

## LAMELLAE OF BADDELEYITE AND Fe-Cr-SPINEL IN ILMENITE FROM THE BASISTOPPEN SILL, EAST GREENLAND

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### ABSTRACT

Ilmenite in the Gabbro Picrite Zone of the Basistoppen sill, East Greenland, contains exsolution lamellae of baddeleyite and Fe-Cr-spinel. The relative proportions of baddeleyite and Fe-Cr-spinel in isolated ilmenite grains indicate that the ilmenite was reduced during subsolidus re-equilibration, although the sill in general was oxidized during cooling. Calculated pre-exsolution compositions for the ilmenite contain up to 2.4% Cr<sub>2</sub>O<sub>3</sub> and up to 7100 ppm Zr. The calculated Zr-content is higher than that of any previously reported lunar or terrestrial ilmenite. The reduction-exsolution of Fe-Cr-spinel is considered the result of the incorporation of Cr in the ilmenite during crystallization at concentrations that exceeded the solubility limit of Cr in ilmenite at the temperatures of re-equilibration. The Zr content of the host ilmenite is well below the solubility limit of Zr in ilmenite, indicating that oversaturation was not the driving force for the exsolution of baddeleyite. The exsolution of baddeleyite in the ilmenite may have partly compensated for the overall reduction of the ilmenite resulting from the exsolution of Fe-Cr-spinel.

**Keywords:** baddeleyite, ilmenite, Fe-Cr-spinel, oxidation, reduction, exsolution, Basistoppen sill, East Greenland, tholeiitic gabbro.

### SOMMAIRE

L'ilménite de la zone Gabbro Picrite du filon-couche de Basistoppen, au Groënland oriental, contient des lamelles d'exsolution de baddeleyite et spinelle-Fe-Cr. Les proportions relatives de baddeleyite et spinelle-Fe-Cr dans des grains d'ilménite isolés indiquent que l'ilménite fut réduite au cours de la ré-équilibration au dessous du solidus, bien que le sill, en général, soit oxydé pendant le refroidissement. La composition pré-exsolution calculée pour l'ilménite contient jusqu'à 2.4% Cr<sub>2</sub>O<sub>3</sub> et jusqu'à 7100 ppm Zr. Le contenu de Zr calculé est plus élevé que celui de n'importe quelle ilménite, lunaire ou terrestre. L'exsolution-réduction du spinelle-Fe-Cr est considérée comme résultat de l'incorporation de Cr dans l'ilménite pendant la cristallisation, à concentration excédant la limite de solubilité de Cr dans l'ilménite aux températures de ré-équilibration. La teneur en Zr de l'ilménite hôte est nettement inférieure à sa limite de solubilité, d'où l'on déduit que la sursaturation n'est pas la force motrice de l'exsolution de la baddeleyite. L'exsolution de la baddeleyite dans l'ilménite peut avoir compensé partiellement la réduction totale de l'ilménite résultant de l'exsolution du spinelle-Fe-Cr.

(Traduit par la Rédaction)

**Mots-clés:** baddeleyite, ilménite, spinelle-Fe-Cr, oxydation, réduction, exsolution, Basistoppen (filon-couche de), Groënland oriental, gabbro tholéitique.

### INTRODUCTION

Baddeleyite is a common accessory phase in lunar basalts (Agrell *et al.* 1970, Ramdohr & El-Goresy 1970, Keil *et al.* 1971, Brown *et al.* 1971, Haggerty 1973) and in undersaturated terrestrial igneous rocks (Widenfalk & Gorbatshev 1971, Keil & Fricker 1974, Fieremans & Ottenburgs 1979, Raber & Haggerty 1979, Clarke 1981, Scatena-Wachel & Jones 1984), and has been reported as a rare constituent in tektites (King 1966, Clarke & Wosinski 1967) and in shock-altered sediments surrounding meteorite-impact structures (El-Goresy 1965, Kleinmann 1969). Baddeleyite is not a common constituent of subalkaline terrestrial basalts or gabbros, but has been previously reported as a late-stage magmatic mineral in two gabbro dykes from Axel Heiberg island (Keil & Fricker 1974) and in ultramafic cumulates from the Rhum intrusive complex (Williams 1978). Analyses of lunar and terrestrial baddeleyite indicate that it is an important reservoir for the incompatible elements Zr, Hf, and U in igneous rocks (Lovering & Wark 1971, Frondel 1975, Williams 1978); as a result, an understanding of the formation and distribution of baddeleyite in igneous rocks is important for modeling the changing concentrations of incompatible elements during the evolution of magmas.

Although baddeleyite is commonly associated with ilmenite in lunar rocks, it has not been previously reported as an exsolution phase in either lunar or terrestrial ilmenite. Cr-rich spinel has been reported as an exsolution phase of ilmenite in lunar rocks (Haggerty *et al.* 1970, Dence *et al.* 1971), in meteorites (Buseck & Keil 1966) and in kimberlites (Danchin & d'Orey 1972, Haggerty & Tompkins 1984), but has not been previously reported in other terrestrial occurrences of ilmenite (Ramdohr & El-Goresy 1970, Danchin & d'Orey 1972).

### GEOLOGICAL SETTING

The tholeiitic Basistoppen sill, in East Greenland, intruded the upper part of the Skaergaard complex

shortly after the solidification of the main Skaergaard magma (Douglas 1964, Naslund 1984a, b, c). The 600-m-thick sill is zoned; a magnesium-rich initial magma differentiated to an iron-rich residual magma. Zone boundaries are based on changes in the assemblage of cumulate minerals during differentiation. The Gabbro Picrite Zone (GPZ), of interest here, is a discontinuous unit located at the base of the sill. The GPZ contains cumulate olivine (Fo<sub>71-79</sub>), orthopyroxene (En<sub>74-79</sub>), and Fe-Cr-spinel, and intercumulate plagioclase (An<sub>63-69</sub>), augite (En<sub>48-50</sub>Fs<sub>7-11</sub>Wo<sub>40-44</sub>), and ilmenite. Biotite may be a minor cumulate phase.

#### TEXTURAL RELATIONSHIPS

Baddeleyite is a common exsolution phase in ilmenite from the GPZ, but is less common in ilmenite from the other zones of the Basistoppen sill. Fe-Cr-spinel is found either adjacent to or as an

exsolution phase in all of the ilmenite grains containing exsolution lamellae of baddeleyite. Three textural relationships were observed: (A) isolated intercumulate ilmenite grains containing exsolution lamellae of baddeleyite and Fe-Cr-spinel, (B) baddeleyite exsolution lamellae within ilmenite lamellae in cumulate Fe-Cr-spinel, and (C) baddeleyite exsolution lamellae in intercumulate ilmenite that precipitated adjacent to Fe-Cr-spinel (Fig. 1). The relative abundances of ilmenite, baddeleyite and spinel in these samples were calculated from the areas of these phases in back-scattered electron images and were corrected for mineral densities. The approximate relative abundances of ilmenite:baddeleyite:spinel are 110:1:11 in texture A, 580:1:3750 in texture B, and 45:1:130 in texture C. The observed grain-size of the baddeleyite ranges up to  $10 \times 3 \mu\text{m}$  in texture A, up to  $1 \times 1 \mu\text{m}$  in texture B, and up to  $20 \times 5 \mu\text{m}$  in texture C. Cumulate Fe-Cr-spinel in the GPZ is up to 1.0 mm across, and intercumulate ilmenite in the GPZ is up to 0.5 mm in maximum dimension.

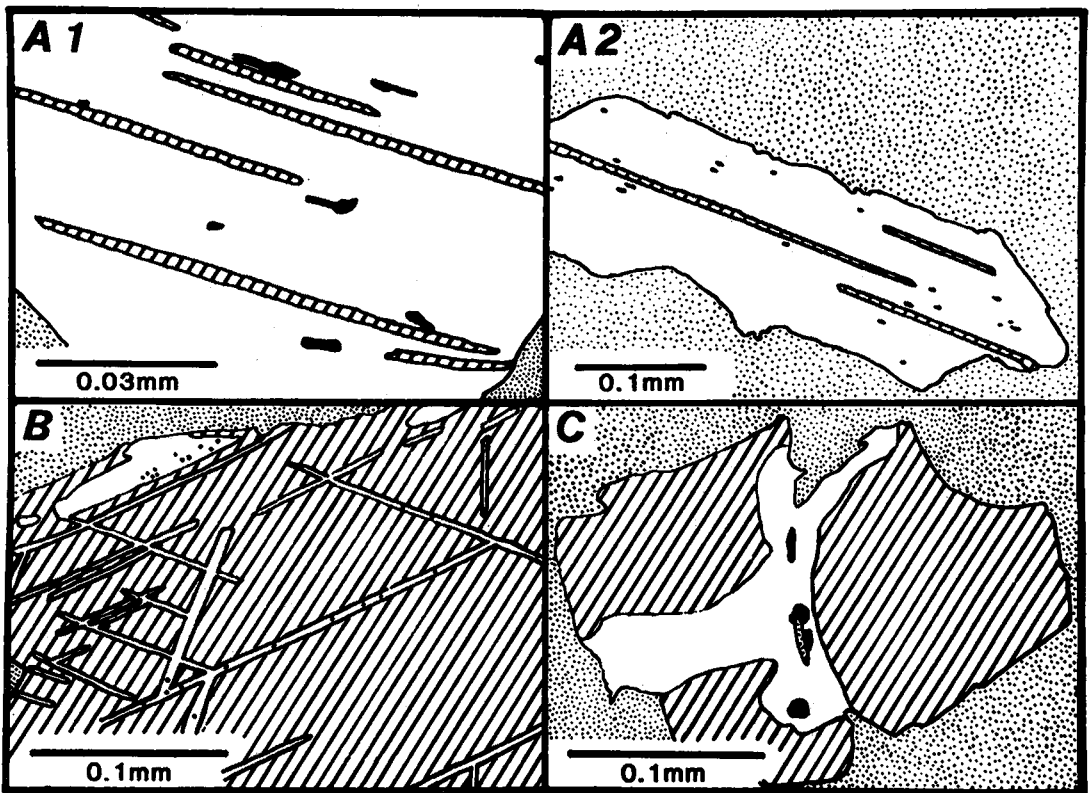


FIG. 1. Textural relationships in ilmenite-baddeleyite-spinel assemblages from the Basistoppen sill, traced from photographs of back-scattered electron images. Ilmenite white, baddeleyite black, Fe-Cr-spinel diagonally ruled; silicates stippled. A1 and A2. Isolated intercumulate ilmenite with exsolution lamellae of baddeleyite and Fe-Cr-spinel (A1 sample 212-5, A2 sample 208-5). B. Isolated cumulate Fe-Cr-spinel with exsolution lamellae of ilmenite containing blebs of baddeleyite (sample 641-3). C. Cumulate Fe-Cr-spinel with adjacent intercumulate ilmenite containing exsolved baddeleyite (sample 208-13).

## MINERAL COMPOSITIONS

Owing to the small grain-size, baddeleyite in textures A and B could only be analyzed qualitatively: energy-dispersion spectra indicate that it is almost pure  $ZrO_2$ . Baddeleyite in texture C was analyzed quantitatively on an electron microprobe with wavelength-dispersion spectrometers using synthetic  $ZrSiO_4$  and  $HfSiO_4$  as standards for Zr and Hf (Table 1). The Basistoppen baddeleyite is similar in composition to that reported from lunar rocks, and is generally poorer in  $ZrO_2$  and richer in  $TiO_2$ ,  $SiO_2$ , and FeO than baddeleyite previously reported from terrestrial gabbroic and ultramafic rocks. FeO and  $TiO_2$  contents in the Basistoppen baddeleyite, however, may be high due to secondary fluorescence effects in the host ilmenite resulting from the small size of the analyzed grains.

In the ilmenite and Fe-Cr-spinel coexisting with baddeleyite, the trivalent cations  $Fe^{3+}$ ,  $Cr^{3+}$ ,  $Al^{3+}$  and  $V^{3+}$  are concentrated in spinel, whereas  $Ti^{4+}$  and the divalent cations  $Fe^{2+}$ ,  $Mn^{2+}$  and  $Mg^{2+}$  are concentrated in ilmenite (Table 2).  $Zr^{4+}$  was not detected in either phase (detection limit  $\sim 750$  ppm). Calculations based on the recalculation scheme of Stormer (1983) and the temperature- $f(O_2)$  curves of Spencer & Lindsley (1981) suggest that the ilmenite and spinel in these rocks last equilibrated at temperatures between 600 and 850°C and oxygen fugacities between  $10^{-13}$  and  $10^{-21}$  bars. All four calculated temperature- $f(O_2)$  pairs are within 1 log unit in  $f(O_2)$  and 35°C of the QFM buffer curve, but the high Cr-content of the spinel requires considerable extrapolation from the curves of Spencer & Lindsley (1981), which are based on Cr-free synthetic compositions.

TABLE 1. CHEMICAL COMPOSITION OF BADDELEYITE

	[1]	[2]	[3]	[4]	[5]	[6]	[7]
Oxide weight per cent							
ZrO <sub>2</sub>	92.59	98.03	97.80	96.40	91.93	92.83	94.70
HfO <sub>2</sub>	1.30	—	0.93	1.72	—	1.95	1.65
TiO <sub>2</sub>	2.52	1.81	0.55	0.3-0.8	4.89	2.14	1.92
SiO <sub>2</sub>	0.34	0.01	0.08	0.0-0.2	0.00	0.24	0.18
Al <sub>2</sub> O <sub>3</sub>	0.18	0.01	—	0.0-0.1	0.31	—	0.54
Cr <sub>2</sub> O <sub>3</sub>	0.17	—	—	0.00	0.15	—	0.13
Nb <sub>2</sub> O <sub>5</sub>	0.00	—	—	0.4-1.3	—	—	0.49
FeO	2.15	0.48	1.30	0.00	0.68	2.05	0.45
MnO	0.09	—	—	—	0.12	0.03	0.17
MgO	0.21	—	—	0.1-0.2	0.14	0.14	0.14
Total	99.55	100.35	100.67	100.30	98.20	99.38	100.27
#	4	2	35	36	2	5	1
Zr/Hf	62	—	105	49	—	48	57

- [1] Basistoppen sill  
 [2] Rhum intrusion (Williams 1976)  
 [3] Axel Heiberg gabbros (Keil & Fricker 1974)  
 [4] Kimberlite (Scatena-Wachel & Jones 1984)  
 [5] Kimberlite (Faber & Haggerty 1979)  
 [6] Apollo 12 (Keil et al. 1971)  
 [7] Luna 20 (Brett et al. 1973)  
 # - number of analyses in average

## DISCUSSION

The presence of ilmenite lamellae in a host spinel is a common feature of slowly cooled igneous rocks. Such textures are the result of oxidation of the spinel during cooling and are sometimes referred to as oxidation-exsolution (Haggerty 1976). Although less common, the oxidation of ilmenite during cooling can result in the oxidation-exsolution of rutile or pseudobrookite (Haggerty 1976). Similarly, lamellae of baddeleyite could form in an ilmenite host during cooling under oxidizing conditions (Fig. 2). The development of spinel lamellae in an ilmenite host, however, requires a reduction of the ilmenite-spinel assemblage (El-Goresy & Ramdohr 1975, Wyatt 1979, Haggerty & Tompkins 1984). The simultaneous exsolution of spinel and baddeleyite could develop in an ilmenite host with no oxidation or reduction if the ratio of spinel:baddeleyite were approximately 3:1 (Fig. 2). In the GPZ of the Basistoppen sill, the ratio of spinel to baddeleyite in

TABLE 2. CHEMICAL COMPOSITION OF ILMENITE AND FE-CR-SPINEL

	208-5 il	208-5 mt	208-13 il	208-13 mt	212-5 il	212-5 mt	641-3 il	641-3 mt
Oxide weight per cent *								
TiO <sub>2</sub>	47.85	6.08	48.84	5.17	49.76	7.27	49.64	9.58
Cr <sub>2</sub> O <sub>3</sub>	0.88	19.31	0.74	17.24	0.34	9.95	1.20	11.74
V <sub>2</sub> O <sub>5</sub>	0.19	1.23	0.00	0.65	0.00	1.35	0.00	0.67
Al <sub>2</sub> O <sub>3</sub>	0.18	3.74	0.12	3.10	0.08	1.84	0.11	2.68
Fe <sub>2</sub> O <sub>3</sub>	8.11	31.61	7.54	38.06	5.52	42.06	3.48	34.52
FeO #	40.10	36.39	40.93	35.35	41.40	37.53	41.66	39.49
MnO	1.57	0.79	1.46	0.55	1.23	0.47	2.76	1.08
MgO	0.67	0.25	0.87	0.72	1.20	0.32	0.14	0.05
total	99.15	99.40	100.50	100.84	99.53	100.59	99.01	99.81
Cations per formula unit								
Ti 4+	0.9103	0.1703	0.9192	0.1371	0.9423	0.2043	0.9511	0.2694
Cr 3+	0.0137	0.5688	0.0146	0.5117	0.0088	0.2939	0.0242	0.3470
V 3+	0.0039	0.0367	0.0000	0.0145	0.0000	0.0404	0.0000	0.0201
Al 3+	0.0054	0.1642	0.0035	0.1392	0.0024	0.0722	0.0033	0.1181
Fe 3+	0.1551	0.8862	0.1420	1.0575	0.1047	1.1827	0.0688	0.9716
Fe 2+	0.8519	1.1334	0.8565	1.0812	0.8718	1.1727	0.8876	1.2348
Mn 2+	0.0338	0.0249	0.0309	0.0184	0.0262	0.0149	0.0600	0.0342
Mg 2+	0.0254	0.0139	0.0325	0.0389	0.0450	0.0178	0.0053	0.0028
Calculations								
X* lim	0.9191	—	0.9259	—	0.9454	—	0.9849	—
X* usp	—	0.4098	—	0.2949	—	0.3111	—	0.4467
temp.	810 C.	—	720 C.	—	680 C.	—	630 C.	—
log f(O <sub>2</sub> )	-14.0	—	-16.0	—	-17.7	—	-17.7	—
texture **	A	—	C	—	A	—	B	—

\* CaO, SiO<sub>2</sub>, ZrO<sub>2</sub>, Nb<sub>2</sub>O<sub>5</sub> < 0.10%; analyses are averages of at least 3 microprobe determinations.

# Fe 2+/Fe 3+ calculated from stoichiometry; \*\* textural types are explained in the text.

X\* lim and X\* usp after Stormer (1983); temp. and log f(O<sub>2</sub>) from curves of Spencer & Lindsley (1981).

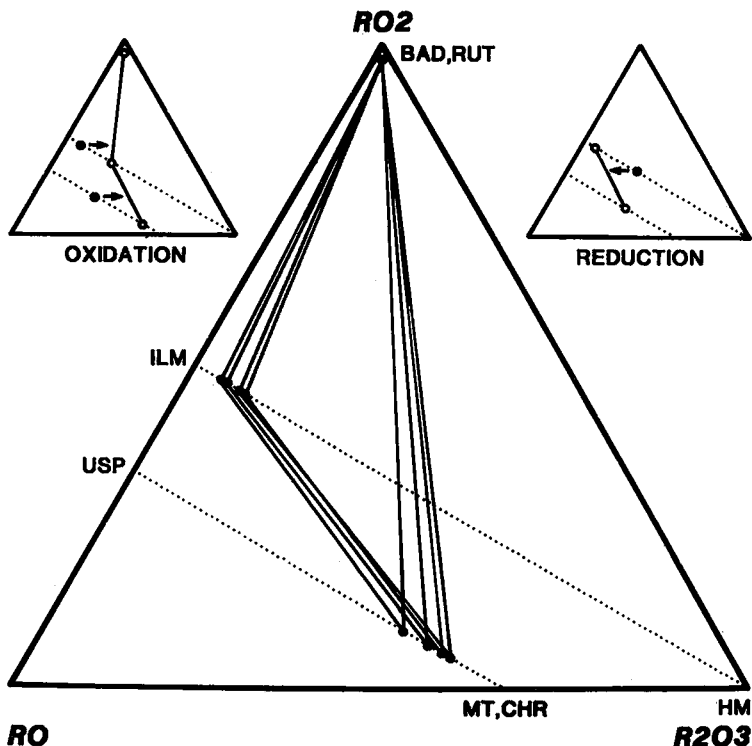


FIG. 2.  $RO-RO_2-R_2O_3$  triangular plot of ilmenite, baddeleyite, and Fe-Cr-spinel compositions from the Basistoppen sill. End-member compositions are indicated: BAD baddeleyite, RUT rutile, ILM ilmenite, USP ulvöspinel, MT magnetite, CHR chromite, and HM hematite. Dotted lines indicate solid-solution series; solid lines connect coexisting phases. The diagram is plotted in cation percent. The upper left insert shows the effect of oxidation-exsolution on ilmenite and spinel. The upper right insert shows the effect of reduction-exsolution on ilmenite.

texture A is approximately 11:1, indicating that the ilmenite-baddeleyite-spinel assemblage must have undergone reduction during cooling. Texture types B and C contain primary Fe-Cr-spinel, so that the ratio of spinel:baddeleyite in these assemblages is not useful for determining whether oxidation or reduction occurred during formation.

Samples of terrestrial ilmenite have systematically lower Zr contents (average 300 ppm) than do samples of lunar ilmenite (average 700–1100 ppm) when comparisons are made between rocks with similar bulk Zr-contents (Arrhenius *et al.* 1971). Lunar ilmenite containing up to 4200 ppm Zr and terrestrial ilmenite containing up to 1100 ppm Zr have been reported (Arrhenius *et al.* 1971, El-Goresy *et al.* 1971a). Experimental results suggest, however, that even the highest concentrations of Zr observed do not represent the solubility limit of Zr in ilmenite, which is in excess of 6000 ppm (Arrhenius *et al.* 1971). Geikielite-ilmenite<sub>ss</sub> ( $Mg_{0.6}Fe_{0.4}TiO_3$ ) containing up to 7600 ppm Zr and ilmenite-pyrophanite<sub>ss</sub>

( $Fe_{0.6}Mn_{0.4}TiO_3$ ) containing up to 1900 ppm Zr have been reported from the groundmass of kimberlite dykes (Tompkins & Haggerty 1985). Ilmenite in ilmenite-baddeleyite-spinel clusters from the Basistoppen sill contains less than 750 ppm Zr, indicating that the driving force for the exsolution of baddeleyite was not oversaturation of Zr in ilmenite. A solubility limit for Zr in ilmenite below 750 ppm would require that previously described examples of ilmenite with higher Zr contents either are metastable or contain unrecognized Zr-rich inclusions.

The distribution coefficient for Zr between ilmenite and silicate melt ( $K_{D,ilm/lq}$ ) has been calculated to be approximately 5.4 for lunar rocks crystallizing at 1200°C, and approximately 2.2–3.0 for the terrestrial Palisades sill crystallizing at 1000–1100°C (Arrhenius *et al.* 1971). A value of 3.0 for  $K_{D,ilm/lq}$  was obtained for Zr in ilmenite megacrysts from a kimberlite (Fugimati *et al.* 1984). Calculated pre-exsolution compositions of ilmenite

from the Basistoppen sill contain up to 7100 ppm Zr. Using the chilled margin composition (175 ppm Zr) for the initial Basistoppen magma and assuming a  $K_D$ ilm/lq between 3.0 and 5.4, the interstitial ilmenite in the GPZ is inferred to have crystallized from a calculated residual liquid enriched in Zr by a factor of 7.5 to 13.5. If Zr behaved as a perfectly excluded element during the crystallization of the Basistoppen sill, this liquid would represent the final 13.3 to 7.4% of crystallization.

Ilmenite in equilibrium with Fe–Cr-spinel from the Basistoppen sill contains 0.3 to 1.2%  $\text{Cr}_2\text{O}_3$ . Calculated pre-exsolution ilmenite from these samples contains up to 2.4%  $\text{Cr}_2\text{O}_3$ . The reduction–exsolution of Fe–Cr-spinel in these grains is considered to be the result of the concentration of Cr in ilmenite during crystallization at magmatic temperatures, followed by a decrease in the solubility limit of Cr in ilmenite during cooling and re-equilibration. The ilmenite was reduced during cooling, in spite of the fact that the rocks as a whole were oxidized during cooling, because Fe–Cr-spinel was the only stable phase that could act as a sink for excess Cr. The exsolution of baddeleyite in the ilmenite may have partly compensated for the overall reduction of the ilmenite–baddeleyite–spinel assemblage.

The stable Zr-bearing mineral in magmas saturated or nearly saturated with silica is zircon (Butterman & Foster 1967, Watson 1979). In the Basistoppen sill baddeleyite is believed to have formed during subsolidus re-equilibration, and therefore was never in contact with silica-saturated magma. Baddeleyite in the Rhum intrusive complex (Williams 1978), the Axel Heiberg gabbros (Keil & Fricker 1974), and lunar basalts (Agrell *et al.* 1970, Lovering & Wark 1971, Simpson & Bowie 1971, Keil *et al.* 1971, El-Goresy *et al.* 1971b) has been interpreted to be a late-stage mineral that crystallized from interstitial Zr-enriched magma. The residual magmas in these rocks, however, are likely to have been nearly saturated in silica, and therefore under equilibrium conditions should have precipitated zircon rather than baddeleyite. In some of these samples, the baddeleyite may have formed during subsolidus re-equilibration of Zr-rich ilmenite or spinel. In some lunar basalts that have been described in detail, nearly all of the observed baddeleyite occurs adjacent to or closely associated with ilmenite (Agrell *et al.* 1970, El-Goresy *et al.* 1971a). Alternatively, the precipitation of baddeleyite in place of zircon may be related to the relative ease of nucleation of the two phases at subsolidus temperatures.

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