Tungsten-bearing yttrobetafite in lunar granophyre

CHARLES MEYER

Planetary Materials Branch SN2, NASA Johnson Space Center, Houston, Texas 77058, U.S.A.

S. VINCENT YANG

Lockheed EMSCO, 2400 NASA Road 1, Houston, Texas 77058, U.S.A.

ABSTRACT

Several relatively large grains (up to 200 μ m) of metamict yttrobetafite have been discovered in a small clast of granophyre in lunar breccia 14321. The mineral formula is calculated as (Ca_{0.61}Y_{0.42}REE_{0.32}Fe_{0.25}Pb_{0.18}Th_{0.12}U_{0.07}La_{0.02}Mn_{0.01})(Ti_{1.24}Nb_{0.58}Fe_{0.14}Ta_{0.02}-Zr_{0.01}W_{0.01})O_{6.80}. This metamict phase contained a significant concentration of initial PbO (3.0 ± 0.2 wt%) at time of formation.

This clast of lunar granophyre may have formed under relatively oxidizing conditions. The yttrobetafite contains significant concentrations of WO₃ (0.44 wt%) and Fe₂O₃ (2.5 wt%). Ilmenite contains high Nb₂O₅ (2.3 wt%).

INTRODUCTION

Numerous small fragments of granophyre have been recognized in lunar soils and breccia (Drake et al., 1970; Meyer, 1972; Ryder et al., 1975; Glass, 1976; Warren et al., 1983, 1987). These fragments of lunar granophyre are characterized by intergrowths of silica with Ba-rich potassium feldspar and various amounts of plagioclase. Ilmenite, fayalite, Fe-rich pyroxene, apatite, zirkelite, and/ or zircon are sometimes present as minor minerals. Hydrous phases have not been reported in lunar granophyre, and it is surprising that new minerals have not been discovered in these trace-element-rich lunar rock fragments.

While searching thin sections of lunar granophyre for zircons that could be dated by the ion-microprobe method, we have discovered several relatively large, homogeneous grains of an unusual mineral phase containing unusually large amounts of Nb, REEs, U, Th, and other elements. This unknown phase was found in a thin section of a coarse-grained, granophyre clast in lunar breccia 14321. We have tentatively identified this phase as yttrobetafite on the basis of electron-microprobe analyses (EMPA).

The granophyric clast (14321,1027) containing this new lunar mineral has been analyzed by Warren et al. (1983) and Shih et al. (1985). The clast is partly brecciated with a high percentage of vesicular, shock-melted glass. However, in some thin sections of this clast there is an unmelted igneous texture consisting of a coarse intergrowth (1 mm) of silica and potassium feldspar (Fig. 1). In thin section 14321,1494, two large (200 μ m) and many smaller grains of yttrobetafite are intergrown in the large potassium feldspars, and other grains are located in the glass. They are transparent, reddish, and isotropic and provide a bright backscattered-electron (BSE) image (Fig. 2). One large grain, included in potassium feldspar, has a vague hexagonal outline. Most lunar rocks are very reduced. However, late-stage differentiates of a lunar magma ocean may be expected to be more oxidized, because Fe³⁺ and other high-valence ions would be concentrated in the residual liquid.

METHOD

Energy-dispersive X-ray analysis showed the presence of large quantities of Nb, Y, Ti, Ca, Fe, U, Th, and Ce in grains of the unknown phase (Fig. 3). Quantitative EMPA results were obtained with an automated CAMECA electron microprobe using 20 kV and 20-nA beam current. The counting time was 20 s for major elements and 60 s for minor elements. Pulse-height analysis was used to discriminate against high-energy, second-order X-ray lines. Each X-ray peak in the wavelength spectra was carefully scanned to select background positions for quantitative analyses. Slow wavelength scans across the X-ray peaks were also made for each element whose presence was suspected. In this way, some elements were shown to be absent (below 0.1 wt%).

Pure element standards (wires) were used for U, Nb, Ta, W, and Zr. ThO_2 , PbS, ilmenite, and kaersutite standards were used for Th, Pb, Ti, Ca, and Fe. Standardized, REE-containing glasses were used for La, Y, and the REEs (Drake and Weill, 1972). Corrections for absorbtion, fluorescence, and atomic number were accomplished using the CAMECA ZAF procedure.

We have chosen to measure the W concentration using the WM α peak because it can be resolved from the TaM β peak with the PET crystal spectrometer (Fig. 4). The WL α peak has a major interference by YbL β . The UM β X-ray peak was used because ThM β peak partially interferes with the UM α peak at high concentrations. The very narrow width (FWHM = 0.008 Å) of the X-ray peak on the LiF crystal spectrometer allowed interference-free measurement of L α peaks for Ce, Sm, Eu, Gd, and Yb. The other REEs had complicated interference problems.

RESULTS

Table 1 gives the chemical composition of yttrobetafite in 14321,1494 as determined by EMPA. For all elements, the standard deviation of 13 analyses of different spots



Fig. 1. Backscattered-electron image illustrating the texture of granophyre clast 14321,1027 with intergrown silica (gray) and Ba-rich potassium feldspar (bright). The black is epoxy, and the speckled areas are recrystallized "glass." The scale bar is 200 μ m.

on several grains was less than or equal to the standard deviation based on counting statistics for an individual analysis. The centers and margins of yttrobetafite were found to be homogeneous for all elements. High-contrast, BSE images of each grain were also uniform, confirming that the grains are homogeneous between the analyzed spots. There are 25 elements in quantities greater than 0.1 wt% in this mineral phase. Na, F, Si, Sc, Mo, and other elements were shown to be absent (less than 0.1 wt%) using slow wavelength scans across the position of their X-ray peaks.

The REE pattern of yttrobetafite was determined by measuring the concentrations of Ce, Sm, Eu, Gd, and Yb (Fig. 5) using the electron microprobe. The total REEs were then calculated using interpolated values of the other REEs from this chondrite-normalized pattern. Altogether, there is a total of 13 ± 2 wt% REE oxide in this phase. The deep negative Eu anomaly is a characteristic signature of trace-element-rich lunar samples. The shape of the REE pattern is generally consistent with the V-shaped pattern of the whole clast (Warren et al., 1983), but the elemental concentrations are, of course, much higher. This granophyre clast also contains apatite and zircons as additional sites for REEs.

We have found that there is a significant concentration of W in lunar yttrobetafite (WO₃ = 0.44 wt%). We have also determined that the ilmenite in this granophyre clast has an unusually high concentration of Nb (Table 2). Lunar ilmenite usually contains less than 0.2 wt% Nb.

DISCUSSION

Nb, Ta, and W are trace elements in lunar samples that are not generally concentrated into mineral phases. However, relatively high Ta values have been reported for lunar granites and felsites (Table 3), and this would indicate that these rocks would contain high Nb. However, the only Nb-rich mineral phases reported in lunar samples include a single $5-\mu m$ grain of "pyrochlore" in 76535, "phase beta" in 12013, and niobian rutile (Table 4). Quick et al. (1981) have previously reported up to 1 wt% Nb in ilmenite in lunar breccia 12013.

Schreiber (1977) has experimentally studied the valence of several multivalent elements under lunar redox conditions. Most lunar samples crystallized under re-



Fig. 2. Backscattered-electron image of thin section 14321,1494. All of the bright grains are yttrobetafite. The Ba-rich potassium feldspar is light gray, silica is dark, epoxy is black, and recrystallized glass is speckled. The large equant grain of yttrobetafite has vague hexagonal outline and is included in potassium feldspar. The scale bar is 100 μ m.

duced conditions with Fe present as Fe^{2+} . There is evidence that Nb is present as Nb⁴⁺ under reducing conditions (Hughes and Schmitt, 1985). We have chosen relatively reduced oxidation states of the elements listed in our analysis (Table 1). The oxidation states of W, U, and Pb are not well known under these conditions. The exact choice of oxidation state makes a considerable dif-

ference to the calculated amount of oxygen in the mineral formula and also leads to uncertainty in ZAF corrections.

It is difficult to identify a mineral species based on composition alone. For the phase we have analyzed, the sum of the large cations (A) appears greater than the sum of the small cations (B) (Table 1) if all the Fe is assumed to be Fe^{2+} . However, there are no known phases of Ti



Fig. 3. Energy-dispersive X-ray spectra of lunar yttrobetafite in 14321,1494. Ti, Nb, Y, Ca, U, Th, and Fe are the most abundant elements.

	wt%	% STD for 13 analyses	% STD for counting statistics	Atoms
	W170		01210100	71001110
		B cations		
TiO₂	22.69	0.8	0.5	1.24
ZrO ₂	0.26	11	22	0.01
Nb ₂ O ₅	17.67	1.3	1.5	0.58
Ta ₂ O ₅	1.16	6	5	0.02
WO₃	0.44	2.3	5.3	0.01
Fe ₂ O ₃				0.14
				2.00
		A cations		
CaO	7.79	1.3	0.8	0.61
Y ₂ O ₃	10.85	2.5	2.7	0.42
FeO	5.91	1.2	1.3	0.25
MnO	0.20	7.9	11	0.01
UO2	4.33	2.2	2.0	0.07
ThO₂	6.95	1.3	1.9	0.11
PbO	8.94	1.4	2.2	0.18
La₂O₃	0.63	8.6	8.6	0.02
Ce ₂ O ₃	3.13	3.3	4.2	
Sm ₂ O ₃	0.76	3.6	5.7	
Eu ₂ O ₃	0.03			
Gd ₂ O ₃	1.11	3.3	3.3	
Yb ₂ O ₃	1.38	3.0	2.7	0.00
REEs*	13.11			0.32
Total	100.93			2.00
Oxygen				6.80

and/or Nb with an excess of large cations over small cat-

ions. Certainly the apparent excess of large cations rules

out phases such as AB_2O_6 (polycrase, aesehynite). This

limits us to the following possibilities-ABO₃ (perov-

skite), ABO₄ (fergusonite), or $A_{2-x}B_2O_6O_{1-y}$ (pyrochlore).

Of these possibilities, the open pyrochlore structure al-

lows the greatest range of complex elemental substitution

(Aleshin and Roy, 1962; Ewing and Chakoumakos, 1982;

Lumpkin et al., 1986). The pyrochlore structure has been

described as a hexagonal tungsten bronze motif with two

offset layers of octahedrally coordinated cations (Ti, Nb) separated by a layer of large cations (Ca, U, REEs) held in eightfold coordination in large, distorted cubic sites

(White, 1984). This open structure allows a substantial number of vacancies at the large cation site and at one of the anion sites. It is relatively forgiving with respect to substitution of multivalent cations. The low Zr concentration (ZrO₂ = 0.26 wt%) shows that this phase is un-

* The REE content is estimated.

 TABLE 1. Electron-microprobe analysis of yttrobetafite in 14321.1494

TABLE 3. Summary of highest Ta and Hf in lunar samples

	K₂O (wt%)	Ta (ppm)	Hf (ppm)	Reference
12013 light 12033,517 14321,1027 15405,152 73215,43 Ap 14 KREEP	6.8 4.7 8.6 2.1 7.0	12 8 8 10 5 3–4	41 37 14 45 26 18–20	Quick et al., 1981 Warren et al., 1987 Warren et al., 1983 Taylor et al., 1980 Blanchard et al., 1977 Hughes and Schmitt, 1985

related to zirkelite $(AZrB_2O_7)$, which is a closely related structure. The lack of any Si or P shows that this phase is not a residual glass. We conclude that the phase we have analyzed belongs to the pyrochlore group.

There is a systematic nomenclature for minerals in the pyrochlore group (Hogarth, 1977). The Ti-rich end member (2Ti > Nb + Ta) is termed betafite. The species name is determined by the second-most-abundant large cation (A) after Ca, in this case Y. Hence, the phase we have analyzed would be termed yttrobetafite. Kalita et al. (1962) have reported a terrestrial occurrence of yttrobetafite with a composition similar to our analysis.

Optically the yttrobetafite in 14321,1494 is isotropic, which is consistent with the cubic structure of pyrochlore. However, this phase is certain to be metamict because of the large amount of radiation damage caused by the high U and Th contents and the old age of this sample. The radiation dose calculated for this phase is 1.3×10^{18} α/mg . Lumpkin et al. (1986) have shown that microlite (Ta-rich pyrochlore) becomes completely amorphous at doses greater than $10^{17} \alpha/mg$. In order to structurally identify this phase, it would be necessary to reconstitute its original structure by heating it to 700–900 °C. We have not yet attempted this critical experiment, because we have only found this phase in a single thin section and we don't want to destroy it until all nondestructive experiments are finished.

In betafite, the eightfold coordination site (A) can accommodate cations in the size range 0.78 to 1.2 Å (Ca, Y, U, Th, Pb, REEs, La, Fe^{2+}), and the sixfold octahedral

TABLE 4. Summary of Nb-rich minerals in lunar samples (wt%)

	Pyro- chlore 76535 (1)	"Phase beta"		Niobia	n rutile	
		76535 12013	12013	12070 (3)	14162 (4)	72275 (5)
TiO ₂	10.0	22.1	87.9	85.3	73	75.5
Nb ₂ O ₅	50.1	7.9	6.4	7.1	20	14
CaO	6.7	2.9		0.2		
Y_2O_3	4.4	8.9				
FeO	0.9	13.8		0.5	1	
ZrO ₂		17.2		0.7	2	
UO2	2.3	3.6				
ThO ₂	16.2	3.5				
PbO ₂	6.8	4.2				
Ce ₂ O ₃		1.6		0.8		
Cr ₂ O ₃			3.2	2.6	4	5.7

Note: References are (1) Dymek et al., 1975; (2) Haines et al., 1971; (3) Marvin, 1971; (4) Hava et al., 1972; (5) Stoeser et al., 1974; (6) Steele, 1975.

 TABLE 2.
 Electron-microprobe analysis (wt%) of ilmenite in 14321,1613

50.34	50.36	53.64	54.20
0.08	0.08	0.00	0.06
2.30	2.22	1.20	1.34
0.11	0.08	0.75	0.79
46.07	45.41	40.05	39.20
0.41	0.34	0.36	0.35
0.41	0.41	3.88	3.89
99.72	98.90	99.88	99.77
	50.34 0.08 2.30 0.11 46.07 0.41 0.41	50.34 50.36 0.08 0.08 2.30 2.22 0.11 0.08 46.07 45.41 0.41 0.34	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$

l	4	2	3



Fig. 4. X-ray wavelength scan with PET crystal spectrometer of lunar yttrobetafite illustrating the resolution of the $WM\alpha$ peak and the Ta $M\beta$ interference.

site (B) can accommodate cations 0.60 to 0.72 Å (Ti, Zr, Nb, Ta, W, Fe³⁺) (Shannon and Prewitt, 1969). An apparent problem of excess number of large cations (A) over small cations (B) can be solved by assigning some of the Fe as Fe³⁺ that is located in the B site. In this way, it is necessary to assign about $\frac{1}{3}$ of the Fe as Fe³⁺ in order to have balanced stoichiometry (Table 1). Oxidation of other elements (Pb or U) will not yield cations small enough to fit into the B site. Additional amounts of Fe³⁺ could be located in the B site, balanced by vacancies in the A site. Although it is unusual for lunar samples to contain Fe³⁺, one might expect that Fe³⁺ would be concentrated in late-stage lunar differentiate. Indeed, there are no metallic iron grains in this clast.

The presence of a significant concentration of W in yttrobetafite may also be evidence that this mineral assemblage originally crystallized under relatively oxidizing conditions. The partitioning of W between metal and silicate is about 25 to 1 under reducing conditions (Newsom and Drake, 1982), and most pristine lunar samples have less than 1 ppb W. One would not expect to find a measureable concentration of W in lunar granite unless conditions were oxidizing.

Further evidence that this clast crystallized under relatively oxidizing conditions is provided by the unusually high Nb content of ilmenite (Nb₂O₅ = 2.2 wt%). Nb⁴⁺ is too large to substitute for Ti⁴⁺ in ilmenite, but coupled substitution, 2 Ti⁴⁺ = Nb⁵⁺ + (Fe,Cr)³⁺, could explain the presence of Nb in ilmenite. Nb-rich ilmenite is only found in lunar granite.

This clast of granophyre also contains minor zircon and apatite. Prior crystallization of zircon explains why there is a depletion of Zr and Hf in yttrobetafite.

There is more Pb in this phase than can be accounted for by the radioactive decay of U and Th. Shih et al. (1985) have determined an age of about 4.1 Ga for this granophyre clast. Using this age, we recalculate the U,





Fig. 5. Chondrite-normalized REE pattern of lunar yttrobetafite in 14321,1494. Data were collected by electron-microprobe analyses. The Eu anomaly is characteristic of trace-element-rich lunar samples.

Th, and Pb that must have been present at the time of crystallization of this phase (Steiger and Jager, 1977). This calculation shows that this phase originally had about 3.0 \pm 0.2 wt% PbO at 4.1 Ga. Even if this mineral was as old as 4.55 Ga, calculations show that there would be a calculated initial PbO of 1.6 \pm 0.2 wt%. It is most unusual to have a significant quantity of initial Pb present in a lunar mineral. However, the large cation-accommodating A site in pyrochlore can accept either Pb²⁺ (1.18 Å) or Pb⁴⁺ (0.77 Å).

Shih et al. (1985) also reported an Ar age of 3.9 Ga for this clast. This is the same as the age for this lunar breccia and may be the date of the glass in this clast. This clast was certain to have been heated at the time of breccia formation and consequently has had the potential for Pb loss at that time. Some of the grains of yttrobetafite are included in the glass matrix. However, we have found that the Pb concentration is homogeneous in all grains (and at the edges of grains) of yttrobetafite so that Pb loss by volume diffusion is unlikely.

The origin of granophyric rock on the Moon is uncertain. Presumably, silicic lunar rocks are late-stage differentiates of a crystallizing magma ocean (Warren, 1985). The presence of rare, silica-rich glass particles (SiO₂ > 72 wt%) in the lunar soils indicates that granophyric rocks may be exposed on the lunar crust (Glass, 1976). However, the great amount of cratering that has occurred on the lunar surface has obliterated much of the original mineralogy of lunar granophyres so that we cannot be certain if these silicic fragments were from plutonic igneous rocks. The relatively coarse grain size of this granophyre clast and the presence of yttrobetafite may indicate that some lunar granophyre may have been originally formed as pegmatite in the early lunar crust.

The apparently highly oxidized state of this clast of

lunar granophyre is important because it may indicate that late-stage differentiates of a dry magma ocean can become naturally oxidizing during the concentration of the most highly charged cations. During fractional crystallization of a dry magma, the more highly charged ions—such as Fe³⁺, Nb⁵⁺, and W⁶⁺—become highly enriched in the residual liquid.

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APPENDIX TABLE 1. Geneology of granite clast 14321,1027

Sample number	Mass (g)	Description
,1027		Parent clast from slab ,46
.1028	0.14	consumed by Warren et al., 1983
.9046	0.02	residue
.1029	0.02	thin section
.993	0.01	thin section
,1047	0.02	thin section
.1030	0.20	chips
.1034	0.27	chips
.1061	0.12	small chips and fines
.1489	0.01	potted butt
.1493	0.01	thin section
.1494	0.01	thin section with yttrobetafite
,1613	0.05	grain mount with zircon
,1062	0.15	consumed by Shih et al., 1985