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Santa Barbara

Lower Crustal Metasediments: Implications for Continental Evolution and Their Utility in
Recording Geologic History

A dissertation submitted in partial satisfaction of the
requirements for the degree Doctor of Philosophy
in Earth Science

by

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Recording Geologic History

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by

Mary Ferrand Ringwood

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ABSTRACT

Lower Crustal Metasediments: Implications for Continental Evolution and Their Utility in Recording Geologic History

by

Mary Ferrand Ringwood

[The Earth is unique among the terrestrial planets in our solar system, and no other known planet has a similarly bimodal distribution of surface elevations. The thicker, lighter continents are intermediate in composition, as opposed to the denser, mafic oceanic crust. All magmatism is ultimately sourced from the mantle; yet, mantle melting alone produces basalt. Multiple hypotheses have been proposed to explain the composition and evolution of the continental crust, including the incorporation of felsic sediments from the surface into the deep crust. How this happens and the amount of metasedimentary material in the lower continental crust remains unknown. There are four documented processes that can transport surface material to depth: (1) tectonic underplating in a forearc setting (e.g., Jacobson et al., 2011), (2) deep thrust faulting in a retroarc (e.g., Chin et al., 2013), (3) relamination via deep subduction and buoyancy-driven diapiric ascent (e.g., Hacker et al., 2011), (4) deep thrust faulting in a continent-continent collision (e.g., Zhao et al., 2001). Petrochronology of deep-seated metasedimentary rocks can distinguish between these scenarios by revealing the timing and conditions of metamorphism and the circumstances under which sediments were

returned to the surface. To that end, three case studies of lower crustal metasedimentary rocks erupted as xenoliths were carried out and are described herein.

Metapelitic xenoliths from New Mexico (USA) and Chihuahua (Mexico) record sediment transport to the lower crust during continent-continent collision associated with the Mesoproterozoic Picuris orogeny. Geothermobarometry and Rio Grande rift-age rutile (Cipar et al., 2020) document equilibration in the local deep crust, which is actively undergoing ultra-high-temperature metamorphism. The original sediments were deposited during the early stages of the Picuris orogeny (1500–1450 Ma), and the transition from detrital igneous zircon accumulation to metamorphic zircon and monazite growth marks the end of subduction and onset of collision. Metamorphic zircon and monazite also grew during Rio Grande rift activity beginning at 32 Ma. Nearly all garnet grains are homogeneous with respect to major and trace elements, and monazite inclusions within garnet indicate that they are Rio Grande rift in age. A single garnet grain is zoned in Zr and Hf, the slowest diffusing elements, with rim concentrations similar to those of other garnets in the sample. The core, however, has lower concentrations, indicating growth at lower temperatures, and is distinct in its abundance of acicular sillimanite inclusions. The presence of sillimanite points to metamorphism under thermal gradients higher than those found in subduction zones, and negative Eu anomalies in all zircon and monazite rule out metamorphism at the ultra-high-pressures that would have preceded relamination. Collectively, these data demonstrate transport of sediments from the surface to the lower continental crust during a collisional orogeny. The sediments resided in the lower deep continental crust for over 1400 Ma, and their presence there defines the location of the suture zone that formed during the Picuris orogeny.

Lower crustal xenoliths from the Great Falls tectonic zone (GFTZ) in Montana (USA) record a multi-billion-year geologic history prior to their entrainment and eruption during the Cenozoic Laramide orogeny. Metapelitic samples equilibrated in the lower crust, and mafic xenoliths with an arc cumulate origin document conditions in excess of the current crustal thickness. Rutile U-Pb ages date the eruption of the host rocks to ~50 Ma and demonstrate residence of the samples in the deep crust until that time. Metamorphic zircon and monazite in all metapelitic samples record sediment transport to depth during a major metamorphic event beginning ~1800 Ma, while one sample also documents an event at 2200–2000 Ma related to burial and metamorphism during rifting. Younger metapelite xenoliths show a clear transition from detrital igneous zircon formation to metamorphic zircon and monazite growth at 1800 Ma, corresponding the end of subduction and beginning of collision during the Paleoproterozoic Great Falls orogeny. Their occurrence in the deep crust of the Great Falls tectonic zone (GFTZ) provides evidence that the GFTZ is a suture zone between the Medicine Hat block and Wyoming craton. U-Pb and trace-element depth profiles of monazite and zircon also record metasomatism in the deep crust at ~60 Ma during the Laramide orogeny, which is consistent with recent suggestions for Farallon slab fluid infiltration at this time. These rocks remained in the lower continental crust for over 1700 Ma.

Metapelitic xenoliths from the Bournac volcanic pipe in the Massif Central (France) record the incorporation of sediments into the lower continental crust during the collisional phase of the Paleozoic Variscan orogeny. Geothermobarometry yields pressure-temperature conditions consistent with a geothermal gradient in excess of those associated with a continental geotherm, and rutile in these samples remained above the Pb closure temperature

until eruption of the xenoliths at 11.6 Ma. Detrital igneous zircon date back to the Archean, and the sediments were deposited after 393 Ma based on the youngest detrital igneous zircon and monazite ages. Metamorphic zircon and monazite document the transport of the sediments to depth beginning ~330 Ma, which is consistent with the known timing of continent-continent collision during the Variscan orogeny just prior to an episode of UHT metamorphism in the lower crust that peaked at 313 Ma (Laurent et al., 2023). Post-orogenic collapse began by 300 Ma, and zircon and monazite crystallization continued until ~265 Ma as the crust thinned and cooled. Later rifting events are recorded by even younger metamorphic zircon and monazite dates (as young as 207 and 74 Ma, respectively). These data demonstrate that the original sediments were transported to depth during continent-continent collision and resided in the stable lower continental crust for over 300 Ma.]

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I. Introduction and Motivations

The continental crust is fundamental to modern life on Earth. Humanity resides almost entirely on the continents, utilizing their resources to maintain modern society. Despite its paramount importance to human civilization, basic characteristics of the continental crust are not fully understood: It is ~35 km thick, felsic, and old, while the oceanic crust is ~6 km thick, mafic, and young. Mantle melting creates basalt, which makes up the oceanic crust, but the continental crust is, on average, andesitic. This begs the question: How is continental crust formed, and what processes explain its evolution toward more felsic compositions over time? Several hypotheses have been proposed to explain the evolved nature of the continental crust and its formation and refinement over Earth's history, including melting of oceanic crust during the hotter Archean, foundering of mafic material from the base of the continental crust, and chemical weathering (e.g., Martin et al., 1986; Arndt & Goldstein, 1989; Lee et al., 2008; respectively).

These processes are unequivocally involved in the generation and evolution of the continents, but how much of a role each plays and how those roles might have changed over time and from one part of the world to another remains unknown. Likewise, sediments are a major source of felsic material formed at the Earth's surface, but we know that they can be metamorphosed under high- or ultra-high-pressure conditions. In some cases, sediments have become stable components of the lower continental crust, which appears to be an important means by which felsic material is incorporated into the deep crust and thus a process that contributes to the growth and evolution of the felsic continents. Examples of sedimentary material in the deep continental crust are found in lower crustal xenoliths carried to the surface by magmatism (e.g., Chin et al., 2013; Reid et al., 1989) and in large,

exhumed terranes (e.g., Dumond et al., 2018). Due to the inaccessibility of such depths, the amount of sedimentary material in the lower continental crust is unknown, but estimates point to as much as 10–20% being possible (Hacker et al., 2011).

Documenting the processes responsible for transporting sediments from the surface to depth and how they differ across time and location promotes a deeper understanding of the ways in which felsic material is added to the lower continental crust. In turn, such processes help explain the formation of the continents, as well as their evolution over Earth's history, which has significant implications for the past and present bulk crustal composition, the lower crust's composition and heterogeneity, and the amount of heat produced in the lower crust compared to the mantle.

There are four main processes that have been proposed for sediment transport to depth: (1) underplating of sediments in the forearc during subduction (e.g., the Pelona-Orocopia-Rand schists in California, USA: Jacobsen et al., 2011), (2) deep-seated thrust faulting in the retroarc of a magmatic arc undergoing compression (e.g., North American cordillera: Chin et al., 2013; DeCelles et al., 2009), (3) relamination, wherein sedimentary material is subducted to ultra-high pressures and then rises buoyantly through the mantle wedge before stalling out at the base of the over-riding plate's crust (Hacker et al., 2011; Kelemen and Behn, 2016), and (4) deep-seated thrust-faulting during continent-continent collision (e.g., Himalayas: Zhao et al., 1993).

To explore the ways sediments can be incorporated into the lower continental crust, high- and ultra-high-pressure metasedimentary rocks, including metapelites, can be studied to answer the following questions: (1) Does the thermobarometry document their equilibration in the deep crust? (2) What metamorphic events do the geochronology record,

and which are responsible for transport to depth? (3) Can the timing and conditions of metamorphism reveal the tectonic setting at the time? (4) Were the sediments actually incorporated into the stable lower crust, remaining there for an extended period of time, or were they rapidly returned to the surface?

Furthermore, metasedimentary samples from the lower continental crust provide excellent opportunities to explore the history of the deep crust in a region, as well as broader tectonic history. Compared to mafic rocks, metasediments are more easily influenced by metamorphic events and thus can better record geologic history. They also contain an abundance of minerals that can be utilized for petrochronology, including zircon, monazite, rutile, apatite, and garnet. These accessory phases are dateable using multiple isotopic systems and are relevant to a number of thermobarometry calculations, enabling the linking of time and pressure-temperature conditions.

Three case studies are presented here that utilize lower crustal metasedimentary xenoliths to understand the transport of felsic sedimentary material from the surface to depth, as well as the local geologic history of their regions, using multiple modern petrochronology and thermobarometry methods. In all three cases, the original sediments were incorporated into the lower continental crust during collisional orogens and remained there for hundreds of millions or even billions of years before being entrained by magmas and returned to the surface.

II. Metasediments from the Lower Crust Reveal the History of the Picuris Orogeny, Southwest USA

A. Abstract

Petrologic and geochronologic data for metapelitic lower crustal xenoliths from New Mexico (USA) and Chihuahua (Mexico) states provide evidence for both a magmatic and collisional component to the enigmatic Mesoproterozoic Picuris orogeny. These garnet-sillimanite-bearing metapelites are found within the southern Rio Grande rift at Kilbourne Hole and Potrillo Maar in southern New Mexico and northern Chihuahua.

Geothermobarometry and rutile with Quaternary U-Pb dates indicate equilibration in the local lower crust, which is actively undergoing ultra-high temperature (UHT) metamorphism (Cipar et al., 2020). The samples contain older detrital igneous zircons dating back the Paleoproterozoic, marking their deposition at the surface. Coupled zircon U-Pb dates and trace-element ratios (e.g., Gd/Yb) show a clear transition from oscillatory-zoned, low-Gd/Yb detrital igneous zircon to featureless, high-Gd/Yb metamorphic zircon between 1500 and 1400 Ma, marking the transition from subduction to collision during this period.

Metamorphic zircon and monazite grew in two major intervals. The first, between ca. 1450 and 1350 Ma, documents the journey of the sediments to depth within the orogen and provides evidence of extended Mesoproterozoic metamorphism in the region. The second corresponds with UHT metamorphism that commenced at ca. 32 Ma and is associated with the Rio Grande rift. Whereas nearly all garnets are homogeneous in both major and trace elements, a single garnet from one sample has a core defined by abundant quartz and acicular sillimanite inclusions. The core and rim of this garnet are homogeneous in major

and most trace elements, but the rim is enriched in the slowest diffusing elements, Zr and Hf, which likely indicates rim growth at higher temperatures. We interpret the garnet core to have grown at the time of emplacement of the sediments into the lower crust. Because this occurred in the sillimanite stability field and because the metamorphic zircon and monazite all have negative Eu anomalies, indicating their equilibration with feldspar (stable at depths of <45 km), we conclude that the sediments were not emplaced via subduction and/or relamination of forearc sediments, but were instead metamorphosed under warmer, shallower conditions in an orogenic setting. Collectively, the data point to a collisional orogen during the inferred timing of the Picuris orogeny. These samples may therefore define the location of the Picuris suture zone, a key feature of this orogenic event.

B. Introduction

The southern margin of Laurentia, which corresponds to the present-day midcontinent region of North America, was an important locus for the growth of continental crust during the Proterozoic (Fig. 1) (Karlstrom et. al, 2001; Whitmeyer and Karlstrom, 2007). This time period was punctuated by a series of well-documented events: the Yavapai, Mazatzal, and Grenville orogenies (Fig. 1) (e.g., Bowring and Karlstrom, 1990; Rivers, 1997; Eisele and Isachsen, 2001; Bartholomew and Hatcher, 2010; Mulder et al., 2017). Despite extensive work on the tectonics of Proterozoic southern Laurentia, less attention has been paid to activity in the time interval between the Mazatzal and Grenville orogenies. Early research focused on the voluminous evolved magmatic rocks emplaced between 1500 and 1340 Ma (including the granite-rhyolite provinces), which have been ascribed an anorogenic origin (e.g., Anderson, 1983; Anderson and Bender, 1989; Anderson and Morrison, 1992).

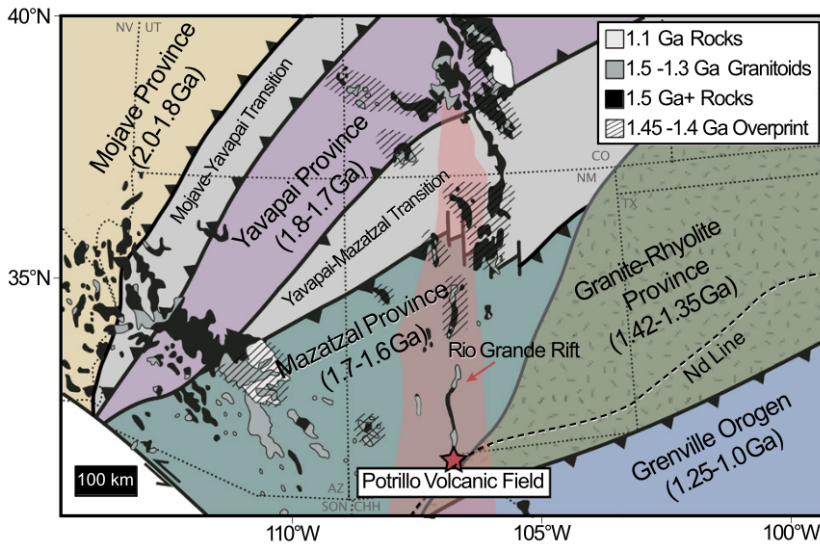


Figure 1. Map of southwest USA showing accreted terranes. Key shows rock outcrops and overprints: locations of 1.5–1.3 Ga granitoids indicated in regions where they occur outside the granite-rhyolite province and the 1.45–1.4 Ga overprint (stippled pattern) showing areas previously identified as having experienced Picuris-aged metamorphism and deformation (Daniel et al., 2013b). Location of Kilbourne Hole and Potrillo Maar marked by star. The Nd line, which marks the boundary determined from Nd isotopic mapping, separates older continental crust (1.8–1.6 Ga Nd model ages) in the north from younger accreted terranes to the south (1.5–1.4 Ga Nd model ages) and is shown as a dashed black line. Rio Grande rift highlighted in red and identified by a red arrow. Modified from Karlstrom et al. (2004), Amato et al. (2008), and Bickford et al. (2015). AZ—Arizona, CHH—Chihuahua, CO—Colorado, NV—Nevada, SON—Sonora, TX—Texas, UT—Utah.

the final docking of the Mazatzal terrane (Daniel et al., 2013b).

A major issue in understanding the Picuris orogeny is the lack of an obvious suture zone. The only potential evidence of this feature was identified through Nd isotopic mapping (Bennett and DePaolo, 1987; Bickford et al., 2015), which showed a somewhat diffuse isotopic boundary between ca. 1600 and ca. 1400 Ma crust (the Nd line, Fig. 1). Additional data linking the Nd line to the Picuris orogeny would shed light on a possible relationship

However, work on a variety of rock types, including metasediments and other deformed units, provides evidence for a distinct orogeny during this time: the Picuris (Daniel et al., 2013a, 2013b; Karlstrom et al., 2016). Some have suggested that the Picuris is an along-strike analog to the Baraboo and Pinware orogens of eastern Laurentia (Aronoff et al., 2016; Daniel et al., 2023) and that it may represent

between the two. Thus, the study of ca. 1500–1400 Ma rocks, especially those that were metamorphosed during this time, is necessary. In particular, lower crustal rocks, which can record tectonic events and changing pressure-temperature (P-T) conditions, have the potential to provide major insight into the extent, timing, and nature of the tectonics involved in the assembly of Laurentia during this time.

Here, we report petrologic and geochronologic data for metapelitic xenoliths carried by magmas erupted within the Potrillo volcanic field from Kilbourne Hole and Potrillo Maar, which are part of the Rio Grande rift in New Mexico (USA) and Chihuahua (Mexico) states (Padovani and Carter, 1977a, 1977b). The data presented include (1) petrography to identify the most appropriate samples for detailed analyses and provide mineralogical and textural context for analyses; (2) U-Pb dates and trace-element analyses of metamorphic zircon, detrital igneous zircon cores, and monazite to determine the timing of metamorphism, as well as maximum depositional age; and (3) mineral and whole-rock geochemistry and thermobarometry utilizing the garnet-aluminosilicate-plagioclase-quartz equilibria (GASP) barometer, the Zr-in-rutile barometer, and pseudosection modeling to explore changing metamorphic conditions. The results provide new insights into the nature of the Picuris orogeny and record a lengthy metamorphic history dating back to the Mesoproterozoic.

C. Geologic Background

Kilbourne Hole and Potrillo Maar are well-known xenolith localities within the Potrillo volcanic field. The lavas erupted during prolonged extension associated with the Rio Grande rift, which has thinned the crust to ~25 km (e.g., Cipar et al., 2020). This area is largely composed of Mazatzal province crust, which makes up much of the southwestern United

States and is part of a broader series of terranes accreted during the Meso- and Paleoproterozoic (Fig. 1) (Bowring and Karlstrom, 1990; Rivers, 1997; Karlstrom et al., 2001; Whitmeyer and Karlstrom, 2007). Most Mazatzal crust was generated between 1680 and 1650 Ma, and the terrane likely collided with the Yavapai Province on or near the southern margin of Laurentia during the Mazatzal orogeny, which lasted from 1650 to 1633 Ma. Related post-orogenic magmatism continued until ca. 1600 Ma (Karlstrom et al., 2001; Whitmeyer and Karlstrom, 2007). Most of the region is overlain by Phanerozoic sedimentary and volcanic rocks (Seager et al., 1984).

Following the Mazatzal orogeny, there was a general lull in tectonic activity from 1600 to 1500 Ma, after which time the southern margin of Laurentia was reactivated, producing the granite-rhyolite provinces that are characterized by extensive A-type magmatism lasting from 1500 to 1340 Ma (e.g., Anderson and Bender, 1989; Barnes et al., 2002; Bickford et al., 2015; Holland et al., 2020). Although A-type granitic magmatism is generally interpreted to originate from mantle-derived melts in an anorogenic setting (Frost and Frost, 1997; Frost et al., 2001), evidence for coeval shortening and crustal thickening is indicative of a convergent plate boundary and regional orogenesis (Nyman et al., 1994; Daniel and Pyle, 2006; Bickford et al., 2015). A variety of data point to metamorphism and deformation related to orogenic activity at this time (1500–1400 Ma): detrital igneous zircon U-Pb dates in exposed metasedimentary rocks indicating deposition during the early Picuris and then subsequent deformation (e.g., Daniel et al., 2013a); thrust shear zones in northern New Mexico recording multiple generations of garnet growth between 1460 and 1400 Ma (Aronoff et al., 2016); and metamorphic ages from other minerals like monazite, hornblende, and muscovite (Aronoff et al., 2016; Bollen et al., 2022). Daniel et al. (2013b)

originally defined the orogenic activity from 1490 to 1450 Ma as the Picuris orogeny and later expanded this to the Pinware-Baraboo-Picuris orogeny lasting from 1520 to 1340 Ma (Daniel et al., 2023). Magmatism and metamorphism continued until ca. 1340 Ma (Anderson and Bender, 1989; Anderson and Morrison, 1992; Aronoff et al., 2016), indicating that the Picuris orogeny may have been a prolonged event, lasting over 150 Ma and involving both subduction and collision, or could have possibly been a series of discrete, shorter-lived collisional events. While the connection between the A-type magmatism, other granitoids, and regional metamorphism from this time may be unclear, it is now evident that the tectonic setting on the southern margin of Laurentia between 1500 and 1400 Ma included a convergent component and saw a transition from subduction to collision.

Following the Picuris, a shorter magmatic lull lasted until the Grenville orogeny began just to the south (Fig. 1) at ca. 1250 Ma. Grenville activity continued until ca. 980 Ma (Mosher, 1998; Bartholomew and Hatcher, 2010; Mulder et al., 2017) and involved terrane accretion from the modern-day southwest United States through eastern Canada, after which time the crust was relatively stable until the onset of extension in the Late Jurassic (e.g., Bilodeau, 1982; Lawton and McMillan, 1999). Between ca. 150 and 90 Ma the region to the southwest of modern-day New Mexico underwent a transition from an actively rifting margin to a retroarc foreland basin (Lawton et al., 2020). Rifting generated significant uplift along the margins, including the area in southern New Mexico (e.g., Chapman and DeCelles, 2021). Renewed uplift continued during the Laramide orogeny (ca. 80–40 Ma) as a result of shallow angle subduction along the western Laurentian margin (Gastil et al., 1992; Chapman et al., 2020). Post-Laramide, the region was relatively quiet until the Rio Grande rift became active at ca. 32 Ma, which is expressed as extension and magmatism at

the surface (Morgan et al., 1986) and ultra-high temperature (UHT) metamorphism in the lower crust (Cipar et al., 2020).

D. Samples and Previous Investigations of Xenoliths

This study focuses on metapelitic xenoliths (Table 1) from Kilbourne Hole and Potrillo Maar, which are found within the ejecta and surge deposits surrounding the two maars. The samples were either collected by the authors or come from the Ph.D. collection of Elaine Padovani, which is currently curated by the Smithsonian Institution. Specimens contain quartz + sillimanite + garnet + potassium feldspar ± plagioclase ± zircon ± monazite ± rutile ± ilmenite ± apatite ± graphite. No hydrous phases were observed in any of the samples, and many feldspars show perthitic textures or are ternary in nature (data in appendix), which is indicative of UHT metamorphic conditions (e.g., Elkins and Grove, 1990). The garnets vary in degree of preservation and can contain inclusions of plagioclase, potassium feldspar, sillimanite, zircon, monazite, or quartz. The majority of samples contain garnet that is entirely kelyphite (a fine-grained symplectite of acicular high-Al orthopyroxene + hercynite + glass), which presumably formed from rapid decompression in the host basalt (Padovani and Carter, 1977a; Dégi et al., 2010). Where fresh garnet is preserved, all have kelyphite rims. The rutile in these samples is unique in that some grains are blue in plane-polarized light (or shades of purple, gray, and green in plane polarized light depending on orientation) (appendix: Fig. S1).

TABLE 1. METAPELITIC XENOLITH SAMPLE INFORMATION

Sample	Locality	Location (°N/°W)	Major phases	Accessory phases	Dated
18KH1	Kilbourne	31.975°/106.967°	40% q, 20% gt, 20% sil, 15% kfs, 5% pl	rt, ap, zrn, ilm, mnz	zrn, mnz
18KH9	Kilbourne	31.975°/106.967°	45% q, 20% gt, 20% sil, 15% kfs, < 1% pl	rt, ap, zrn, ilm, mnz	zrn, mnz
117848-286	Potrillo	31.769°/106.999°	40% sil, 30% gt, 20% q, 5% kfs, 5% pl	rt, ap, zrn, ilm, mnz	zrn, mnz
117848-309	Kilbourne	31.975°/106.967°	45% q, 20% gt, 15% kfs, 15% sil, 5% pl	rt, ap, zrn, ilm, mnz	zrn

Notes: ap—apatite; gt—garnet; ilm—ilmenite; kfs—potassium feldspar; mnz—monazite; pl—plagioclase; q—quartz; rt—rutile; sil—sillimanite; zrn—zircon. Modal mineralogy estimated optically. Gt in sample 286 entirely kelyphitized.

Padovani and Carter (1977b) estimated that these xenoliths equilibrated between 750–1000 °C and 0.6–0.9 GPa, broadly consistent with the deep crust in the region. Furthermore, the seismic P-wave velocity of these xenoliths (calculated to be 6.4–8 km/s; 7 km/s avg.) overlaps the seismic velocity of the local deep crust (Reid et al., 1989; Hamblock et al., 2007).

Early geochronology data for metapelite xenoliths from the Potrillo volcanic field (e.g., Padovani and Reid, 1989; Reid et al., 1989) assumed that the sediments were transported to depth at ca. 1600 Ma, based on Sr, Nd, and Pb isochrons. Scherer et al. (1997) later dated mafic garnet granulites using the Lu-Hf isotope system and identified a major re-equilibration event at ca. 25 Ma, which is consistent with the onset of UHT metamorphism associated with the Rio Grande rift. Scherer et al. (1997) also documented a ca. 1400 Ma inherited zircon from one of these samples, as well as evidence of Cenozoic zircon growth during Rio Grande rift activity. Cipar et al. (2020) dated zircon and rutile from the metasedimentary xenoliths (including sample 18KH9 from this study), as well as calculated Zr-in-rutile and Ti-in-zircon temperatures. The zircon in that study showed Rio Grande rift ages and ultra-high temperatures (>800 °C), and the rutile yielded essentially zero Ma dates (corresponding to eruption <100 ka) and even higher temperatures (>930 °C).

Forty-seven metasedimentary xenoliths were examined in hand specimen and thin section. Of these, four samples were selected for detailed petrochronologic analyses based on the degree of preservation of the garnet, sample size, and representativeness in terms of mineralogy and texture.

E. Methods

1. Major- and Trace-Element Analyses of Whole Rocks and Major Minerals

Major-element data for rutile, garnet, and plagioclase were collected using the Cameca SX-100 electron probe micro-analyzer (EPMA) at the University of California, Santa Barbara (UCSB), California, USA. Transects of 2 μm spots, spaced 10–20 μm apart, were made across grains, using an accelerating voltage of 15 kV and a beam current of 10 nA. Zirconium contents in rutile were also determined via EPMA. Transects of 5 μm spots, spaced 10–20 μm apart, were done across grains, with an accelerating voltage of 20 kV and a beam current of 80 nA.

Trace elements in garnet were obtained with either the Agilent quadrupole inductively coupled plasma mass spectrometer (Q-ICP-MS) at UCSB or the Thermo Scientific Element2 sector field inductively coupled plasma mass spectrometer (SF-ICP-MS) at California State University, Northridge. The Agilent was coupled with a Teledyne CETAC Excimer Excite laser system and the Element2 with a Teledyne CETAC Analyte G2 laser system. 40 μm spots spaced 50 μm apart were used to make transects across grains along the same axes as analyzed by the EPMA. Manganese or Si concentrations from the EPMA were used as the internal element standards, NIST-612 was the calibrating standard, and BHVO-2G and GSD-1G were used as secondary standards (data in appendix).

Whole-rock major elements were measured on glass beads made by flux fusion of whole-rock powders and analyzed via a Thermo-ARL automated X-ray fluorescence spectrometer (XRF) at Washington State University, Washington, USA, which typically has an uncertainty better than 2% (2σ).

2. Thermobarometry

Whole-rock major-element data were used to construct pseudosections (Fig. 2) with the Perple_X software (Connolly, 1990) using the internally consistent thermodynamic database of Holland and Powell (2011). Calculations assumed an anhydrous composition based on the lack of hydrous phases in

the samples, as well as the extended time over which these samples experienced UHT metamorphism (Cipar et al., 2020).

Zr-in-rutile temperatures were calculated using the Ferry and Watson (2007) and Tomkins and Powell (2007) calibrations of this thermometer. These temperatures were used in

conjunction with the garnet-aluminosilicate-plagioclase-quartz equilibria (“GASP”) barometer to determine equilibration pressures and temperatures (using THERMOCALC v.3.3: Holland and Powell, 2011; White et al., 2014).

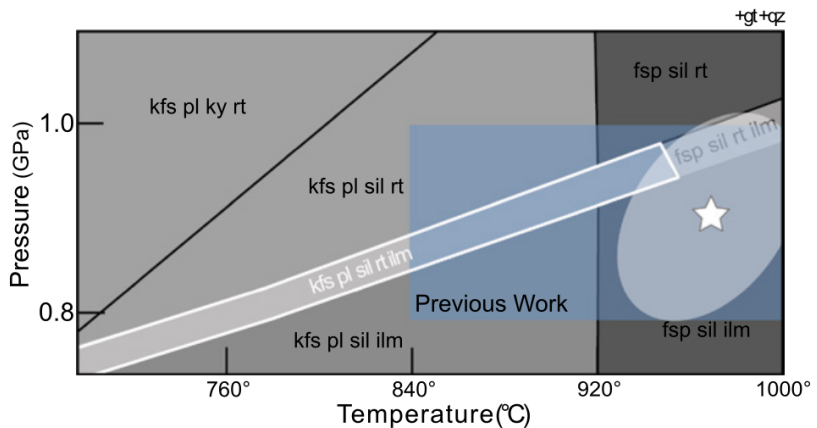


Figure 2. Pseudosection for sample 18KH1, which was collected from Kilbourne Hole: Field outlined in white represents the observed mineral assemblage for the samples studied here. White star shows pressure and temperature obtained from Zr-in-rutile thermometer and garnet-aluminosilicate-plagioclase-quartz equilibria barometer, and ellipse around star represents its uncertainty. Blue shading is the range of pressures and temperatures estimated by Padovani and Carter (1977b). All three samples for which X-ray fluorescence (XRF) analyses were obtained yielded the same pseudosection fields within uncertainty. fsp–feldspar, gt–garnet, ilm–ilmenite, kfs–K-feldspar, ky–kyanite, rt–rutile, pl–plagioclase, qz–quartz, sil–sillimanite

3. U-Pb Dating and Trace-Element Analyses of Accessory Phases

Zircon was separated from three xenoliths (18KH1, 18KH9, and 117848-286) and mounted in epoxy. (Zircon was also analyzed in thin section for another well-preserved, but small, sample, 17848-309.) Laser ablation spots were positioned based on cathodoluminescence images obtained via the FEI Quanta 400f scanning electron microscope at UCSB, and the images were also used to distinguish between oscillatory-zoned detrital igneous cores and generally featureless metamorphic zircon (Vavra, 1990; Shore and Fowler, 1996).

U-Pb and trace-element data in both detrital igneous and metamorphic zircon were collected simultaneously via laser ablation split-stream ICP-MS (LASS: Kylander-Clark et al., 2013) at UCSB, using either a Nu Plasma or Nu Plasma 3D and an Agilent 7700x quadrupole connected to a Teledyne CETAC Excimer Excite laser system. One sample (117848-309) was not large enough for mineral separates, and so zircon was analyzed in thin section in this case. Initially, 35 μm spots were used, but after determining that there was sufficient U, 12 μm spots were utilized to target the small oscillatory-zoned cores identified in the cathodoluminescence (CL) images (rarely larger than 15 μm in diameter). Metamorphic zircon rims (appearing feature-less in CL) were analyzed with 15 μm spots. Reference zircon 91500 was the primary U-Pb standard (1065 Ma: Wiedenbeck et al., 1995), and secondary standards included GJ1 (also the primary trace-element standard; 609 Ma: Jackson et al., 2004), Plešovice (337 Ma: Sláma et al., 2008), R33 (419 Ma: Black et al., 2004), Temora-2 (417 Ma: Black et al., 2004), and Mud Tank (732 Ma: Black and Gulson, 1978). Error propagation follows that outlined in Horstwood et al. (2016), including a typical uncertainty of 1%–2% (2σ) on individual $^{238}\text{U}/^{206}\text{Pb}$ and $^{207}\text{Pb}/^{206}\text{Pb}$ ratios (such

that the standards produce single populations). Secondary reference materials yielded the following concordia ages (2σ) within 2% of accepted values: GJ1 (597.8 ± 1.4 Ma), Plešovice (336.5 ± 1.2 Ma), R33 (415.7 ± 2.5 Ma), Temora-2 (417.3 ± 2.3 Ma), and Mud Tank (732.2 ± 3.9 Ma). All standard data are provided in the appendix. Silicon was used as the internal standard for trace-element determinations, assuming a concentration of 14.76%, and external uncertainties for trace-element measurements were generally $<10\%$ (2σ). Because of the smaller spot size necessary for targeting specific zircon zones, $\sim 30\%$ of the analyses yielded a 2σ uncertainty of 15% or greater on either the $^{206}\text{Pb}/^{238}\text{U}$ or $^{207}\text{Pb}/^{206}\text{Pb}$ ratios; these data generate little to no benefit in geologic interpretation and were thus omitted from further consideration.

Monazite from 18KH1, 18KH9, and 117848-286 was analyzed in thin section for Th-U-Pb ages and select trace elements via LASS. Analyses were made using 8 μm spots on grains in polished thin sections. Grains were identified through optical microscopy, as well as backscatter electron scans and Ce maps of the thin sections made using the EPMA at UCSB. Th-Pb dates were used for the Rio Grande-rift-age monazite (inset Fig. 3B) to get around the stronger effect of common Pb on younger U-Pb dates. Reference monazite 44069 (424 Ma: Aleinikoff et al., 2006) was the primary U-Th-Pb standard, and secondary standards included Stern (also the primary trace-element standard; 512 Ma: Palin et al., 2013), FC1 (55.7 Ma: Paces and Miller, 1993), and Trebilcock (273 Ma: Tomascak et al., 1996). Error propagation follows that outlined in Horstwood et al. (2016). Secondary reference materials yielded the following concordia ages (2σ) within 2% of accepted values: Stern (503.7 ± 2.2 Ma), FC1 (56.3 ± 0.7 Ma), and Trebilcock (277.1 ± 1.3 Ma). See the appendices for all standard data. Phosphorous was used as the internal standard for trace-

element determinations, assuming a concentration of 12.89%, which is the typical phosphorus concentration in monazite based on the idealized formula. Trace elements for secondary reference materials reproduce to better than 10% (2σ). Dates with $>15\%$ 2σ uncertainty ($\sim 30\%$ of the data set) were discarded. No common lead correction was applied to either the zircon or monazite data due to their highly discordant nature.

F. Results

Mineral and whole-rock compositions used for thermobarometry are reported in the appendices. Modal mineralogy is provided in Table 1. Pseudosection modeling using the major-element composition of 18KH1 indicates equilibration at 760–965 °C and 0.8–1.0 GPa in the garnet-sillimanite-quartz-potassium feldspar-plagioclase-rutile-ilmenite stability field (Fig. 2). Zr-in-rutile yielded an average temperature of 970 ± 50 °C, corresponding to a pressure of 0.9 ± 0.1 GPa using the GASP barometer (star with surrounding error ellipse in Fig. 2). These values are similar to P-T estimates from previous work (e.g., Padovani and Carter, 1977b; Hamblock et al., 2007; Cipar et al., 2020) and are consistent with equilibration under UHT conditions in the lower crust of the Rio Grande rift, as well as with rutile being open to Pb diffusion at the time of eruption (Cipar et al., 2020).

Over 1000 potential detrital igneous zircon cores were identified in CL images of the grains mounted in epoxy and then analyzed. However, the vast majority of these were

strongly discordant (see reasons for this in Discussion), and the 12 μm spot size used to

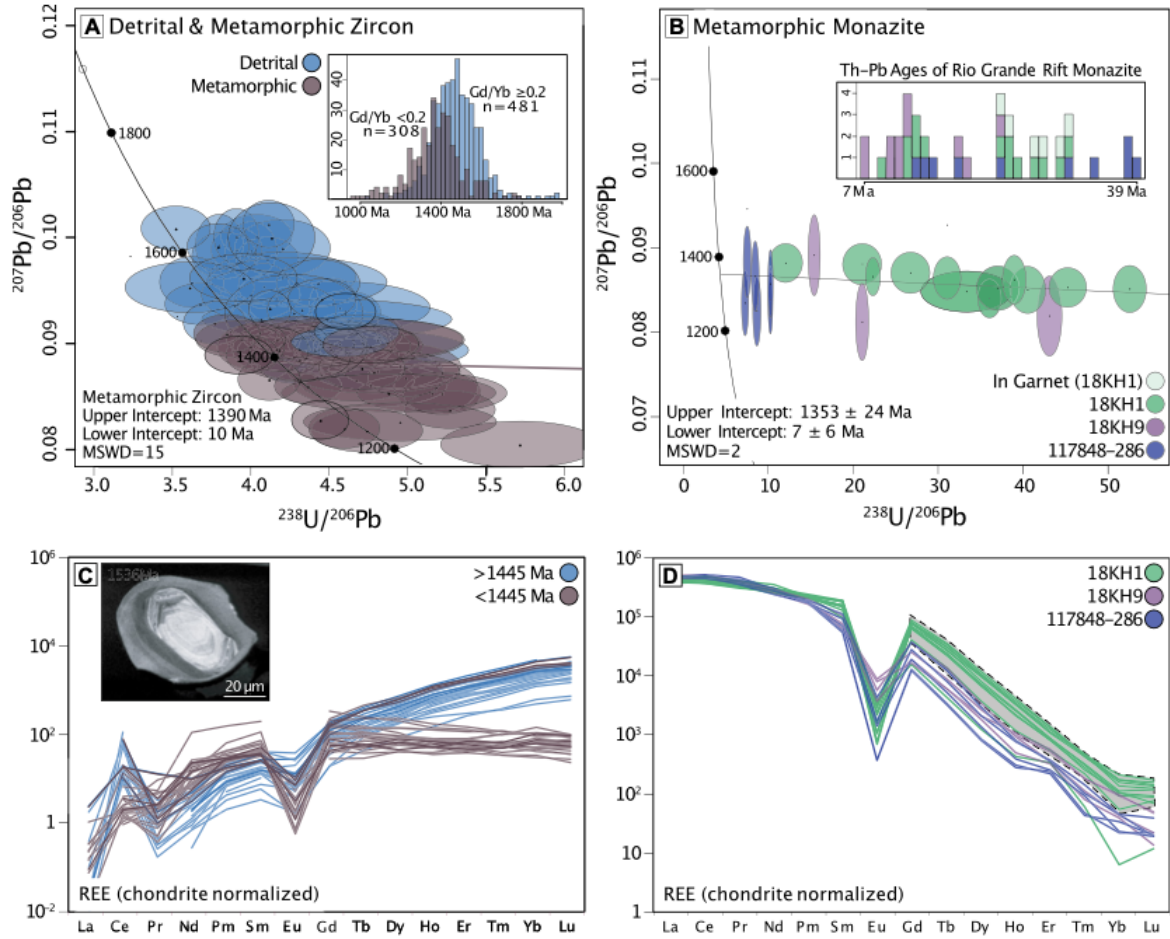


Figure 3. Geochronology results: (A) Tera-Wasserburg concordia plot of detrital zircon cores ($n = 45$) and metamorphic zircon ($n = 33$) from the four samples in Table 1 (as determined by cathodoluminescence [CL] images and Gd/Yb ratios) that fall within 15% of concordia and have uncertainties less than 15% (2σ). Inset shows upper intercept ages for each individual zircon with a $^{238}\text{U}/^{206}\text{Pb}$ ratio <100 classified solely on Gd/Yb ratio (data and associated uncertainties used to make this inset can be found in the appendix). Scale does not show younger ages or the eight analyses >2000 Ma. These data document a clear transition from detrital to metamorphic zircon production during the Picuris orogeny. MSWD—mean square of weighted deviates. **(B)** Tera-Wasserburg concordia plot of metamorphic monazite. All dates are for matrix monazites except for the six pale green data points for sample 18KH1 in the histogram, which are monazites within garnet. Error ellipses show 2σ total uncertainty for individual spot analyses. **(C)** Rare earth element (REE) plot for concordant zircon sorted by age, showing a clear transition at 1450 Ma from steep to flat heavy HREE. Inset cathodoluminescence image is an example of a zircon from sample 18KH1 with an oscillatory-zoned detrital core surrounded by a metamorphic rim. **(D)** REE plot of metamorphic monazite. Gray field highlights the higher HREE of the Rio Grande rift age monazite, indicating these younger grains are not simply the result of Pb loss from Proterozoic monazite and are instead a separate population.

collect both U-Pb and trace-element data yielded many analyses with extremely large uncertainties.

Approximately 1000 analyses of metamorphic zircon were collected from each of the samples shown in Table 1, except for 117848-309, analyses of which were limited by the number of zircon successfully targeted in thin section.

Detrital igneous zircon U-Pb dates (identified by oscillatory zoning in CL) range from ca. 1650–1450 Ma for concordant analyses (data in appendix) and up to ca. 2500 Ma for discordant analyses (assuming Pb-loss or recrystallization at 16 Ma for the latter; for calculated upper intercept ages and uncertainties based on a 16 Ma lower intercept, see appendix) (Fig. 3A). The discordant nature of the detrital igneous zircon, as well as their larger uncertainties, makes it difficult to identify a precise maximum depositional age. Featureless metamorphic zircon rims lie on a discordia line between Rio Grande rift dates (≤ 32 Ma: Morgan et al., 1986; Cipar et al., 2020) and concordant Proterozoic dates between ca. 1450 Ma and 1350 Ma, with eight concordant analyses < 1250 Ma (Fig. 3A).

Metamorphic monazite U-Pb and Th-Pb dates also record growth during these time periods (Fig. 3B), with an upper intercept of 1353 ± 24 Ma (2σ) and lower intercept of 7 ± 6 Ma (2σ), though monazite inclusions in garnet ($n = 6$) yield only Rio Grande rift ages, and

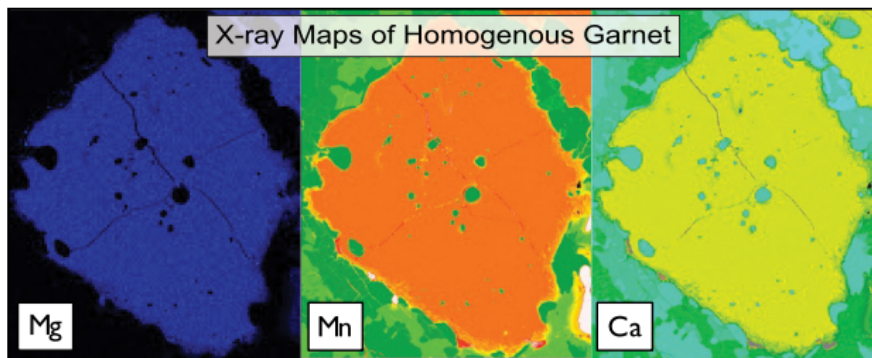


Figure 4. Representative X-ray maps of a chemically homogeneous garnet grain from sample 18KH1, a metapelitic xenolith collected at Kilbourne Hole.

the majority of matrix monazite are Rio Grande rift age (Fig. 3B inset). The Cenozoic monazite have higher heavy

rare earth elements (HREE) than the older grains (Fig. 3D), indicating that they are recrystallized grains and not simply Proterozoic monazite that experienced Pb loss. Both the featureless (in CL) metamorphic zircon and the monazite have negative Eu anomalies (Eu/Eu^*) and generally flat or depleted HREE, respectively (Figs. 3C and 3D).

Garnets are homogeneous with respect to their major- and trace-element compositions (Fig. 4), which precludes their use in determining the prograde path. Only a single grain (in sample 117848-309) is zoned in Zr and Hf (Fig. 5), corresponding to a distinct inclusion-rich core containing fibrous sillimanite, as well as quartz, and an inclusion-poor rim (no dateable phases were found within the core of this garnet). All other garnets in the samples have compositions similar to those of the inclusion-poor rim (including Zr and Hf content). Garnet data are provided in the appendix, and major-element compositions are plotted in Fig. S4.

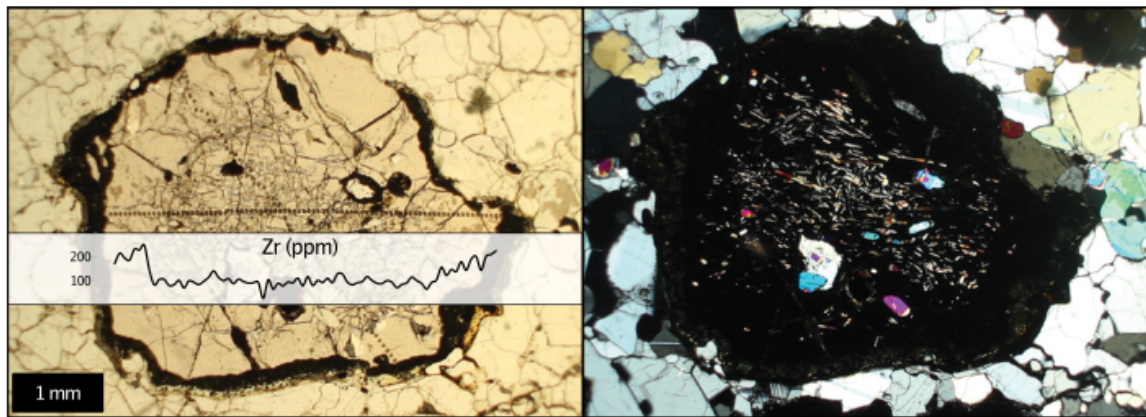


Figure 5. Texturally and chemically zoned garnet with a sillimanite inclusion-rich core indicating metamorphic conditions within the sillimanite field (sample 117848-309, a metapelitic xenolith collected at Kilbourne Hole). Only Zr and Hf preserve chemical zoning, with higher concentrations in the rim, while all other elements are unzoned. Garnet grew at $\sim 800^\circ\text{C}$ and rim at $\sim 1050^\circ\text{C}$ based on the Zr-in-garnet thermometer of Degeling (2002).

G. Discussion

1. Timing of Sedimentation and Metamorphism

Detrital igneous and metamorphic zircon were primarily distinguished using CL imaging, assuming oscillatory zoning reflects igneous cores and that featureless zircon grains/rims are metamorphic (see inset to Fig. 3C). Rare earth element patterns (in particular, Gd/Yb ratios) were also utilized to distinguish the two populations and generally correlate with grain structure: Oscillatory zoned (detrital igneous) zircon have low Gd/Yb, and featureless (metamorphic) zircon mostly have high Gd/Yb, indicating equilibration with garnet (e.g., Taylor et al., 2015). Where CL-distinct cores did not clearly show oscillatory zoning, the Gd/Yb ratio was used to determine whether they were igneous or metamorphic.

Most detrital igneous zircon show a large degree of discordancy (appendix: Fig. S3), which is likely due to one or more of the following: (1) Pb loss and/or recrystallization during initial and subsequent Proterozoic metamorphic events at ca. 1400 Ma (Picuris orogeny) or ca. 1200 Ma (Grenville orogeny), (2) Pb loss and/or recrystallization during Rio Grande rift UHT metamorphism (Cipar et al., 2020), and/or (3) mixing of detrital igneous and metamorphic domains in the analysis spot. The last of these was a particular problem due to the small size of most cores, and many analyses aimed at cores incorporated some rim material (this issue was mitigated in Iolite as much as possible, but may not have resulted in removal of all mixed analyses and cannot compensate for inheritance/incomplete recrystallization). The detrital igneous zircon plotted in Figure 3A represent those that are within 15% of concordia and have uncertainties less than 15% (2σ). These detrital cores mainly record ages corresponding to magmatism associated with the Mazatzal orogeny (>1600 Ma) and early stages of the Picuris orogeny (<1500 Ma). Based on the youngest

dates of detrital igneous zircon shown in Figure 3A, the sediments were deposited after 1500 Ma (during the earliest phase of the Picuris).

There is significant overlap in concordant dates of the oscillatory zoned cores and the featureless zircon grains and rims interpreted as metamorphic, which is potentially due to Pb loss from detrital zircon during transport to depth, mixing of these two domains during analysis, and/or uncertainties in individual dates. The inset in Figure 3A shows calculated/restored upper intercept ages for each individual zircon analysis (with $^{238}\text{U}/^{206}\text{Pb}$ ratios <100) assuming a lower intercept of 16 Ma and sorted by Gd/Yb ratio. We generated this inset image to show the probable upper limit of detrital zircon dates, rather than the transition from detrital igneous to metamorphic zircon. The lower intercept age was chosen because there is a clear discordia between the Mesoproterozoic and Rio Grande rift ages, and 16 Ma is the median age of Rio Grande rift activity (e.g., Cipar et al., 2020). With restored ages, the increasing influence of garnet on zircon composition can be seen overtime, though the concordant zircon analyses exhibit a significantly more robust relationship between age and Gd/Yb ratio (Fig. 3C).

Metamorphic zircon and monazite yield upper intercept dates primarily between 1450 and 1350 Ma (Fig. 3), which coincides with the Picuris orogeny and records peak metamorphism and therefore the transport of the sediments to depth. There is a clear transition from detrital zircon dates to metamorphic zircon growth at ca. 1450 Ma, which is indicative of a major change in tectonic setting at this time. The metamorphic monazite upper intercept of 1353 ± 24 Ma (discussed below) provides additional evidence for Picuris-aged transport of the samples to depth.

Due to the difficulty in dating very young monazite with the long-lived U-Pb systems, we present the Rio Grande rift-age monazite using their Th-Pb ages (Fig. 3B inset) and the older monazite using the U-Pb systems. The two populations have differing trace-element content, especially in the HREE, indicating that they are likely the product of recrystallization and not simply Pb loss. The ca. 1350 Ma upper intercept may represent mineral crystallization during retrograde metamorphism if it was accompanied by partial melting (e.g., Hacker et al., 2015). Though less likely, this upper intercept may instead show Pb loss during slow cooling, but that would require extended UHT conditions during the Mesoproterozoic, which are not documented in the literature and are difficult to assess using this data set due to the UHT metamorphism associated with the Rio Grande rift. Instead, the spread in metamorphic zircon dates and the monazite upper intercept at ca. 1350 Ma likely reflects an extended period of metamorphism, which is consistent with earlier research that documents protracted metamorphism during the Picuris orogeny (e.g., Aronoff et al., 2016; Bollen et al., 2022). There are also eight nearly concordant (<15% discordancy) metamorphic zircon with upper intercepts of ca. 1250–1000 Ma (out of a total of over 300 high Gd/Yb analyses). These are interpreted to reflect metamorphism associated with the Grenville orogeny, which occurred just to the south (Fig. 1) and would have likely affected the deep crust below the modern Potrillo volcanic field.

Although the recent UHT metamorphism makes interpretation of the Proterozoic history of these rocks challenging, the data presented here demonstrate that the sediments accumulated at the surface before eventually being transported to depth at ca. 1450 Ma, with metamorphism likely extending to ca. 1350 Ma (monazite upper intercept). This time

interval corresponds to the Picuris orogeny, which was most recently defined by Daniel et al. (2023) as 1500–1340 Ma in modern southwest USA.

2. P-T-Time Path of the Sediments and Tectonic Setting

Understanding how the metapelites were transported to the lower crust has important implications for interpreting the nature of the Picuris orogeny and its role in the assembly of Laurentia. Several processes may transport sediments to the lower crust: (1) forearc thrusting/underplating of oceanic sediments in a subduction zone (e.g., Pelona-Orocopia-Rand schists, western USA: Jacobson et al., 2011), (2) deep-seated thrust faulting on the continental/inboard side of an arc (retroarc) undergoing crustal shortening (e.g., North American cordillera: DeCelles et al., 2009; Chin et al., 2013), (3) deep-seated thrust faulting in a continent-continent collision zone after subduction ceases (e.g., Himalayas: Zhao et al., 1993), and (4) relamination, wherein buoyant diapirs of subducted continental crust and/or sediments rise through the mantle wedge to the base of the lower crust (e.g., Hacker et al., 2011; Kelemen and Behn, 2016). The petrochronologic data reported here can be used to distinguish between these different scenarios and provide important insight into the tectonic processes responsible for sediment emplacement at depth, as well as evidence for the tectonic setting at the time.

The transport of sediments to the lower crust could only have been achieved in a contractional setting via one of the scenarios previously discussed, thereby providing strong evidence for a major orogenic event at this time. This is consistent with previous work concluding that there was accretion of new crust south of the Nd line (Bickford et al., 2015), which divides older crust to the north and younger to the south, and has long been suspected as an important crustal boundary. The Picuris orogeny partly coincided with mantle-

dominated A-type magmatic activity (the extensive granite-rhyolite provinces) (Fig. 1; Bickford et al., 2015, and references therein). Previous work suggests that subduction during the Picuris ceased mid-1400s Ma (e.g., Daniel et al., 2013a; Karlstrom et al., 2016) and that slab breakoff or lower crustal foundering may have occurred, thereby potentially generating the A-type magmatic rocks from this period (Karlstrom et al., 2016). Subduction ceasing at mid-1400s Ma is consistent with the transition from igneous to metamorphic zircon production at ca. 1450 Ma and the onset of monazite growth at ca. 1350 Ma seen in the Potrillo metapelites.

The trace-element composition of accessory phases in the deep crustal metapelites provides clues about the nature of coexisting metamorphic minerals. HREE depletion in metamorphic monazite and zircon indicates growth in the presence of garnet (Rubatto et al., 2013; Taylor et al., 2015). Both phases also have negative Eu anomalies, indicating growth within the feldspar stability field (e.g., Rubatto et al., 2013; Dumond et al., 2015) and therefore at crustal depths, ruling out the higher pressures expected for subduction into the mantle followed by diapiric ascent and relamination (Hacker et al., 2011).

The rim of the single zoned garnet matches the inclusion-poor appearance and Zr and Hf contents of other garnet in these samples (Fig. 4). Thus, the rim is likely Rio Grande rift age, and the unique core may be a relict of the original prograde metamorphism that occurred during initial transport of the sediment to depth. If so, the abundant sillimanite in the core records a relatively warm metamorphic field gradient at the time of transport—warmer than expected in forearc subduction settings where kyanite is usually the stable aluminosilicate (see fig. 4 in Chapman, 2017). As there are no zircon or monazite inclusions in the garnet core, the hypothesis that this is a relict from the Proterozoic is based on its unique

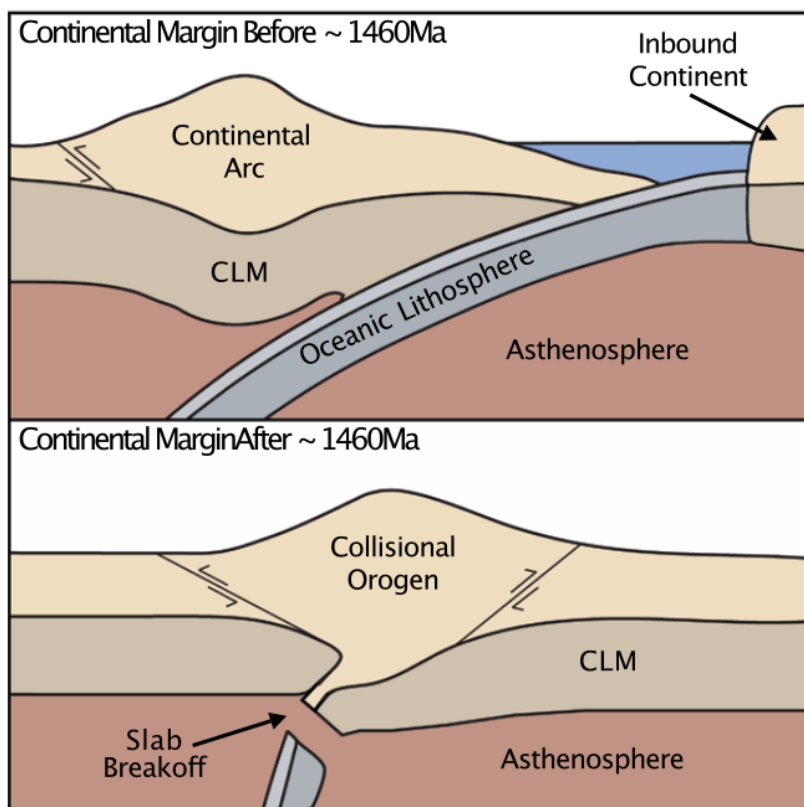


Figure 6. Simplified north-south cross section of the Picuris orogen along what was the Proterozoic southern margin of Laurentia: (A) Before ca. 1460 Ma. (B) Continent-continent collision after ca. 1460 Ma; this date lies within the time window of the transition from detrital/igneous to metamorphic zircon shown in Figure 3A. As the subduction polarity during the Picuris is unknown, it is not possible to determine north from south on the cross section. CLM—continental lithospheric mantle.

diffusion profile), this zoning likely reflects garnet rim growth. The Zr concentrations are compatible with growth of the core at 800 °C and the rim at 1050 °C (using the Zr-in-garnet thermometer of Degeling, 2002), with the latter temperature being consistent with the UHT metamorphism associated with the Rio Grande rift as determined by Zr-in-rutile thermometry (Cipar et al., 2020; this study) and the presence of ternary feldspars. The 800 °C domain may therefore represent a much earlier metamorphic history.

appearance (full of acicular sillimanite inclusions) and Zr and Hf concentrations, which differ from both the rim of the grain as well as the other garnet in the samples we examined. As Zr and Hf are some of the slowest diffusing elements in garnet (e.g., Chakraborty and Ganguly, 1991; Li et al., 2018; Bloch et al., 2020), and the core-rim boundary is relatively sharp (unlike a typical

These lines of evidence suggest transport of the sediments to depth in either a backarc setting or within a continent-continent collision zone. If subduction was no longer active along the margin after the mid-1400s Ma (e.g., Daniel et al., 2013a; Karlstrom et al., 2016), the transport of sediments to the lower crust after this period must have occurred within a continent-continent collision, wherein sediments transported to the base of the overriding plate are inserted into the lower continental crust after removal of the down-going slab through rollback, foundering, or breakoff (Fig. 6). This setting may have been similar to modern-day thick continent-continent collisions or to the hotter, thinner, and more ductile orogens that may be typical of the Meso- to Paleoproterozoic Earth (Spencer et al., 2021) and which could have involved the transport of sediments to the deep crust through burial and stacking of upper crustal “pop downs” (essentially inverted diapirs: Cagnard et al., 2006). Though this study’s data do not provide evidence for the polarity of subduction, the timing of this sequence of events fits with slab break-off typically occurring after subduction ceases and continent-continent collision begins (e.g., Davies and von Blanckenburg, 1995; van Hunen and Allen, 2011), as well as with the increasing influence of garnet on zircon trace-element contents (Figs. 3A and 3C). If subduction did indeed stop by ca. 1450 Ma, these metasediments were transported to depth within a collisional orogen.

The array of detrital zircon ages (those between 1600 and 1500 Ma) do not match any known sedimentary sequence in the southwest USA (e.g., Daniel et al., 2013a, 2013b; Amato et al., 2018; Amato, 2019). This may be evidence of a non-Laurentian provenance for these sediments, as has been suggested for other rocks in New Mexico (e.g., Piedra Lumbre Formation: Daniel et al., 2013b). In that case, these sediments may have been

transported to the southern margin of Laurentia by whatever terrane accreted there during the Picuris orogeny.

H. Conclusions

Detrital zircon dates from the metasedimentary xenoliths of the Potrillo volcanic field point to ca. 1650–1450 Ma source rocks, which are distinct from any sedimentary rocks known in New Mexico. The ages of metamorphic zircon and monazite show that the sediments were transported to the lower crust between 1450 and 1350 Ma, during the latter (collisional) part of the Picuris orogeny. The chronology, mineral trace-element data, textural and mineralogical clues, and the fact that it involved the transport of sediments to the deep crust indicate that the Picuris was, indeed, a contractional orogen and not the result of purely extensional or anorogenic activity near the southern margin of Proterozoic Laurentia. Furthermore, the occurrence of these lower crustal metapelites in south-central New Mexico provides corroboration of previous suggestions that the “Nd-line” may represent the location of a suture zone. By providing evidence for the tectonic setting at the time of sediment transport to depth, as well as the possible location of the Picuris suture zone, these data provide insight into an otherwise poorly understood yet major event in the Proterozoic assembly of North America.

III. A 3-billion-year history of magmatism, metamorphism, and metasomatism recorded by granulite-facies xenoliths from central Montana, USA

A. Abstract

Lower crustal xenoliths from the Missouri Breaks diatremes and Bearpaw Mountains volcanic field in Montana record a multi-billion-year geologic history lasting from the Neoproterozoic to the Cenozoic. Unusual kyanite-scapolite-bearing mafic granulites equilibrated at 1.8 GPa and 890 °C and 2.3 GPa and 1000 °C (67 and 85 km depth) and have compositions pointing to their origin as arc cumulates, while metapelitic granulites record peak conditions of 1.3 GPa and 775°C (48 km depth). Rutile from both mafic granulites and metapelites have U-Pb dates that document the eruption of the host rocks at ca. 46 Ma (Big Slide in the Missouri Breaks) and ca. 51 Ma (Robinson Ranch in the Bearpaw Mountains). Detrital igneous zircon in metapelites date back to the Archean, and metamorphic zircon and monazite record a major event beginning at 1800 Ma. Both zircon and monazite from a xenolith from Robinson Ranch also document an earlier metamorphic event at 2200–2000 Ma, likely related to burial/ metamorphism in a rift setting. Metapelites from Big Slide show a clear transition from detrital igneous zircon dates to metamorphic zircon and monazite growth around 1800 Ma, recording arc magmatism and subsequent continent-continent collision during the Great Falls orogeny, supporting suggestions that the Great Falls tectonic zone is a suture between the Wyoming craton and Medicine Hat block. U-Pb and trace-element depth profiles of zircon and monazite record metasomatism of the lower crust

during the Laramide orogeny at ~60 Ma, supporting recent suggestions for Farallon slab fluid infiltration during the orogeny.

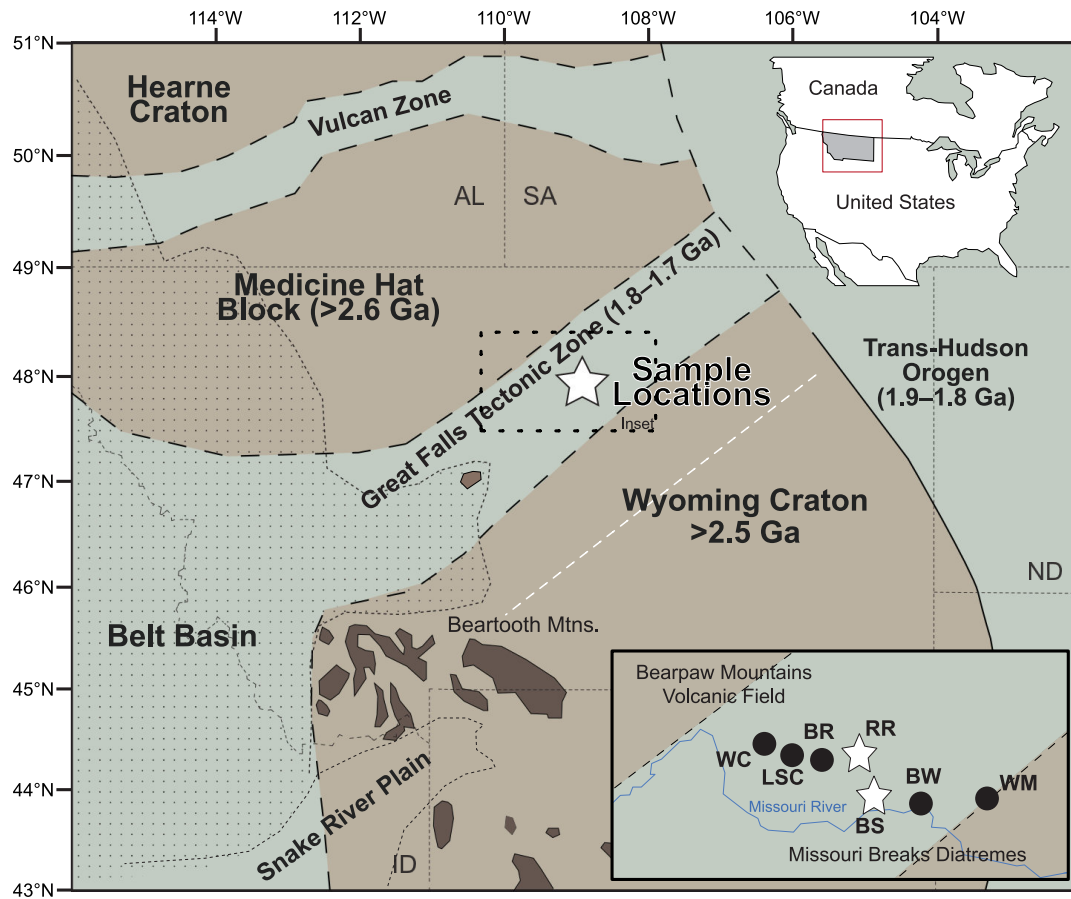


Figure 1. Map of region: Archean cratons shown in light brown, and younger regions shown in green. Darker brown areas represent surface exposures of Precambrian basement. State and province borders marked by dashed lines. Dotted region represents the Belt Basin. Inset box shows sample locations and other diatremes in this area of the Great Falls tectonic zone. Dashed white line is the northern boundary of the Wyoming craton proposed by Bedrosian & Frost (2022). Abbreviations for xenolith localities are as follows: BR = Brewer Ranch, BS = Big Slide, BW = Bullwhacker Diatreme, LSC = Little Sand Creek, RR = Robinson Ranch, WC = Warrick Creek, WM = Williams Kimberlite. Modified from previous studies (Ross 1991; Condie 1992; Vogl et al. 2004; Foster et al. 2012; Gifford et al. 2014).

B. Introduction

The ancient core of North America is largely composed of Archean blocks assembled during the Paleoproterozoic (Hoffman 1988; Mueller et al. 2002; Whitmeyer and Karlstrom 2007; Chapman et al. 2020). Two such crustal blocks, the Wyoming craton and Medicine Hat block, experienced ~8 My of alkalic volcanism during the Laramide orogeny beginning ca. 50 Ma, including the intrusion of the Missouri Breaks diatremes and Bearpaw Mountains volcanic field (Marvin et al. 1980; Hearn 1989; O'Brien et al. 1991; Carlson and Irving 1994; Facer et al. 2009). Many of the volcanic centers host xenoliths from both the crust and mantle, which can provide important insight into the history of the deep lithosphere.

The Big Slide and Robinson Ranch volcanic centers are located within the Missouri Breaks diatremes and the Bearpaw Mountains volcanic field, respectively. Both are within the larger Great Falls tectonic zone (Fig. 1), which is bounded by the Medicine Hat block to the north and the Wyoming craton to the south, though the exact relationship between the two and the nature of this boundary have been debated. Garnet- and aluminosilicate-bearing xenoliths, including both metapelites and metaigneous rocks, provide an opportunity to document the geologic history of this region, determine how and which geologic events have shaped these ancient cratons over time, and constrain the ways in which this history has manifested in the lower crust.

This study utilizes geochronologic, geochemical, and petrologic data for garnet-aluminosilicate bearing xenoliths from central Montana, USA, to address the following questions: (1) Is there evidence of subduction and an ocean basin between the Wyoming craton and Medicine hat block? (2) Can the end of subduction and beginning of continent-continent collision be seen and its timing determined? (3) What are the protoliths of the

xenoliths, and where did they originate? (4) At what depth did the samples equilibrate, and, in the case of the metapelites, how were they transported from the surface to depth? (5) What happened to the the metapelites following emplacement in the lower crust? (6) When did the Robinson Ranch and Big Slide magmas erupt?

C. Geologic Background

The core of North America/ Laurentia (Fig. 1) is composed of a series of Archean crustal blocks—the Wyoming, Superior, Hearne, Sask, Slave, and Rae cratons and the Medicine Hat block—that collided during a succession of orogens in the Proterozoic (Hoffman 1988; Bowring et al. 1989; Mueller et al. 2002; Chamberlain et al. 2003; Downes 2004; Gifford et al. 2014; Chapman et al. 2020). The earliest of these are some of the oldest well-documented orogens in the world, such as the Wopmay and Trans-Hudson orogens at ca. 2000-1800 Ma (Hoffman 1988; Bickford et al. 1990; Hildebrand et al. 2010; Weller and St-Onge 2017). Much of the remainder of modern North America was accreted along the southern margin of Laurentia during subsequent major orogenic events, including the Yavapai (ca. 1800 Ma), Picuris (ca. 1400 Ma), and Grenville (ca. 1000 Ma) orogenies (Hoffman 1988; Karlstrom et al. 2001; Whitmeyer and Karlstrom 2007; Bartholomew and Hatcher 2010; Daniel et al. 2013; Mulder et al. 2017; Ringwood et al. 2024).

Between 2000–1800 Ma, the eastern margins of both the Medicine Hat block and the Wyoming craton collided with and were sutured to the Superior craton along their eastern boundaries during the Trans-Hudson orogeny (Bickford et al. 1990; Lucas et al. 1994; Corrigan et al. 2009; Weller and St-Onge 2017). The Trans-Hudson may also be responsible for the suturing of the Hearne craton to the Medicine Hat block along its northern border.

This boundary is referred to as the Vulcan zone (Eaton et al. 1999) and is demarcated by subsurface E-W trending gravity and magnetic anomalies (Kanasewich et al. 1969; Hoffman 1988; Eaton et al. 1999). The Vulcan zone has been interpreted as both a failed rift and a collisional boundary (Kanasewich et al. 1969; Clowes and Kanasewich 1972; Hoffman 1988), though research comparing the two scenarios has provided more evidence for the latter (Eaton et al. 1999; Hope and Eaton 2002). The Medicine Hat block is less well studied than the Wyoming craton due to the fact that it is largely obscured by younger sedimentary sequences, and the boundary between the two, the Great Falls tectonic zone, is poorly exposed at the surface (Gifford et al. 2018, 2020). The Great Falls tectonic zone was active in the Paleoproterozoic until ca. 1700 Ma and may have been repeatedly reactivated, potentially even into the Cenozoic (O'Neill and Lopez 1985). Some view it as an intraplate shear zone and consider the Vulcan zone to the north of the Medicine Hat block to be the primary collisional boundary in the area (Boerner et al. 1998; Eaton et al. 1999), while others have concluded that the Great Falls tectonic zone is a collisional boundary between the Medicine Hat block and the Wyoming craton and refer to the event that formed it as the “Great Falls orogeny” (Mueller et al. 2002; LaDouceur 2019; Gifford et al. 2020). The poor surface exposure within the Great Falls tectonic zone and the Medicine Hat block have hindered consensus in this debate, though the collisional orogen hypothesis has come to dominate in recent years (e.g., LaDouceur 2019; Gifford et al. 2020). Furthermore, recent research based on geophysical data has pushed the southern boundary of the Great Falls tectonic zone (and thus the northern boundary of the Wyoming craton) further to the south (Bedrosian and Frost 2023) (white dashed line in Fig. 1).

Surface exposures of the Great Falls tectonic zone are rare (Fig. 1), but those that do exist contain 1900–1800 Ma granitic rocks (Holm and Schneider 2002; Mueller et al. 2002; Foster et al. 2012), documenting a probable continental arc setting (Mueller et al. 2002). Lower crustal xenoliths provide additional insight into the Proterozoic history of the zone. Granulite-facies xenoliths from Little Sand Creek, Eagle Buttes, Robinson Ranch, Brewer Ranch, and the Homestead Kimberlite contain igneous zircon formed at ca. 1850 Ma (Bolhar et al. 2007; Barnhart et al. 2012; Thakurdin et al. 2019a, b). Thakurdin et al. (2019a) demonstrated that some ~1870 Ma xenoliths from the Bearpaw Mountains have arc-like trace-element patterns (e.g., enrichment in large-ion lithophile and depletion of high field strength elements), suggesting that subduction was occurring in the region until at least 1870 Ma.

Metamorphic zircon and monazite provide possible evidence for the collision of the Medicine Hat block and Wyoming craton at ca. 1770 Ma. (Barnhart et al. 2012; Thakurdin et al. 2019a, b). Some drill core samples (Villeneuve et al. 1993), as well as outcrops from the Priest River and Clearwater complexes in Idaho (Wang et al. 2022), record ca. 1850 Ma magmatism and ca. 1770 metamorphism. Archean rocks found in the Tobacco Root and Highland mountains similarly document granulite-facies metamorphism and partial melting at ca. 1770 Ma (Cheney et al. 2004; Harms et al. 2004; Mueller et al. 2004). Based on the inferred arc setting and subsequent metamorphic event, many of the studies cited herein infer the closure of an ocean basin before metamorphism began. Mantle xenoliths carried in alkaline magmas erupted within the Great Falls tectonic zone also reveal a history of metasomatism in the deep lithosphere at ca. 1800 Ma. This major collisional event has been referred to by multiple names in the literature, including the Great Falls orogeny (Gifford et

al. 2018; Mogk et al. 2020), the Big Sky orogeny (Brady et al. 2004; Harms and Baldwin 2020), and the Trans-Montana orogeny (Sims et al. 2004). Herein, it will be referred to as the Great Falls orogeny.

The collisional component of the Great Falls orogeny is generally considered to have peaked at 1770 Ma and ended by 1700 Ma (Bolhar et al. 2007; Gifford et al. 2020; Thakurdin et al. 2019a, b; Wang et al. 2022). Despite this, some deep-seated xenoliths show evidence of continued metamorphism in the lower crust, including monazite as young as 1300 Ma (Barnhart et al. 2012), garnet dates of 1502 ± 13 Ma (Lu-Hf) and 1363 ± 12 Ma (Sm-Nd) (Scherer et al. 2000), and metamorphic zircon dates from ~ 1700 – 1500 Ma (Scherer et al. 2000; Thakurdin et al. 2019b). These post-Great Falls orogeny ages are likely related to metamorphism and fluid flow associated with the beginning of rifting in the Belt Basin (Chamberlain et al. 2003; Barnhart et al. 2012). This extensional regime generated subsidence and basin formation in western Montana and resulted in the deposition of the massive (>15 km thick) Mesoproterozoic Belt Supergroup (Lonn et al. 2020 and references therein). Early rift activity is also recorded in episodic, primarily tholeiitic, magmatism at 1470–1400 Ma (Anderson and Davis 1995; Evans et al. 2000) and ca. 1370 Ma (Doughty and Chamberlain 1996) in Idaho, Montana, and British Columbia. Some refer to this failed rift as the Montana Aulacogen (e.g., Nelson 1995; Shepard 1993).

Western Montana again experienced magmatic activity in the Neoproterozoic. U-Pb zircon dates of 780–740 Ma from mafic dikes (Baadsgaard and Mueller 1973; Park et al. 1995; Harlan et al. 1997, 2003, 2008; Mackinder et al. 2019) record the emplacement of the Gunbarrel Large Igneous Province in Idaho, Montana, and Wyoming at ca. 778, followed by

extension-related magmatism likely connected to the initial fragmentation of Rodinia (Harlan et al. 2003, 2008; Mackinder et al. 2019; Macdonald et al. 2023).

The breakup of Rodinia lasted until ca. 600 Ma (e.g., Li 2011; Macdonald et al. 2023 and references therein), and subsidence in Montana continued into the Cambrian with the development of a passive margin (Norwood 1965; Yonkee et al. 2014; Link et al. 2017). New subduction along the western margin of North America began in the Permian (Ward 1991) and continued sporadically. By 80 Ma, the subduction angle of the Farallon plate began to decrease, initiating the Laramide orogeny (ca. 75–30 Ma), which was characterized by flat slab subduction and extensive contractional deformation in the western United States and Canada, potentially related to the subduction of an oceanic plateau (Liu et al. 2010). The transition to flat slab subduction resulted in the shut-off of the Sierra Nevada arc and migration of magmatism inboard along a north-south axis running through modern Utah, Nevada, Montana, Idaho, and Alberta (Dickinson and Snyder 1978; Bird 1998; Chapman et al. 2020).

The Wyoming craton, Great Falls tectonic zone, and Medicine Hat block experienced deformation associated with the Laramide orogeny, which was accompanied by alkalic magmatism in Montana (Marvin et al. 1980; Hearn 1989; O'Brien et al. 1991; Carlson and Irving 1994; Facer et al. 2009). Unlike many Archean cratons, which are typically >200 km thick (Gung et al. 2003; Lee et al. 2011 and references therein), the Wyoming craton was extensively deformed and thinned during the Cenozoic (Gilbert 2012; Dave and Li 2016) and has crustal and lithospheric thicknesses of ~45 and ~150 km, respectively (Schmandt et al. 2015). Multiple studies indicate that thinning likely occurred due to the Laramide orogeny, wherein the shallow angle of subduction caused an influx of fluids into the lower

lithosphere. Subduction-related metasomatism has long been associated with the alkalic nature of the Eocene magmatism in the Wyoming craton and surrounding areas (Volmer et al. 1984; O'Brien et al. 1991; MacDonald et al. 1992). More recently, Apen et al. (2024) documented multiple metasomatic events in mafic lower crustal xenoliths from central Montana, which occurred between 62 and 52 Ma and, in some cases, predate the age of the host magmas. They proposed that eclogitization of the Farallon slab led to its foundering, generating an influx of asthenosphere from which the alkalic volcanism derived via melting of the ancient, metasomatized mantle lithosphere.

The Eocene volcanic centers in central Montana erupted between ca. 54–46 Ma (Hearn, 1989; Hearn and McGee, 1983; Marvin et al. 1980), though Robinson Ranch and Big Slide diatreme have not previously been dated. The broader area is referred to as the Central Montana Alkalic Province and is composed of a variety of alkalic volcanic and intrusive rocks, including kimberlites, minettes, and latites, as well as Phanerozoic sedimentary sequences (Hearn et al. 1989). The magmatic rocks host a variety of xenolith types, and a significant amount of work has been carried out on the mafic and ultramafic lithologies (Downes et al. 2004; Bolhar et al. 2007; Facer et al. 2009; Barnhart et al. 2012; Blackburn et al. 2012; Thakurdin et al. 2019a, b). However, granulite-facies metapelitic xenoliths and kyanite-bearing mafic xenoliths are uncommon among these volcanic centers and have yet to be studied. The metapelites provide an opportunity to understand the processes by which sediments are transported to lower crustal depths and become part of stable lower continental crust, while the kyanite-bearing mafic xenoliths offer insights into formation of these relatively rare rock types. Moreover, metapelites contain a multitude of minerals useful for petrochronology, including zircon, monazite, rutile, apatite, and garnet, and thus

allow for further study of the region's tectonic history, especially that of the Great Falls tectonic zone.

D. Samples

Robinson Ranch diatreme in the Bearpaw Mountains and Big Slide in the Missouri Breaks diatremes erupted through the Great Falls tectonic zone (Fig. 1). Xenoliths from these localities are typically quite small, with the largest no more than 5-8 cm along their longest axis. The samples described herein fall into two groups: mafic pyroxene-kyanite-scapolite-bearing rocks and metapelitic granulites. Information about the samples is provided in Table 1, and selected photomicrographs are shown in Fig. 2.

Sample	Locality	Location (°N/°W)	Major phases	Accessory phases
<u>Metapelites</u>				
20BS3	Big Slide	47.830°, 109.268°	kfs, grt, ky, pl, qz, bt, rt	mnz, zrn, ap
20BS4	Big Slide	47.830°, 109.268°	grt, kfs, ky, pl, qz, bt, rt	mnz, zrn, ap
Rob-2	Robinson Ranch	48.032°, 109.324°	qz, grt, kfs, sil, pl, bt, rt	mnz, zrn, ap
<u>Mafic</u>				
20BS19	Big Slide	47.830°, 109.268°	pl, grt, cpx, ky, scp, amp, czo	ap, rt
H67-14-5	Big Slide	47.830°, 109.268°	pl, grt, scp, cpx, ky, czo, qz, rt	ap, brt, spl

Table 1. Sample Information: Mineral abbreviations: amp = amphibole, ap = apatite, brt = barite, bt = biotite, cpx = clinopyroxene, czo = clinozoisite, grt = garnet, kfs = potassium feldspar, ky = kyanite, mnz = monazite, pl = plagioclase, qz = quartz, rt = rutile, sil = sillimanite, scp = scapolite, spl = spinel, zrn = zircon. Major phases are listed in order of abundance (from greatest to least). Rutile is included as an accessory phase for sample 20BS19, because it is not present as macroscopic grains.

Mafic samples include 20BS19 and H67-14-5, which are both from Big Slide. Sample 20BS19 is largely composed of garnet, clinopyroxene, plagioclase, kyanite, silvialite, clinozoisite, and amphibole. Both garnet and plagioclase are crosscut by unusual intergrowths of elongate, radial kyanite and clinopyroxene, the latter of which rims the amphibole in 20BS19. H67-14-5 has two major domains: The first contains garnet,

scapolite, clinopyroxene, and plagioclase, while the second is composed of garnet, scapolite,

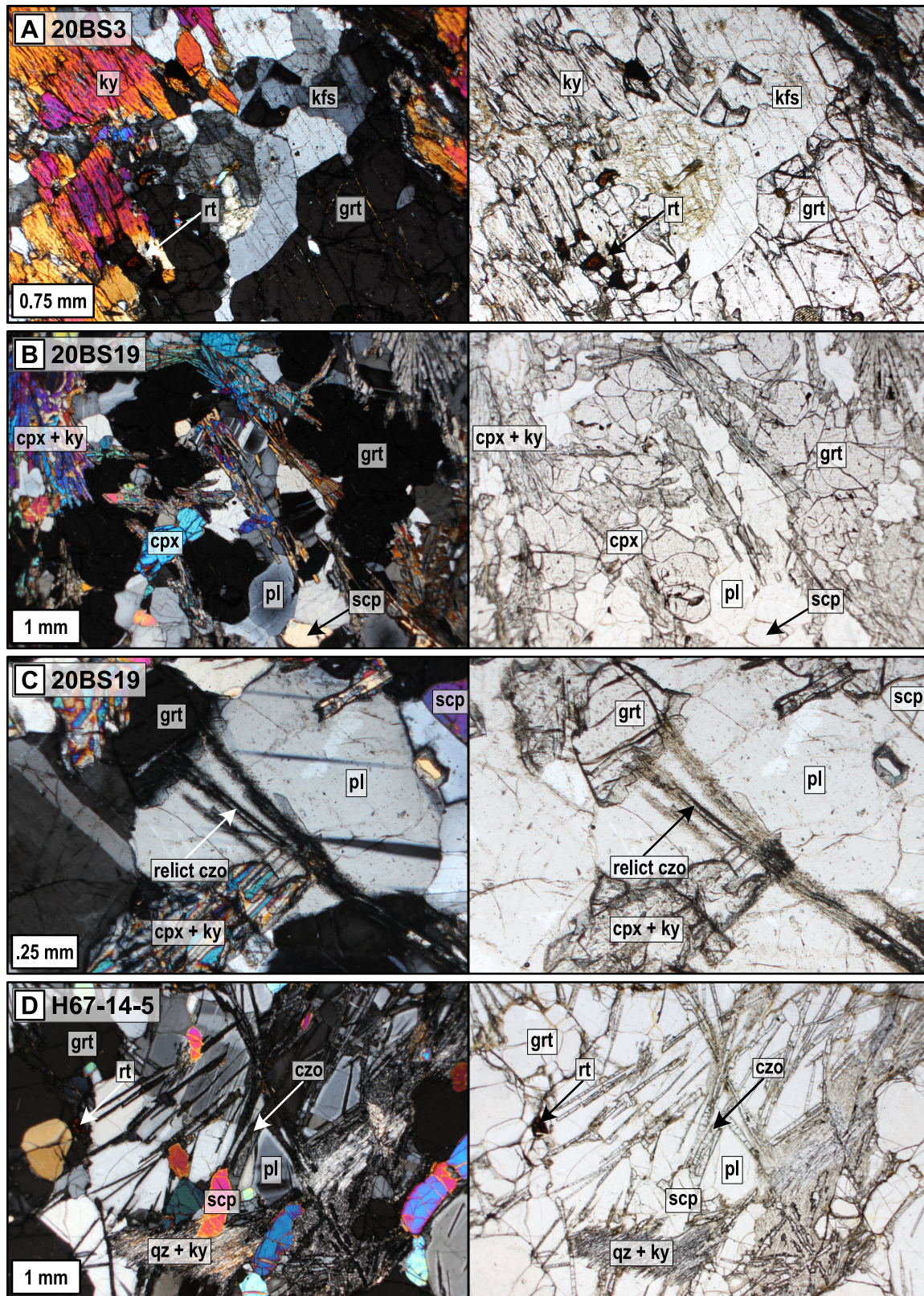


Figure 2. Photomicrographs of selected samples, with crossed polar (XPL) images on the left and plane-polarized light (PPL) images on the right: (a) metapelite 20BS3, (b) mafic granulite 20BS19, (c) higher magnification of 20BS19 showing relict clinozoisite, (d) mafic granulite H67-14-5. Mineral abbreviations: cpx = clinopyroxene, czo = clinozoisite, grt = garnet, kfs = potassium feldspar, ky = kyanite, pl = plagioclase, qz = quartz, rt = rutile, scp = scapolite. Additional photomicrographs are provided in the supplement.

and plagioclase, as well as elongate clinozoisite and fine-grained areas of quartz and kyanite.

Both contain apatite and <10- μ m diameter rutile, though H67-14-5 also has larger rutile visible to the naked eye, as well as minor amounts of barite and spinel, primarily in altered areas.

The metapelitic granulites include samples 20BS3 and 20BS4 from Big Slide and sample ROB-2 from Robinson Ranch. All three consist of garnet, plagioclase, potassium feldspar, quartz, aluminosilicate, and biotite, though ROB-2 is more quartz-rich and is the sole sample with sillimanite, while 20BS3 and 20BS4 both contain kyanite and are mineralogically indistinguishable from each other in hand specimen. The same aluminosilicates are found in both the matrix and as inclusions in garnet. All three contain rutile, monazite, apatite, and zircon.

E. Sampling and Analytical Methods

1. Modal Mineralogy

Modal mineralogy was estimated using back-scatter electron images (BSE) and elemental maps of thin sections obtained via the Cameca SX-100 electron probe microanalyzer (EPMA) at the University of California, Santa Barbara (UCSB). These images were converted to grey-scale, and ImageJ software (Rasband 1997) was used to

calculate percentages of each mineral in the BSE images and elemental maps to quantitatively estimate mineral modes.

2. Whole-Rock Major and Trace-Element Analyses

Whole-rock major- and selected trace-elements were determined by X-ray fluorescence (XRF) at Franklin & Marshall College. Major elements were measured on homogenized glass beads, while trace-elements were analyzed on compressed briquettes of homogenized powder, using a Malvern PANalytical Inc. Zetium X-ray fluorescence spectrometer, which generally yields uncertainties less than 2% (2σ) (Rousseau 2001).

Whole-rock major-element compositions were also calculated using the mineral major-element data collected via EPMA and modal abundances calculated using ImageJ (Rasband 1997). In the case of sample H67-14-5, only the calculated whole-rock estimate is available due to a lack of sufficient whole-rock material for XRF analysis. Calculated whole-rock compositions lie within a few percentage points of the XRF results.

Whole-rock trace-element data were obtained by analyzing the glasses produced for XRF via laser ablation quadrupole inductively coupled plasma mass spectrometry (LA-Q-ICP-MS) at UCSB using an Agilent 8900 Q-ICP-MS connected to a CETAC Teledyne Excimer Excite laser system. Forty-second line analyses were used with an 85- μm spot diameter and the following laser settings: $\sim 2 \text{ J/cm}^2$ and a fixed dosage of 50 at a 10-Hz repetition rate. Si content (as determined by XRF) was used as the internal element standard, and trace-element data were processed using the Iolite4 software (Paton et al. 2010, 2011). Trace-elements obtained via LA-Q-ICP-MS typically yield results for secondary reference materials within 10% of accepted values (full reference material data in supplement).

3. Mineral Major- and Trace-Element Analyses

Major-element data for amphibole, biotite, garnet, clinopyroxene, clinozoisite, plagioclase, potassium feldspar, and scapolite were measured using the EPMA at UCSB and Probe Software, Inc. Probe for EPMA data acquisition and analysis software. Data were acquired as transects (typically 10 spots per grain) across grains of interest using an accelerating voltage of 15 kV, an electron beam defocused to a 2- μm diameter, and a 10-nA current. Zirconium contents in rutile were also determined using transects of 5- μm spots (10 spots per grain), an accelerating voltage of 20 kV, and a beam current of 80 nA.

Trace-element data for garnet were collected via LA-Q-ICP-MS using an Agilent 7700x Q-ICP-MS connected to a CETAC Teledyne Excimer Excite laser system at UCSB. Laser settings used were $\sim 1 \text{ J/cm}^2$ for 180 pulses and a 10-Hz repetition rate. Transects of 150- μm spots were done across grains. Si content (as determined by EPMA) was used as the internal element standard, and trace-element data were processed using the Iolite4 software (Paton et al. 2010, 2011). Trace-elements obtained via LA-Q-ICP-MS typically yield results for secondary reference materials within 10% of accepted values (full reference material data in supplement).

4. Thermobarometry

The Perple_X software package (Connolly 1990) was used to construct pseudosections illustrating the stable mineral assemblages at different pressure-temperature conditions for metapelites 20BS3, 20BS4, and ROB-2. These calculations used the internally consistent thermodynamic database of Holland and Powell (2011). Perple_X was also used to determine the predicted garnet volume % at different pressure-temperature conditions in the metapelitic samples. Pseudosections were not created for the two mafic granulites 20BS19

and H67-14-5, because no activity model for S-bearing scapolite (silvialite) exists. Instead, thermobarometers using the garnet and plagioclase rim compositions were compared to and plotted over pressure-temperature diagrams showing the phase stability field of S-bearing scapolite as determined by Goldsmith (1976). A variety of thermobarometers were utilized for the mafic samples, including the garnet-clinopyroxene thermometers of Ellis & Green (1979), Powell (1985), and Krogh (1988); the garnet-clinopyroxene barometer of Mukhopadhyay (1991); the clinopyroxene-plagioclase-quartz barometer of McCarthy & Patiño Douce (1998); the garnet-aluminosilicate-plagioclase-quartz equilibria (“GASP”) barometer of Holdaway (2001); the garnet-clinopyroxene-plagioclase-quartz barometer of Eckert et al. (1991); and the hornblende-plagioclase barometer of Molina et al. (2015).

Zr-in-rutile equilibration temperatures were calculated using the Ferry & Watson (2007) calibration of this thermometer for the metapelitic samples 20BS3, 20BS4, and ROB-2. The garnet-biotite thermometer of Holdaway (2000) and the GASP barometer of Holdaway (2001) were also utilized for these samples and, along with the Zr-in-rutile results, plotted on the pseudosections. The average garnet compositions were used for thermobarometry calculations, as there was no clear core-rim structure in the metapelitic samples. The calibration of Watson et al. (2006) was utilized to calculate Ti-in-Zircon temperatures.

5. LASS Spot Analyses of Accessory Phases

U-Pb and trace-element data were simultaneously collected for zircon, monazite, and rutile via laser ablation split-stream ICP-MS (LASS: Kylander-Clark et al. 2013) at UCSB,

using either a Nu Plasma or Nu Plasma 3D multi-collector ICP-MS (MC-ICP-MS) and an Agilent 7700x Q-ICP-MS connected to a CETAC Teledyne Excimer Excite laser system.

Zircon was separated from two metapelitic xenoliths, 20BS3 and ROB-2, and mounted in epoxy. Metapelite 20BS4 also contains zircon, but there was less material for separates, and so zircon grains were analyzed in-situ in a polished thin section. Cathodoluminescence (CL) images of zircon grains were obtained with a KE Developments ‘Centaurus’ Panchromatic CL detector mounted on a FEI Quanta 400f scanning electron microscope (SEM) at UCSB. CL images were used to determine suitable positions for laser ablation spots and to distinguish between oscillatory-zoned detrital igneous cores and homogeneous

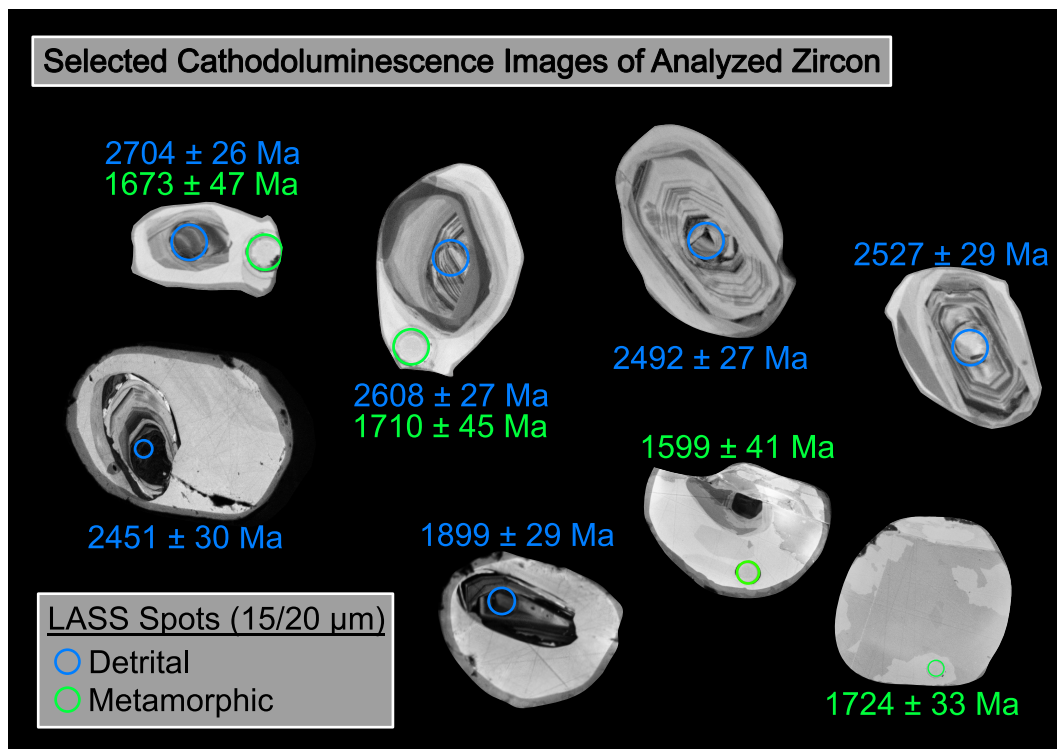


Figure 3. Selected cathodoluminescence images of zircon: Spot sizes shown are 15 or 20 microns in diameter. Detrital cores are indicated by hollow blue circles and show oscillatory zoning, while metamorphic domains are shown with hollow green circles and are generally featureless or patchy in appearance. $^{207}\text{Pb}/^{206}\text{Pb}$ dates shown.

or patchy metamorphic zircon (Vavra 1990; Shore and Fowler 1996). Representative CL images of zircon are shown in Fig. 3.

U-Pb and trace-element data were simultaneously collected for both detrital igneous cores and metamorphic zircon via LASS. Igneous cores (rarely >20 μm in diameter) were analyzed using 15- μm spots, while 20- μm spots were used for metamorphic zircon domains as size permitted. Laser settings were $\sim 1 \text{ J/cm}^2$ for 60 pulses at a 5-Hz repetition rate.

Matrix-matched reference materials were used to quantify the accuracy and precision of analyses. Reference zircon 91500 was used as the primary standard for U-Pb analyses (1065 Ma: Wiedenbeck et al. 1995), and secondary standards included GJ1 (also the primary TE standard; 609 Ma: Jackson et al. 2004), Plešovice (337 Ma: Sláma et al. 2008), OG1 (3465 Ma: Stern et al. 2009), and Mud Tank (732 Ma: Black and Gulson 1978). Secondary reference materials yielded the following concordia ages that, with 2σ uncertainty, are within 2% of the accepted values (all data in supplement): GJ1 ($599.3 \pm 1.8 \text{ Ma}$), Plešovice ($336.3 \pm 1.5 \text{ Ma}$), OG1 ($3472.7 \pm 7.4 \text{ Ma}$), and Mud Tank ($717.1 \pm 12.8 \text{ Ma}$). Silicon in zircon was used as an internal standard for determining trace-element concentrations, assuming a stoichiometric concentration of 14.8% for unknowns. Trace-element concentrations for secondary reference materials using LASS are typically within 10% of accepted values.

LASS was used to collect U-Th-Pb and trace-element data for monazite in thin section for the metapelites. Grains were analyzed using 8- μm spots and identified using optical microscopy and Ce element maps created via EPMA. Laser settings were $\sim 1 \text{ J/cm}^2$ for 45 pulses at a 3-Hz repetition rate. Reference monazite Stern (512 Ma: Palin et al. 2013) served as the primary U-Pb and trace-element reference material, and secondary reference materials

included 44069 (424 Ma: Aleinikoff et al. 2006), FC1 (55.7 Ma: Paces and Miller 1993), Trebilcock (273 Ma: Tomascak et al. 1996), and Manangotry (555 Ma, 558 Ma: Horstwood et al. 2003). Secondary reference materials yielded the following concordia ages that, with 2σ uncertainty, are within 2% of accepted values (all data in supplement): 44069 (429.8 ± 4.5 Ma), FC1 (55.9 ± 2.0 Ma), Trebilcock (267.4 ± 2.0 Ma), and Manangotry (558.3 ± 8.0). Phosphorous was used as the internal standard for trace-elements, assuming an idealized stoichiometric concentration of 12.9%. Trace-element concentrations for secondary reference materials using LASS are typically within 10% of accepted values.

Rutile from samples 20BS3, H67-14-5, and ROB-2 were analyzed in-situ using polished thin sections for U-Pb and trace-elements via LASS (while 20BS19 does contain rutile, the grains are $<10 \mu\text{m}$ and are considered too small to analyze). Spot sizes were $50 \mu\text{m}$, and grains were identified using optical microscopy. Laser settings were $\sim 2 \text{ J/cm}^2$ for 60 pulses at a 5-Hz repetition rate. Reference rutile Kragero (1087 Ma: Kellett et al. 2018) served as the primary U-Pb reference material, and secondary reference materials included 9826J (382 Ma: Kylander-Clark et al. 2008), LJ04 (498 Ma: Apen et al. 2020), and RMJG (1750 Ma: Zhang et al. 2020) for U-Pb. BHVO-2G (Jochum et al. 2005) served as the primary trace-element reference material. Secondary reference materials yielded the following concordia ages that, with 2σ uncertainty, are within 3% of accepted values (all data in supplement): 9826J (388.3 ± 4.4 Ma), LJ04 (498.2 ± 5.4 Ma), and RMJG (1792.4 ± 9.6 Ma). Titanium was used as the internal standard for trace-elements, assuming a stoichiometric concentration of 59.9%.

Raw U-Th-Pb and trace-element data were processed using the Iolite4 software (Paton et al. 2010, 2011), which allowed for data correction for elemental fractionation, the drift of

instrument signals over time, and the background/baseline signal. Error propagation follows that outlined in Horstwood et al. (2016). Data were plotted using the software IsoplotR (Vermeesch 2018). All data, including for reference materials, are reported in the supplementary materials.

6. LASS Depth Profiling of Accessory Phases

Zircon from 20BS3 and ROB-2 and monazite from 20BS3, 20BS4, and ROB-2 were mounted in epoxy and analyzed for U-Pb dates and trace-elements via LASS depth profiling on unpolished mineral surfaces in order to target thin (μm -scale) changes near rims. Others have used this technique to resolve both narrow zones of Pb loss and rim growth that would be otherwise impossible to analyze using conventional laser ablation techniques (e.g., Smye and Stockli 2014; Stearns et al. 2016).

Zircon and monazite grains were analyzed at UCSB. Before analysis, grain surfaces were cleaned using low power ablation runs on spots significantly larger than the analysis spots (50 μm for zircon and 20 μm for monazite). The laser settings for the lower power runs were $\sim 0.2 \text{ J/cm}^2$ for 10 pulses at a 10-Hz repetition rate.

Zircon grains were analyzed using 20- μm laser spots placed on the unpolished mineral surfaces and laser settings of $\sim 1 \text{ J/cm}^2$ for 80 pulses at a 3-Hz repetition rate. Reference zircon 91500, GJ1, and Plešovice were analyzed along with unknowns. Uncertainties were quantified using the method outlined in Cottle et al. (2012) wherein the relative standard deviation of the U-Th-Pb isotopic ratios of the primary reference material are added in quadrature with the counting statistics uncertainty for each individual analysis.

Monazite grains were analyzed using 8- μm laser spots placed on the unpolished mineral surfaces and laser settings of $\sim 1 \text{ J/cm}^2$ for 60 pulses at a 3-Hz repetition rate. Reference

monazites 44069, Stern, FC1, Trebilcock, and Manangotry were analyzed along with the unknowns (all data in supplement). Uncertainties were also quantified via the method described in Cottle et al. (2012).

F. Results

1. Geochemistry and Thermobarometry

Minerals in all samples show some major-element zoning, though it is more pronounced in the mafic samples, H67-14-5 and 20BS19. Garnet cores in all samples have lower Ca and higher Mg and Fe concentrations, while plagioclase cores in the mafic samples have higher Ca and lower Na concentrations. Garnet trace-elements are also zoned in the metapelites, 20BS3, 20BS4, and ROB-2, with cores containing more Y + HREE than the rims. Trace-elements in garnet in the mafic samples have minimal zoning, though their major element-zoning corresponds to a clearer core-rim structure (Fig. 4). Zoning in the metapelites' garnet grains is more convex or concave, as one might expect of a diffusion profile. Representative major-element compositions for minerals are shown in Table 2, and all mineral compositional data are included in the supplementary material.

Whole-rock compositions for all samples are shown in Table 3 and were used to generate pseudosections for the metapelites (20BS3, 20BS4, and ROB-2) (Fig. 5). The stability fields for the assemblages in these samples (garnet, potassium feldspar, kyanite/sillimanite, plagioclase, quartz, biotite, and rutile) are large. In the case of the kyanite-bearing samples (20BS3 and 20BS4), the whole stability field on the pseudosection spans from approximately 300 to 850 °C and 0.5 to 1.8 GPa. The entire stability field for

ROB-2, which contains sillimanite, is between approximately 540 to 920°C and 0.45 to 1.1 GPa.

Individual thermobarometers applied to the metapelites return P-T conditions that are in

Sample	SiO ₂	Al ₂ O ₃	TiO ₂	MnO	Na ₂ O	MgO	FeO	F	CaO	SO	Cl	Cr ₂ O ₃	K ₂ O
METAPELITES													
<u>20BS3</u>													
Garnet	39.0	22.1	--	0.299	0.008	9.75	26.1		2.34			0.029	0.002
Plagioclase	64.2	22.2	0.003	--	9.30	0.0006	0.040		3.24			--	0.783
Gt Inclusion	63.7	23.2	0.004	--	8.67	0.013	0.138		3.70			--	0.828
K-Feldspar	63.6	19.3	--	--	2.46	--	0.019		0.057			0.001	12.8
Gt Inclusion	63.6	19.3	--	--	2.23	0.026	0.126		0.125			0.0002	12.9
Biotite	37.9	16.2	3.90	0.004	0.140	16.7	7.94		0.003			0.314	10.0
Gt Inclusion	38.5	16.5	3.26	0.007	0.169	17.8	7.92		0.021			0.066	9.86
<u>20BS4</u>													
Garnet	39.2	22.1	--	0.301	0.014	10.2	25.7		2.21			0.026	0.007
Plagioclase	63.3	22.7	0.001	--	9.04	0.006	0.035		3.31			--	0.870
Gt Inclusion	63.5	23.5	0.016	--	8.64	0.007	0.06		3.96			--	0.866
K-Feldspar	63.9	19.2	--	--	2.37	--	0.023		0.074			--	13.3
Gt Inclusion	64.3	19.3	--	--	2.25	--	0.072		0.120			--	13.4
Biotite	38.1	16.1	3.21	0.008	0.205	18.2	7.11		0.010			0.050	9.86
Gt Inclusion	37.9	15.8	2.95	0.007	0.180	18.2	7.44		0.009			0.060	9.87
<u>Rob-2</u>													
Garnet	39.1	22.9	0.037	0.283	0.023	11.5	24.9		1.47			0.053	0.022
Plagioclase	62.0	24.5	0.007	--	7.93	0.023	0.045		5.28			0.0004	0.944
K-Feldspar	63.5	19.3	--	0.001	2.54	--	0.018		0.106			0.003	12.9
Biotite	38.2	16.2	4.46	0.005	0.286	17.6	8.23		0.010			0.093	9.70
Gt Inclusion	37.9	16.5	3.82	0.011	0.315	18.5	7.10		0.014			0.177	9.68
MAFIC													
<u>20BS19</u>													
Garnet	40.0	23.2	0.049	0.209	0.017	11.6	12.1		12.3			0.019	0.013
Cores	40.1	23.1	0.054	0.206	0.020	12.4	12.1		11.3			0.020	0.015
Rims	39.7	22.8	0.027	0.225	0.009	8.31	11.9		16.6			0.020	0.009
Plagioclase	55.7	27.8	--	--	5.97	0.013	0.021		9.18			0.001	0.374
Cores	55.0	28.6	0.001	--	5.58	0.013	0.011		10.0			0.002	0.288
Rims	57.4	26.3	0.0002	--	6.85	0.088	0.043		7.27			0.009	0.536
Clinopyroxene	49.4	13.0	0.468	0.017	2.69	11.0	2.96	0.225	19.2	0.004	0.006	0.016	0.276
Scapolite	46.1	26.8	0.0005	0.003	3.90	0.016	0.041	0.018	16.3	2.10	0.030	0.002	0.116
Amphibole	40.9	19.4	0.746	0.007	2.09	15.0	3.66	2.18	11.6	0.003	0.014	0.021	3.04
<u>H67-14-5</u>													
Garnet	39.0	22.6	0.122	0.177	0.036	8.25	15.9		13.3			0.007	0.009
Cores	39.1	22.5	0.127	0.184	0.036	8.58	16.3		12.7			0.009	0.009
Rims	39.2	22.9	0.065	0.170	0.022	7.85	14.8		14.8			0.001	0.004
Plagioclase	55.6	28.6	0.003	0.001	5.74	0.009	0.006		9.93			--	0.296
Cores	55.6	28.6	0.003	0.001	5.73	0.009	0.005		9.94			--	0.296
Rims	58.2	26.5	--	0.006	6.71	0.030	0.089		7.70			0.005	0.676
Gt Inclusion	55.5	27.8	0.005	0.001	6.18	0.004	0.152		9.15			0.001	0.351
Clinopyroxene	50.6	10.5	0.705	0.006	2.51	11.3	3.28	0.044	20.4	0.004	0.008	0.008	0.007
Scapolite	45.8	27.3	0.001	--	3.74	0.026	0.040	--	16.9	2.86	0.011	--	0.094

Table 2. Representative mineral compositions (oxide %): If there are cores, rims, and/ or inclusions within a given mineral, those values are listed below the average value for said mineral. Dashes indicate content was below detection limit.

Sample	SiO ₂	TiO ₂	Al ₂ O ₃	FeO	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	LOI
<u>Metapelites</u>											
20BS3	44.9	1.02	28.9	14.4	0.19	5.44	1.61	1.13	2.65	0.06	0.53
20BS4	47.6	0.98	23.9	14.2	0.18	5.52	1.92	1.40	3.92	0.09	0.67
Rob-2	61.6	0.92	19.8	8.26	0.08	4.05	1.22	1.16	2.46	0.03	0.65
<u>Mafic</u>											
20BS19	45.5	0.10	25.2	7.35	0.20	8.54	10.6	1.75	0.30	0.03	2.62
H67-14-5*	45.8	0.09	28.4	5.28	0.06	3.33	12.1	2.66	0.12		

Table 3. Whole-rock sample compositions (oxide %): Data collected via XRF for samples with enough material to make homogenized glass beads. *H67-14-5's composition was estimated using mineral compositions obtained via EPMA.

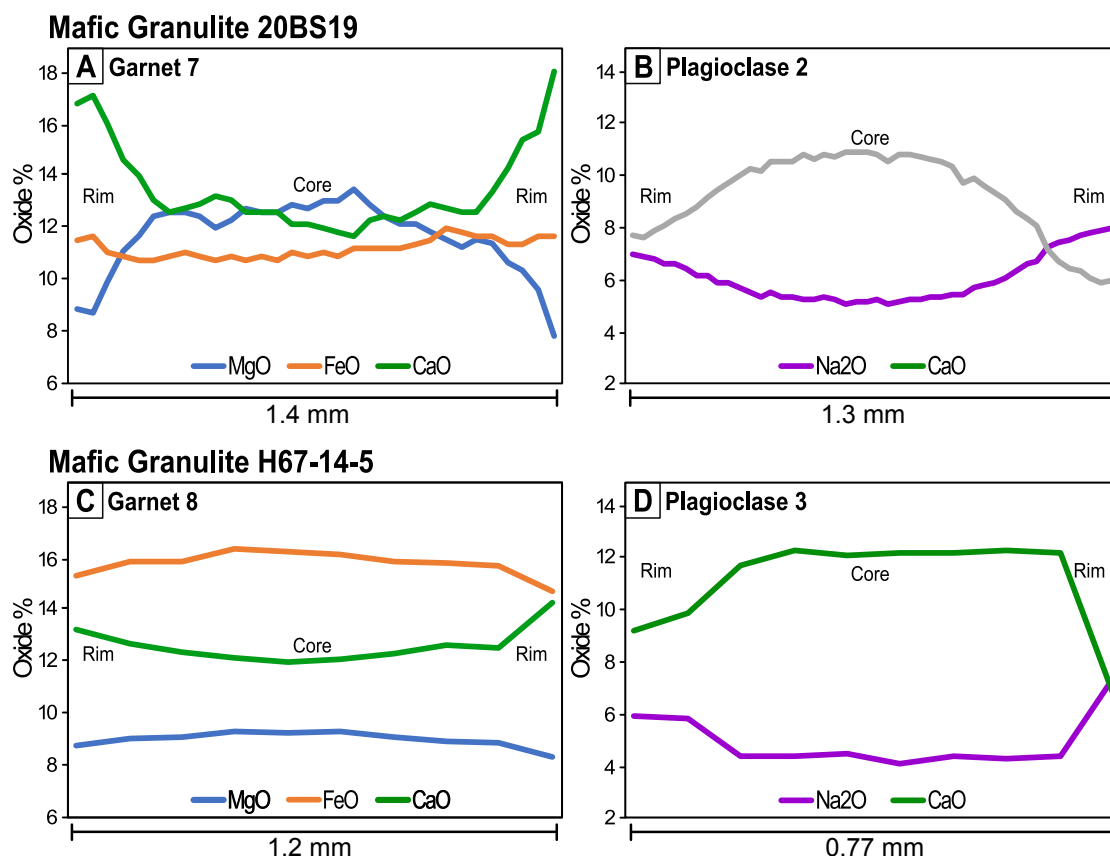


Figure 4. Representative compositional transects across garnet and plagioclase grains for mafic granulites, 20BS19 (a,b) and H67-14-5 (c,d): Oxide weight % shown for MgO, FeO, and CaO in garnet and Na₂O and CaO for plagioclase. Analyses were conducted via EPMA.

general agreement with the pseudosections (Fig. 5). The Zr-in-rutile thermometer and GASP barometer yielded the following average pressures and temperatures: 750 °C and 1.3 GPa for 20BS3, 775 °C and 1.3 GPa for 20BS4, and 790 °C and 0.9 GPa for ROB-2. Typical

uncertainties for these thermobarometers are 50 °C and 0.1 GPa. The garnet-biotite Fe-Mg exchange thermometer yielded temperatures approximately 200 to 300 °C lower than the average Zr-in-rutile temperature. The intersection of the GASP barometer and Zr-in-rutile thermometer lines fall within the appropriate pseudosection fields for all three samples. exchange thermometer yielded temperatures approximately 200 to 300 °C lower than the average Zr-in-rutile temperature. The intersection of the GASP barometer and Zr-in-rutile thermometer lines fall within the appropriate pseudosection fields for all three samples.

Thermobarometry calculations for the mafic granulites, 20BS19 and H67-14-5, produced eclogite-facies peak pressure and temperature conditions (Fig. 6). Sample H67-14-5 yielded an approximate temperature and pressure of 890 °C and 1.8 GPa, respectively, based on the

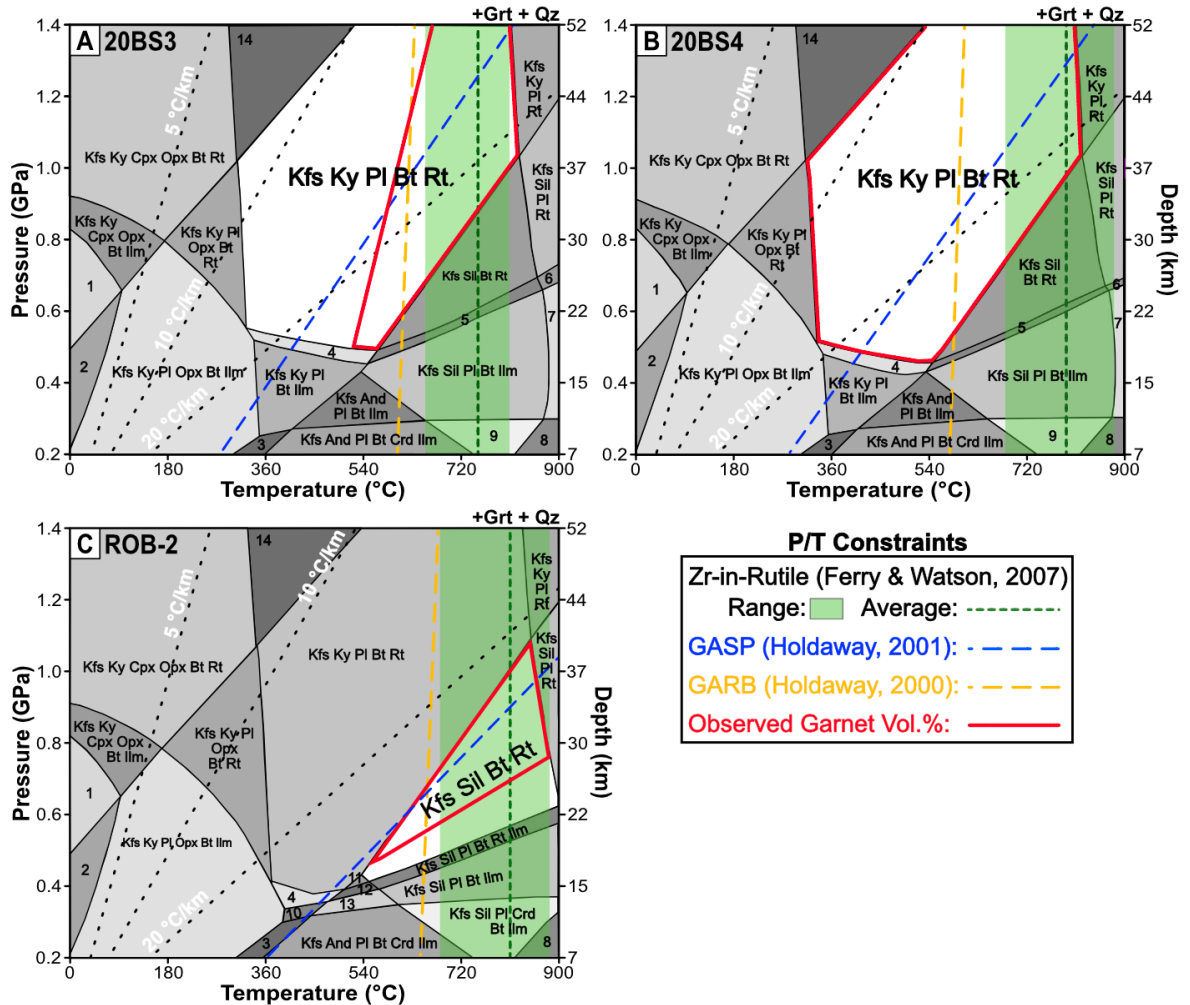


Figure 5. Pseudosections for metapelites 20BS3 (a), 20BS4 (b), and ROB-2 (c). The lines representing the GASP barometer (blue) and GARB thermometer (gold) are shown on the pseudosections, along with the range of temperatures calculated using the Zr-in-rutile thermometer (green field) and the line representing the average Zr-in-rutile temperature (black). White fields are those with the observed mineral assemblage. Field highlighted by a thick red line encompasses observed garnet volume % for each sample. All fields of the pseudosections include garnet and quartz. Geothermal gradients shown with black dashed lines and white text. Subduction zone metamorphism occurs between 5 and 10 °C/km. Mineral abbreviations are as follows: and = andalusite, bt = biotite, crd = cordierite, ilm = ilmenite, kfs = potassium feldspar, ky = kyanite, mu = muscovite, pl = plagioclase, rt = rutile, and sil = sillimanite. Fields denoted by numbers are as follows: 1 = Kfs Ky Cpx Opx Amp Ilm, 2 = Kfs Ky Pl Opx Amp Ilm, 3 = Kfs Ky Pl Bt Crd Ilm, 4 = Kfs Ky Pl Bt Rt Ilm, 5 = Kfs Sil Pl Bt Rt Ilm, 6 = Kfs Sil Rt Ilm, 7 = Kfs Sil Pl Ilm, 8 = Kfs Sil Pl Crd Ilm, 9 = Kfs Sil Pl Crd Bt Ilm, 10 = Kfs Ky Pl Bt Ilm, 11 = Kfs And Pl Bt Rt, 12 = Kfs And Pl Bt Rt Ilm, 13 = Kfs And Pl Bt Ilm, 14 = Kfs Ky Cpx Bt Rt. Note that the intersection of the GASP and Zr-in-rutile equilibria fall within the observed mineral assemblage field and provide the best estimate of peak metamorphic conditions.

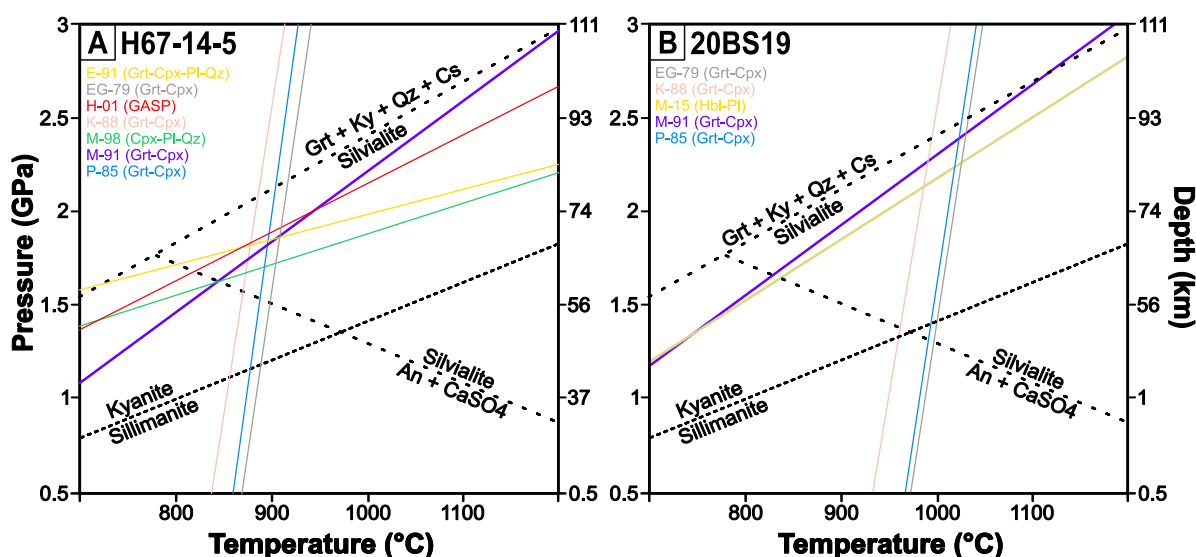


Figure 6. Pressure-temperature diagrams for mafic granulites, H67-14-5 (a) and 20BS19 (b): Various thermobarometers are plotted, with the silicalite stability field and the transition between the kyanite and sillimanite stability fields shown by dashed lines. The abbreviations for the individual thermobarometers are as follows: E-91 = Eckert *et al.* (1991), EG-79 = Ellis & Green (1979), H-01 = Holdaway (2001), K-88 = Krogh (1988), M-98 = McCarthy *et al.* (1998), M-15 = Molina (2015), M-91 = Mukhopadhyay (1991), P-85 = Powell (1985).

garnet-clinopyroxene thermobarometers, GASP barometer, garnet-clinopyroxene-

plagioclase-quartz barometer, and clinopyroxene-plagioclase-quartz barometer. Sample

20BS19 yielded even higher-grade conditions at approximately 1000 °C and 2.3 GPa as determined by the garnet-clinopyroxene thermobarometers and the amphibole-plagioclase barometer. These calculated temperatures and pressures are in general agreement for all thermobarometers and within the conditions at which S-bearing scapolite (silvialite) is stable (Fig. 6).

2. U-Th-Pb Dates and Trace-Elements of Accessory Phases

The U-Pb zircon dates (Fig. 7) are generally concordant, with the vast majority falling within 10% of concordia. $^{207}\text{Pb}/^{206}\text{Pb}$ dates for detrital igneous cores, as distinguished by oscillatory zoning in CL (Fig. 3) and HREE enrichment, range from ca. 2700–1800 Ma for 20BS3 ($n = 74$), 1950–1800 Ma for 20BS4 ($n = 10$), and 3300–2200 for ROB-2 ($n = 174$). In all three samples, metamorphic zircon exhibit more depleted HREE, lack oscillatory zoning in CL, and have negative Eu anomalies. $^{207}\text{Pb}/^{206}\text{Pb}$ dates for metamorphic zircon mostly fall between 1800 and 1600 Ma, though there are analyses as young as 1350 Ma for 20BS3 and 1580 Ma for 20BS4. ROB-2 also contains an older population of metamorphic zircon between 2200 and 2000 Ma. All three metapelites return a subset of spot analyses that appear to be a mix of igneous and metamorphic zones, which was determined based on REE patterns, CL images, Th/U ratio, and age. For samples 20BS3 and 20BS4, the mixed analyses have steep HREE patterns corresponding to the detrital igneous zircon, but appear homogenous (or have partially faded zoning) in CL images. These may be analyses that mixed igneous and metamorphic material in the laser spot and/ or zircon that lost lead or recrystallized due to metamorphism. ROB-2 has a similar mixed population of zircon with steep HREE, but also has a sub-population of mixed analyses with flat HREE patterns that are similar to those of the older metamorphic population in that sample. Metamorphic zircon

from the metapelites exhibit peak Ti-in-Zircon temperatures of 800–850 °C and decreasing temperatures after 1800 Ma.

Monazite U-Pb dates (Fig. 8) are consistent with those of the metamorphic zircon, with the majority of dates from all three samples falling between 1800 and 1600 Ma. ROB-2 also contains monazite with 2200–2000 Ma ages, and samples 20BS3 and ROB-2 yielded concordant dates as young as 1400 Ma, while the former includes discordant analyses

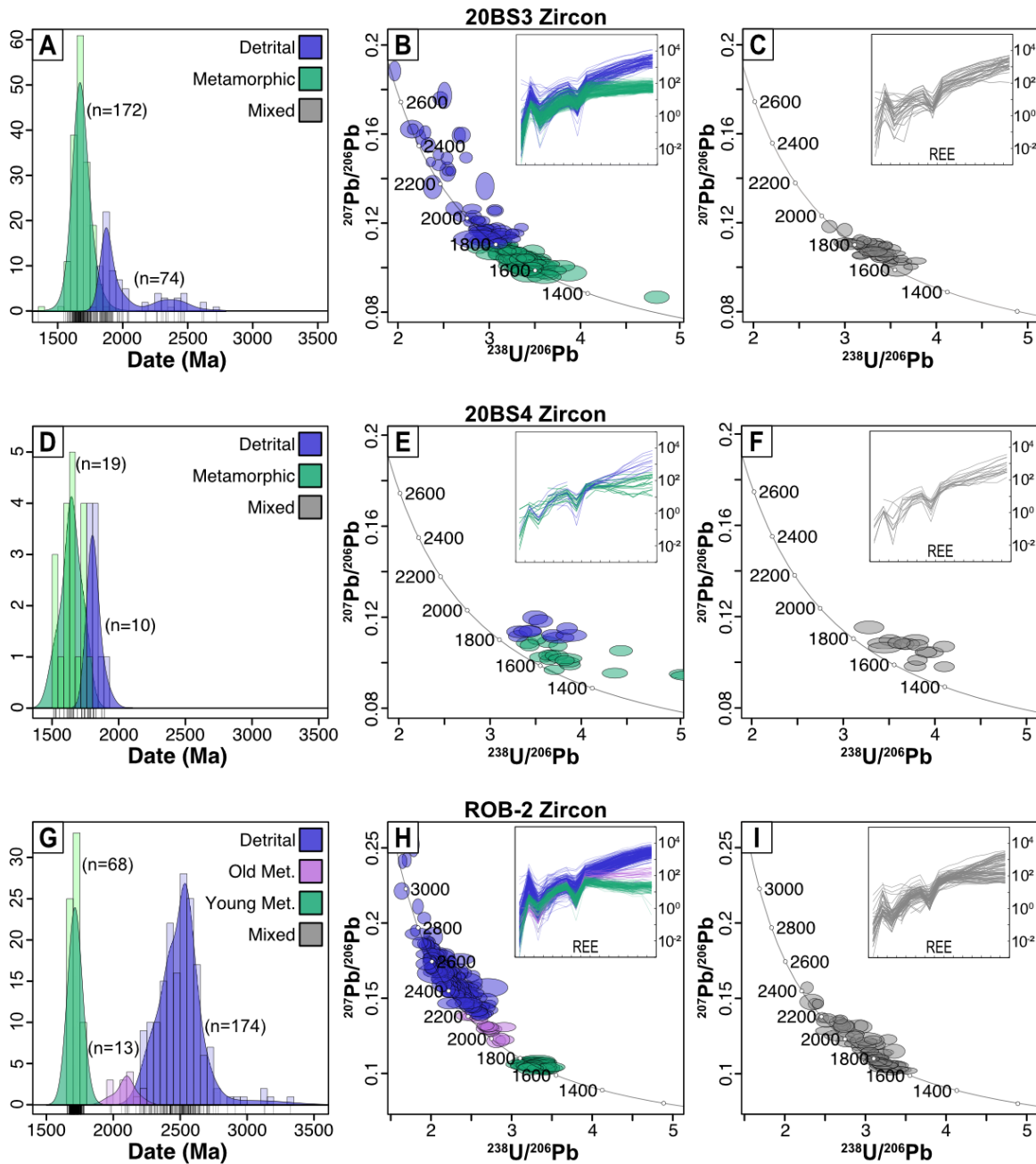


Figure 7. Zircon geochronology results: (a-c) Sample 20BS3 includes 74 detrital, 172 metamorphic, and 42 mixed analyses. (d-f) Sample 20BS4 includes 10 detrital, 19 metamorphic, and 16 mixed analyses (20BS4 was only analyzed in thin section.). (g-i) Sample ROB-2 includes 174 detrital, 76 mixed, and 68 metamorphic analyses with similar metamorphic dates to the other samples, as well as an older metamorphic population. Mixed analyses are those which incorporated both metamorphic and detrital material or that appeared to be older zircon partially recrystallized by subsequent events. This type of analysis was determined by comparing REE patterns, dates, and cathodoluminescence images of the zircon. Data shown includes kernel density estimate plots for $^{207}\text{Pb}/^{206}\text{Pb}$ ages (left panels), Tera-Wasserburg concordia (TW) plots for detrital and metamorphic zircon (central panels), and TW plots for mixed zircon analyses (right panels). REE for analyses are included as insets in the TW plots. Ellipses on TW plots show 2σ total uncertainty for individual spot analyses. For all samples, indigo is used for detrital analyses, green for metamorphic analyses, and gray for mixed analyses, as well as light purple for the older metamorphic population seen in ROB-2. The mixed analyses and the significant smearing along concordia are likely due to the numerous metamorphic and metasomatic events these rocks experienced.

(>50% discordancy) with $^{207}\text{Pb}/^{206}\text{Pb}$ ages as young as 627 Ma. All monazite grains have negative Eu anomalies. In all three samples, there is a population of monazite with less pronounced negative Eu anomalies. These analyses mostly correspond to the period between 1800 and 1600 Ma or 2200 and 2000 Ma. However, not all analyses from this time period exhibit this trend, and there is no clear single correlation between these analyses and age or other factors, such as proximity to plagioclase.

Zircon U-Pb and trace-element depth profiles were obtained for 51 spots from 20BS3 and 38 spots from ROB-2. Individual profiles from 20BS3 primarily yield concordant or near-concordant Proterozoic dates as old as 1600 Ma and discordant analyses trending from younger Proterozoic ages (~1200 Ma) toward Mesozoic or Cenozoic dates on concordia. Profiles for ROB-2 zircon are more generally concordant. Some depth profiles show an increasing common Pb influence in the data points near the surface of grains. All zircon depth profiling data are provided in the supplement, along with additional plots.

Monazite U-Pb and trace-element depth profiles were collected for 12 spots from 20BS3, 4 spots from 20BS4, and 4 spots from ROB-2 and were limited by the number of available grains. Individual profiles for 20BS3 and 20BS4 generally include concordant or near concordant Proterozoic dates and discordant dates trending toward Mesozoic or Cenozoic dates on concordia, though some individual profiles record concordant Proterozoic

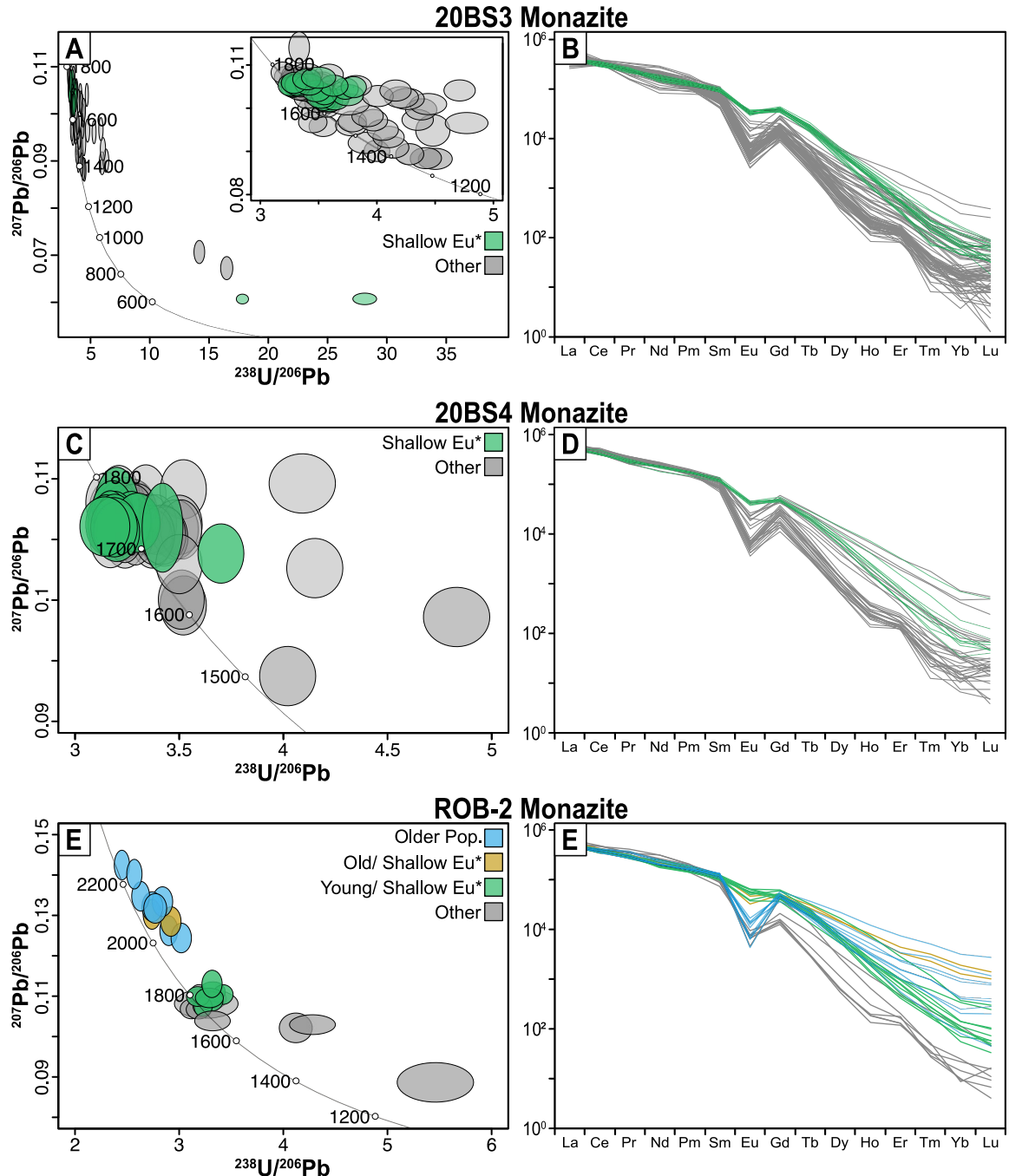


Figure 8. Monazite geochronology results: (a,b) Sample 20BS3 includes 88 analyses. (c,d) Sample 20BS4 includes 53 analyses. (e,f) Sample ROB-2 includes 28 analyses. Data shown includes Tera-Wasserburg concordia plots (TW) (left panels) and chondrite normalized REEs (right panels). Ellipses on TW plots show 2σ total uncertainty for individual spot analyses. Samples with distinctly higher Eu concentrations (and thus shallower Eu* values) are shown in green, while the remainder of analyses are shown in grey. For sample ROB-2, the older monazite population is also shown in blue. In the case of two analyses from ROB-2 that are both old and have higher Eu concentrations, they are shown in gold.

dates as young as 1000 Ma. Some profiles are highly discordant, with a strong common-Pb influence for the data points closest to the surface of grains. One profile from 20BS3 records concordant Cretaceous and Cenozoic dates, along with discordant analyses that fall along a discordia array between Laramide ages and common-Pb (Stacey and Kramers 1975) (Fig. 9a). The concordant Laramide and Proterozoic dates from this profile have REE patterns that are distinct from each other, with the former having less pronounced negative Eu anomalies (Fig. 9b). Profiles from ROB-2 monazite mostly yield concordant or near-concordant Proterozoic dates with some common-Pb influence. All monazite depth profiling data are provided in the supplement, along with additional plots.

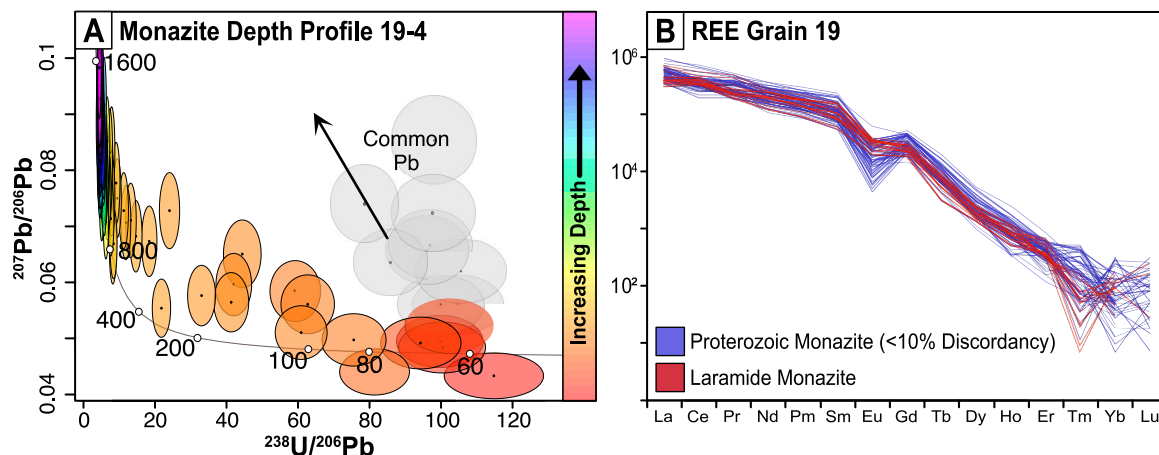


Figure 9. Monazite depth profiling: (a) A single profile from sample 20BS3 showing concordant Laramide ages toward the rim of the grain. Color ramp corresponds to depth of analysis (colder colors = deeper, hotter colors = shallower). Error ellipses show 2σ total uncertainty for individual analyses. (b) Chondrite normalized REE showing a distinct difference in the Eu anomaly between concordant Proterozoic analyses and concordant Laramide analyses in a

U-Pb rutile analyses are displayed in Fig. 10. The ages presented herein were calculated using the analyses that form a discordia array between a common Pb upper intercept and the youngest possible lower intercept with an MSWD of ~ 1 –2. Rutile analyses in metapelite ROB-2, from Robinson Ranch, yield a lower intercept age of 51.2 ± 1.0 Ma (2σ), but show inheritance dating back to the Proterozoic. Rutile in mafic granulite H67-14-5 and metapelite 20BS3, from Big Slide, have lower intercept ages of 46.8 ± 1.9 Ma (2σ) and 46.0 ± 1.0 Ma (2σ), respectively. The analyses from H67-14-5 all roughly fall along the discordia array between common Pb and the lower intercept, whereas rutile in 20BS3 show some inheritance dating back to the Proterozoic. It is noteworthy that H67-14-5 shows higher peak P-T conditions than 20BS3 (890 °C and 1.8 GPa vs. 750 °C and 1.3 GPa: Figs. 5, 6).

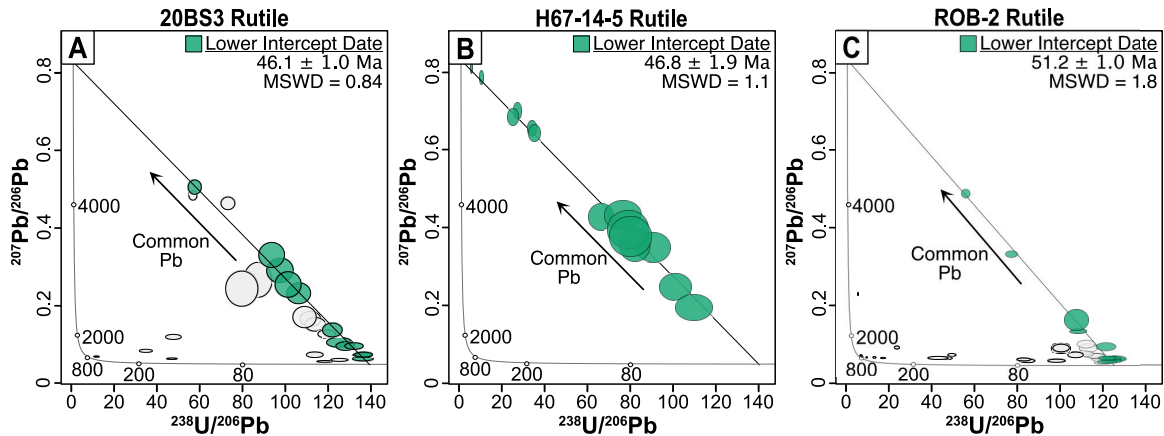


Figure 10. Rutile geochronology results for metapelites: (a) 20BS3 includes 28 analyses. (b) H67-14-5 includes 15 analyses. (c) ROB-2 includes 34 analyses. Tera-Wasserburg concordia plots are shown with the analyses used to calculate the lower intercept ages in green and other analyses in gray. Lower intercepts were calculated using discordia arrays between common Pb (Stacey and Kramers 1975) and the youngest possible dates that formed a reasonable single population. All analyses were used for H67-14-5. Ellipses show 2σ total uncertainty for individual spot analyses.

G. Discussion

1. Provenance of Metapelites

Both the Medicine Hat block and Wyoming craton are potential sources of Archean zircon, which are found in abundance in sample ROB-2 and to a lesser extent in 20BS3. None are observed in 20BS4, though this may be a function of the limited number of analyses given that zircon in the sample were analyzed in a polished thin section. The sedimentary protolith of the three metapelites could be sourced from either the Medicine Hat block or the Wyoming craton. However, the Medicine Hat Block has a dearth of known Paleoproterozoic rocks (e.g., 2500–1900 Ma: Mueller et al. 2002; Downes 2004; Gifford et al. 2014; Chapman et al. 2020; Gifford et al. 2020), and both ROB-2 and 20BS3 contain zircon from this time interval, with the former recording a 2200–2000 Ma metamorphic event. The poor basement surface exposure in the Medicine Hat block means that Paleoproterozoic source rocks could exist but simply be unexposed. Within the context of existing data, however, it is more likely that the sedimentary protoliths were sourced from the Wyoming, which contains known sources of appropriate age detrital zircon.

The detrital igneous zircon ages smear along concordia, likely due to a combination of uncertainty in the data, Pb loss, and domain mixing. There is, however, documented geologic activity in the Wyoming craton that could be the source of the detrital zircon in the metapelites. A major period of crust formation started at 3600 Ma and peaked between ca. 3300–3100 Ma, as recorded in both detrital zircon from multiple locations across the Wyoming craton and the voluminous calc-alkaline rocks from the Beartooth-Bighorn magmatic zone (Mueller et al. 1996; Frost and Fanning 2006; Frost et al. 2017; Mogk et al. 2023). There was further calc-alkaline magmatism between 2900 and 2600 Ma that has been

described as the earliest continental arc activity in the Wyoming craton, though the extent, timing, and how many discrete events this represents are not certain (Frost and Fanning 2006; Mueller et al. 2010; Frost et al. 2017; Frost 2022). Another stage of melting, which may correspond to the final stabilization of the Wyoming craton, occurred between 2600–2400 Ma (Mogk et al. 1988; Gosselin et al. 1990; Frost and Da Prat 2021), overlapping with the ~2500 Ma detrital cores in 20BS3 and ROB-2. The youngest detrital igneous zircon dates in samples 20BS3 and 20BS4 (<1900 Ma) correspond to known magmatism associated with subduction zone magmatism preceding the collisional Great Falls orogeny (Holm and Schneider 2002; Mueller et al. 2002; Bolhar et al. 2007; Barnhart et al. 2012; Foster et al. 2012; Thakurdin et al. 2019a, b).

2. Pre-2 Ga Metamorphism and Rifting

Unlike the two metapelites from Big Slide, ROB-2 zircon and monazite record metamorphism between ca. 2200–2000 Ma (Figs. 7, 8). While it is possible that the older monazite grains are igneous, metamorphic zircon of the same age are also present in ROB-2.

The period before 2000 Ma is not well documented, but the northern margin of the Wyoming craton is thought to have experienced an episode of continental rifting between 2200 and 2000 Ma, a time when various late-Archean “supercratons” were breaking up (Bleeker 2003; Harms et al. 2004; Bowers and Chamberlain 2006; Kilian et al. 2016; Mammone et al. 2022). Others have identified metamorphic domains within monazite or zircon from this time period in xenoliths from the Bearpaw Mountains (Scherer et al. 2000; Bolhar et al. 2007; Barnhart et al. 2012).

If the protolith sediments that ROB-2 was sourced from were buried in the rift setting, they would have been metamorphosed at lower pressures, which may be recorded in the

zircon and monazite. The zircon population has nearly flat HREE with a slightly positive slope, unlike the younger metamorphic zircon (<1800 Ma), which have negatively sloped HREE and lower overall abundances. Generally flat or negatively sloped HREE elements in zircon indicate growth in the presence of garnet (Rubatto et al. 2013; Taylor et al. 2015), so the negatively sloped HREE in the younger metamorphic zircon population may suggest a stronger garnet influence. The younger monazite population in ROB-2 (~1800 Ma) exhibits more depleted HREE patterns than those in the older population (2200-2000 Ma), which also may indicate a stronger garnet influence during the ~1800 Ma metamorphism than during the ca. 2200–2000 Ma metamorphism. It is unlikely that the rift setting transported the ROB-2 protolith sediment to 0.9 GPa; it would then follow that they were later transported to the deeper crustal levels documented in Fig. 5 (0.9 GPa) during the metamorphism beginning ca. 1800 Ma.

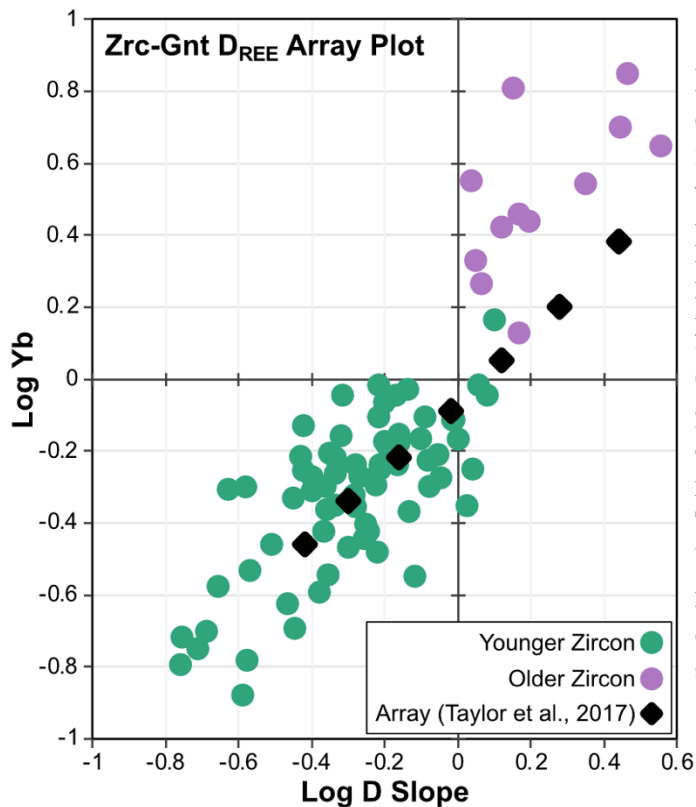


Figure 11. Garnet and zircon data plotted using the array plot method of Taylor et al. (2017), wherein the log of the Yb partition coefficient ($\log(D_{Yb})$) is plotted against the log of a parameter representing the slope from D_{MREE} to D_{HREE} ($\log(D_{Yb}/D_{Gd})$). Zircon and garnet in equilibrium with each other should fall along the array indicated by the black diamonds. The younger zircon population better matches the array, indicating that it is in equilibrium with garnet in the sample.

The garnet in ROB-2 appear to be in equilibrium with the metamorphic zircon that began growing ca. 1800 Ma and therefore more likely represent the equilibrium conditions associated with this younger event. Zircon and garnet that are in equilibrium with each other have characteristic REE partition coefficients (D_{REE}). Using the array plot method of Taylor et al. (2017), the D_{REE} of zircon–garnet from the older zircon population can be compared to that of the younger (Fig. 12). In this method, the log of the Yb partition coefficient ($\log(D_{\text{Yb}})$) is plotted against the log of a parameter representing the slope from D_{MREE} to D_{HREE} ($\log(D_{\text{Yb}}/D_{\text{Gd}})$). If zircon is in equilibrium with garnet, the plotted values should fall along an array (shown in black diamonds in Fig. 12) that was determined through strain modelling and both empirical and experimental data (Taylor et al. 2015, 2017). Data for the younger zircon population in ROB-2 (green circles in Fig. 12) generally fall along the array, while those of the older population do not, indicating that is more likely that garnet is in equilibrium with the younger metamorphic zircon that grew starting at 1800 Ma.

3. Continued Proterozoic Metamorphism and Peak P-T Conditions

Metapelites 20BS3 and 20BS4 show a sharp transition from detrital igneous zircon accumulation to metamorphic zircon growth around 1800 Ma, pointing to a very short time between deposition and metamorphism. Magmatic arc activity has been documented in the region prior to 1800 Ma (e.g., Thakurdin et al. 2019a), so it is likely that these zircon grains were originally sourced from the arc. While both subduction and collision can transport sedimentary material to lower crustal or even mantle depths (Decelles et al. 2009; Hacker et al. 2011; Jacobson et al. 2011; Chin et al. 2013; Ringwood et al. 2024), the high temperatures (relative to pressure) recorded by the thermobarometry, as well as the presence of sillimanite in ROB-2, is not characteristic of a subduction zone geothermal gradient,

which corresponds to 5–10 °C/km (Fig. 5). This suggests that the transport to depth occurred within a retro-arc or collisional orogen setting, though it is possible that the thermobarometry is not representative of peak P-T conditions. However, the timing of the transition from igneous zircon to metamorphic zircon and monazite crystallization at 1800 Ma generally coincides with the assumed cessation of subduction and beginning of collision inferred in the literature (e.g., Barnhart et al. 2012; Thakurdin, et al. 2019a,b; Wang et al. 2022). Even if the sediments were transported to depth within the earlier arc setting, this would have occurred just prior to the onset of collision. The data presented herein clearly document both magmatic and continent-continent collision phases for the Great Falls orogeny.

While the Great Falls orogeny is thought to have largely ended by 1700 Ma, with post-orogenic metamorphism continuing until approximately 1600 Ma, the lower crust clearly records episodic metamorphism until the end of the Proterozoic. Others have also documented younger dates in lower crustal xenoliths from the area (Barnhart et al. 2012; Scherer et al. 2000; Thakurdin et al. 2019b; Apen et al. 2024). Collectively, these data show that the lower crust of the Great Falls tectonic zone was affected by multiple events between the Great Falls and Laramide orogenies. The younger zircon and monazite (1500–1300 Ma: Figs. 7, 8) likely record rifting that lasted from ca. 1470–1350 Ma (Anderson and Davis 1995; Evans et al. 2000; Chamberlain et al. 2003; Barnhart et al. 2012) and which some refer to as the Montana Aulacogen (Shepard 1993; Nelson 1995). The metapelites also contain discordant monazite ages (Figs. 8, 10) marking a discordia array between Proterozoic dates <1000 Ma and some much younger Mesozoic or Cenozoic dates, and these may represent a perturbation of the lower crust due to the emplacement of the Gunbarrel

Large Igneous Province at ca. 778 Ma and associated extension-related magmatism lasting to 740 Ma (Baadsgaard and Mueller 1973; Park et al. 1995; Harlan et al. 1997, 2003, 2008; Mackinder et al. 2019). Among the older rutile dates for 20BS3 and ROB-2 (Fig. 10), the majority intersect concordia between roughly 1000 and 800 Ma. These may record heating associated with the emplacement of the Gunbarrel Large Igneous Province, as well. The crustal thinning in the region associated with the Montana Aulacogen and the emplacement of the Gunbarrel Large Igneous Province likely exhumed the metapelites to lower pressure and temperature conditions more consistent with the depth at which they were entrained prior to eruption (Chamberlain et al. 2003; Barnhart et al. 2012).

4. Origin of the Mafic Granulites

Mafic granulites 20BS19 and H67-14-5 have unusual textures that document their high-grade metamorphic and metasomatic histories (Fig. 2). Garnet and plagioclase are both zoned in major elements, with garnet rims enriched in Ca and depleted in Mg and Fe and plagioclase rims enriched in Na and depleted in Ca (Fig. 4), indicating rim growth at higher pressures. H67-14-5 contains two domains (one with clinozoisite, quartz, and kyanite and one without). Both garnet and plagioclase in the areas with clinozoisite are zoned in the same manner as that in 20BS19. Clinozoisite crosscuts plagioclase, and the plagioclase in contact with the clinozoisite contains more Na and less Ca than the cores. The fine-grained kyanite and quartz appear to be growing at the expense of the clinozoisite and plagioclase. Based on these textural relationships between minerals, the garnet and plagioclase rims were used for the thermobarometry calculations for the mafic samples, which record temperatures and pressures consistent with eclogite-facies conditions: 1000 °C and 2.3 GPa for 20BS19 and 890 °C and 1.8 GPa for H67-14-5. Using the average garnet and plagioclase

compositions yields similar, though slightly less, high-grade pressures and temperatures: 1030 °C and 2.1 GPa for 20BS19 and 840 °C and 1.6 GPa for H67-14-5. These pressures correspond to depths of 85 and 67 km, respectively. Like the thermobarometry calculations, the mineralogy of these samples provides evidence of high pressures and temperatures, though the presence of plagioclase implies that 2.3 GPa is likely too high of an estimate and the result of minerals being out of equilibrium. Both samples contain silvialite, which is only stable at high pressure (> 1 GPa) and temperature conditions consistent with granulite- or eclogite-facies metamorphism (Fig. 6: Newton and Goldsmith 1975; Goldsmith 1976; Teertstra et al. 1999) and which can be an important reservoir for volatiles at deeper crustal levels, including arc lower crust (Hammerli et al. 2017; Wales 2022).

Unlike silvialite, clinozoisite is stable at lower pressures and temperatures, but can also exist at eclogite-facies conditions, where the pressure is high enough in comparison to the temperature (Comodi and Zanazzi 1997). Quartz and kyanite may not be intuitively associated with mafic rocks, but they can be found in granulites (Mansur et al. 2014) and eclogites (Castelli et al. 1998), primarily due to the breakdown of the anorthite component of plagioclase to form kyanite, quartz, and grossular and, at even higher pressures, the breakdown of albite to form jadeite and quartz (the basis of the GASP barometer: e.g., Holdaway 2001). Silvialite will also break down at high enough pressures (Fig. 6) to form quartz, kyanite, and grossular, along with calcium-sulfate (Newton and Goldsmith 1975; Goldsmith 1976; Brunsmann et al. 2000; Alderton 2021), though neither 20BS19 or H67-14-5 document the breakdown of silvialite or record high enough P-T conditions.

These samples equilibrated at great depths (85 km: 20BS19, 67 km: H67-14-5) and likely represent peak crustal thickness prior to crustal thinning associated with the Montana

Aulacogen and the emplacement of the Gunbarrel Large Igneous Province (Chamberlain et al. 2003; Barnhart et al. 2012).

Both mafic samples are silica-poor (Table 2) and contain abundant plagioclase. This anorthosite-like composition indicates that they could be arc cumulates, which form through the accumulation of igneous minerals at depth as melt escapes the system. Arc cumulates are generally plagioclase-rich and mafic to ultramafic in bulk composition (Kelemen et al. 2007; Chapman et al. 2014; Jagoutz 2014; Lee and Bachmann 2014; Ducea et al. 2015; Chin et al. 2021). In the Montana samples there are a number of different geochemical signatures that could point to a cumulate origin. For example, Jagoutz et al. (2011) and Jagoutz (2014) classify arc cumulates as those that have (1) $\text{SiO}_2 < 50 \text{ wt\%}$ and/ or $\text{Si/Al} < 2.9$ AND (2) $\text{Sr/Nd} > 0.50$, $\text{Eu/Eu}^* > 1$, and/ or $\text{Eu/Sm} > 0.46$. Sample 20BS19 meets both criteria 1 and 2 (Fig. 13, supplementary tables). While trace-element concentrations for H67-14-5 could not

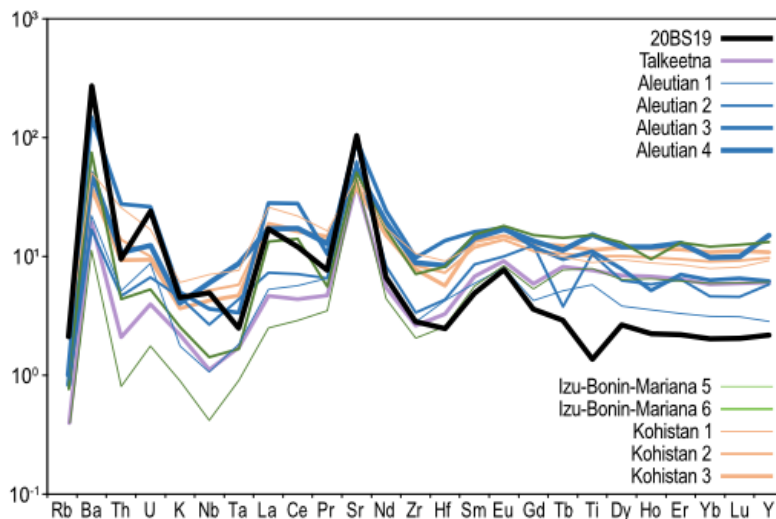


Figure 12. Whole-rock trace-elements for 20BS19 (chondrite normalized: McDonough and Sun 1995). Whole-rock trace-elements of Talkeetna, Aleutian, Kohistan, Izu-Bonin-Mariana arc cumulates as calculated/compiled by Kelemen and Behn (2016) shown for comparison. 20BS19's trace-elements are generally comparable to the other arc cumulates, though the HREE are slightly more depleted.

be determined due to a lack of whole rock powder, it does meet criteria 1. Arc cumulates generally also have higher Nb/Ta ratios than shallower magmatic arc rocks, due to the accumulation of magmatic rutile, and have an average Nb/Ta ratio of ~19 (Tang

et al. 2019). Sample 20BS19 has an even higher Nb/Ta ratio of ~38 (supplementary tables). The high Ca content of the garnet rims is points to increasing pressure over time, indicating that lithospheric thickening due to contractional orogenesis played a role in their metamorphism.

The timing of the lithospheric thickening responsible for the high pressures recorded in 20BS19 and H67-14-5 is uncertain due to a lack of dateable phases in these samples (other than rutile, which does not retain Pb at the high temperatures these rocks experienced, Fig. 10b). However, the original cumulates most certainly formed prior to the Laramide orogeny, as it did not manifest as a thick continental arc in central Montana. The magmatic arc that preceded collision during the Great Falls orogeny was the last documented active arc in this region (Bolhar et al. 2007; Barnhart et al. 2012; Thakurdin et al. 2019a,b; Wang et al. 2022) and is therefore the youngest event that would have generated arc cumulates.

5. Melt Extraction and Laramide-age Metasomatism

Both mafic granulites, 20BS19 and H67-14-5, have distinctive textures and mineral assemblages (Fig. 2b, d), and both contain clinozoisite and silvialite. These phases are associated with metasomatic alteration of rocks and are often found in skarns or metamorphosed limestones (Newton and Goldsmith 1975; Goldsmith 1976; Brunsmann et al. 2000; Alderton 2021). Sample 20BS19 contains intergrowths of kyanite and clinopyroxene, and there are few documented examples of this in the literature. Both examples discussed here are the product of high-grade metamorphic conditions combined with metasomatism. Meyer and Brookins (1976) described radial patterns/symplectites of sillimanite and clinopyroxene replacing plagioclase at 1.0–1.4 GPa and 800–1000°C. The clinopyroxene produced by this reaction is aluminous (10.1% Al_2O_3 in a spinel-pyroxenite

and 7.6% Al_2O_3 in a metagabbro: Meyer and Brookins 1976). The clinopyroxene intergrown with kyanite in 20BS19 is similarly aluminous (10.3% Al_2O_3 ; Table 2). Daczko et al. (2002) also documented a series of reactions under similar lower crustal conditions wherein hydration of a garnet-clinopyroxene-quartz assemblage forms hornblende and clinozoisite, and later dehydration causes the breakdown of the hornblende and clinozoisite to form clinopyroxene-kyanite symplectites.

If 20BS19 and H67-14-5 indeed have similar origins, their individual mineral assemblages may be due to their slightly differing whole-rock compositions (Table 2) and/or metamorphic grade (Fig. 2b, d). Sample 20BS19 contains more Mg and Fe, while H67-14-5 has more Al and Ca. If the series of reactions described by Daczko et al. (2002) played a role, it would follow that clinozoisite would be the limiting reactant for 20BS19 (lower Al) and amphibole the limiting reactant for H67-14-5 (more Al). There is, however, a small amount of relict clinozoisite that is starting to break down in 20BS19 (Fig. 2c). The process described by Meyer and Brookins (1976) may have also played a role, with the clinopyroxene-kyanite intergrowths forming at the expense of plagioclase in 20BS19 (there is no evidence of current or former symplectites in H67-14-5.).

Further evidence of metasomatism is seen in the metapelites. U-Pb geochronology of accessory phases in these rocks, as well as titanite growth around rutile in mafic granulite xenoliths from nearby localities (Apen et al. 2024), also clearly document metasomatism during the Laramide orogeny prior to entrainment of the xenoliths by their host. Depth profiles for both zircon and monazite yielded Proterozoic ages, along with discordant analyses generally trending back toward Mesozoic or Cenozoic ages (see supplementary material). A single monazite in 20BS3 yielded multiple concordant Laramide dates toward

the rim, the majority of which are between 70–60 Ma (Fig. 9a), suggesting that the discordant analyses in other monazites may also reflect rim growth or recrystallization during the Laramide, prior to the time of entrainment. The swath of discordant ages could be the result of either Pb loss at the lower intercept date or mixing between older material and thin rims that crystallized during a Laramide-age event. The latter is far more likely, as there is no documented UHT metamorphism at that time, and both zircon and monazite have high Pb closure temperatures in the absence of significant radiation damage ($>900^{\circ}\text{C}$: Cherniak and Watson, 2001; Cherniak et al., 2004). 20BS3 and ROB-2 contain rutile with only partially reset Proterozoic ages, while ROB-2 includes a few near-concordant Proterozoic analyses (Fig. 10), and the Pb closure temperature for rutile is $\sim 600^{\circ}\text{C}$ at most in natural rutile (Cherniak 2000; Ewing et al. 2013; Smye et al. 2018), significantly less than that of zircon and monazite. If these samples had been sitting above 900°C during the Laramide, the rutile would likely record only Laramide ages. Furthermore, the REE patterns of the concordant Laramide monazite domains are distinct from the concordant Proterozoic analyses (Fig. 9), suggesting that the rims are indeed new growth. Others (Egger, 1988; Chin et al. 2021; Apen et al. 2024) also documented metasomatism of the deep lithosphere of the Wyoming craton during the Laramide orogeny. Therefore, it is likely that the metapelites experienced new growth or recrystallization of both monazite and zircon rims in the presence of fluid and that the swath of discordant dates seen on concordia plots represents mixing of the rims and the Proterozoic domains during analyses.

Metapelites 20BS3 and 20BS4 have unusually low SiO_2 (~ 45 and 48 wt%) and high Al_2O_3 (~ 29 and 24 wt%) (Table 2), which suggests that they may be residues of extensive melting and/ or sourced from extremely aluminous sediments. Crustal melting has been

documented during the Great Falls orogeny (Cheney et al. 2004; Mueller et al. 2005), and melting of these metapelites likely occurred at that time, though the thermobarometry does not record temperatures quite that high. There is no evidence for extreme crustal thickening and melting during the Laramide orogeny, which would have dehydrated the samples discussed herein.

6. Eruption of Big Slide and Robinson Ranch Diatremes

Eruption ages of the Big Slide and Robinson Ranch diatremes were previously unknown, and the rutile ages presented herein (~46 and 51 Ma for Big Slide and Robinson Ranch, respectively: Fig. 10) are consistent with the age of alkalic volcanism that occurred in Montana during the Laramide orogeny (Dickinson and Snyder 1978; Marvin et al. 1980; Volmer et al. 1984; Hearn et al. 1989; MacDonald et al. 1992; Bird 1998; Facer et al. 2009; Chapman et al. 2020). Thermobarometry of these samples (Fig. 5, 6) indicates residence prior to eruption in the lower crust (for the metapelites) and upper mantle (for the mafic granulites). While it is possible that heating during entrainment could yield Pb loss from rutile, the time frame is typically too short for any significant Pb loss (Schmitz & Bowring, 2003). As such, these samples likely resided above the closure temperature of rutile and were losing lead prior to entrainment and eruption (Cherniak 2000; Schutt et al. 2018; Apen et al. 2020).

H. Conclusions

Detrital igneous zircon data indicate that granulite-facies metapelitic xenoliths from the central Montana alkali province were likely sourced from Archean and Paleoproterozoic

Wyoming craton rocks and that their protoliths were deposited by ca. 2200 Ma (ROB-2, Robinson Ranch) and 1800 Ma (20BS3 and 20BS4, Big Slide). Metamorphic zircon and monazite in ROB-2 record 2200–2000 Ma metamorphism, likely in a rift setting where it experienced metamorphism at moderate crustal depths. All of the metapelites record a major metamorphic event between 1800 and 1700 Ma, documenting their transport to the deep crust under high dT/dP conditions, which likely occurred during continent-continent collision. The transition from detrital igneous zircon crystallization (in 20BS3 and 20BS4) to metamorphic mineral growth around 1800 Ma demonstrates an arc source for many of the detrital zircon in these samples as the intervening ocean basin between the Wyoming craton and Medicine Hat block closed and collision began. The Great Falls orogeny is part of the Proterozoic assembly of Laurentia and largely ended by 1700 Ma, but younger U-Pb dates for metamorphic zircon and monazite in these samples document significant post-orogenic metamorphism until ca. 1600 Ma, as well as the impact of additional episodic thermal events, including metasomatic rim growth associated with the Laramide orogeny. The mafic samples also document metasomatism at depths in excess of the present Moho, which, in concert with their whole rock compositions, suggest an origin as arc cumulates that were subsequently metamorphosed as the lithosphere thickened during orogenesis (likely the Great Falls orogeny). Collectively, these samples provide insight into the 3000-Ma geologic history of Laurentia and underscore the importance of metasedimentary samples and their multitude of dateable phases in revealing the geologic history of a given region.

IV. The Addition of Felsic Sediments to the Lower Continental Crust during the Variscan Orogeny

A. Abstract

Lower crustal metapelitic xenoliths (garnet-sillimanite granulites) from the Bournac volcanic pipe in the Massif Central, France, provide a robust example of sediments transported to depth and incorporated into stable lower continental crust during a collisional orogeny. Dates for detrital igneous zircon range from Archean (up to 3300 Ma) to Devonian in age and record sedimentation just prior to onset of the collisional phase of the Variscan orogeny. Metamorphic zircon and monazite document the beginning of sediment transport to depth at ca. 330 Ma during collision. Zircon and monazite crystallization continued within the lower crust until ca. 265 Ma, corresponding to a period of slow cooling following an episode of UHT metamorphism that peaked at 313 Ma. Zr-in-rutile thermometry and GASP barometry from these samples record peak conditions of 0.63–0.77 GPa and 830–910°C, which correspond to Ti-in-zircon temperatures from ~300 Ma and geotherms in excess of typical continent-continent collisions. Rutile in these samples remained open to Pb loss until their eruption at ca. 11.6 Ma, providing an indirect date of the Bournac eruption. These rocks record incorporation of felsic sedimentary material into the stable deep continental crust during a collisional orogen and their residence there for over 300 Ma. More broadly, the addition of sediments to stable lower crust contributes to changes in crustal composition and has significant implications for the heterogeneity of the deep continental crust, as well as overall crust and mantle heat flow and production.

B. Introduction

1. Motivations

Humanity lives on the continental crust, and nearly all of the resources that sustain our civilization are derived from it. Yet, its formation and evolution over time is still not fully understood. In particular, the continental crust provides one of the greatest conundrums in Geoscience: If mantle melting generates basalt, why is the average continental crust composition andesitic (Rollinson, 2008; Rudnick, 1995)? Furthermore, the oceanic crust is young, ~6 km thick, and mafic and forms by mantle-derived melt addition. By contrast, the continental crust is old, ~35 km thick, and felsic and cannot be formed by single-stage melting of the mantle. Multiple hypotheses have been invoked to explain the more felsic composition of the continental crust (see review by Rudnick, 1995): addition of slab melts in a hotter Archean Earth, delamination/foundering of mafic material from the base of the continental crust, and chemical weathering of the continents (e.g., Arndt & Goldstein, 1989; Lee et al., 2008; Martin, 1986).

Each of these processes almost certainly play a role, but how much each contributes to the generation of the continental crust and how the role of these processes might change from location to location and over time is uncertain. Likewise, it is known that clay-rich terrigenous sediments that form at Earth's surface can be metamorphosed under high or ultra-high-pressure conditions and even become stable components of the deep continental crust (e.g., Dumond et al., 2018; Reid et al., 1989). This is potentially an important way to add felsic material with more heat-producing elements to the lower crust, and examples of metapelites that resided at depth for long time periods can be found both in exhumed terranes (e.g., Dumond et al., 2018) and in lower crustal xenoliths carried to the surface by

volcanism (e.g., Chin et al., 2013; Reid et al., 1989; Ringwood, et al., 2024). Estimates of the amount of metasedimentary rocks in the deep continental crust are as high as 10–20% (Hacker et al., 2011), but quantifying the amount of sediment in the stable lower continental crust is difficult due to its inaccessibility and the wide range of seismic velocities observed in lower crustal metapelites (Hacker et al., 2015; Rudnick & Fountain, 1995). By better understanding the conditions under which sediments can be transported to depth and the mechanisms responsible for said transport, researchers can begin unraveling the important Earth processes that add felsic material to the lower crust. In turn, these processes may help explain how the continental crust evolved to its present state, which has significant implications for bulk crustal composition, the heterogeneity of the lower crust, the crustal contribution to surface heat flow, the Moho heat flux, and the origin of modern continents.

To understand how sediments are added to stable lower continental crust, the authors have been investigating metasedimentary rocks metamorphosed under high-pressure conditions to assess the following: (1) Are the pressure and temperature conditions documented by their mineral assemblages consistent with that of the lower crust? (2) Which metamorphic events do they record, and which one is responsible for their transport to depth? (3) Can the timing and pressure-temperature conditions of metamorphism provide insights into the tectonic setting? (4) How long did the metasediments reside in lower crust?

The Neogene basaltic breccia pipe at Bournac in the Massif Central, France (Fig. 1), hosts garnet-bearing metapelitic xenoliths well-suited for studying the mechanisms of sediment transport to depth (Downes & Leyreloup, 1986; Laurent et al., 2023; Leyreloup et al., 1977; Rossi et al., 2006). Not only are such lithologies relatively abundant, but the tectonic history of the region has been well-studied: The Bournac pipe erupted through the

Massif Central, which was formed by stacking of a series of nappes during the Paleozoic Variscan orogeny (Faure et al., 2008; Ledru et al., 1989)—sometimes referred to as the Hercynian or Armorican orogeny. The Variscan was primarily generated by the collision between Laurentia-Baltica (modern North America and Europe) and Gondwana (modern Africa) and correlates along-strike to the North American Alleghanian (or Appalachian) and Ouachita orogenies, which are generally associated with the assembly of Pangaea and closure of the Iapetus and Rheic oceans (Faure et al., 2008, 2009; Matte, 2001; Schulmann et al., 2014; Simancas et al., 2005; Stampfli et al., 2013).

2. Geologic Background

For most of the Cambrian period, Baltica, Laurentia, and Gondwana were separated by the Iapetus Ocean (Cocks & Torsvik, 2005; Matte, 1986; Stampfli & Borel, 2002), and during the late-Cambrian to early Ordovician (510–480 Ma), several peri-Gondwanan terranes broke away from its northern margin. These included Avalonia, Armorica, and Carolinia, among others, and their separation from Gondwana resulted in the formation of the Rheic ocean (Murphy et al., 2006; Nance & Linnemann, 2008; Stampfli & Borel, 2002; Winchester et al., 2002). Eventually, the Iapetus Ocean closed as Avalonia, Baltica, and Laurentia collided to form Laurussia during the Caledonian orogeny (ca. 490–390 Ma) (Schulmann et al., 2014; van Staal et al., 2021; Stampfli & Borel, 2002; Winchester et al., 2002).

The Massif Central is largely composed of Gondwanan-affinity terranes that were juxtaposed via the collision of Laurussia and Gondwana during the Variscan orogeny (ca.

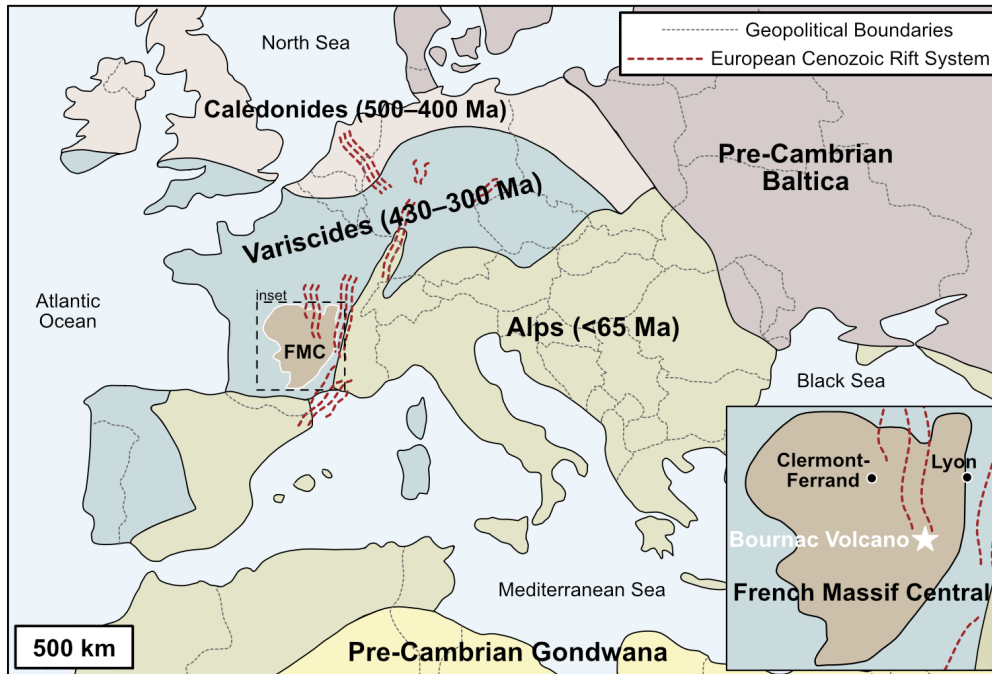


Figure 1. Map of region: Pre-Cambrian Baltica shown in purple and Pre-Cambrian Gondwana in yellow. Regions accreted to Baltica during orogens are indicated in the following colors: blue (Variscan orogeny), gray (Caledonian orogeny), and green (Alpine orogeny). The Massif Central is shown in brown, and major cities, as well as the Bournac Volcano, are visible in the inset map. The red dashed lines indicate major areas of extension due to the Western European rift system. Modified from Artemieva et al. (2006) and Martí & Bolós. (2019).

370–290 Ma) (Faure et al., 2008; Ledru et al., 1989). The earliest stage—sometimes called the Eo-Variscan—involved subduction of the intervening oceanic crust and associated high-pressure metamorphism beginning as early as 430 Ma (Berger et al., 2010; Lotout et al., 2018; Matte, 2001; Nance et al., 2012; Pin et al., 1982).

Previous research identified a high-pressure event recorded in Massif Central rocks at ca. 415 Ma and attributed this to subduction-related metamorphism (Lardeaux et al., 2001; Paquette et al., 1995; Peucat et al., 1982; Pin & Lancelot, 1982). However, later research has cast doubt on these dates. Lotout et al. (2018) points out that these ages are based on whole-grain or multi-grain analyses that would have incorporated both detrital igneous core and metamorphic rim material. For example, many of the 420–400 Ma dates come from whole

zircon grains analyzed by TIMS and yielded discordant analyses. Paquette et al. (2017) redated previously analyzed zircon via LA-ICP-MS and did not find any 420–400 dates in the same material. Berger et al. (2010) also used LA-ICP-MS to analyze zircon and obtained a 412 Ma date, though this is only based on three analyses, and it is not clear that they truly represent a distinct 412 Ma metamorphic event. More recent studies have used both garnet and zircon to document high-pressure metamorphism during the late Devonian (~383–377 Ma) and attribute it to eclogite-facies conditions associated with subduction (Lotout et al., 2018; Paquette et al., 2017). Many of the samples from the earlier studies that identified 420–400 Ma dates also show these Late Devonian or early Carboniferous ages (Berger et al., 2010; Peucat et al., 1982; Pin & Lancelot, 1982), meaning that the older ages could be the result of mixing between the younger metamorphic material and an older detrital igneous component.

In addition to uncertainty about the timing of the onset of subduction as recorded in the Massif Central, there is further disagreement in the literature about the nature of the continental margin prior to the primary collision between Gondwana and Laurussia. Some researchers hypothesize that one or more of the peri-Gondwanan terranes that rifted from Gondwana formed small, short-lived ocean basins and then collided with Laurussia (Franke et al., 2017; Kroner & Romer, 2013; Martínez Catalán et al., 2021). Alternatively, terranes may have re-sutured to Gondwana before the final collision with Laurussia, in which case subduction would have continued along the Laurussian margin uninterrupted through the early Devonian (e.g., Lardeaux et al., 2001; Matte, 2001; Matte, 1986). The multi-stage collision hypothesis has come to dominate in more recent years, and it is nearly universally

agreed upon that the primary collision between Laurussia and Gondwana began by the early Carboniferous (~360 Ma) (Arenas et al., 2007; Kroner et al., 2007; Nance et al., 2012).

Numerous studies have documented the collisional phase of the Variscan orogeny, which was broadly characterized by extensive faulting and the large-scale stacking of thrust nappes (Faure et al., 2008; Fornelli et al., 2020; Kroner & Romer, 2013; Laurent et al., 2023; Matte, 1986; Rossi et al., 2006). Significant Barrovian metamorphism and crustal thickening is recorded by rocks in the Massif Central beginning at ca. 360 Ma, signaling the transition to a tectonic regime dominated by collision (Duguet et al., 2007; Faure et al., 2005, 2009; Lardeaux, 2014; Martínez Catalán et al., 2021), though the Variscan orogeny also involved extensive strike-slip faulting (Kroner & Romer, 2013; Matte, 2007; Mazur et al., 2020) and episodes of lower crustal melting (Downes et al., 1990; Finger et al., 2022; Förster et al., 1999; Žák et al., 2014). Modest extension, the intrusion of voluminous granitoids, and the onset of high-temperature metamorphism is recorded in the Massif Central beginning at ca. 340 Ma (Be Mezeme et al., 2006; Faure et al., 2010, 2014; Laurent et al., 2017). Extensive migmatization of local rocks occurred after 320 Ma (Poujol et al., 2017; Trap et al., 2017; Vanandois et al., 2022), and peak lower crustal temperatures of 970–940 °C were reached at 313 ± 3 Ma (Laurent et al., 2023).

Post-orogenic collapse began at ca. 310–300 Ma (Faure, 1995; Lardeaux et al., 2001; Laurent et al., 2023; Malavieille et al., 1990). This period saw continued melting in the lower crust and the creation of major granite-migmatite zones (Barbey et al., 1999; Chelle-Michou et al., 2017; Cocherie et al., 2005; Downes et al., 1997; Laurent et al., 2017), the largest of which is the Velay dome in the Massif Central (Barbey et al., 1999; Ledru et al.,

2001). Related magmatism persisted until ca. 285 Ma, and the lower crust cooled to about 800 °C by 265 Ma (Laurent et al., 2023; Rossi et al., 2006).

Extension continued, locally contributing to the eventual breakup of Pangaea, which was underway worldwide during the early Triassic (e.g., Morra et al., 2013; Peace et al., 2020; Schettino & Turco, 2009). By the middle Jurassic, the Alpine Tethys Ocean, of which the Mediterranean is a remnant, began opening as the African and European plates separated (Handy et al., 1999; Lardeaux & Spalla, 1991; Le Breton et al., 2021; Marotta et al., 2009; Roda et al., 2019).

As the Atlantic Ocean opened toward the end of the Cretaceous, Africa started rotating in a counter-clockwise direction, leading to the onset of convergence between the Eurasian and African Plates (Angrand & Mouthereau, 2021; van Hinsbergen et al., 2020; Schmid et al., 2008) and the beginning of the on-going Alpine orogeny (Dewey et al., 1973; Lardeaux, 2014; Rosenbaum et al., 2002). The Alpine orogeny led to the formation of the Western European rift system – also called the European Cenozoic rift system – which stretches from the North Sea to the Mediterranean Sea perpendicular to the Alpine Front (Lustrino & Wilson, 2007; Wilson & Downes, 2006; Ziegler, 1992).

The majority of the Cenozoic volcanism in the Massif Central is associated with the Western European rift system (Martí & Bolós, 2019; Michon & Merle, 2001; Wilson & Downes, 2006), though scattered volcanism began in the early Cenozoic (Bellon et al., 1974; Lenoir et al., 2000; Vincent et al., 1977). Since major E-W extension, crustal thinning, graben formation, and magmatism did not start until the upper Eocene, the earlier stage of volcanism is referred to as “pre-rift” (Bergerat, 1985; Lacombe et al., 1993; Michon & Merle, 2001). There was a major rift-associated magmatic event beginning at 15 Ma in the

southern part of the massif and at 9 Ma in the north (Baubron & Demange, 1982; Feraud & Campredon, 1983), as well as a second episode occurring from 2.5 to 0.5 Ma in both the north and south (Gourgaud, 2016; Rochette et al., 1993). This period of volcanism has been associated with crustal thinning, and the average Moho depth in the Massif Central is now ~28–30 km (Barth et al., 2007; Zeyen et al., 1997). The alkali basalt breccia pipe at Bournac has been dated to ca. 7 Ma via K-Ar geochronology (Couthures et al., 1991), though some authors urge caution in interpreting the K-Ar ages from the Massif Central's Cenozoic volcanic rocks (e.g., Michon and Merle, 2001), as some have been proven inaccurate by more precise dating methods (Bellon & Hernandez, 1979; Lenoir et al., 2000).

3. Previous Work

Work on the wide variety of xenoliths found at Bournac, including metaigneous rocks, upper mantle peridotites, and metasedimentary granulites, began in earnest in the 1970s (e.g., Dupuy et al., 1979; Leyreloup, 1974; Leyreloup et al., 1977) and continued into the 1990s (e.g., Downes et al., 1990; Downes & Leyreloup, 1986), after which time the metasedimentary samples were neglected until the 21st century (Laurent et al., 2017, 2023; Rossi et al., 2006).

Leyreloup et al. (1977) and Downes & Leyreloup (1986) demonstrated that metasedimentary granulite xenoliths from Bournac were sourced from terrigenous sediments and that these rocks underwent intermediate- to high-pressure metamorphism (0.9–1.5 GPa), but were later uplifted to lower pressures (0.4–0.9 GPa). Rossi et al. (2006) analyzed zircon in situ from the metasedimentary xenoliths via ion microprobe and identified age populations of 630–430 Ma and 380–150 Ma, with a major peak at 285 Ma. They concluded that the >430-Ma zircon are detrital igneous and sourced from pre-Variscan rocks, based on

Th/U ratios, and that the younger population formed as the result of metamorphism in the lower crust. Rossi et al. also identified a period of prograde heating and melt loss between 340–310 Ma, as well as an episode of cooling from 315–265 Ma. Based on these data, it was inferred that the sediments were buried during the gap in zircon dates between 430 and 380 Ma. In 2010, a study by some of the same authors suggested that the youngest detrital igneous zircons could have been sourced from material subducted during the early stages of the Variscan (Melleton et al., 2010).

Laurent et al. (2023) used laser ablation-based zircon petrochronology on both metasedimentary and mafic granulite xenoliths from Bournac and identified similar age populations as Rossi et al (2006). They concluded that partial melting of the lower crust occurred during ultra-high-temperature (UHT) metamorphism between 340–310 Ma and that this generated the upper crustal granitic rocks from this time period, leaving behind dry metasedimentary assemblages in the local lower crust. According to their data, the peak of UHT metamorphism occurred at 313 ± 3 Ma under conditions of 940–970 °C and 0.8 GPa before crustal thinning and cooling to 800 °C at 0.7 GPa by 265 Ma.

2. Sediments in Stable Lower Continental Crust

Felsic upper crustal material, including sediments, can be tectonically transported to high- or ultra-high-pressure conditions in the lower crust or upper mantle without being incorporated into the stable lower crust. Subducted sediments can be melted and assimilated into magmas (Förster & Selway, 2021; Plank, 2005; Turner & Langmuir, 2022), and there are well-documented examples of this in areas such as the Lesser Antilles in the Caribbean, where large-volumes of sediment collect in the oceanic trench, and arc magmas have geochemical signatures demonstrating the incorporation of melted sediments (e.g., White &

Dupré, 1986). Similarly, felsic material can be transported to high pressures during subduction or thrust-faulting, but remain there only ephemerally before buoyancy-driven exhumation occurs during the same orogenic cycle (Beaumont et al., 2009; Ernst et al., 1997). In these cases, sediments are not incorporated into the stable lower continental crust. Conversely, there are well-documented examples of sedimentary material remaining at lower crustal depths for hundreds of millions or even billions of years before being exhumed or entrained by magmas and transported to the surface (e.g., Chin et al., 2013; Ringwood, et al., 2024). How, then, can sedimentary rocks become part of stable lower continental crust? Four main mechanisms have been proposed for transporting sediments to lower crustal or even mantle depths that can lead to their incorporation into stable lower continental crust.

During subduction, sediments collected in the trench may be thrust/ underplated beneath the overriding plate. An archetypal example is the Pelona-Orocopia-Rand schists in California, USA, which were sea-floor sediments subducted beneath North America during the Late Cretaceous to early Cenozoic (Chapman, 2017, and references therein). Their sedimentary component was primarily sourced from material shed by the western side of the Sierra Nevada magmatic arc (Jacobson et al., 2011); the schists resided in the lower crust for tens of millions of years and were partially exhumed as a result of extension and faulting associated with evolution of the plate boundary as it transitioned to strike-slip movement (Goodman & Malin, 1992; Grove et al., 2016; Jacobson et al., 2007).

Rather than underplating in the forearc, sediments can be subducted into the mantle where they are buoyant compared to the surrounding peridotite. If these sediments did not melt and get incorporated into magmas, they would rise as diapirs through the mantle wedge and could eventually stall out at the base of the over-riding plate's crust. This process, along

with other scenarios in which subducted felsic material ends up in the lower crust, is referred to as relamination (Hacker et al., 2011, 2015; Kelemen & Behn, 2016). Herein, the term is only used to describe the process by which sediment is subducted to ultra-high pressures and rises to the base of the crust via buoyant diapirs. Proposed examples of relaminated sediments are the Swakane and Skagit gneisses in Washington, USA (Gordon et al., 2017; Sauer et al., 2018), which now reside at the surface.

While a magmatic arc is active, compressional forces can generate deep-seated thrust faulting in the retroarc that can transport sediments to deeper levels. For example, Chin et al. (2013) examined metaquartzite xenoliths erupted through the Sierra Nevada batholith in western North America during the late-Miocene and concluded that the quartzites were transported to depth at ca. 103 ± 10 Ma in the retroarc as continental crust was thrust beneath the arc during compression. They remained there for nearly 100 Ma before being entrained within magmas associated with delamination of the local lower lithosphere (DeCelles et al., 2009). These rocks record metamorphism during a period of well-documented arc magmatism, but the high temperatures documented (700–800 °C) rule out subduction as the mode of transport to depth. Furthermore, subduction was to the east at this time, and the detrital zircon were sourced from the North American side of the arc, providing additional evidence that these sediments were transported to depth in the retroarc.

Similarly, after subduction ceases and continent-continent collision begins, deep-seated thrust faulting can bring surface material to lower crustal depths (e.g., Zhao et al., 1993). Ringwood et al. (2024) demonstrated that metapelitic xenoliths from the Potrillo volcanic field were emplaced into the lower crust during the collisional phase of the Picuris orogeny

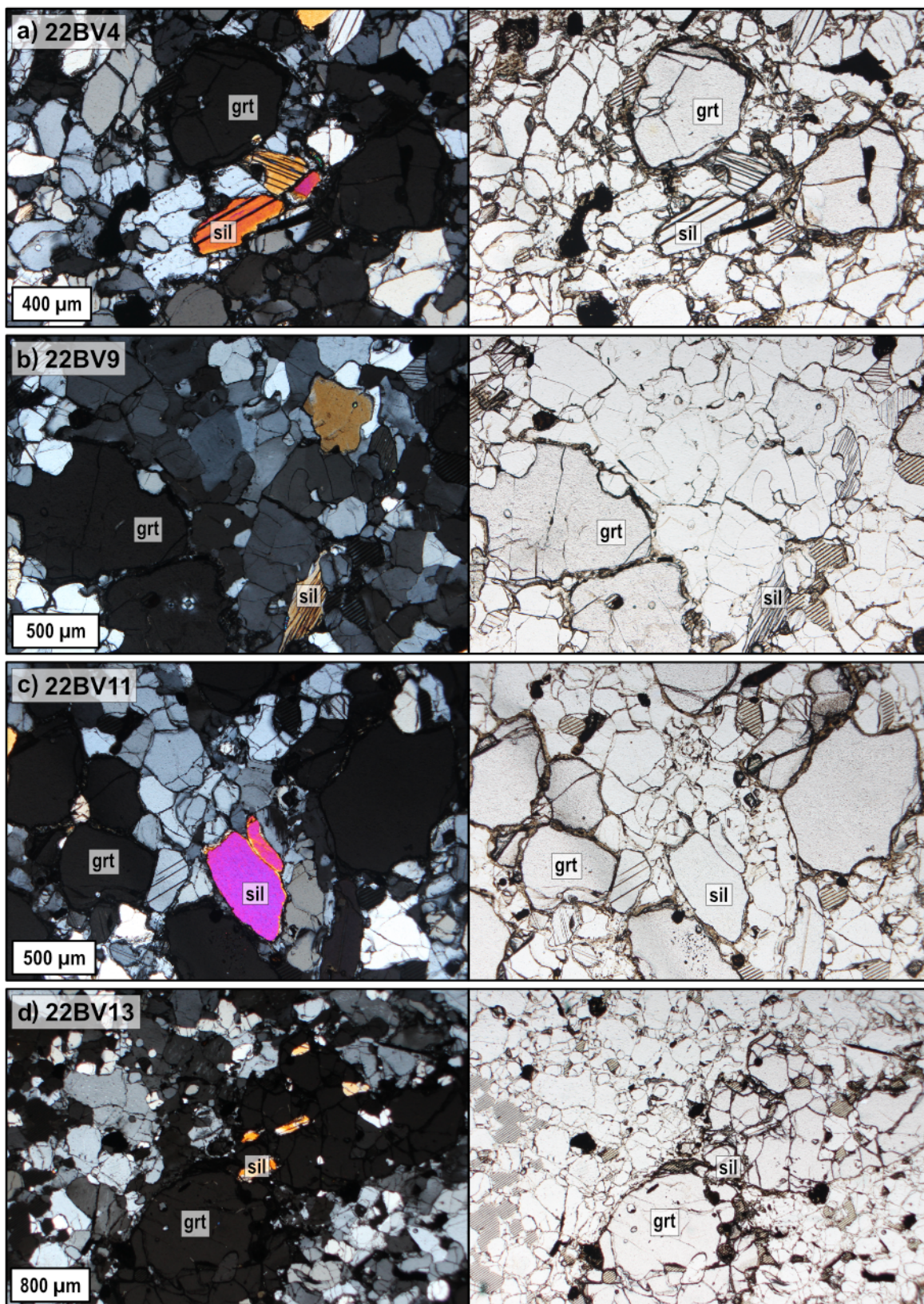
at ca. 1450 Ma and that those metasediments resided in the lower crust for ~1400 Ma and, indeed, remain there today.

C. Bournac Metapelitic Xenoliths

Eight metapelitic xenoliths from the Bournac volcanic pipe were investigated in this study (Table 1): 22BV4, 22BV9, 22BV11, 22BV13, BE330, BE331, Bo.93.8, and Bo.94. All samples are primarily composed of garnet, plagioclase, sillimanite, potassium feldspar, quartz, rutile, and ilmenite, though BE331 also contains minor biotite, which occurs only on grain boundaries and thus appears secondary (Fig. 2f). Zircon, monazite, rutile, and apatite are present as accessory phases. Representative photomicrographs are shown in Fig. 2, and plane polarized, crossed polarized, and backscattered electron scans of sample thin sections are included in the supporting information.

Sample	Qz	Kfs	Grt	Pl	Sil	Bt	Rt	Ilm	Accessory Phases
22BV4	31%	29%	16%	16%	7%		1%	<1%	zrc, ap, mnz
22BV9	34%	25%	21%	12%	7%		1%	<1%	zrc, ap, mnz
22BV11	31%	25%	16%	22%	5%		1%	<1%	zrc, ap, mnz
22BV13	23%	31%	20%	18%	7%		1%	<1%	zrc, ap, mnz
BE330	33%	13%	23%	26%	4%		1%	<1%	zrc, ap, mnz
BE331	34%	11%	26%	13%	9%	5%	<1%	2%	zrc, ap, mnz
Bo.93.8	30%	27%	15%	22%	5%		1%	<1%	zrc, ap, mnz
Bo.94	27%	27%	14%	21%	10%		1%	<1%	zrc, ap, mnz

Table 1. Sample mineralogy: All samples were collected at the Bournac volcanic pipe (44.985 °N, 4.090 °E). Mineral abbreviations are as follows: ap = apatite, bt = biotite, grt = garnet, ilm = ilmenite, kfs = potassium feldspar, mnz = monazite, sil = sillimanite, pl = plagioclase, qz = quartz, rt = rutile, zrc = zircon.



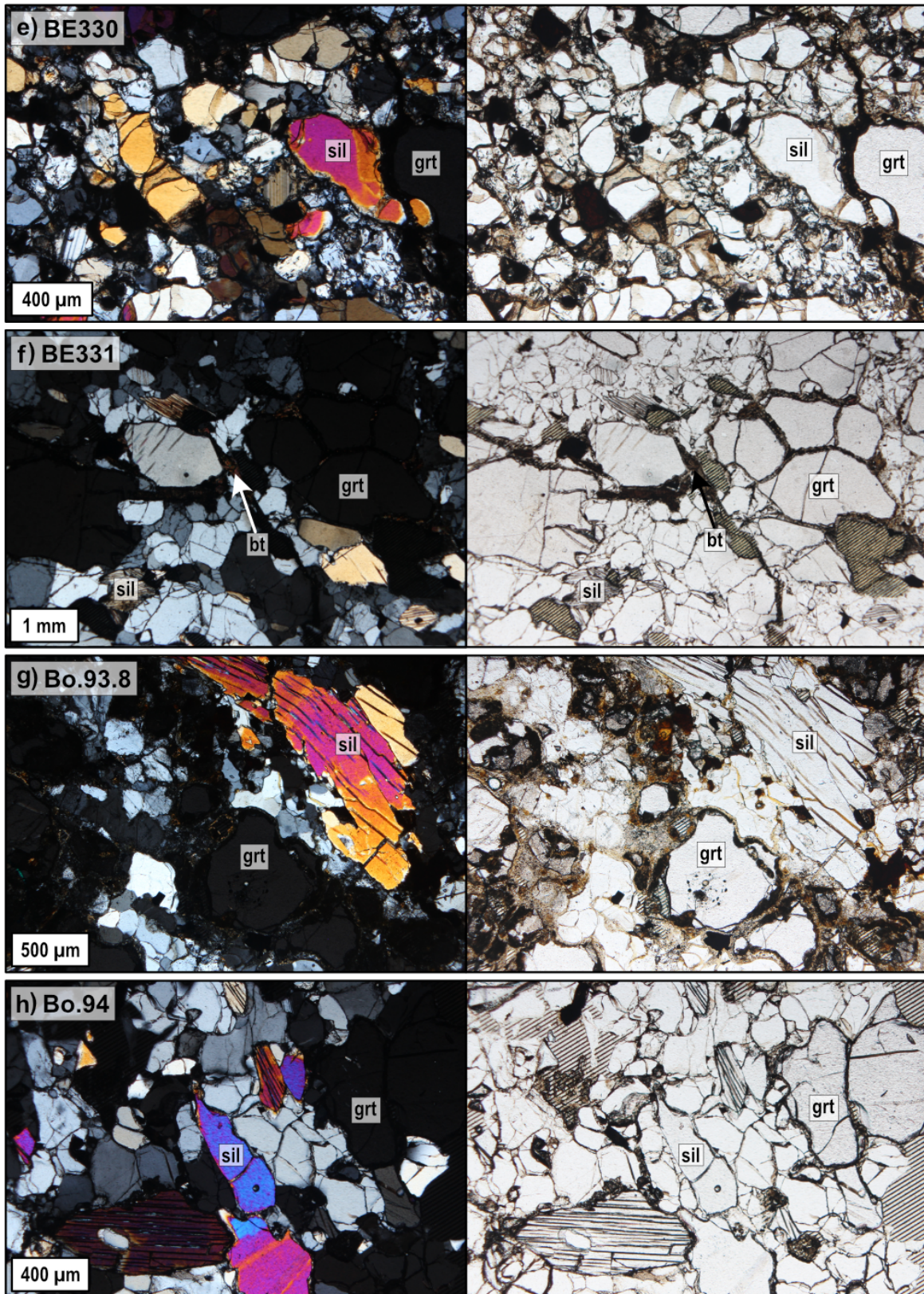


Figure 2. Photomicrographs of xenoliths, with crossed polarized (XPL) images on the left and plane polarized light (PPL) images on the right: (a) 22BV4, (b) 22BV9, (c) 22BV11, (d) 22BV13, (e) BE330, (f) BE331, (g) Bo.93.8, (h) Bo.94. Mineral abbreviations: bt = biotite, grt = garnet, sil = sillimanite. Brown areas with diagonal stripes are regions where EPMA scans damaged the epoxy.

D. Analytical Methods

1. Modal Mineralogy

Backscattered electron (BSE) images and element maps of polished thin sections and the ImageJ software (Rasband, 1997) were used to quantitatively estimate the modal mineralogy of the samples. These BSE and element maps were collected at the University of California, Santa Barbara (UCSB), using a Cameca SX-100 electron probe microanalyzer (EPMA).

2. Mineral Major-Element Analyses

Garnet, plagioclase, biotite, and potassium feldspar major-element data were collected via EPMA at UCSB and processed using Probe Software, Inc.'s data acquisition and analysis software. Ten spot analyses were made on each of ten grains per mineral, using an accelerating voltage of 15 kV with an electron beam defocused to a 2- μ m diameter and a 10-nA current. Zirconium contents in rutile were determined for use in thermobarometry calculations using an accelerating voltage of 20 kV with an electron beam defocused to a 2- μ m diameter and 100-nA current.

3. Whole-Rock Major- and Trace-Element Analyses

Franklin & Marshall College's Malvern PANalytical Inc. Zetium X-ray fluorescence spectrometer (XRF) was used to collect whole-rock compositional data. Homogenized glass

beads were analyzed for major-elements, and briquettes of homogenized powder were used for trace-element analyses. XRF typically yields 2σ uncertainties $<2\%$ for major elements (Rousseau, 2001). Samples 22BV4, 22BV9, 22BV11, 22BV13, Bo.93.8, and Bo.94 were analyzed via XRF, while samples BE330 and BE331 lacked sufficient material for such analyses. Therefore, the whole-rock compositions of BE330 and BE331 were estimated using their modal mineralogies and individual mineral compositions obtained via EPMA.

Additional trace-elements from 22BV4, 22BV9, 22BV11, and Bo.94 were analyzed on the glasses produced for XRF via laser ablation quadrupole inductively coupled plasma mass spectrometry (LA-Q-ICP-MS) at UCSB using an Agilent 7700x Q-ICP-MS connected to a CETAC Teledyne Excimer Excite laser system. Forty-second line analyses were used with an 85- μm spot diameter and laser settings of $\sim 2 \text{ J/cm}^2$ and a fixed dosage of 50 at a 10-Hz repetition rate. Si content (as determined by XRF) was used as the internal element standard, and trace-element data were processed using the Iolite4 software (Paton et al., 2010, 2011). Trace-elements obtained via LA-Q-ICP-MS typically yield results for secondary reference materials within 10% of accepted values (Kylander-Clark et al., 2013). Reference material data is included in supporting information (<https://doi.org/10.17605/OSF.IO/AWD87>).

4. LASS Analyses of Accessory Phases

Zircon grains were separated from six samples: 22BV4, 22BV9, 22BV11, BE330, BE331, and Bo.94. Grains were mounted in epoxy, polished, and imaged via cathodoluminescence (CL) using a KE Developments “Centaurus” Panchromatic CL detector on an FEI Quanta 400f scanning electron microscope (SEM) at UCSB. CL images enabled the identification of oscillatory-zoned domains, which generally belong to detrital

igneous zircon, and more homogeneous domains characteristic of metamorphic zircon (Shore & Fowler, 1996; Vavra, 1990). Figure 3 shows examples of CL images of detrital igneous and metamorphic zircon.

All zircon were analyzed via laser ablation split stream (LASS: Kylander-Clark et al., 2013), wherein U-Pb geochronology and trace-element data are collected simultaneously. Analyses were performed at UCSB using a Nu Plasma 3D multi-collector ICP-MS and an Agilent 7700x Q-ICP-MS coupled with a CETAC Teledyne Excimer Excite laser. Twenty- μm spots and the following laser settings were used: $\sim 1 \text{ J/cm}^2$ for 60 pulses at a 5-Hz repetition rate.

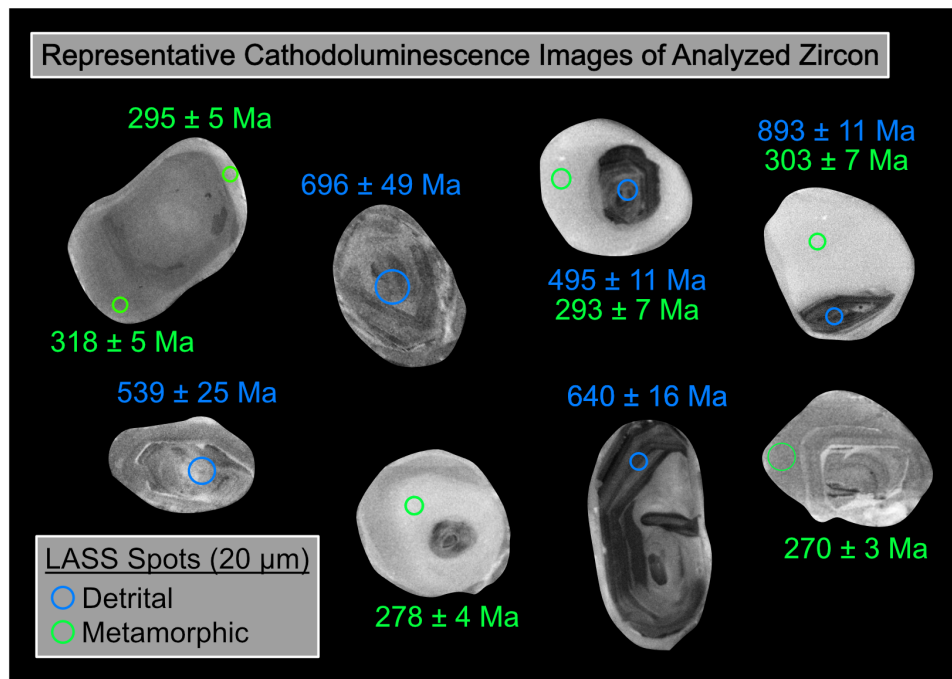


Figure 3. Representative cathodoluminescence images of zircon: Spot sizes shown are 20 microns in diameter. Detrital cores are indicated by blue circles and show oscillatory zoning, while metamorphic domains are shown with green circles and are generally featureless or patchy in appearance. $^{207}\text{Pb}/^{206}\text{Pb}$ spot dates are shown.

Zircon 91500 (1065 Ma: Wiedenbeck et al., 1995) was utilized as the primary U-Pb reference material, and GJ1 (609 Ma: Jackson et al., 2004) was used as the primary trace-

element reference material. The following secondary reference zircons were also analyzed: Plešovice (337 Ma: Sláma et al., 2008), OG1 (3465 Ma: Stern et al., 2009), and Temora-2 (418 Ma: Black et al., 2004). Secondary reference materials yielded concordia ages within 2% of the accepted values (all data in supplement): GJ1 (600 ± 12 Ma), Plešovice (336 ± 7 Ma), OG1 (3473 ± 69 Ma), and Temora-2 (420 ± 8 Ma). An assumed stoichiometric concentration of 14.8% Si was used as an internal standard for calculating trace-element concentrations for unknowns. LASS typically produces trace-element concentrations within 10% of accepted values for secondary reference materials (Kylander-Clark et al., 2013).

Monazite grains were analyzed in-situ in polished thin sections for U-Th-Pb and trace-elements collected via LASS. Grains were identified using optical microscopy and Ce and P element maps produced via EPMA. LASS data were collected at UCSB using 8- μ m spots and the following laser settings: ~ 1 J/cm² for 45 pulses at a 3-Hz repetition rate. Stern (512 Ma: Palin et al., 2013) was utilized as the primary U-Th-Pb and trace-element reference monazite. The following secondary reference materials were also used: 44069 (424 Ma: Aleinikoff et al., 2006), FC1 (55.7 Ma: Paces & Miller, 1993), Trebilcock (273 Ma: Tomascak et al., 2015), and Manangotry (555 Ma, 558 Ma: Horstwood et al., 2003). Secondary reference materials yielded concordia ages within 2% of the accepted values (all data in supplement): 44069 (425 ± 9 Ma), FC1 (57 ± 3 Ma), Trebilcock (274 ± 6 Ma), and Manangotry (554 ± 11). An idealized stoichiometric concentration of 12.9% P was used as an internal standard for calculating trace-element concentrations for unknowns.

Rutile grains were identified via optical microscopy and analyzed for U-Pb and trace-elements in-situ in polished thin sections via LASS or for U-Pb only using LA-MC-ICP-MS. Data were collected at UCSB using 35- or 50- μ m spots and the following laser settings: ~ 2

J/cm² for 60 pulses at a 5-Hz repetition rate. Note that rutile in BE331 was not analyzed, as grains were too extensively infiltrated by ilmenite. Kragero (1087 Ma: Kellet et al., 2018) was utilized as the primary U-Pb reference rutile, and BHVO (Jochum et al., 2005) was used as the primary trace-element reference material. The following secondary reference rutiles were also analyzed: 9826J (382 Ma: Kylander-Clark et al., 2008), LJ04 (498 Ma: Apen et al., 2020), and RMJG (1750 Ma: Zhang et al., 2020). Secondary reference materials yielded lower intercept ages within 3% of accepted values (all data in supplement): 9826J (399 ± 22 Ma), LJ04 (503 ± 40 Ma), and RMJG (1784 ± 36 Ma). A stoichiometric concentration of 59.9% Ti was used as an internal standard for calculating trace-element concentrations for unknowns.

The Iolite4 software (Paton et al., 2010, 2011) was used to process all U-Th-Pb and trace-element data for accessory phases, allowing for correction of data based on instrument signal drift, the baseline signal, and elemental fractionation. IsoplotR (Vermeesch, 2018) was used to plot U-Th-Pb data. All geochronology and trace-element data for reference materials is available in the supporting information (<https://doi.org/10.17605/OSF.IO/AWD87>).

5. Thermobarometry

Pseudosections illustrating the stable mineral assemblages at different pressure-temperature conditions based on whole-rock composition were constructed for all samples using the Perple_X software package (Connolly, 1990) and the internally consistent thermodynamic database of Holland & Powell (2011). Additionally, the garnet-aluminosilicate-quartz-plagioclase “GASP” barometer of Holdaway (2001), the Zr-in-rutile thermometer of Ferry & Watson (2007), and the Ti-in-zircon thermometer of Watson et al.

(2006) were utilized for thermobarometry calculations. Note that only one rutile grain was available for analysis in BE331 due to the prevalence of ilmenite. Also, while BE331 does contain biotite, the garnet-biotite thermometer was not utilized under the assumption that garnet is not in equilibrium with the secondary biotite. Titanium concentrations for zircon were collected via LASS. Typical uncertainties for these thermobarometers are 0.1 GPa and 50 °C. Unlike EPMA analyses, however, LASS allows for Ti-in-zircon measurements on the same material that is being dated.

E. Results

1. Geochemistry and Thermobarometry

Minerals used for thermobarometry calculations and estimating modal mineralogy (Table 1)—garnet, potassium-feldspar, biotite, plagioclase, and rutile—do not show any major element compositional zoning, and representative mineral compositions are shown in Table 2. All compositional data are included in the supporting information (<https://doi.org/10.17605/OSF.IO/AWD87>).

Oxide (%)	22BV4	22BV9	22BV11	22BV13	BE330*	BE331*	Bo.93.8	Bo.94	PAAS**
SiO₂	62.4	61.7	64.8	60.2	64.9	61.1	61.1	70.0	62.8
TiO₂	1.20	1.14	1.01	1.18	1.45	2.21	1.14	1.08	1.00
Al₂O₃	18.3	19.7	17.6	18.9	17.4	17.1	19.3	16.0	18.9
FeO	10.3	9.42	7.68	9.52	6.72	11.2	8.08	5.99	6.50
MnO	0.070	0.080	0.080	0.100	0.100	0.210	0.530	0.050	0.110
MgO	3.64	3.60	3.20	3.55	3.55	4.15	2.14	2.64	2.20
CaO	0.850	0.740	1.44	1.38	1.70	1.23	2.02	0.820	1.30
Na₂O	0.780	0.730	1.34	1.67	1.89	1.08	2.16	0.980	1.20
K₂O	2.11	2.62	2.54	3.47	2.21	1.42	3.22	2.17	3.70
P₂O₅	0.050	0.050	0.080	0.050			0.180	0.060	0.160
LOI	1.36	1.03	1.04	0.430			2.48	1.01	
TE (ppm)									

Sc	18.0	19.0	18.0	22.0	19.0	20.0	16.0
V	234	244	179	179	223	207	150
Co	25.0	18.0	14.0	14.0	52.0	7.00	
Ni	72.0	45.0	20.0	30.0	162	12.0	55.0
Cr	175	159	154	147	149	117	110
Rb	32.6	48.4	48.4	70.8	106.7	41.1	160
Sr	130	109	159	151	207	130	200
Y	40.4	43.1	36.4	50.6	20.4	39.1	27.0
Zr	257	261	212	214	208	228	210
Nb	13.1	12.6	12.6	13.1	19.5	10.6	19.0
Cu	60.0	31.0	17.0	34.0	106	30.0	
Zn	108	104	76.0	94.0	84.0	73.0	
Ga	23.3	23.2	22.0	23.7	27.1	18.9	
Ba	852	899	824	669	1280	1035	650
La	30.0	40.0	34.0	44.0	36.0	36.0	38.0
Ce	54.0	76.0	64.0	74.0	77.0	53.0	80.0
Pb	14.0	17.0	13.0	16.0	16.9	19.5	20.0
Th	3.30	11.3	3.90	3.00	0.800	6.30	14.6
U	1.20	<0.500	<0.500	1.10	0.900	0.500	3.1

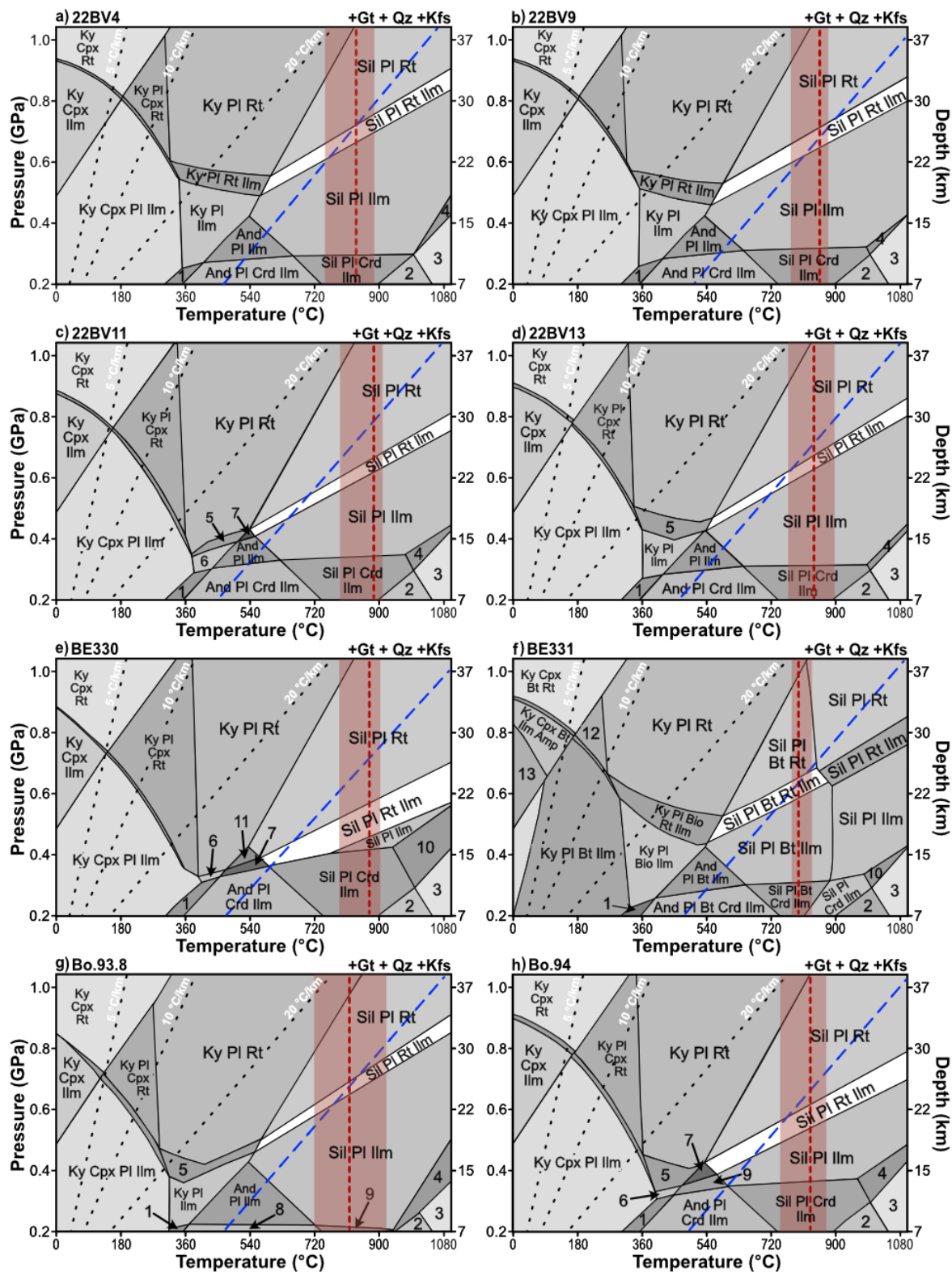
Table 2. Whole-rock compositions: Data include major- and selected trace-elements collected via XRF. Compositions for BE330 and BE331 were estimated using modal mineralogy and mineral compositions. Post-Archean Australian Shale (PAAS) from Taylor and McLennan (1995) shown for comparison.

	SiO₂	TiO₂	Al₂O₃	FeO	MnO	MgO	CaO	Na₂O	K₂O	An	Or
22BV4											
Garnet	38.8	0.022	22.4	24.8	0.241	11.9	1.11	bdl	bdl		
Plagioclase	59.5	0.008	24.4	bdl	bdl	bdl	6.06	7.60	1.85	31	
22BV9											
Garnet	38.6	0.03	22.9	25.5	0.26	11.4	1.16	bdl	bdl		
Plagioclase	60.2	0.01	24.7	bdl	bdl	bdl	6.29	6.47	1.95	35	
22BV11											
Garnet	38.8	0.027	22.4	24.5	0.305	11.9	1.29	bdl	bdl		
Plagioclase	58.1	0.022	25.4	bdl	bdl	bdl	7.18	7.19	1.34	36	
22BV13											
Garnet	38.6	0.02	22.1	25.8	0.326	11.1	1.13	bdl	bdl		
Plagioclase	59.5	0.02	24.5	bdl	bdl	bdl	6.15	7.45	2.01	31	
BE330A											
Garnet	39.3	0.040	22.5	23.7	0.366	12.5	1.18	bdl	bdl		
Plagioclase	59.9	0.02	25.4	0.076	0.003	0.030	6.68	6.53	1.74	36	
K-Feldspar	65.1	bdl	18.8	0.054	bdl	bdl	0.61	3.8	11.0		66
BE331B											
Garnet	38.8	0.05	22.0	25.8	0.628	10.8	1.26	bdl	bdl		
Plagioclase	59.5	0.01	26.0	bdl	bdl	bdl	6.99	7.05	1.09	35	

K-Feldspar	65.0	0.01	18.7	bdl	bdl	bdl	0.366	3.56	11.4	68
Biotite	35.4	5.45	17.8	16.7	0.033	10.8	0.037	0.687	9.18	
Bo.93.8										43
Garnet	38.4	0.05	22.6	25.0	2.72	9.7	1.60	bdl	bdl	
Plagioclase	56.5	0.01	26.9	bdl	bdl	bdl	8.33	6.16	1.02	
Bo.94										33
Garnet	38.9	0.02	23.0	25.2	0.25	11.7	1.11	bdl	bdl	
Plagioclase	59.4	0.01	24.7	bdl	bdl	bdl	6.07	6.83	1.79	

Table 3. Representative mineral compositions: Data include garnet, K-feldspar, plagioclase, and biotite major elements collected via EPMA.

Pseudosections (Fig. 4) were generated using whole-rock compositional data (Table 3) obtained via XRF or calculated using individual mineral compositions and modal mineralogy. The stability fields for the observed mineral assemblages are shown in white. The GASP barometer and average Zr-in-rutile thermometer lines are plotted on the pseudosections (dashed blue and red lines, respectively) and intersect at the following pressures and temperatures: 0.72 GPa and 840 °C (22BV4), 0.68 GPa and 850 °C (22BV9), 0.77 GPa and 890 °C (22BV11), 0.71 GPa and 840 °C (22BV13), 0.71 and 870 °C (BE330), 0.64 and 800 °C (BE331), 0.68 GPa and 830 °C (Bo.93.8), and 0.66 GPa and 830 °C (Bo.94). For samples 22BV4, 22BV9, BE331, and Bo.93.8, these results fall within the garnet-quartz-potassium feldspar-sillimanite-plagioclase-rutile-ilmenite (+biotite for BE331) field on their pseudosections and are in agreement with the observed mineral assemblages. Samples 22BV11, 22BV13, BE330, and Bo.94 yielded P-T conditions that fall within the garnet-quartz-potassium feldspar-sillimanite-plagioclase-rutile field, though all samples contain ilmenite rimming and exsolving from rutile (Fig. 5). In the case of 22BV13 and Bo.94, the GASP barometer line does intersect with the range of Zr-in-rutile temperatures within the stability field for both rutile and ilmenite.



Zr-in-Rutile (Ferry & Watson, 2007)
 Range: Average: - - - - - GASP (Holdaway, 2001): - - - - - Geothermal Gradients:

Figure 4. Pseudosections for samples: (a) 22BV4, (b) 22BV9, (c) 22BV11, (d) 22BV13, (e) BE330, (f) BE331, (g) Bo.93.8, (h) Bo.94. All fields contain garnet, potassium feldspar, and quartz, and the observed mineral assemblage fields are shown in white. The GASP barometer line is shown in blue, and the Zr-in-rutile thermometer is shown in red (shading = range, dashed line = average). Black dashed lines show geothermal gradients of 5, 10, and 20 °C/km. Mineral abbreviations are as follows: amp = amphibole, and = andalusite, bt = biotite, cpx = clinopyroxene, crd = cordierite, hc = hercynite, ilm = ilmenite, kfs = potassium feldspar, ky = kyanite, osm = osumilite, pl = plagioclase, rt = rutile, sil = sillimanite. Fields denoted by numbers are as follows: 1 = Ky Pl Crd Ilm, 2 = Sil Pl Crd Hc Ilm, 3 = Sil Pl Hc Osm Ilm, 4 = Sil Pl Hc Ilm, 5 = Ky Pl Rt Ilm, 6 = Ky Pl Ilm, 7 = And Pl Rt Ilm, 8 = And Pl Crd Ilm, 9 = And Pl Ilm, 10 = Sil Pl Ilm Osm, 11 = And Pl Rt, 12 = Ky Pl Cpx Amp Ilm, 13 = Ky Cpx Amp Ilm.

Results from Ti-in-zircon thermometry generally overlap with those of the Zr-in-rutile thermometer and show decreasing temperatures between 320 and 265 Ma, though significant scatter in some samples makes identifying trends difficult (Fig. 6).

All mineral and whole-rock major- and trace-element data is available in the supporting information (<https://doi.org/10.17605/OSF.IO/AWD87>).

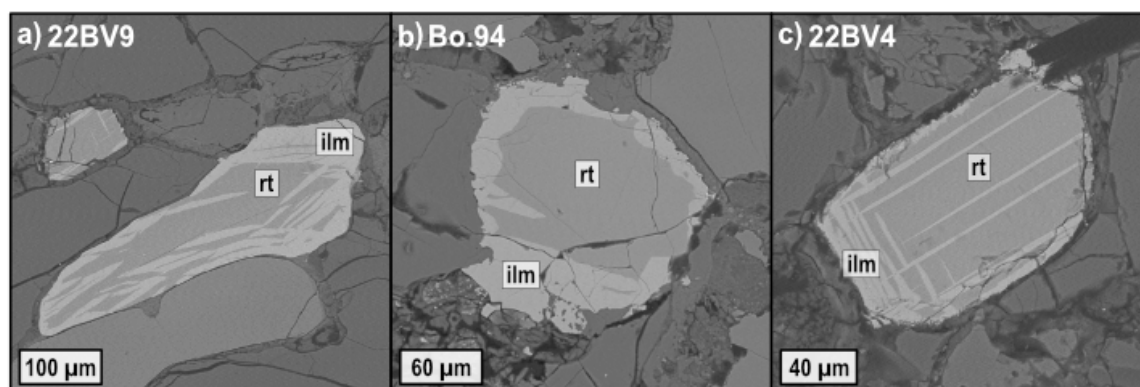


Figure 5. Examples of ilmenite rimming and exsolving from rutile: Backscattered electron images (BSE) were obtained via EPMA.

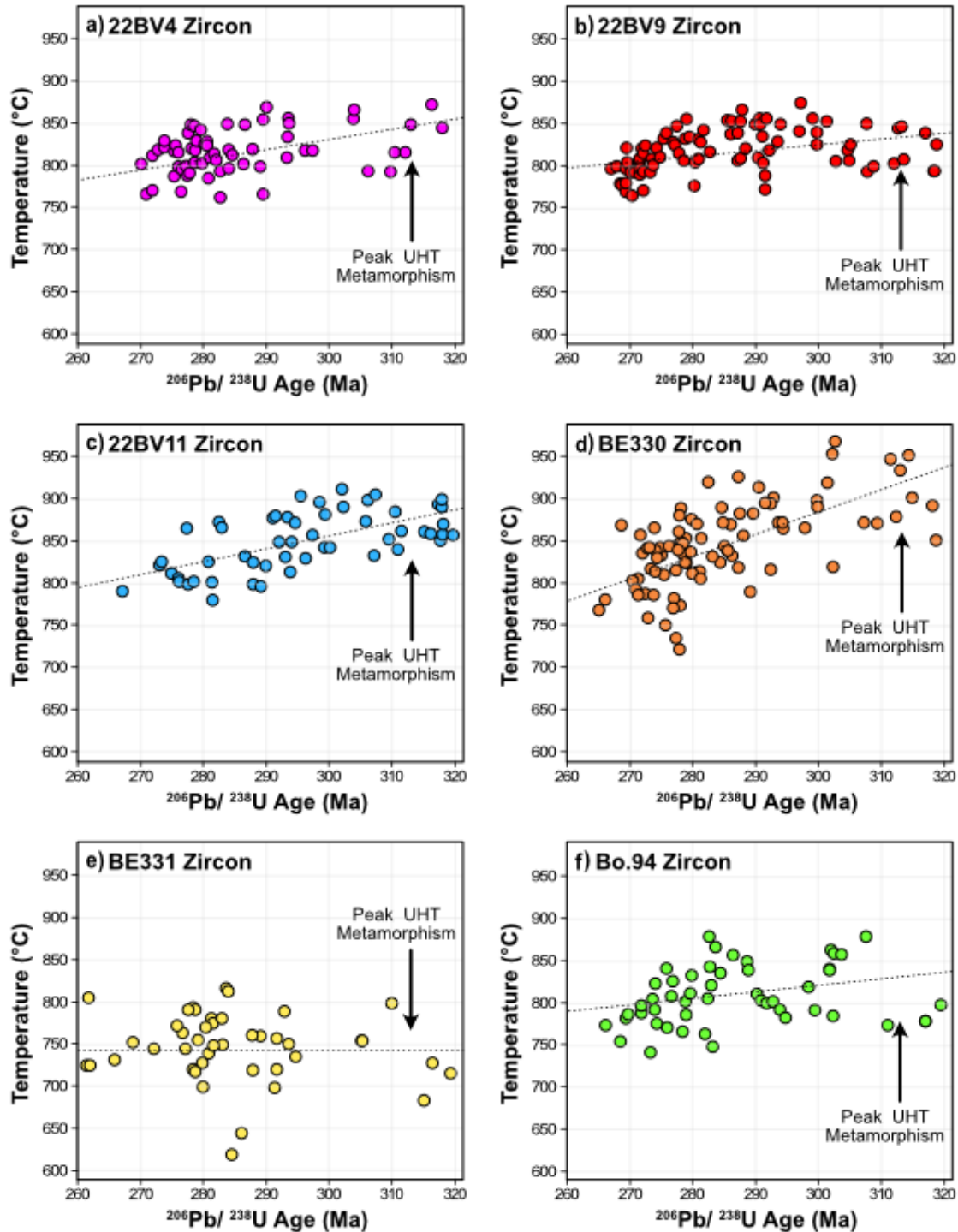


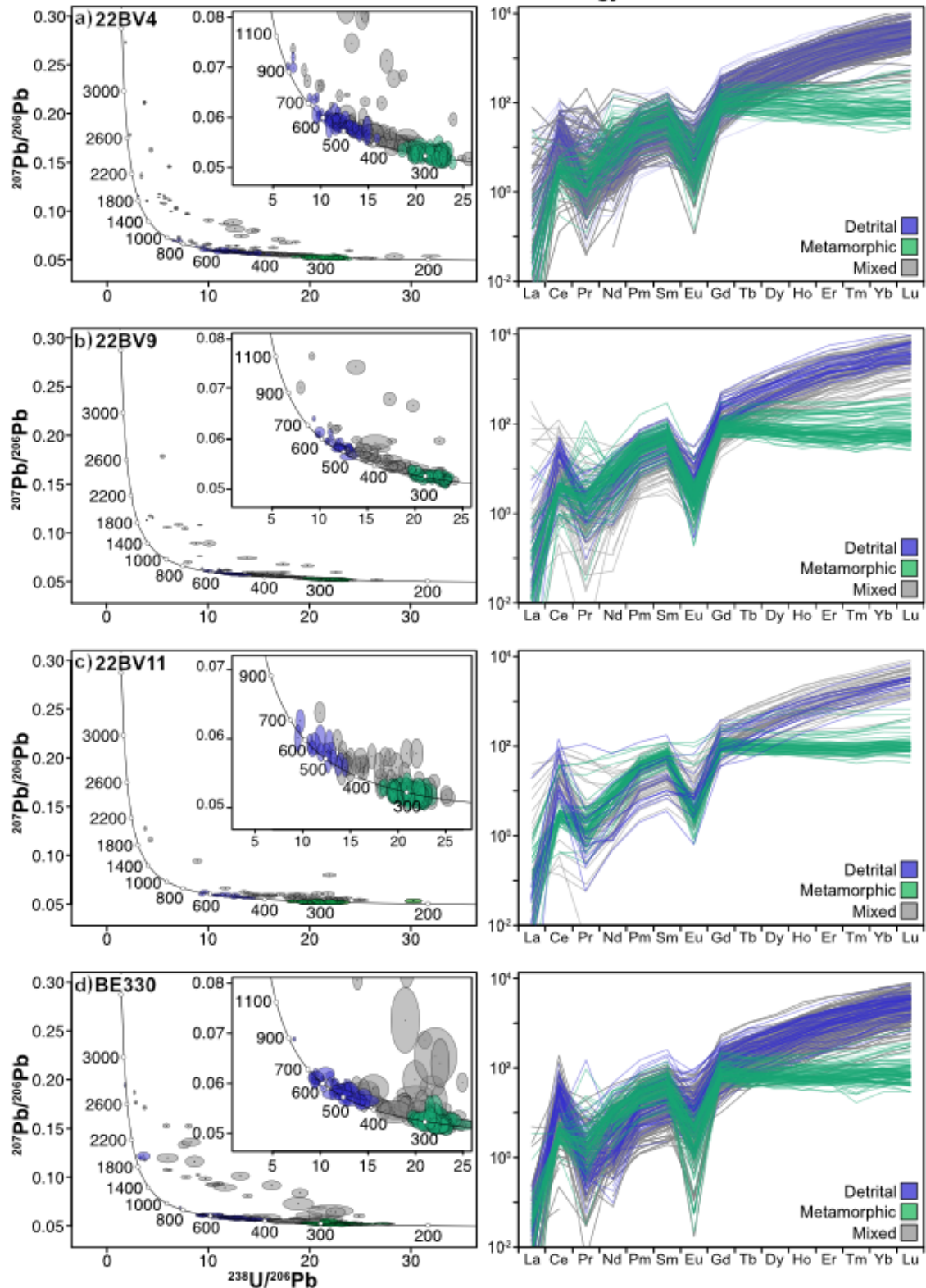
Figure 6. Ti-in-zircon results from 320–260 Ma analyses: (a) 22BV4, (b) 22BV9, (c) 22BV11, (d) BE330, (e) BE331, (f) Bo. 94. The calibration of Watson et al. (2006) was used, and zircon show decreasing temperatures between 320–265 Ma, which is consistent with previous work (e.g., Laurent et al. 2023). The peak of UHT metamorphism in the lower crust at 313 Ma is indicated by an arrow. Black dashed lines show trends.

2. U-Th-Pb and Trace-Elements of Accessory Phases

The majority of U-Pb zircon dates (Fig. 7) are concordant, though there appears to be mixing between detrital igneous and metamorphic populations. All samples contain zircon that have small igneous cores, as determined by HREE enrichment (Fig. 7) and oscillatory zoning in CL (Fig. 3), surrounded by thick, relatively homogenous metamorphic rims with flat HREE patterns. The concordant detrital igneous populations are largely composed of 650–430 Ma dates, though concordant dates as old as 2800 Ma (BE330) and discordant dates as old as 3300 Ma (22BV4) do occur. The youngest detrital zircon analyses have $^{206}\text{Pb}/^{238}\text{U}$ ages of 392 Ma (22BV4 and Bo.94). The number of detrital core analyses varied significantly between samples (5–86 per or an average of 19% of analyses per sample) is likely a function of the number of zircon, the small size of many cores, and the probable impact of metamorphism on the cores. Metamorphic zircon in Fig. 7 are defined as analyses that are <15% discordant with depleted/ flat HREE patterns, the majority of which have dates between 330–260 Ma. Beyond this age peak at 330–260 Ma, there are also nine analyses of metamorphic zircon across all samples that are as old as 348 Ma (Bo.94) and as young as 207 Ma (22BV11). The number of analyses classified as metamorphic were more consistent across samples (between 43–92 or an average of 35% of analyses per sample) than the number of detrital igneous analyses. All samples also contain significant numbers of analyses categorized as “mixed” based on conflicting REE patterns, CL imaging, Th/U ratio, and age. Many of these appear igneous or metamorphic based on HREE and CL imaging, but are >15% discordant and generally fall along a discordia array between Paleozoic or Proterozoic (in a few cases, Late Archean) and metamorphic dates. These analyses may be an actual mixture of igneous and metamorphic material or detrital igneous

grains that lost lead or experienced recrystallization. There are also mixed analyses that are concordant or nearly concordant between ~500–350 Ma but lack or have faded oscillatory

Zircon Petrochronology



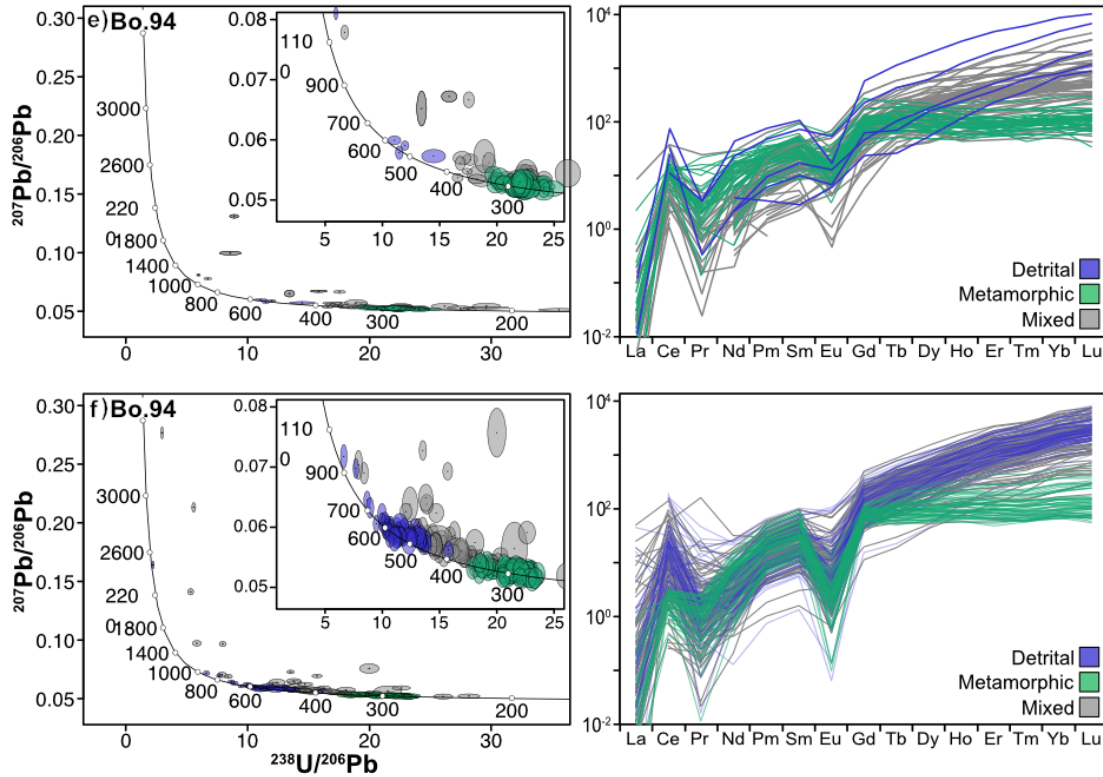
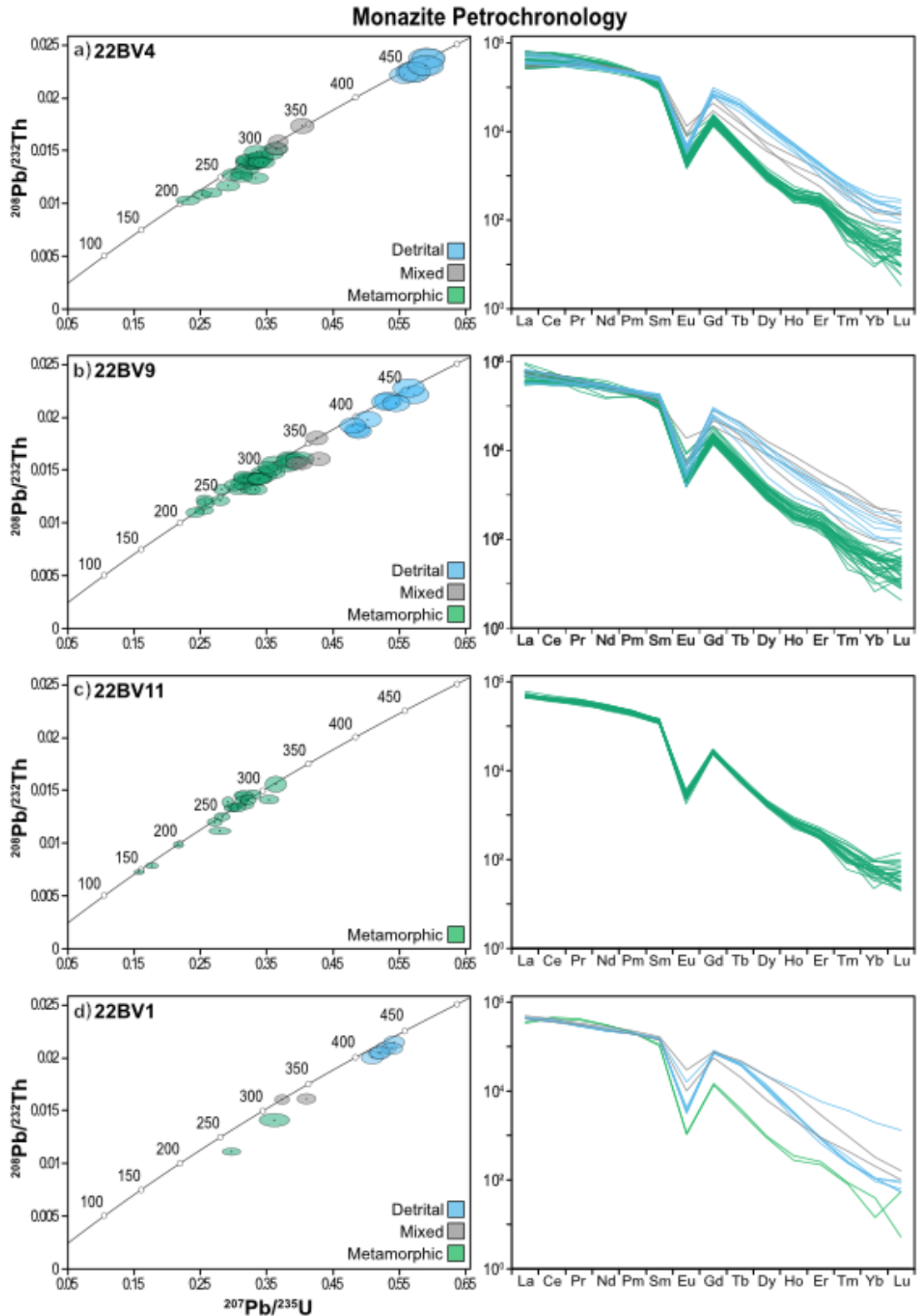


Figure 7. Zircon geochronology and trace element results: (a) 22BV4, (b) 22BV9, (c) 22BV11, (d) BE330, (e) BE331, (f) Bo. 94. Detrital analyses are shown in purple, metamorphic in green, and mixed in gray. Mixed analyses are those which are interpreted to have incorporated both metamorphic and detrital material or that appear to be older zircon influenced by subsequent events. This type of analysis was determined by comparing REE patterns, dates, and cathodoluminescence images of the zircon. Data shown includes Tera-Wasserburg concordia plots (TW) (left panels) and chondrite normalized REEs (right panels). Ellipses on TW plots show 2σ total uncertainty for individual spot analyses. Trace-elements are normalized to primitive mantle (McDonough & Sun, 1995).

zoning and HREE and/or Th/U ratios that are not in agreement. Significant numbers of analyses fall into this category (between 51–162 or an average of 46% of analyses per sample). All zircon have negative Eu anomalies.

Monazite U-Th-Pb isotopes do not yield concordant $^{207}\text{Pb}/^{206}\text{Pb}$ and $^{206}\text{Pb}/^{238}\text{U}$ dates for all analyses (Terra-Wasserburg diagrams in appendix), and many appear to contain an excess of ^{206}Pb , which can make calculated $^{207}\text{Pb}/^{206}\text{Pb}$ and $^{206}\text{Pb}/^{238}\text{U}$ dates incorrect, generating reversely discordant dates or even negative $^{207}\text{Pb}/^{206}\text{Pb}$ ages. Generally, monazite

grains preferentially incorporate Th over U (Cheng et al., 2000; Montel et al., 1996; Suzuki et al., 1994) with extreme Th/U ratios as high as 629 documented in the literature (Janots et



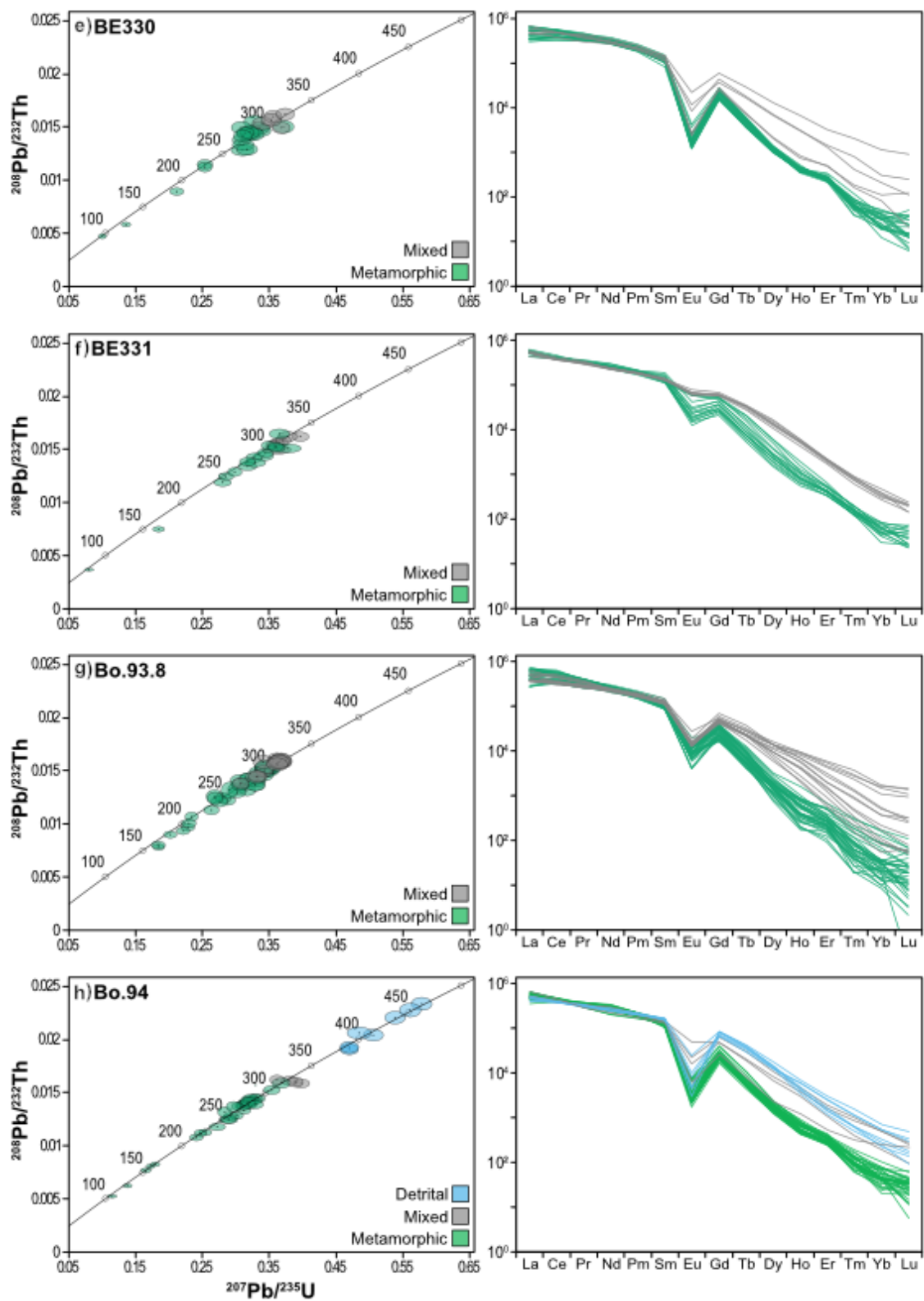


Figure 8. Monazite geochronology results: (a) 22BV4, (b) 22BV9, (c) 22BV11, (d) 22BV13, (e) BE330, (f) BE331, (g) Bo.93.8, (h) Bo.94. Detrital analyses are shown in blue, the primary metamorphic population in green, and “mixed” analyses in gray. Data shown includes modified concordia diagrams (left panels) and chondrite normalized REEs (right panels). Ellipses on TW plots show 2 σ total uncertainty for individual spot analyses. Trace-elements normalized to primitive mantle (McDonough & Sun, 1995).

al., 2012). The average Th/U ratio for monazite analyzed in this study is 21, with some analyses having extreme values of <1. Excess ^{206}Pb in monazite is caused by the incorporation of ^{230}Th , an intermediate decay product of ^{238}U . In this case, it is most appropriate to utilize $^{207}\text{Pb}/^{235}\text{U}$ and/ or $^{208}\text{Pb}/^{232}\text{Th}$ ages, which are largely consistent with each other (Fig. 8a) and have been plotted on modified concordia diagrams using the $^{207}\text{Pb}/^{235}\text{U}$ and $^{208}\text{Pb}/^{232}\text{Th}$ ratios (Fig. 9). The vast majority of monazite record ages from ca. 330–250 Ma (almost an exact match for the zircon U-Pb age peak), with scattered dates as young as 74 Ma (BE331) and as old as 361 Ma (22BV9). Samples 22BV4, 22BV9, 22BV13, and Bo.94 also contain a population of monazite with $^{207}\text{Pb}/^{235}\text{U}$ and/ or $^{208}\text{Pb}/^{232}\text{Th}$ ages ≥ 393 Ma. All monazite analyses show negative Eu anomalies and depleted HREE, though BE331 has a population with higher abundances of HREE and less pronounced negative Eu anomalies, which are all from monazite inclusions within garnets. The older monazite population (≥ 393 Ma) have the least depleted HREE, while the majority of young monazite have more depleted HREE. The number of monazite analyses was dependent on the number and size of grains in thin sections and ranged from 10–45 per sample.

U-Pb rutile ages presented here (Fig. 10) were calculated based on discordia arrays between a lower intercept and common Pb (Stacey & Kramers, 1975). Samples yielded the following ages (2 σ uncertainty): 22BV4 (11.5 ± 0.3 Ma), 22BV9 (11.6 ± 0.2 Ma), 22BV11 (11.0 ± 0.4 Ma), 22BV13 (11.5 ± 0.2), BE330 (11.3 ± 0.2 Ma), Bo.93.8 (11.5 ± 0.2 Ma), and

Bo.94 (11.5 ± 0.2 Ma). All samples have MSWDs of 1 or less, except BE330, which has an MSWD of **4.9**. However, if Stacey and Kramer's (1975) common Pb values are not used for this sample, a date with a lower MSWD is obtained: 12.6 ± 0.4 Ma (MSWD = 0.8). The average age using Stacey and Kramer (1975) for all samples except BE330 is $11.6 \text{ Ma} \pm 0.7$ Ma. The number of rutile analyses per sample was dependent on the number and size of grains in thin sections and ranged from 30–58 per sample.

F. Discussion

1. Pressure-Temperature Conditions and Derivation Depth

The Bournac volcanic pipe erupted through the Massif Central, which is composed of a series of nappes juxtaposed during the Variscan orogeny, as well as related migmatites and upper crustal granitoids (Faure et al., 2008; Fornelli et al., 2020; Kroner & Romer, 2013; Laurent et al., 2023; Matte, 1986; Rossi et al., 2006). Regional metamorphic rocks present at the surface contain assemblages of varying metamorphic grade, including granulite-facies or migmatized metasediments. High-grade metasediments that equilibrated at $\sim 850^\circ\text{C}$ and 0.6 GPa are fairly common in the Upper and Lower Gneiss Units (e.g., G belin et al., 2009; Schulz, 2009; Schulz et al., 1996). It is therefore conceivable that the metasedimentary xenoliths found at Bournac could be sourced from high-grade metasedimentary material currently residing in the upper crust. However, the rutile data presented herein demonstrate that these rocks were open to Pb loss until their entrainment by magmas and must therefore have been sitting at temperatures in excess of 600° (Apen et al., 2020; Cherniak, 2000; Schutt et al., 2018) in the lower crust. While some studies report lower Pb closure

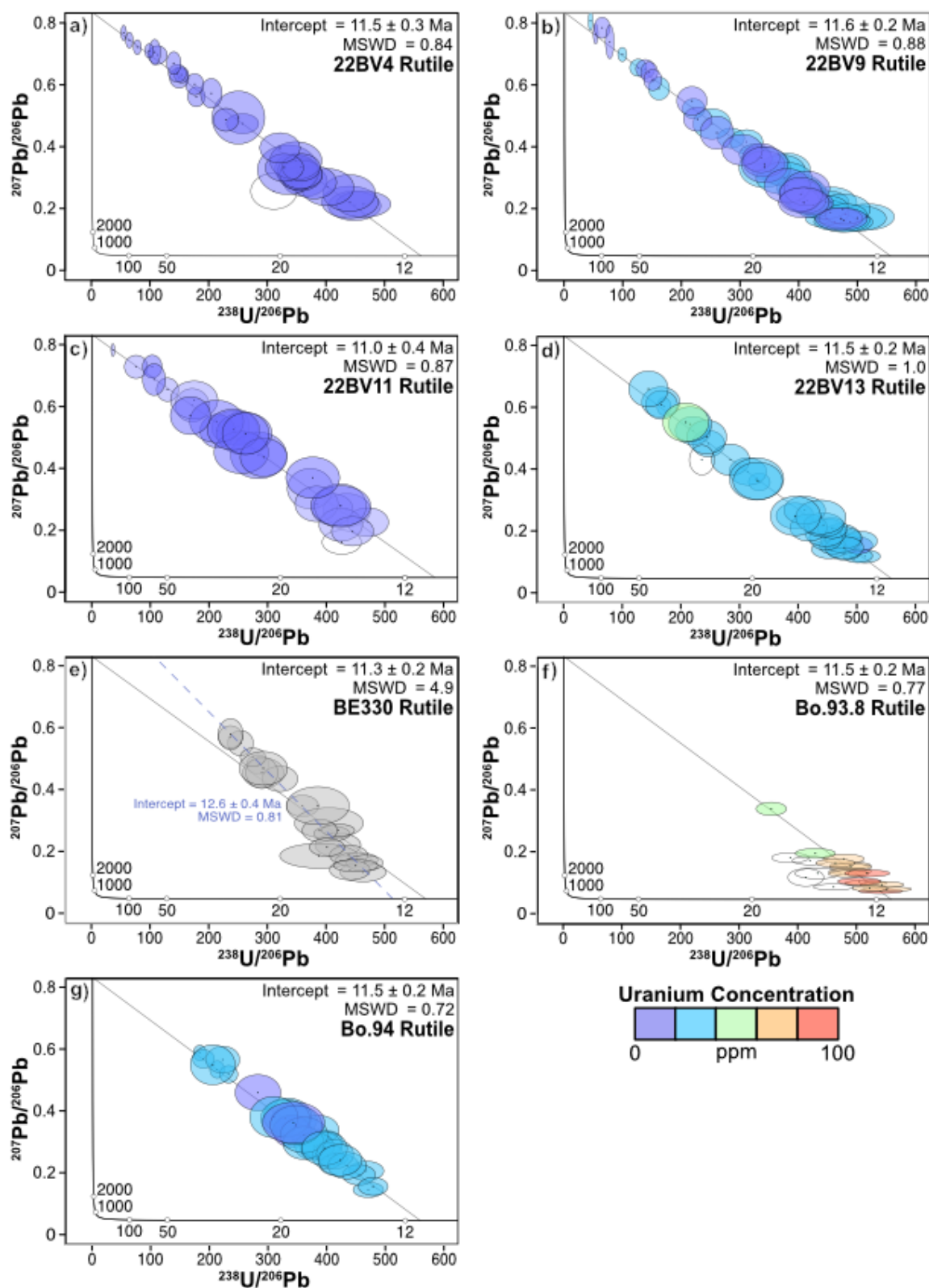


Figure 9. Rutile geochronology results for metapelites: (a) 22BV4, (b) 22BV9, (c) 22BV11, (d) 22BV13, (e) BE330, (f) Bo.93.8, (g) Bo.94. Tera-Wasserburg concordia plots are shown with analyses colored by U concentration (warmer colors = higher, colder colors = lower). Analyses excluded from the intercept calculation are shown without a fill color. Lower intercepts were calculated using discordia arrays from common Pb (Stacey & Kramers, 1975). Discordia calculated based on best fit are shown for BE330 in purple, and U concentrations are not shown for BE330, due to the concentrations being measured via MC-ICP-MS, which generally produces incorrectly low values. Based on the Uranium concentrations in reference materials, concentrations in BE330 are likely ~20–40 ppm. Ellipses show 2σ total uncertainty for individual spot analyses.

temperatures for rutile (Cherniak, 2000, and references therein), the fact that the data tend to form clear discordia arrays without evidence of older, inherited dates indicates that the rutile would have likely resided at temperatures significantly higher than the Pb closure temperature.

The average pressures and temperatures obtained via the GASP barometer and Zr-in-rutile thermometer are between 0.63–0.77 GPa and 800–890 °C (Fig. 4, summary shown in Fig. 11), which are generally consistent with equilibration of the metasediments within the lower crust and likely represent near-peak metamorphic conditions. However, not all P-T estimates fall within the observed mineral assemblages on the pseudosections. In all cases where there is a mismatch, the barometer and thermometer lines intersect in the rutile (sans ilmenite) fields. Because all samples contain ilmenite rimming and exsolving from rutile (Fig. 5), it can be inferred that they experienced a decrease in pressure and/ or an increase in temperature following rutile growth (see schematic dashed arrow in Fig. 11). Therefore, the thermobarometry may simply record conditions prior to this change in pressure and/ or temperature.

Previous studies of Bournac metapelites demonstrated that peak metamorphic temperatures and pressures of 0.8 GPa and 970–940 °C in the lower crust were reached at 313 Ma (open grey circle in Fig. 11 labeled “313 Ma”) (Laurent et al., 2023) before cooling and crustal thinning left the lower crust at 0.7 GPa and 800 °C by 265 Ma (open grey circle labelled “265 Ma”) (Laurent et al., 2023; Rossi et al., 2006). The maximum P-T conditions recorded in this study are generally lower than those of the peak conditions previously documented (Fig. 11) and are very similar to those inferred at 265 Ma. However, our Ti-in-zircon thermometry also yielded lower temperatures than those described previously for peak conditions, and our Zr-in-rutile temperatures generally correspond to the Ti-in-zircon results from ca. 300–290 Ma, though there is significant scatter in the data (Fig. 6).

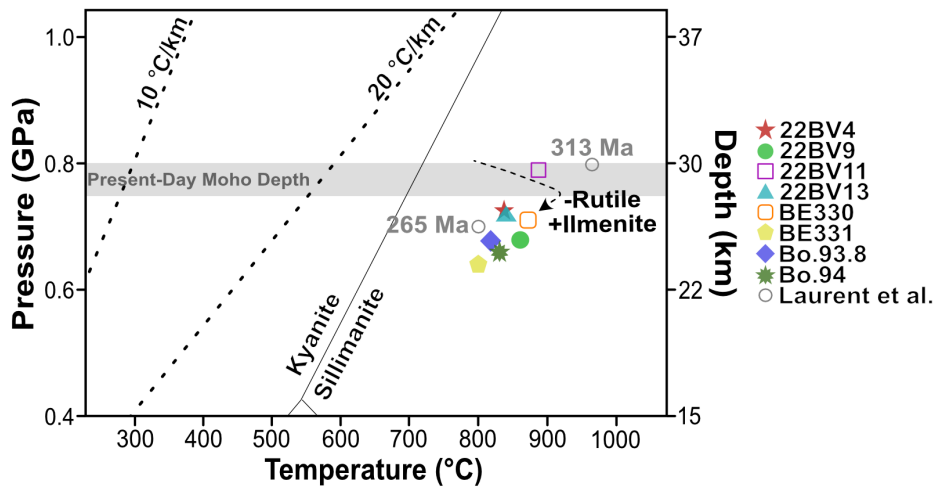


Figure 10. Summary of sample thermobarometry. Samples that plot in the ilmenite + rutile stability field on pseudosections are shown with filled symbols, and samples that plot in the rutile field are shown with open symbols. The P-T conditions as determined by Laurent et al. (2023) at 313 and 265 Ma are shown by grey circles. Geothermal gradients of 10 and 20 °C/km are indicated by dashed black lines, while the current Moho depth is shown by a gray band. Typical subduction geotherms are <10 °C/km (Syracuse et al., 2010).

Overall, the thermobarometry presented here appears to record conditions from the period just after peak UHT metamorphism in the lower crust, around the time of the onset of

post-orogenic collapse or, though less likely, conditions right after the end of the Variscan orogeny. Either way, we find no evidence for the conditions of prograde metamorphism during the sediment's transport to depth, likely due to the UHT metamorphism experienced by the samples. These original prograde conditions would have likely involved higher pressures and lower temperatures than recorded by the thermobarometry. Such a transition fits within the known context of the Variscan orogeny collision involving significant crustal thickening between 360–340 Ma before UHT metamorphism of the lower crust and eventual post-orogenic collapse, which commenced around 310–300 Ma.

2. Sedimentary Provenance

The major and trace-element chemistry of the metapelites is similar to that of post-Archean shales (Fig. 12, Fig. S1 in supporting information), pointing to their likely origin as terrigenous ocean-floor sediments prior to incorporation into the lower continental crust.

The majority of concordant detrital igneous zircon grew between ca. 600–420 Ma (Fig. 7), though there appear to be discordia arrays trending from metamorphic dates toward ~1900 and 2800 Ma, and scattered discordant analyses may represent additional Proterozoic and/or Archean detrital populations. Such mixtures are likely the result of Pb loss associated with UHT metamorphism or mechanical mixing during analysis due to the small size of detrital cores compared to the laser spot.

Theoretically, zircon with these ages could derive from Laurussia (Baltic-Laurentia) or Gondwana, but the latter is far more plausible, as the Massif Central is known to be composed of Gondwana-derived crust (Faure et al., 2008; Ledru et al., 1989). Furthermore, there are intervening peri-Gondwanan terranes, such as Armorica and Avalonia, between the Massif Central and Precambrian Baltica (Franke et al., 2017; Kroner & Romer, 2013;

Martínez Catalán et al., 2021), and sources of ~2800, ~2300, ~1900, ~1300 Ma, and ~640–450 Ma detrital igneous zircon can be found within the Massif Central itself (Alexandrov et al., 2001; Melleton et al., 2010; Pin & Lancelot, 1982). The majority of the detrital zircon likely originated from Gondwana (or the peri-Gondwanan terranes), with those less than 450 Ma in age sourced from the subduction zones responsible for the closure of the Rheic Ocean between Laurussia and Gondwana (Murphy et al., 2006; Nance et al., 2012).

In addition to detrital zircon, there also appear to be detrital monazite in these samples. The older monazite population (≥ 393 Ma) generally have the least depleted HREE patterns (Fig. 9a, b, d, h), indicating that they grew in the presence of fewer HREE-rich minerals like garnet (Rubatto et al., 2013; Taylor et al., 2015). Furthermore, these rocks have no metamorphic zircon of this age; rather, they contain detrital igneous zircon that are the same age as many of the older monazite (Figs. 7 and 9). These monazite grains must be detrital and therefore provide an additional constraint on the maximum deposition age of the original sediment, placing that date at ca. 390 Ma, and they could be sourced from either igneous or metamorphic rocks associated with subduction as the Rheic Ocean closed, though the more abundant HREE point to the former.

3. Variscan Metamorphism

The onset of major metamorphic zircon and monazite growth occurred at ca. 330 Ma, which corresponds to a period of collision during the Variscan orogeny (Be Mezeme et al., 2006; Faure et al., 2010, 2014; Laurent et al., 2017). The scattered metamorphic zircon and monazite older than 330 Ma likely represent mixing of detrital and metamorphic ages. In fact, the monazite analyses with $^{208}\text{Pb}/^{232}\text{Th}$ dates between ca. 361 and 330 Ma are found in grains that also have analysis spots from the older detrital population, and these analyses

have less depleted HREE patterns than the majority of the metamorphic population. Some, but not all, of the metamorphic monazite <330 Ma also have less depleted HREE, but these too may represent mixing with the older population. In samples that contain the older population, the vast majority of <330-Ma analyses that have higher HREE come from grains that also have analyses from the older population. In samples that do not contain the older population, the vast majority of <330-Ma analyses with higher HREE come from grains that also have analyses >330 Ma in age and may thus still represent mixing. Both metamorphic zircon and monazite demonstrate that the original sediments were definitively transported to the lower crust during the collisional phase of the Variscan orogeny, which began by 360 Ma, and before post-orogenic collapse, which commenced at ca. 310–300 Ma (Faure, 1995; Laurent et al., 2023; Ledru et al., 2001; Malavieille et al., 1990).

Metamorphic zircon and monazite continued growing until at least 260 Ma, reflecting an extended period of crystallization during slow cooling until the lower crust reached 800 °C at 265 Ma (Laurent et al., 2023; Rossi et al., 2006). Some monazite grown during this period exhibit an excess of ^{206}Pb , which is caused by the incorporation of ^{230}Th into the crystal structure. As ^{230}Th is an intermediate decay product of ^{238}U , the excess ^{206}Pb points to a U-rich precursor mineral such as xenotime or coffinite, though the ultimate bulk composition and processes responsible remain enigmatic.

4. Post-Variscan Metamorphism

Younger metamorphic zircon and monazite (<260 Ma) presumably represent mixing of older material with younger domains influenced by subsequent perturbations of the local lower crust, including episodic rifting associated with the separation of the African and Eurasian plates between 250 and 175 Ma (Rossi et al., 2006; Simonetti et al., 2023), as well

as the on-going Alpine orogeny (Dewey et al., 1973; Lardeaux, 2014; Rosenbaum et al., 2002). Earlier work on the Bournac metasedimentary xenoliths also yielded zircon dates as young as 140 Ma and attributed them to these post-Variscan events (Laurent et al., 2023; Rossi et al., 2006), which may have caused Pb loss or recrystallization of older zircon and monazite.

5. Dating the Bournac Volcanic Pipe

The rutile ages recorded by these metapelites can be used to indirectly date the eruption of the Bournac volcanic pipe. If rutile present in the lower crust resides above its Pb closure temperature (~ 600 °C at most in natural samples: Apen et al., 2020; Cherniak, 2000; Schutt et al., 2018), it will not retain radiogenic Pb until it cools via exhumation or rapid transport to the surface after entrainment by a magma. In the case of xenolith entrainment, rutile will begin retaining radiogenic Pb immediately following eruption and therefore yield an age corresponding to the time of eruption. While it is possible that heating during entrainment could yield Pb loss from rutile, the time frame is typically too short for any significant Pb loss (Schmitz & Bowring, 2003).

The alkali basalt pipe at Bournac was dated to ca. 7 Ma by Couthures et al. (1991) using K-Ar dating. However, similar ages for other Massif Central volcanic pipes have proven incorrect by application of more modern dating techniques, and some authors advise caution when interpreting the older dates (e.g., Lenoir et al., 2000). Indeed, the data presented herein point to an older eruption age for the Bournac volcanic pipe of $11.6 \text{ Ma} \pm 0.7 \text{ Ma}$, the average of rutile U-Pb dates for seven samples (Fig. 10).

6. Sediments in the Lower Continental Crust

The onset of metamorphism at ca. 330 Ma marks the beginning of sediment transport to the lower crust. Thermobarometry from these samples points to equilibration in the deep continental crust and conditions consistent with a warmer geothermal gradient, as opposed to the cooler geotherm of a subduction zone (Fig. 11). The presence of sillimanite is also inconsistent with subduction. Perhaps most importantly, the first period of metamorphism recorded by the xenoliths was clearly during the collisional phase of the Variscan orogeny. The fact that the rutile were losing Pb up until eruption at 11.6 Ma shows that the samples remained at depth until then and were part of the lower continental crust for over 300 Ma. Presumably, the bulk of the metasediment remains in the lower crust to this day.

These results are similar to other work by the authors (Ringwood et al., 2024) from North America. Metapelitic xenoliths from New Mexico, USA, and Chihuahua, Mexico, represent lower crustal metasediments that were transported to depth during continent-continent collision. Interestingly, these metapelites also experienced UHT metamorphism in the deep crust (Cipar et al., 2020), though the timing of collision there was uncertain and could only be inferred using the transition from detrital to metamorphic dates. In contrast, the well-constrained history of the Variscan orogeny allows for an even more robust demonstration of sediment transport to depth occurring within a collisional orogen.

Continent-continent collisions appear to be an important setting for adding felsic material to the stable lower continental crust, which has implications for continental evolution and composition. Under known constraints (seismic velocity/ density, surface heat flow data, etc.), the lower continental crust may be made up of as much as 10–20% sedimentary material (Hacker et al., 2011). Significant sediment in the deep crust would

mean a more heterogeneous lower crust composition than is generally assumed, and such a lower crust would contribute more towards crustal heat production. This would, in turn, imply less heat flux from the mantle to the crust for a given surface heat flow and lower Moho temperatures (Apen et al., 2020). The inaccessibility of the deep crust makes determining its bulk composition challenging, but better understanding how and when sediments can be incorporated into the lower continental crust furthers this endeavor.

G. Conclusions

Detrital igneous zircon from metapelitic xenoliths found at the Bournac volcanic pipe in the Massif Central, France, were sourced from Gondwanan affinity rocks and deposited as early as ca. 393 Ma. Metamorphic monazite and zircon record major metamorphism of the metapelites beginning around 330 Ma and continuing to 260 Ma, reflecting an extended period of recrystallization as the hot lower crust cooled from peak temperatures of 940–970 °C at 313 Ma down to 800 °C at 265 Ma (Laurent et al., 2023; Rossi et al., 2006). The onset of metamorphism at 330 Ma documents the beginning of transport of the sediments to the deep crust during the collisional phase of the Variscan orogeny. These samples remained part of the stable lower continental crust in the Massif Central until the eruption of their host magma in the Neogene, though they did experience minimal perturbation by subsequent metamorphic events that impacted the lower crust, including episodic rifting between 250–175 Ma and earlier phases of the Cenozoic Alpine orogeny. Rutile from the metasediments record their eventual entrainment and eruption at ca. 11.6 ± 0.7 Ma, significantly earlier than the previously determined date of 7 Ma for the basaltic breccia (Couthures et al., 1991). Moreover, the fact that rutile was open to Pb diffusion until entrainment and eruption

demonstrates that these samples derive from present-day lower crust rather than nearby high-grade metamorphic rocks of the upper crust. This study provides yet another example of sediments being added to lower continental crust during a collisional orogeny and their long-term residence at depth (in this case, for over 300 Ma). The incorporation of felsic material into the lower crust via collisional orogeny helps explain the continental crust's evolution over time and points to significant heterogeneity of the present-day lower continental crust.

V. Summary and Future Work

A. Summary and Conclusions

Collectively, the case studies documented herein provide examples of how felsic sedimentary material is transported to depth and incorporated into the lower continental crust. In all three cases, continent-continent collision was the process responsible, and examples of sediment transport to depth via shallow underplating in the forearc, UHP metamorphism during relamination, or deep-seated thrust faulting in a retroarc were not found.

Garnet-sillimanite metapelites from the Potrillo volcanic field provided insight into the enigmatic Picuris orogeny. The youngest detrital zircon in these samples must have been sourced from an arc during the subduction phase of the Picuris orogeny, and the end of detrital zircon production and beginning of metamorphic zircon and monazite growth records the transition of the Picuris from a magmatic arc to continent-continent collision. Transport of the original sediments to the lower crust occurred during this collision, and their presence in the local deep crust points to the Nd line being the suture zone created by the Picuris orogeny. The sediments remained in the deep continental crust for over 1400 Ma, until magmatism within the Rio Grande rift returned them to the surface.

Both metapelitic and mafic granulite xenoliths from the Missouri Breaks diatremes and Bearpaw Mountains volcanic field revealed a geologic history over 3000 Ma in length. The unusual kyanite-scapolite-bearing mafic samples were sourced from arc cumulates and recorded a history of crustal thickening during orogenesis, and rutile from both the mafic and metapelitic samples were used to date their volcanic host rocks to ca. 46 Ma (Big Slide) and ca. 51 Ma (Robinson Ranch). The sample from Robinson Ranch chronicled a 2200–

2000 Ma metamorphic event under conditions consistent with burial/ metamorphism in a rift setting, while younger samples documented the transition from subduction to collision during the Great Falls orogeny at ca. 1800 Ma. Additionally, zircon and monazite from the metapelitic samples reveal a history of metasomatism in the local lower crust during the Laramide orogeny, prior to their eventual entrainment by magmas after 1700+ Ma in the lower crust.

Garnet-sillimanite metapelitic xenoliths from Bournac were incorporated into the lower continental crust during the Variscan orogeny. This is perhaps the most robust example of the case studies, as the Variscan orogeny is extremely well-studied, and the tectonic setting at the time of metamorphism recorded in the samples is unequivocal. These samples contain detrital zircon from as long ago as the late-Archean, with the bulk of these being sourced from Gondwana or other terranes that derived from Gondwana, while the youngest detrital component (as young as 393 Ma) probably formed during subduction as the Rheic ocean closed between Gondwana and Laurussia. Metamorphic zircon and monazite growth began at ca. 330 Ma, recording the transport of the original sediments from the surface to depth during the collisional phase of the Variscan orogeny and before post-orogenic collapse began. These rocks remained in the lower continental crust for over 300 Ma.

B. Future Work

These case studies demonstrated the transport of sediments from the surface to the deep crust during collisional orogenies at ca. 1450, ca. 1800, and ca. 330 Ma. Continent-continent collisions are therefore likely an important mechanism for adding felsic material to the stable lower continental crust and contribute to the overall growth and evolution of the

continents over time. However, the ubiquity of this and other processes are still unknown. It would be prudent to study additional high and ultra-high pressure metasedimentary xenoliths or terranes to explore any differences across Earth's history and tectonic settings. Useful data may already exist, as other studies have investigated the same rocks and generated similar data (e.g., Laurent et al., 2023, for Bournac), but are simply not asking the question: "How did the sediments get to depth in the first place?"

Furthermore, seeking out samples that could represent sedimentary material being transported to depth by relamination, retroarc thrusting, or forearc underplating would be useful to explore mechanisms other than collision. Relamination, in particular, is worth examining, because it is the least well-demonstrated of these processes in the real world. It is possible that most sediments subducted to ultra-high pressures are melted and assimilated into magmas and never have the opportunity to relaminate. Similarly, it is likely that the local mantle lithosphere needs to be removed in order for sediments to relaminate to the base of the crust, in which case it may happen less often simply due to a lack of the necessary conditions at the time of orogenesis.

Nonetheless, understanding the ways in which felsic material can be added to the continental crust, particularly the lower crust, and how this has changed over time and differs across regions, contributes to understanding the formation and evolution of the continents. How has their composition changed over time? What are the processes responsible? Have these changed over time? How much metasediment is in the lower crust? How does that affect bulk composition and crustal heat production? Synthesizing a single answer to all of these questions may only be done through continued work exploring the multitude of smaller questions explored by studies such as those described here.

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Appendix

A. Chapter 2 Supplementary Text

1. Petrographic Information for All Samples Analyzed

Sample	Major Phases	Accessory Phases	Notes
18KH1 (K)	gt (20%), q (40%), sil (20%), kfs (15%), pl (<5%)	rt, ap, zrn, mnz, ilm, oxides	Gts mostly unaltered; one of samples in best shape
18KH9 (K)	gt (20%), q (45%), sil (20%), kfs (15%)	rt, ap, zrn, mnz, ilm, oxides	Relatively inclusion-free gts
18KH10 (K)	gt (15%), q (50%), sil (15%), kfs (20%), pl (1%)	rt, ap, zrn, mnz, ilm, sulfides	Gts small (~2mm); intact gt cores; perthitic kfs
117848-125 (K)	gt (20%), q (40%), sil (15%), kfs (20%), pl (<5%)	rRt, ap, mnz, zrn, oxides, sulfides	Only rims of gt altered
117848-136A (K)	gt (25%), q (40%), sil (5%), kfs (20%), pl (4%), bio (1%)	rt, bio, zrn, oxides, pyx, melt	Gts heavily altered; perthitic kfs; symplectites; only sample with biotite (intermingled with pyx and melt?); bio and pyx possibly secondary
117848-286 (P)	gt (30%), q (20%) sil (45%), kfs (5%)	rt, zrn, mnz, ilm, sulfides	Gts mostly altered; blue rutile
117848-309 (K)	gt (15%), q (45%), sil (10%), kfs (15%), pl (10%)	rt, ap, mnz, zrn, ilm, oxides, sulfides	More pl than typical; lots of large relatively intact gt cores
* K = Kilbourne Hole, P = Potrillo Maar. Ap = apatite, bio = biotite, gt = garnet, ilm = ilmenite, kfs = potassium feldspar, mnz = monazite, pl = plagioclase, pyx = pyroxene q = quartz, rt = rutile, sil = sillimanite, zrn = zircon			

2. Blue Rutile

Blue rutile was discovered in four of the metapelite samples (Fig. S1), two of which also have typical brown rutile. Both varieties of rutile were analyzed using the Cameca SX-100 electron microprobe (EPMA) at UCSB to determine whether any unusual chemistry could explain the blue color. Magnesium, Al, Si, Fe, Ti, Zr, V, Cr, Nb, Ta were all analyzed.

The blue and brown rutile have variable Nb contents. The blue grains contain as much as 1.1 wt% Nb, whereas the brown grains all have less than 0.5 wt% (Fig. S2). There is also significant variation among the blue rutile. The mean and standard deviation of Nb content

Blue-type Rutile

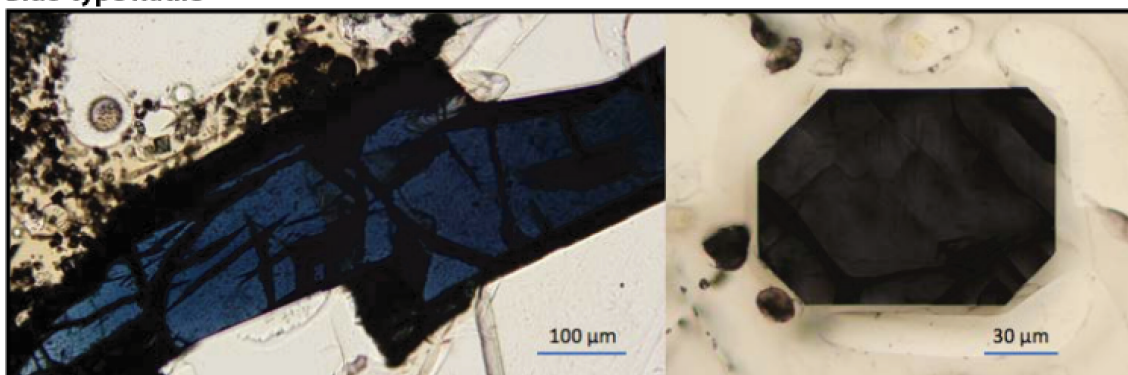


Figure S1. Examples of the blue-type rutile found in sample 286. Panel B is looking down the c-axis.

of the pure blue (e.g., left grain in Fig. S1) is 4990 ± 2320 ppm and ranges from approximately 2100 to 11,000 ppm. However, including all of the blue-type rutile (blue, purple, green, gray), the average Nb content is 4190 ± 2280 ppm. Though there appears to be a difference between the pure blue and other blue-type rutile, with the purple and green grains having less Nb, it is also possible that this is a coincidence and that the color difference is only the result of optic axis orientation. By applying an unpaired t-test to this limited set of data, it was determined that these two populations are not statistically different.

Whereas the brown rutile has an average Nb concentration of 3550 ± 1000 ppm and a maximum measured Nb concentration of < 500 ppm, it is clear that the blue-type rutile can contain significantly more Nb (Fig. S2). However, it is probable

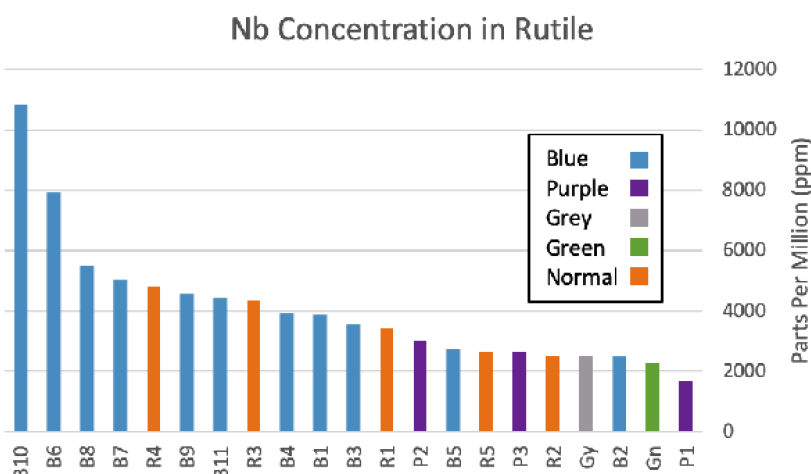


Figure S2. Nb concentration in rutile grains measured via EPMA and sorted by perceived color.

that the blue color is not due to the presence of additional Nb. Rather, the two are likely the result of the same thing: the presence of Ti^{3+} in the rutile crystal lattice. While there is only one other documented occurrence of natural terrestrial blue rutile (Mposkos and Kostopoulos, 2001¹), there are multiple instances of synthetic blue rutile containing Ti^{3+} documented in the materials science literature (e.g., Khomenko et al., 1998²). If Ti^{3+} is the cause of the blue color in these rutile, it would be possible to explain the high Nb content by a coupled substitution of Nb^{5+} and Ti^{3+} for 2Ti^{4+} .

3. All Zircon Data

All zircon data collected from the four samples (both detrital and metamorphic) are shown in Fig. S3 to demonstrate the strongly discordant nature of most analyses.

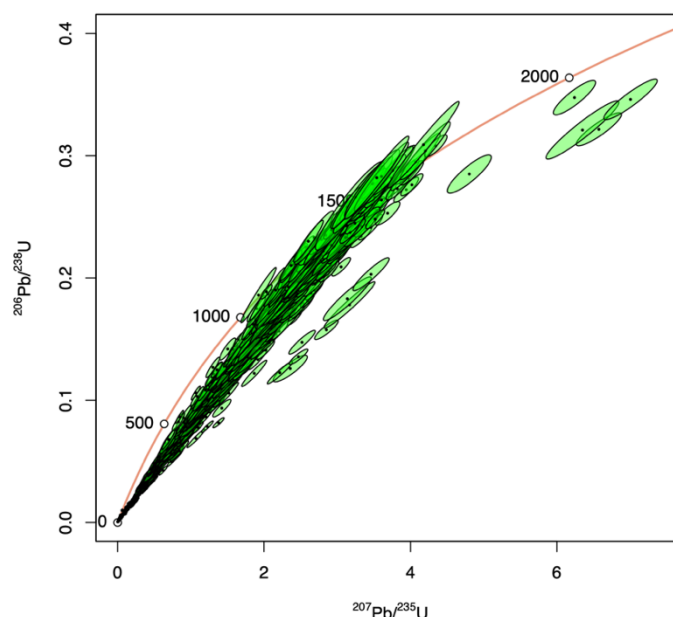


Figure S3. All zircon U-Pb data collected having <15% uncertainty.

¹ Mposkos, E.D., and Kostopoulos, D.K., 2003, Diamond, former coesite and supersilicic garnet in metasedimentary rocks from the Greek Rhodope: a new ultrahigh-pressure metamorphic province established: *Earth and Planetary Science Letters*, v. 214, p. 675–678, doi:10.1016/S0012-821X(03)00378-9.

² Khomenko, V.M., Langer, K., Rager, H., and Fett, A., 1998, Electronic absorption by Ti^{3+} ions and electron delocalization in synthetic blue rutile: *Physics and Chemistry of Minerals*, v. 25, p. 338–346.

4. Garnet Compositions

All samples for which data was collected have been plotted in Fig. S4 to show composition (Alm-Py-Grs content).

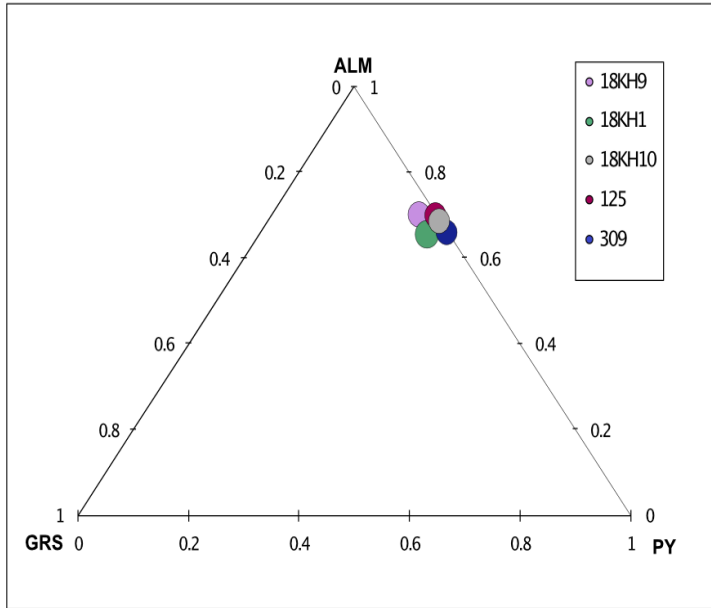


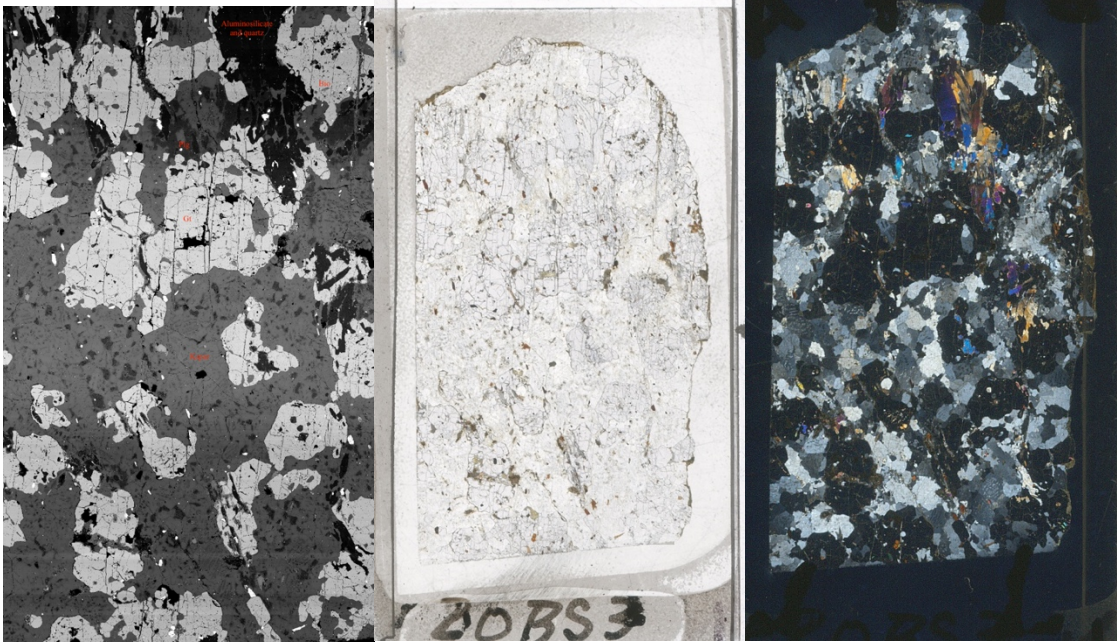
Figure S4. Composition of garnet grains by sample. Dots are averages for individual samples and encompass uncertainty. Samples 125 and 309 are SI samples with the prefix 117848.

B. Chapter 3 Supplementary Text

1. Metasedimentary Sample Information

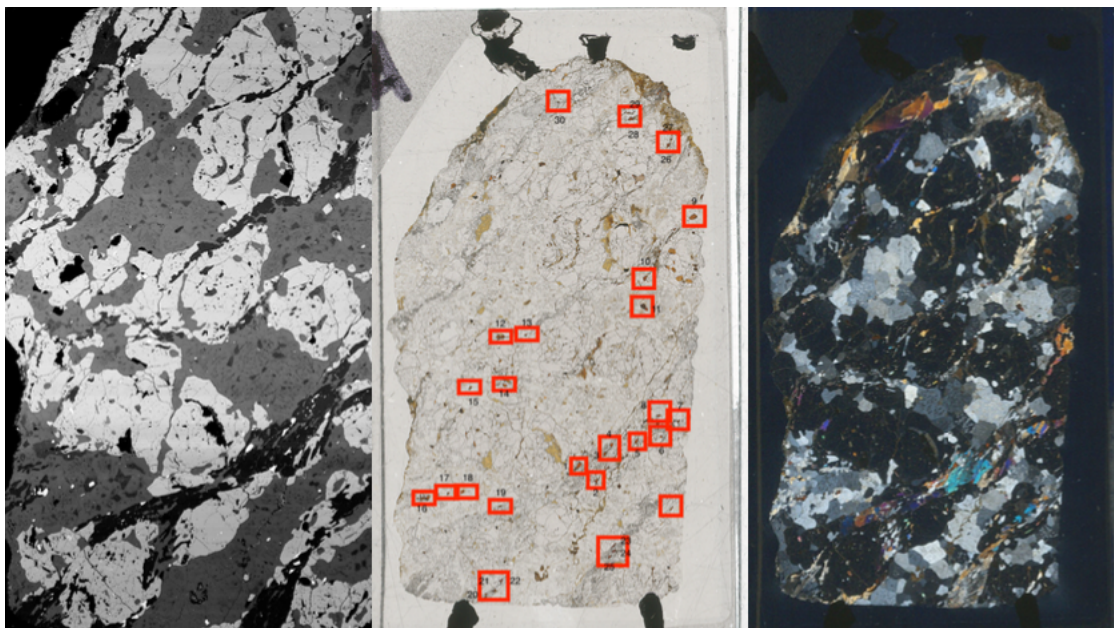
Backscattered electron maps of thin sections, plain-polarized photomicrographs, and cross-polarized photomicrographs are shown above the descriptions of each sample. Labelled plain-polarized and cross-polarized photomicrographs taken at higher magnification are shown below sample descriptions for samples 20BS4 and ROB-2.

20BS3:

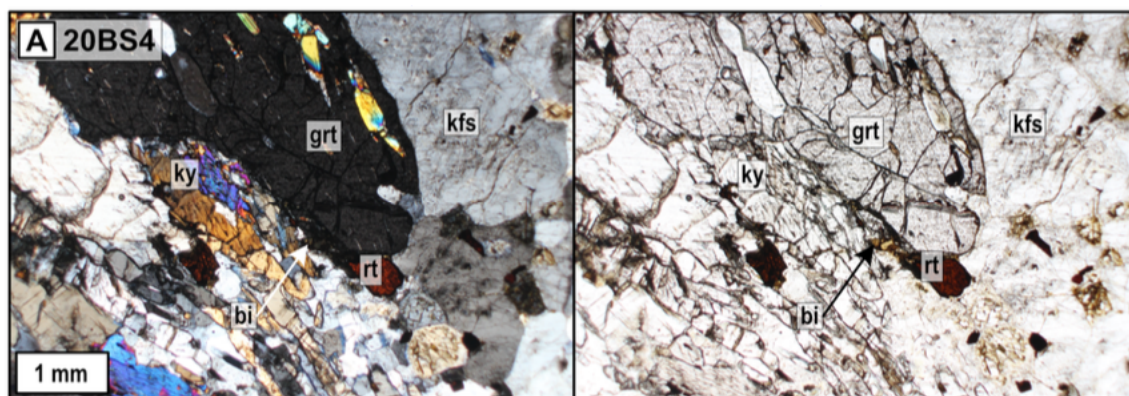


Description: 20BS3 is slightly deformed and primarily contains garnet, plagioclase, potassium feldspar, quartz, kyanite, biotite, and rutile. Monazite, zircon, and apatite are found both within the matrix and as inclusions in other minerals. Kyanite and quartz are mostly found together and are generally oriented parallel with the long axis of the thin section. Potassium feldspar is found throughout and contains patchy areas of exsolved plagioclase. Garnet grains contain numerous inclusions, but almost non-existent rim alteration. Generally, there is very minimal alteration along grain boundaries.

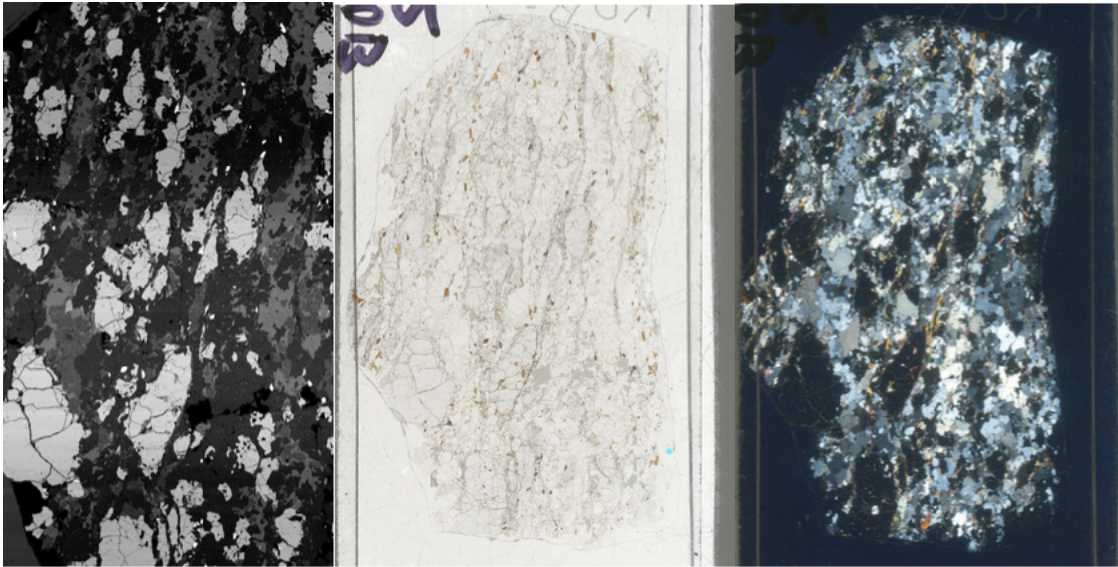
20BS4:



Description: 20BS4 is slightly deformed and primarily contains garnet, plagioclase, potassium feldspar, quartz, kyanite, biotite, and rutile. Monazite, zircon, and apatite are found both within the matrix and as inclusions in other minerals. Kyanite and quartz are mostly found together and are generally oriented diagonal to the long axis of the thin section. Potassium feldspar is found throughout and contains patchy areas of exsolved plagioclase. Garnet grains contain numerous inclusions, but almost non-existent rim alteration. Generally, there is very minimal alteration along grain boundaries.



ROB-2:

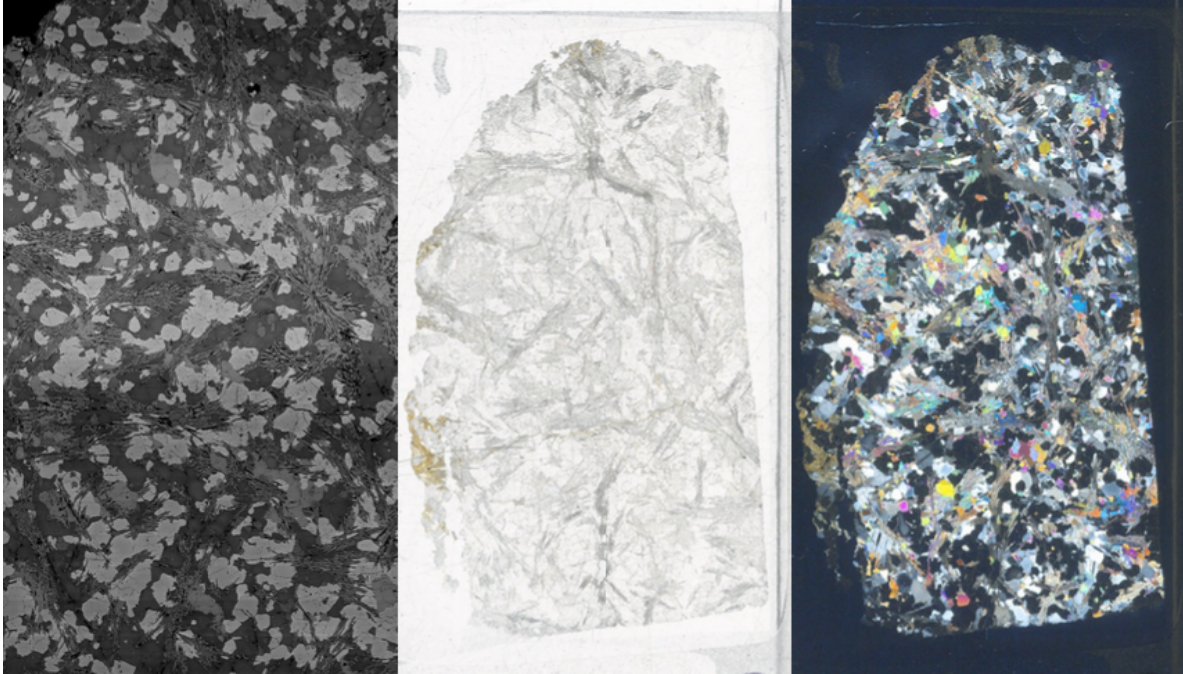


Description: ROB-2 is slightly deformed, with minerals largely aligned parallel to the long axis of the thin section. It primarily contains garnet, plagioclase, potassium feldspar, quartz, sillimanite, biotite, and rutile. Monazite, zircon, and apatite are found both within the matrix and as inclusions in other minerals. There is an order of magnitude more quartz than 20BS3 and 20BS4, and grain sizes tend to be smaller than in those samples. Potassium feldspar is found throughout and, in some places, appears to surround plagioclase. Garnet grains contain numerous inclusions, but almost non-existent rim alteration. Generally, there is very minimal alteration along grain boundaries.

2. Mafic Sample Information

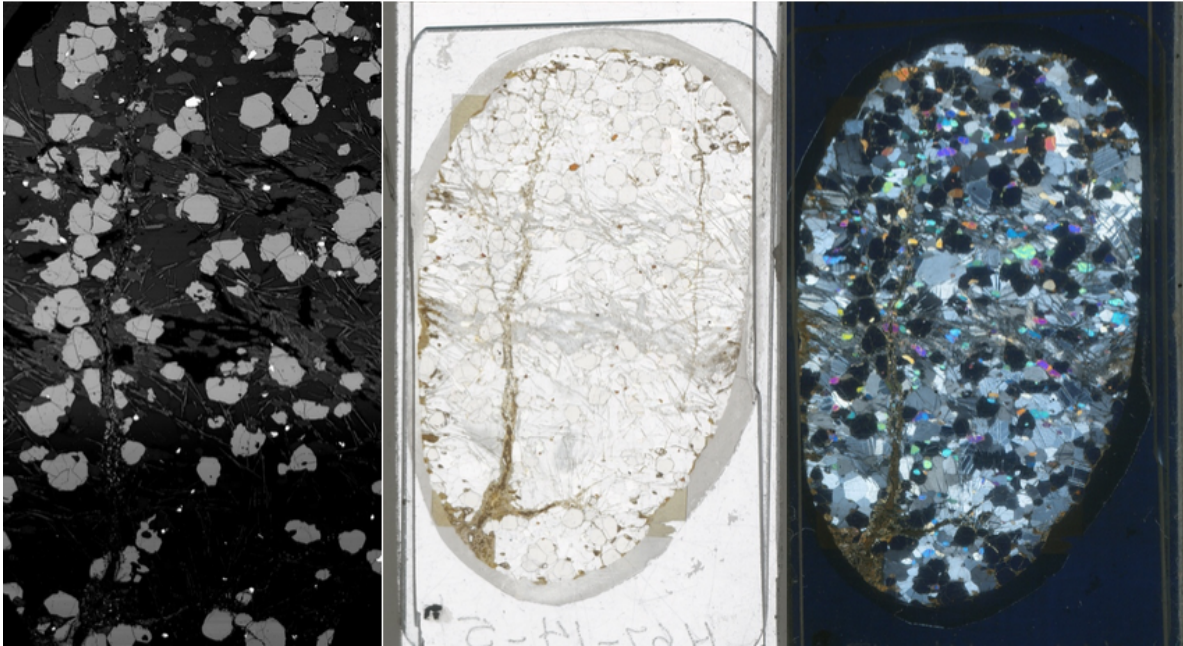
Backscattered electron maps of thin sections, plain-polarized photomicrographs, and cross-polarized photomicrographs are shown above the descriptions of each sample.

20BS19:



Description: 20BS19 primarily contains garnet, clinopyroxene, plagioclase, kyanite, and silvialite, with minor amounts of clinozoisite and amphibole, as well as apatite and rutile. Both garnet and plagioclase are crosscut by intergrowths of kyanite and clinopyroxene, though blockier clinopyroxene unassociated with kyanite is also present. Clinozoisite is found in a single area and appears to be breaking down, and there are only a few grains of amphibole present, which tend to be rimmed by the clinopyroxene-kyanite intergrowths. Like H67-14-5, 20BS19 contains rutile, but only as <10 micron grains.

H67-14-5:



Description: H67-14-5 contains garnet, clinopyroxene, plagioclase, kyanite, silvialite, clinozoisite, quartz, and rutile, as well as apatite, barite, and spinel. Veins of alteration cut across the sample, and the fresher areas contain two zones: The first contains garnet, scapolite, clinopyroxene, and plagioclase, while the second is also composed of garnet, scapolite, and plagioclase, as well as elongate clinozoisite and fine-grained areas of quartz and kyanite. Clinozoisite crosscuts plagioclase, and the fine-grained kyanite and quartz appear to be growing at the expense of the clinozoisite and plagioclase. Like 20BS19, H67-14-5 contains rutile, but as much larger macroscopic grains.

3. Supplementary Figures

Zircon and monazite depth profiles are largely confined to nearly concordant Proterozoic analyses starting to trend back toward younger dates, except for a single profile containing concordant Laramide-age analyses (Fig. S1).

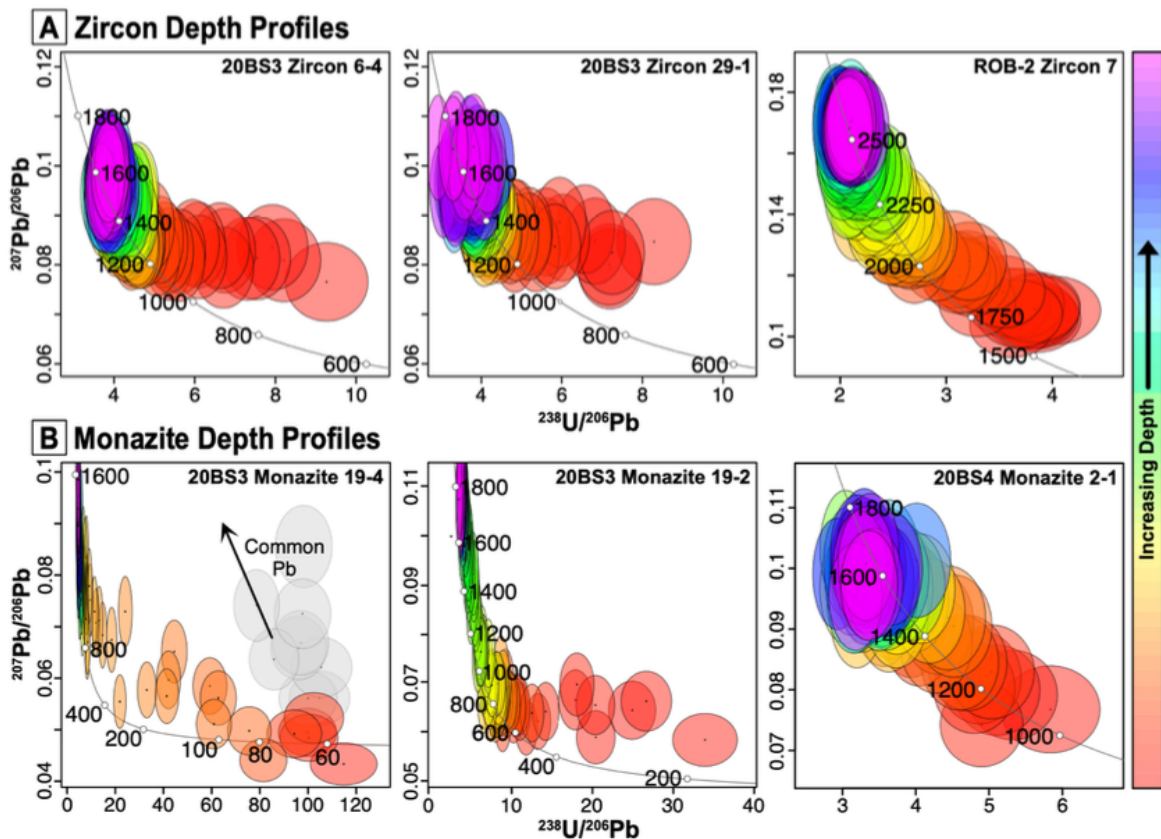


Figure S1. Examples of zircon and monazite depth profiling results: (A) Zircon profiles from 20BS3 (left and central panel) and ROB-2 (right panel). (B) Monazite profiles from 20BS3 (left and central panels) and 20BS4 (right panel). Color ramp corresponds to depth of the analyses: Colder colors = deeper. Hotter colors = shallower. Error ellipses show 2σ total uncertainty for individual analyses. All samples record concordant or near-concordant Proterozoic analyses, and 20BS3 demonstrates the most significant discordancy, though there is variability from profile to profile.

Trace element patterns for 20BS3, 20BS4, and ROB-2 are show in Figure S2.

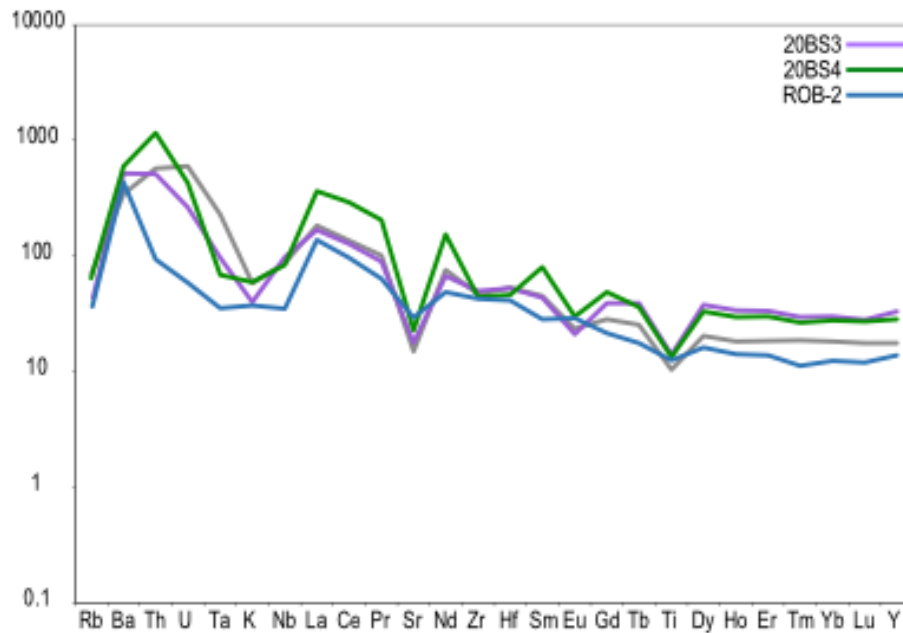


Figure S2. Whole-rock trace-elements for the metapelite samples (chondrite normalized: McDonough and Sun, 1995). Post-Archean Shale plotted for comparison using the compilation of Li et al. (2016).

C. Chapter 4 Supplementary Material

1. Supplementary Text

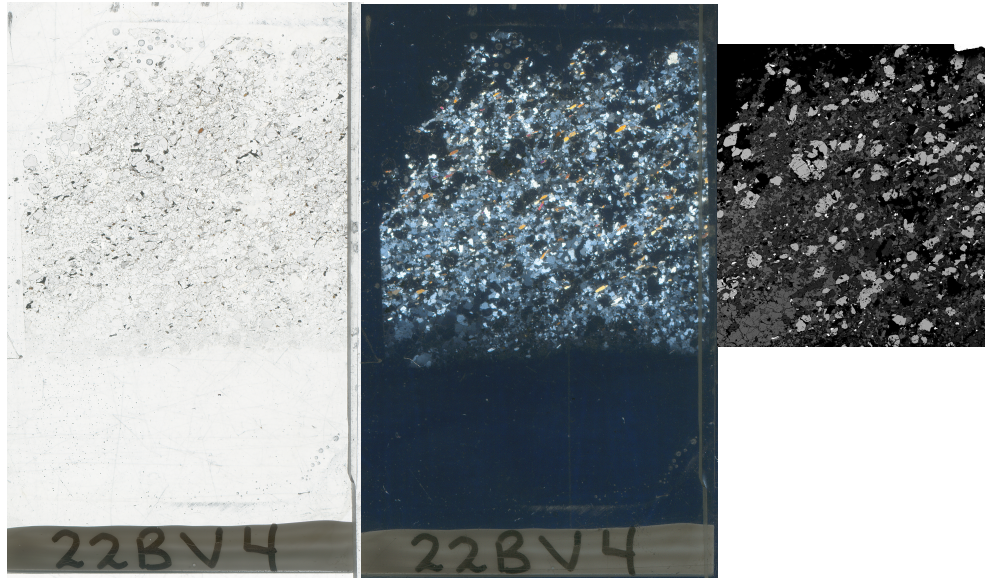
Comparison of whole-rock compositions obtained via XRF to those calculated from modal mineralogy and mineral composition shows that they are consistent.

Oxide (%)	SiO ₂	TiO ₂	Al ₂ O ₃	FeO	MnO	MgO	CaO	Na ₂ O	K ₂ O
22BV4 (XRF)	62	1.2	18	10	0.07	3.6	0.85	0.78	2.1
22BV4 (calc)	64	1.7	19	6.6	0.06	3.0	1.3	2.0	2.9
22BV9 (XRF)	62	1.1	20	9.4	0.08	3.6	0.74	0.73	2.6
22BV9 (calc)	64	1.4	18	7.2	0.07	3.2	1.1	1.5	2.6
22BV11 (XRF)	65	1.0	18	7.7	0.08	3.2	1.4	1.3	2.5

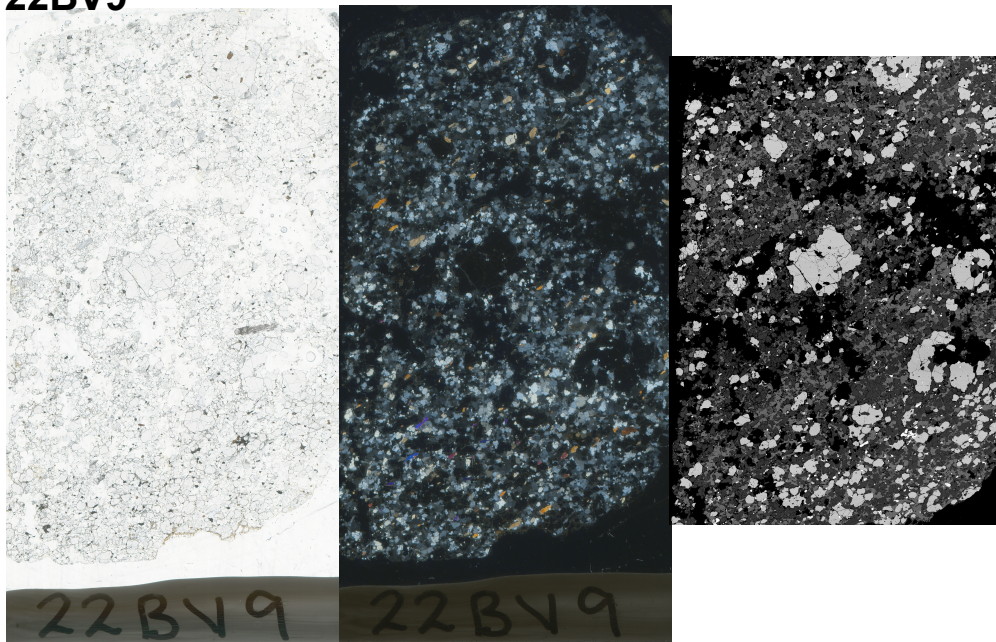
22BV11 (calc)	65	1.5	18	5.4	0.07	2.6	1.8	2.3	2.7
22BV13 (XRF)	60	1.2	19	9.5	0.10	3.6	1.4	1.7	3.5
22BV13 (calc)	61	1.7	19	7.9	0.10	3.3	1.5	2.2	3.2
Bo.93.8 (XRF)	61	1.1	19	8.1	0.53	2.1	2.0	2.2	3.2
Bo.93.8 (calc)	62	1.5	19	6.2	0.67	2.4	2.2	2.1	2.9
Bo.94 (XRF)	70	1.1	16	6.0	0.05	2.6	0.82	1.0	2.2
Bo.94 (calc)	68	1.0	17	5.9	0.06	2.7	1.2	1.7	2.4

Table 1. Comparison of whole-rock composition obtained via XRF and calculated from modal mineralogy and mineral composition

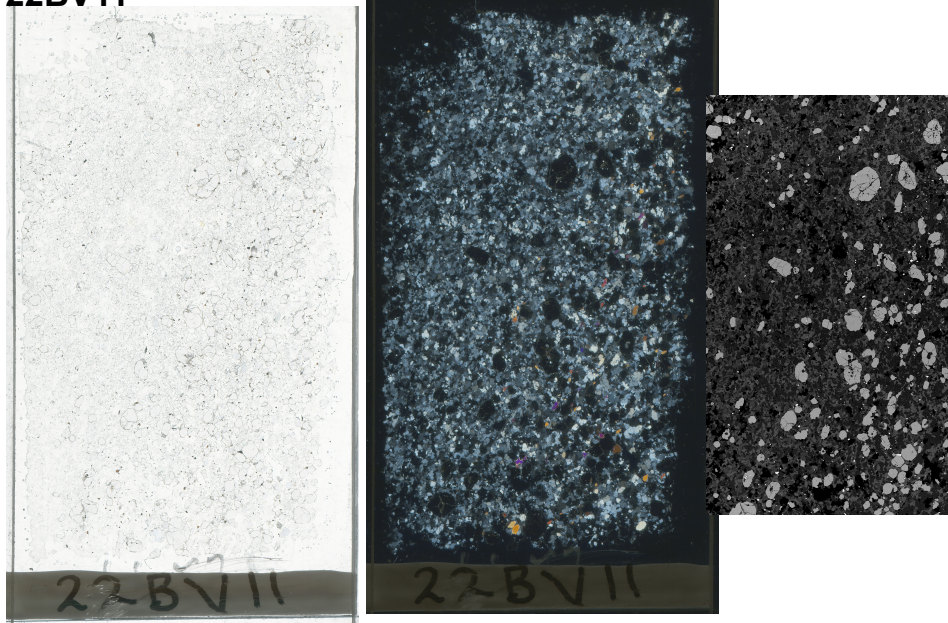
22BV4



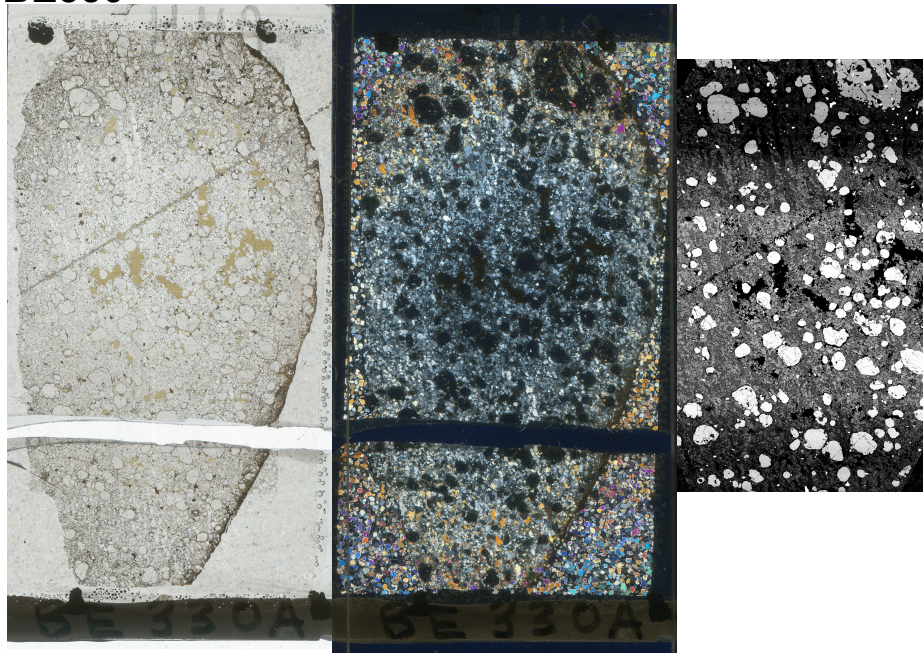
22BV9



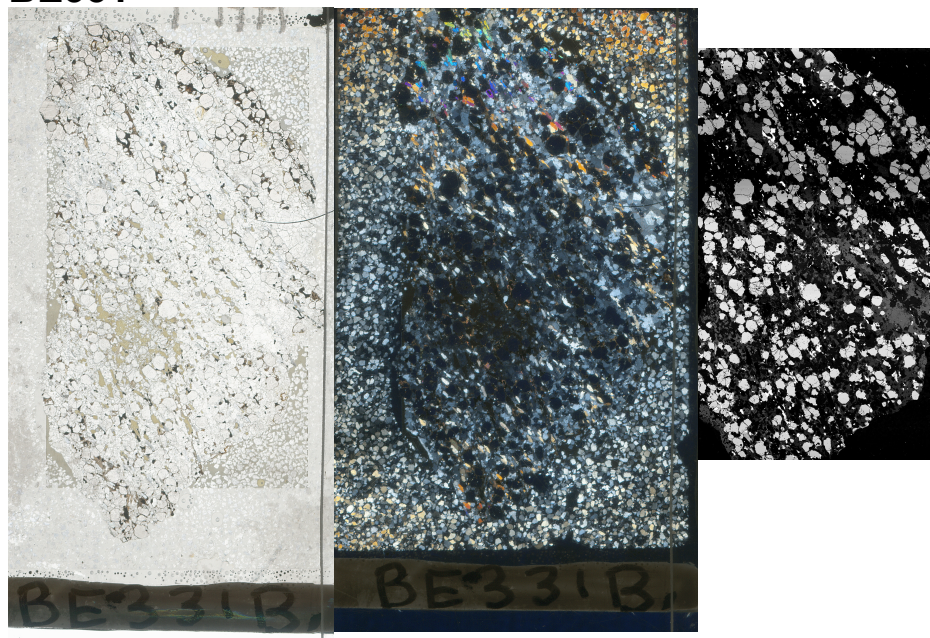
22BV11



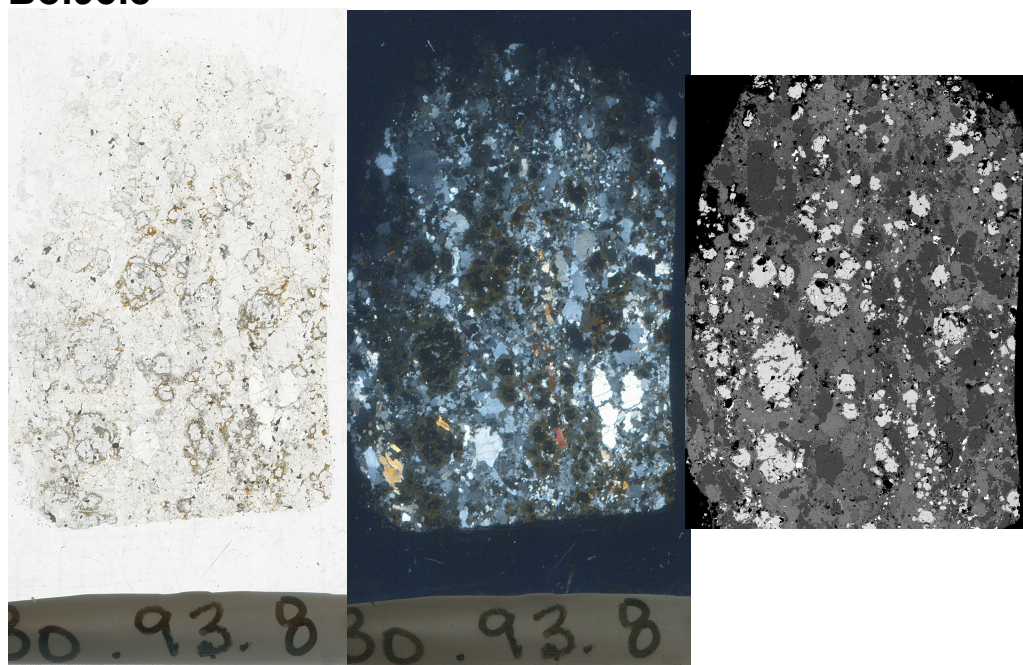
BE330



BE331



Bo.93.8



Bo.94

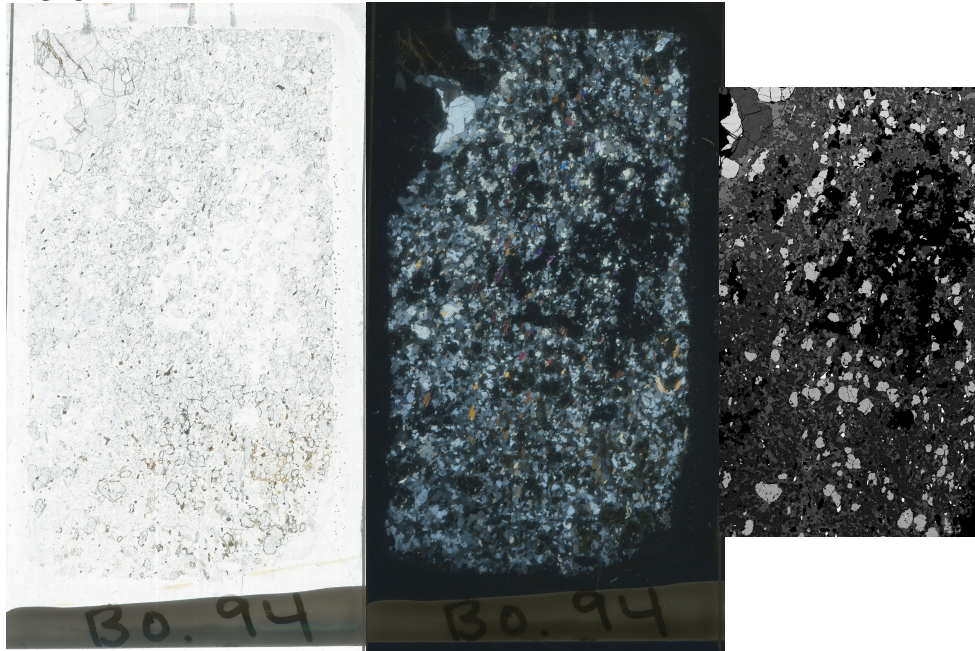


Figure 1. Images of sample thin sections: Left images are PPL scans, center are XPL scans, and right are BSE scans

Whole-rock trace-elements from samples are consistent with post-Archean Australian shale, indicating that they may be related (Fig. S2).

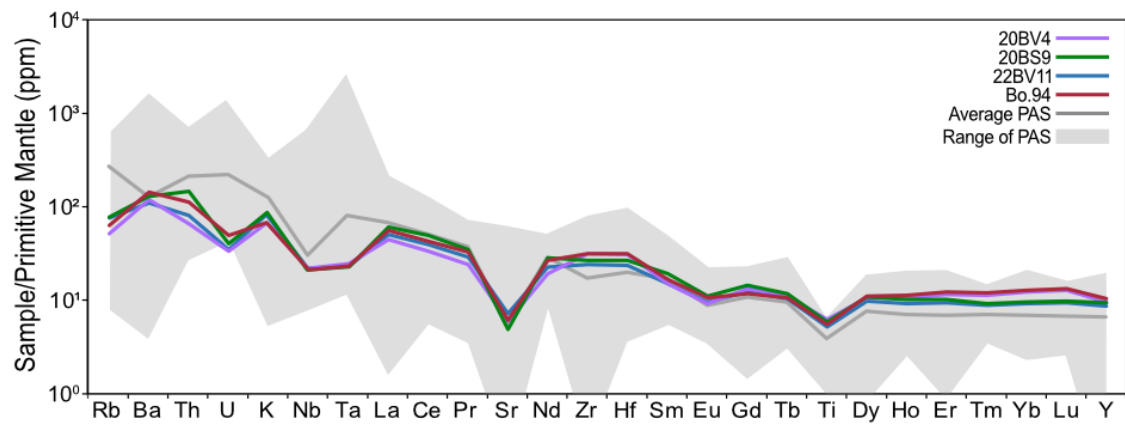


Figure S2. Whole-rock trace-elements compared to post-Archean Australian Shale.

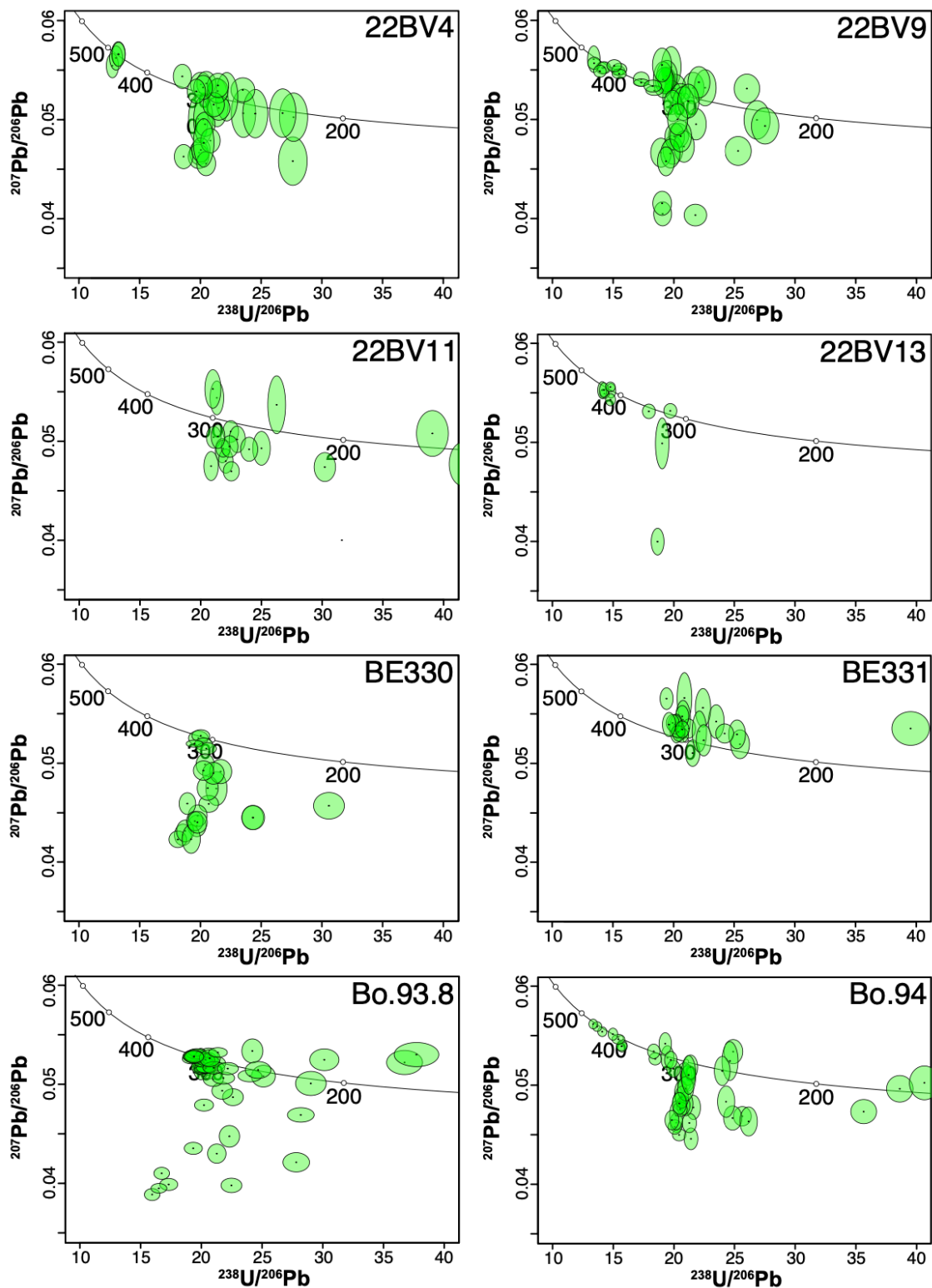


Figure S3. Terra-Wasserburg concordia diagrams for monazite. Ellipses show 2σ total uncertainty for individual spot analyses. Reversely discordant data points trending toward the bottom left are due to excess ^{206}Pb .

D. Full Data Tables

1. Chapter 2 Data

Full data tables for chapter 3 are included in the Excel file named “Chapter_2_Tables.”

These include the following: (1) monazite LASS data, (2) monazite LASS standard data, (3) zircon LASS data, (4) zircon LASS standard data, (5) XRF whole-rock data, (6) rutile EPMA data, (7) feldspar and garnet EPMA data, (8) garnet LA-ICP-MS data.

2. Chapter 3 Data

Full data tables for chapter 4 are included in the Excel file named “Chapter_3_Tables.”

These include the following: (1) monazite LASS spot data, (2) monazite standard LASS spot data, (3) zircon LASS spot data, (4) zircon standard LASS spot data, (5) rutile LASS data, (6) rutile standard LASS data, (7) rutile EPMA data, (8) garnet, feldspar, clinopyroxene, biotite, scapolite, clinozoisite, and amphibole EPMA data, (9) garnet LA-ICP-MS data, (10) whole-rock LA-ICP-MS data, (11) monazite depth profile LASS U-Pb data, (12) monazite depth profile LASS trace-element data, (13) monazite standard depth profile LASS U-Pb data, (14) monazite standard depth profile LASS trace-element data, (15) zircon depth profile LASS U-Pb data, (16) zircon depth profile LASS trace-element data, (17) zircon standard depth profile LASS U-Pb data, (18) zircon standard depth profile LASS trace-element data.

3. Chapter 4 Data

Full data tables for chapter 4 are included in the Excel file named “Chapter_4_Tables.”

These include the following: (1) monazite LASS data, (2) monazite LASS standard data, (3) zircon LASS data, (4) zircon standard LASS data, (5) rutile LASS and LA-MC-ICP-MS

data, (6) rutile standard LASS and LA-MC-ICP-MS data, (7) rutile EPMA data, (8) garnet, feldspar, and biotite EPMA data, (9) whole-rock LA-ICP-MS data.