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## HIGH VOLTAGE ELECTRON MICROSCOPY OF LUNAR SAMPLES

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## GENERAL SUMMARY

Based on earlier work with Dr. Stefan S. Hafner and his colleagues of the Dept. of the Geophysical Sciences at The University of Chicago, during the 2-year funding period covered by this grant, my associates and I have worked closely with co-investigator Dr. David Virgo of the Carnegie Institution Geophysical Laboratories to carry out correlated investigations of lunar pyroxenes from Apollo 11, 12, 14 and 15 specimens and reference samples from selected terrestrial sources.

The iron-rich and magnesium-rich pyroxene specimens were crushed to a grain size of ca. 50 microns and studied by a combination of x-ray and electron diffraction, electron microscopy, $^{57}$Fe Mössbauer spectroscopy and x-ray crystallography techniques. Additional heating experiments were performed under controlled conditions.

Crystals cleaved and sectioned by diamond knife ultramicrotomy were examined by both bright and dark field high resolution electron microscopy at voltages ranging from 75 kV to 200 kV and temperatures of 290°K and 4.2°K using a special liquid helium cold stage. Highly ordered, uniform electron-dense bands, corresponding to exsolution lamellae, with average widths of ca. 230Å to 1000Å dependent on the source specimen were observed in most of the separate preparations. These were separated by wider, less-dense interband spacings with average widths of ca. 330Å to 3100Å. In heating experiments, splitting of the dense bands into finer structures, leading finally to obliteration of the exsolution lamellae was recorded. The extensive exsolution is evidence for significantly slower cooling rates, or possibly annealing, at temperatures in the subsolidus range, adding evidence that annealing of rock from the surface of the moon took place at ca. 600°C.

Correlation of the band structures with magnetic ordering at low temperatures and iron clustering within the bands was studied. Specimens of iron-rich pyroxenes from Apollo 11, 12 and 14 were examined by electron microscopy and selected-area diffraction using several forms of decoration techniques for the study of ferromagnetic domain boundaries by the deposition of iron in a high vacuum or in an argon atmosphere. The structures, probably indicating enhanced concentrations of iron within the bands. This has subsequently been at least partially confirmed by independent investigators.

Dr. Virgo is now characterizing the 15076 pigeonite specimens by x-ray crystallography techniques. When this research is complete, a comprehensive paper embodying the details of this entire research project will be submitted to an appropriate scientific journal. It will include data and comparisons with electron optical finger-printing of the pigeonites based on the work carried out in our laboratories at The University of Chicago and reported in the various papers which we have presented at the Lunar Science Conferences and summarized here.

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## INTRODUCTION

In 1970, we undertook a series of preliminary high resolution electron microscopy and electron diffraction studies of pyroxenes from Apollo 11 type B specimens in an attempt to clarify and supplement data derived from $^{57}Fe$ Mössbauer absorption studies conducted by Dr. Stefan S. Hafner and his associates in the Department of the Geophysical Sciences at The University of Chicago. Based on the results of this work and on the demonstrated potential of the high resolution electron microscope to directly reveal the ultrastructural details of these lunar samples, we applied for a research grant in the lunar science Apollo program with co-investigator Dr. David Virgo of the Carnegie Institution Geophysical Laboratories in Washington, D. C.

From our first observation and characterization of extensive pigeonite-augite exsolution lamellae in these iron-bearing chain silicates to our recent correlation of magnetic ordering and iron-iron clustering within the band structures, our results have now been confirmed by several independent researchers in the field. Thus, we have been able to make a significant contribution to the present knowledge and understanding of the ultrastructural organization of the lunar rocks and to obtain new insights into the evolution and cooling history of the moon.

The electron microscopy and electron diffraction work on the Apollo 11, 12, 14 and 15 samples was carried out by our interdisciplinary research team under controlled conditions in the specially-

designed facilities at The University of Chicago Research Institutes. It has been systematically supplemented by complementary Mössbauer, x-ray diffraction and x-ray crystallographic studies conducted by co-investigator Dr. David Virgo at the Carnegie Institution Geophysical Laboratories.

## STUDIES OF APOLLO 11 SAMPLES

Pyroxenes from lunar rock 10044,25 (composition $En_{36}Fs_{34}Wo_{30}$) were cleaved and sectioned by diamond knife ultramicrotomy and mounted directly on ultra-thin carbon films or platinum or copper grids.

Standard (75 kV) and high voltage (200 kV) electron microscopy and diffraction studies of iron-rich separates from this rock sample revealed exceptionally regular, periodically-spaced dense bands 100Å to 600Å wide (average width ca. 300Å). At the first Lunar Science Conference, we reported that these straight-edged bands, corresponding to single crystal domains, closely resembled images of magnetic domain walls seen in layers of ferromagnetic materials. (1) Lattice spacings of 2.5Å could be detected within the bands, probably corresponding to d(200).

These findings are consistent with the observation of extensive exsolution lamellae as described by Radcliff, Bailey and Ross.(2-4) Working with Virgo and Hafner, Mössbauer resonant absorption studies of $^{57}Fe$ iron-rich specimens from 10044,25 were carried out at liquid helium temperatures. (5) This revealed magnetic hyperfine splitting, and a relatively sharp transition temperature was found to exist between 20°K and 30°K, interpreted as a Neel point. The spin orientations below this point are assumed to be ferrimagnetic. However, the iron-bearing chain silicates are generally not magnetically ordered, even at very low temperatures, especially if the amount of

diamagnetic cations (magnesium, calcium) substituting for iron at the octahedrally coordinated positions is larger than 25% as in the iron-rich lunar augite.

This data was then correlated with our high resolution electron microscopy results, and we were able to speculate that the rather unusual magnetic ordering in the lunar pyroxene could be due to iron-iron clustering in the single crystal domain bands. This would imply a substantial enrichment of the bands in iron as compared to the interband regions. (Later magnetic decoration studies of lunar pyroxenes from Apollo 12 and 14 tended to confirm this hypothesis, as will be described in a subsequent section of the current report.)

In the domains between the bands, clustering of the Mg,Ca seems likely. If one assumes the extreme case that the ferrous ions are entirely located within the bands, one is led to the pigeonite composition $En_{0.19}Fs_{0.68}Wo_{0.47}$ for the bands and an almost pure diopside of the composition $En_{0.53}Wo_{0.47}$ for the interband domains (molecular percent). If one assumes complete ordering of the Mg and Ca in the bands, the $Fe^{2+}$ site occupancy in M1,M2 is 0.62, 0.73 respectively. This would certainly produce magnetic ordering at low temperatures due to M1-oxygen-M2 superexchanges. (5)

This preliminary work indicated the potential contribution of correlated electron optical and crystallographic studies to a better understanding of the intrinsic atomic organization of pyroxenes and their possible bearing on the crystallization and cooling history of the lunar igneous rocks.

## APOLLO 12 STUDIES

Based on the results of this preliminary work, inter-disciplinary investigations were continued on Apollo 12 clinopyroxenes. Cores of

yellow-green, calcium-poor pigeonite $Wo_9En_{60}Fs_{32}$ from coarse-grained Apollo 12 lunar basalt 12021 were separated and supplied to us by Virgo, Hafner and Warburton. (6) The crystals were again cleaved and sectioned by essentially non-destructive diamond knife ultramicrotomy techniques or fragmented and mounted directly on thin film substrates.

High resolution electron microscopy and diffraction studies were carried out at 290°K, 4.2°K and 1.8°K at voltages ranging from 75 kV to 200 kV. Using a special specimen stage at liquid helium temperatures (i.e. 1.8° to 4.2°K), reduced radiation damage and contamination were observed. Specimen cooling was provided by the world's only functioning Collins closed-cycle superfluid helium refrigeration system which is fully integrated with the modified high voltage (200 kV) electron microscope.

Arrays of uniform, electron-dense lamellae of exsolved augite ca. 200Å wide were observed in most of the crystal fragments examined. The bands appeared to be oriented mainly along (001) and (100) and other planes, and they displayed tapered ends, dense borders and relatively light central regions. The bands are separated by wide, less-dense interband regions (ca. 500Å to 600Å) of apparently homogeneous host. (7) These single crystal domain bands appeared to be less regular and slightly smaller than the ca. 300Å bands initially observed in the Apollo 11 specimens. Many of them also displayed splitting and serrated or scalloped edges. (8)

Contrast changes dependent on the orientation of the specimen to the incident electron beam were evident. This can be ascribed to antiphase domain boundaries in the pigeonite. Bands in certain other regions showed no modifications when the specimen was tilted ca. 1° to 5°, perhaps indicating that they represent localized areas of different compositions.

1. Heating Experiments. Experimental modifications involving heating of Apollo 11 and Apollo 12 specimens were also carried out. Electron optical studies of heated Apollo 11 specimens revealed a splitting of the bands into finer structures, leading finally to obliteration of the exsolution lamellae. (7)

Apollo 12 specimens heated by Virgo and his colleagues for 8 days at 1125°C in a $10^{-5}$mm Hg vacuum indicated the absence of exsolved augite. However, sharp diffraction patterns could still be observed, indicating a crystalline homogenization. This may be due to a more disordered Mg and Fe distribution between the crystal sites. (8)

2. Experiments Involving Magnetic Decoration. Through the application of new techniques for the study of ferromagnetic domain boundaries, we have now been able to directly visualize differences between the bands and the interband regions. By condensation of evaporated Fe, small ferromagnetic single crystals can be formed. These preferentially deposit on the traces of Block walls on the surface of ferromagnetic crystals. This technique yields a permanent picture of the domain pattern. In view of the increased penetration power of the 200 kV electron microscope, no replica patterns were needed.

Our preliminary examinations of the "decorated" specimens at room temperature indicated the presence of magnetic domains related to the band structures, probably indicative of an enhanced concentration of iron within the electron dense thin exsolution lamellae. This supports our earlier suggestion that the band structures are similar to images of magnetic domain walls seen in thin layers of ferromagnetic materials. (1) It also provides evidence for the assumption that the rather unusual magnetic ordering in the lunar pyroxene could be due to iron-iron clustering in the single crystal domains. (5, 13)

The domains and any lattice imperfections can also be directly resolved through the application of cryo-electron microscopy at liquid helium temperatures. In this way, we were able to observe trapped flux patterns by Träuble and Essmann's decoration techniques (9,10) or through the distribution and detection of regular arrays of flux lines in the electron diffraction patterns of the lunar clinopyroxenes.

3. Low Temperature Experiments. Additional studies were carried out at low temperatures in the liquid helium range. Examination of the native yellow-green pigeonite specimens at 1.8° to 4.2°K revealed substructures in certain local areas which were not present in the corresponding regions of the same specimens at 290°K. (7,8) Together with related Lorentz microscopy findings, these results are tentatively interpreted as being consistent with the assumption of greater ordering of iron atoms in these areas.

4. Color Recording Techniques. In addition to the conventional high resolution plates, new types of photo-sensitive recording media, developed in collaboration with my colleague Charles Hough, based on the differential cross-linking of organic polymers and organometallic layers were used to obtain high resolution color electron micrographs. Images could be recorded as thickness variations of the cross-linked and insoluble polymer layers, and interference colors are then presented as a function of the image-dependent thickness variations. In some cases, this method of color recording makes it possible to quantitatively analyze electron beam interaction with the specimens.

In addition, this color translation process brings out heretofore unobservable structural detail by translating normally indistinguishable shades of gray into clearly distinct color differences.

Using similar developmental electron optical techniques, we

were also able to make "synthetic aperture" color holograms of three-dimensional image quality from a series of stereo micro-holograms. Preliminary results of these studies indicated that they may provide new insights into the three-dimensional relationships within these lunar specimens. (8)

5. Lunar Cooling History. This observation of extensive exsolution in the apparently homogeneous, calcium-poor pigeonite and correlated Mössbauer absorption studies suggest that this coarse-grained lunar basalt must have been subjected to an exceedingly slow cooling rate in the subsolidous temperature range. (11)

Virgo, Hafner and Warburton have observed considerable long range ordering within the crystal fragments at temperatures of 810° to 480°C. Taken together with the submicroscopic unmixing and ordering of Mg and Fe among the cation sites, this indicates that annealing of the rock from the surface of the moon took place at about 600°C. (11) Hawaiian lavaflows and certain other terrestrial orthopyroxenes do not demonstrate this type of characteristic long range ordering when cooled to 480°C. However, similar effects are known in ancient terrestrial igneous rock, particularly in dykes cutting genisses in continental shield regions, where the rock has been rapidly cooled and then reheated or held for long periods of time at moderate temperatures.

## STUDIES OF APOLLO 14 AND 15

Moon rocks from Apollo 15, chemically zoned clinopyroxene 15535, of heterogeneous composition $En_{49.80\pm9,90}Fs_{34.46\pm5.52}Wo_{15.60\pm6.50}$, and pigeonite fraction 15076 of homogeneous composition $En_{65.84\pm1.49}Fs_{27.83\pm0.90}Wo_{5.83\pm0.58}$ were examined by electron optical techniques. (12) Our sample 15535 of 16 mg of small grains of chemically zoned clinopyroxene was found to contain different mineral

phases, still intergrown. We crushed the grains to a finer size and removed the black opaque particles. The remaining grains were then cleaved further to sizes of 1μ to 5μ in diameter by the cleavage preparation technique. Again, the black impurities were removed with very fine tungsten needles by micromanipulation, leaving a purer clinopyroxene. The crystalline layers, varying in thickness from 200Å to 600Å, were placed on ultrathin carbon coated formvar films about 150Å thick.

These specimens were examined by both 75 kV and 200 kV electron microscopy and selected-area electron diffraction at room temperature and at 4.2°K. Of all the crystals examined, we observed exsolution lamellae in 20% of sample 15535 and in 32% of sample 15076. The remainder showed no band structures. The lamellar structure consisted of a dense band and an interband spacing which are single crystal domains oriented mainly along the (001) or (100) with uniform boundaries.

In sample 15535, dense bands with widths of 25Å to 550Å (average width 250Å) were observed directly. Corresponding interband regions ranged in width from 50Å to 900Å, with an average width of 330Å. Approximately 43% of the total crystal volume of this specimen sample is dense band.

In pigeonite sample 15076, the width of the dense bands varied from 100Å to 1800Å, with an average width of 1000Å. The interband width ranged from 300Å to 6200Å with an average width of 3100Å. In this specimen sample, approximately 34% of the total crystal volume is dense band.

These measurements of the dense band widths of sample 15535 are slightly smaller than those from Apollo 11 pyroxene 10044,25 which averaged between 250Å and 300Å in width. The interband measurements

from sample 15535 were about the same as those from Apollo 11 pyroxene 10044,25, which ranged from ca. 100Å to 700Å with an average of ca. 330Å. Of the total crystal volume, approximately 43% is dense band in sample 15535 and 49% is dense band in sample 10044,25.

The band widths of specimens from sample 15076, averaging ca. 1000Å in dense band and ca. 3100Å in interband width, are much greater than those measured by the same techniques in Apollo 12 sample 12021. These latter averaged ca. 230Å in the dense band and ca. 650Å in the interband. However, the two pigeonite samples show a similarity in their percentage of dense band content of the total crystal volume, being 24% in 15976 and 26% in 12021.

A lattice spacing of 2.51Å was also detected within these bands in both of the Apollo 15 specimens examined by selected-area electron diffraction. The spacing is oriented mainly parallel to the (001) or (100). This is also the direction of the domain boundary of the band.

Dr. Virgo is now completing the x-ray crystallography studies of sample 15076. To date, he has been able to confirm the pigeonite structure, but in the positions where the exsolved phase augite occurs, he has gotten only diffuse streaks. (13)

Several researchers have reported extremely thin exsolution lamellae in 15076 pigeonite not more than 1 or 2 unit cells thick. ($14,15$) This is in contrast to our recorded observation of 1000Å wide bands within the specimen sample. Thus our findings may represent a repeat distance of these fine lamellae or some feature other than lamellae which has heretofore been unobserved. It should also be considered that our high resolution electron micrographs possibly depict a small percentage of large exsolution lamellae which are not detectable by the photography techniques used by Virgo in the precision study of the six crystals from 15076. This work is now being completed and

will be reported in detail in a subsequent paper now in preparation.

Virgo has also been able to obtain evidence of the P21/c domain size data on exsolution phenomenon and $Fe^{2+}$, Mg exchange between the crystal sites which are necessary to any interpretation of the cooling history of this particular basalt. (13) We are now completing the final stages of this comprehensive research. The results will be presented in a detailed paper which will present a comparison and correlative evaluation of Dr. Virgo's crystallographic data and our own electron optical results. A copy of this manuscript will be sent to the proper NASA offices when the data is complete.

## CONCLUSION

Electron microscopy and diffraction studies of specimens from Apollo 11, 12, 14 and 15 have opened the way for yet more extensive research which must be conducted before we can have a true understanding of the intrinsic atomic organization of the lunar pyroxenes and their bearing on the cooling history of the moon. Essentially, the data obtained through systematic correlated investigations during our two-year research program, have provided us with a "finger-printing" of the lunar pyroxenes. The extensive exsolution lamellae, which can only be directly observed through the use of electron optical techniques, such as those reported here, and the results of correlated Mössbauer absorption studies and related x-ray investigations suggest slow cooling rates, or possibly annealing, at temperatures in the subsolidous range.

In addition, the possible correaltion of the band structures with the magnetic domains observed in thin layers of ferromagnetic materials and the possible iron-iron clustering, which we first suggested in our studies of Apollo 11 samples 10044,25 three years ago, can now be systematically studied through the application of new techniques.

Thus, we have begun to unravel the intrinsic chemical compositions of the submicroscopic domains within the lunar clinopyroxenes, but we have only begun.

Through continued correlated electron microscope and diffraction studies, supplemented and extended by other techniques, it may be possible to come eventually to a better understanding of the complex organization of the pyroxenes and their significance.

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1. First Lunar Science Conference, Houston, Texas. January, 1970.
2. Ninth National Meeting of the Society of Applied Spectroscopy, New Orleans, Louisiana, October, 1970.
3. Second Lunar Science Conference, Houston, Texas. January, 1971.
4. Third Lunar Science Conference, Houston, Texas. January, 1972.
5. Fourth Lunar Science Conference, Houston, Texas. March, 1973.

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## LECTURES, EXHIBITS AND RADIO & T.V. PRESENTATIONS OF LUNAR WORK

1. Fernandez-Moran, H. "Wissenschaftliche Untersuchungen und Aufnahmen bei der letzten Apollo-Missions." Paper presented at Biophysics Colloquium, Institut fur Biochemie, Gesellschaft fur Strahlen und unweltforschung MbH, West Germany. June, 1972.

2. C.O.S.P.A.R. Exhibit touring Russia-at the invitation of Dr. John Pomeroy.

3. C.O.S.P.A.R. Exhibit in Seattle, Washington-at the invitation of Dr. John Pomeroy.

4. Special exhibit at the Musuem of Science and Industry, Chicago, Illinois in connection with the installation of the permanent exhibit of the Apollo 8 Command Module.

5. W.I.N.D. Radio's "CONTACT"-Discussion program on research with the Apollo Samples, Chicago, Illinois. November, 1971.

6. "Perspectives"-T.V. program sponsored by The University of Chicago-Presentation of work with the lunar samples. May, 1972.