ɪNN and G. SIMMONS

ND ACRONYMS

| | |
|---|---|
| plg | plagioclase |
| pxf | pyroxferroite |
| rut | rutile |
| spn | spinel |
| tro | troilite |
| ulv | ulvöspinel |
| ves | vesicle |
| vug | vug |

to return the lunar samples to earth.
ıstronauts out of the LM and on to the

he moon.

group advised NASA on all aspects of

This group examined the lunar samples
.in data needed for the intelligent distri-

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# Armalcolite: A new mineral from the Apollo 11 samples*

A. T. ANDERSON
Department of Geophysical Sciences, University of Chicago, Chicago, Illinois 60637

T. E. BUNCH
Space Science Division, NASA, Ames Research Center, California 94035

E. N. CAMERON
Department of Geology and Geophysics, University of Wisconsin, Madison, Wisconsin 53706

S. E. HAGGERTY, F. R. BOYD and L. W. FINGER
Geophysical Laboratory, Carnegie Institution of Washington, Washington, D.C. 20008

O. B. JAMES
U.S. Geological Survey, Washington, D.C. 20242

K. KEIL and M. PRINZ
Department of Geology, Institute of Meteoritics, University of New Mexico, Albuquerque, New Mexico 87106

and

P. RAMDOHR and A. EL GORESY
Max-Planck Institut für Kernphysik, Heidelberg, Germany

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**Abstract**—Armalcolite, $(Fe^{2+}_{0.5}Mg_{0.5})Ti_2O_5$, is a new mineral from Tranquillity base with the pseudobrookite structure ($a = 9{\cdot}743$ Å, $b = 10{\cdot}024$ Å, $c = 3{\cdot}738$ Å, $V = 365{\cdot}077$ Å$^3$). Theoretical density is 4·64 g/cm$^3$. The mineral is opaque, blue-gray in reflected light, and distinctly anisotropic. Reflectivity values in air at 450 nm are $R_1 = 14{\cdot}1$ per cent, $R_2 = 15{\cdot}2$ per cent, and at 640 mn are $R_1 = 13{\cdot}0$ per cent, $R_2 = 14{\cdot}1$ per cent. Synthesis of the end members $Fe^{2+}Ti_2O_5$ and $MgTi_2O_5$ and armalcolite has been accomplished at 1300°C.

## INTRODUCTION

ARMALCOLITE ($Fe_{0.5}Mg_{0.5}Ti_2O_5$), a new magnesium-rich opaque oxide related to the pseudobrookite series, was discovered independently by six research groups in their examination of different samples of the lunar material collected from Mare Tranquillitatis.

Compositionally the mineral is intermediate between two end members, $Fe^{2+}Ti_2O_5$ and $MgTi_2O_5$. Neither of these end members is known to occur naturally. Armalcolite is proposed for the composition $FeMgTi_2O_5$; new mineral names for the end members are not proposed. Ferro-armalcolite is rich in $Fe^{2+}TiO_5$, and magnesian

* This report by S. E. HAGGERTY is a compilation of analytical data on a armalcolite reported by six groups of investigators at the Apollo 11 conference and published in *Science*, January 30, 1970. Discussion of the paragenesis and origin of this mineral has been omitted, thus allowing free expression and interpretation by the various authors in their individual papers.

armalcolite is rich in $MgTi_2O_5$. Analyses of lunar armalcolite show that in addition to $Fe^{2+}$, Ti and Mg, minor concentrations of Cr, Al, Mn, V, Ca and Zr are present. The phase is sufficiently different from related minerals in terrestrial occurrences to warrant a new mineral name.

The name armalcolite is derived from the initial letters of the three astronauts, Neil A. *Arm*strong, Edwin E. *Al*drin and Michael *Col*lins, who were responsible for collection and return of the Apollo 11 material. Type material is deposited at the Lunar Science Institute, Houston, Texas.

## OCCURRENCE

Isolated grains of armalcolite have been identified in the crystalline rocks (10022-37; 10071-28) and in rock fragments within the microbreccias (10059-27; 10067-8; 10068-25; 10084-64). In polished thin sections, grains were observed to be generally rectangular in outline, with the longest dimensions in the range 100–300 $\mu$m. In all but one case (an inclusion in a feldspar-rich fragment, 10084-12) the mineral is mantled by a well defined overgrowth of ilmenite. In all cases armalcolite is optically homogeneous and free of exsolution or alteration products.

Table 1. Reflectivity values of armalcolite for selected wavelengths between 450 and 640 nm* in air

| | Armalcolite | |
|---|---|---|
| Wavelength (nm) | $R_1$ | $R_2$ |
| 450 | 14·1 | 15·2 |
| 470 | 14·0 | 15·0 |
| 500 | 13·8 | 14·7 |
| 520 | 13·7 | 14·5 |
| 546 | 13·4 | 14·4 |
| 589 | 13·3 | 14·3 |
| 620 | 13·2 | 14·2 |
| 640 | 13·0 | 14·1 |

* All values in per cent. Accuracy $\pm$0·2 per cent Measurements carried out by E. N. Cameron, University of Wisconsin.

## OPTICAL PROPERTIES

Armalcolite is opaque, gray and distinctly pleochroic. Its reflection anisotropy in oil varies from pale gray (minimum) to dark blue-gray (maximum) on rotation of the stage. It is free of internal reflections, with estimated white light reflectivity values in oil in the range 15–18 per cent. Quantitative reflectivity data for selected wavelengths between 450 and 640 nm in air are given in Table 1. The reflectivity and microhardness of armalcolite are less than that of ilmenite (Plate 8).

## CHEMISTRY

Electron microprobe analyses (Table 2) indicate that the new mineral has the generalized formula $Fe^{2+}_{0.5}Mg_{0.5}Ti_2O_5$. The range in major element concentrations reported by the various investigating groups is as follows: 71·1–75·6 per cent $TiO_2$,



Table 2. Electron microprobe analyses for armalcolite

| Investigating group*: Sample No.: | 1 10022-37 Grain $a_1$ | 1 10022-37 Grain $a_2$ | 1 10022-37 Grain $b_1$ | 1 10022-37 Grain $b_2$ | 2 1007128 | 3 100 Grain *a* | 3 100 Grain *b* | 4 10059-27 | 5 10068 | 6 10084-12 |
|---|---|---|---|---|---|---|---|---|---|---|
| $TiO_2$ | 70·9 | 71·6 | 71·2 | 71·0 | 73·4 | 72·36 | 71·63 | 75·6 | 72·0 | 71·9 |
| $Al_2O_3$ | 1·8 | 1·8 | 2·0 | 1·8 | 1·62 | 2·01 | 2·18 | 1·87 | 1·48 | 0·97 |
| $Cr_2O_3$ | 1·3 | 1·3 | 1·25 | 1·02 | 2·15 | 1·37 | 1·38 | 1·81 | 1·94 | 1·26 |
| FeO | 16·9 | 15·7 | 16·0 | 17·3 | 15·3 | 16·95 | 18·01 | 11·9 | 14·7 | 11·32 |
| MnO | 0·02 | 0·02 | 0·03 | 0·03 | 0·08 | 0·02 | 0·05 | | 0·07 | 0·01 |
| MgO | 8·6 | 8·9 | 8·7 | 8·0 | 7·70 | 5·98 | 5·52 | 8·12 | 8·7 | 11·06 |
| CaO | | | | | 0·01 | 0·13 | | | 0·32 | |

Table 2. Electron microprobe analyses for armalcolite

| Investigating group*: Sample No.: | 1 10022-37 Grain $a_1$ | 1 10022-37 Grain $a_2$ | 1 10022-37 Grain $b_1$ | 1 10022-37 Grain $b_2$ | 2 1007128 | 3 100 Grain *a* | 3 100 Grain *b* | 4 10059-27 | 5 10068 | 6 10084-12 |
|---|---|---|---|---|---|---|---|---|---|---|
| $TiO_2$ | 70·9 | 71·6 | 71·2 | 71·0 | 73·4 | 72·36 | 71·63 | 75·6 | 72·0 | 71·9 |
| $Al_2O_3$ | 1·8 | 1·8 | 2·0 | 1·8 | 1·62 | 2·01 | 2·18 | 1·87 | 1·48 | 0·97 |
| $Cr_2O_3$ | 1·3 | 1·3 | 1·25 | 1·02 | 2·15 | 1·37 | 1·38 | 1·81 | 1·94 | 1·26 |
| FeO | 16·9 | 15·7 | 16·0 | 17·3 | 15·3 | 16·95 | 18·01 | 11·9 | 14·7 | 11·32 |
| MnO | 0·02 | 0·02 | 0·03 | 0·03 | 0·08 | 0·02 | 0·05 | | 0·07 | 0·01 |
| MgO | 8·6 | 8·9 | 8·7 | 8·0 | 7·70 | 5·98 | 5·52 | 8·12 | 8·7 | 11·06 |
| CaO | | | | | 0·01 | 0·13 | | | 0·32 | |
| $V_2O_3$ | | | | | $<$0·5 | | | | 0·07 | |
| CoO | | | | | $<$0·02 | | | | | |
| NiO | | | | | $<$0·01 | | | | | |
| CuO | | | | | $<$0·01 | | | | | |
| ZnO | | | | | $<$0·01 | | | | | |
| Totals | 99·52 | 99·32 | 99·18 | 99·15 | 100·25 | 98·82 | 98·77 | 99·30 | 99·28 | 96·52 |
| | | | | | Atomic proportions based on 5 oxygens | | | | | |
| Ti | 1·897 | 1·909 | 1·934 | 1·910 | 1·968 | 1·986 | 1·940 | 2·012 | 1·917 | 1·938 |
| Al | 0·076 | 0·076 | 0·085 | 0·076 | 0·068 | 0·085 | 0·093 | 0·077 | 0·062 | 0·041 |
| Cr | 0·037 | 0·037 | 0·036 | 0·029 | 0·061 | 0·039 | 0·040 | 0·051 | 0·055 | 0·036 |
| Fe | 0·506 | 0·469 | 0·483 | 0·521 | 0·456 | 0·512 | 0·547 | 0·352 | 0·438 | 0·342 |
| Mn | 0·001 | 0·001 | 0·001 | 0·001 | 0·002 | 0·001 | 0·001 | | 0·002 | |
| Mg | 0·459 | 0·473 | 0·468 | 0·430 | 0·409 | 0·322 | 0·299 | 0·428 | 0·462 | 0·595 |
| Ca | | | | | 0·0004 | 0·005 | | | 0·012 | |
| V | | | | | | | | | 0·002 | |
| Totals | 2·976 | 2·965 | 3·000 | 2·967 | 2·914 | 2·920 | 2·920 | 2·950 | 2·952 | |

* Investigating groups: (1) University of Chicago, (2) Geophysical Laboratory, (3) University of Wisconsin, (4) Max–Planck Institut, (5) University of New Mexico, (6) U.S. Geological Survey.

The positions of the analytical data points by Anderson (group 1) for grains *a* and *b* are shown in Fig. 2.

11·90–18·01 per cent FeO, 5·52–11·06 per cent MgO. Minor amounts of $Cr_2O_3$ (1·3–2·15 per cent), $Al_2O_3$ (1·48–2·18 per cent), MnO (0·01–0·08 per cent), and CaO (0·01–0·32 per cent) were also reported. The variations and spread in major element concentrations, in terms of the end members $Fe^{2+}Ti_2O_5$ and $MgTi_2O_5$, are graphically expressed in the ternary diagram $FeO–MgO–TiO_2$ in Fig. 1.. Detailed analyses by

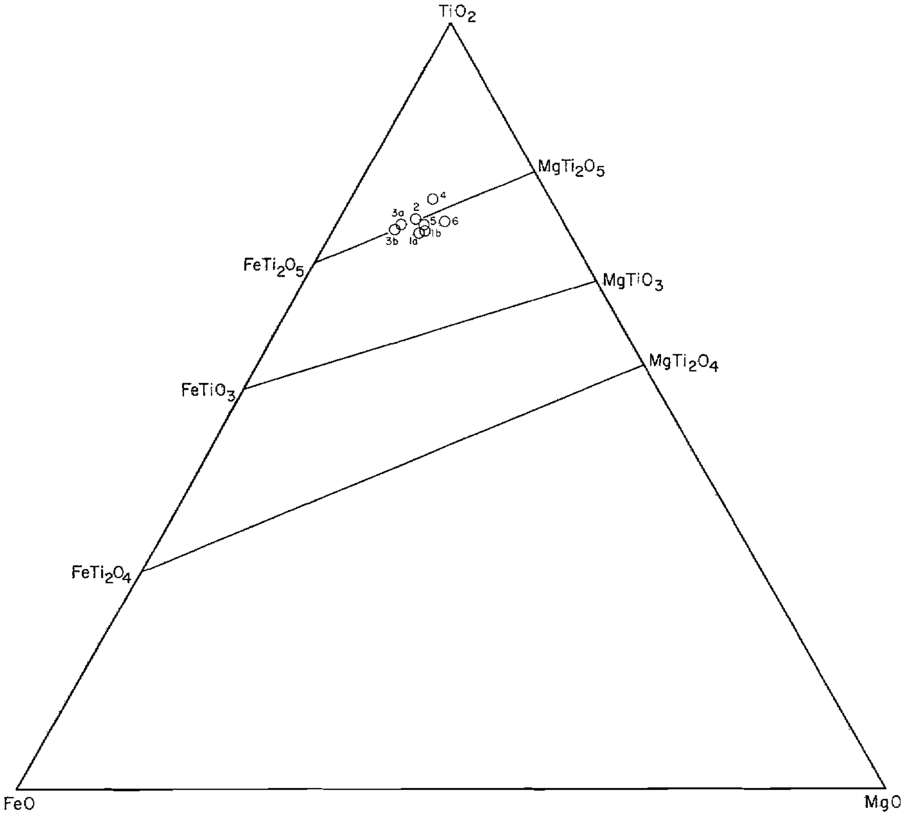



Fig. 1. Armalcolite analyses (wt. %) plotted on the join $FeTi_2O_5–MgTi_2O_5$ in the system $FeO–MgO–TiO_2$. The tie lines represent possible solid solution series. Investigating groups: (1) University of Chicago, (2) Geophysical Laboratory, (3) University of Wisconsin, (4) Max-Planck Institut, (5) University of New Mexico, (6) U.S. Geological Survey.

ANDERSON (Table 2, Fig. 2) indicate that armalcolite in section 10022-37 is unzoned, but similar electron microprobe analyses by KEIL, PRINZ and BUNCH (Fig. 3) of an armalcolite grain in section 10059-27 show slight core-to-boundary variations in Fe, Ti and Mg; in both of these examples armalcolite is mantled by ilmenite and apparent zoning may be due to geometry effects.

## SYNTHESIS

Thermal stability experiments similar to those described by HAGGERTY and LINDSLEY (1970) for the series $Fe_2TiO_5–FeTi_2O_5$ are in progress for members of the join $FeTi_2O_5$ (F)–$MgTi_2O_5$ (M) at the Geophysical Laboratory. LINDSLEY (1965)




Fig. 2. Armalcolite (medium gray
probe analyses were carried out a
Anderson. These data are listed
immersi

and HAGGERTY and LINDSLEY (197
the $FeTi_2O_5$ end member breaks
predicted decomposition products f
whereas intermediate members sho

Synthetic armalcolite with com
end members $FeTi_2O_5$ and $MgTi_2O$
the intermediate members represen
The experimental products have a
polish. The synthetic minerals are
in tones of pale gray with a tan tin
phases are comparable with lunar
texture with some well developed lat

* Experiments were carried out in se
quenched. Reactants were chemically pure
thoroughly mixed and ground under alcoh


Fig. 2. Armalcolite (medium gray) rimmed by ilmenite (light gray). Electron microprobe analyses were carried out at points 1 and 2 in each of grains *a* and *b* by A. T. Anderson. These data are listed in Table 2 (group 1). Rock section 10022-37. Oil immersion, plane polarized light.

and HAGGERTY and LINDSLEY (1970) have established in reversible experiments that the $FeTi_2O_5$ end member breaks down to $FeTiO_3 + TiO_2$ at 1140° ± 10°C. The predicted decomposition products for the end member $MgTi_2O_5$ are $MgTiO_3 + TiO_2$, whereas intermediate members should break down to $FeTiO_3$–$MgTiO_3$ ss + $TiO_2$.

Synthetic armalcolite with compositions $F_{40}M_{60}$, $F_{50}M_{50}$, $F_{60}M_{40}$, as well as the end members $FeTi_2O_5$ and $MgTi_2O_5$, have been successfully synthesized* at 1300°C; the intermediate members represent the span of analyses for the lunar armalcolite. The experimental products have a black metallic luster and provide an excellent polish. The synthetic minerals are gray and distinctly anisotropic in oil immersion in tones of pale gray with a tan tint. Estimated reflectivity values for the synthetic phases are comparable with lunar armalcolite (15–18 per cent). An equigranular texture with some well developed laths, and a large number of twinned grains (Fig. 4)

* Experiments were carried out in sealed, evacuated silica glass tubes reacted for 2 hr. and quenched. Reactants were chemically pure and dried Fe, $Fe_2O_3$, $TiO_2$, and MgO. Compositions were thoroughly mixed and ground under alcohol for 2–4 hr before firing.

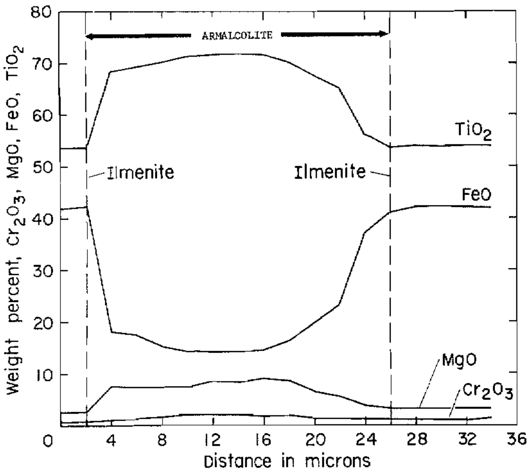



Fig. 3. Electron microprobe traverse across ilmenite–armalcolite–ilmenite, showing variations in $Cr_2O_3$, MgO, FeO and $TiO_2$ contents (University of New Mexico group).

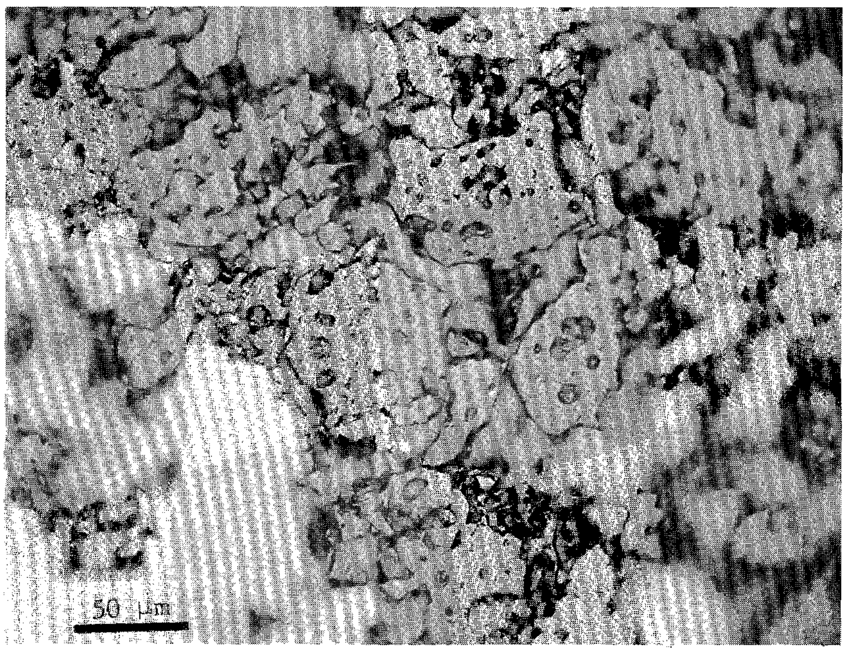



Fig. 4. An intergranular aggregate of synthetic armalcolite produced at 1300°C. Note the bladed form of some of the grains and also the twinned crystals. Oil immersion and polarized light, nicol ×10° (S. E. Haggerty).



are produced in each of the e presented in Table 3.

X-R

A small amount of armalcoli 10071-28 using the microsampl Debye–Scherrer X-ray pattern us material. These data (Geophysi

Table 3. X-ray d

| | Synthetic armalcolite | |
|---|---|---|
| *hkl* | *d* (obs) | *d* (c |
| 020 | 5·019 | 5·0 |
| 200 | 4·879 | 4·8 |
| 101, 220 | 3·493 | 3·4 |
| 230 | 2·762 | 3·7 |
| 301 | 2·452 | 2·4 |
| 400 | 2·438 | 2·4 |
| 131 | 2·415 | 2·4 |
| 240 | 2·233 | 2·2 |
| 420 | 2·194 | 2·1 |
| 430 | 1·972 | 1·97 |
| 250 | 1·858 | 1·85 |
| 341 | 1·755 | 1·75 |
| 060 | 1·675 | 1·67 |
| 521 | 1·634 | 1·63 |
| 600 | 1·625 | 1·62 |
| 232, 620, 450 | 1·549 | 1·54 |
| 531 | 1·537 | 1·53 |

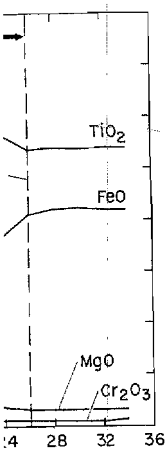
$a = 9{\cdot}752 \pm 0{\cdot}003$ Å
$b = 10{\cdot}048 \pm 0{\cdot}003$ Å
$c = 3{\cdot}736 \pm 0{\cdot}004$ Å
$V = 366{\cdot}071 \pm 0{\cdot}322$ Å$^3$

* Diffractometer patterns and an i $(Fe^{2+}_{0.5}Mg_{0.5}Ti_2O_5$. A Debye–Scherrer patte taminated by adhering ilmenite. Reflection (Geophysical Laboratory).

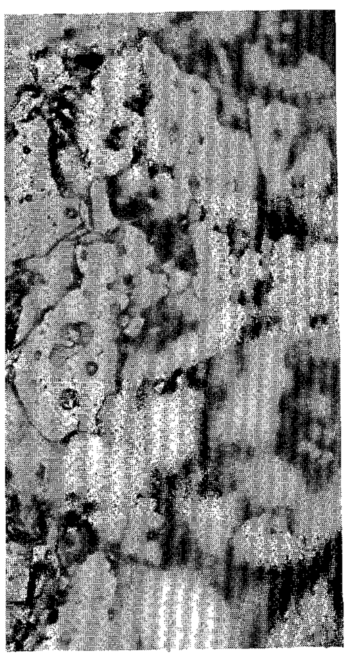
electron microprobe analysis for th fractometer data on synthetic arm CuKα radiation. A powdered Si edge ($a = 5{\cdot}4301$ Å) was previous Hägg–Guinier camera and a Man unit cell was initiated for both the proximated from PAULING's (1930) and unit-cell volume data for lunar a $FeTi_2O_5$ and $MgTi_2O_5$ are presented

The theoretical density of armalc for synthetic armalcolite, $F_{0.5}Mg_{0.5}$ volume data presented above.

al.




ite–armalcolite–ilmenite, showing
Jniversity of New Mexico group).



armalcolite produced at 1300°C.
the twinned crystals. Oil immersion
S. E. Haggerty).



are produced in each of the experiments. X-ray diffraction data for $F_{50}M_{50}$ are presented in Table 3.

## X-Ray Diffraction Data

A small amount of armalcolite was extracted from the surface of polished section 10071-28 using the microsampling technique described by Kingston (1966). A Debye–Scherrer X-ray pattern using Mn filtered $FeK_\alpha$ radiation was obtained on this material. These data (Geophysical Laboratory) are presented in Table 3, and the

Table 3. X-ray data for synthetic and lunar armalcolite*

| Synthetic armalcolite | | | | Lunar armalcolite | | |
|---|---|---|---|---|---|---|
| *hkl* | *d* (obs) | *d* (cal) | *I* | *d* (obs) | *d* (cal) | *I* |
| 020 | 5·019 | 5·024 | 40 | | | |
| 200 | 4·879 | 4·876 | 80 | | | |
| 101, 220 | 3·493 | 3·499 | 100 | 3·468 | 3·483 | 100 |
| 230 | 2·762 | 3·761 | 80 | 2·763 | 2·755 | 25 |
| 301 | 2·452 | 2·452 | 10 | 2·454 | 2·452 | 25 |
| 400 | 2·438 | 2·438 | 5 | | | |
| 131 | 2·415 | 2·416 | 10 | 2·414 | 2·428 | 10 |
| 240 | 2·233 | 2·233 | 15 | 2·235 | 2·228 | 15 |
| 420 | 2·194 | 2·193 | 4 | 2·199 | 2·191 | 15 |
| 430 | 1·972 | 1·971 | 17 | 1·958 | 1·968 | 80 |
| 250 | 1·858 | 1·858 | 8 | | | |
| 341 | 1·755 | 1·755 | 8 | 1·751 | 1·752 | 10 |
| 060 | 1·675 | 1·675 | 10 | 1·669 | 1·669 | 10 |
| 521 | 1·634 | 1·635 | 28 | 1·632 | 1·634 | 10 |
| 600 | 1·625 | 1·625 | 13 | | | |
| 232, 620, 450 | 1·549 | 1·546 | 18 | | | |
| 531 | 1·537 | 1·536 | 8 | | | |
| | $a = 9{\cdot}752 \pm 0{\cdot}003$ Å<br>$b = 10{\cdot}048 \pm 0{\cdot}003$ Å<br>$c = 3{\cdot}736 \pm 0{\cdot}004$ Å<br>$V = 366{\cdot}071 \pm 0{\cdot}322$ Å$^3$ | | | $a = 9{\cdot}743 \pm 0{\cdot}03$ Å<br>$b = 10{\cdot}024 \pm 0{\cdot}02$ Å<br>$c = 3{\cdot}738 \pm 0{\cdot}03$ Å<br>$V = 365{\cdot}077 \pm 0{\cdot}619$ Å$^3$ | | |

* Diffractometer patterns and an internal Si standard were used for synthetic armalcolite ($Fe^{2+}_{0.5}Mg_{0.5}Ti_2O_5$. A Debye–Scherrer pattern was used for the lunar armalcolite. This pattern was contaminated by adhering ilmenite. Reflections considered to be due to the armalcolite alone are presented (Geophysical Laboratory).

electron microprobe analysis for the same grain is listed in Table 2 (group 2). Diffractometer data on synthetic armalcolite (Table 3) were obtained with Ni-filtered $CuK\alpha$ radiation. A powdered Si internal standard was used whose absolute cell edge ($a = 5{\cdot}4301$ Å) was previously measured (J. Whitney, Dupont Co.) with a Hägg–Guinier camera and a Mann photometer. Refinement by computer of the unit cell was initiated for both the natural and synthetic armalcolite on values approximated from Pauling's (1930) structure for pseudobrookite. Lattice constants and unit-cell volume data for lunar and synthetic armalcolite and for the end members $FeTi_2O_5$ and $MgTi_2O_5$ are presented in Table 4.

## Density

The theoretical density of armalcolite is 4·94 g/cm³. This is the value determined for synthetic armalcolite, $F_{0.5}Mg_{0.5}Ti_2O_5$; the calculation is based on the unit-cell volume data presented above.

Table 4. Comparison of lattice constants and unit-cell volume data for natural and synthetic armalcolite and for the members $FeTi_2O_5$ and $MgTi_2O_5$

| | $a$ (Å) | $b$ (Å) | $c$ (Å) | $V$ (Å$^3$) |
|---|---|---|---|---|
| Lunar armalcolite* | 9·743 | 10·024 | 3·738 | 365·077 |
| Synthetic armalcolite† | 9·752 | 10·048 | 3·736 | 366·071 |
| Synthetic $FeTi_2O_5$‡ | 9·798 | 10·041 | 3·741 | 368·046 |
| Synthetic $MgTi_2O_5$§ | 9·8 | 10·0 | 3·7 | 362·60 |

* Rock section 10071-28 (Type A lava).
† Geophysical Laboratory.
‡ AKIMOTO *et al.* (1957).
§ ZHDANOV and RUSAKOV (1952). ASTM Index File No. 9-16.

## DISCUSSION

Neither $Fe^{2+}Ti_2O_5$ ("ferropseudobrookite")* nor $MgTi_2O_5$ ("karrooite")* has previously been found as a discrete end member in nature. Both phases have been synthesized, but a complete solid solution series between them has not yet been experimentally demonstrated. The formula of pure pseudobrookite is $Fe^{3+}{}_2TiO_5$, and solid solution of $MgTi_2O_5$ or $Fe^{2+}Ti_2O_5$ with this phase is accomplished by the coupled substitution (Mg, $Fe^{2+}$)–Ti for $Fe^{3+}$–$Fe^{3+}$. $Fe^{2+}Ti_2O_5$ and $MgTi_2O_5$ have been found as subordinate components in various pseudobrookite-like minerals in terrestrial occurrences (SMITH, 1965). An example of such a mineral is kennedyite ($Fe^{3+}{}_2MgTi_3O_{10}$), a phase found in the Karroo basalts (VON KNORRING and COX, 1961). Kennedyite is isostructural with pseudobrookite and contains 28·77 per cent $Fe_2O_3$ and 60·33 per cent $TiO_2$. The MgO content of kennedyite (6·45 per cent) is within the range of the lunar phase (5·52–11·06 per cent), but the apparent absence of $Fe_2O_3$ and higher $TiO_2$ content in armalcolite make its composition distinctive.

A low content of $Fe_2O_3$ in armalcolite is obviously essential for its recognition as a new mineral. There are several reasons that lead to the strong presumption that the concentration of $Fe^{3+}$ is very small in this lunar phase. If the electron probe analyses of armalcolite are recalculated to an ionic formula where $O = 5$ (Table 2), Ti is found to vary between 1·897 and 2·012 (mean 1·941), in comparison with the maximum possible value of 2·00. Inasmuch as half of any $Fe^{3+}$ present must replace Ti, there is obviously not much possibility of significant $Fe^{3+}$. Moreover, native iron coexists with armalcolite in all samples. In conclusion, therefore, the presence of metallic iron, the occurrence of $Cr^{2+}$ in olivine (HAGGERTY *et al.*, 1970), the compositions of other solid solutions (e.g. titanian chromite and chromian ulvöspinel; AGRELL *et al.*, 1970), and the lack of ferric iron in chemical analyses of lunar rock samples (e.g. ROSE *et al.*, 1970) indicate that these rocks crystallized under highly reducing conditions and that ferric iron is absent.

* "Ferropseudobrookite" (AGRELL and LONG, 1960) and "karrooite" (VON KNORRING and COX, 1961) have no standing as mineral names.

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