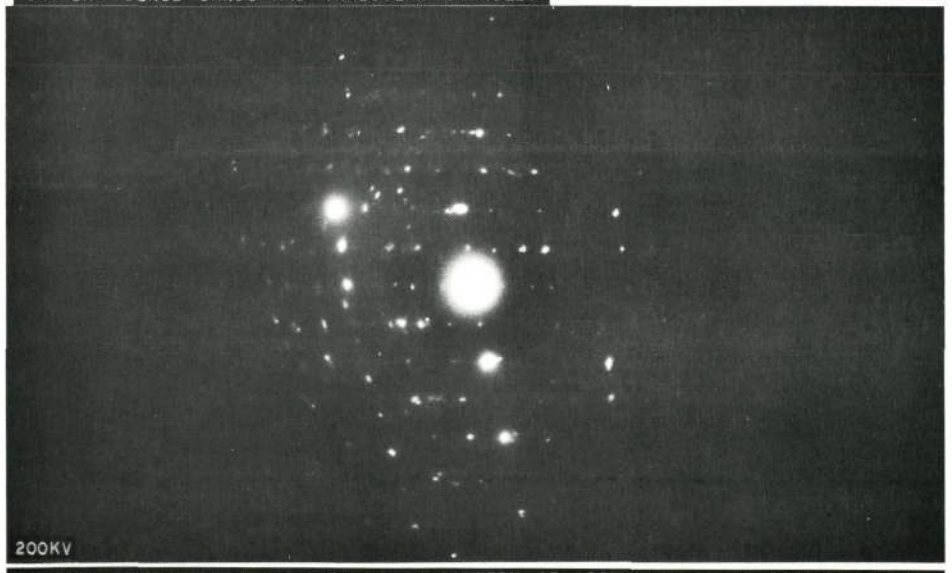


HIGH RESOLUTION ELECTRON MICROGRAPH AND SELECTED AREA DIFFRACTION PATTERN OF TERRESTRIAL PYROXENE XYZ SHOWING 18.2 A LATTICE SPACING

Fig-4

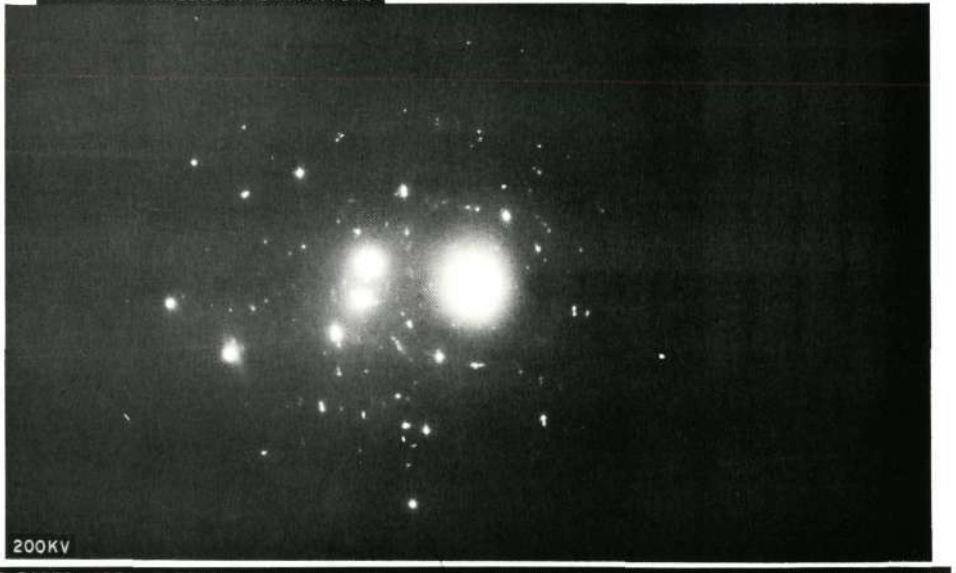


HIGH VOLTAGE ELECTRON MICROGRAPH OF IRON RICH LUNAR PYROXENE 10044 SHOWING UNIFORM DENSE BANDS AND IRREGULAR GRANULES



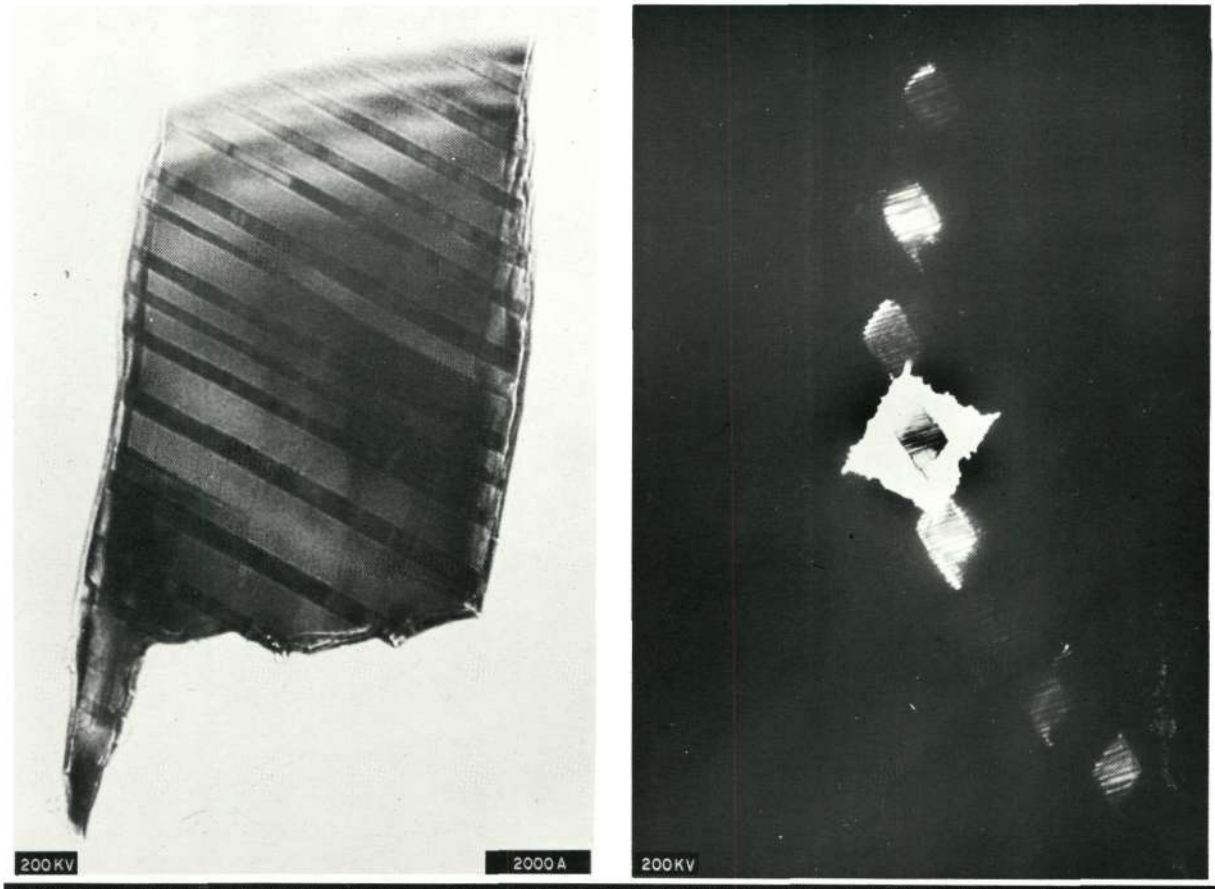
SELECTED AREA ELECTRON DIFFRACTION PATTERN OF IRON RICH LUNAR PYROXENE 10044

HIGH VOLTAGE ELECTRON MICROGRAPH OF MAGNESIUM RICH LUNAR PYROXENE 10044 SHOWING IRREGULAR STRIATIONS

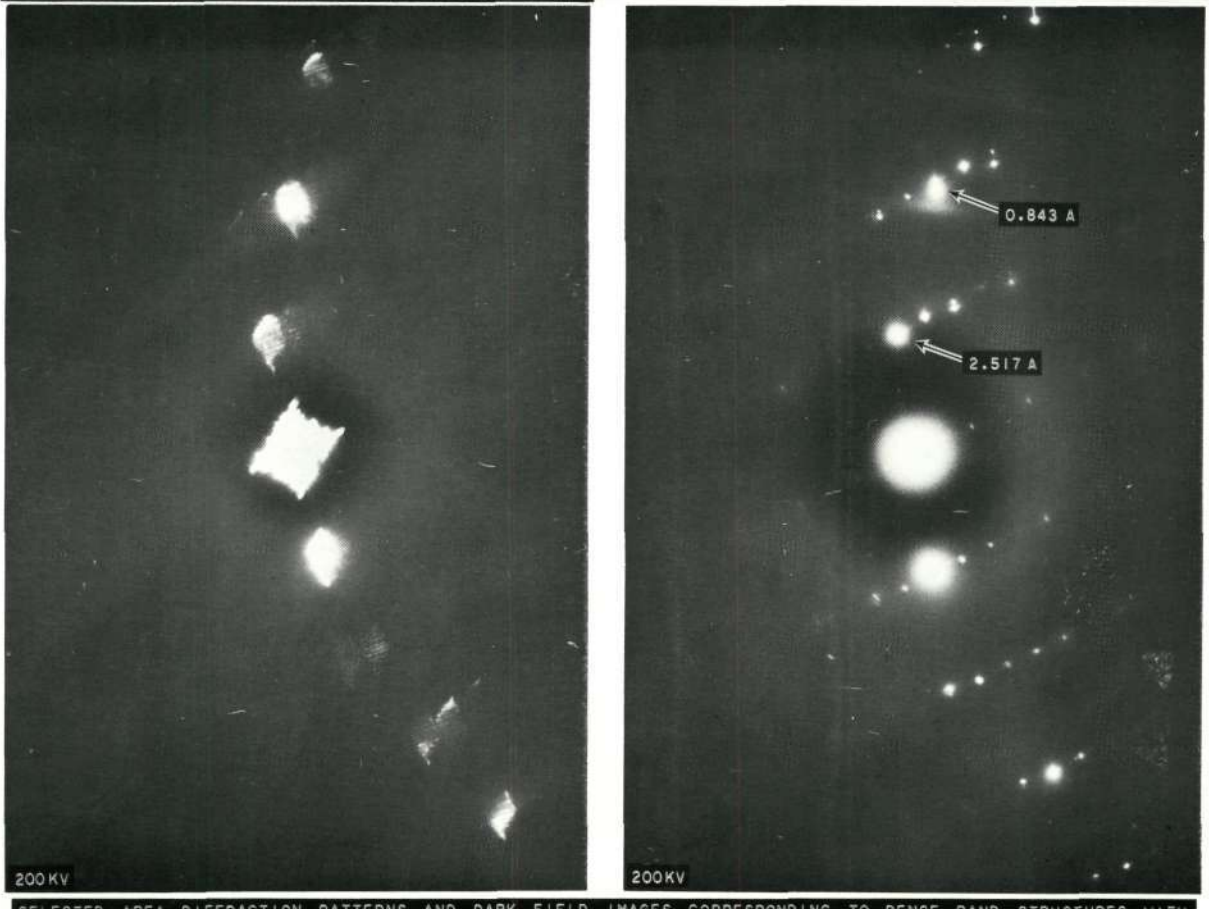


SELECTED AREA ELECTRON DIFFRACTION PATTERN OF MAGNESIUM RICH LUNAR PYROXENE 10044

Fig.5

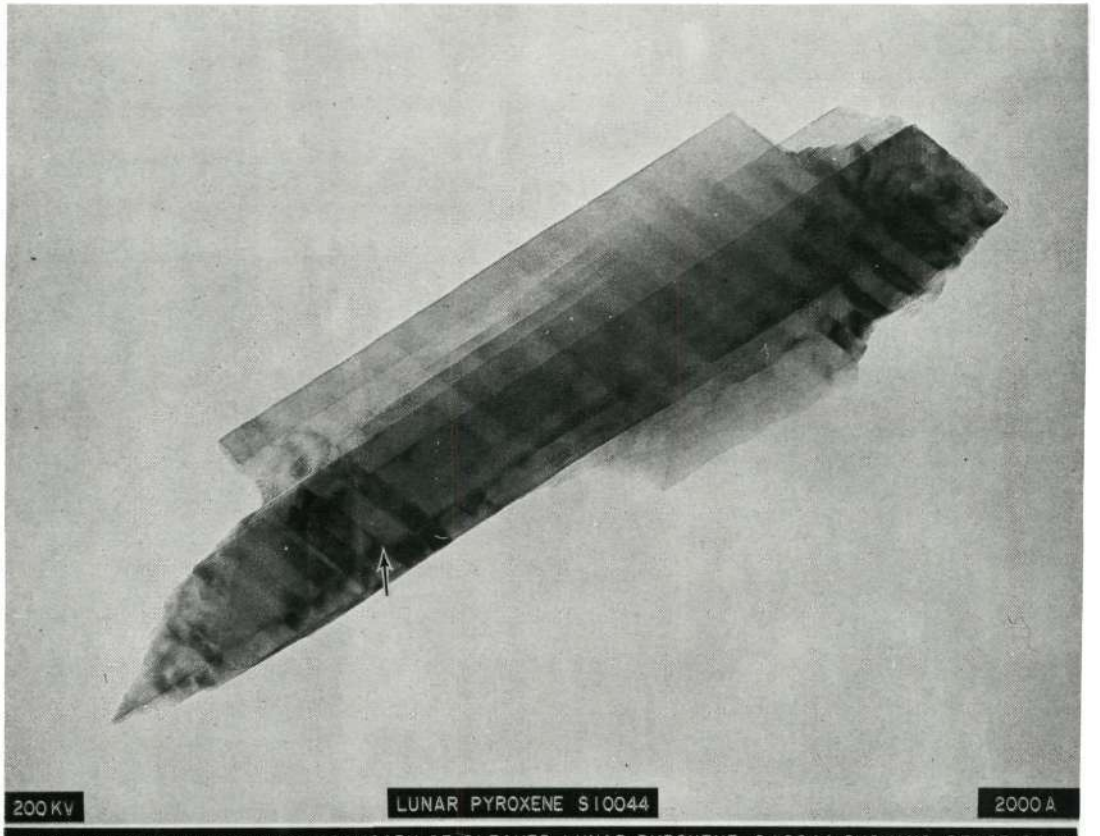


ELECTRON MICROGRAPH OF LUNAR PYROXENE 10044 SELECTED FOR COMPARING IMAGES OF BAND STRUCTURE WITH DIFFRACTION PATTERNS CENTRAL BRIGHT FIELD IMAGE OF LUNAR PYROXENE TAKEN WITH SELECTOR APERTURE ON (000) AND DARK FIELD IMAGES CORRESPONDING TO ENLARGED DIFFRACTION SPOTS

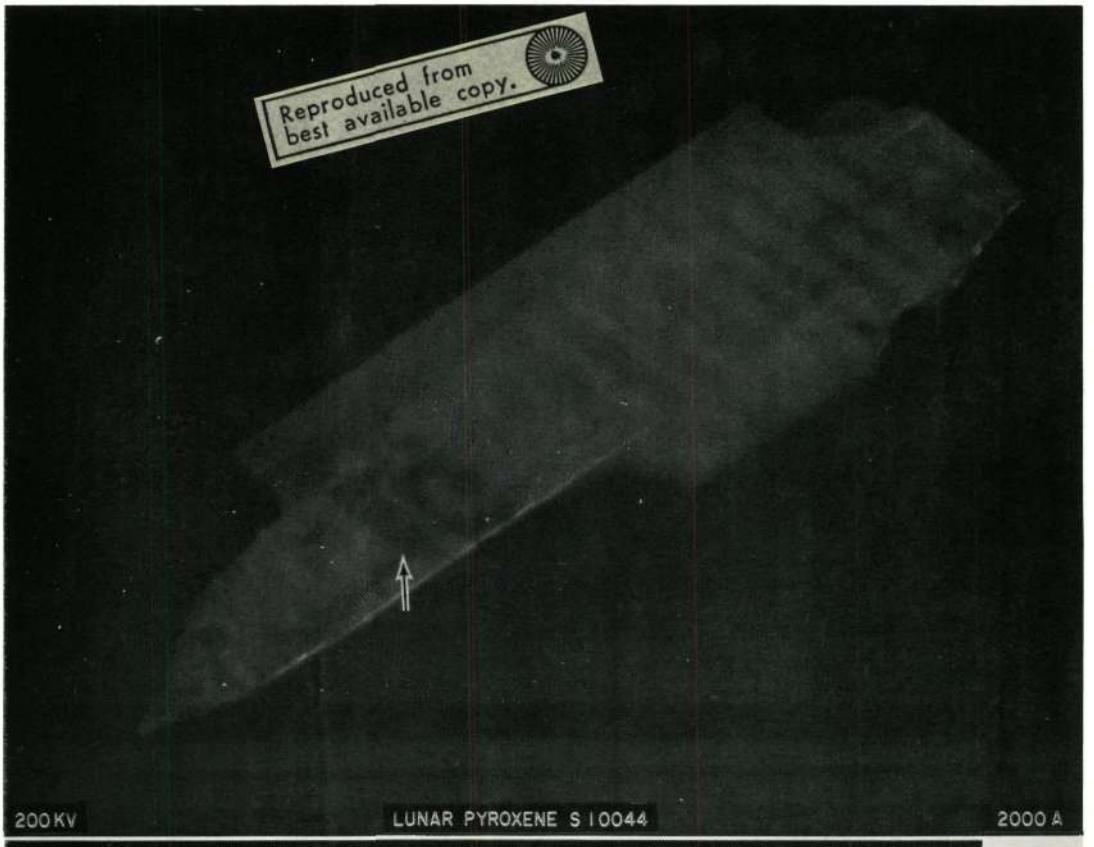


SELECTED AREA DIFFRACTION PATTERNS AND DARK FIELD IMAGES CORRESPONDING TO DENSE BAND STRUCTURES WITH CRYSTALLINE LATTICE SPACINGS OF 2.517 A AND 0.843 A

Fig.6

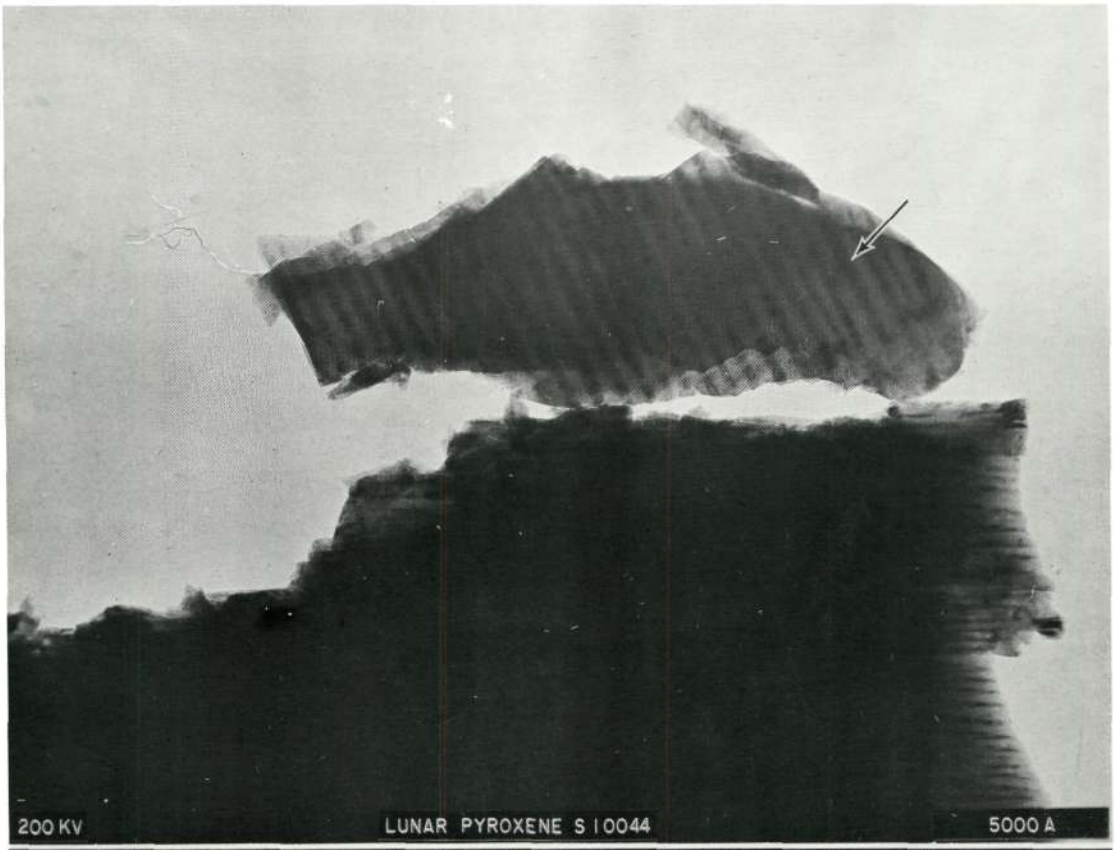


HIGH VOLTAGE ELECTRON MICROGRAPH OF CLEAVED LUNAR PYROXENE S 10044 SHOWING DENSE BAND STRUCTURE ON CRYSTALLINE LAMELLAE.

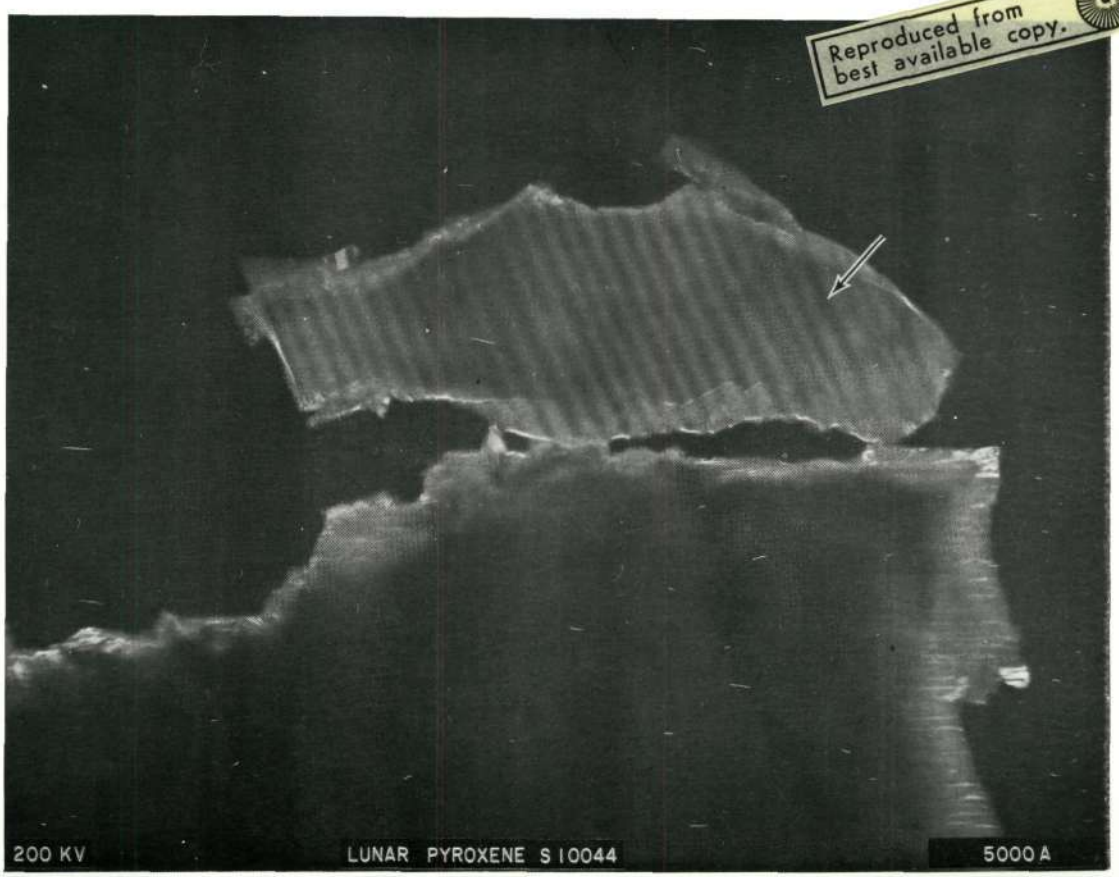


DARK FIELD HIGH VOLTAGE MICROGRAPH OF CLEAVED LUNAR PYROXENE S 10044 SHOWING CHARACTERISTIC DENSE BAND STRUCTURE.

Fig.7

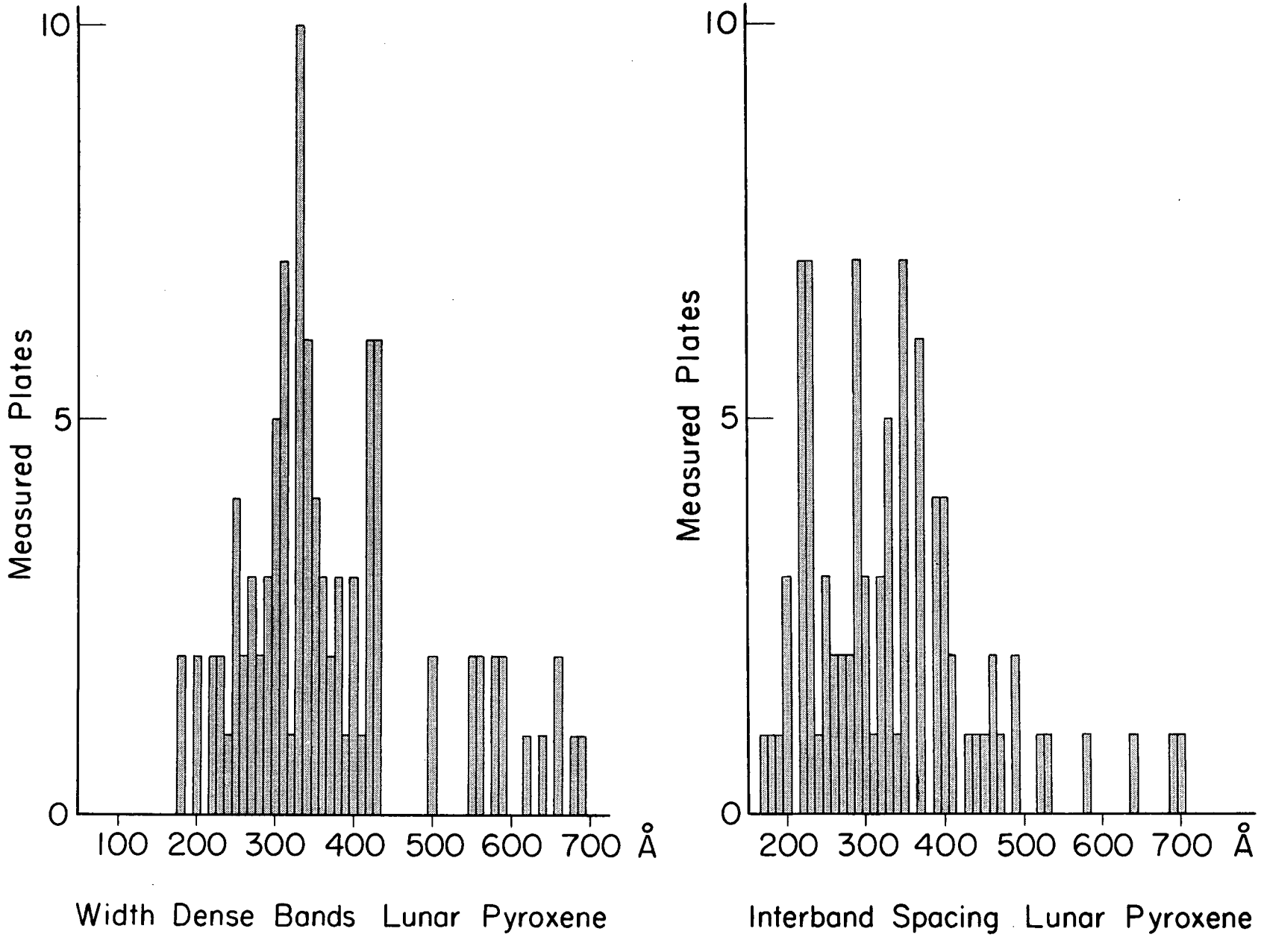


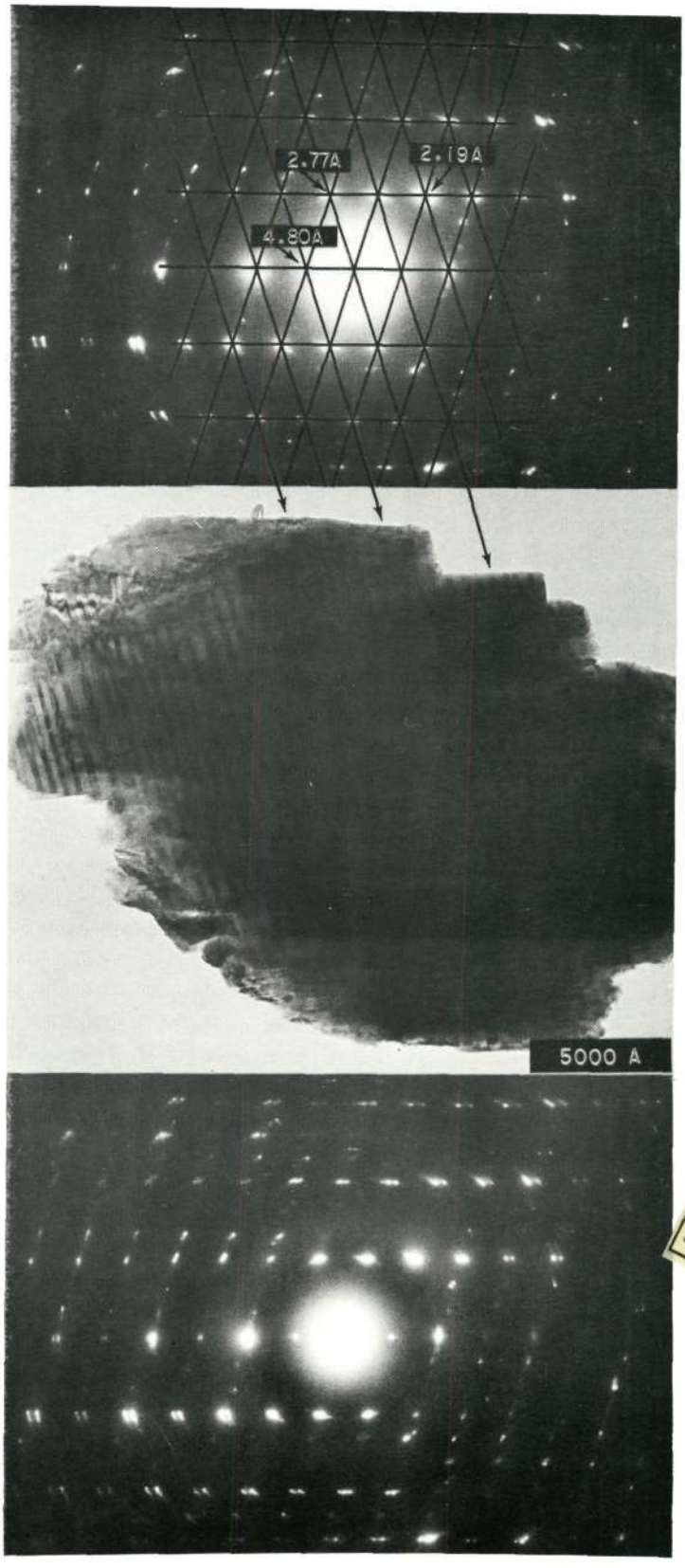
HIGH VOLTAGE BRIGHT FIELD ELECTRON MICROGRAPH OF CLEAVED LUNAR PYROXENE S 10044 SHOWING UNIFORM DENSE BAND STRUCTURE



HIGH VOLTAGE DARK FIELD ELECTRON MICROGRAPH OF CLEAVED LUNAR PYROXENE S 10044

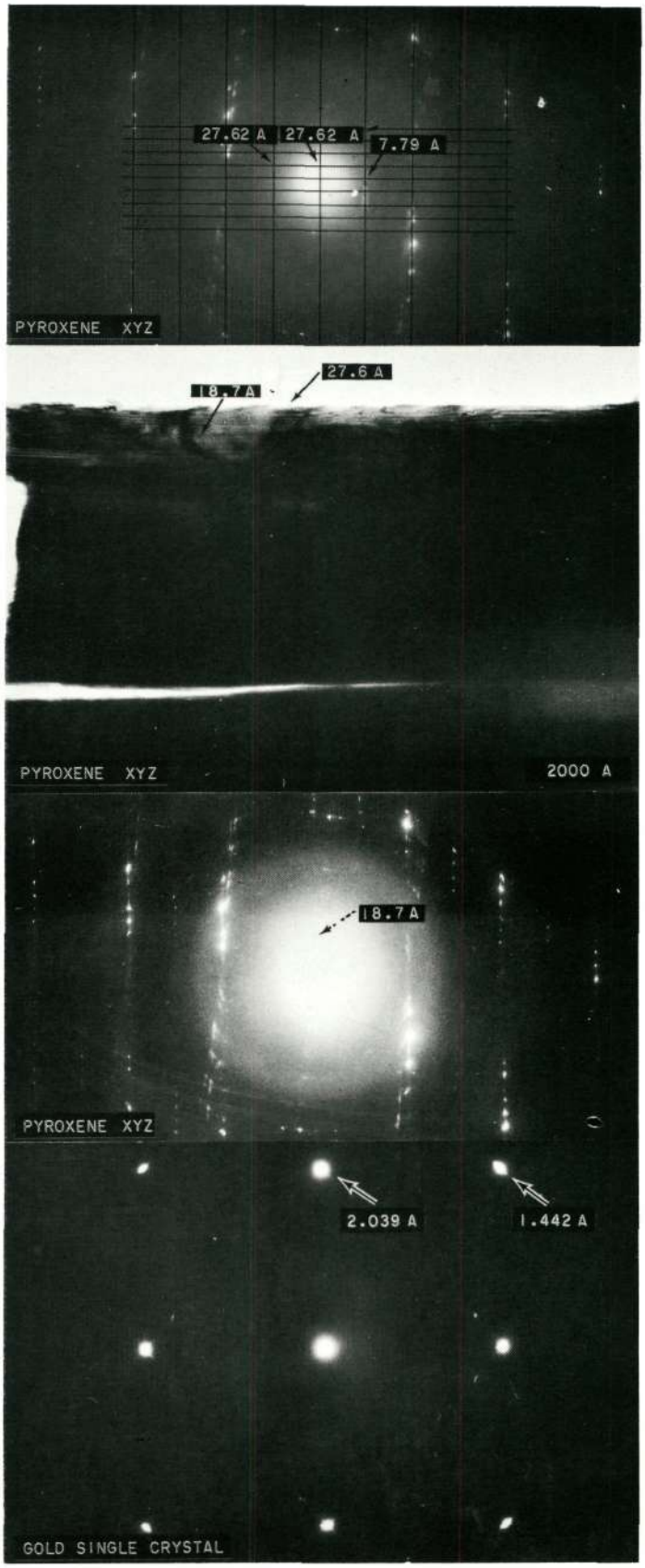
Fig.8


10
5
0
Measured Plates
100 200 300 400 500 600 700 Å
Width Dense Bands Lunar Pyroxene
10
5
0
Measured Plates
200 300 400 500 600 700 Å
Interband Spacing Lunar Pyroxene



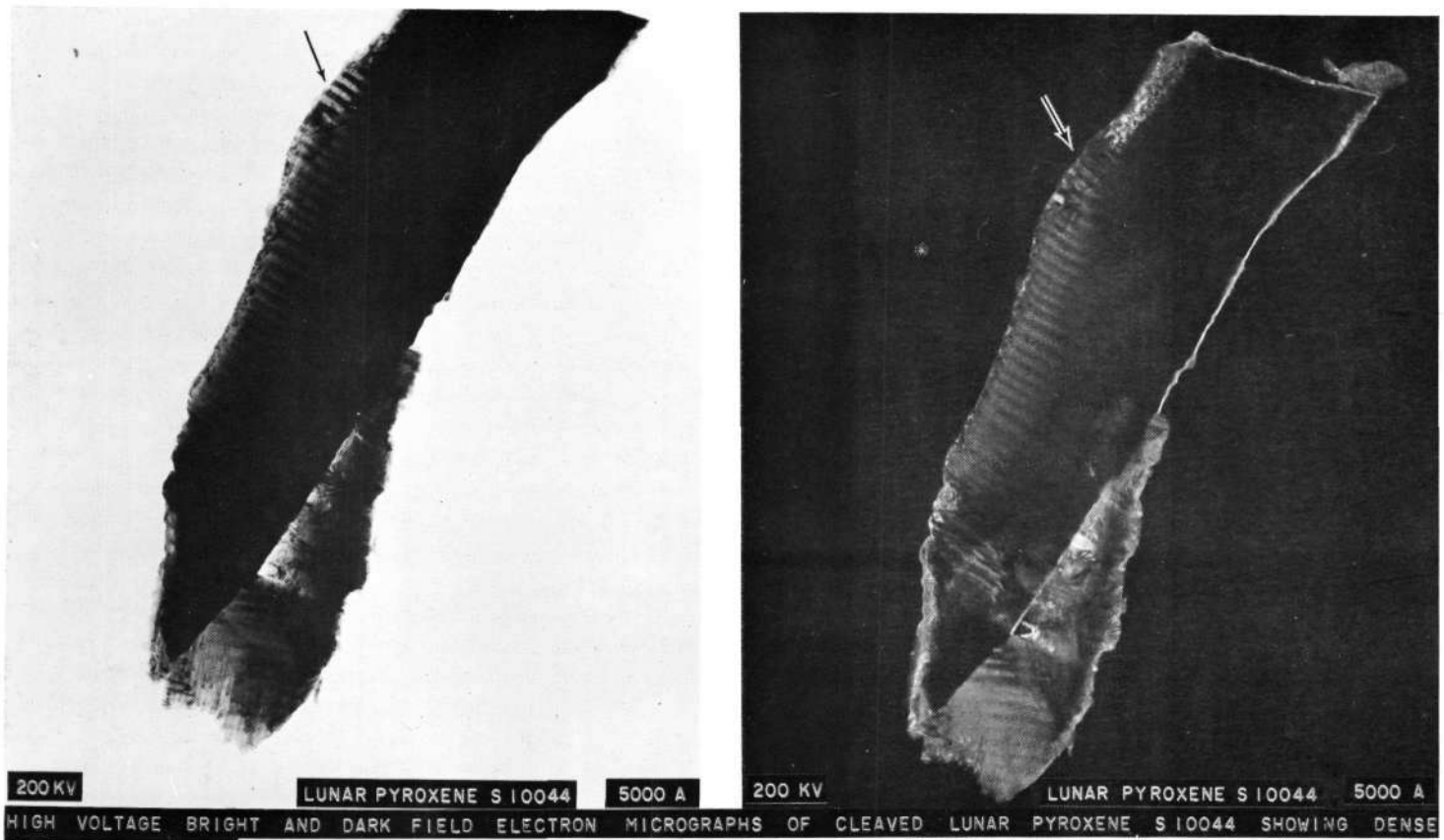
CORRELATED HIGH VOLTAGE ELECTRON MICROSCOPY AND INDEXED SELECTED AREA ELECTRON DIFFRACTION PATTERN OF CLINOPYROXENE CRYSTAL OBTAINED FROM LUNAR SAMPLE 10044 RETURNED BY APOLLO 11. SPACINGS OF 4.8 A 2.77 A 2.19 A RECORDED BY MITSUO OHTSUKI ELECTRON MICROSCOPY LABORATORY H. FERNANDEZ MORAN UNIVERSITY OF CHICAGO

Fig.9

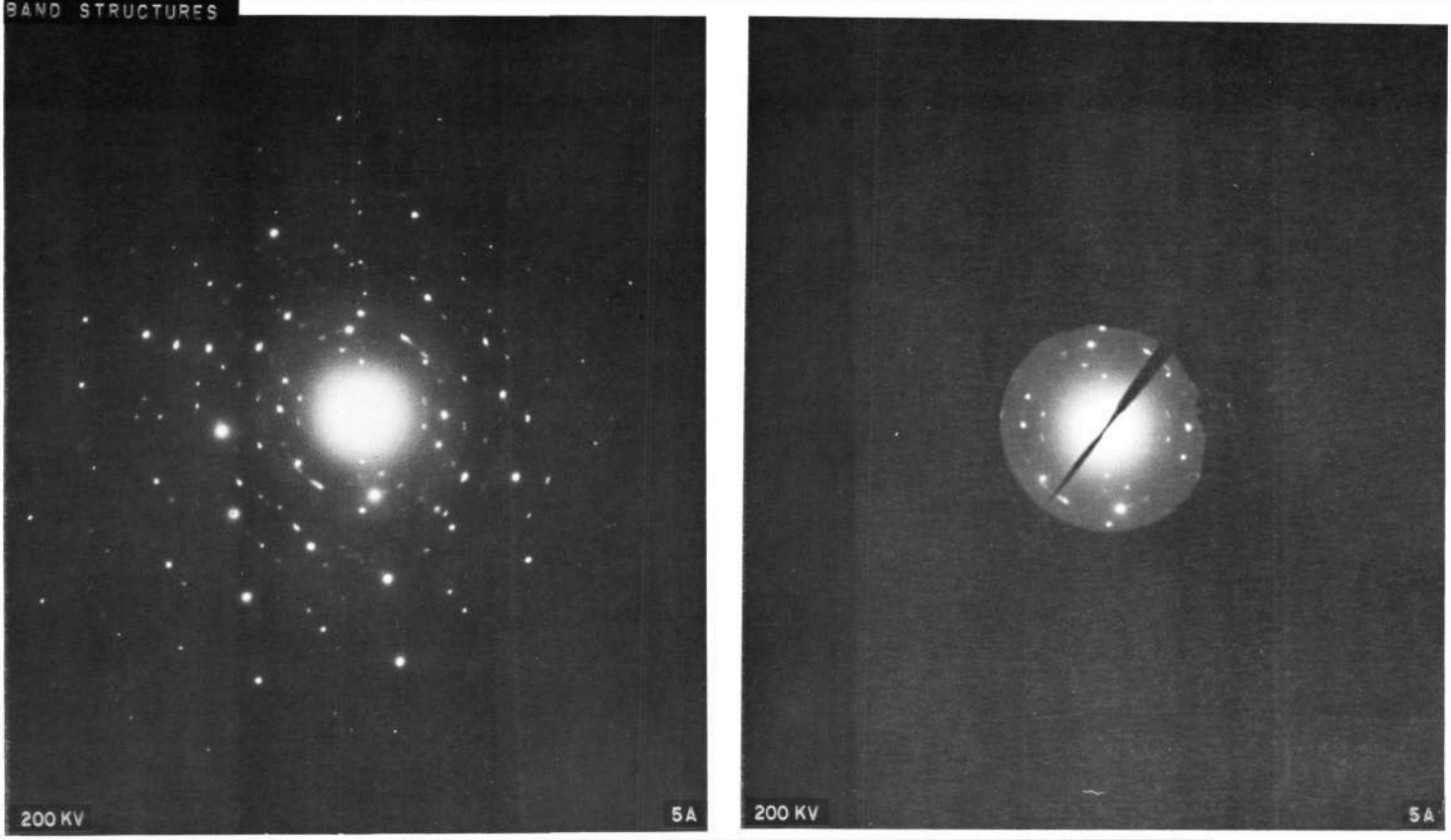


CORRELATED HIGH VOLTAGE ELECTRON MICROSCOPY AND INDEXED SELECTED AREA ELECTRON DIFFRACTION OF TERRESTRIAL PYROXENE XYZ OBTAINED FROM DR. HAFNER. SPACINGS OF 27.62 A 7.79 A 7.53 A RECORDED BY MITSUO OHTSUKI . CALIBRATION WITH GOLD SINGLE CRYSTAL 200 PLANES 2.039 A AND 220 PLANES 1.442 A ELECTRON MICROSCOPY LABORATORY H. FERNANDEZ MORAN UNIVERSITY OF CHICAGO OCT. 1969

Fig.10

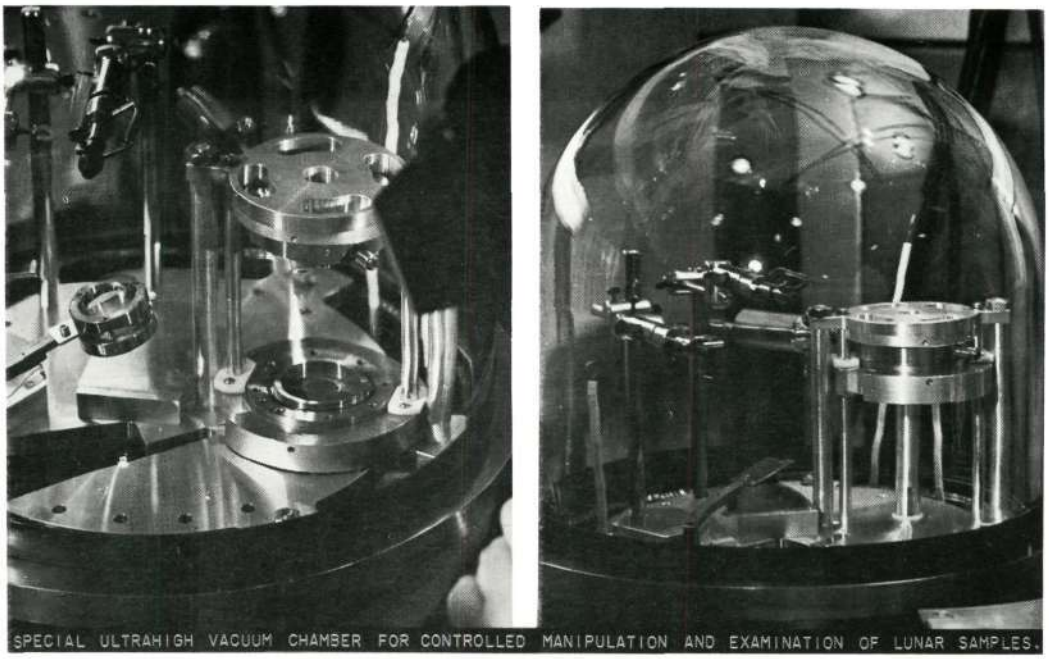


HIGH VOLTAGE BRIGHT AND DARK FIELD ELECTRON MICROGRAPHS OF CLEAVED LUNAR PYROXENE S 10044 SHOWING DENSE BAND STRUCTURES

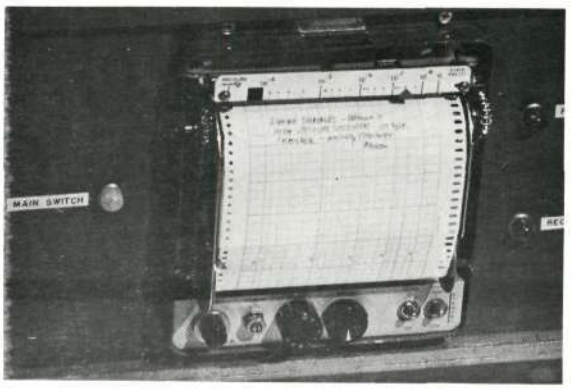


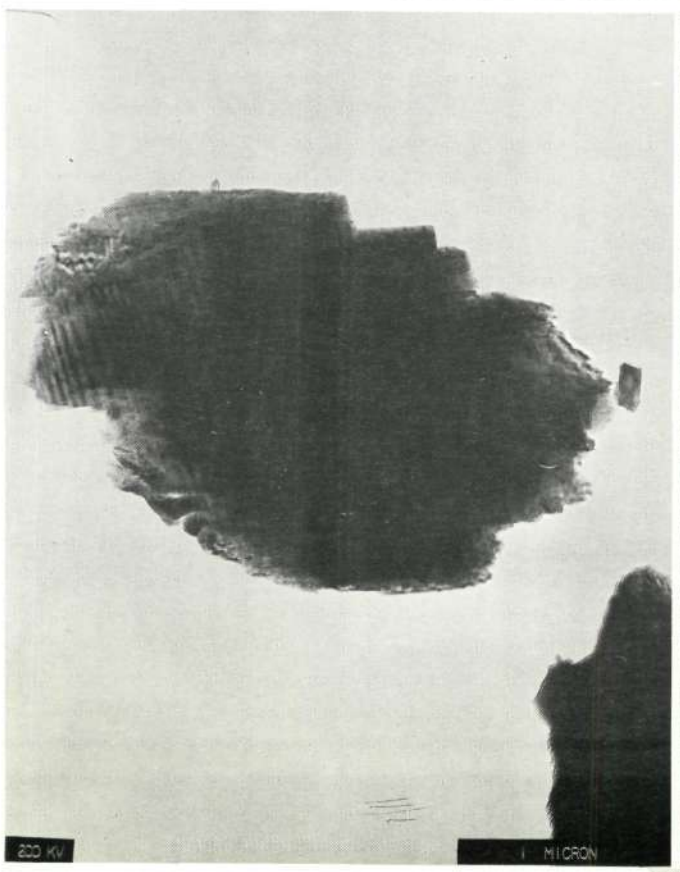
CORRESPONDING SELECTED AREA ELECTRON DIFFRACTION PATTERNS OF LUNAR PYROXENE S 10044

Fig.11



SPECIAL ULTRAHIGH VACUUM CHAMBER FOR CONTROLLED MANIPULATION AND EXAMINATION OF LUNAR SAMPLES.





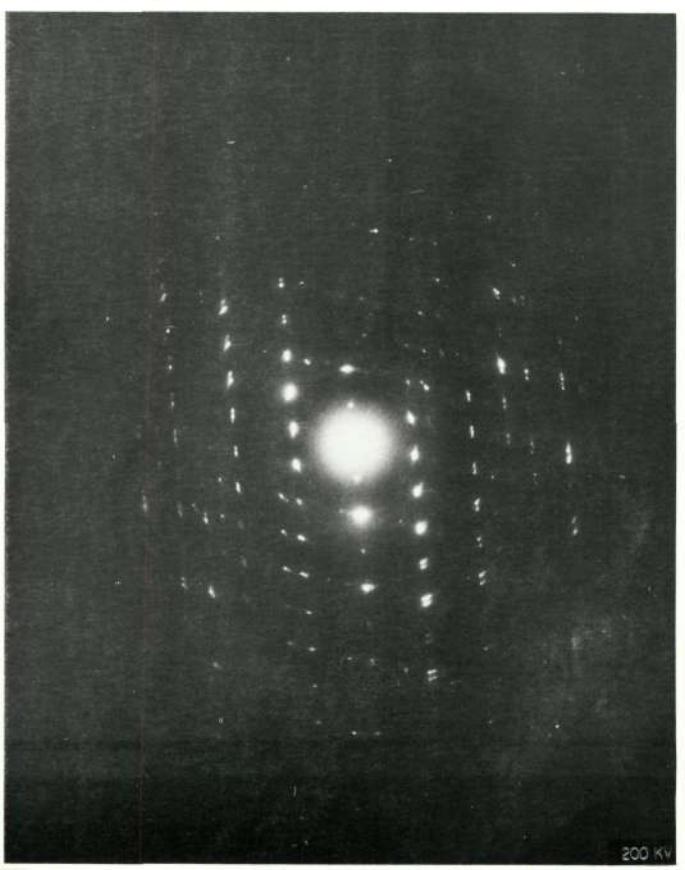
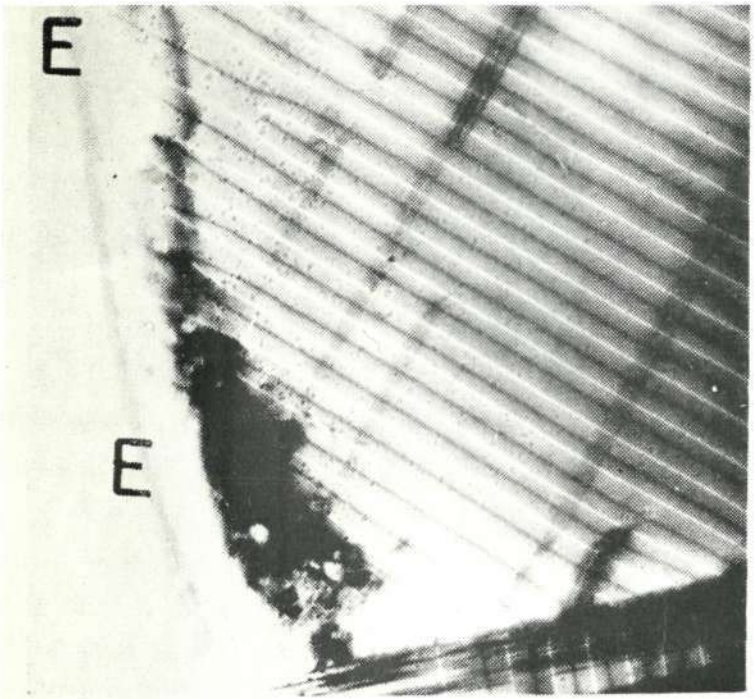


Fig.12

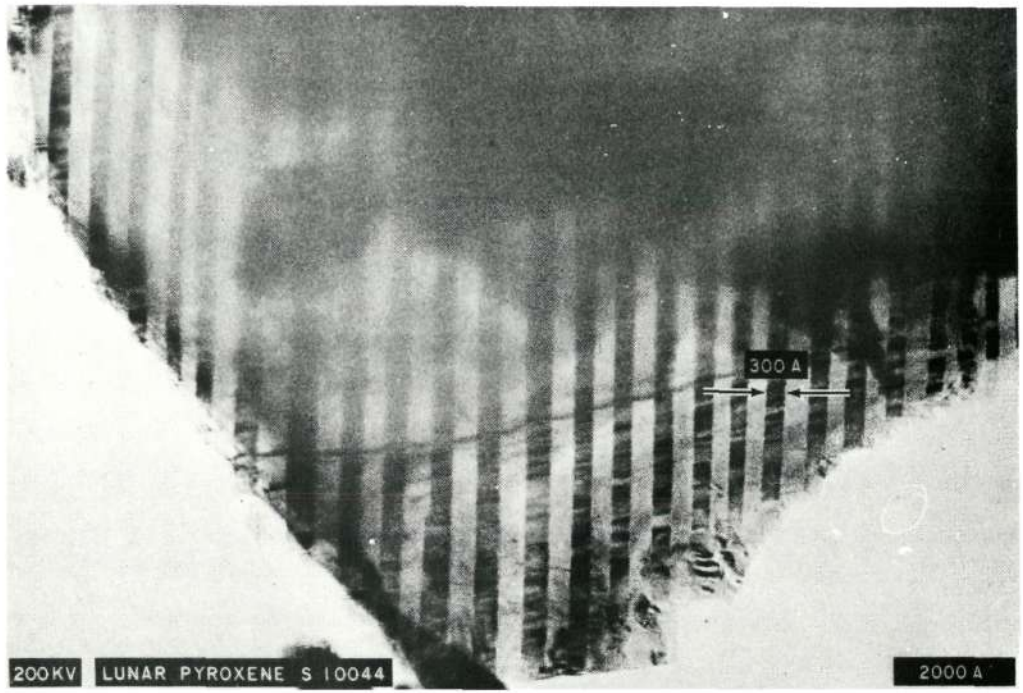


1 μ

The nucleation of domains in cobalt as the foil becomes thicker. The edge of the foil is shown as E.

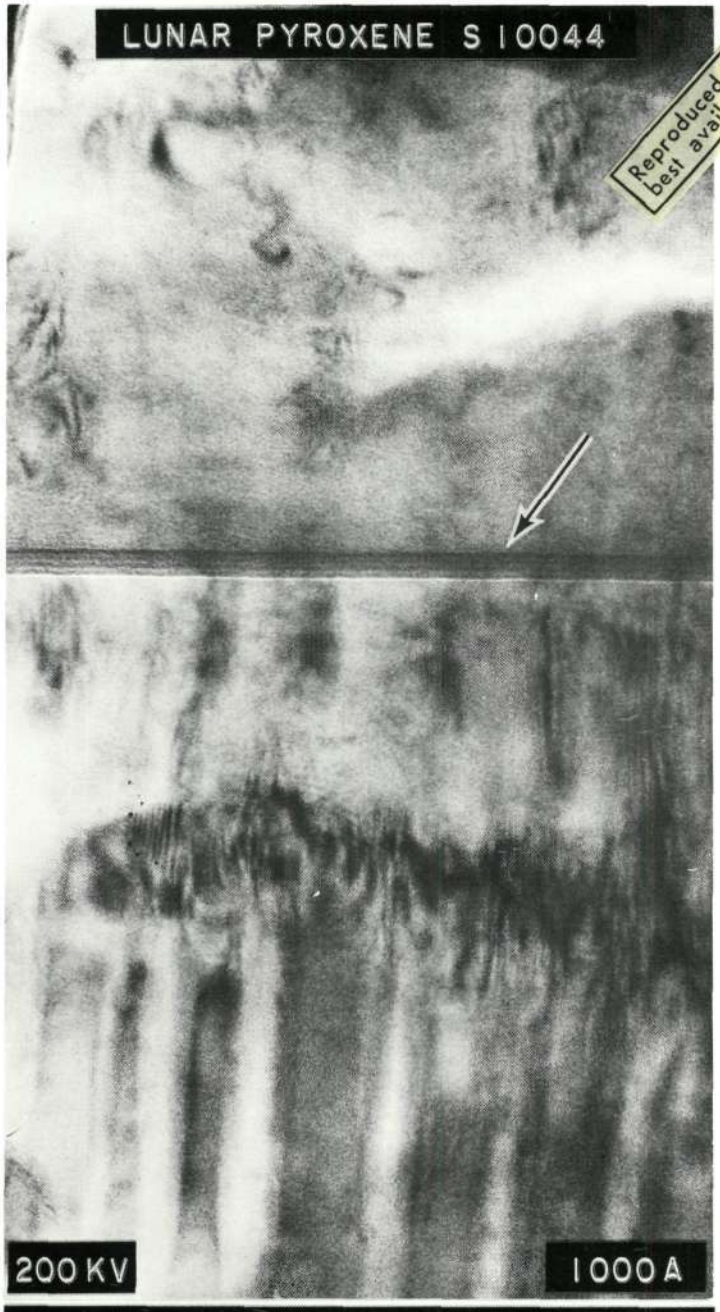
Magnetic Domain Walls in Thin Films of Nickel and Cobalt By J. Silcox 1963

PHILOSOPHICAL MAGAZINE VIII /7

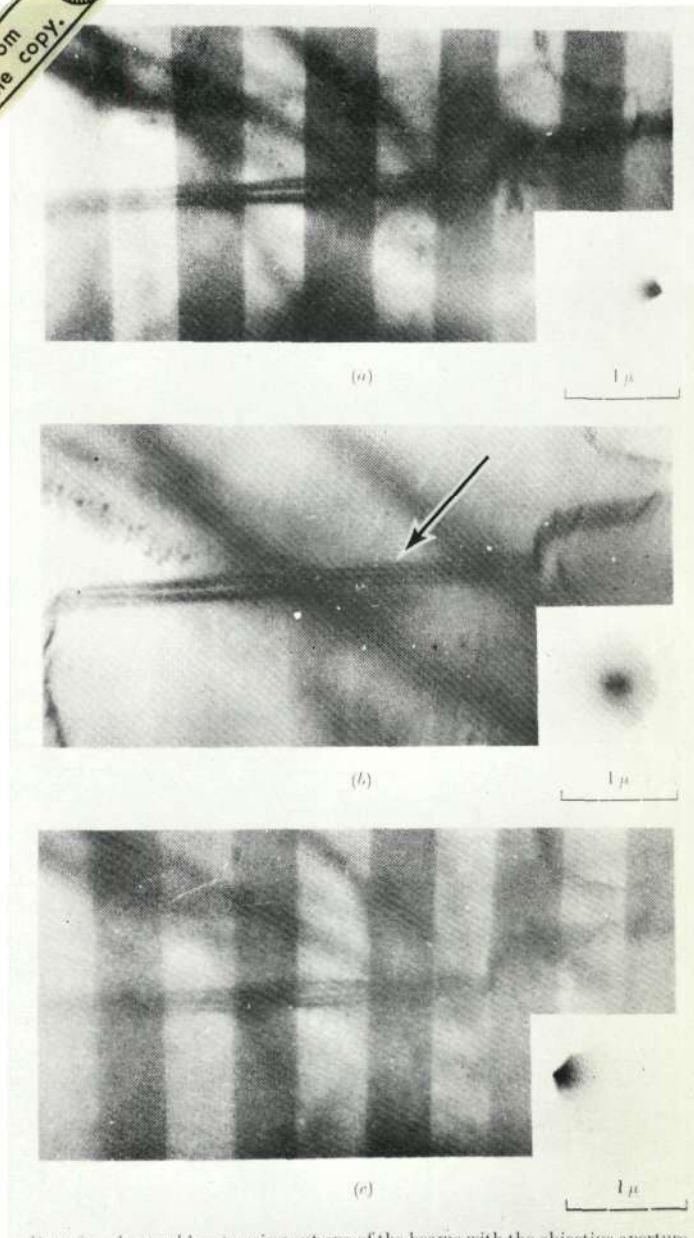


LUNAR PYROXENE CRYSTALLINE LAYERS WITH STRUCTURES RESEMBLING MAGNETIC DOMAIN WALLS AS REVEALED BY HIGH VOLTAGE ELECTRON MICROSCOPY 1969

H. FERNANDEZ MORAN AND M. OHTSUKI



LUNAR PYROXENE CRYSTALLINE LAYERS WITH STRUCTURES RESEMBLING MAGNETIC DOMAIN WALLS AND STACKING FAULTS AS REVEALED BY SPECIAL ELECTRON MICROSCOPY TECHNIQUES



Domains observed by stopping out one of the beams with the objective aperture. The position of the objective aperture in relation to the diffraction spots for each micrograph is shown in the inset. Magnification ×21 000.

Magnetic Domain Walls in Thin Films of Nickel and Cobalt

By J. Silcox

Cavendish Laboratory, Free School Lane, Cambridge†

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