

Mineral ages for multi isotope system in phlogopite-bearing pyroxene granulite and felsic gneiss, the Howard Hills, Enderby Land, East Antarctica: Possible Proterozoic tectonothermal events in the Napier Complex

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Abstract: Large mafic to ultramafic blocks in felsic gneisses on the northern part of the Howard Hills, Napier Complex, East Antarctica are accompanied by phlogopite-bearing pyroxene granulite occurring in the margins of the block. In order to understand the crustal evolution of the Napier Complex, especially regarding the thermal history after peak metamorphism, Rb-Sr, Sm-Nd and U-Pb isotopic analyses have been carried out on different minerals from the phlogopite-bearing pyroxene granulite and adjacent orthopyroxene felsic gneiss.

Zircon grains from the orthopyroxene felsic gneiss yielded near concordant U-Pb isotopic ages of about 2.5 Ga by conventional isotope dilution methods and defined a discordia with 2.44 ± 0.02 Ga lower intercept age. This age shows the waning stage of UHT granulite facies metamorphism in the Howard Hills. Rutile fractions from pyroxene granulite yielded a near concordant U-Pb isotopic age of about 1.5 Ga. This age is interpreted as the final thermal episode, excepting local igneous intrusions, in the Howard Hills region.

Fluorophlogopite fractions from pyroxene granulite yielded Rb-Sr model ages of about 1.85 Ga, although evidence of retrograde metamorphism with fluid activity or deformation were poor in the pyroxene granulite. An internal Sm-Nd isochron of whole rock and orthopyroxene and feldspar separated from the same sample shows 1.85 ± 0.15 Ga. The Rb-Sr phlogopite model age, along with the Sm-Nd internal isochron age, records the time when the rocks of the Howard Hills underwent medium to high grade metamorphism at temperatures well above the currently accepted closure temperature of biotite (about 300–350°C).

key words: Napier Complex, UHT metamorphism, zircon, fluorophlogopite, rutile

1. Introduction

The Napier Complex in Enderby Land, East Antarctica, consists of ultra-high temperature (UHT) granulite-facies metamorphic rocks that are characterized by

osumilite-bearing, sapphirine-quartz and spinel-quartz assemblages (Sheraton *et al.*, 1987). The temperature and pressure conditions of peak metamorphism have been estimated at over 1000°C and 7–11 kbar through analyses of phase relations and geothermobarometry (*e.g.*, Harley and Hensen, 1990; Motoyoshi, 1998). The complex is surrounded by the Proterozoic Rayner Complex, which is composed of upper-amphibolite to granulite-facies metamorphic rocks (Sheraton *et al.*, 1987).

The Napier Complex is an important area for research into the structure and evolution of the Archean continental lithosphere. Many isotopic dating studies have been conducted on metamorphic rocks from the complex to reveal its geothermal history (Fig. 1). Zircon grains with ion-microprobe (SHRIMP) U-Pb ages up to 3.8 Ga have been found in orthogneiss from Mt. Sones (Williams *et al.*, 1984; Black *et al.*, 1986). Harley and Black (1997) suggested that UHT granulite-facies metamorphism occurred at 2.82–2.84 Ga deduced from their SHRIMP U-Pb geochronology. However, recent SHRIMP and CHIME dating of zircon and monazite in the granulite have yielded abundant Late Archean~Early Proterozoic ages of 2.4 to 2.5 Ga (Hokada *et al.*, 2001, 2003, 2004; Asami *et al.*, 2002; Carson *et al.*, 2002a, b; Suzuki *et al.*, 2006). Hokada *et al.* (2001, 2003, 2004), Asami *et al.* (2002) and Suzuki *et al.* (2006) suggest that most rocks in the Napier Complex underwent UHT granulite-facies metamorphism at about 2.4–2.5 Ga. On the other hand, Harley *et al.* (2001), Crowe *et al.* (2002) and Kelly and Harley (2005) infer it to be somewhat older, >2.52 Ga, and up to \cong 2.59 Ga. In their research, Kelly and Harley (2005) reported U-Pb ages of zircon from metamorphic rocks on Gage Ridge and Dallwitz Nunatak where there are easterly fields in the western Napier Complex. A reason for the disagreement might be the local difference of geothermal history in the Napier Complex. Further regional chronological research is required to understand the thermal history of the Napier Complex.

Many chronological studies have been performed to decide the timing of peak metamorphism and the ages of older igneous events from detrital materials in the Napier Complex; however, chronological studies for post-peak thermal evolution are few (Fig. 1). The Napier Complex has matured as a continental lithosphere after UHT granulite facies metamorphism. Research on geothermal history after peak metamorphism is important for revealing the structure and evolution of the Archean continental crust. Only a few geochronological studies at Mount Riiser Larsen, Tonagh Islands, and some outcrops around Casey Bay, however, have suggested the possibility of a geothermal event after peak metamorphism (Grew *et al.*, 2001; Owada *et al.*, 2001, 2003; Suzuki *et al.*, 2001, 2006; Carson *et al.*, 2002a, Fig. 1). The outcrops around Casey Bay are close to the Rayner Complex, and are probably influenced by Proterozoic metamorphism at the complex. Mount Riiser Larsen and the Tonagh Islands are far from the Rayner Complex; however, these crops are probably also influenced by the Proterozoic tectonothermal event of the complex (Owada *et al.*, 2003). We can find their trace in metamorphic rock samples in the Napier Complex. Therefore, further systematic research on the thermal history after peak metamorphism is required to understand the history of transition from dynamic to static conditions during the maturation of the continental crust. In order to constrain the timing of later thermal events, especially finding guideposts for the temperature-time relation after peak metamorphism, in the thermal history of the Napier Complex, Rb-Sr, Sm-Nd and U-Pb analyses were done on

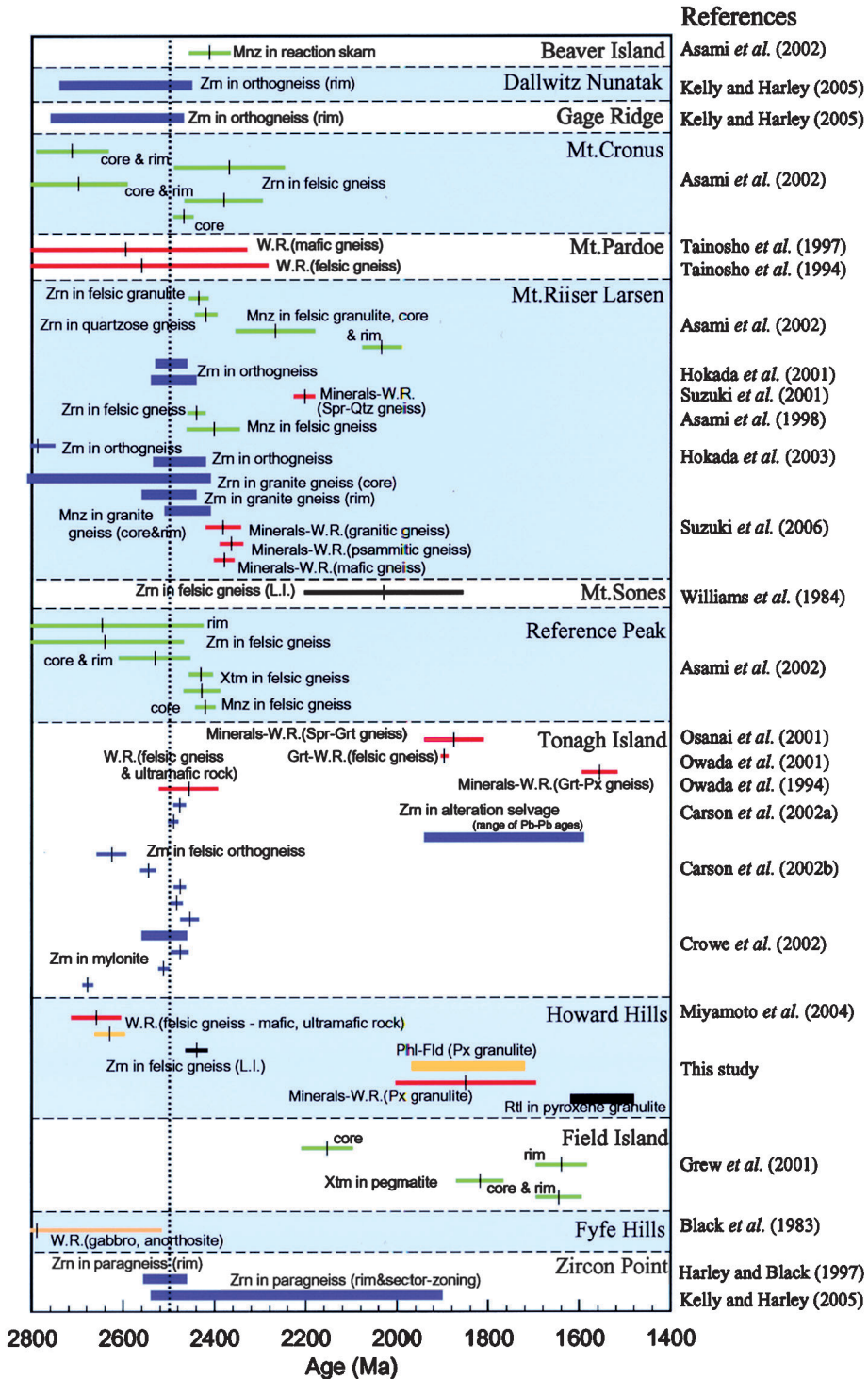


Fig. 1.

mineral fractions from metamorphic rocks in the Howard Hills, Enderby Land, East Antarctica, and the relation between dating results and geothermal conditions considered. The mineral fractions used are zircon, rutile, orthopyroxene, feldspar, and phlogopite which have different closure temperatures (T_c). Their dating results probably give us guideposts on the thermal history of the Napier Complex, even if texture and mineral assemblages in metamorphic rocks were poorly modified after peak metamorphism.

2. General geology and sample description

2.1. General geology and occurrence of dated sample

The Howard Hills lie on the eastern shore of Amundsen Bay in Enderby Land (Fig. 2), and consist mainly of UHT granulite-facies metamorphic rocks (Yoshimura *et al.*, 2000). The basement rocks include alternating layers of garnet-bearing felsic gneiss and orthopyroxene-bearing felsic gneiss, with minor amounts of metamorphosed mafic to ultramafic rocks occurring sporadically as thin lenticular layers (tens of centimeters to meters across). Peak metamorphic conditions of the gneisses in the Howard Hills have reached 1150–1200°C (Yoshimura *et al.*, 2000). Metamorphic rocks are intruded by unmetamorphosed basaltic to doleritic Amundsen dykes at *ca.* 1.2 Ga (Sheraton and Black, 1981; Sheraton *et al.*, 1987).

We investigated felsic gneisses cropping out in the northern part of the Howard Hills. The felsic gneiss contains an over 5 m elongated block of metamorphosed mafic to ultramafic rocks (Fig. 3). The metamorphosed ultramafic rock is composed of olivine with subordinate orthopyroxene, spinel and phlogopite, and is locally penetrated by fine serpentine veins (Miyamoto *et al.*, 2004). Pyroxene granulite divides the ultramafic core from the felsic gneiss and comprises orthopyroxene mainly with small clinopyroxene and plagioclase, with granoblastic texture. A phlogopite-rich section is present in the boundary zone with the felsic gneiss (Fig. 3b). Small patches of orthopyroxene felsic gneiss also occur in the pyroxene granulite.

2.2. Petrography and mineral remarks of dated samples

Phlogopite-bearing pyroxene granulite (TM981229-03E) and orthopyroxene felsic gneiss (TM981229-03Ef) were taken from the edge of the mafic block (Fig. 3b). Sample TM981229-03E is composed mainly of orthopyroxene, with interstitial phlo-

Fig. 1 (opposite). Compiled age data between 2.8 and 1.4 Ga for metamorphic rocks from the Napier Complex. A vertical dotted line shows the Archean-Proterozoic boundary. Red: Sm-Nd isochron age, Orange: Rb-Sr isochron age, Green: U-Th-total Pb CHIME age, Blue: U-Pb SHRIMP age, and Black: U-Pb age defined by conventional method. Thin colored lines represent the data values of one analysis with error ranges. Thick colored lines show the dated ranges involving a series of analyses. "U.I." and "L.I." represent the upper and lower intercept ages for discordant U-Pb age. Fld: feldspar, Grt: garnet, Mnz: monazite, Phl: phlogopite, Px: pyroxene, Qtz: quartz, Rtl: rutile, Spr: sapphirine, Xtm: xenotime, Zrn: zircon, and W.R.: whole rock sample. It is clear that dating results for SHRIMP U-Pb dating and U-Th-Pb CHIME dating methods of zircon and monazite are concentrated between the Late Archean and the Early Proterozoic. Other methods mostly show Proterozoic.

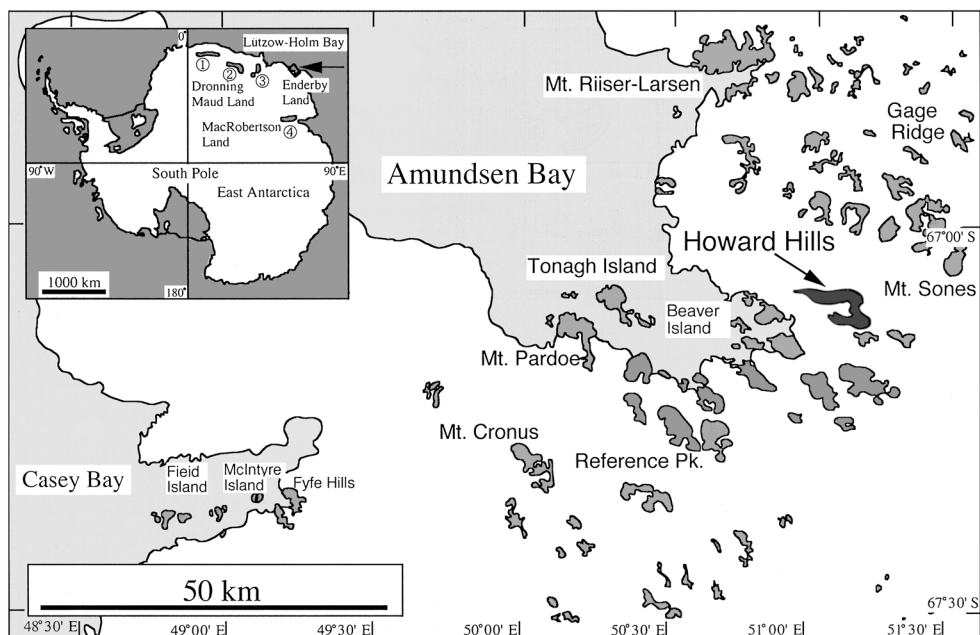


Fig. 2. Location map of the Howard Hills in western Enderby Land, East Antarctica. Inset, ①: Schirmacher Hills, ②: Sør Rondane Mountains, ③: Yamato-Belgica Complex, and ④: Prince Charles Mountains.

gopite, alkali feldspar, plagioclase, sapphirine and spinel. Sapphirine occurs as porphyroblasts with core parts of greenish spinel and as thin films along the grain boundaries of granoblastic orthopyroxene. This spinel-sapphirine corona was synthesized artificially for this granulite sample at high-pressure (9 kbar) and high-temperature (1100°C) during an annealing experiment involving heating to 1200°C (Sato *et al.*, 2004a). The sapphirine films locally contact quartz (Miyamoto *et al.*, 2004). Alkali feldspar is sanidine and/or perthite. Rutile is present as a minor phase, and occurs along grain boundaries and as acicular inclusions in orthopyroxene. Orthopyroxene shows aluminous composition, and the content decreases from the core (9.9 wt% of Al₂O₃, 0.42 per 6-oxygen formula unit) to the rim (5.3 wt%, 0.22 per 6-oxygen formula unit) although their Mg/Fe ratios have small differences (Mg/(Mg + Fe_{total}) = 0.76–0.79) (Miyamoto *et al.*, 2004). Large orthopyroxene grains include rutile, phlogopite and unidentified fine minerals. Phlogopite is rich in fluorine (nearly 4 wt%; Sato *et al.*, 2004b) and poor in H₂O (H₂O(–) = 0.06 wt%, H₂O(+) = 0.54 wt%, as determined by the Karl-Fischer method (Muroi, 1979)).

Sample TM981229-03Ef consists of quartz, plagioclase, alkali feldspar and orthopyroxene. Alkali feldspar represents perthite to mesoperthite. Rutile and zircon occur as accessory minerals in the matrix and as inclusions in orthopyroxene. Some zircon grains show oscillatory zoning without an inherited-core (Fig. 4a), and others have an inherited core with mosaic texture (Fig. 4b). Quartz, feldspar, pyroxene and biotite were found in some zircon grains as inclusions (Figs. 4a, b and c). Chemical

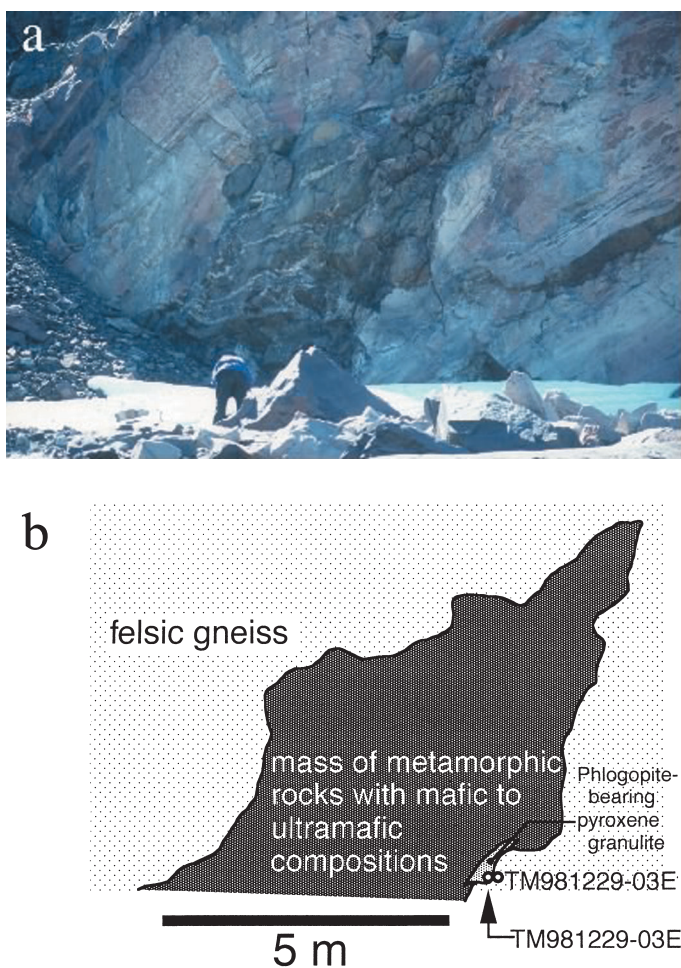


Fig. 3. Occurrence of metamorphosed ultramafic block and granulite in felsic gneiss (a; photograph, b; sketch, with sampling points of TM981229-03E and TM981229-03Ef). Their petrographies and compositions are described in Miyamoto *et al.* (2004).

compositions of pyroxene and biotite included in zircon are listed in Table 1. Evidence of hydrothermal activity at a later stage after the UHT metamorphism was not found, except for occurrence of serpentine in the associated metamorphosed ultramafic rock.

2.3. Compositional remarks of the rock samples

Major and minor element compositions of phlogopite-bearing pyroxene granulite (TM981229-03E), orthopyroxene felsic gneiss (TM981229-03Ef) and associated rocks are reported by Miyamoto *et al.* (2004). Estimated Nd model ages of the orthopyroxene felsic gneiss (TM981229-03Ef; Miyamoto *et al.*, 2004) are $t_{\text{Nd,CHUR}} = 3.37 \pm 0.16$ Ga and $t_{\text{Nd,DM}} = 3.50 \pm 0.17$ Ga (based on $^{143}\text{Nd}/^{144}\text{Nd}$ (present) = 0.512638 and $^{147}\text{Sm}/^{144}\text{Nd}$ (present) = 0.1966 for chondritic uniform reservoir (CHUR) parameters

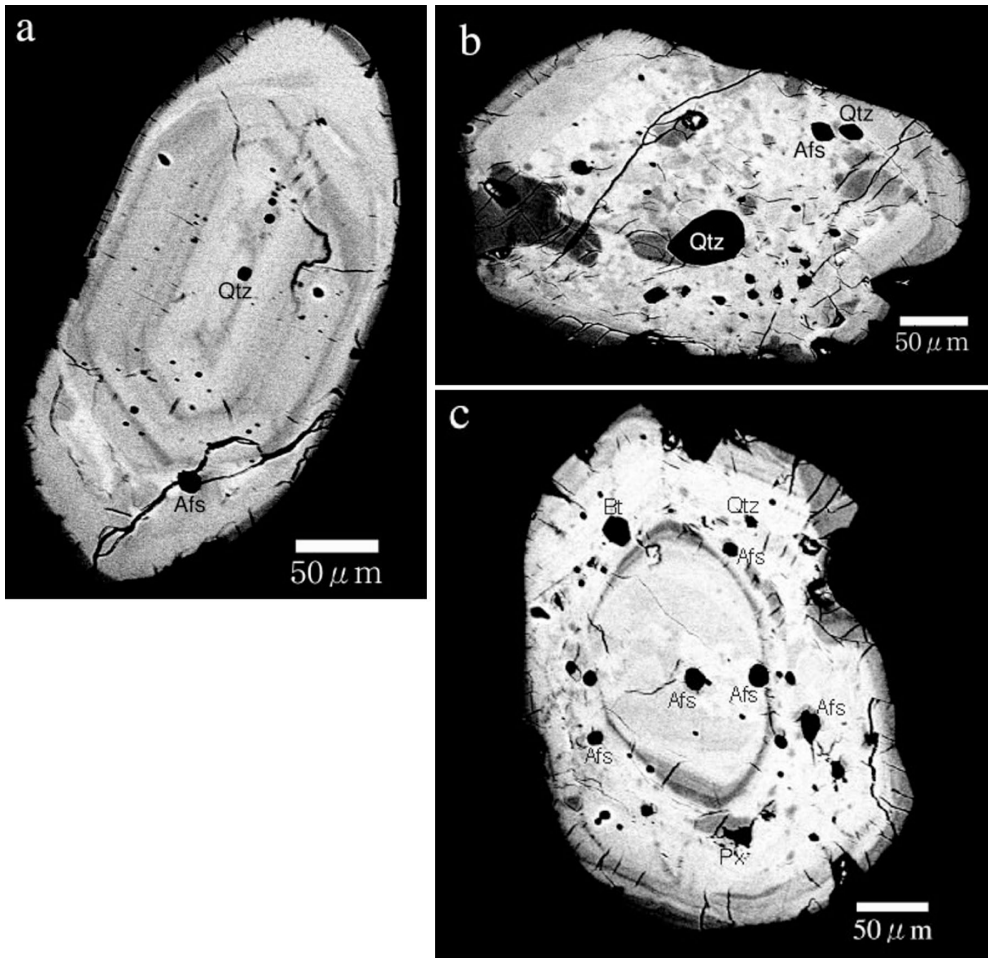


Fig. 4. Back scattered electron (BSE) images of zircon grain prepared for U-Pb isotopic analyses from orthopyroxene felsic gneiss (TM981229-03Ef). The images were pictured by a scanning electron microscope (SEM: JEOL JSM-5800LV) with an energy dispersive X-ray analytical system (EDS: OXFORD Link ISIS) at Kyushu University. For chemical zoning and mosaic core in the images, light and dark areas are Hf rich and poor, respectively. Zoning (a) and mosaic core (b) are faintly visible. (c) Zircon with many kind of inclusions in the same grain. Afs: alkali-feldspar, Bt: biotite, Px: pyroxene, Qtz: quartz. Chemical compositions of biotite and pyroxene inclusions are listed in Table 1.

(Hamilton *et al.*, 1983), and $^{143}\text{Nd}/^{144}\text{Nd}$ (present) = 0.51315 and $^{147}\text{Sm}/^{144}\text{Nd}$ (present) = 0.2137 for depleted mantle (DM) parameter (Peucat *et al.*, 1988)). In that study, Rb-Sr and Sm-Nd isotope data of the metamorphic rocks were arranged as lines on the isochron diagrams with contemporary apparent ages of about 2.65 Ga within analytical errors with ordinate intercepts with $^{87}\text{Sr}/^{86}\text{Sr} = 0.7357 \pm 0.0024$ and $^{143}\text{Nd}/^{144}\text{Nd} = 0.50856 \pm 0.00004$ ($\epsilon_{\text{Nd,CHUR}}^{\text{isochron,t}} = -12.3 \pm 0.9$). Miyamoto *et al.* (2004) interpreted these whole rock isochron ages as the time of UHT granulite facies metamorphism with

Table 1. Chemical compositions of pyroxene and biotite included in a zircon from TM981229-03Ef (Fig. 4c).

	pyroxene	biotite
wt%		
SiO ₂	47.13	41.01
TiO ₂	0.06	4.97
Al ₂ O ₃	14.61	13.57
FeO*	12.51	6.93
MnO	0.12	0.10
MgO	21.29	19.60
CaO	2.46	
Na ₂ O	1.02	1.01
K ₂ O		9.36
Total	99.20	96.55
Cations	(O=6)	(O=22)
Si	1.697	5.801
Ti	0.002	0.529
Al	0.620	2.262
Fe	0.377	0.820
Mn	0.004	0.012
Mg	1.143	4.133
Ca	0.095	
Na	0.071	0.276
K		1.690
Total Cations	4.009	15.523

Chemical compositions were determined using a scanning electron microscope (SEM: JEOL JSM-5800LV) with an energy dispersive X-ray analytical system (EDS: OXFORD Link ISIS) at Kyushu University. * Total Fe as FeO.

partial melting of LILE-enriched protoliths, after denying the possibility of pseudoisochrons due to mechanical mixing among the dated samples in terms of petrochemical evidence.

3. Analytical procedure

Phlogopite, orthopyroxene, feldspar and rutile were separated from phlogopite-bearing pyroxene granulite (TM981229-03E). Zircon was also taken from the orthopyroxene felsic gneiss (TM981229-03Ef). The minerals were concentrated by magnetic separation with a Frantz isodynamic separator and by gravity separation with tetrabromoethane and methylene iodide. All mineral fractions were purified by hand-picking under a binocular-microscope.

For Rb-Sr and Sm-Nd isotopic analysis, 100–200 mg of each mineral fraction was used. They were cleaned with acetone and distilled water before decomposition. Conventional isotope dilution methods were applied to determine Rb, Sr, Sm and Nd concentrations of the samples. A decomposed sample fraction was resolved in a

mixture of hydrochloric acid (HCl) and oxalic acid ((COOH)₂), and passed through DOWEX 50W-X8, 200 mesh cation exchange resin to separate Rb, Sr and REE. Samarium and neodymium were dissolved by 0.2N 2-methylactic acid, and separated from the REE by passing through DOWEX 50W-X8, 200 mesh cation exchange resin adjusted with ammonia, following the method of Kubota (1992) modified from Notsu *et al.* (1973). Rubidium concentrations were determined with a HITACHI RMU5G mass spectrometer with a single collector. Strontium and neodymium isotope compositions and total Sr, Sm and Nd concentrations were determined with a JEOL JMS05RB mass spectrometer with a single collector. The strontium standard of Eimer and Amend gave $^{87}\text{Sr}/^{86}\text{Sr}=0.7080\pm 0.0001$ (1σ) and standard NBS983 gave $^{87}\text{Sr}/^{86}\text{Sr}=0.7103\pm 0.0001$ (1σ). The neodymium standard of JNdi-1 (GSJ (Geological Survey of Japan) standard with recommended value of $^{143}\text{Nd}/^{144}\text{Nd}=0.512115\pm 0.000007$; Tanaka *et al.*, 2000) gave $^{143}\text{Nd}/^{144}\text{Nd}=0.51212\pm 0.00004$ (1σ). Relative analytical errors in Rb, Sr, Sm and Nd concentrations are 2%, 1%, 0.5% and 1%, respectively. Contamination levels of Rb, Sr, Sm and Nd are under 1×10^{-10} g, 3×10^{-10} g, 1×10^{-10} g and 1×10^{-10} g per sample.

For U-Pb isotopic analysis, 10–30 dark reddish-brown grains of rutile were picked for each run (Nos. 593, 599, and 602 in Table 2). For zircon analysis, one (Nos. 638, 642, 644 and 645), two (Nos. 637 and 641), and four (No. 636) grains were used as sample fractions. All zircon grains chosen are dark-colored and translucent with smooth surfaces. All rutile fractions and one zircon fraction (No. 636) were cleaned with warmed dilute nitric acid to remove common lead contamination on grain surfaces. Other zircon fractions were cleaned by warming in distilled water for an hour for surface cleaning before decomposition. Conventional isotope dilution methods were applied to determine U and Pb compositions of the samples. Chemical treatment and analytical method for U-Pb isotope determination in zircon and rutile followed principally the conventional method of Krogh (1973), and is described in Miyamoto and Yanagi (1996).

The decay constants used for age calculations are 6.54×10^{-12} year⁻¹ for ^{147}Sm (Lugmair and Marti, 1978), 1.42×10^{-11} year⁻¹ for ^{87}Rb , 9.8485×10^{-10} year⁻¹ for ^{235}U and 1.55125×10^{-10} year⁻¹ for ^{238}U , and the present $^{238}\text{U}/^{235}\text{U}$ ratio is 137.88 (Steiger and Jäger, 1977). Samarium-neodymium isochrons were determined by the least squares regression method of York (1966). Error estimation with correlations for U-Pb isotope ratios follows Ludwig (1980). A discordia was defined from the U-Pb isotope ratios of zircon by the least squares regression method of York (1969) for data with correlated errors.

4. Results

Analytical U-Pb isotope data for rutile fractions from phlogopite-bearing pyroxene granulite (TM981229-03E) and zircon fractions from orthopyroxene felsic gneiss (TM 981229-03Ef) are listed in Table 2. A concordia diagram for the U-Pb compositions is shown in Fig. 5. Some of the zircon fractions have almost concordant U-Pb compositions that correspond to ages of around 2.45 Ga (Fig. 5a). Forced extrapolation of U-Pb compositions of zircon fractions except No. 636 make us estimate a discordia with

Table 2. Analytical data for rutile fractions in phlogopite-bearing pyroxene granulite (TM981229-03E) and zircon fractions in the orthopyroxene-bearing felsic gneiss (TM981229-03Ef) from the Howard Hills, Enderby Land, East Antarctica.

Sample	U (ppm)	Pb (ppm)	$^{204}\text{Pb}/^{206}\text{Pb}^*$	$^{207}\text{Pb}/^{206}\text{Pb}^*$	$^{208}\text{Pb}/^{206}\text{Pb}^*$	t_{206} (Ga)*	t_{207} (Ga)*	$t_{207-206}$ (Ga)*
Rutile fractions in phlogopite-bearing pyroxene granulite (TM981229-03E)								
No.593	63.4	16.1	0.0003 ± 0.0003	0.1022 ± 0.0087	0.0104 ± 0.0050	1.51 ± 0.49	1.54 ± 0.32	1.58 ± 0.25
No.599	63.9	14.9	0.0001 ± 0.0001	0.0949 ± 0.0026	0.0058 ± 0.0010	1.42 ± 0.17	1.46 ± 0.11	1.50 ± 0.09
No.602	66.6	15.0	0.0003 ± 0.0003	0.1026 ± 0.0026	0.0095 ± 0.0007	1.35 ± 0.08	1.45 ± 0.05	1.58 ± 0.09
Zircon fractions in Opx-bearing felsic gneiss (TM981229-03Ef)								
No.636	1240	572	0.0001 ± 0.0001	0.1711 ± 0.0002	0.0488 ± 0.0002	2.36 ± 0.04	2.47 ± 0.02	2.56 ± 0.01
No.637	672	322	0.0002 ± 0.0002	0.1631 ± 0.0002	0.0582 ± 0.0005	2.43 ± 0.03	2.45 ± 0.02	2.46 ± 0.01
No.638	819	427	0.0010 ± 0.0001	0.1772 ± 0.0003	0.1001 ± 0.0001	2.48 ± 0.02	2.49 ± 0.01	2.51 ± 0.01
No.641	452	220	0.0002 ± 0.0001	0.1671 ± 0.0001	0.0555 ± 0.0001	2.47 ± 0.01	2.49 ± 0.01	2.51 ± 0.01
No.642	245	118	0.0001 ± 0.0001	0.1620 ± 0.0002	0.0623 ± 0.0003	2.44 ± 0.02	2.45 ± 0.01	2.46 ± 0.01
No.644	1081	529	0.0001 ± 0.0001	0.1746 ± 0.0001	0.0409 ± 0.0002	2.50 ± 0.02	2.56 ± 0.01	2.60 ± 0.01
No.645	500	244	0.0003 ± 0.0003	0.1724 ± 0.0003	0.0368 ± 0.0002	2.49 ± 0.02	2.53 ± 0.02	2.56 ± 0.01

*Errors given are $\pm 1\sigma$. For error calculation, see Ludwig (1980).

lower intercept age of 2.44 ± 0.02 Ga and upper intercept age of 3.9 ± 0.5 Ga. Rutile fractions have near concordant U-Pb compositions (Fig. 5b) and correspond to ages of 1.5–1.6 Ga, which is the youngest age estimation among U-Pb, Sm-Nd, and Rb-Sr results in this study.

Analytical Rb-Sr isotopic compositions for feldspar and phlogopite fractions from phlogopite-bearing pyroxene granulite are shown in Table 3. Phlogopite fractions contain abundant rubidium, and show extremely high Sr isotope ratios. Model ages of phlogopite (t_{model}) were determined as 2-points isochron ages for all fractions by assuming an initial ratio equal to the Rb-Sr isotopic ratio of the feldspar fraction (Fig. 6). The t_{model} values are 1.79–1.90 Ga with analytical errors of about 0.06 Ga.

Analytical Sm-Nd isotopic data for whole-rock, feldspar and orthopyroxene fractions from phlogopite-bearing pyroxene granulite are shown in Table 4. Isotope ratios approximate a regression line with a slope corresponding to an age of 1.85 ± 0.15 Ga with an initial ratio of $(^{143}\text{Nd}/^{144}\text{Nd})_{\text{I.R.}} = 0.50907 \pm 0.00011$ on an isochron diagram (Fig. 7).

5. Discussion

The pyroxene granulite and felsic gneiss on the Howard Hills were residue and/or influenced by melt generated during partial melting of LILE-enriched protoliths with high Sr isotope ratio (Yoshimura *et al.*, 2000; Miyamoto *et al.*, 2004). Partial melting under granulite-facies conditions can cause growth of zircon grains in metamorphic rocks (Rubatto *et al.*, 2001). It is significant that zircon includes biotite and pyroxene in addition to quartz and feldspar (Fig. 4c). The biotite inclusion might be evidence of a protolith of metamorphic rocks since biotite was not found in the host felsic gneiss matrix (TM981229-03Ef). The pyroxene has aluminous composition (14.61 wt% of Al_2O_3 , corresponding to 0.62 Al atoms per 6 oxygens) with $\text{Mg}/(\text{Mg} + \text{Fe}_{\text{total}}) = 0.75$.

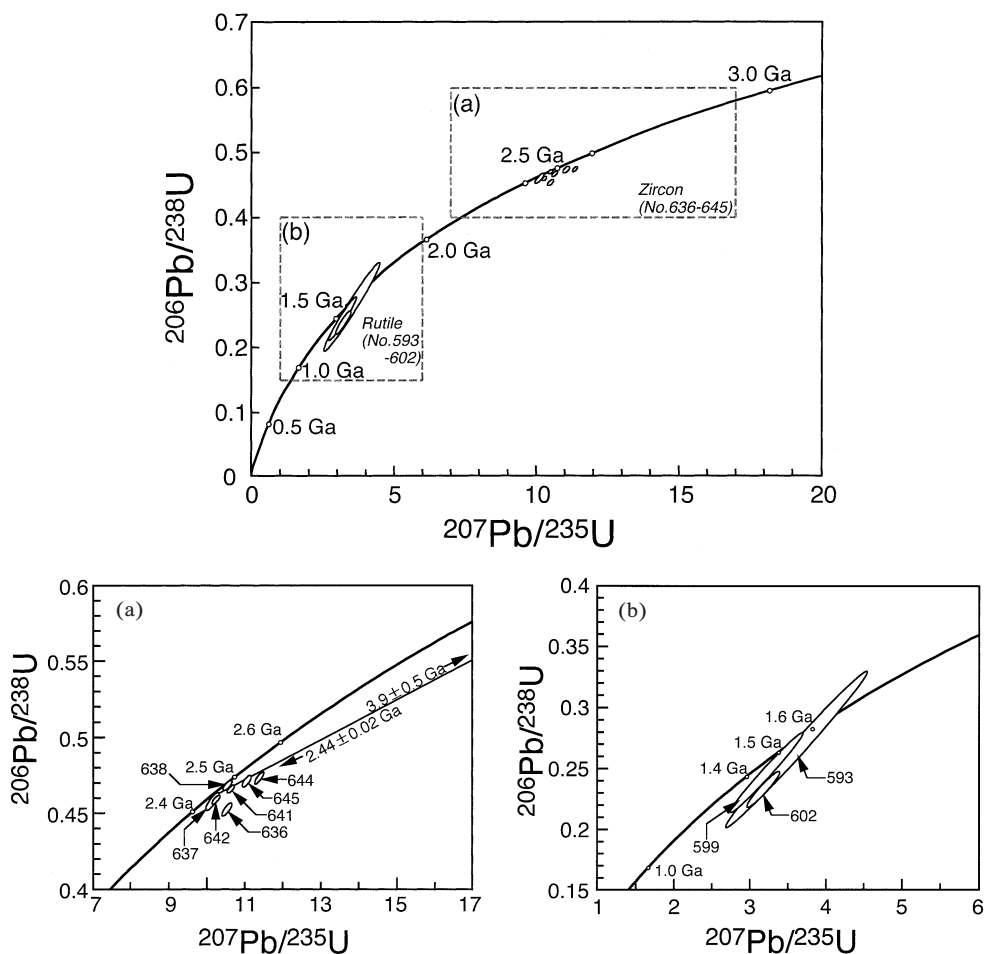


Fig. 5. U-Pb concordia diagram for zircon grains from orthopyroxene felsic gneiss (TM981229-03E_f) and rutile fractions from phlogopite-bearing pyroxene granulite (TM981229-03E). Figures 5a and 5b are expanded diagrams for the zircon and rutile fractions. Ellipses express data points for each fraction with correlated errors. Rutile fractions which are dark reddish-brown under the microscope comprise 10–30 grains (Nos. 593, 599, and 602). For zircon fractions (Nos. 636–646), Nos. 638, 642, 644 and 645 fractions are single grain, Nos. 637 and 641 fractions comprise two grains, and the No. 636 fraction comprises four grains. Forced extrapolation of U-Pb compositions of zircon fractions except No. 636 estimates a discordia with lower intercept age of 2.44 ± 0.02 Ga and upper intercept age of 3.9 ± 0.5 Ga.

These characteristics are consistent with composition of porphyroblastic orthopyroxene in the gneiss matrix. Therefore, the zircon potentially grew before or at the time of reaction with biotite by melting and/or after production of pyroxene.

The composition of No. 636 fraction lies between those of the others and the origin on the concordia diagram (Fig. 5). Since this zircon fraction was rinsed with warmed nitric acid diluent, the discordant composition is probably attributed to Pb loss from

Table 3. Analytical data for feldspar and phlogopite fractions in phlogopite-bearing pyroxene granulite (TM981229-03E) from the Howard Hills, East Antarctica.

	Rb (ppm)	Sr (ppm)	$^{87}\text{Rb}/^{86}\text{Sr}$	$^{87}\text{Sr}/^{86}\text{Sr}^*$	t_{model} (Ga)*
Feldspar	61.5	305	0.589	0.79958 ± 0.00008	
Phlogopite					
0.50–0.71 mm	1382	24.3	297	8.8971 ± 0.0004	1.90 ± 0.06
0.35–0.50 mm	1802	24.0	486	13.296 ± 0.003	1.79 ± 0.06
0.25–0.35 mm	1700	19.8	704	19.497 ± 0.002	1.85 ± 0.06

* Error given are $\pm 1\sigma$. Model ages (t_{model}) of phlogopite fractions were estimated on from the pairs of feldspar and phlogopite fractions.

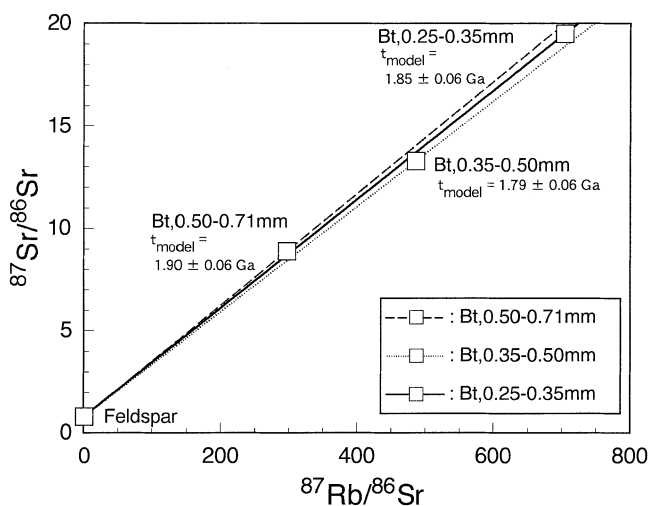


Fig. 6. Rb-Sr isotope ratios of phlogopite and feldspar from pyroxene granulite (TM981229-03E) on an isochron diagram. Three model isochron ages (t_{model}) connecting with the feldspar fraction and three phlogopite fractions are also given in Table 3.

metamict zircon during this cleaning process.

An extrapolation of U-Pb compositions of zircon fractions except No. 636 shows 3.9 ± 0.5 Ga as the upper intercept age (Fig. 5a). Unfortunately, this upper intercept age has large uncertainty although it is similar to the Nd model age of the host rock ($t_{\text{Nd,CHUR}} = 3.37 \pm 0.16$ Ga and $t_{\text{Nd,DM}} = 3.50 \pm 0.17$ Ga for TM981229-03E; Miyamoto *et al.*, 2004); therefore, it does not require detailed examination. One thing, however, is certain: this discordance is consistent with the presence of an inherited domain in some zircons (Fig. 4b). The discordant U-Pb composition is probably followed by the existence of a surviving old domain in zircon as an inherited core.

The extrapolation shows 2.44 ± 0.02 Ga as the lower intercept age (Fig. 5a). This age probably shows an age of UHT granulite facies metamorphism. However, Miyamoto *et al.* (2004) reported simultaneous 2.65 Ga by Rb-Sr and Sm-Nd whole rock isochron ages using metamorphic rocks with ultramafic to felsic compositions on the

Table 4. Analytical data for whole rock and mineral fractions of phlogopite-bearing pyroxene granulite (TM981229-03E) from the Howard Hills, East Antarctica.

	Sm (ppm)	Nd (ppm)	$^{147}\text{Sm}/^{144}\text{Nd}$	$^{143}\text{Nd}/^{144}\text{Nd}^*$
Whole rock (W.R.)	1.33	6.89	0.117	0.51058 ± 0.00005
Orthopyroxene (Opx)				
0.50–0.71 mm	0.489	1.68	0.176	0.51116 ± 0.00006
0.35–0.50 mm	0.456	2.13	0.126	0.51063 ± 0.00008
Feldspar	2.22	22.6	0.0595	0.50976 ± 0.00002

* Error given are $\pm 1\sigma$.

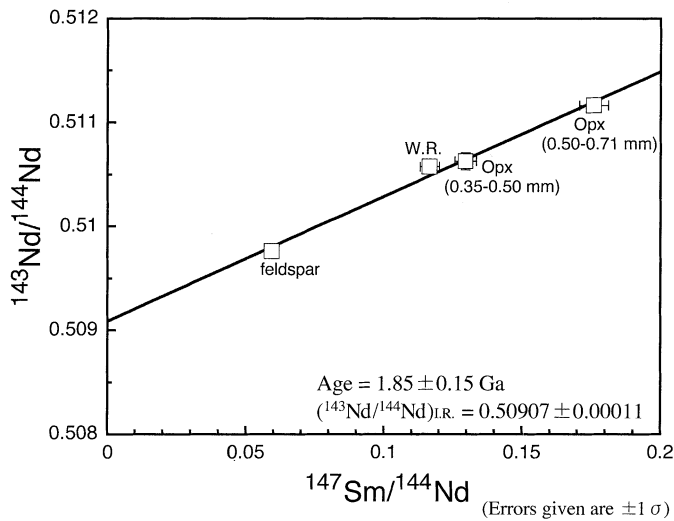


Fig. 7. Sm-Nd isochron diagram for mineral fractions and whole rock samples. Errors given are $\pm 1\sigma$. 3% errors were estimated for Sm/Nd ratios of each sample as a convention for isochron determination. MSWD for the isochron was estimated below 1.0. Letters identifying points refer to the sample. Small deviation of whole rock sample (W.R.) from regression may be caused by occurrence of spinel which is regarded as a container of REE in a rock sample.

Howard Hills. He concluded that the 2.65 Ga whole rock age indicates the time of UHT granulite facies metamorphism with partial melting of LILE-enriched protoliths with high Sr isotope ratio, after eliminating the possibility of pseudoisochrons by mechanical mixing of the analyzed samples. Ellis and Green (1985) and Harley (1985, 1998) proposed that isobaric cooling took place in the UHT metamorphic rocks after peak metamorphic condition in the Napier Complex. In addition, Yoshimura *et al.* (2000) also suggested the same possibility for metamorphic rocks from the Howard Hills. Cherniak and Watson (2000) suggested that zircon with $100\mu\text{m}$ radius would lose over 90% of Pb if residing at 1000°C for 10^7 – 10^8 years, at 1100°C for 10^6 years or at 1200°C for 10^5 years. Occurrence of fluid activity produces much Pb-loss from zircon crystal during metamorphism at even lower temperature condition (Pidgeon *et al.*,

1966; Sinha *et al.*, 1992). Consequently, the radiogenic-Pb clock of zircon can be reset to a high temperature ($>1000^{\circ}\text{C}$) if such high temperature is continued over 10 million years. The younger U-Pb zircon age than whole rock ages for metamorphic rocks from the Howard Hills can be explained by continuation of the UHT granulite facies metamorphism from 2.65 Ga to 2.44 Ga.

The period of UHT metamorphism for the Napier Complex is estimated as 0.1 billion years by Kelly and Harley (2005) and as 0.06 billion years or shorter by Suzuki *et al.* (2006). In this study, the time-interval between the U-Pb zircon age and the Rb-Sr and Sm-Nd whole rock ages is 0.21 billion years, and corresponds to twice the period of UHT metamorphism estimated by Kelly and Harley (2005) and over three times that estimated by Suzuki *et al.* (2006). However, previously reported ages of UHT metamorphism range from 2.59 to 2.40 Ga (Fig. 1). The length of this period is equivalent to the time-interval between the U-Pb zircon age and the whole rock ages for the Howard Hills. The length of this time-interval is not unreasonable for the period of UHT metamorphism in the Napier Complex. We should consider the continuation of the UHT granulite facies metamorphism after obtaining more regional and systematic dating results from the Napier Complex.

Rutile fractions have near concordant U-Pb ages of *ca.* 1.5 Ga. Mezger *et al.* (1989) estimated the closure temperature (T_c) of rutile as 350–450 $^{\circ}\text{C}$ from field evidence. This T_c is lower than those in other minerals used for U-Pb dating, such as garnet, zircon, monazite, xenotime or titanite. Cherniak (2000) has also suggested from the diffusion parameter that Pb can be moved easily from rutile crystals under low-temperature geothermal activity. Therefore, we interpret the *ca.* 1.5 Ga rutile age as representing the last stage of the regional thermal condition with low-temperature in the Napier Complex, except for intrusion of the Amundsen dykes or pegmatite (Sheraton and Black, 1981; Osanai *et al.*, 2001). The fine serpentine vein penetrated in metamorphosed ultramafic rocks in the mafic to ultramafic block (Miyamoto *et al.*, 2004) is evidence of minor geothermal activity at low temperature in the Howard Hills. No other obvious trace of retrograde metamorphism with reaction nor deformation texture was found in the samples and associated rocks. This minor hydrothermal activity might have occurred at around 1.5–1.6 Ga.

Phlogopite occurs with feldspar (perthite, sanidine, and plagioclase) in the interstices of granoblastic orthopyroxene in pyroxene granulite. Feldspar is the most suitable mineral for diffusive exchange of Sr with phlogopite since other minerals can contain negligible strontium. A Rb-Sr model age for phlogopite, therefore, shows timing of equilibration between phlogopite and feldspar in the sample.

Phlogopite fractions from pyroxene granulite yielded Rb-Sr model ages (t_{model}) of around 1.85 Ga (Fig. 6), although the phlogopite also shows different Rb compositions in differing grain sizes (Table 3). Biotite grains can locally preserve heterogeneous compositions corresponding to different ages of their crystallization (Hart, 1964; Yamashita and Yanagi, 1994). The compositional variation is probably originated from their heterochronous crystallization. Biotite and phlogopite are susceptible to homogenization of Sr isotopic ratios under relatively low-temperature conditions. Similarity of t_{model} between phlogopite fractions of differing grain sizes reflects an influence of re-equilibration of Sr isotope ratio between phlogopite and feldspar. The phlogopite,

however, contains much fluorine. Enhancing fluorine content in phlogopite can raise the closure temperature (T_c) depending on the effect of Sr diffusion (Hammouda and Cherniak, 2000; Kühn *et al.*, 2000), as well as expanding the stability field toward higher temperature (Munoz, 1984; Motoyoshi and Hensen, 2001). The presence of fluorophlogopite with *ca.* 1.85 Ga gave us the highest limit of temperature from its T_c , which metamorphic rocks suffered at that time.

There is a difference in Sm/Nd ratio between large and small orthopyroxene fractions (Table 4; Fig. 7). The orthopyroxene includes fine rutile, phlogopite, and unidentified minerals. Suzuki *et al.* (2006) suggests that the existence of inclusions such as monazite modifies Sm/Nd ratios of host mineral fractions. The compositional difference among orthopyroxene fractions probably depends on quantitative variation of the inclusions as monazite, apatite, or zircon, although their influences were not analyzed either quantitatively or qualitatively for this sample. Neodymium composition of the mineral is generally believed to be resistant to disturbance by metamorphism and hydrothermal alteration. Neodymium isotopic compositions of orthopyroxene in granulite or peridotite, however, sometimes deviate from precisely determined internal isochrons (“cryptic retrogression”, Burton and O’Nions, 1990; Pan *et al.*, 1999; Zhang *et al.*, 2001). Re-equilibration of Nd isotope ratios in orthopyroxene is believed to take place at amphibolite to granulite-facies conditions.

The *ca.* 1.85 Ga age from Rb-Sr and Sm-Nd mineral dating significantly postdates the UHT granulite facies metamorphism. Previous geochronological research at Mount Riiser Larsen, Tonagh Islands, and some outcrops around Casey Bay have suggested a possibility of a geothermal event following peak metamorphism regionally in the Napier Complex (Grew *et al.*, 2001; Osanai *et al.*, 2001; Owada *et al.*, 2001, 2003; Suzuki *et al.*, 2001, 2006, Fig. 1). Especially, Osanai *et al.* (2001) and Owada *et al.* (2001, 2003) reported Sm-Nd internal isochron ages with *ca.* 1.9 Ga and 1.6 Ga. Geochronological results in this study are consistent with the previous works. Suzuki *et al.* (2000), and Owada *et al.* (2001, 2003) have suggested the possibility of a tectonothermal event involving tholeiitic magmatism at *ca.* 1.9 Ga that affected gneiss at Mount Riiser Larsen and on Tonagh Island, the Napier Complex. Unmetamorphosed basaltic to doleritic dykes that occur in the Howard Hills are considered to be much younger (*ca.* 1.2 Ga, Sheraton and Black, 1981). Although no field evidence of igneous activity at *ca.* 1.9 Ga has been found in the Howard Hills, Rb-Sr and Sm-Nd mineral ages support a possibility of regional tectonothermal event that influenced the Napier Complex after UHT granulite-facies metamorphism.

Grew *et al.* (2001) reported a CHIME U-Th-Pb apparent age of 1819 ± 48 Ma for xenotimes in a beryllium pegmatite and another age of 2154 ± 53 Ma for a xenotime in another pegmatite from Christmas point on the Napier Complex (Fig. 1). The mineral age of 1.85 Ga agrees with the xenotime CHIME ages. A closure temperature for the U-Pb system of the xenotime is estimated at $\geq 650^\circ\text{C}$ (Heaman and Parrish, 1991) and up to about 800°C (Kamber *et al.*, 1998). Suzuki *et al.* (2001, 2006) reported Sm-Nd internal isochron ages of 2.2–2.4 Ga and regard the age as a record of cooling below $600\text{--}700^\circ\text{C}$. These ages are older than the tectonothermal event involving tholeiitic magmatism at *ca.* 1.9 Ga in Mount Riiser Larsen (Suzuki *et al.*, 2000). The Sm-Nd internal isochron age dated in this study is 1.85 Ga for the Howard Hills. The

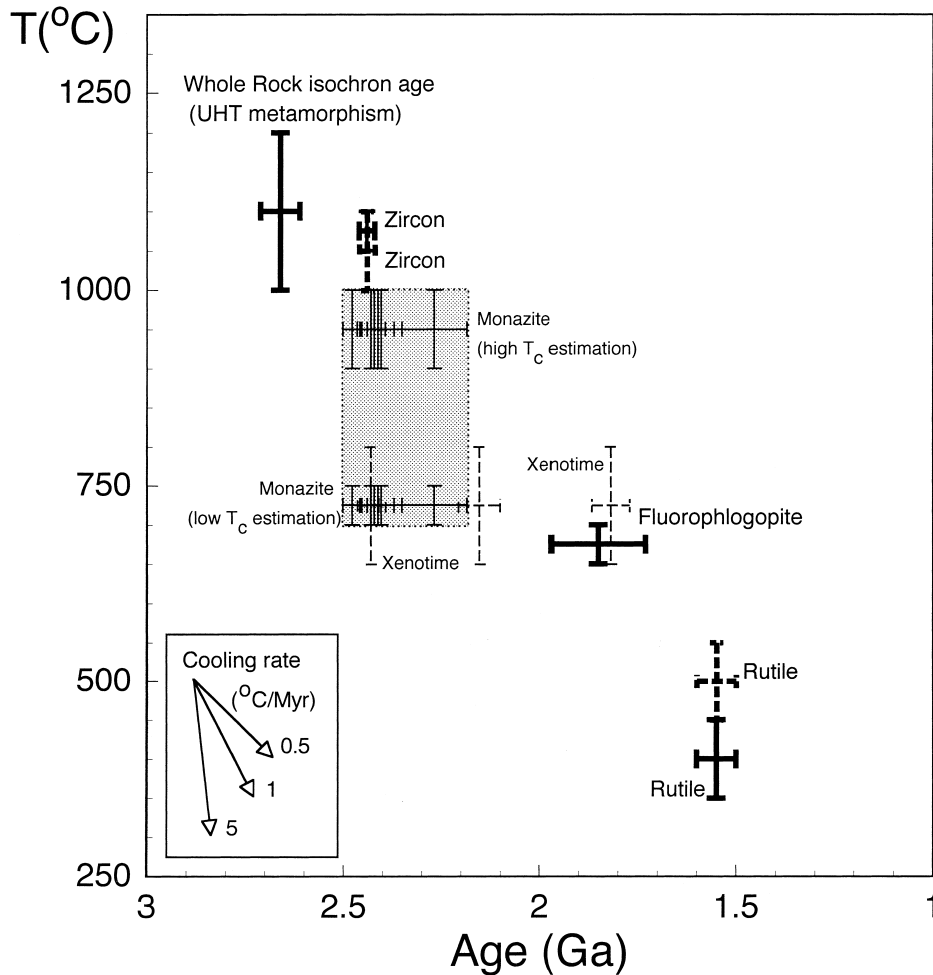


Fig. 8. Apparent temperature-time ($T-t$) relation for metamorphic rocks from the Howard Hills. Age data are taken from the result of this study and Miyamoto et al. (2004). The $Sm-Nd$ whole-rock isochron age is regarded as the time of peak metamorphism for the dated sample (Miyamoto et al., 2004). The metamorphic temperature (1000° – $1200^{\circ}C$) is quoted from Yoshimura et al. (2000). Estimations of closure temperatures for each mineral are as follows: 1050° – $1100^{\circ}C$ for closure temperature of zircon age (Cliff, 1985); 650° – $700^{\circ}C$ for fluorophlogopite under dry condition (Kühn et al., 2000); 350° – $450^{\circ}C$ for rutile age (Mezger et al., 1989), and based on diffusion parameters (thick and broken lines): 1000° – $1100^{\circ}C$ for zircon (Cherniak and Watson, 2000); 450° – $550^{\circ}C$ for rutile (Cherniak, 2000). The $Th-U-Pb$ CHIME ages for monazite and xenotime are obtained from Grew et al. (2001), Asami et al. (2002), and Suzuki et al. (2006). Their temperature estimations are based on the diffusion parameter: 700° – $750^{\circ}C$ for monazite (Parrish, 1990; low T_c estimation in the figure) and 900° – $1000^{\circ}C$ for monazite (Cherniak et al., 2004, high T_c estimation in the figure); 650° – $800^{\circ}C$ for xenotime (Heaman and Parrish, 1991; Kamber et al., 1998). The shaded square is an area of $T-t$ points for monazite.

deviation between these Sm-Nd internal isochron ages is probably caused by the strong influence of a tectonothermal event at *ca.* 1.9 Ga, which is unidentified on the Howard Hills; otherwise, it might be caused by local difference of the cooling process, that is to say, faster cooling of metamorphic rocks in Mount Riiser Larsen than other outcrops.

Apparent temperature-time (*T-t*) relations for zircon, fluorophlogopite and rutile from metamorphic rocks on the Howard Hills are shown in Fig. 8. *T-t* relations of monazite and xenotime from other localities in the Napier Complex (Asami *et al.*, 1998, 2002; Grew *et al.*, 2001; Suzuki *et al.*, 2006) are also shown in the same figure. Temperature constraints were given by adoption of UHT granulite-facies condition (Yoshimura *et al.*, 2000) for whole rock age, and closure temperatures evaluated from field evidences or diffusion parameters (Cliff, 1985; Mezger *et al.*, 1989; Parrish, 1990; Heaman and Parrish, 1991; Kamber *et al.*, 1998; Kühn *et al.*, 2000; Cherniak, 2000; Cherniak and Watson, 2000; Carson *et al.*, 2002a; Cherniak *et al.*, 2004) for zircon, monazite, xenotime, fluorophlogopite, and rutile ages. The apparent *T-t* relations for the Howard Hills are linear within the uncertainties of age and estimated temperature.

We suggest two plausible explanations for the *T-t* relation: one suggests slow cooling by passing each *T-t* point in turn after UHT granulite facies metamorphism. Alternatively, dating results might detect individual heated episodes on the jagged cooling history of the Howard Hills. Osanai *et al.* (2001) and Owada *et al.* (2001, 2003) reported two individual geothermal events at *ca.* 1.9 Ga and 1.6 Ga, and concluded that these two events reflect the regional scale of tectonothermal events among the East Gondwana fragments. Geochronological results in this study might also show the Proterozoic episodic thermal events in the Napier Complex.

6. Conclusions

Analytical U-Pb data for zircon fractions from orthopyroxene felsic gneiss in the Howard Hills, Enderby Land, define a discordia with lower intercept age of 2.44 ± 0.02 Ga. This age shows the time of termination of the UHT granulite facies metamorphism that has occurred several times since 2.65 Ga at the Napier Complex. Analytical U-Pb data for rutile fractions in phlogopite-bearing pyroxene granulite from the same outcrop show nearly a concordant 1.5 Ga age. This age represents the last stage of thermal activity, with the exception of the later intrusion of tholeiitic dykes or pegmatites.

The rubidium-strontium model age is about 1.85 Ga for phlogopite in pyroxene granulite. Samarium and neodymium compositions of whole-rock, feldspar and orthopyroxene fractions of the same pyroxene granulite were regressed on an isochron with an age of 1.85 ± 0.15 Ga. Although field evidence of igneous activity at 1.9 Ga has not been found in the Howard Hills, the correspondence of Rb-Sr and Sm-Nd ages probably records a regional tectonothermal event which influenced the Napier Complex after the peak metamorphism at Archean.

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