



A magnetite reference material for LA-ICP-MS U–Pb geochronology from the Troodos Ophiolite, Cyprus

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Abstract. Magnetite is an increasingly important target material for in situ geochemical analysis and has recently emerged as a promising U–Pb geochronometer. However, the wider application of the method is hindered by the lack of a widely available matrix-matched reference material. Here we propose TR41, a hydrothermal magnetite from the Troodos ophiolite (Cyprus), as a candidate reference material for LA-ICP-MS U–Pb magnetite geochronology. Petrographic, geochemical, and isotopic data demonstrate that TR41 is sufficiently homogeneous for laser ablation analyses and yields clearly defined regressions in Tera–Wasserburg diagram despite U concentrations below 1 $\mu\text{g g}^{-1}$. As independent ID-TIMS characterisation of magnetite is currently not feasible, the age of TR41 was constrained using complementary geochronological approaches. U–Pb dating of coexisting hydrothermal andradite yields a lower-intercept age of 92.04 ± 2.23 Ma. Assuming that the andradite and magnetite formed during the same hydrothermal event, four blind-test samples containing coexisting magnetite and garnet or apatite were analysed. In all cases, magnetite ages obtained using TR41 as the primary reference material agree, within uncertainty, with independently determined ages of the associated minerals. These results demonstrate that TR41 provides an appropriate matrix correction for LA-ICP-MS U–Pb analyses of magnetite and represents a practical reference material for expanding the magnetite U–Pb geochronology. TR41 is available for distribution to interested laboratories upon request from the corresponding author.

1 Introduction

Magnetite is a ubiquitous accessory mineral in igneous, metamorphic, hydrothermal, and sedimentary environments and is widely used as a tracer of redox conditions, fluid-rock interaction, or ore-forming processes (e.g., Grigsby, 1990; Dupuis and Beaudoin, 2011; Dare et al., 2014; Nadoll et al., 2014; Coltat et al., 2021; Maher, 2024; Xu et al., 2024; Peters et al., 2026). In ultramafic rocks, magnetite commonly forms during serpentinization and related oxidation reactions, recording the interaction between fluids and oceanic lithosphere under a wide range of physicochemical conditions (Bach et al., 2006; Frost and Beard, 2007; Evans, 2008; Beard et al., 2009; Klein et al., 2014; McCollom et al., 2016). Likewise, during mafic to



intermediate magmatism, magnetite is generally ubiquitous (e.g. Hector et al., 2024; Patten et al., 2024a; 2024b). Because these processes often lack minerals suitable for conventional geochronology, direct dating of magnetite has the potential to provide unique temporal constraints on serpentinization, hydrothermal alteration, and associated mineralising events (e.g., Courtney-Davies et al., 2020).

Over the last decades, magnetite has become an increasingly important target for in situ geochemical analysis, and laser ablation-inductively coupled mass spectrometry (LA-ICP-MS) is now routinely used to determine its trace-element composition in a wide variety of geological settings (e.g. Dare et al., 2014; Coltat et al., 2021). These studies have demonstrated that magnetite can incorporate small concentrations of U (usually $< 1 \mu\text{g g}^{-1}$), highlighting its potential as a geochronometer. The possibility of dating magnetite was already recognised since the early 1990s (Burton and O’Nions, 1990; Erel et al., 1997; Gelcich et al., 2005). However, these early investigations did not lead to widespread application of the method, largely because of the analytical complexity of the required procedures and the relatively large uncertainties in the results. Recent advances in analytical capabilities have facilitate the development of both (U–Th)/He thermochronology (Blackburn et al., 2007; Cooperdock and Stockli, 2016; Schwartz et al., 2020; Corre et al., 2022) and U–Pb geochronology (Courtney-Davies et al., 2020; Rodríguez-Mustafa et al., 2024; Tang et al., 2024) are being developed.

The application of in situ U–Pb geochronology has expanded far beyond the traditional U-rich accessory minerals, and it is now routinely applied to a growing number of low-U minerals (e.g. Ring and Gerdes, 2016; Piccione et al., 2019; Millonig et al., 2020; Ring et al., 2024; Beranoaguirre et al., 2026). The suitability of a mineral for U–Pb geochronology, however, depends not only on its U and Pb concentrations, but also on the distribution and behaviour of these elements within the mineral and the ability of the U–Pb system to remain closed, or to be reset, so it records specific geological processes. In this respect, magnetite has considerable potential as a geochronometer, despite its generally low U concentrations, because it is widespread in geological environments and can record hydrothermal, metamorphic, and fluid–rock interaction events. Nevertheless, the application of LA-ICP-MS U–Pb geochronology depends critically on the availability of well-characterised, matrix-matched reference materials, which are essential to correct for laser-induced elemental and isotopic fractionation and to ensure the accuracy and reproducibility of U–Pb ages (Schaltegger et al., 2015; Horstwood et al., 2016). Although magnetite U–Pb geochronology is increasingly being explored, no widely available and community-adopted magnetite reference material currently exists. The lack of such a material therefore represents a major practical limitation to the wider application and inter-laboratory comparability of magnetite U–Pb geochronology.

Here we present a natural magnetite from the Troodos Ophiolite, Cyprus, as a candidate reference material for LA-ICP-MS U–Pb magnetite geochronology. The magnetite, formed during the oxidation of ultramafic rocks associated with ocean-floor serpentinization, is accessible in sufficient quantity to allow distribution to the wider community. Although the material has some limitations, it represents a practical solution to the limitations for the widespread application of magnetite U–Pb geochronology. We propose that this material may serve as a reference material for method development and inter-laboratory comparison until additional magnetite reference materials become available.



67 2 Methodology

68 2.1 LA-ICPMS

69 U-Pb data on magnetite were acquired *in-situ* from a polished mount and thin section, both prepared after the same
 70 hand specimen (Fig. 1), using a RESOLution 193 nm ArF excimer laser (CompexPro 102) equipped with a two-volume
 71 ablation cell (Laurin Technic S155) coupled to a single collector SF-ICPMS (Element XR, ThermoScientific) at the Frankfurt
 72 Isotope and Element Research Center (FIERCE), Goethe University Frankfurt. The instrumentation and methodology are the
 73 same as described in Beranoaguirre et al. (2022), and further analytical details are given in the supplementary material. Soda-
 74 lime glass NIST-SRM 612 (Jochum et al., 2011) was used as the primary standard for correcting the fractionation and the drift
 75 over the session of the $^{206}\text{Pb}/^{238}\text{U}$ and $^{207}\text{Pb}/^{206}\text{Pb}$ ratios. Raw data were corrected off-line using an in-house Microsoft Excel
 76 spreadsheet program (Gerdes and Zeh, 2006).

77 2.2 (U-Th)/He

78 For the (U-Th)/He analyses, the sample was gently crushed, and the fine dust was removed by rinsing in ethyl alcohol.
 79 The sizes of the selected fragments were between 150 and 250 μm . Three to six fragments were wrapped in platinum capsules
 80 of ca. 1x1 mm size. The Pt capsules were heated in the full-metal extraction line by an infrared laser in high vacuum. The
 81 extracted gas was purified using a SAES Ti-Zr getter kept at 450 $^{\circ}\text{C}$. The chemically inert noble gases and a minor amount of
 82 other residual gases were then expanded into a Hiden triple-filter quadrupole mass spectrometer equipped with a positive ion
 83 counting detector. Beyond the detection of helium, the partial pressures of some residual gases were continuously monitored
 84 (H_2 , CH_4 , H_2O , N_2 , Ar and CO_2). Helium blanks were estimated using the same procedure on empty Pt tubes (max. 0.0008 and
 85 0.002 ncc ^4He ; cold and hot blanks, respectively). Aliquots were checked for degassing of He by sequential reheating and He
 86 measurement.

87 In order to achieve quantitative dissolution, we have performed digestion tests on unheated multi-fragment aliquots
 88 applying 30% suprapur HCl and an HCl-HF mixture of a 10:1 ratio. Only the HCl-HF mixture dissolved the accompanying
 89 silicates of the samples; thus, we applied this acid mixture to the degassed aliquots. Important to note that those aliquots
 90 containing chromite were not dissolved completely. Following degassing, samples were retrieved from the gas extraction line,
 91 spiked with calibrated ^{230}Th and ^{233}U solutions and were digested by the above-described HCl-HF mixture in closed Savillex
 92 Teflon vials on a hot plate set for 125 $^{\circ}\text{C}$ in 72 hours. Each sample batch was prepared with a series of procedural blanks to
 93 check the purity and calibration of the reagents and spikes. The U and Th blanks are usually constant in a session and below 1
 94 pg. Spiked solutions were analysed by a Thermo iCAP Q ICP-MS coupled to an autosampler; seven cycles were recorded per
 95 sample. The actinides were determined by isotope dilution, while the concentration of major matrix elements of the dated
 96 minerals (Fe, Mn, Ti), samarium and the REEs were determined by external calibration.

97 The ejection correction factors (e.g. Farley et al., 1996) can be calculated only by a coarse approximation to be 0.85,
 98 as the magnetite crystal size distribution is rather wide. The sheet silicate matrix between the magnetite grains is frequently



very thin (1-2 μm); thus, the ejected alpha particles can be implanted in the adjacent magnetite crystal. This introduces additional bias in the ejection correction. The (U-Th-Sm)/He ages were calculated by the Taylor Expansion Method (after Des Patterson). The Sm content was also considered for age calculation.

2.2.1 Actinide evaporation

Some iron oxides, mostly hematite and goethite, undergo phase transformation at higher temperatures during the thermal extraction of helium (e.g. Hoffmann et al., 2020). During this transformation, the actinide element can evaporate from the Fe-oxide, and it can cause erroneously old (U-Th)/He ages. To check this potential effect on magnetite, degassing tests were performed at different temperatures, and also, unheated aliquots were dissolved and measured. The concentrations of the U obtained at different temperatures are similar, which indicates that the actinide loss during the He extraction was negligible, and thus the ages do not need correction.

2.3 SEM and EPMA

To further characterise the sample, Energy Dispersive X-ray Spectrometry (EDS) images were performed using a JEOL JSM-6010LV scanning electron microscope at the Institute of Mineralogy and Petrography, University of Innsbruck. Mineral chemistry for silicate minerals was determined using a JEOL JXA-iSP100 electron microprobe (EMPA) at the same institution. The JEOL PC-EPMA application software version 2.4.1.0 was used, and the running conditions were 15 kV and 20 nA, with a beam size of 3-5 μm .

Mineral recalculation (molecular and $\mu\text{g g}^{-1}$) is performed with a homemade spreadsheet, in which Fe^{3+} is calculated using the difference between the ideal charge (8) and the sum of the calculated charge assuming $\text{Fe}^{\text{tot}} = \text{Fe}^{2+}$. The temperature of magnetite formation is determined from empirical magnetite thermometry using Al trace elements (Canil and Lacourse, 2020). Magnesium in magnetite geothermometer would be preferred for use in hydrothermal systems; however, a strong correlation of Si vs. Mg shows contamination of the serpentinite matrix or inheritance from the protolith, preventing the use of the Mg geothermometer for this case. Aluminium concentration in magnetite is dependent on temperature and suitable for a hydrothermal system (eq. 1).

$$T_{\text{Al-mag}} (^{\circ}\text{C}) = -6050(\pm 540)/(\ln X_{\text{Al}} - 2.88(\pm 0.47)) - 273.15, \text{ in which } X_{\text{Al}} \text{ is } \text{Al}/(\text{Al} + \text{Cr} + \text{Fe}^{3+}) \quad (\text{eq. 1})$$

A second empirical thermometer using Ti and V content in magnetite is applied (eq. 2; Gao et al., 2026). The incorporation of Ti and V in magnetite is strongly dependant of temperature in hydrothermal systems (Nadoll et al., 2014) and reflects the thermal condition during magnetite formation.

$$T_{\text{Ti+V-mag}} (^{\circ}\text{C}) = -7699(\pm 73)/(\ln(X_{\text{Ti+V}}) - 4.68(\pm 0.1)) - 273.15, \text{ with } X_{\text{Ti+V}} = \text{molar (Ti+V)}/(\text{Ti+V} + \text{Fe}^{\text{tot}}) \quad (\text{eq. 2})$$

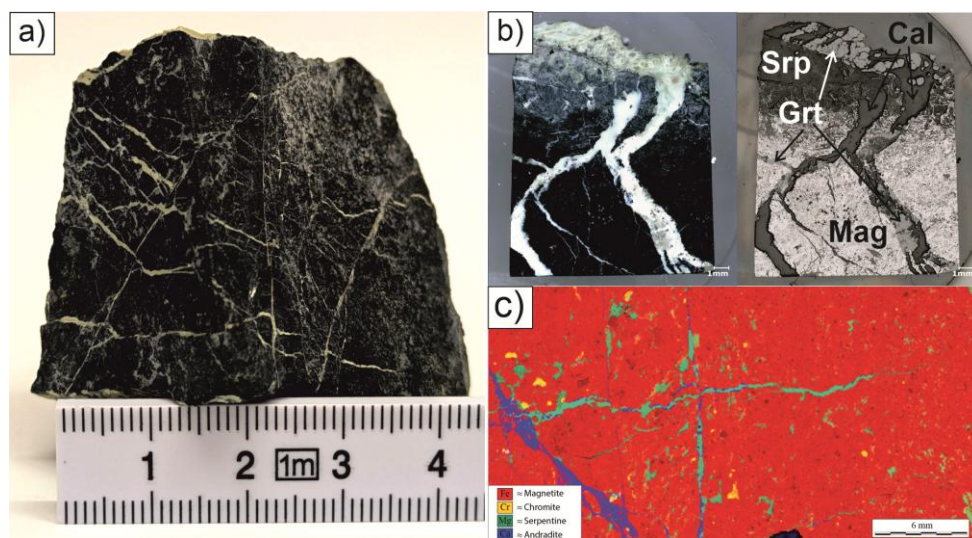
Individual temperature uncertainty is derived from formula uncertainties. Final temperature is given as the median and standard deviation (2SD) of the dataset.



129 3 Samples

130 3.1 TR41 magnetite: description

131 The Troodos ophiolite in Cyprus is a well-preserved example of fossil oceanic crust that preserves a classic Penrose
 132 oceanic crustal sequence formed in a supra-subduction environment during the Late Cretaceous (Moores and Vine, 1971;
 133 Robertson and Dixon, 1984; Gass et al., 1994). It is divided into the northern massif, representing the main Troodos ophiolite,
 134 and the south-eastern massif, also known as the Limassol Forest Complex. The two massifs are separated by the Arakapas
 135 transform fault zone. These two ophiolite blocks have been interpreted to represent two different oceanic ridge segments (Gass
 136 et al., 1994; McLeod and Murton, 1995). The exhumation of mantle rocks within the Limassol Forest Complex has been
 137 attributed either to deformation associated with the Arakapas transform fault and subsequent Miocene thrusting (Murton and
 138 Gass, 1986; MacLeod and Murton, 1995) or to the development of a detachment fault at a ridge-transform intersection (Cann
 139 et al., 2001). Regardless of the exhumation mechanism, the serpentinisation of the mantle rocks in the Limassol Forest
 140 Complex occurred soon after the creation of the oceanic crust during the Late Cretaceous. Zircon U–Pb ages from
 141 plagiogranites indicate that magmatism in the Troodos ophiolite took place between approximately 95 and 89 Ma (Mukasa
 142 and Ludden, 1987; Morag et al., 2016; 2020; Chen et al., 2020). Zircon (U–Th)/He isochron age of 92.6 ± 1.4 Ma from gabbro,
 143 diabase and plagiogranite in Troodos massif indicates early cooling of the oceanic crust (Morag et al., 2016). Similarly, Sr
 144 isotopes from seawater precipitation of calcite veins within basalts in S-Limassol resulted in an age of 90.5 ± 2.5 Ma, indicating
 145 that seafloor alteration was contemporaneous with volcanic activity (Quandt et al., 2019).



146
 147 **Figure 1. Representative images of sample TR41. a) Hand specimen of the hydrothermal magnetite from which the grain mount (b)**
 148 **and polished thin section (c) were prepared. The hand specimen is sufficiently large to be subdivided and distributed to interested**
 149 **laboratories, upon request. (b) Photograph (left) and corresponding reflected-light image (right) of the grain mount. (c) μXRF map**
 150 **of the polished thin section showing the distribution of magnetite (red), serpentine (green), andradite (blue), and chromite (yellow).**
 151 **The sample is dominated by massive magnetite cross-cut by serpentine-, calcite-, and andradite-bearing veins.**



Sample TR41 (34.822258°; 33.091867°) was collected in the Limassol Forest Complex of the Troodos ophiolite. It is composed of massive magnetite crosscut by calcite, andradite and serpentine veins. Magnetite forms a 5-10 cm metasomatic rim on serpentinite clasts (~1 m) within a shear zone (Fig. 2a). Petrological observations show that the sample is composed of magnetite, with minor chromite, serpentine, calcite, andradite and pyrite (Fig. 1 and 2). The overall texture indicates that magnetite formed through replacement of serpentinite, as evidenced by abundant serpentine inclusions in magnetite (Fig. 2b). Likewise, magnetite is cross-cut by multiple, network-like carbonate, serpentine and andradite veins (Fig. 1), suggesting that magnetite precipitation, serpentinization, and veining occurred during a prolonged episode of fluid circulation rather than as discrete events. Such textures are consistent with the widely recognised formation of secondary magnetite during serpentinization, whereby hydration and oxidation of Fe-bearing olivine and pyroxene release Fe^{2+} that is subsequently oxidised to Fe^{3+} and incorporated into newly formed magnetite (e.g., Beard et al., 2009; McCollom et al., 2016). Pyrite is observed as a small inclusion both in magnetite (1-30 μm) and in the matrix (~30 μm), suggesting that pyrite is cogenetic with magnetite (Fig. 2b,c,e). Chromite is sub-idiomorphic (~250-500 μm) and shows a rim of ferry-chromite (Fig. 2d). Andradite, mainly found in the veins, occurs as idiomorphic to sub-idiomorphic grains (50-100 μm) or forming aggregates (Fig. 2c,d,e).

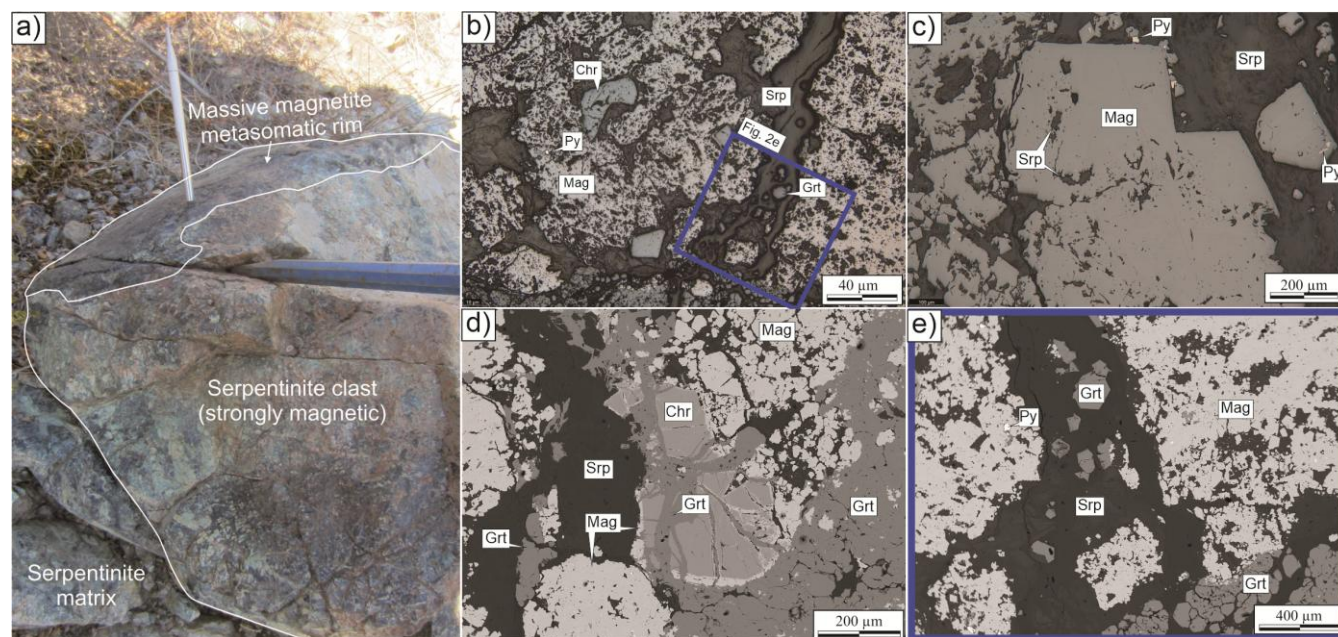
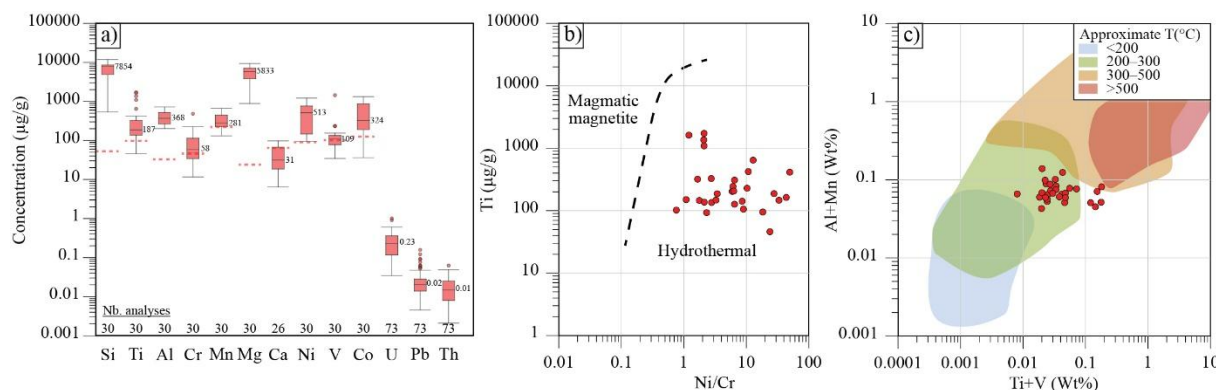


Figure 2. Representative field and petrographic images of sample TR41. a) Outcrop photograph showing the massive metasomatic magnetite developed around a serpentinite clast. b) Reflected-light image showing the typical texture of TR41. Magnetite contains abundant serpentinite inclusions, indicating replacement of the serpentinite protolith, whereas later serpentinite and andradite veins cross-cut the magnetite. The blue rectangle indicates the area enlarged in (e). c) Detailed micro-photograph showing sub-idiomorphic magnetite hosting intragranular serpentine and pyrite inclusions, interpreted as evidence for contemporaneous mineral growth during hydrothermal alteration. d) BSE images showing partly replaced and fractured chromite infill by andradite. e) Close look of Fig. 2b showing later veins of serpentine and andradite.



173 The magnetite is chemically homogeneous at the major-element scale, with a median Fe content of ~90 wt.% and
174 calculated $X\text{Fe}^{3+}$ values ranging from 0.63 to 0.67 (supplementary material, Table 1). Trace-element analyses reveal relatively
175 high Si (median 7854 $\mu\text{g g}^{-1}$) and Mg (5833 $\mu\text{g g}^{-1}$) concentrations (Fig. 3a). The positive correlation between Si and Mg is
176 interpreted to reflect minor contamination from the surrounding serpentinite or from the precursor silicates. Ti (187 $\mu\text{g g}^{-1}$), Al
177 (368 $\mu\text{g g}^{-1}$), Cr (58 $\mu\text{g g}^{-1}$), Mn (281 $\mu\text{g g}^{-1}$), Ni (513 $\mu\text{g g}^{-1}$), V (109 $\mu\text{g g}^{-1}$) and Co (324 $\mu\text{g g}^{-1}$) are relatively homogeneous
178 and occur at similar concentrations, whereas Ca (median 31 $\mu\text{g g}^{-1}$; most analyses below detection) and Zn (below detection)
179 are depleted (Fig. 3a). High-contrast BSE images reveal oscillatory rims within magnetite grains, which are accompanied by
180 a systematic decrease in Si (from ~7700 to ~600 $\mu\text{g g}^{-1}$) and Mg (from ~6700 to ~1600 $\mu\text{g g}^{-1}$) and a slight increase in $X\text{Fe}^{3+}$
181 (from 0.65 to 0.67). Despite these variations, the zoning is gradual and reproducible between grains, and no petrographic
182 evidence exists for multiple generations of magnetite. Petrographic characteristics and trace element compositions are
183 consistent with a hydrothermal origin of the magnetite (Fig. 3b; Dare et al., 2014). The discrimination diagram of Nadoll et al.
184 (2014) suggests crystallisation temperatures of approximately 200–300 °C (Fig. 3c), while independent estimates based on Al
185 contents (Canil and Lacourse, 2020) and the Ti–V geothermometer (Gao et al., 2026) yield slightly higher temperatures of 378
186 ± 55 °C and 358 ± 85 °C (2 σ), respectively. Overall, all the independent data indicate formation under epi- to mesothermal
187 hydrothermal conditions. Coltat et al. (2025) showed that Fe–Ca silicates such as andradite commonly crystallise during the
188 early stages of serpentinization at temperatures of approximately 300–360 °C. These temperatures overlap with those of the
189 magnetite, consistent with formation during the same hydrothermal system, even if the veining post-dated the main stage of
190 magnetite precipitation.



191
192 **Figure 3. a) Element concentration in magnetite.** The box represents the interquartile range (IQR), with the median indicated by
193 the horizontal line. Whiskers extend to 1.5 $\times$ IQR from the first and third quartiles, and values beyond this range are plotted as
194 outliers. Concentrations of U, Pb and Th are determined using LA-ICP-MS, while the rest of the elements measured are from EPMA.
195 Red dashed lines show the limit of detection for EPMA. Detection limits for U, Pb and Th are not shown because they are below the
196 graphic representation. b) Plot of Ti ($\mu\text{g g}^{-1}$) vs. Ni/Cr ratio in magnetite (Dare et al., 2014 showing the hydrothermal origin of the
197 magnetite. c) Ti + V vs. Al + Mn plots (Nadoll et al., 2014) showing relative temperature formation of the magnetite.



3.2 Validation samples

As no routine methodology currently exists for the independent characterisation of magnetite by isotope dilution thermal ionisation mass spectrometry (ID-TIMS, see Discussion), the proposed age of TR41 was evaluated through a series of validation analyses. These include an internal validation based on the U–Pb dating of coexisting andradite veins, together with an external validation using magnetite-bearing samples for which independent geochronological constraints are available. Although the andradite veins post-date magnetite replacement, both minerals are interpreted to record the same hydrothermal event. Consequently, the andradite age provides an independent constraint on the timing of magnetite formation. In addition, four blind-test samples provided by external collaborators were analysed without prior knowledge of their geological origin or expected ages. These include one skarn sample containing magnetite and garnet in textural equilibrium and three magnetite–apatite samples (Fig. 4). In all cases, magnetite was dated using TR41 as the primary reference material, whereas the coexisting garnet or apatite was dated independently using established analytical protocols at the FIERCE laboratory (e.g., Reinhardt et al., 2022; Binder et al., 2022).

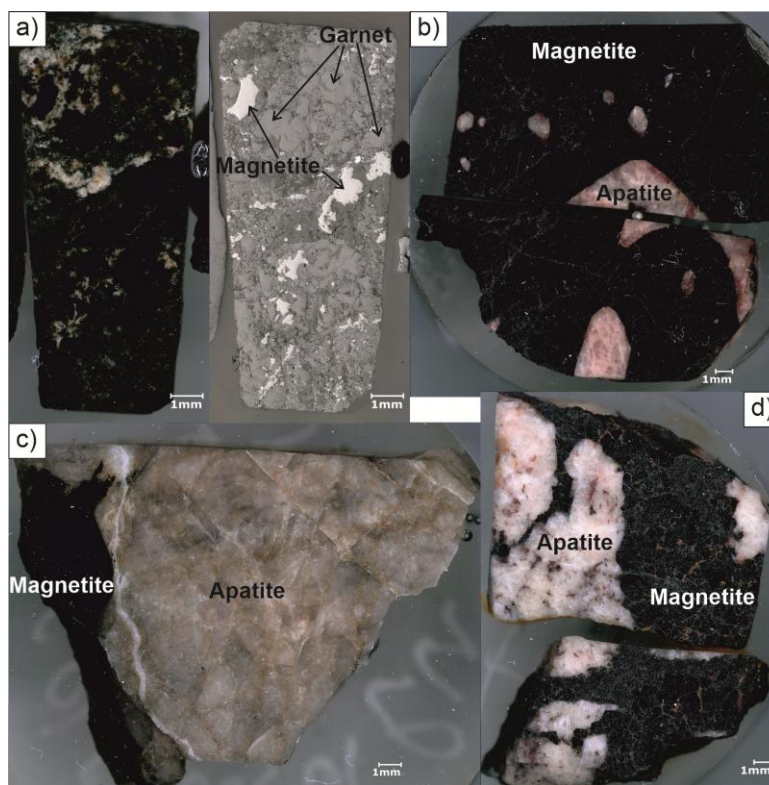


Figure 4. Representative photographs of the four blind-test samples used to evaluate TR41 as a matrix-matched reference material for magnetite U–Pb geochronology. a) Skarn containing magnetite and garnet in textural equilibrium (sample FT-4 in Fig. 7). The reflected-light image (right) highlights the textural relationship between both minerals. b), c) and d) Magnetite–apatite samples (CB-2, CB-1 and CB-3, respectively). The geological origin of these samples, provided by external collaborators, was not disclosed during the analytical work, allowing an unbiased evaluation of the proposed reference material.



216 4 Results and discussion

217 Traditionally, the proposal of a potential reference material is accompanied by an independently determined ID-TIMS
218 age. However, ID-TIMS analysis of magnetite is particularly challenging due to several factors, which have so far precluded
219 the establishment of a routine methodology. U concentrations in magnetite are commonly below $1\mu\text{g g}^{-1}$ (Tang et al., 2024;
220 this work), which results in even lower radiogenic Pb content. Additionally, magnetite may also contain significant common
221 Pb. Therefore, obtaining enough U and Pb for high-precision ID-TIMS analysis requires dissolving large quantities of
222 magnetite, introducing considerable amounts of matrix Fe into the analytical procedure. This may reduce the chromatographic
223 purification efficiency, as well as increase procedural blanks. Moreover, the incomplete removal of the matrix may influence
224 thermal ionisation behaviour, affecting ion beam stability and ionisation efficiency during TIMS analysis (Parrish et al., 1992;
225 Davis et al., 1997). To circumvent this limitation, the suitability of a magnetite reference material must therefore be evaluated
226 through alternative criteria, including the validation against independently dated minerals.

227 The absence of well-characterised magnetite reference material also raises the question of how matrix-dependent
228 elemental and isotopic fractionation should be corrected during LA-ICP-MS analyses. Tang et al. (2024) demonstrated that
229 U–Pb ages of magnetite can be obtained using cassiterite, zircon, or garnet as primary reference materials. Although this
230 approach may yield geologically meaningful ages, numerous studies have shown that non-matrix-matched reference materials
231 may introduce systematic biases in U/Pb ratios owing to differences in ablation behaviour, aerosol transport, and plasma
232 ionisation (e.g. Thompson et al., 2016, 2021; Carr et al., 2023). Consequently, the development of a dedicated magnetite
233 reference material remains a critical step towards accurate and reproducible U–Pb magnetite geochronology.

234 Preliminary U–Pb analyses of TR41 were performed to evaluate whether magnetite could be dated using the analytical
235 protocols at the FIERCE laboratory. Despite the low U and Pb concentrations ($\text{U} < 1\mu\text{g g}^{-1}$), the analyses show a broad spread
236 in both the $^{238}\text{U}/^{206}\text{Pb}$ and $^{207}\text{Pb}/^{206}\text{Pb}$ ratios, allowing a well-constrained regression line to be calculated (Fig. 5a). The resulting
237 internal precision is approximately 1%, demonstrating that the isotopic variability of the sample is sufficient for precise LA-
238 ICP-MS U–Pb analyses. Furthermore, analyses performed on both a polished thin section and a grain mount (Fig.1) plot along
239 the same regression line (Fig. 5a), indicating that the isotopic composition is comparable in different aliquots. This observation
240 is consistent with the petrographic and trace-element characterisation, which indicates that TR41 consists of a relatively
241 homogeneous hydrothermal magnetite population, with no evidence for multiple generations of magnetite. Although Fig. 5a
242 does not report any age because the data can not be matrix corrected, the analytical reproducibility between different aliquots
243 and isotopic spread observed in the sample already suggest that TR41 possesses the characteristics required of a potential
244 matrix-matched reference material.

245 During the petrographic characterisation of TR41, hydrothermal andradite veins cross-cutting the magnetite were
246 identified (Fig. 2 and 3). Although the andradite post-dates magnetite precipitation and thus, the two minerals do not crystallise
247 simultaneously in a strict petrographic sense, both minerals formed during the same hydrothermal episode associated with
248 serpentinization. Independent geothermometric estimates indicate magnetite crystallisation temperatures of approximately



300–380 °C, which overlap with the 300–360 °C formation temperatures proposed for hydrothermal andradite during serpentization (Coltat et al., 2025). Consequently, any time difference between magnetite replacement and andradite veining is likely to be small relative to the analytical resolution of the U–Pb method. U–Pb analyses of the andradite, performed using the routine garnet analytical protocol employed at the FIERCE laboratory, yield a lower-intercept age of $92.04 \pm 1.27/2.23$ Ma (Fig. 5b). This agrees with the zircon (U–Th)/He age of 92.6 ± 1.4 Ma for the main Troodos ophiolite published by Morag et al. (2016). Although this age does not directly date the magnetite, it provides the first independent geochronological constraint on the timing of the hydrothermal event recorded by TR41 and therefore represents the best available age constraint for evaluating TR41 as a candidate magnetite reference material.

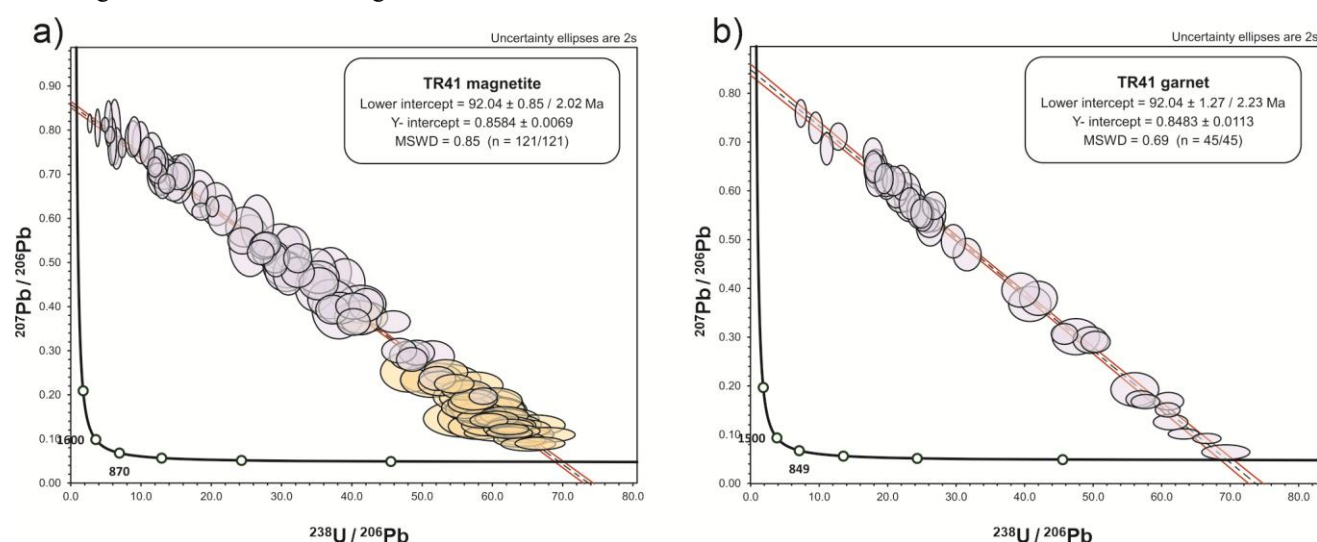


Figure 5. U–Pb data obtained for the proposed reference material TR41. (a) Tera–Wasserburg diagram showing all magnetite analyses. Grey ellipses correspond to analyses performed on the polished thin section, whereas green ellipses represent analyses from the grain mount. Both aliquots plot on the same regression line, demonstrating the reproducibility of the isotopic composition between independently prepared samples. The magnetite age was calculated using the U–Pb age of the andradite in (b) as the assigned age for TR41. (b) Tera–Wasserburg diagram for hydrothermal andradite veins associated with the magnetite. The lower-intercept age of 92.04 ± 2.23 Ma provides the best independent age constraint currently available for the hydrothermal event responsible for magnetite formation and is therefore adopted for the evaluation of TR41 as a candidate magnetite reference material.

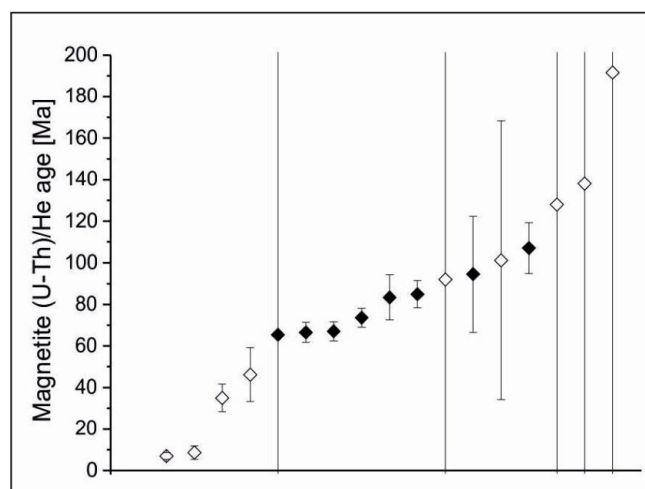


Figure 6. The ejection-corrected magnetite (U-Th)/He ages obtained for TR41. Filled symbols correspond to the eight aliquots included in the age calculation, whereas open symbols indicate rejected analyses. Rejected aliquots are characterised either by large analytical uncertainties resulting from extremely low actinide contents or by poor Fe recovery during chemical digestion, suggesting incomplete dissolution of magnetite. The Tukey's Biweight Mean of the 8 aliquots is 80 ± 13 Ma (Isoplot, Ludwig, 2012).

As an additional and fully independent analysis, TR41 was also investigated using magnetite (U-Th)/He thermochronology. Although the analyses yield relatively large uncertainties, the ejection-corrected Tukey's Biweight mean age of 80 ± 13 Ma overlaps, within uncertainty, with the andradite U-Pb age obtained in this study (92.04 ± 2.23 Ma) (Fig. 6). The magnetite (U-Th)/He thermochronology is still at an early stage of development and is affected by several analytical limitations, including (i) the opacity of magnetite, which prevents the identification of mineral inclusions (e.g. apatite; Blackburn et al., 2007), and (ii) the generally low U contents of magnetite, which make the analyses susceptible to alpha implantation from neighbouring U-rich phases (Schwartz et al., 2024). Despite these limitations, the obtained age is geologically reasonable. The magnetite (U-Th)/He system has an estimated closure temperature of 200–280 °C (Blackburn et al., 2007; Bassal et al., 2022), slightly lower than the hydrothermal temperatures estimated for TR41 (up to 350–400 °C). Therefore, the (U-Th)/He system may record cooling following magnetite formation rather than the timing of crystallisation itself. Nevertheless, considering the large analytical uncertainties, the magnetite (U-Th)/He age is interpreted as a low-precision constraint that is consistent with Late Cretaceous cooling of the Troodos ophiolite.

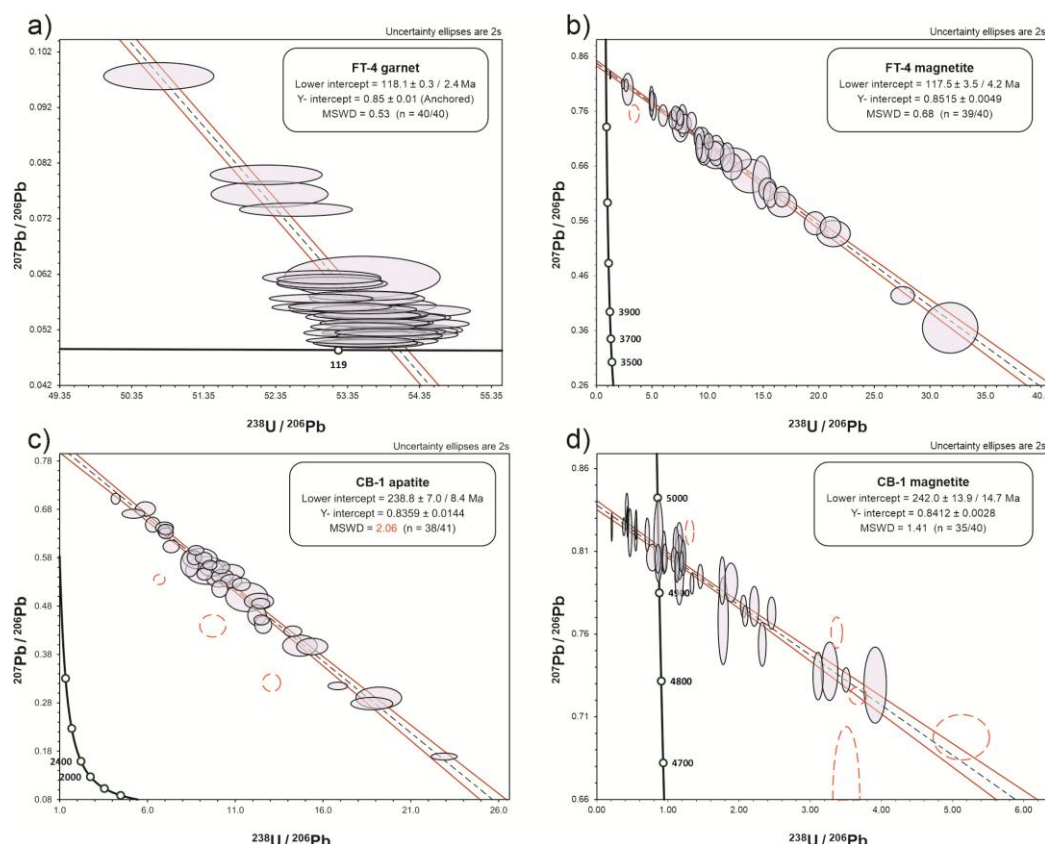


Figure 7. U–Pb Tera–Wasserburg diagrams for the first two blind-test samples. (a) Garnet and (b) magnetite from the FT-4 skarn sample, and (c) apatite and (d) magnetite from sample CB-1. Magnetite analyses were calibrated using TR41 as the primary reference material, assuming an age of 92.04 ± 2.23 Ma. In both samples, magnetite yields ages indistinguishable from those obtained from the coexisting datable minerals. Rejected analyses are shown as open symbols and are discussed in the text.

The final test performed to evaluate the suitability of TR41 as a matrix-matched reference material was to assess whether it reproduces independently determined ages from other geological samples. As described above, this assessment was carried out using four blind-test samples (Fig. 4), in which TR41 was analysed as the primary reference material for magnetite U–Pb dating, assuming an age of 92.04 ± 2.23 Ma based on the coexisting andradite.

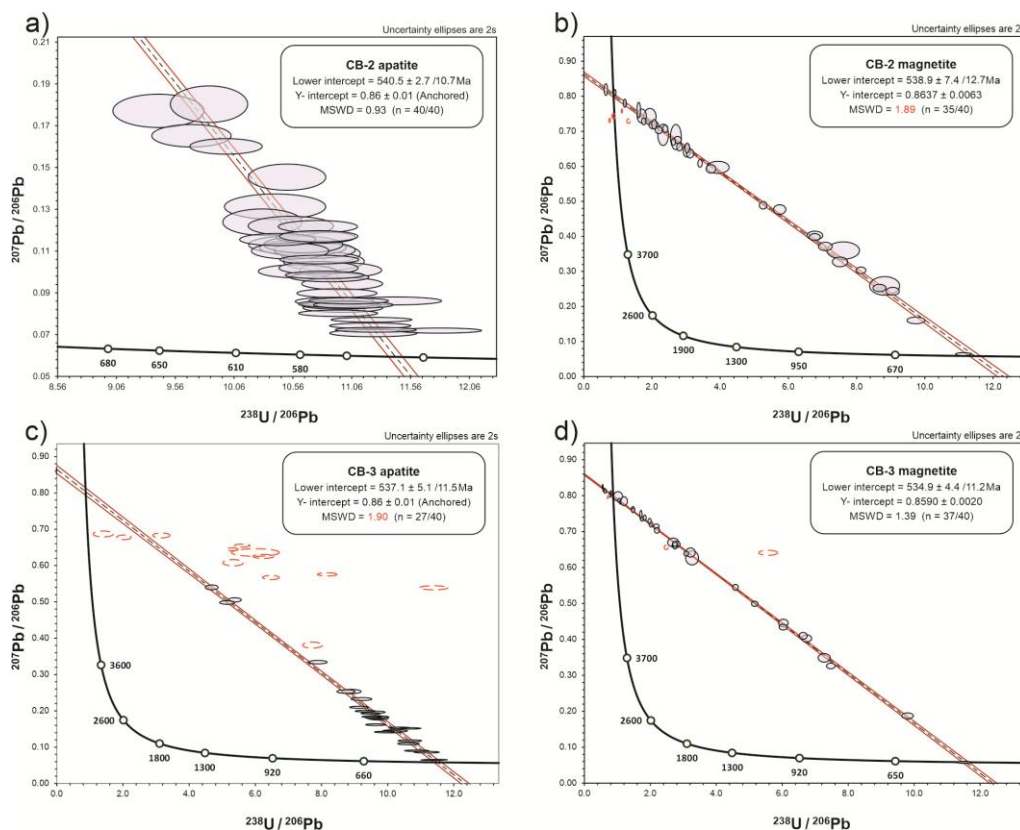


Figure 8. U–Pb Tera–Wasserburg diagrams for the remaining blind-test samples. (a) Apatite and (b) magnetite from sample CB-2, and (c) apatite and (d) magnetite from sample CB-3. Magnetite analyses were calibrated using TR41 as the primary reference material, assuming an age of 92.04 ± 2.23 Ma. In both samples, magnetite ages agree within uncertainty with those obtained from the coexisting apatite. Rejected analyses are shown as open symbols and are discussed in the text.

The four blind-test samples span a wide range of geological ages, from the Cretaceous to the (Pre?)-Cambrian, and include minerals with markedly different U and Pb concentrations (supplementary material). Whereas the coexisting garnet and apatite typically contain U in the $\mu\text{g g}^{-1}$ concentrations, the analysed magnetite ranges only 0.02–1.11 $\mu\text{g g}^{-1}$ U. Nevertheless, all magnetite data define robust regression lines in the Tera–Wasserburg diagram (Figs. 7 and 8) and yield lower-intercept ages that are indistinguishable from those obtained from the coexisting minerals. The agreement is observed despite differences in common-Pb contents, spread in $^{206}\text{Pb}/^{238}\text{U}$ and $^{207}\text{Pb}/^{206}\text{Pb}$ ratios, or geological age. A few data points were excluded based on analytical or geological criteria. For example, two of the analyses in the CB-1 magnetite were rejected, even if they plot on the regression line, because of their anomalously high Th concentrations (ca. 30 $\mu\text{g g}^{-1}$), which likely indicate the ablation of apatite. Likewise, eleven analyses from CB-3 apatite define a poorly constrained younger array, suggesting a partial isotopic resetting. However, the remaining analyses still define a robust crystallisation age consistent with the magnetite. In other cases, isolated outliers were discarded when they clearly deviated from the main regression, due to probable micrometric inclusions



or disturbances observed in the time-resolved analysis spectra. Nevertheless, all data are reported in the supplementary material, including both accepted and rejected spots.

These observations demonstrate that the absence of an independently characterised ID-TIMS age does not necessarily preclude the development of a robust magnetite reference material. Instead, the combination of petrographic, geochemical and isotopic characterisation, and successful validation through blind tests provides a practical framework for evaluating candidate materials. In the case of TR41, the agreement between magnetite ages and independently dated coexisting minerals across four blind-test samples strongly supports the proposed age assignment and demonstrates that the material may be considered as an appropriate matrix-matched reference material for LA-ICP-MS U–Pb analyses. TR41 is therefore proposed as a reference material for magnetite U–Pb geochronology. Additional reference materials and methodological developments will further strengthen and expand the approach.

5 Conclusions

This study presents TR41, a hydrothermal magnetite from the Troodos ophiolite (Cyprus), as a candidate matrix-matched reference material for LA-ICP-MS U–Pb magnetite geochronology. Petrographic observations, major- and trace-element composition, and U–Pb data indicate that the material is sufficiently homogeneous to produce well-defined regressions in the Tera–Wasserburg diagram despite containing less than $1 \mu\text{g g}^{-1}$ U. Although an independent age determination of magnetite by ID-TIMS is currently not feasible because of significant analytical challenges, the age of TR41 was constrained through complementary geochronological approaches. U–Pb geochronology of hydrothermal andradite veins genetically associated with the magnetite yields a lower-intercept age of 92.04 ± 2.23 Ma, consistent with the timing of hydrothermal activity in the Troodos ophiolite. Additional magnetite (U–Th)/He thermochronology provides a lower-precision age of 80 ± 13 Ma, which overlaps with the U–Pb age within uncertainty. Using the proposed age of TR41, four blind-test samples containing coexisting datable minerals (garnet or apatite) were analysed. In all cases, magnetite ages obtained using TR41 as the primary reference material are indistinguishable from the ages determined for the coexisting minerals. This agreement demonstrates that the proposed age of TR41 provides an appropriate matrix correction for LA-ICP-MS U–Pb analyses of magnetite. Although additional community reference materials would further strengthen the method, the availability of TR41 overcomes one of the principal limitations that has so far hindered the wider application of magnetite U–Pb geochronology and provides a practical foundation for future methodological developments.

Approximately 1×1 cm wafers of TR41, cut from the massive magnetite shown in Fig. 1a, are available for distribution to the scientific community upon request from the corresponding author, Aratz Beranoaguirre (beranoaguirre@fierce.uni-frankfurt.de).

Data availability

The data presented in this manuscript are available in the supplementary material.



338 **Author contributions**

339 AB conceptualised the study, obtained funding, carried out the formal U-Pb analyses and wrote the original draft. AP,
340 CGCP, SH and MJ collected the sample in the field and characterised it. ID performed the (U-Th)/He analyses. JK and AG
341 helped throughout the whole process and during the manuscript writing. All authors reviewed and approved the final
342 manuscript

343 **Competing interests**

344 The authors declare that they have no competing interests.

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349 **Declaration of AI Use**

350 During the preparation of this manuscript, the authors used OpenAI software to improve the clarity, grammar, and
351 readability of the English text. These tools were used exclusively for language editing. All scientific content, figures,
352 interpretations, analyses, and conclusions were developed and verified by the authors, who take full responsibility for the
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