

High-P metamorphism in the Mesoproterozoic: petrochronological insights from the Grenville Province

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1	High-P metamorphism in the Mesoproterozoic: petrochronological insights
2	from the Grenville Province
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5	Caroline Lotout ^{1,2} , Aphrodite Indares ² , Jeffrey Vervoort ³ , Etienne Deloule ¹
6	
7	¹ Université de Lorraine, CNRS, CRPG, F-54000 Nancy, France
8	² Department of Earth Sciences, Memorial University of Newfoundland, St. John's, Canada
9	³ School of the Environment, Washington State University, United States
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11	Corresponding author: Caroline Lotout, caroline.lotout@uca.fr
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23 Abstract:

In the Grenville Province, high-P rocks that are discontinuously exposed along the margin of 24 25 the allochthonous belt preserve the record of deep crustal processes and are an essential puzzle piece to understanding Mesoproterozoic geodynamics. The Manicouagan Imbricate 26 zone (MIZ) is one of the three high-P domains from the Grenville Province. It is located in the 27 Central Grenville between the Parautochthonous Belt to the North and the orogenic hinterland 28 29 to the South, that were metamorphosed during the 1005-980 Ma Rigolet and 1080-1020 Ma Ottawan orogenic phases, respectively. In the western MIZ, the Lelukuau Terrane (LT) 30 31 mostly consists of Labradorian-age (~1650 Ma) mafic suites with a fringe of aluminous rocks at its southern edge. Metamafic samples from the Western and Eastern parts of the MIZ 32 33 display a peak assemblage of garnet, clinopyroxene, plagioclase, rare pargasite or edenite, and quartz \pm kyanite. Pseudosection modelling suggests high-P granulite peak conditions at ca. 14 34 35 to 16 kbar and 800–900°C, with the scarcity of hydrous phases and quartz explaining the lack of evidence for partial melting. Zircon cores from the Western LT sample show a maximum 36 37 magmatic age of ca 1.6 Ga. Lu–Hf and Sm–Nd dating on garnet from this sample yield ages 38 of 1020 ± 7 Ma and 1005 ± 13 Ma, respectively, overlapping within error and inferred to represent peak metamorphic conditions followed by fast cooling. In the Eastern LT sample, 39 garnet Lu-Hf dating yields two ages that are consistent with a petrographically preserved two 40 stage growth, at 1033 ± 6 Ma and 1013 ± 6 Ma, while the Sm–Nd age indicates cooling at 41 1003 ± 8 Ma. The recorded high-P granulite facies conditions highlight a late Ottawan to 42 43 Rigolet-age localized crustal thickening at the margin of the hinterland during the propagation of the orogen to the NW, with a possible younging of the high-P granulite-facies 44 45 metamorphism from the Eastern to Western LT. These new results indicate that the high-P belt in the Central Grenville does not represent the exhumed base of an Ottawan age orogenic 46 47 plateau, as previously proposed, and that no tectonic hiatus exists between the two orogenic

- 48 phases, as generally thought. Finally, this publication highlights the diversity and
- 49 diachronicity of the high-P domains in the Grenville Province.
- 50
- 51 **Keywords:** Garnet geochronology, Zircon geochronology, high-P granulite, Grenville
- 52 Orogeny, Mesoproterozoic

53 **1. Introduction**

High-pressure (high-P) rocks, namely eclogite, high-P granulite, and kyanite-bearing 54 migmatite provide important evidence for active plate tectonics, recording the burial of the 55 crust and crustal thickening in convergence zones. In those settings, timescales and conditions 56 of metamorphism are crucial in deciphering the evolution of the lithosphere, as the tectonic 57 scenarios invoked largely rely on the rates of burial and exhumation of metamorphic terranes 58 (e.g., Warren, 2013), their corresponding size, and their temporal and spatial distribution 59 within an orogen (e.g., Kylander-Clark et al., 2012). In this regard, the advent of 60 petrochronology (see RIMG 2017 special volume, Kohn et al., 2017) has provided valuable 61 insights on the petrological evolution of chronometers by integrating their textural, isotopic, 62 and elemental context. Zircon and monazite are often targeted to access time information 63 during metamorphism (e.g., Baldwin and Brown, 2008; Gasser et al., 2012; Štípská et al., 64 65 2016; Wang et al., 2017), as their potential equilibrium with garnet can be assessed by their trace element record (e.g., Rubatto, 2002, 2017; Whitehouse and Platt, 2003). In recent years, 66 67 an increasing number of publications have highlighted the need to combine multiple 68 petrochronological tools in high-grade terranes (e.g., Manzotti et al., 2018; Lotout et al., 2018, 2020; Wang et al., 2019; Du et al., 2021; Li et al., 2022). This is supported by the fact that 69 prograde and/or retrograde metamorphic overprints can result in a complex record of 70 71 accessory minerals reflecting lack of equilibrium at the rock scale, and incomplete U-Pb resetting or trace element inheritance (e.g., Harley et a., 2007; Yakymchuk and Brown, 2014; 72 Whitehouse et al., 2014; Kohn et al., 2015; Taylor et al., 2016; Lotout, et al., 2018; Pitra et al., 73 2022), thus making it difficult to link ages to specific P-T stages. Petrochronological 74 investigation of both rock-forming minerals (like garnet) and accessory minerals (like zircon) 75 76 provides a strategy to obtain significant timing information. Combined with P-T

thermodynamic modelling, this has proven to be one of the most reliable approaches todecipher the style of tectonic processes.

79 Exhumed cores of ancient orogens give direct access to formerly deep and hot levels of the crust offering opportunities to both assess the condition and duration of high-T and high-P 80 processes and investigate the tectonic evolution of orogenic roots. The Grenville Province, 81 82 exposed in eastern North America from Canada to the Adirondack Mountains of the United States (Fig. 1a), is recognized as the first Large Hot Orogen (LHO) on Earth (Rivers, 2009), 83 84 and is attributed to the collision between Laurentia and Amazonia in the late Mesoproterozoic. Its orogenic core (Allochthonous belt, or hinterland) is an assembly of 85 86 crustal segments that were metamorphosed, mostly at granulite-facies conditions, at different 87 depths during the 1090–1020 Ma Ottawan phase of the Grenvillian orogeny (Fig. 1b) and is inferred to represent remnants of a collapsed orogenic plateau (Rivers, 2008; 2012). In 88 contrast, the Parauthochtonous belt of the Grenville Province (Fig. 1b) was deformed and 89 90 metamorphosed later, during the final propagation of the orogen to the NNW (Rigolet Phase; 1005–980 Ma), and after a presumed tectonometamorphic hiatus of about 15 Myr (e.g., 91 92 Rivers, 2009, 2012).

Although geodynamic modelling has addressed key tectonic features of the Grenville orogen
(e.g., Jamieson et al., 2007, 2010), metamorphic data, crucial for tectonic interpretations, are
patchy and incomplete (see Indares and Dunning, 2018, and Indares, 2020 for a review), and
petrochronological studies remain scarce. For instance, garnet ages are only reported from
mid-P and high-T rocks from the Virginia Blue Ridge area (Johnson et al., 2018, 2020), a
tectonic window within the Appalachian Orogen.

99 In addition, recent debate on Mesoproterozoic global tectonic regimes, and changes of

100 orogenic style with time, has been spirited (e.g., Cawood and Hawkesworth, 2014; Tang et al.,

101 2021; Roberts et al., 2022), and some aspects of the orogen are questioned by recent studies.

For instance, Spencer et al. (2021), made the case that the orogenic crust was thin in the 102 103 Mesoproterozoic and Brudner et al., (2022) disputed the existence of a long-lived highly 104 elevated orogenic plateau in the Grenville. Considering this, high-P rocks exposed in the Grenville Province are essential pieces to the Mesoproterozoic geodynamic puzzle as they 105 106 record orogenic processes operating at depth. These rocks are documented on either side of the Allochthon Boundary Thrust (Fig. 1b). In the hinterland, they are grouped within the 107 108 "high-P belt" (Rivers et al., 2002) and occur (i) as mafic pods of eclogite retrogressed to 109 granulite, with early Ottawan metamorphic ages, dispersed in orthogneiss (e.g., Davidson1990; Marsh & Culshaw, 2014; western Grenville), and (ii) in coherent lithotectonic 110 111 packages dominated by mafic rocks with well-preserved high-P granulite to eclogite facies 112 assemblages (Indares 1997, Indares et al., 1998, 2000; central Grenville). In addition, eclogitized metagabbros are reported in the Parautochthonous Molson Lake terrane (Indares 113 114 1993; Indares and Rivers 1995). 115 This contribution focuses on high-P mafic rocks from the Manicouagan Imbricate zone (MIZ) of the Central Grenville. The MIZ is located structurally below the mid-P belt of the 116 117 hinterland, (Fig. 1b, c) and based on this configuration is well suited for comparing processes

operating in deep to intermediate levels of the Ottawan orogenic crust. Based on poorly

119 constrained Ottawan ages reported in the early literature (Indares et al., 2000), the MIZ was

120 inferred to represent the extruded base of the thickened Ottawan crust (Rivers 2008), and

therefore it is a key area to bring new fundamental constraints on the timing and duration of

122 high-P metamorphism and subsequent tectonic evolution.

123 The samples were collected in the early 90s in the context of the Lithoprobe project (see

124 Clowes et al., 1999; Rivers et al., 2012). In the context of the present study, they were

125 investigated using textural observation, P–T thermodynamic modelling, garnet Lu–Hf and

126 Sm–Nd dating, zircon U–Pb dating, and trace element analysis in garnet and zircon. This

study shows that the high-P rocks of the Central Grenville experienced peak high-P
metamorphism straddling the interval between the Ottawan and Rigolet phases and reveals a
continuum where a tectonometamorphic hiatus was previously inferred. It also highlights that
this high-P granulite metamorphism reflects localized crustal thickening at the margin of the
hinterland, as the orogen was progressing towards the Grenville front. By bringing new P–T–t
information on this high-P metamorphic event of the Grenville Orogen, this study also
highlights the tectonically active character of the lithosphere during Mesoproterozoic times.

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135 2. Geological background

136 The MIZ is a 2000 km² area of stacked high-P crustal slices (e.g., Indares, 1997), located in the central Grenville between the structurally lower Parautochthonous Gagnon terrane to the 137 north, and structurally higher lithotectonic units of the hinterland, to the south (Fig. 1b, c). 138 The Gagnon terrane consists of Archean basement and Paleoproterozoic cover 139 metamorphosed, in the vicinity of the MIZ, at amphibolite to high-P granulite facies 140 conditions at ~990 to 980 Ma (Jordan et al. 2006; Jannin et al., 2018a). South of the MIZ, the 141 mid-P belt of the hinterland consists of ~1.5–1.4 and 1.2 Ga plutonic and supracrustal rocks 142 143 metamorphosed under mid-P granulite-facies conditions between 1.08 and 1.04 Ga (Dunning 144 and Indares 2010; Lasalle et al. 2014). However, its contact with the MIZ is obscured by the Triassic Manicouagan impact crater. In contrast, to the SE, the MIZ is in direct contact with 145 the Hart Jaune terrane, which represents the highest crustal levels of the hinterland in this area 146 147 that largely escaped Grenvillian-age metamorphism and deformation (part of the Orogenic Lid; Rivers 2008). 148

149 The MIZ is subdivided into two terranes, Lelukuau (LT, lower unit) and Tshenukutish (TT,

upper unit; Fig. 1c). Both terranes are dominated by plutonic units, with a discontinuous

fringe of supracrustal rocks, including anatectic aluminous gneisses, exposed in the southern
MIZ along the shores of the Manicouagan reservoir (Indares and Dunning 2001, Dunning and
Indares, 2010).

The LT is the largest component of MIZ and is mostly composed of Labradorian-age 154 anorthosite-mangerite-charnockite-granite (AMCG) bodies emplaced at 1650-1630 Ma 155 156 (Indares et al., 1998). In the eastern MIZ, the structurally higher TT is an assembly of highly 157 deformed tectonic slices, composed of Labradorian-age AMCG, Pinwarian-age granites 158 emplaced at ca. 1450 Ma, and ca. 1190 Ma mafic sills (Cox and Indares, 1999; Indares et al., 2000). Mafic rocks in the two terranes show different metamorphic textures. Coronitic or 159 160 granoblastic textures, based on the protolith type (sill vs other types of magmatic bodies), are 161 observed in the TT (Indares, 2003; Cox and Indares, 1999), while mafic rocks from the LT are pervasively recrystallized in a granoblastic assemblage of garnet-clinopyroxene-162 plagioclase \pm amphibole \pm quartz, up to the point that the magmatic archive is only deduced 163 164 from the presence of microdomains mimicking igneous textures (Indares, 2003). P-T estimates are mostly restricted to traditional thermobarometry results, with P-T conditions in 165 the range of 14–18 kbar and 800–900°C in the LT (Indares, 1997, 2003; Indares and Dunning, 166 2004), and 780–930°C and 16–19 kbar in the TT (Cox and Indares, 1999). The P-T 167 conditions of the LT are consistent with results of subsequent pseudosection modelling (Yang 168 169 and Indares, 2005). This was, however, limited by the lack of thermodynamic melt data suited for mafic compositions, only recently provided by Green et al. (2016), to appropriately 170 bracket the temperature range. Metamorphic ages on mafic rocks, that represent the main rock 171 172 types of the MIZ, are scarce and mostly restricted to lower intercepts obtained by single zircon grain dissolution using ID-TIMS, at 1052 ± 19 Ma (Indares et al., 1998), and 173 174 1030 ± 12 Ma (Cox et al., 1998; Fig. 2). The best approximation for the age of high-P metamorphism is provided by a set of syn-orogenic dykes ("Themines dykes"; Indares and 175

Dunning 2004) cropping out in southern MIZ, which display high-P granoblastic assemblages
(garnet-clinopyroxene-plagioclase-quartz) and are interpreted to have been emplaced at
high-P conditions. One of these dykes, in the TT, was dated at 1039 ± 2 Ma (Indares et al.,
1998).

The high-P anatectic aluminous rocks exposed in the southern MIZ have kyanite-garnet-K-180 feldspar-plagioclase-quartz \pm biotite assemblages, with textures consistent with peak and 181 melt crystallization P–T conditions in the kyanite stability field. Pseudosection modelling has 182 183 constrained the peak P-T conditions at 15-16 kbar and 875°C (Indares et al., 2008; Lasalle and Indares, 2014; Kendrick and Indares, 2018). In the southern LT, monazite from one 184 sample was analyzed by both ID-TIMS on single grains (Dunning and Indares, 2010) and 185 186 later by in situ Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICPMS) (Lasalle et al., 2014). Both methods yielded complex age data and the age of Grenvillian 187 metamorphism remains difficult to interpret owing to strong inheritance. LA-ICPMS ages 188 189 spread from 1362 ± 26 Ma to 1025 ± 22 Ma, with a cluster at 1070-1050 Ma, and a poorly supported weighted average age of 1061 ± 9.6 Ma while ID-TIMS data yielded an average 190 age for Grenvillian metamorphism at 1044 ± 1.5 Ma. These data are interpreted to reflect 191 protracted growth of monazite during anatexis, and, in the case of ID-TIMS, varied mixing of 192 193 distinct monazite age domains (Lasalle et al., 2014). In contrast, monazite ages obtained in 5 194 samples from the southern boundary of the TT (single-grain dissolution, ID-TIMS, Indares 195 and Dunning, 2001) are better constrained as they are tightly clustered at the sample scale, 196 ranging from 1040 ± 2 Ma to 1017 ± 2 Ma from West to East (Fig. 2). These late Ottawan 197 ages likely indicate diachronism of the metamorphism, however they only represent the southern fringe of the MIZ, while the timing of the high-P metamorphism in the interior of the 198 199 MIZ, that is dominated by mafic rocks, remains poorly constrained.

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201 **3. Analytical Methods**

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The analytical techniques were carried out at the Core Research Equipment and Instrument Training (CREAIT) Network at Memorial University of Newfoundland (MUN, St John's, Canada), Centre de Recherches Petrographiques et Géochimiques (CRPG, Nancy, France), and Radiogenic and Isotopic Geochronology Lab (RIGL, Washington State University,

206 Pullman, USA). All operating conditions, standards, chemical procedures, and solid solution

207 models used and carried out are described in detail in the Appendix.

208 Bulk rock powders were produced at MUN and major and trace elements analyses were

209 performed by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) and

210 Inductively Coupled Plasma Mass Spectrometry (ICPMS), respectively, at the SARM

211 laboratory of CRPG, following the procedure described in Carignan et al. (2001;

Supplementary Table 1). Thin sections were imaged by Scanning Electron Microscope (SEM)

equipped with Mineral Liberation Analysis (MLA) software to provide false color mineral

214 maps illustrating textures, X-Ray maps of garnet were obtained using an Electron Probe

215 Microanalyzer (EPMA), and zircon were imaged using a SEM equipped with a Deben

216 Centaurus CathodoLuminescence (CL) detector at CREAIT (MUN). Quantitative mineral

analyses for major elements were carried out using a EPMA (Supplementary Table 2) and

trace elements in garnet were measured by LA-ICPMS (Supplementary Table 3) at CREAIT

219 (MUN). P–T pseudosections have been calculated in the Na₂O–CaO–FeO–MgO–Al₂O₃–

220 SiO₂-H₂O-Fe₂O₃ (NCFMASHO) system using the software Theriak/Domino (de Capitani

and Petrakakis, 2010), and the internally consistent thermodynamic data set 6.2 (Holland and

Powell, 2011, update ds62, 6th February 2012). Garnet and zircon mineral separation was

223 performed at MUN following standard procedure to concentrate heavy minerals and picking.

224 Lu-Hf and Sm-Nd garnet dating was performed at RIGL, following procedures described in

225 Cheng et al., (2008) and Johnson et al., (2018). Isotope data and corresponding calculated

dates were produced using isoplot (Ludwig, 2003) and ages are reported at 95% confidence 226 227 level (Table 2, Fig. 9.). U-Pb and REE analyzes in zircon were performed at the National Ion 228 Probe facility hosted by the CRPG, following Deloule et al. (2002) and Jeon & Whitehouse (2014). Isotope data are reported in Supplementary Table 3 and corresponding calculated 229 dates, produced using isoplot (Ludwig, 2003), are shown in Fig.10 and reported at 95% 230 confidence level. Abbreviations used in this manuscript are: apfu = atom per formula unit; g =231 232 garnet; cpx = clinopyroxene; di = diopside; amp = amphibole; prg = pargasite; edn = edenite; mu = muscovite; pl = plagioclase; scp = scapolite; q = quartz; ru = rutile; bi = biotite; ky =233 kyanite; zrn = zircon; ap = apatite; liq = liquid. Other symbols used (mole/atomic proportion) 234 235 are: $X_{alm} = Fe/(Ca + Fe + Mg + Mn)$; $X_{py} = Mg / (Ca + Fe + Mg + Mn)$; $X_{grs} = Ca / (Ca + Fe + Mg$ $Mg + Mn); X_{sps} = Mn / (Ca + Fe + Mg + Mn) (garnet); X_{Jd} = Na / (Na + Ca); CaT = Al^{IV} - X_{Jd}$ 236 (cpx); $X_{Mg} = Mg / (Fe + Mg)$ (garnet, clinopyroxene, amphibole); $X_{An} = Ca / (Ca + Na + K)$; 237 $X_{Ab} = Na / (Ca + Na + K)$ (plagioclase); $X_{CO3} = CO3 / (CO3 + SO4 + Cl)$; $X_{eqAn} = 100 (Al - Cl)$ 238 3)/3; $X_{Me} = Ca / (Ca + Na)$ (scapolite); Mg# = 100*Mg / (Mg+Fe) (bulk rock). 239

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241 **4. Results**

242 **4.1. Sample description**

Three mafic rocks were selected for this study. They belong to distinct levels of the LT following Indares (1997) and all have a gabbroic composition with high Al₂O₃ content (17 to 20 wt%) and Mg# ranging from 41.7 to 61.7% (Supplementary Table 1). All samples are pervasively recrystallized showing a medium to fine grained granoblastic texture and containing the mineral assemblage garnet–clinopyroxene–plagioclase–amphibole–scapolite with varied mineral proportions (Fig.3 and 4, Table 1).

249 4.1.1. Western Lelukuau: sample 135A

Sample 135A is a homogeneous granoblastic rock which, in addition to the main mineral 250 assemblage, also contains rare biotite, quartz, zircon, and apatite (Figs. 3a and 4a, Table 1). 251 252 Garnet is subhedral, ranging in size from 2 to 4 mm and containing rare inclusions of kyanite, zircon, or apatite. It is chemically characterized by $X_{Mg} = 0.54-0.57$ and $X_{Grs} = 0.21-0.29$, both 253 parameters decreasing towards the rims (Figs 4a, 5a, 6a, b, c). Clinopyroxene is light green 254 255 subhedral and up to 1 mm in size (Fig.5c). It has a diopside composition, with up to 10 % Ca-Tschermak component, regardless the textural position, and 7–13% jadeite, uniform at the 256 257 grain scale. Clinopyroxene in contact with garnet systematically shows a higher Si content (1.90 –1.92 apfu), and all grains display Al enrichment in the core and Mg enrichment in the 258 259 rims. Plagioclase is subhedral with a grain size ranging from 0.2 to 0.5 mm. Its anorthite 260 component ($X_{An} = 0.35 - 0.50$) is varied at the thin-section scale, but single grains are homogeneous (e.g., $X_{An}pl_1 = 0.43-0.44$; $X_{An}pl_6 = 0.35-0.36$; $X_{An}pl_3 = 0.45-0.47$; Fig. 5d). 261 Amphibole, 0.2 to 0.4 mm in size, is exclusively associated with clinopyroxene. It is calcic 262 263 $(Ca^{B} = 1.84 - 1.93, Na + K = 0.77 - 0.92, Ti = 0.09 - 0.22 apfu)$, Al rich (Al M1=0.76 - 1.27), and magnesian ($X_{Mg} = 0.73 - 0.78$; Fig. 5b). Scapolite is disseminated across the thin section and 264 has a sylvialitic composition, with $X_{SO4} = 0.48 - 0.66$ (Fig.5e and Supplementary Table 2). 265 When the chemical formulae of the analyzed grains are recalculated following Ellis (1978) to 266 be compared to plagioclase, they show a significantly distinct composition of XeqAn of 0.59-267 268 0.70 (Fig.5d), homogeneous at the grain scale. Rare biotite (1.5%) is observed without specific textural position. Quartz, zircon, and apatite are dispersed at the thin section scale. 269

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4.1.2. Eastern Lelukuau: samples KW4A and KW4C

Samples KW4C and KW4A are metagabbros collected a few meters away from each other on
the same outcrop and have broadly similar chemical compositions (Supplementary Table 1).
Texturally, both are granoblastic but KW4A is homogeneous and fine-grained (<0.5 mm)

while KW4C is medium-grained (0.5–3 mm) and has polycrystalline plagioclase domains and 274 275 garnet porphyroblasts, as illustrated by the SEM-MLA maps (Fig.3b and c). Sample KW4C – Subhedral plagioclase (0.15–0.5 mm) forms polycrystalline domains (up to 276 1 cm) that are zoned with $X_{An} = 0.52 - 0.80$, decreasing from the cores toward the rims 277 (Fig.5d, Supplementary Table 2), but have a homogeneous grain size. The inner zones of the 278 279 domains contain rare kyanite inclusions and are locally altered to sericite. Garnet forms subhedral grains of 0.5–2 mm, clustered into up to 4 mm composite grains (Fig.3b; Fig.6e, f, 280 281 h, i). Garnet cores have abundant inclusions of kyanite needles (up to 250 µm) and rare quartz 282 inclusions while the rims are inclusion-free (Fig.4b; Fig.6d to i). Garnet cores are Ca-rich with 283 $X_{Grs} = 0.26 - 0.29$, $X_{Prp} = 0.46 - 0.48$, and $X_{Alm} = 0.22 - 0.24$ while the borders (including those 284 of the subgrains) are enriched in Fe–Mg and show Ca depletion ($X_{Grs} = 0.19-0.21$, $X_{Prp} = 0.52 - 0.57$, $X_{Alm} = 0.25 - 0.29$) with this trend being more pronounced at the outer rims 285 of the composite grains (Figs. 5a, 6e, f, h, i). Clinopyroxene forms anhedral grains of 0.5 to 1 286 287 mm, that are locally associated with quartz and plagioclase and/or amphibole in large aggregates of 1 to 2 mm. It has a diopside composition, with 5 to 8% jadeite and 2 to 10% Ca-288 Tschermak (Fig.4b, 5c). They all display Al enrichment in cores and Mg enrichment in rims. 289 Amphibole forms subhedral grains with embayed borders, closely associated to clinopyroxene 290 and, rarely, in the middle of plagioclase domains. Single grains are ca. 0.15–0. 40 mm in size, 291 292 but locally form 2 mm elongated aggregates. Amphibole has edenitic composition with $Ca^{B} = 1.88 - 1.93$, Na+K = 0.53-0.64, Ti = 0.09-0.2 apfu, Al M1 = 0.97-1.32 and $X_{Mg} = 0.85 - 1.02$ 293 0.87 (Fig.5b). The Si content varies from 6.50 to 6.89 apfu in all the analyzed grains but 294 295 doesn't reflect any general trend or textural position specificity. Quartz is only found in three textural positions, either as rare inclusions in garnet, entrapped in the inner zones of garnet 296 297 composite grains, or associated to clinopyroxene (Fig. 6d, g). Scapolite grains are dispersed in the thin section and commonly associated with garnet or plagioclase. They have a meionite 298

composition with $X_{SO4} = 0.28-0.37$ (Fig. 5e and Supplementary Table 2). When recalculated 299 300 following Ellis (1978) to be compared to plagioclase (Fig., 5d), scapolite shows an intermediate composition of X_{eqAn} of 0.65–0.69 and are homogeneous at the scale of the grain. 301 Kyanite is found in two textural positions: i) as needle inclusions in garnet and ii) locally in 302 303 the center of high-Ca plagioclase domains. 304 <u>Sample KW4A</u> – Plagioclase forms euhedral grains, 0.2–0.4 mm in size, that are homogeneous throughout the thin section, with $X_{An} = 0.35-0.24$ (Fig. 3c, 5d, 6j to 1) and 305 306 Supplementary Table 2). Locally, K-feldspar exsolutions are observed in the plagioclase domains (<1%). Garnet forms grains mostly 0.5 mm in size that are commonly clustered into 307 308 3 mm-wide composite grains, homogeneously distributed across the thin section. Grains show 309 complex zoning (Fig.5a, 6j to 1), some subgrains being Ca-rich ($X_{Grs} = 0.23 - 0.25$, $X_{Prp} = 0.35 - 0.25$ 0.36, $X_{Alm} = 0.39-0.41$) while others are Fe-Mg-rich ($X_{Grs} = 0.19-0.22$, $X_{Prp} = 0.38-0.41$, 310 $X_{Alm} = 0.37 - 0.41$). Clinopyroxene forms subhedral grains, 0.2-0.5 mm in size. It has diopside 311 312 composition, 7 to 11% jadeite and up to 10% Ca-Tschermak component, while total Al is low (0.18–0.41 apfu). Scapolite is euhedral, 0.2 to 0.5 mm in size, and is homogeneously 313 distributed at the thin section scale. It has a sylvialitic composition ($X_{SO4} = 0.50-0.70$, Fig. 5e) 314 and commonly clusters in larger domains up to 3 mm. Rare amphibole or quartz are dispersed 315 at the thin section scale. 316 4.1.3. Mineral assemblages and background to the phase equilibria modelling 317

The three samples are composed of varied amounts of garnet, clinopyroxene, and plagioclase that are characteristic of high-P granulite facies conditions. Samples 135A and KW4C, that are medium grained, also show a X_{Alm} increase in the garnet rim that is consistent with diffusional re-equilibration of the rims during cooling. Because of high peak temperatures attained and/or maintained in granulite facies rocks, the prograde history can be deciphered mostly by mineral inclusions. To this end, KW4C provides a snapshot in the crystallization of

kyanite and quartz during an early part of the P–T path associated with plagioclase
destabilization and garnet crystallization following the reaction anorthite = grossular +
kyanite + quartz, at high-P conditions. Moreover, in this sample, the large, recrystallized
plagioclase domains are interpreted to pseudomorph former igneous plagioclase, preserving
original Ca-rich core and Na-rich rims. Rare kyanite included in garnet of sample 135A,
suggests a similar reaction history.

According to the literature, scapolite, and specifically S-scapolite, can either be (i) an igneous 330 331 phase (e.g., Boivin & Camus, 1981), (ii) result from plagioclase alteration (e.g. Touret & Nijland, 2013), or (iii) be a metamorphic mineral (e.g., by replacement of igneous plagioclase: 332 Austhreim, 2013). Given the textural evidence of pervasive granoblastic recrystallisation, the 333 334 magmatic phase option can be ruled out. If the scapolite were an alteration phase of 335 metamorphic plagioclase, then the X_{eqAn} would be spread along the same range given that scapolite grains are not observed in one specific textural position. In all samples, scapolite 336 X_{eqAn} is different from X_{An} of plagioclase, and they are consequently interpreted as part of the 337 main metamorphic assemblage. This would require either a S-bearing mineral breakdown 338 during prograde metamorphism, or a S-bearing fluid phase at high-pressure. 339

340

4.2. P-T estimates

Samples 135A and KW4A were used to calculate P–T pseudosections. MnO, K₂O and TiO₂ were removed from the bulk-rock composition since their amounts are low in the bulk rock (less than 0.65 wt.%; see Supplementary Table 1), and they consequently will not impact the modelled stability of the main mineral assemblage present. For both calculations, Fe³⁺ /Fe^{tot} was set at 0.1 while H₂O was calculated based on the proportion of amphibole in the rock. Scapolite is part of the mineral assemblage and represents ca. 5 %. As no solid-solution model was available, two approaches were considered: (i) scapolite was treated as plagioclase; and

(ii) scapolite was removed from the bulk-rock composition by subtracting the equivalent 349 amount of CaO, Na₂O, Al₂O₃ and SiO₂. Considering all scapolite as plagioclase results in 350 excess Al and Si, that impacts the Al-Si-bearing-mineral stability, either over-estimating 351 quartz or kyanite, but also over or under-estimating garnet, plagioclase, and clinopyroxene 352 modal proportions. Subtracting a mineral from a bulk rock composition is a common practice 353 to account for the lack of a solution model for some phases and is often used to correct for the 354 355 Ca content of apatite (see LaSalle and Indares, 2014, Lotout et al., 2018, 2020). Subtracting a minor phase from the bulk is unlikely to influence rock-forming reactions, but it is an 356 oversimplification for major phase that *de facto* modifies the thermodynamic equilibrium state 357 358 of each assemblage. Both solutions have been tested, and we present results calculated from 359 an unmodified bulk, i.e., not removing scapolite from the bulk-rock composition, as it provides the best fit. 360

Western-Lelukuau - For sample 135A, the stability field of the peak assemblage garnet-361 362 diopside-plagioclase-amphibole is limited by the appearance of liquid at higher T and kyanite at higher P–T conditions (Fig. 7a). Quartz is stable at P–T conditions between 700°C/12 kbar 363 and 900°C/14 kbar, with modal proportion < 2 %, in good agreement with the observed 364 mineral proportion (Table 1). In the P-T pseudosection, amphibole has a pargasitic 365 composition, with modal proportions (12 to 8 %) decreasing gradually towards the melt-in 366 367 line. The plagioclase-out line at ca. 700°C/16 kbar and 850°C/19 kbar marks the limit 368 between the high-P granulite and the eclogite facies. The calculated modal proportions of garnet and clinopyroxene (Fig. 7a, Table 1), the X_{Mg} of garnet and the grossular isopleth (Fig. 369 370 7b), together with the kyanite and melt boundary lines, restrict the stability field of the assemblage between ca. 770°/14 kbar and 870°C/16 kbar, with garnet rims crystallizing in the 371 372 lower P and T part of this stability field. The calculated modal proportion of plagioclase is

however higher than the measured proportions, which is attributed to the exclusion ofscapolite from the modelling.

Eastern-Lelukuau – Sample KW4C could not be successfully modelled because it is too
dominated by local equilibria texture. In contrast, sample KW4A is well equilibrated at the
thin section scale and the garnet–clinopyroxene–plagioclase–quartz assemblage is stable in a
large P–T field from 700 to 900°C and up to 17 kbar, delimited by the kyanite-in and liquid-in
lines at lower and higher temperatures, respectively (Fig.7c). Calculated modal proportions of
clinopyroxene, garnet and plagioclase don't match the observed mineral proportion in thin
section. This could be the result of re-equilibration during cooling.

382

383 **4.3. Garnet petrochronology**

Samples 135A and KW4C were analyzed using the Lu–Hf and Sm–Nd garnet dating method. 384 A summary of the results is presented in Table 2 and plotted in Figure 9. Garnet fractions 1 to 385 386 4 were spiked for both Lu-Hf and Sm-Nd, while garnet fractions 5 and 6 were only spiked for Lu-Hf analysis. For each sample, two representative whole rocks were also spiked for Lu-387 Hf and Sm-Nd, and followed partial (WRS - Whole Rock Savillex; tabletop) and complete 388 389 (WRB – Whole Rock Bombed; Parr-bomb vessel) digestion procedures. One clinopyroxene fraction was also analyzed for sample 135A. Single Lu-Hf or Sm-Nd dates were calculated 390 for each mineral fraction by pairing them to the WRB (2-pt ages). For each sample, a 391 representative trace element (TE) profile normalized to chondrite is presented in Fig.8. 392

393

4.3.1. Western Lelukuau: sample 135A

The Lu, Sm and Nd contents from sample 135A are mostly below 1 ppm across garnet grains,

and increase from core to rim, from 0.2 to 1 ppm, 0.4 to 1.6 ppm and 0.4 to 1.1 ppm,

respectively (Fig. 8a and Supplementary Table 3). Hf contents range from below the detection

limit to 0.3 ppm through garnet grains, at the limit of detection and with no specific pattern.
Gd/Yb ratios are systematically highest at the rims, at 0.9 to 2.2, while internal zones vary
from 0.2 to 0.8. The atypical Lu-depleted core and Lu-enriched rim could be the result of a
Lu-rich phase (such as zircon) reacting out during garnet growth. Garnet residues from the
dissolution procedure were mostly composed of rare quartz or clinopyroxene inclusions,
except of fraction 4 where a single zircon grain was found (z-Rg4, later subjected to U–Pb *in situ* dating).

404 The six garnet aliquots represent a well averaged population, with Lu and Hf contents (0.58–

405 0.60 ppm and 0.13–0.14 ppm respectively; Table 2) similar to those obtained by *in situ*

406 measurements. The WRS and WRB show a significant difference in both Lu and Hf content

407 highlighting the contribution of zircon (e.g., Scherer et al., 2000). Two-point isochrons, using

408 the WRB fraction and garnet fractions 2, 3, 5 and 6, range from 1022.2 ± 6.1 to

409 1018.4 ± 6.3 Ma, and generate a mean date of 1020.3 ± 3.0 (MSWD = 0.34, Table 2).

410 Fractions 1 and 4 show slightly younger dates of 1009.3 ± 6 Ma and 997.1 ± 6 Ma,

respectively. With respect to fraction 4, all the two point garnet-whole rock isochron dates

412 yielded similar results within error when paired with the clinopyroxene, the WRS, or the

413 WRB fractions, at ca. 1018 ± 6.8 Ma (MSWD = 3.6; Fig. 9a). Garnet fractions 1, 2, 3, 5 and

6, the clinopyroxene fraction, and both whole rocks define an eight-point isochron date of

415 1019.9 ± 6.8 Ma (MSWD = 2.7; Fig 9a). Including fraction 4 in the calculation results in a

date of 1016 ± 14 Ma with higher MSWD of 8.7 reflecting excess scatter. Excluding fraction

417 1 and 4 results in a 7-point isochron age of 1022 ± 3.2 Ma with a slightly lower MSWD of

418 0.94.

Sm and Nd contents from the four garnet aliquots range from 1.54 to 1.60 ppm and from 2.19

420 to 2.74 ppm respectively (Table 2). The WRS and WRB fractions have significantly distinct

421 Sm and Nd concentrations, but almost identical ¹⁴⁷Sm/¹⁴⁴Nd and ¹⁴³Nd/¹⁴⁴Nd. This highlights

that, despite a contribution from a Sm- and Nd-rich refractory phase during the dissolution procedure in bomb, the sample was well equilibrated and wasn't later disturbed by alteration or Sm- or Nd-rich fluid. Altogether, the data align on an isochron that corresponds to a date of 1005 ± 13 Ma (MSWD = 0.25; Fig 9b), overlapping within error with the Lu–Hf results.

426

4.3.2. Eastern Lelukuau: sample KW4C

427 Chondrite-normalized REE patterns for sample KW4C show HREE-enriched cores and MREE-enriched rims across garnet grains (Fig. 8b), typical of preferential incorporation of 428 429 HREEs during garnet early-growth (e.g., Otamendi et al., 2002). Garnet cores also show 430 higher LREE concentrations compared to the mantle and rim zones. The Lu contents decrease from core to rim, from 0.4 to 0.1 ppm, and Hf content was systematically below 0.1ppm to 431 below the detection limit. Sm systematically shows two pulses of concentration increases, 432 433 separated by a lower concentration, from ca. 0.4 to 1 ppm, followed by a 0.2 to 1.2 ppm zone, and 0.1 to 1 ppm (Supplementary Table 3). The positive Eu anomaly in the whole rock (1.86) 434 is not observed consistently in the garnet, with maximum values in the garnet cores (1.1 to 435 436 2.0) and lowest values in garnet rims (0.9 to 1.4). Finally, the rim shows distinct REE pattern differences from the core of garnet, and lack continuous depletion; instead, two population are 437 observed (Fig. 8b). 438

439 All residues from garnet dissolution procedures included variable amount of kyanite and

440 quartz needles, fractions 5 and 6 being 10 times richer than fractions 1, 4 and 5, while fraction

441 2 has an intermediate amount. Lu and Hf contents for the six garnet fractions are similar, at

442 0.21–0.22 ppm and 0.07–0.08 ppm respectively, and corresponding $^{176}Lu/^{177}$ Hf and

443 ¹⁷⁶Hf/¹⁷⁷Hf show little variation (Table 2). WRS and WRB fractions yield equivalent Lu and

444 Hf contents of 0.06 ppm and 0.32–0.34 ppm, showing that no Hf or Lu-rich phase

445 contributed. However, isotopic ratios are not identical, possibly resulting from an incomplete

spike-sample equilibration in the savillex tabletop fraction (WRS). The garnet Lu–Hf 2-point

447	isochron dates for fractions 2, 5 and 6 are 1023.9 ± 7.8 Ma, 1029.1 ± 7.9 Ma and
448	1036.8 \pm 7.9 Ma, resulting in an average date of 1029.9 \pm 4.6 Ma (MSWD = 2.7). Garnet
449	fractions 1, 3 and 4 resulted in 2-point isochron dates of 1014.6 \pm 7.8 Ma, 1011.9 \pm 6.6 Ma
450	and 1013.0 \pm 7.8 Ma, respectively, and corresponding average date of 1013.0 \pm 4.2 Ma
451	(MSWD = 0.87). Including all 6 of these dates result in a mean date of 1021 ± 11 Ma, with an
452	expectedly high MSWD of 7. The 2-point date of garnet fraction 2 is intermediate at
453	1023.9 ± 7.9 Ma, overlapping within error with all garnet fractions, corresponding to an
454	averaged population of two garnet endmembers. Altogether, the 9 fractions define an isochron
455	age of 1018 ± 18 Ma (MSWD = 8.1), reflecting scatter along the isochron due to the
456	variability observed in the garnet isotopic compositions. If we consider only the fractions with
457	similar 2-point dates to calculate two separate isochrons, garnet fractions 1, 3, and 4
458	correspond to an isochron date of 1013.2 ± 5.6 Ma (MSWD = 0.14) and garnet fractions 5 and
459	6 correspond to an isochron date of 1033 ± 6.3 Ma (MSWD = 2.4; Fig. 9c).
460	Sm and Nd contents of the four garnet aliquots are low, 0.70–0.75 ppm and 0.77–0.86 ppm
461	respectively (Table 2). WRS and WRB aliquots yielded almost identical Sm and Nd
462	concentrations of 1.08 and 1.09 ppm and 4.88 and 4.93 ppm, respectively, and corresponding
463	isotopic ratios are also identical within error. This indicates there was no contribution from a
464	Sm or Nd rich phase during the dissolution procedure in bomb, and Sm-Nd homogeneity
465	within the sample. Moreover, this is also independent evidence for reproducibility of the
466	results, emphasizing the absence of contamination in the overall dissolution and column
467	procedure. All fractions correspond to an isochron date of 1003.0 ± 8.3 Ma (MSWD = 0.07;
468	Fig. 9d), with a good alignment of the 8 points; the limited spread in the data is responsible
469	for the elevated error and very low MSWD.

4.4. Zircon petrochronology

472	Zircon was only found in sample 135A from western-Lelukuau. Six zircon grains were
473	analyzed directly in thick section and 14 zircon grains were handpicked from whole rock
474	mineral separate. Colorless zircon crystals are 60 to 200 μ m in size, with variable shapes from
475	subhedral and elongated to anhedral grains. They all have a CL-bright rim of 1 to 60 $\mu m,$ and
476	1 to 2 inner darker CL zones, some with a well-defined core. Some zircon grains show
477	complex textures, including fluid pathways across CL-zones (Fig.10, 135A-2-Z2; Z4), and
478	cracks in the external rim (Fig.10, 135A-1z3, 135A-5-z2b). Few cores present an oscillatory
479	zonation (Fig.10, Z1, Z6, Z8 and Z10). Forty-seven U-Pb analyses and 34 REE analyses were
480	acquired on distinct CL zones from these 20 zircon grains. Amongst the U-Pb data, 3
481	analyses were excluded due to high common lead contents (Pbc $> 3\%$), the remaining 44
482	analyses showing a Pb_c content < 0.7% in most cases, with one outlier at 2.2 %. Uranium
483	contents are variable, spreading from 12 to 2623 ppm and Th/U ratios range from 0.02 to
484	1.83, this ratio showing the maximum spread for cores (ca. 0.4 to 1.7, Fig.11c). Analyzed
485	zircon zones are discordant to sub-concordant (92 to 99 % concordance, Fig 11a), and the
486	207 Pb/ 206 Pb ages spread from ca 1.00 to 1.65 Ga (Fig. 11b, d). In cores and rims, the Lu
487	concentrations vary from 0.5 to 96 ppm independently from the corresponding Eu* anomaly
488	(Fig.11f), which spreads from 0.7 to 1.1. Chondrite-normalized REE patterns are similar with
489	enriched HREE and no to small Eu*, in both cores and rims (Eu = $0.78-1.08$, Fig.11e). Three
490	spectra obtained in ca. 1.2 Ga rims show flat HREE pattern (Fig. 11e and Fig 10).
491	Zircon cores are composed of two ²⁰⁷ Pb/ ²⁰⁶ Pb population, at ca. 1.6 and 1.4 Ga, without any
492	specific distinction in U, Th, or REE contents (Fig. 11c, d, e, f). Average ²⁰⁷ Pb/ ²⁰⁶ Pb dates
493	calculated on ca. 1.6 Ga cores yields a mean date of 1630 ± 12 Ma (N = 13, MSWD = 53) and
494	an upper intercept date of 1653 ± 33 Ma (N = 13, MSWD = 32). Two cores and 4 rims
495	(Fig.10, core: z2 and z12, rims z3, z8, z9, z2b) yield an average 207 Pb/ 206 Pb date of
496	1415 \pm 9 Ma (N=6; MSWD = 3.3) and an upper intercept of 1429 \pm 9 Ma (MSWD = 0.69).

Six rims provided an average 207 Pb/ 206 Pb date of 1184 ± 9.1 Ma (MSWD = 0.72, N = 6) and corresponding upper intercept of 1239 ± 76 Ma (MSWD = 0.13). However, the corresponding trace element patterns are variable, with either enriched HREE or flat HREE patterns (Fig. 11e), and their Th/U ratio is also varied (Th/U = 0.11 to 0.47, U = 17-264 ppm). No consistency could be observed between the remaining zircon analysis.

503 **5. General discussion and geodynamic implications**

504 5.1. P-T conditions and equilibrium in HP mafic granulite

The studied samples KW4A, KW4C, and 135A are textbook high-P granulites. However, assessing their peak P–T conditions is limited by: (a) the preservation of microdomains with restricted scale of equilibration likely due to a low amount of H₂O, which is a catalyst in mineral reactions; and (b) the scarcity of quartz, which promotes mineral reactions in mafic rocks, leading to wide P–T stability fields of the peak assemblage.

Among the two modelled rocks, sample 135A, from the western Lelukuau, provided the
tightest P–T constrains. Modal proportions of phases calculated using P–T pseudosections of
the bulk rock composition including scapolite (which is inferred to be part of the main

assemblage) are consistent with the mineral proportions observed in the sample. Only slight

discrepancies in plagioclase (25–30 % instead 22 %), and quartz (up to 1.5 % instead of

< 0.5 %) are noted while the calculated clinopyroxene and garnet modal proportions are

within <10% compared to the proportions derived from SEM–MLA. Isopleths of X_{Mg} and

517 X_{Grs} in garnet are compatible with EMPA measurements and support the calculated P–T

estimates in the range between $770^{\circ}/14$ kbar and $870^{\circ}C/16$ kbar. These P–T estimates are in

519 line with available data on kyanite bearing anatectic rocks from the southern LT (e.g.,

520 14.5 kbar and 870–900°C, Lasalle and Indares, 2014), and with petrographic observations that

place the assemblages of both aluminous and mafic rocks in the high-P granulite field. In the calculated pseudosections, the liquid-in line at P<18 kbar, is between 900 and 950°C, close to the minimum peak T of 875°C for aluminous anatectic rocks from southern LT (Lasalle and Indares, 2014). The lack of partial melting in the samples is attributed to the scarcity of H₂Obearing phases. The slight discrepancy between the calculated temperature in metamafic and aluminous migmatite is within the range of uncertainty of P–T modelling.

The sample from eastern Lelukuau (KW4C), that preserves inherited zonation of the original igneous plagioclase, is dominated by local equilibria that makes it unsuitable for modelling, but it provides clues on the prograde history. Quartz and kyanite inclusions in garnet cores, together with garnet major element zoning, are consistent with a prograde portion of the P–T path, through the kyanite stability field. In addition, the sharp boundaries of the inclusionbearing cores, in conjunction with the REE zoning, are consistent with two-stage garnet growth (Fig. 6e, f, h, i; Fig 8b).

Textural equilibrium in the absence of chemical equilibrium suggests short-term residency under high-grade conditions. Preservation of garnet prograde zoning, absence of retrograde textures, preservation of S-scapolite and preservation of igneous zoning in recrystallized plagioclase domains is consistent with fast cooling following the thermal peak. In addition, the absence of retrograde textures testifies to a fluid-poor environment during cooling and decompression.

540

- 541 **5.2. From ages to stages**
- 542 5.2.1. Garnet petrochronology

543 Western Lelukuau: sample 135A

In this sample, WR, clinopyroxene, and garnet fractions (excluding g4 for Lu-Hf) align and 544 545 yield a low MSWD isochron age for the two isotopic systems, which indicates closed system evolution following equilibration. Together with petrographic and P-T evidence for high-P-546 granulite-facies conditions, and with the Lu–Hf and Sm–Nd ages overlapping within error at 547 548 1019.9 ± 6.8 Ma (MSWD = 0.94) and 1005 ± 13 Ma (MSWD = 0.25), respectively, this provides an age for the peak metamorphism of the Western Lelukuau Terrane. The Lu-Hf 549 550 ages are systematically skewed toward garnet early growth (e.g., see Anczkiewicz et al, 2007; Lotout et al., 2018, Tual et al., 2022) and the Lu-Hf isotopic system has a higher closure 551 temperature than Sm-Nd (see Smit et al., 2013), which is particularly important in high-T 552 553 terranes. Given the ca. \pm 7 Ma uncertainty on the isochron and the ~ 800-900°C peak 554 metamorphic temperatures, and despite that Lu-Hf and Sm-Nd ages slightly overlap, we interpret the garnet Lu-Hf age to approximate garnet growth and crystallization at ca. 555 556 1020 Ma, and the garnet Sm-Nd age to approximate subsequent, fast cooling at ca. 1005 Ma at lower P. The preserved zoning pattern in garnet with increasing Lu towards the rims, is 557 atypical, compared to the classic Lu-rich core to Lu-poor-rim resulting from Rayleigh 558 fractionation. Breakdown of a HREE-rich phase during garnet growth is most likely 559 560 responsible for this pattern. Given the low Lu concentration in garnet, and the fact that zircon 561 cores are rich in Lu (up to 1000 times more), zircon breakdown during garnet growth could be 562 sufficient to release enough Lu inducing such garnet zoning.

563 Eastern Lelukuau : sample KW4C

564 The Eastern Lelukuau sample preserves petrographic evidence of two-stage garnet growth,

that is corroborated by trace element zoning. The variable amount of kyanite needles and

566 quartz crystals in the residues from the different garnet fractions, together with the two mean

- 567 Lu-Hf age clusters, allow the interpretation of the older age of 1033 ± 6.3 Ma as
- approximating first garnet growth, within the kyanite stability field. The younger Lu–Hf date

of 1013 ± 5.6 Ma is interpreted to estimate the age of the second garnet growth. The latter also overlaps within error with the Sm–Nd date of 1003 ± 8.3 Ma. Given the petrographic record, this date is interpreted to reflect cooling of the terrane at lower pressure.

572

573 **5.3. Zircon ages**

Zircon was only retrieved in sample 135A, and shows a complex history. The U-Pb dataset is 574 consistent with continuous or multiple lead loss event, and, potentially, several ages of zircon 575 576 growth. The oldest age of 1.6 Ga from 16 zircon cores is in good agreement with the ca. 1631 Ma age of a gabbro-troctolite from the AMCG suite in the same area (Indares et al., 577 1998). Additional zircon growth episodes at ca. 1.4 Ga and 1.2 Ga are deduced from the 578 textures and the ²⁰⁷Pb/²⁰⁶Pb ages. The similarities between the REE patterns in the different 579 CL zones with different ²⁰⁷Pb/²⁰⁶Pb dates, and the large variability in the elemental record of 580 581 CL zones recording similar dates, could be attributed to partial dissolution-reprecipitation of igneous zircon below the solidus. This would reset the age but preserve the initial REE 582 signature and new growth, both variably affected by lead loss during metamorphism. 583 584 Moreover, the Eu* and Lu content are not linked to a specific CL texture.

585 Only a few zircon rims are of Grenvillian age, and they are mostly discordant. The Eu* anomaly is consistently low to absent, from 0.8 to 1.2. This could suggest absence of 586 plagioclase (e.g., Rubatto 2002, 2017; Whitehouse and Platt, 2003) and/or growth of garnet 587 that fractionate Fe^{2+} over Fe^{3+} , changing the overall oxidation state and converting Eu^{2+} to 588 Eu³⁺ (e.g., Tang et al., 2018). In both cases, it would imply crystallization of zircon under 589 590 high-pressure, at 1.6, 1.4 and 1.2 Ga. However, zircon show classic HREE-enrichment, typical of crystallization in a garnet-absent assemblage, which contradicts this interpretation. 591 Another way to interpret this dataset would either be for zircon to 1) crystallize before or 592 during early plagioclase crystallization, 2) crystallize in an oxidized magma, or 3) crystallize 593

from kinetic disequilibrium, as has been invoked for Eu* anomalies in clinopyroxene (see Tilhac et al., 2023). Option 1 can easily be ruled out, as late-stage crystallization of zircon from a local high differentiated pocket of magma are needed to crystallize zircon in Zr undersaturated mafic magmas. Given the homogeneous REE patterns, kinetic disequilibrium seems unlikely, and an oxidized magma appears to be a preferable option.

599 Ages at 1.4 and 1.2 Ga are well known in Tshenukutish Terrane and in the Mid-P belt to the 600 south. 1.4 Ga corresponds to a major crust forming event (Pinwarian), whereas 1.2 Ga is the 601 time of localized extensional episodes (see review in Indares, 2020). However, it seems unlikely that these events would have promoted metamorphic zircon growth in the dry MIZ 602 protoliths. Considering that sample 135A has no record of inherited gabbroic textures, an 603 604 alternate explanation for the variability in the zircon dataset would be that the protolith of this 605 sample is a mafic supracrustal rock with a sedimentary component rather than a plutonic rock, 606 and that the zircons are detrital. This is also supported by the large range of Th/U ratio at 607 distinct dates, that could suggest variable zircon sources.

Finally, regardless of the protolith nature of the sample and only looking at recorded dates,
zircon did not successfully recrystallize during the high P–T event. The absence of partial
melting and the dryness inherent to mafic high-P granulite is a plausible explanation for
zircon's rare reactions despite the high P–T metamorphism that occurred at 1020 Ma.

612

5.4. Overall metamorphic patterns in the MIZ and tectonic implications

The new garnet ages reported in this study provide important constraints on the
metamorphism of the MIZ since they are derived from mafic rocks from the LT, that make up
its largest lithotectonic component. The Lu– Hf ages, at ca. 1033 Ma, with a second garnet
growth at ca 1013 Ma in the eastern LT sample and at ca. 1020 Ma in the western LT sample,

suggest that the high-P granulite assemblage formed during the late Ottawan straddling the
Ottawan-Rigolet hiatus. They also suggest that there may be diachronism in garnet growth
from East to West. In addition, the Sm-Nd isotope data are consistent with rapid cooling at ca.
1005 – 1003 Ma.

These ages are younger than the monazite dates from an aluminous anatectic rock in southern

LT (ca. 1060 – 1044 Ma, Dunning and Indares 2010; Lassalle et al., 2014), however, the

624 previously published ages are difficult to interpret because of strong inheritance.

625 Nevertheless, taken together, these data suggest a potential younging of the age of

626 metamorphism from the southern boundary of LT to the north, towards the Parautochthonous

627 Belt.

628 Anatectic aluminous rocks from the Tshenukutish Terrane, at the SSE margin of the MIZ, 629 were subjected to similar high-P metamorphic conditions (Indares et al., 2008; Lasalle and Indares, 2014; Kendrick and Indares, 2018) and show well constrained monazite ages from 630 ca. 1040 to 1017 Ma, interpreted to approximate the age of high-P metamorphism. These ages 631 are younging to the east, towards the boundary of the Tsenukutish Terrane against the Hart 632 Jaune terrane (Fig. 1c), and were interpreted to represent progressively later incorporation of 633 tectonic slivers in the slice stack (Indares 2020). In contrast, ages of mid-P granulite facies 634 metamorphism, in the structurally overlying mid-P belt in the Manicouagan area, range 635 636 between ca. 1080 and 1040 Ma, i.e. they are early to mid-Ottawan (Dunning and Indares, 637 2010; Lassalle et al., 2014).

The high-P belt of the Grenville Province has been traditionally attributed to the extrusion of
lower crust from beneath an orogenic plateau that developed during the early stages of the
Ottawan phase, prior to ca. 1050 Ma (e.g., Rivers, 2008, and reference therein). In this
scenario, the MIZ would show similar or older metamorphic ages than the overlying mid-P
belt. However, the diachronism observed in the metamorphic age pattern of the hinterland in

the central Grenville contradicts this interpretation, as younger, late Ottawan transitional to 643 644 Rigolet ages are recorded in the MIZ relative to the early to mid-Ottawan ages in the structurally overlying Mid-P belt. This highlights a general younging of the metamorphic ages 645 towards the NW that is best explained by local crustal thickening in the margin of the 646 647 orogenic front at the time the orogen was progressing (e.g., see Fig. 16b and c of Indares, 2020). This local thickening would have been facilitated by the presence of the rigid Archean 648 649 lithosphere of the Parautochthonous belt, as proposed by Indares (2020). Although the presence of a thick orogenic plateau in the central Grenville cannot be excluded, the new data 650 show that the MIZ does not provide tangible evidence, and are in line with recent proposals 651 652 for overall thin orogenic crust in the Mesoproterozoic (e.g., Spencer et al., 2021). 653 These new metamorphic ages still predate the incorporation of the Parautochthonous Gagnon

terrane in the Grenville orogen during the Rigolet phase, as suggested by the ~1000–980 Ma
zircon and monazite metamorphic ages from the footwall of the MIZ (Jordan et al. 2006;
Jannin et al. 2018a, b). However, these ages indicate a continuum in the tectonic activity of
the orogen, and discount the tectonic hiatus hypothesis traditionally inferred between the
Ottawan and Rigolet orogenic phases.

659

5.5. High-P domains in the Grenville Province

The characteristics of the MIZ are markedly distinct from the other two high-P segments 660 documented in the Grenville Province (Fig.1b). In the western high-P segment, eclogite pods 661 662 in orthogneisses (Davidson, 1990) have metamorphic ages as old as 1090 Ma, broadly 663 coincident with early stages of the continental collision (Hynes and Rivers, 2010), followed by a 1080 Ma granulite-facies overprint (zircon; Marsh & Culshaw, 2014). Both the western 664 high-P segment and MIZ belong to the Allochthonous belt and are located structurally above 665 666 the Parautochthonous belt. They have been referred in the literature as the 'high-P belt', however, it now appears that the ages in the western segment are much older than in the MIZ. 667

They consequently correspond to different orogenic episodes and, because of this, the term "high-P belt" is not appropriate. Finally, east of the MIZ, the high-P Molson Lake terrane is part of the Parautochthonous belt, structurally below the Orogenic Lid in that area (St Joseph terrane, Connelly et al., 1995), with preliminary metamorphic ages at 1.00 Ga (see Rivers et al. 2012).

Collectively, the three high-P segments provide evidence of significant crustal thickening in a
Mesoproterozoic orogenic setting. Although the tectonic significance of the other two
segments is poorly understood, they are all exposed at the frontal parts of the orogen, and
formed diachronously, defining a pattern of younging towards the Eastern part of the
Grenville Province, while also getting closer to the Grenville Front.

678

679 6. Conclusions

High-P domains exposed in the Grenville Province provide unequivocal evidence of crustal
thickening in a Mesoproterozoic orogenic setting; their characteristics, however, are far from
uniform. The metamorphic patterns and tectonic significance of the Manicouagan Imbricate
Zone (MIZ) explored in this study provide new insights on high-P processes in the
Mesoproterozoic. The key outcomes are summarized below:

685
1. Mafic rocks from the Manicouagan Imbricate Zone underwent high-P granulite
686 metamorphism of 14–16kbar and 800–900°C.

687
2. This study in the MIZ provides the first garnet Lu–Hf and Sm–Nd dating of high-P
688
689
689 to 1020 Ma and followed by subsequent fast cooling, with an apparent younging of
690 the ages towards the Grenville Front.

- 691 3. Zircon, in dry high-P mafic granulites appears to not successfully recrystallize due692 to lack of partial melt and hydrous phases.
- 4. The high-P granulite rocks in the Manicouagan Imbricate Zone are not related to
 the extrusion of metamorphosed lower crust during collision and building of a
 high-plateau during the early stage of the Ottawan phase as previously thought.
 Instead, it is related to local crustal thickening as the orogen propagates towards
 the Grenville front. Therefore, the existence of a thick orogenic plateau in the
 Grenville is questioned.
- 5. The results from this study support a tectonic continuum in between the Ottawan
 and Rigolet phases, and highlight the long-lived active tectonic character of the
 Grenville Orogeny.

Finally, this study highlights the diachronicity of the high-P domains of the Grenville Province, and calls for a better understanding of the overall pattern of crustal thickening at the broad regional scale of the Province.

Debate on the Mesoproterozoic geodynamic styles continues and spans from single lid to
active tectonics. The multiplicity of tectonic scenarios for the Grenville Orogeny, and for
Mesoproterozoic orogenies in general, highlights that the scarcity of P-T-t data is a barrier to
comprehensive large-scale reconstructions and geodynamic interpretations.

709

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725 CAPTIONS

Figure 1 - A. Schematic map of the Grenville Orogen in North America, B. Schematic map

- highlighting the tectono-metamorphic divisions of the Grenville Province, modified after
- Rivers et al. (2008) and Indares (2020). ABT Allochton Boundary Thrust. C. Schematic
- map highlighting the tectono-metamorphic divisions of the Manicouagan Imbricate Zone
- 730 (MIZ, Central Grenville Province), modified after Indares (2020), sample location indicated.

731 Light pink in the LT shows locations of the anatectic aluminous outcrops.

- Figure 2 Available ages compiled from Cox et al. (1998), Indares et al. (1998), Indares and
- 733 Dunning (2001, 2004), Dunning and Indares (2010), Lasalle et al. (2014), modified from
- T34 Indares and Dunning (2017). Error bars less than 5 my are not shown.
- Figure 3 False color mineral maps generated by Scanning Electron Microscopy and Mineral
- Tiberation Analysis of polished thin sections, and corresponding mineral abundances. A.
- sample 135a, western Lelukuau terrane. B. sample KW4C, eastern Lelukuau terrane.
- 738 Plagioclase: light grey (low Ca) to dark grey (high Ca). Garnet: pink (low Ca) to burgundy
- (high Ca). C. sample KW4A, eastern Lelukuau terrane. Corresponding proportions are shown
- 740 in Table 1.
- Figure 4 Photomicrographs of samples A. 135a, B. KW4C, C. KW4A
- Figure 5- Compositional profiles (A) and diagrams (B to E) for the minerals from samples
- 135A (pink), KW4C (green) and KW4A (orange). A. Garnet. B. Amphibole (CaB \geq 1.5 and
- 744 (Na+K)^A \ge 0.5) following the Leake et al. (1997) classification. C. Clinopyroxene. D.
- 745 Plagioclase. The lighter colored symbols represent the scapolite composition recalculated as
- 746 plagioclase, following Ellis (1978). E. Scapolite.

Figure 6 - SEM pictures (A, D, G and J) and X-ray maps for Ca (B, E, H, K), Fe (C, F, L) and
Mg (I) of garnet grains for samples 135A, KW4C, KW4A. Abbreviations are listed in the
methodology section.

Figure 7 : P–T pseudosection calculated in the CNFMASHO system for samples 135A (A, B)
and KW4A (C).

Figure 8 - Rare Earth Element patterns normalized to chondrite (after Sun and Mcdonough
1989) acquired in garnet (yellow, magenta and blue) and for the bulk rock (black). Limit of
detection in grey for the corresponding set of analysis.

Figure 9– (A, C) Lu–Hf and (B, D) Sm–Nd isochron diagrams for samples 135A and KW4C.

756 WRB, whole rock bomb dissolution; WRS whole rock tabletop dissolution; g garnet; 1–6,

757 garnet fractions; cpx, clinopyroxene. Inserts show the mean age of garnet-WRB 2-point

isochrons. Zooms on the isochrons (B and D) show the reproducibility of the whole-rocks

759 Sm-Nd analysis and (C) the scatter amongst the Lu–Hf garnet fractions and the whole rocks.

"Zrn" in (A) indicate the sample in which a single grain of zircon was recovered after partialdissolution.

Figure 10 - Cathodoluminescence images of the analyzed zircon crystals from sample 135a

(western Lelukuau), in thick section and epoxy mounts. Color-coded circles show the location
 of the U–Pb and REE analyses, carried out on top of each other at the SIMS. ²⁰⁷Pb/²⁰⁶Pb dates

are quoted with 2σ uncertainty in Ma, with concordance > 90%. Th/U ratio and common lead (italic) are shown.

Figure 11 – Zircon U–Pb, Th/U, and Rare Earth Element data for the Western-Lelukuau

sample (135A). A. Concordia diagram with theoretical recent lead loss and 1.0 Ga-1.6 Ga

discordia line. B. Kernel density plot of ²⁰⁷Pb/²⁰⁶Pb ages. C. ²⁰⁷Pb/²⁰⁶Pb dates as a function of

Th/U. D 207 Pb/ 206 Pb dates, selected data used for average date calculation are highlighted. E.

Rare Earth Element patterns normalized to chondrite (after Sun and Mcdonough 1989), with
color code for cores, rims and rims with an atypical pattern. F. Lu concentration as a function
of the Eu*.

774

- Table 1 Summary of the mineral proportion obtained by SEM–MLA on thin section of the
- samples 135A, KW4C and KW4A, and corresponding to Fig.3. Kyanite, rutile, zircon and
- apatite are below 0.5 % in all samples.

778

- 779 Table 2 Summary of Lu-Hf and Sm-Nd isotopic data. Dates are produced using isoplot
- 780 (Ludwig, 2003) and are reported at 95% confidence level.
- ^a g garnet fraction, cpx clinopyroxene fraction, wrs whole-rock savillex tabletop
- 782 dissolution, wrb whole-rock bomb dissolution.

^bLu, Hf, Sm, Nd concentration (ppm) obtained via isotope dilution (ca. 0.5% se).

- ^c Uncertainty on the 147 Sm/ 144 Nd and 176 Lu/ 177 Hf ratios is estimated to be 0.5%.
- ^d Within-run 2σ standard error (6th decimal), combined in quadrature with external
- reproducibility of spiked whole-rock samples for age regression.
- re ¹⁷⁶Lu decay constant used for age calculation is 1.867×10^{-11} a⁻¹ (Söderlund et al., 2004).
- ¹⁴⁷Sm decay constant used for age calculation is 6.54×10^{-12} a⁻¹ (Lugmair & Marti, 1978). 2-
- point dates are the date obtained from a two-point isochron pairing of a single garnet fraction

790 with wrb

- Supplementary 1 Whole rock major (in mol.% oxide) and REE (in ppm) composition.
- 792 Supplementary 2 Representative microprobe analyses of minerals.

- Supplementary 3 Summary of the U-Pb SIMS and REE analysis for zircon (sheet1 and 2,
- respectively), and REE analysis in garnet (sheet 3).
- Appendix Details on operating conditions, standards, chemical procedures, and solid
- solution models used or carried out.

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Figure 2



Figure 3







Figure 5









KW4A



*Figure 6



Figure 7





Figure 8



Figure 9



Figure 10



Figure 11

Table 1

	garnet	clinopyroxene	plagioclase	amphibole	scapolite	quartz	biotite
135A	30	30	22	10	5	< 0.5	1.5
KW4C	21	14	50	8	2.5	4.5	< 0.5
KW4A	47	28	17	< 0.5	5	< 0.5	< 0.5

Sample ^a	Lu ^b (ppm)	Hf ^b (ppm)	¹⁷⁶ Lu/ ¹⁷⁷ Hf ^c	¹⁷⁶ Hf/ ¹⁷⁷ Hf	± ^d	2-point date (Ma) ^e	Sm ^b (ppm)	Nd ^b (ppm)	¹⁴⁷ Sm/ ¹⁴⁴ Nd ^c	¹⁴³ Nd/ ¹⁴⁴ Nd	± ^d	2-point date (Ma) ^e
135A												
g1	0.582	0.134	0.6187	0.293805	31	1009.3 ± 6.2	1.60	2.74	0.3526	0.513367	27	999 ± 26
g2	0.594	0.136	0.6234	0.294034	31	1021.4 ± 6.2	1.59	2.61	0.3692	0.513492	27	1010 ± 24
g3	0.600	0.132	0.6448	0.294456	31	1022.2 ± 6.1	1.54	2.19	0.4237	0.513839	27	1003 ± 20
g4	0.601	0.133	0.6433	0.294129	31	997.1 ± 6.0	1.56	2.29	0.4124	0.513757	27	998 ± 21
g5	0.583	0.140	0.5895	0.293350	31	1018.4 ± 6.3	*	*	*	*	*	*
g6	0.589	0.130	0.6466	0.294454	29	1019.1 ± 6.1	*	*	*	*	*	*
срх	0.014	1.96	0.0010	0.282014	28		2.54	8.57	0.1791	0.512222	27	
wrs	0.160	0.783	0.0290	0.282572	28		1.11	5.45	0.1229	0.511853	27	
wrb	0.419	3.59	0.0166	0.282351	28		2.74	13.5	0.1232	0.511862	26	
KW4C												
g1	0.219	0.078	0.4002	0.289660	31	1014.6 ± 7.8	0.733	0.847	0.5232	0.514536	27	1006 ± 16
g2	0.217	0.077	0.4012	0.289747	32	1023.9 ± 7.9	0.702	0.789	0.5385	0.514635	27	1006 ± 15
g3	0.216	0.078	0.3956	0.289554	35	1011.9 ± 6.6	0.711	0.774	0.5563	0.514744	27	1002 ± 15
g4	0.221	0.079	0.3948	0.289546	30	1013 ± 7.8	0.754	0.856	0.5325	0.514593	27	1004 ± 16
g5	0.214	0.078	0.3879	0.289527	30	1029.1 ± 7.9	*	*	*	*	*	*
g6	0.205	0.074	0.3940	0.289699	30	1036.8 ± 7.9	*	*	*	*	*	*
cpx	0.006	1.15	0.0008	0.281973	28		5.44	22.4	0.1466	0.512056	27	
wrs	0.055	0.342	0.0228	0.282495	29		1.08	4.88	0.1343	0.511975	27	
wrb	0.057	0.321	0.0251	0.282488	28		1.09	4.93	0.1343	0.511968	26	

Table 2