

A new natural occurrence of zirconolite (CaZrTi₂O₇) and baddeleyite (ZrO₂) in basic cumulates: the Laouni layered intrusion (Southern Hoggar, Algeria)

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Abstract

Four grains of zirconolite and baddeleyite have been found in two plagioclase-rich adcumulates from the Laouni layered intrusion (Southern Hoggar, Algeria). Zirconolite is poor in REE; baddeleyite is chemically close to ZrO₂ and contains minor amounts of Hf. They are considered to be crystallization products of pockets of intercumulus liquid. The preferential distribution of zirconolite in anorthosite is explained in terms of expulsion of the intercumulus liquid by sintering or annealing of plagioclase. The crystallization of baddeleyite reflects the sharp increase of Zr in the trapped intercumulus liquid.

KEYWORDS: zirconolite, baddeleyite, cumulate texture, Laouni, Hoggar, Algeria

Introduction

ZIRCONOLITE (CaZrTi₂O₇) and baddeleyite (ZrO₂) are rare in terrestrial environments. To date, fourteen occurrences of zirconolite are known (Purtscheller and Tessadri, 1985; Fowler and Williams, 1986; Giéré, 1986; Platt *et al.*, 1987; Agrell *et al.*, 1986). It occurs mainly in alkaline, silica-undersaturated rocks such as sanidinites, kimberlites, carbonatites, and syenites, as well as in hydrothermal and alluvial deposits, altered pyroxenite and metamorphic rocks. Baddeleyite is encountered in the same environments as zirconolite, especially in kimberlites (Raber and Haggerty, 1979; Scatena-Wachel and Jones, 1984). Both minerals occur only rarely in basic and ultrabasic plutonic rocks. Baddeleyite has been described in the Axel Heilberg gabbro, Arctic Canada (Keil and Fricker, 1974), and in a Norwegian anorthosite (Gierth and Kraus, 1974). To our knowledge, the only layered basic intrusion that contains both zirconolite and baddeleyite is that of Rhum, Inverness-shire, Scotland (Williams, 1978).

The two minerals have been identified in the course of a detailed investigation of opaque mineral assemblages in the Laouni layered intrusion, Algeria, 250 km south of Tamanrasset. Here, we present mineralogical relationships, phase chemistry, and the crystallization order of zirconolite and baddeleyite with reference to those of the Rhum occurrence; particular features of our samples are discussed with respect to the volume of intercumulus liquid entrapped in mafic cumulates.

Main petrographic features of the investigated samples

The Laouni layered intrusion is a tholeiitic igneous complex of late Pan-African age (560–660 Ma) intrusive in granitic and metamorphic rocks of the Central Hoggar (Donville, 1962; Cottin, 1985). The investigated samples are from the layered ultramafic cumulates of the massif defined as 'Western Laouni' (Donville, 1962). This massif is composed of olivine-chromite cumulates, bronzite

cumulates, and plagioclase-olivine cumulates, which are rhythmically associated over a few hundred metres beneath a main mass of massive olivine-gabbro, troctolite and two-pyroxene gabbros (Cottin and Lorand, 1987). Zirconolite and baddeleyite have been observed in two plagioclase cumulates but have not been seen to coexist on the hand-specimen scale.

Zirconolite occurs in an anorthosite sample (2H45) from the top of the layered cumulates. Its magmatic assemblage consists of more than 98% by volume of a labradorite-bytownite plagioclase and small amounts of interstitial clinopyroxene and orthopyroxene. This anorthosite sample has a typical adcumulate texture, with less than 5% by volume intercumulus material, and grain boundaries of plagioclase displaying well defined triple junctions having interfacial angles of 120° (Irvine, 1982). Biotite and other hydrous minerals are very rare. Plagioclase is altered to chlorite, epidote and thompsonite along the fractures and grain boundaries (identifications by X-ray diffraction patterns).

Baddeleyite has been found in a plagioclase-olivine cumulate (2H80) in which the rock-forming minerals are plagioclase (Pl) and olivine (Ol) as primocrysts and orthopyroxene (Opx), clinopyroxene (Cpx) pargasitic amphibole (Amph) phlogopite (Phl) and ilmenite (Ilm) as intercumulus minerals. Its modal composition as estimated by point-counting method (2500 points) is Pl 67%, Ol 18%, Opx 8%, Cpx 3%, Amph 3%, Phl and Ilm 1%. Sample 2H80 differs from sample 2H45 by higher volume of intercumulus material so that its texture grades locally into poikilitic adcumulate (Irvine, 1982).

Analytical methods

Zirconolite and baddeleyite have been studied in polished thin sections both in transmitted and reflected light at high magnifications ($\times 500$ to $\times 1000$). Quantitative microprobe data have been obtained at the Museum National d'Histoire Naturelle (in Paris) with a fully automated CAMEBAX equipped with wavelength dispersive spectrometers (WDS). The elements to be analysed were selected after a complete WDS scan on each grain of zirconolite and baddeleyite. This was carried out at an accelerating voltage of 30 kV to enhance detection of heavy elements. Synthetic glasses, monazite, uraninite were used as standards for the LREE, Th and U respectively and pure yttrium for Y. The results are listed in Tables 1 and 2. Quantitative microprobe analyses of heavy rare earth elements have not been attempted due to the complexity of peak overlaps (Roeder, 1985);

Table 1. Representative electron microprobe analyses for zirconolite.

| Weight % | 1 | 2 | 3 |
|--------------------------------------|-------|--------|-------|
| SiO ₂ | - | - | 0.25 |
| TiO ₂ | 35.67 | 35.73 | 35.76 |
| ZrO ₂ | 43.58 | 44.18 | 37.60 |
| HfO ₂ | 0.34 | 0.57 | 0.80 |
| Al ₂ O ₃ | 0.59 | 0.50 | 0.31 |
| Cr ₂ O ₃ | 0.05 | 0.05 | n.a. |
| FeO* | 4.85 | 4.87 | 5.14 |
| MgO | 0.19 | 0.20 | 0.35 |
| MnO | n.d. | 0.16 | 0.1 |
| CaO | 10.67 | 11.15 | 10.80 |
| Y ₂ O ₃ | 2.5 | 2.45 | 2.04 |
| Ce ₂ O ₃ | 0.18 | 0.13 | 0.92 |
| La ₂ O ₃ | 0.02 | 0.01 | 0.17 |
| Ni ₂ O ₃ | 0.17 | 0.25 | 1.28 |
| Pr ₂ O ₃ | 0.15 | 0.13 | 0.19 |
| Sm ₂ O ₃ | n.d. | n.d. | 0.55 |
| Eu ₂ O ₃ | n.d. | n.d. | 0.1 |
| Gd ₂ O ₃ | n.d. | n.d. | 0.53 |
| Dy ₂ O ₃ | n.d. | n.d. | 0.48 |
| Er ₂ O ₃ | n.d. | n.d. | 0.18 |
| Yb ₂ O ₃ | n.d. | n.d. | 0.13 |
| Ta ₂ O ₅ | - | - | 0.23 |
| Nb ₂ O ₅ | 0.22 | 0.25 | - |
| UO ₂ | n.d. | 0.06 | 0.18 |
| ThO ₂ | 0.41 | 0.59 | 0.48 |
| Total | 99.59 | 101.28 | 98.72 |
| Zr/Hf | 128 | 77 | 47 |
| Number of cations based on 7 oxygens | | | |
| Ti | 1.620 | 1.603 | 1.647 |
| Zr | 1.283 | 1.285 | 1.123 |
| Hf | 0.006 | 0.010 | 0.014 |
| Al | 0.042 | 0.035 | 0.023 |
| Cr | 0.002 | 0.002 | - |
| Fe | 0.245 | 0.243 | 0.263 |
| Mg | 0.017 | 0.018 | 0.032 |
| Mn | n.d. | 0.008 | - |
| Ca | 0.690 | 0.713 | 0.709 |
| Y | 0.080 | 0.078 | 0.290 |
| Ce | 0.004 | 0.003 | 0.021 |
| La | 0.000 | 0.000 | 0.004 |
| Ni | 0.004 | 0.005 | 0.028 |
| Pr | 0.003 | 0.003 | 0.005 |
| Sm | - | - | 0.012 |
| Eu | - | - | - |
| Gd | - | - | 0.011 |
| Dy | - | - | 0.010 |
| Er | - | - | 0.004 |
| Yb | - | - | 0.003 |
| Ta | - | - | 0.004 |
| Nb | 0.006 | 0.007 | 0.010 |
| U | - | 0.001 | 0.003 |
| Th | 0.006 | 0.008 | 0.007 |
| Σ | 4.009 | 4.021 | 4.015 |

1, 2 : This study (sample 2H45) ; 3 : Fowler and Williams (1986) : zirconolite from the Rhum intrusion (Inverness Shire-Scotland).
* All Fe as FeO n.d. : below the detection limit of the microprobe. n.a. : not analysed.

qualitative analyses suggest that they are present in very low concentrations, well below the detection limit of this microprobe procedure.

Zirconolite

To date, the nomenclature and formula of zirconolite are still matters of discussion (Williams, 1978). Two formulae (and names) have been proposed, namely zirconolite— AB_3O_7 and zirkelite— AB_2O_5 . Moreover, crystallochemical studies of Mazzi and Munno (1983) have revealed that zirconolite, zirkelite and a third mineral, polymignite, are three polymorphs of the same chemical compound [AB_3O_7 ($CaZrTi_2O_7$)] which are difficult to

Table 2. Representative electron microprobe analyses of baddeleyite.

| Weight % | 1 | 2 | 3 | 4 | 5 |
|--------------------------------------|--------|-------|--------|--------|-------|
| TiO ₂ | 0.98 | 1.13 | 0.56 | 1.73 | 0.60 |
| ZrO ₂ | 98.73 | 96.26 | 97.80 | 98.12 | 99.00 |
| HfO ₂ | 0.76 | 1.15 | 0.93 | n.d. | n.d. |
| Al ₂ O ₃ | n.d. | n.d. | n.d. | 0.01 | n.d. |
| Cr ₂ O ₃ | n.d. | 0.03 | n.d. | n.d. | n.d. |
| Fe ₂ O ₃ | n.d. | n.d. | n.d. | n.d. | n.d. |
| FeO | 0.50 | 0.22 | 1.30 | 0.54 | 0.20 |
| HgO | n.d. | n.d. | n.d. | n.d. | n.d. |
| MnO | 0.04 | n.d. | n.d. | n.d. | n.d. |
| CaO | 0.02 | 0.03 | n.d. | n.d. | n.d. |
| Na ₂ O | n.d. | n.d. | n.d. | n.d. | n.d. |
| K ₂ O | n.d. | n.d. | n.d. | n.d. | n.d. |
| ZnO | n.d. | n.d. | n.d. | n.d. | n.d. |
| Ce ₂ O ₃ | 0.07 | n.d. | n.d. | n.d. | n.d. |
| La ₂ O ₃ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Nd ₂ O ₃ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Pr ₂ O ₃ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Y ₂ O ₃ | 0.27 | 0.28 | n.d. | n.d. | n.d. |
| UO ₂ | n.d. | n.d. | n.d. | n.d. | n.d. |
| ThO ₂ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Nb ₂ O ₅ | n.d. | n.d. | n.d. | n.d. | n.d. |
| V ₂ O ₅ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Zr/Hf | 101.37 | 99.10 | 100.59 | 100.40 | 99.80 |
| Number of cations based on 2 oxygens | | | | | |
| Ti ₄ ⁺ | 0.015 | 0.018 | 0.009 | 0.026 | 0.009 |
| Zr ₄ ⁺ | 0.973 | 0.971 | 0.975 | 0.969 | 0.989 |
| Hf ₄ ⁺ | 0.004 | 0.007 | 0.005 | n.d. | n.d. |
| Al ₃ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Cr ₃ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Fe ₃ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Fe ₂ ⁺ | 0.008 | 0.004 | 0.022 | 0.009 | 0.003 |
| Mg ₂ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Mn ₂ ⁺ | 0.001 | n.d. | n.d. | n.d. | n.d. |
| Ca ₂ ⁺ | n.d. | 0.001 | n.d. | n.d. | n.d. |
| Na ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| K ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Zn ₂ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Ce ₃ ⁺ | 0.001 | n.d. | n.d. | n.d. | n.d. |
| La ₃ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Nd ₃ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Pr ₃ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Y ₃ ⁺ | 0.003 | 0.003 | n.d. | n.d. | n.d. |
| U ₄ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Th ₄ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Nb ₅ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| V ₅ ⁺ | n.d. | n.d. | n.d. | n.d. | n.d. |
| Σ | 1.006 | 1.003 | 1.011 | 1.005 | 1.002 |

1, 2 : this study ; 3 : Kell and Fricker (1974) ; 4 : Gierth and Kraus (1974) ; 5 : Williams (1978). n.d. : not detected.

distinguish by means of their X-ray powder diffraction patterns. In this study, we have attempted to obtain X-ray data on two grains of zirconolite which were large enough to be picked out of a polished thin section. Both were mounted in a Gandolfi camera (Philips PW 1050/25 diffractometer; Cu-K α Ni filter) and exposed for 96 hours. Although repeated three times, this procedure proved unsuccessful suggesting that, as for many terrestrial occurrences (Borodin *et al.*, 1960), the Laouni zirconolite is metamict. Owing to the lack of X-ray diffraction data, we have retained the name zirconolite for the oxide mineral containing Ca-Zr-Ti as major oxides roughly totalling four cations for seven oxygens per formula unit (Table 1). This is in agreement with the nomenclature widely accepted in previous papers devoted to zirconolite (Fron del, 1975; Williams, 1978).

Zirconolite is present in the sample 2H45 as four grains, up to 200 μ m in length but less than 50 μ m wide. It is usually opaque in transmitted light and only the thinner sections are translucent with a deep red-brown colour. Zirconolite belongs to the intercumulus material and is always found to occur between the cumulus plagioclase. One grain is euhedral and partly enclosed in a plagioclase crystal (Fig. 1a). The others are anhedral and moulded against the plagioclase grain boundaries (Fig. 1b). The other accessory minerals include Fe-Ni-Cu sulphides, ilmenite, chrome spinel, rutile, apatite, zircon and the rare mineral loweringite, a complex titanate belonging to the crichtonite group and containing up to 8 wt. % ZrO₂ (Lorand *et al.*, 1987). Zirconolite is neither observed in close contact with these minerals nor as grains adjacent to biotite or other hydrous phases.

Compositional variations within and between grains of zirconolite are small; all the grains are devoid of core-rim zoning. The Laouni samples are compositionally similar to zirconolite from the Mothae kimberlites (Raber and Haggerty, 1979) in terms of the major oxides CaO, TiO₂ and ZrO₂ (Table 1). They differ from the Rhum samples by higher Zr/Ti, Zr/Hf and Nb/Ta ratios and low contents of highly incompatible elements, especially REE (Table 1). The light REE pattern normalized to chondrite abundances is similar to those of the terrestrial zirconolites (Fowler and Williams, 1986) but shows a marked depletion in absolute concentrations. It remains unclear why Y is strongly fractionated with respect to heavy REE in the Laouni zirconolite. Nevertheless, quantitative microprobe analyses which total near 100 wt. % (Table 1), clearly indicate that, although they may be present in very small amounts, the heavy REE depletion of the Laouni samples cannot be explained by analytical error.

Baddeleyite

Baddeleyite is easily misidentified in reflected light because of its similarities with rutile, both having a weak reflectance and yellowish internal reflections. In transmitted light, baddeleyite can be distinguished from rutile by its strong relief and polarizing tints reaching the third order.

Four grains of baddeleyite, ranging in size from 10 to 20 μ m, are present in the sample 2H80. As for zirconolite, this mineral, often euhedral, has crystallized from intercumulus material and forms pockets with ilmenite and biotite at the triple junctions of plagioclase crystals (Fig. 1c and d). Biotite systematically surrounds the baddeleyite-ilmenite composite grains. Other accessory minerals include trace amounts of Al-Cr spinel and

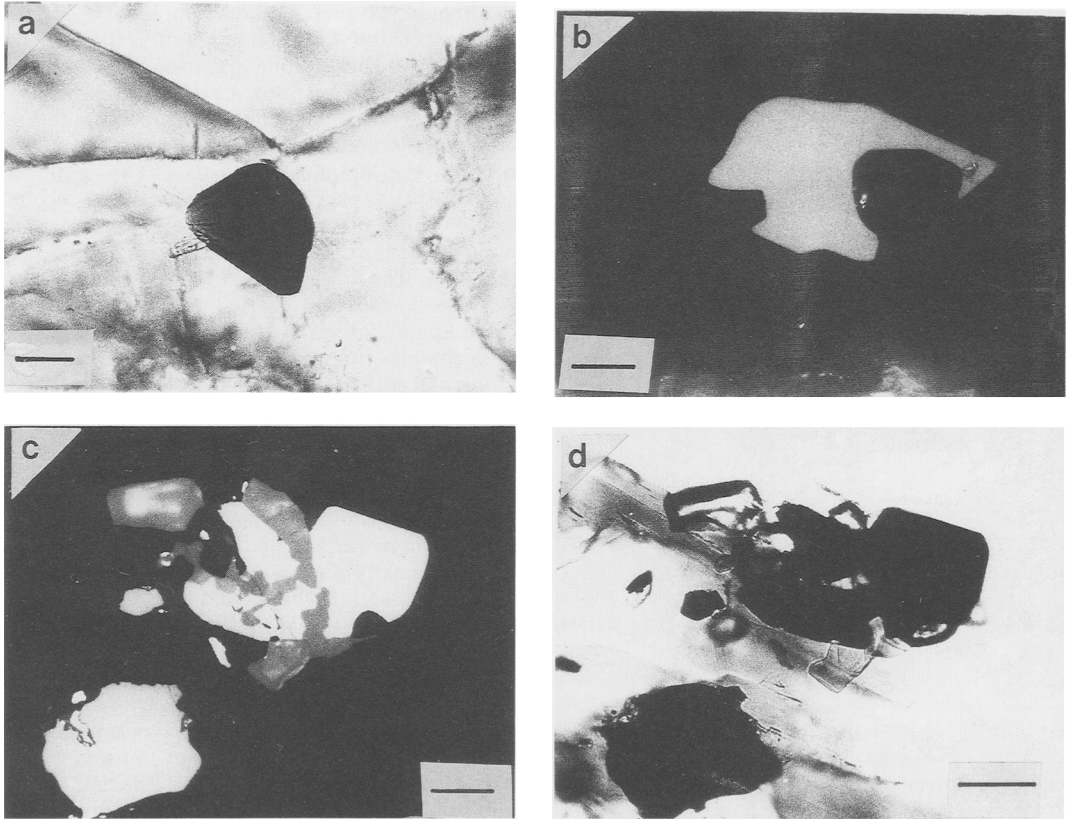


FIG. 1. (a) Euhedral zirconolite (dark) partly enclosed in cumulus plagioclase (white). Sample SH45. Plane polarized transmitted light. (b) Anhedral zirconolite (light grey) from the sample 2H45. Plane polarized reflected light; oil immersion. (c) Complex baddeleyite (translucent)-ilmenite (light grey) intergrowth. Sample 2H80. Plane polarized reflected light; oil immersion. (d) The same as Fig. 1c in plane-polarized transmitted light. Dark grey is interstitial biotite. Scale bar on each photomicrograph is 30 micrometres.

Fe-Ni-Cu sulphides always crystallizing away from the baddeleyite-bearing assemblages.

The composition of the Laouni baddeleyite is very similar to those described by Keil and Fricker (1974) in the Axel Heilberg gabbro, Arctic Canada (Table 2). In both localities, baddeleyite has similar Zr/Hf ratios and contains small amounts of Fe and Ti; the heavy elements detected in the Laouni samples are minor amounts of Ce and Y (Table 2). However, Gierth and Kraus (1974) and Williams (1978) did not provide quantitative microprobe data for Hf, making further discussion on the compositional variability of baddeleyite difficult.

Discussion

The Zr mineral in most mafic rocks is usually zircon while zirconolite and baddeleyite preferentially occur in silica-undersaturated rocks of alkali

affinity (Williams, 1978). Consequently, the occurrence of these minerals in plagioclase cumulates from a tholeiitic intrusion cannot be explained by a simple model of fractional crystallization.

Textural evidence precludes any subsolidus decomposition of zircon + ilmenite + rutile to form baddeleyite (Raber and Haggerty, 1979) or metasomatic replacement of baddeleyite + calcite + Fe-Ti oxide into zirconolite (Purtscheller and Tessadri, 1985). In the Rhum intrusion, zirconolite and baddeleyite are associated with hydrous minerals such as uralite and kaersutite forming late crystallization products of olivine-plagioclase cumulates (Williams, 1978); thus this author has ascribed the crystallization of zirconolite to small pockets of intercumulus liquids isolated from the main mass of magma at the end of the solidification and highly enriched in incompatible elements (U, Nb, Zr, REE, Y). At this stage, each pocket behaves as a closed

system so that local physico-chemical conditions are established which favour the crystallization of Si-deficient minerals (Keil and Fricker, 1974).

Crystallization models of Keil and Fricker (1974) and Williams (1978) are partly valid for the Laouni baddeleyite; indeed this mineral is closely associated with ilmenite and biotite which are late magmatic minerals of olivine-plagioclase cumulates (Cottin and Lorand, 1986). On the other hand, the presence of zirconolite together with zircon, Zr-rich loveringite, ilmenite and rutile in the same polished thin section is evidence for the occurrence of very localized equilibrium conditions, especially regarding the activities of Ti and Zr, during the crystallization of the sample 2H45. However, differences in mineralogical relationships and composition between the Laouni and the Rhum zirconolites suggest that this mineral could form under a great diversity of conditions; in particular, it crystallized at Laouni from intercumulus liquid not enriched in incompatible elements. A remarkable feature of the Laouni samples is that zirconolite is found in adcumulates nearly devoid of intercumulus material compared to a sample containing baddeleyite. It is well known that pockets of intercumulus liquid in a semi-consolidated cumulus pile may be displaced during compaction (Irvine, 1980). Consequently, the crystallization and chemistry of either of the two minerals seems to be strongly dependent on the timing of this expulsion, or, in other words, on how long they have been equilibrated with the intercumulus liquid.

The anorthosite that contains zirconolite displays signs of annealing such as grain boundaries of plagioclase with triple junctions meeting at 120° (Spry, 1969). Kruger and Marsh (1985) have underlined the fact that annealing (often referred to as sintering), coupled with filter-pressing, is a powerful mechanism for expelling pockets of intercumulus liquid. We propose that this process is responsible for both the absence of hydrous silicates associated with zirconolite and for its low content of incompatible elements. Indeed, our interpretation implies that this mineral crystallized at an early stage of the solidification of the intercumulus liquid, before this liquid pocket was expelled. Unfortunately, any accurate determination of the crystallization temperature of zirconolite is precluded because methods based on the Ti/Zr ratio (Wark *et al.*, 1973) are now believed to be unreliable (Purtscheller and Tessadri, 1985).

Zr behaves as a nearly perfect incompatible element during magmatic differentiation (Eales and Robey, 1976). If plagioclase annealing is arrested, then the content of Zr sharply increases in pockets of intercumulus liquid entrapped within cumulates so that baddeleyite crystallization becomes

possible. The ZrO₂ compound is known as several polymorphs and may be stable up to 2400 °C and 100 kbar (Roth *et al.*, 1971). As for zirconolite, it is impossible to accurately determine the crystallization temperature for baddeleyite without a detailed crystallographic study. The close association between ilmenite, biotite and baddeleyite suggest that the latter mineral should have been formed at about 900–1000 °C.

Conclusion

The present study indicates that zirconolite and baddeleyite are not as rare as believed in cumulates from layered igneous intrusions but are probably overlooked during routine ore microscopy. They are typical minerals crystallizing from pockets of intercumulus liquids. However, zirconolite may form under a great diversity of conditions as suggested by its compositional variations between each occurrence.

The crystallization of baddeleyite instead of zirconolite seems to be dependent on the ejection of the intercumulus liquid from the cumulus pile by annealing or sintering of the host rocks. If this process is complete, zirconolite low in REE, Y, Nb, would be stable. If the intercumulus liquid can persist over a wide temperature range, baddeleyite would be crystallized.

Our data are in good agreement with earlier conclusions of Fowler and Williams (1986), e.g. zirconolite and baddeleyite are minor phases which could be powerful petrogenetic guides for study of post-magmatic processes.

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