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# Zr-in-rutile geothermometry of sapphirine + quartz–bearing ultrahigh-temperature granulites from Rajapalaiyam in the Madurai Block, southern India

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**Abstract**

We report for the first time the results of Zr-in-rutile geothermometry of sapphirine + quartz–bearing ultrahigh-temperature (UHT) granulites from the Southern Granulite Terrane, India, where the records of extreme crustal metamorphism related to the Neoproterozoic collisional orogeny during Gondwana assembly is well preserved. Microprobe analyses reveal that rutile in equilibrium with quartz in quartzo-feldspathic granulites from Rajapalaiyam in the southern part of the Madurai Block contains up to 3909 ppm Zr. Application of the geothermometer to the high-Zr rutiles yielded a temperature range of 880–900°C, which is broadly consistent with previous temperature estimates by various geothermometers, as well as the stability field of sapphirine + quartz assemblage. In contrast, rutiles in a pelitic granulite show slightly lower temperatures of 870-880°C (Zr = 3131–3242 ppm). Such rutile grains often show granular exsolution of fine-grained zircons and their distribution along rutile grain margins, suggesting their recrystallization during post-peak cooling. Some rutile grains included in quartz and garnet display the lowest Zr contents of Zr = 1954–2731 ppm and 874–3057 ppm, that translate into lower temperatures of 820-860°C and 740-870°C, respectively. Those isolated rutile grains were probably not in equilibrium with zircons. The results of this study confirmed that Zr-in-rutile geothermometer is applicable to UHT granulites from Rajapalaiyam, although the calculated results correspond to the minimum estimates of peak metamorphic temperature.

**Keywords:** Zr-in-rutile geothermometer; Ultrahigh-temperature metamorphism; Sapphirine + quartz; Granular exsolution; Neoproterozoic to Cambrian; Gondwana Supercontinent

## Introduction

Pressure-temperature (*P–T*) conditions and paths recorded in metamorphic rocks give us valuable information on the burial and exhumation processes of crustal materials in convergent plate margins. Particularly, detailed petrological and mineralogical studies on granulites, which have been regarded as major components of lower crust, are useful for investigating material transportation in the deeper part of orogenic belts (e.g., Brown, 2002). A variety of geothermometers have been applied to estimate peak temperatures of granulite-facies metamorphism in different high-grade terranes worldwide. However, most of the methods are based on Fe-Mg cation exchange between minerals, and they often yield temperatures significantly lower than the peak conditions because of the effect of retrograde metamorphism (e.g., Pattison et al., 2003). Therefore, characterization of peak metamorphic *P–T* conditions in granulite-facies rocks, particularly those that underwent ultrahigh-temperature (UHT) metamorphism at >900°C or event >1100°C, using conventional geothermobarometry is subjected to considerable uncertainty.

In contrast, isopleth geothermometers (e.g., Al in orthopyroxene) are regarded to be more suitable for the estimation of near-peak conditions of UHT metamorphism (e.g., Harley, 2004) because the elements adopted for such geothermometers are relatively less mobile at high-temperature conditions, due to which the effect of thermal overprinting has been regarded as minimum.

Among the available isopleth thermometers, Zr-in-rutile geothermometer is regarded to be one of robust techniques for temperature estimations of near-peak conditions for UHT metamorphic rocks (e.g., Kelsey and Hand, 2015). Rutile is a common mineral in high-grade metasediments, often formed by prograde dehydration of biotite and/or staurolite, and subsequently witnessed peak metamorphic events. Therefore, the geothermometer has been applied to many UHT granulites worldwide (e.g., Zack et al., 2004). However, there has been no attempt so far to apply this method to the UHT granulites from the Southern Granulite Terrane in India.

The Southern Granulite Terrane is characterized by the occurrence of Late Neoproterozoic to Early Cambrian high-grade metamorphic rocks that underwent high to UHT ($T$ >900°C) peak metamorphism related to the collisional orogeny during the final stage of assembly of Gondwana supercontinent (Santosh et al., 2009, and references therein). Among the various UHT localities in the terrane, Rajapalaiyam in the southern Madurai Block (Fig. 1) is well known for the first report of equilibrium sapphirine + quartz assemblage from southern India (Tateishi et al., 2004; Tsunogae and Santosh, 2006, 2010; Braun et al., 2007). We therefore applied Zr-in-rutile geothermometer to rutile grains in sapphirine + quartz–bearing and related granulites from this locality to confirm UHT metamorphism in this region, and also for evaluating the usage of the method.

**Petrography**

For the calculation of near-peak temperatures, we selected four samples of rutile-bearing quartzo-feldspathic and pelitic granulites from Rajapalaiyam. The quartzo-feldspathic granulites (samples MD6-2L, K) are

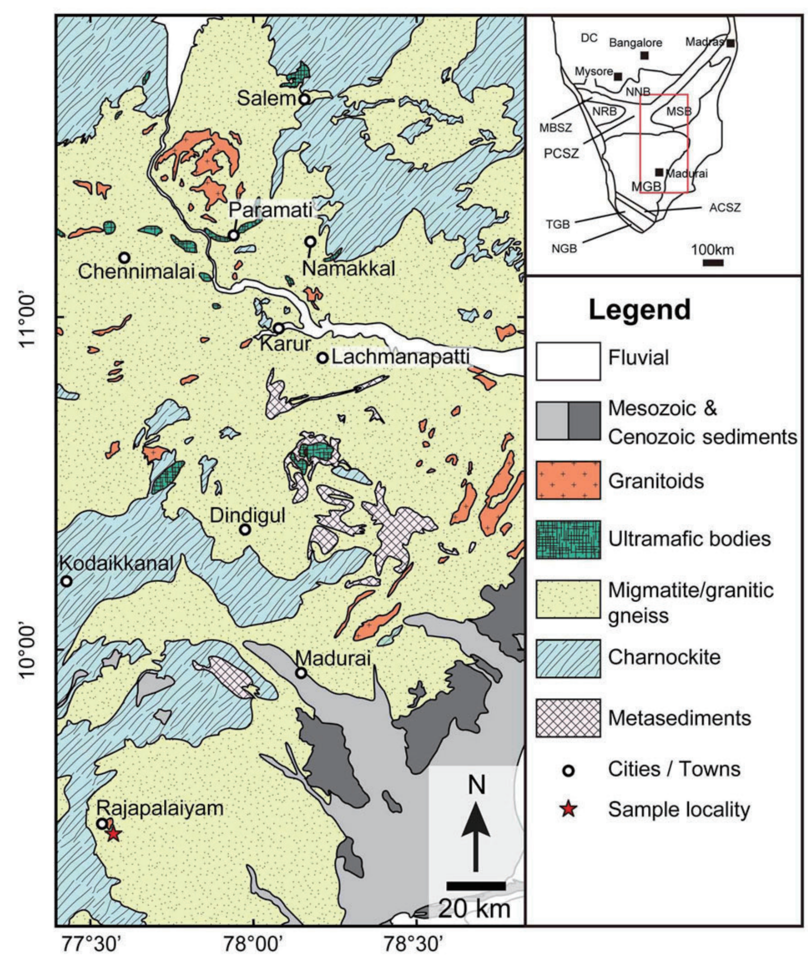


Fig. 1. Geological map of the southern Madurai Block and the Palghat-Cauvery Suture Zone (based on 1: 500,000 map of Tamil Nadu, GSI, 1995) showing the locality of Rajapalaiyam (star) discussed in this study. DC: Dharwar Craton, MGB: Madurai Granulite Block, MSB: Madras Block, NNB: Northern Block, NGB: Nilgiri Block, TGB: Trivandrum Granulite Block, ACSZ: Achankovil Shear Zones, PCSZ: Palghat-Cauvery Suture Zone, P: Phanerozoic cover.

composed of quartz (30–50 %), mesoperthite (20–40 %), garnet (20–10 %), sillimanite (2–10 %), biotite (2–10 %), and cordierite (2–5 %) with accessory sapphirine, zircon, rutile, monazite, and apatite. The rock exhibits weak foliation defined by elongated sillimanite and ribbon quartz. The garnet is poikiloblastic and contains numerous inclusions of sillimanite, sapphirine, quartz, biotite, apatite, zircon, spinel, and rutile. The sapphirine inclusions show direct contact with quartz (Fig. 2a), suggesting that the peak metamorphism probably exceeds 900°C. Sample MD6-2K contains orthopyroxene + sillimanite corona around garnet, which indicates near-isobaric cooling after the peak metamorphism (Tsunogae and Santosh, 2010). Rutile is very rare in the quartzo-feldspathic samples; only a few grains are found in one thin section, whereas zircons are abundant in the rocks. All the rutile grains are in contact with or completely surrounded by quartz (Figs. 2b, 2c).

The pelitic granulite (sample MD6-2E) comprises garnet (20–30 %), biotite (10–20 %), mesoperthite (10–20 %), orthopyroxene (5–10 %), quartz (5–10 %), cordierite (5–10 %), plagioclase (5–10%), and sillimanite (2–5 %) with accessory sapphirine, rutile, and zircon. The rock contains abundant coarse-grained (up to 25 mm) poikiloblastic garnet with numerous inclusions of sapphirine (+ quartz), rutile, apatite, K-feldspar, plagioclase, zircon, and sillimanite. The garnet is in part mantled by coronae of cordierite + orthopyroxene, suggesting post-peak decompression. Pale brownish to yellowish pleochroic orthopyroxene occurs as medium- to coarse-grained (up to 1.2 mm) porphyroblasts in the matrix of quartz and cordierite. In some places, sillimanite coexists with the matrix orthopyroxene. Rutile in the sample is often fine grained (0.2–0.4 mm) and coexists with quartz (Fig. 2d) and feldspars or is included in garnet (Fig. 2e).

Sapphirine-free and quartzo-feldspathic orthopyroxene granulite (sample MD6-1L) from an adjacent locality was also chosen for comparison. The rock is composed of quartz (70–75 %), mesoperthite (10–15%), orthopyroxene (5–10%), and biotite (5–10%) with accessory zircon and rutile. It is characterized by very coarse-grained (up to 2 cm) subidioblastic quartz intergrown with medium-grained (~1.5 mm) orthopyroxene and mesoperthite. The biotite is fine-grained and idioblastic to subidioblastic. It occurs along grain boundaries of the above minerals, and defines the major foliation of the rock. Rutile occurs as fine-grained (<0.25 mm) and dark reddish minerals (Fig. 2f) or rarely as very fine-grained yellowish euhedral minerals within quartz.

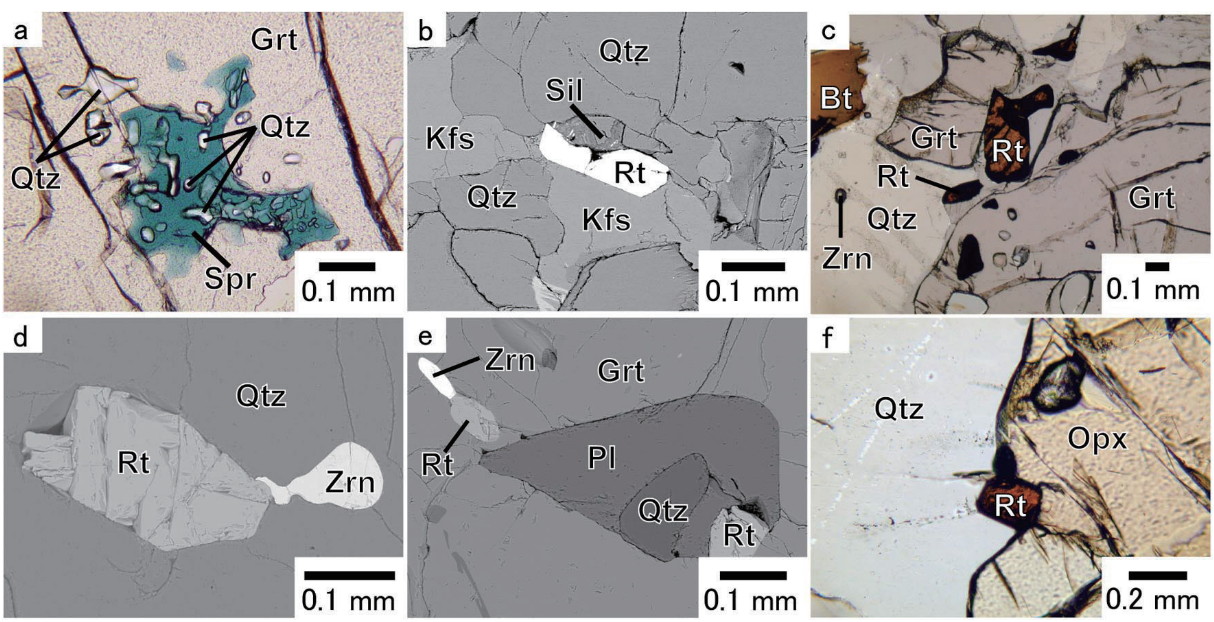


Fig. 2. Photographs of granulites discussed in this study. (a), (c) and (f) are taken by polarized light and (b), (d) and (e) are back-scattered electron image photographs. (a) Sapphirine + quartz assemblage within garnet in sample MD6-2L (quartzo-feldspathic granulite) as an evidence of peak UHT metamorphism. (b) Rutile in contact with sillimanite, K-feldspar, and quartz in sample MD6-2K (quartzo-feldspathic granulite). (c) Rutile in contact with quartz and garnet in sample MD6-2L. (d) Rutile and zircon grains coexisting with quartz in sample MD6-2E (pelitic granulite). (e) Rutile + plagioclase inclusion in garnet in sample MD6-2E. (f) Dark reddish rutile coexisting with orthopyroxene and quartz in sample MD6-1L (sapphirine-absent quartzo-feldspathic granulite).

**Rutile chemistry**

*Analytical technique*

Chemical analyses of rutile were carried out using an electron microprobe analyzer (JEOL JXA8530F) at the Chemical Analysis Division of the Research Facility Center for Science and Technology, the University of Tsukuba, under an accelerating voltage of 15 kV, beam currents of 100 nA for rutiles and 10 nA for standard materials, and a beam diameter of 3 micron. The data were regressed using an oxide-ZAF correction program supplied by JEOL. The standard materials adopted for quantitative analysis of rutile are synthetic $TiO_2$ for Ti, quartz for Si, corundum for Al, chromite for Cr, hematite for Fe, $ZrO_2$ for Zr, and synthetic $NdPO_4$ for Nd. Peak and background counting times for Zr is 100 sec and 50 sec, respectively. Analytical accuracy for Zr in rutile is better than 18%. Compositions of rutiles in the analyzed samples are given in Table 1.

*Results*

All the analyses were performed on rutile grains coexisting with quartz. Zircon is present in all samples, although it is not always in contact with the rutiles. Rutile grains present as inclusions in quartz and garnet were also analyzed. The compositional data of rutile in Table 1 suggest that Zr content varies depending on samples and coexisting minerals. Rutile grains occurring along quartz-garnet grain boundaries in sample MD6-2L (sapphirine + quartz–bearing quartzo-feldspathic granulite) show the highest Zr content of 3501-3909 ppm, whereas those in sample MD6-2K, which coexist with quartz and K-feldspar, show slightly lower Zr content of 3131–3242 ppm. Rutile inclusions in quartz from a sapphirine-absent quartzo-feldspathic granulite (sample MD6-1L) show moderate Zr contents of 3057–3546 ppm, whereas rutiles coexisting with orthopyroxene and quartz in the same sample display lower Zr content of 2495–2510 ppm. A coarse-grained rutile in the matrix of sample MD6-2E

Table 1. Representative electron microprobe analyses of rutile.

| Sample No. | MD6-2E | MD6-2E | MD6-2E | MD6-2E | MD6-2E | MD6-2E | MD6-2L |
|---|---|---|---|---|---|---|---|
| Spot No. | 1 | 2 | 3 | 4 | 5 | 6 | 1 |
| Remarks | In Qtz | In Qtz | With Grt & Qtz | In Grt | In Grt | With Grt & Qtz | With Grt & Qtz |
| $TiO_2$ | 98.89 | 99.34 | 98.91 | 98.15 | 99.00 | 97.92 | 97.14 |
| $SiO_2$ | 0.04 | 0.03 | 0.03 | 0.02 | 0.01 | 0.01 | 0.01 |
| $Al_2O_3$ | 0.04 | 0.02 | 0.02 | 0.04 | 0.03 | 0.02 | 0.03 |
| FeO | 0.40 | 0.41 | 0.39 | 0.57 | 0.55 | 0.48 | 0.58 |
| $Cr_2O_3$ | 0.16 | 0.12 | 0.09 | 0.10 | 0.10 | 0.11 | 0.14 |
| $Nb_2O_5$ | 0.46 | 0.45 | 0.51 | 0.34 | 0.28 | 0.59 | 0.72 |
| $ZrO_2$ | 0.27 | 0.36 | 0.42 | 0.41 | 0.13 | 0.50 | 0.50 |
| Total | 100.25 | 100.73 | 100.35 | 99.64 | 100.10 | 99.63 | 99.10 |
| Zr (ppm) | 1969 | 2635 | 3072 | 3057 | 977 | 3694 | 3672 |

| Sample No. | MD6-2L | MD6-2K | MD6-2K | MD6-1L | MD6-1L | MD6-1L |
|---|---|---|---|---|---|---|
| Spot No. | 2 | 1 | 2 | 1 | 2 | 3 |
| Remarks | With Grt & Qtz | With Qtz, Sil & Kfs | With Qtz & Kfs | With Opx & Qtz | In Qtz | In Qtz |
| $TiO_2$ | 97.69 | 94.42 | 94.42 | 98.06 | 98.56 | 98.76 |
| $SiO_2$ | 0.01 | 0.07 | 0.08 | 0.02 | 0.12 | 0.05 |
| $Al_2O_3$ | 0.06 | 0.11 | 0.10 | 0.03 | 0.02 | 0.02 |
| FeO | 0.78 | 1.41 | 1.48 | 0.47 | 0.38 | 0.41 |
| $Cr_2O_3$ | 0.07 | 0.05 | 0.07 | 0.11 | 0.10 | 0.05 |
| $Nb_2O_5$ | 0.83 | 2.69 | 2.71 | 0.24 | 0.10 | 0.10 |
| $ZrO_2$ | 0.53 | 0.43 | 0.43 | 0.34 | 0.43 | 0.47 |
| Total | 99.97 | 99.18 | 99.29 | 99.26 | 99.70 | 99.86 |
| Zr (ppm) | 3909 | 3206 | 3206 | 2495 | 3183 | 3465 |

(pelitic granulite) shows higher Zr content (3657–3694 ppm) than a fine-grained type (2939–3072 ppm) and inclusion phases in quartz and garnet (1954–2731 ppm and 874–3057 ppm, respectively).

**Zr-in-rutile geothermometry**

Zack et al. (2004) evaluated the incorporation of Zr in rutile coexisting with zircon and quartz based on the reaction:

$$ZrSiO_4 = ZrO_2 \text{ (in rutile)} + SiO_2 \quad (1)$$

at high temperatures, and empirically calibrated a Zr-in-rutile geothermometer using natural rutile data from blueschists, eclogites, and granulites. Degeling (2003) and Watson et al. (2006) improved the thermometer using new experimental data, and Tomkins et al. (2007) examined the effect of pressure and revised the thermometer. The method of Tomkins et al. (2007) indicates that rutile with >4000 ppm Zr records UHT metamorphic conditions ($T$ >900°C). Previous studies (e.g., Zack et al., 2004; Harley, 2008) discussed that overestimation of temperatures with the rutile thermometer is only possible where mineral assemblages in equilibrium with rutile are quartz-free, while underestimation can happen in zircon-absent and/or in partially reset mineral assemblages. We thus applied the method of Tomkins et al. (2007) to rutile coexisting with quartz in zircon-bearing rocks, thereby attempt to estimate the minimum temperature of the peak metamorphism. The calculated results are shown in Table 2.

High-temperature conditions in the range of 889–902°C (at 9 kbar) were obtained for rutiles in a sapphirine + quartz–bearing quartzo-feldspathic granulites (samples MD6-2L). Similar temperature ranges were obtained from different methods such as 898–910°C (Watson et al., 2006) and 896–910°C (Ferry and Watson, 2007), although slightly higher temperatures were obtained from the method of Zack et al. (2004) (1033–1047°C). Rutiles in sample MD6-2K (pelitic granulite) and sample MD6-1L (sapphirine-absent quartzo-feldspathic granulite) also gave consistent temperatures of 876–879°C and 848–890°C, respectively. In contrast, a fine-grained rutile occurring along garnet-quartz grain boundaries in sample MD6-2E (pelitic granulite) yielded lower temperatures of 867–872°C. Rutile inclusions in quartz and garnet in the same sample also display lower temperatures of 820–858°C and 738–872°C, respectively.

**Discussion**

Rutiles in equilibrium with quartz in sapphirine + quartz–bearing quartzo-feldspathic granulites from Rajapalaiyam in the Madurai Block, southern India, contain up to 3909 ppm Zr, while those included in quartz and garnet in pelitic granulites show lower Zr contents of 1954–2731 ppm and 873–3057 ppm, respectively. Application of Zr-in-rutile geothermometer to the former rutile grains yielded high-temperature conditions of 889–902°C (sample MD6-2L; quartzo-feldspathic granulite), 876–879°C (sample MD6-2K; pelitic granulite), 867–895°C (sample MD6-2E; pelitic granulite), and 848–890°C (sample MD6-1L, sapphirine-absent quartzo-feldspathic granulite). Slightly lower temperatures of 738–872°C

Table 2. Zr concentration of analyzed rutiles with calculated temperatures.

| Sample No. | Spot No. | Occurrence | Zr (ppm) | T (°C) | | | |
|---|---|---|---|---|---|---|---|
| | | | | FW07 | W06 | T07* | Z04 |
| MD6-2E | 1 | In Qtz | 1954-2050 | 824-830 | 826-831 | 820-815 | 958-065 |
| | 2 | In Qtz | 2635-2731 | 860-864 | 862-866 | 854-858 | 997-1001 |
| | 3 | With Grt and Qtz | 2939-3072 | 873-879 | 876-881 | 867-872 | 1011-1016 |
| | 4 | In Grt | 2450-3057 | 851-878 | 853-888 | 847-872 | 987-1016 |
| | 5 | In Grt | 874-977 | 738-750 | 739-750 | 738-749 | 856-870 |
| | 6 | With Grt and Qtz | 3657-3694 | 899-903 | 902-906 | 891-895 | 1036-1040 |
| MD6-2L | 1 | With Grt and Qtz | 3501-3672 | 896-902 | 898-905 | 889-894 | 1033-1039 |
| | 2 | With Grt and Qtz | 3827-3909 | 908-910 | 907-910 | 900-902 | 1044-1047 |
| MD6-2K | 1 | With Qtz, Sil, and Kfs | 3153-3228 | 882-885 | 885-888 | 876-878 | 1020-1023 |
| | 2 | With Qtz and Kfs | 3131-3242 | 881-886 | 884-888 | 875-879 | 1019-1023 |
| MD6-1L | 1 | With Opx and Qtz | 2495-2510 | 853-854 | 855-856 | 848 | 990 |
| | 2 | In Qtz | 3057-3183 | 878-883 | 881-886 | 872-877 | 1016-1021 |
| | 3 | In Qtz | 3465-3546 | 894-897 | 897-900 | 887-890 | 1032-1035 |

FW07: Ferry and Watson (2007), W06: Watson et al., (2006), T07: Tomkins et al. (2007), Z04: Zack et al. (2004)

* Temperatures calculated at 9 kbar.

were obtained from rutile inclusions in quartz and garnet within sample MD6-2E probably because rutiles were isolated from the matrix minerals, therefore not in equilibrium with zircons.

The high-temperature conditions obtained in this study are consistent with recent pseudosection analysis of sapphirine granulites from this locality in NCKFMASHTO system that suggested peak metamorphism at $T$ = 900–940°C (Shimizu, unpublished data), and also the stability of sapphirine + quartz assemblage in the rock (>900°C; e.g., Tateishi et al., 2004; Tsunogae and Santosh, 2010). However, the conditions are slightly lower than the previous temperature estimates based on garnet-orthopyroxene (930–990°C), ternary-feldspar (950–1050°C), and Al-in-orthopyroxene (990–1070°C) geothermometers for the sapphirine granulites. Such slightly lower temperature conditions obtained by Zr-in-rutile geothermometry could be related to exsolution of zircon and baddeleyite ($ZrO_2$) during retrograde metamorphism. Pape et al. (2016) applied Zr-in-rutile geothermometry to UHT granulites from the Ivrea Zone, Italy, for evaluating its usefulness and reliability to record and preserve high temperature conditions. Although the geothermometry yielded peak temperatures of up to 1050°C, which is consistent with the published peak conditions, they obtained a wide range in Zr concentrations that translate into a wide range in apparent formation or equilibration temperatures (650°C to 1050°C). Their detailed investigations in rutile textures indicate the presence of lamellae of baddeleyite ($ZrO_2$) in the rutile grains and fine-grained zircons distributed along the grain margins. These textures suggest exsolutions of baddeleyite and zircon during post-peak cooling. Mitchell and Harley (2017) also evaluated rutiles in UHT granulites from the Napier Complex, East Antarctica, and obtained similar granular exsolution of fine-grained zircons within rutile grains. They obtained temperatures of 606°C and 780°C from the recrystallized rutiles, which are significantly lower than the inferred peak temperature of the rock (>1000°C). Our textural observations also confirmed the occurrence of fine-grained zircons along the margin of rutile grains (e.g., Fig. 3) probably formed by granular exsolution during post-peak cooling. In contrast, no zircon exsolution has been observed from high-temperature rutiles except the presence of some Fe-oxide exsolution (either magnetite of hematite) crystallized along later cracks.

The results of this study confirmed that Zr-in-rutile geothermometer is applicable to sapphirine + quartz–bearing UHT granulites from Rajapalaiyam, although the calculated results indicate the minimum estimates of peak metamorphic temperature. Rutiles present along grain boundaries of matrix minerals preserve higher temperatures than those occurring as inclusions. Those free from exsolution textures tend to preserve near-peak conditions. Zr-in-rutile geothermometer should be therefore applied to natural minerals after careful evaluation of textures.

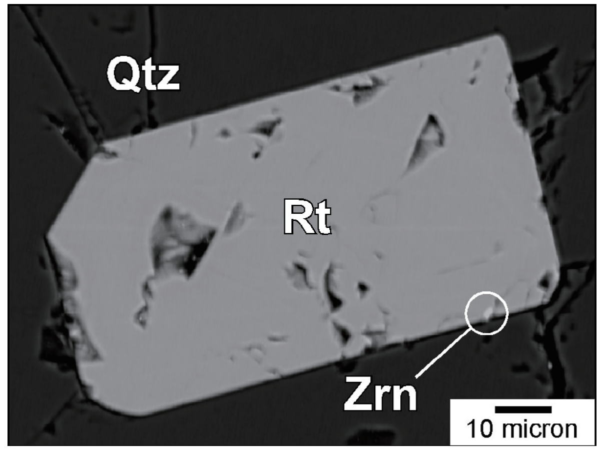


Fig. 3. A back-scattered electron image photograph showing an exsolved zircon grain along the margin of a rutile in sample MD6-2E (pelitic granulite).

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