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2 Ultra-high pressure inclusion in Archean ophiolitic  
3 podiform chromitite in mélangé block suggests deep  
4 subduction on early Earth

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15 **ABSTRACT**

16 The discovery of ultrahigh pressure (UHP) minerals in orogenic belts has revolutionized our  
17 understanding of subduction and the return flow of previously deeply subducted material to  
18 Earth's surface as part of the cycling and interaction of crustal and mantle systems. One class of  
19 UHP minerals is found as inclusions in orogenic peridotite-hosted podiform-chromite systems,  
20 within Phanerozoic ophiolites and ophiolitic mélanges. Such inclusions have opened a window  
21 into processes of recycling of crustal materials to the deep mantle and back through subduction  
22 and mantle convection in Phanerozoic orogens. Here, we document the first occurrence of an

23 UHP mineral hosted in an ophiolitic podiform chromitite mélangé from the Neoproterozoic paired  
24 metamorphic belt of the Central (Taihang) Orogenic Belt, Northern China. Numerous inclusions  
25 of rutile, apatite, dolomite, and amphibole are interpreted to be crustal-derived; they occur in  
26 podiform chromite grains hosted in a 2.6-2.5 Ga ophiolitic mélangé now part of the North China  
27 Craton and formed by subduction of oceanic and continental material. Microstructures and phase  
28 relationships in a multi-phase inclusion of TiO<sub>2</sub>(II), rutile, apatite, and tremolite yield minimum  
29 P-T conditions of 7.5 GPa at 1000°C, indicating that the crustal host, including carbonates, were  
30 subducted to depths > 270 km, transferred to the mantle of the overriding plate, and returned to  
31 the surface by 2.5 Ga. We suggest that slab rollback forced upward mantle flow, incorporating  
32 entities from the lower plate, perhaps in serpentinite diapirs, resulting in adiabatic melting that  
33 allowed crustal material to be trapped in chromite grains crystallizing in high-Mg melts.  
34 Contrasting bulk moduli and thermal contraction of the inclusions and host chromite protected  
35 the inclusions from P-induced back-reaction during exhumation. Together, these features show  
36 that the 1600 km long Central (Taihang) Orogenic Belt is emerging as the world's first well-  
37 documented Phanerozoic style orogen, with classic tectonic zonation, ophiolitic mélanges, paired  
38 metamorphism, local evidence for UHP conditions, foreland basins, and late to post orogenic  
39 magmatism. Applying the null hypothesis, we explain this high degree of similarity by invoking  
40 the operation of Phanerozoic style plate tectonics, at least throughout the 1600 km length of the  
41 COB, and by geological comparison, in other similar aged geological terrains globally. From this  
42 we infer modern-style plate tectonics was operating in the Neoproterozoic.

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45 Y., and Polat, A., Ultra-high pressure inclusion in Archean ophiolitic podiform chromitite in mélangé  
46 block suggests deep subduction on early Earth, *Precambrian Research*, [106318](#)

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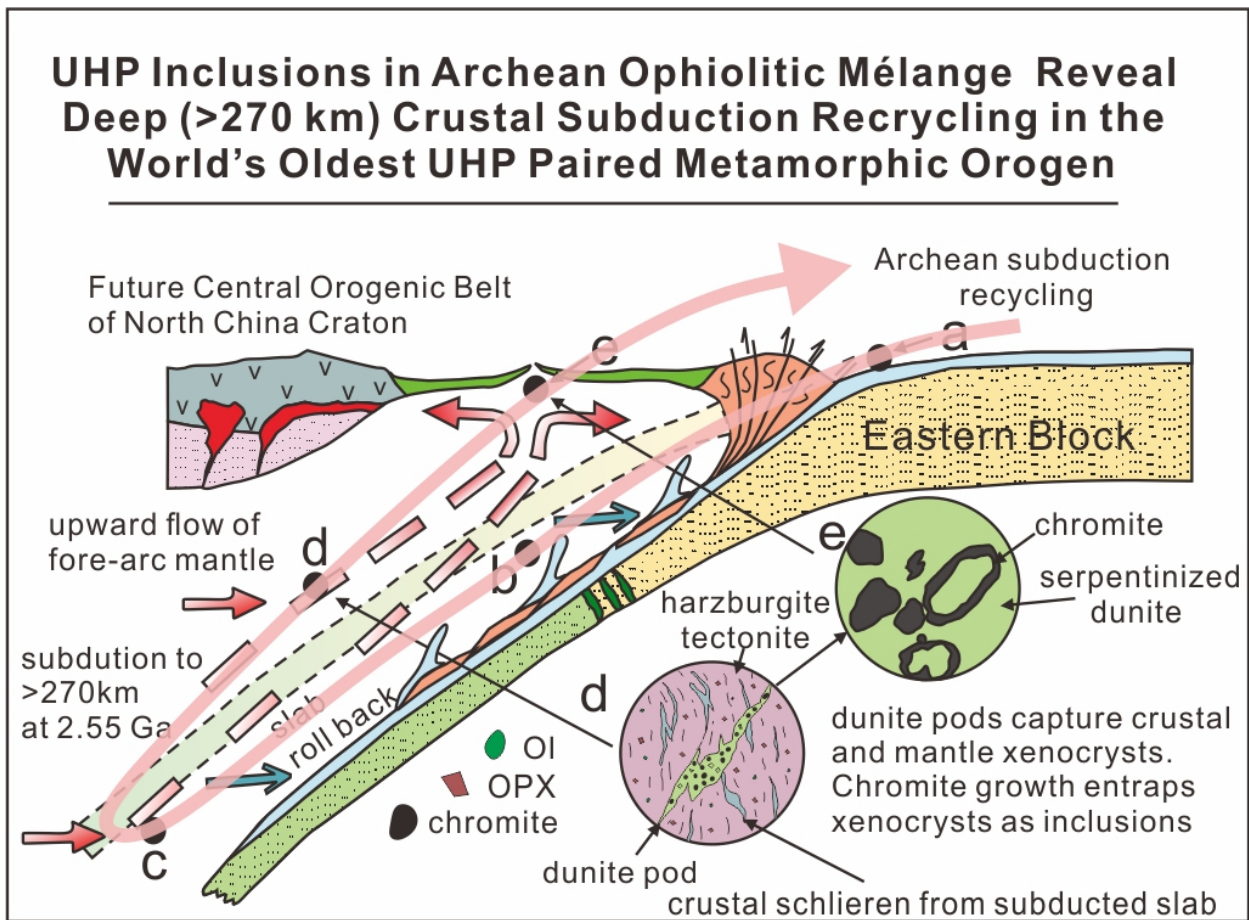
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49 **Keywords**

50 Archean; subduction; ultrahigh pressure metamorphism; deep carbon cycle; North China Craton

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52 **Graphical abstract**



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## 59 Highlights

- 60 • Ultra-high pressure TiO<sub>2</sub> II identified in Archean ophiolitic podiform chromite mélange
- 61 • Crustal minerals subducted to >270 km at 2.55 Ga, then recycled to surface
- 62 • UHP mélange is part of paired metamorphic Central (Taihang) Orogenic Belt of North
- 63 China
- 64 • Data shows operation of subduction-related deep carbon cycle in Archean
- 65 • Convergent margin processes in Archean were similar to those of Phanerozoic

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## 67 1. Introduction

68 One of the most controversial issues in Earth and Planetary Sciences today is determining  
69 when the style of modern plate tectonics developed on Earth, and for how long the subduction-  
70 driven, life-sustaining deep carbon cycle has been in operation ([National Academies of Sciences,](#)  
71 [2020](#)). Field-based structural, petrological, sedimentological, and geochemical evidence is  
72 consistent with modern style tectonics operating since the Eoarchaean ([Harrison, 2009; Polat,](#)  
73 [2012; Kusky et al., 2018; Windley et al., 2021](#)) or Mesoarchaean ([Cawood et al., 2018](#)), but  
74 some have argued that the metamorphic record is not consistent with modern style tectonics,  
75 because of the absence of paired metamorphism (low  $dT/dP$  with high  $dT/dP$ ) and orogenic ultra-  
76 high pressure (UHP) minerals in Archean orogenic belts (c.f. [Stern, 2008; Brown and Johnson,](#)  
77 [2019; Zheng and Zhao, 2020; Kusky, 2020](#)). Recent studies have shown that the Central  
78 (Taihang) Orogenic Belt (COB) of the North China Craton (NCC) contains geological records of

79 a late Archean arc/continental collision, that includes hallmark features of Phanerozoic  
80 collisional orogens including accretionary wedges with accreted ocean plate stratigraphy,  
81 ophiolitic mélanges, fore-arc ophiolites, supra-subduction zone magmatic systems, sea-floor  
82 hydrothermal systems, zones of Alpine-style nappes, a hinterland of high-grade metamorphic  
83 and magmatic rocks, and foreland basins filled contemporaneously with late stages of collision  
84 (Kusky et al., 2016, 2020; Deng et al., 2018; Wang et al., 2019; Huang et al., 2019; Peng et al.,  
85 2020; Ning et al., 2020; Jiang et al., 2020). Most recently the COB has been shown to contain a  
86 clear record of late Archean spatially and temporally linked paired metamorphism (low-medium  
87  $dT/dP$  with high  $dT/dP$ ) associated with this collision (Huang et al., 2020), which is one of the  
88 diagnostic indicators of plate tectonics in old rocks (Brown and Johnson, 2019).

89 UHP minerals ( $> 2.7$  GPa, the pressure needed to stabilize coesite) have been documented in  
90 young orogens from about twenty localities around the world (Hacker and Gerya, 2013; Gilotti,  
91 2013; Gonzalez et al, 2020), resulting in a paradigm shift in understanding how deeply  
92 continental and oceanic crustal material can be subducted, then returned to the surface. Some  
93 very significant developments in this field demonstrate that continental and oceanic material can  
94 be subducted to depths exceeding 150 km, and then returned to the surface in UHP orogenic  
95 belts (Hacker et al., 2013; Wang et al., 2014; Xia et al., 2018; Gonzalez et al., 2020). However,  
96 to-date, no orogenic UHP rocks have been discovered in Archean orogens.

97 UHP inclusions in chromites from podiform chromitites have been documented in ophiolites  
98 throughout the Phanerozoic (Dobrzhinetskaya et al., 2009; Robinson et al., 2015; Yang et al.,  
99 2007, 2014; Zhou et al., 2014; González-Jiménez et al., 2017; Lian et al., 2020) and represent  
100 windows into the P-T-t conditions operating on Earth. They have led to numerous models  
101 involving interaction of subducted lithosphere, deep mantle convection, and return of old crustal

102 material to be preserved within younger oceanic lithosphere (Robinson et al., 2015; González-  
103 Jiménez et al., 2017; Lian et al., 2020). We report the first unambiguous occurrence of an UHP  
104 mineral from an inclusion in chromite from a podiform chromitite block in a well-characterized  
105 Neoproterozoic ophiolitic mélange (Li et al., 2002; Kusky et al., 2016; 2020; Wang et al., 2019;  
106 Ning et al., 2020; Huang et al., 2021). We interpret this result to relate to plate tectonic processes  
107 that must have operated before 2.5 Ga and is significant for characterizing plate tectonic  
108 processes on early Earth, constraining the minimum depths of subduction, and tracking the  
109 interaction of surface tectonic-and deep mantle convective-recycling processes through deep  
110 time.

## 111 2. Geological background

112 Ophiolites and ophiolitic mélanges are both tectonic indicators of sutures where oceans  
113 have closed through subduction and collision (Festa et al., 2019; Kusky et al., 2018, 2020). The  
114 Zunhua ophiolitic mélange is a key component of the Central (Taihang) Orogenic Belt (COB)  
115 that separates the Eastern and Western Blocks of the NCC (Fig. 1a; Kusky et al., 2016, 2020).  
116 The mélange is composed of strongly deformed metasedimentary rocks (biotite-plagioclase-  
117 quartz (BPQ) gneiss), with structurally included blocks that show a strong affinity to rocks of the  
118 modern oceanic realm (Fig. 1b). These exotic mafic-ultramafic blocks include meta- pillow  
119 basalt, gabbro, pyroxenite, harzburgite and dunite, and podiform chromitites are common in  
120 dunite pods within the harzburgite blocks (Li et al., 2002; Huang et al., 2004; Kusky et al., 2004,  
121 2007, 2016, 2020; Wang et al., 2019). Together with lenticular structural slices of Banded Iron  
122 Formation (BIF) (submarine volcanogenic exhalative deposits), the map patterns and structural  
123 relationships (Fig. 1b) are indistinguishable from those of typical modern ophiolitic mélanges  
124 (Kusky et al., 2020). The most abundant mineral of the chromitite-bearing dunites and

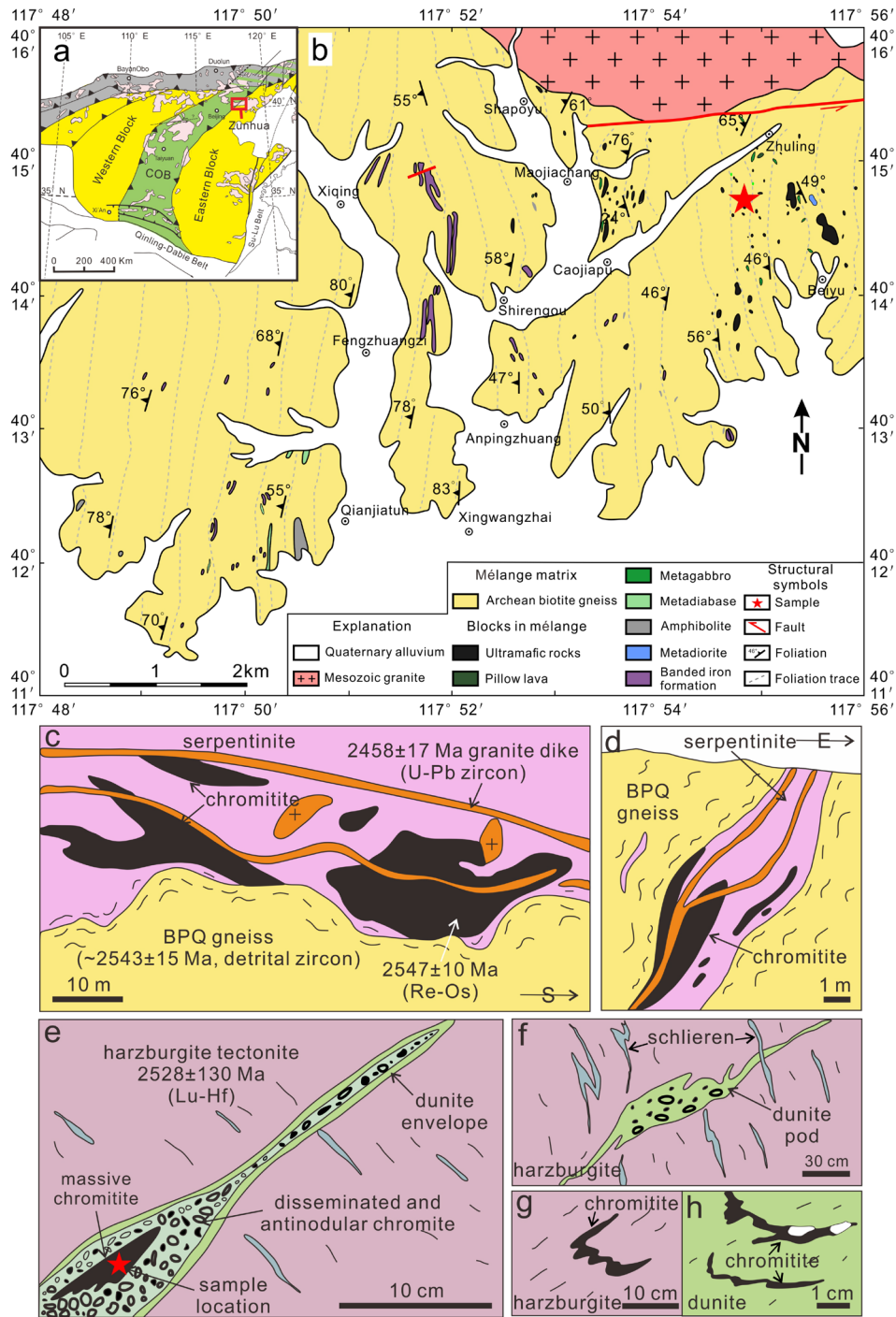
125 harzburgites is serpentine, with lesser amounts of chromite, talc, and magnesite accompanied by  
126 minor olivine (Ning et al., 2020). Thus, nearly all primary minerals of the host harzburgites and  
127 dunites have been altered to serpentine. However, mineral inclusions are found in the chromites  
128 and may preserve primary compositions if armoured by their host.

129 Most of the podiform chromite bodies at Zunhua are small, between 0.5-2 m thick, 1-2 m  
130 long, and extend up to 30 m in depth (Figs. 1c-f). Some however are larger, exceeding 100  
131 meters in length, and have been extensively mined (Kusky et al., 2007). Most of the larger bodies  
132 form irregular lenses of dunite including chromite in pod-like bodies in the host serpentinized  
133 harzburgite blocks (Fig. 1e), and within some of the pods, some of the chromites form igneous  
134 layers that locally show grading, magmatic folds, and asymmetric fabrics indicative of flow (Li  
135 et al., 2002; Huang et al., 2004; Kusky et al., 2007). Chromite layers within the pods are  
136 typically 1-2 cm thick, alternating with 10-50 cm thick dunite layers. The chromites have  
137 disseminated, nodular and orbicular textures that locally form cumulate layers within small  
138 magma chambers defined by the dunite pods (Huang et al., 2004). In addition to these chromites,  
139 the host harzburgite foliations are cut by thin veins of chromite with dunitic rims (Figs. 1g, h),  
140 suggesting a melt-host harzburgite reaction may have left residual chromite-dunite as pods (Li et  
141 al., 2002; Kusky et al., 2007). Ning et al. (2020) and Huang et al. (2021) show that the chromite  
142 has chemical signatures indicating that the infiltrating melt was Mg-rich, and likely boninitic.

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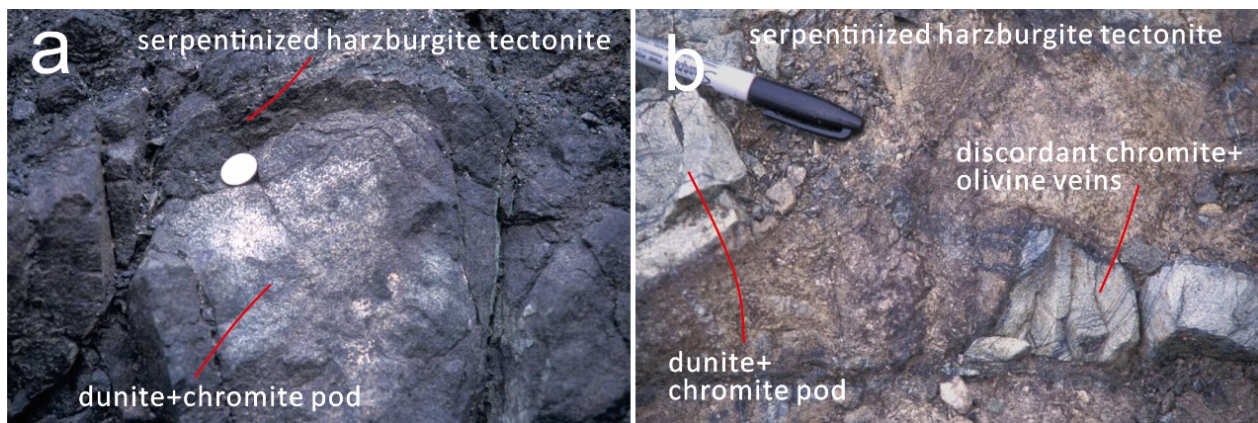


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146 **Fig.1.** Maps and outcrop sketches showing important geological relationships in the study area. (a) Tectonic map  
 147 showing the division of North China Craton (NCC) into the Eastern and Western Blocks, separated by the Central  
 148 (Taihang) Orogenic Belt (COB), and the location of the study area (after Kusky et al., 2016). (b) Detailed geological  
 149 map of the study area (mapping by the authors: Wang et al., 2019; Ning et al., 2020). The podiform chromitites

150 occur in blocks of serpentinized harzburgite tectonite and rare lherzolite (grouped as ultramafic rocks in legend).  
151 (c)-(h) Outcrop sketches of key relationships from map area of Fig. 1b. (c) Outcrop map and (d) cross section of the  
152 Maojiachang podiform chromite block (e) pod of chromite-bearing dunite cutting mantle tectonite fabric in  
153 harzburgite at Zhuling, showing a rim of dunite, and internal zones of disseminated and antinodular chromite, and  
154 massive chromitite, Red star shows location of sample with UHP inclusion. (f) dunite pods with disseminated  
155 chromite cutting mantle harzburgite tectonite with schlieren at Zhuling, (g) chromitite vein with dunitic envelope  
156 cutting harzburgite at Zhuling, (h) chromitite vein within serpentinized dunite at Zhuling. Outcrop sketches c-h  
157 modified after Huang et al., (2004). Outcrop TK-NCC-2002-580 is from the Zhuling body (location: N 40 14.739; E  
158 117 54.794), from which sample (massive chromitite, MC-20, see panel e) containing the UHP TiO<sub>2</sub> II inclusion  
159 was taken from the position shown in panel e.

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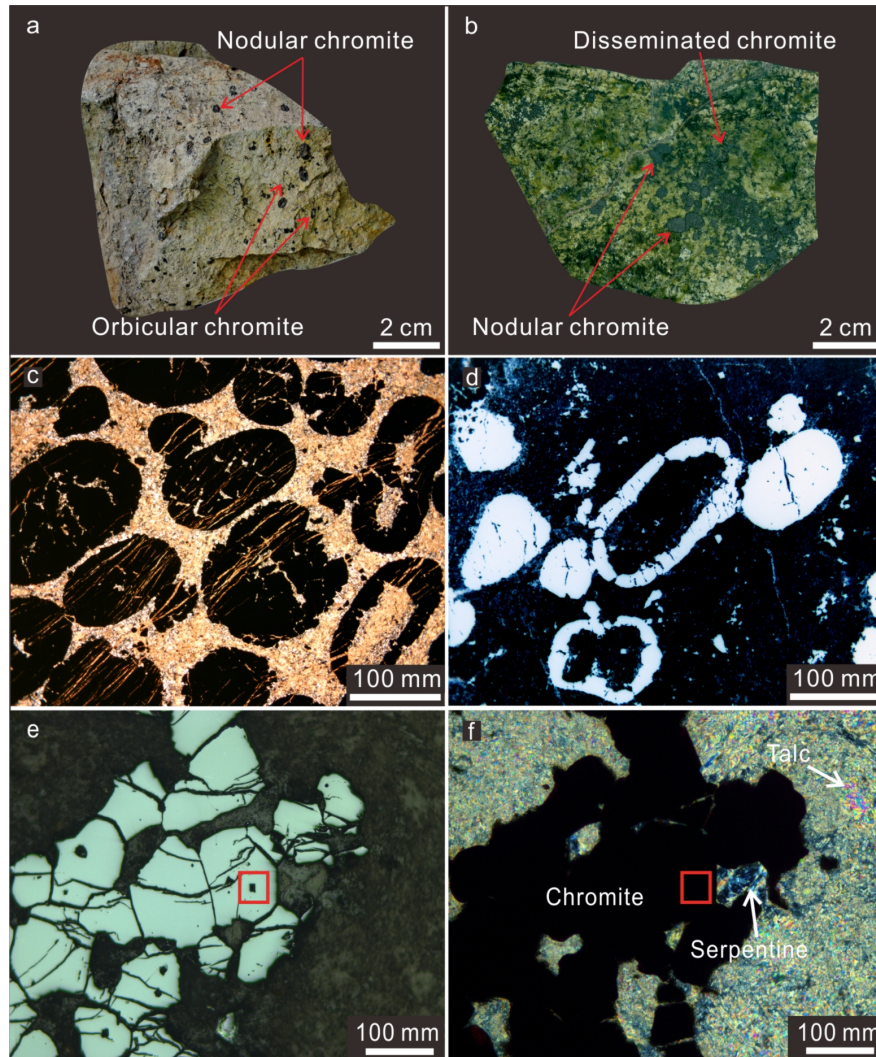


162  
163 **Fig. 2.** (a) Chromite pod with disseminated chromite surrounded by strongly foliated harzburgite tectonite; (b)  
164 chromitite in dunite in tabular pod in sheared serpentinized harzburgite, cut by thin discordant veins of chromitite  
165 with dunitic rims.

166

167 Podiform chromitites are only known to form in supra-subduction zone ophiolites (Li et  
168 al., 2002; Kusky et al., 2007). Those in the Zunhua ophiolitic mélange are hosted in dunite pods  
169 within harzburgites and show disseminated, massive, nodular and orbicular textures (Figs. 2, 3),  
170 clearly meeting the definition of typical ophiolitic podiform chromitites (Thayer, 1964).

171 Moreover, the mineral chemistry and platinum group element (PGE) geochemistry of the Zunhua  
172 chromitites ([Kusky et al., 2007](#); [Ning et al., 2020](#); [Huang et al., 2021](#)) are also similar to those of  
173 well-known Phanerozoic podiform chromitites worldwide ([Zhou, 2005](#); [Arai and Ahmed, 2017](#)).  
174 Outcrop-scale structures show high-temperature deformation in schlieren and chromite-  
175 mylonites, whereas microstructural studies of the Zunhua chromites ([Li et al., 2002](#); [Huang et al.,](#)  
176 [2004](#); [Polat et al., 2006](#); [Kusky et al., 2007](#)) documented high-temperature grain boundary pull-  
177 aparts, preferred crystallographic slip on (010)[100] slip systems in olivine inclusions,  
178 asymmetric recrystallized tails on orthopyroxene porphyroclasts, high-temperature deformation  
179 bands in inclusions in olivine, and used established paleothermometers ([Holtzman, 2000](#);  
180 [Nicolas, 1989](#); [Nicolas and Azri, 1991](#)), to estimate the crystallization temperatures of the  
181 chromite to have been between 1000 °C and 1250°C, consistent with recent high-pressure  
182 experimental work ([Raterron et a., 2012](#); [Wang et al., 2017](#); [Wallis et al., 2019](#)).  
183



184

185 **Fig. 3.** Photographs of nodular and orbicular textures of podiform chromitite from the Zunhua ophiolitic mélange at

186 Zhuling (**Fig. 1b**). (a) Hand specimen picture showing nodular chromites. (b) Scanning image of rock sample

187 showing chromites with nodular and disseminated textures. (c) Photomicrograph of a thin section showing nodular

188 and orbicular chromites (plane polarized light). (d) Photomicrograph of a thin section showing nodular and orbicular

189 chromites (reflected light). (e) Reflected light photomicrograph of the massive chromite band from the Zhuling

190 podiform location (TK-2002-NCC-580, thin section MC20). Red square indicates the FIB foil cutting position of the

191  $\text{TiO}_2$  II bearing chromite grain. (f) Thin section photomicrograph of the same area as (e) in cross-polarized light.

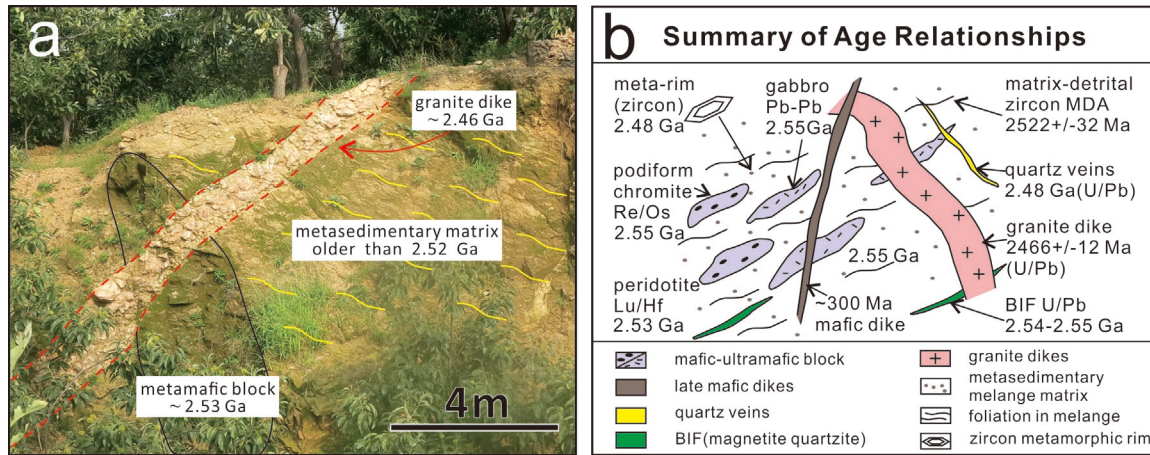
192 **Figs. 2e** and **2f** show the same position and viewing range, but with different light sources, causing slightly different

193 appearance of the chromite grains.

194

195 Age constraints on the formation of the mélangé, the blocks contained therein, and of the  
196 chromite grains are described in detail in [W.B. Ning et al. \(2020\)](#) and [J.P. Wang et al. \(2019\)](#), and  
197 briefly summarized here ([Fig. 4](#)). Blocks in the Zunhua mélangé have all yielded ages greater than  
198 2.5 Ga, with most, using various methods and multiple labs, falling between 2.55 and 2.52 Ga  
199 (review in [Kusky et al., 2020](#)). These ages include detrital zircons from magnetite quartzite blocks  
200 and lenses (2541-2553 Ma; [Zhang et al., 2012](#)), and  $^{207}\text{Pb}/^{206}\text{Pb}$  zircon ages from blocks of gabbro  
201 ranging from 2.51-2.55 Ga ([Kusky et al., 2020](#); [Wang et al., 2019](#); [Kusky et al., 2016](#)) and Lu/Hf  
202 ages on blocks of peridotite of 2528 +/- 130 Ma ([Polat et al., 2006](#)). Detrital zircons analyzed from  
203 the metasedimentary matrix of the mélangé (BPQ gneiss) yield age peaks between 2522 to 2633  
204 Ma, and the youngest detrital zircon has an age of 2522 +/- 32 Ma ([Wang et al., 2019](#)). Statistically  
205 the youngest group of detrital zircons (n=29) yields a weighted mean  $^{207}\text{Pb}/^{206}\text{Pb}$  age of 2543 +/-  
206 15 Ma ([Ning et al. 2020](#)), which we take as the maximum depositional age. Metamorphic rims on  
207 the detrital zircons from different sample sets yield a weighted means  $^{207}\text{Pb}/^{206}\text{Pb}$  age of 2467 +/-  
208 27 Ma ([Wang et al., 2019](#)), and 2481 +/- 32 Ma ([Ning et al., 2020](#)), within error of each other.  
209 Undeformed syenogranite dikes that cut the foliation in the mélangé have yielded ages of 2458 +/-  
210 17 Ma ([Wang et al., 2019](#)). The syenogranite, and metamorphic rims of the zircons all broadly  
211 overlap in age, and are associated with cross-cutting quartz veins, which have also yielded  
212 indistinguishable  $^{207}\text{Pb}/^{206}\text{Pb}$  ages of 2466 +/- 12 Ma ([Wang et al., 2019](#)).

213



214

215 **Fig. 4.** (a) Outcrop photo showing 2.46 Ga undeformed granitic dike, cutting circa 2.53 Ga mafic block, in a  
 216 metasedimentary mélangé matrix with an age > 2.52 Ga. (b) Schematic diagram summarizing the cross-cutting  
 217 relationships and ages determined from multiple methods that demonstrate the Archean age of the Zunhua ophiolitic  
 218 mélangé and contained ophiolitic podiform chromitites.

219

220 Of particular interest are the Re-Os range of the chromite grains, which were dated in two  
 221 different labs (Carnegie, DTM in Washington D.C., and University of Science and Technology of  
 222 China), with the result being an average Re-Os age of 2547 +/- 10 Ma for all the chromite grains  
 223 analyzed (Kusky et al., 2007). However, since these results did not yield a well-constrained  
 224 isochron, the individual model ages need to be considered since it is possible that there may be  
 225 chromites or material of different ages or sources recycled from the deep mantle in the chromite  
 226 deposits. With that in mind, we note that the model ages reported by Kusky et al. (2007) have one  
 227 group that clusters around 2.5 Ga (within errors), and a few model age determinations that are  
 228 clustered around 2.65-2.72 Ga. They report the best data from three massive chromitites (with Os >  
 229 300 ppb) yields chondritic osmium isotopic composition for the mantle at 2.6 Ga, of 0.110126 +/-  
 230 0.00004.

231 Taken together, the data indicates the mélangé formed between 2.55 and 2.52 Ga, incorporates  
 232 some older material, and metamorphic effects of the collision of the arc and continent lasted until

233 2.48-2.46 Ga (Kusky et al., 2020). The ages of the chromite grains is generally the same as the  
234 ages of the host peridotites and their derived melts, but a possibility that awaits further testing is  
235 whether there may be an older age population of chromite grains within the sample set.

236

### 237 **3. Methods**

#### 238 *3.1. Mapping and Sample collection*

239 Samples were collected in the field during detailed structural mapping of the area shown  
240 in Fig. 1b. Outcrop sketches and detailed sample locations are shown on Fig. 1c-g. Methods used  
241 for mapping (Li et al., 2002; Huang et al., 2004; Kusky et al., 2007; Wang et al., 2019),  
242 structural analysis (Li et al., 2002; Huang et al., 2004; Kusky et al., 2007), geochronology  
243 (Kusky et al., 2007; Polat et al., 2006; Wang et al., 2019; Ning et al., 2020), geochemistry (Ning  
244 et al., 2020; Huang et al., 2021), paleo-thermometry (Li et al., 2002), and petrography (Huang et  
245 al., 2004; Ning et al., 2020) are described in the cited papers above. Specialized techniques used  
246 in this work include Scanning Electron Microscopy (SEM), Focused Ion Beam (FIB) milling,  
247 and Transmission Electron Microscopy (TEM). Detailed analytical procedures are described in  
248 supplementary text S1 "Analytical Methods."

249

### 250 **4. Results**

#### 251 *4.1. Trapping of mineral inclusions in chromite grains*

252 Figure 5 shows textural relationships which with geochemical and other data (Ning et al.,  
253 2020; Huang et al., 2021) we use to propose a petrogenetic model in which an olivine-saturated  
254 melt (boninite) derived from partial melting above a slab reacted with orthopyroxene in depleted  
255 harzburgite of the mantle wedge to produce replacive dunite pods containing chromite. The  
256 dynamic flow of a hydrous melt in the initial dikes in lead to formation of nodular and orbicular

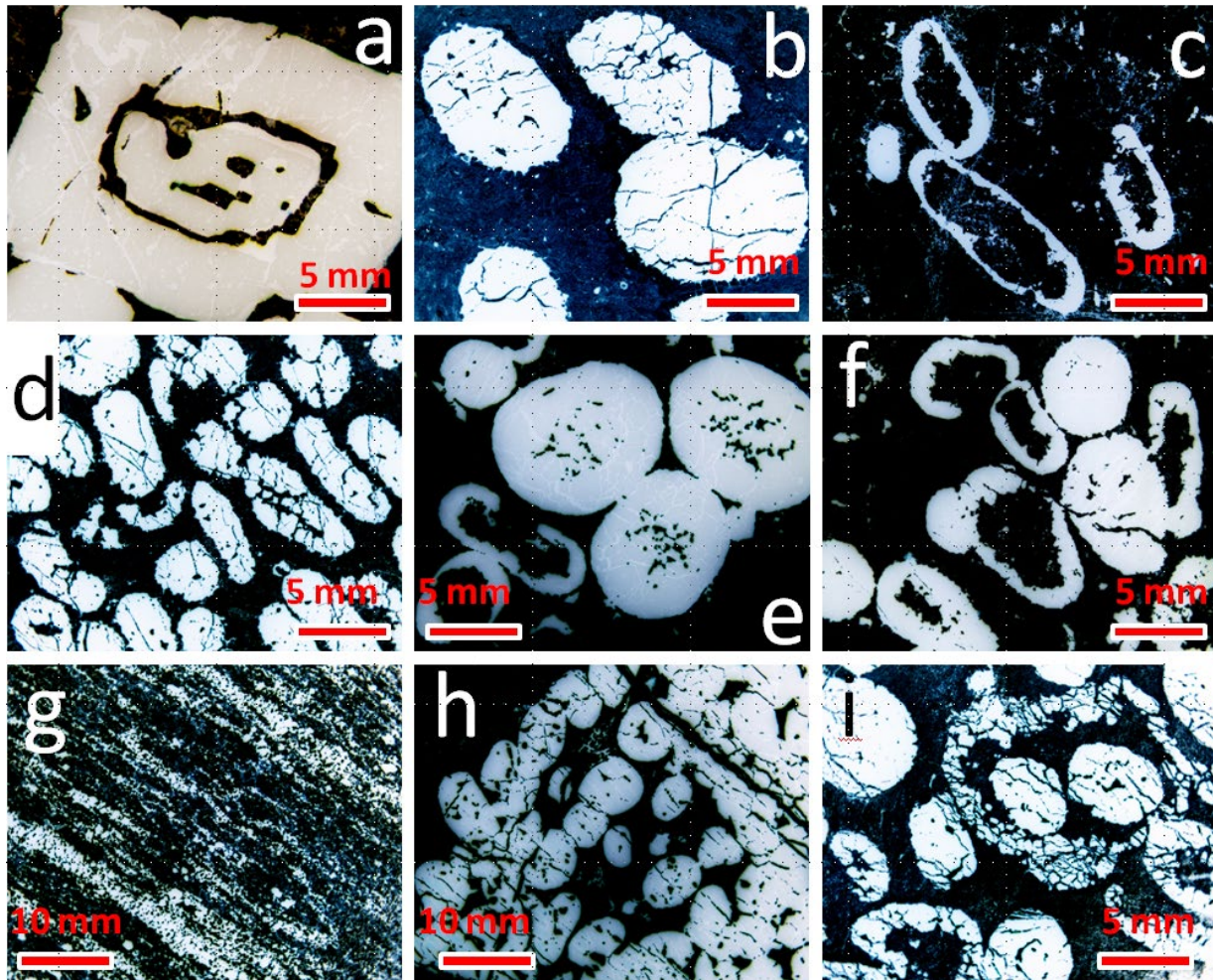
257 chromite initially as disseminated immiscible blobs, that formed inward-growing orbicules (Fig.  
258 5) then nodules that trapped host minerals and liquids. Eventually, the nodules became  
259 concentrated by melts or formed cumulate layers depending on the dynamics of the flow of the  
260 early melts and replacive reactions.

261 Both single and multi-phase mineral inclusions within the Zunhua chromite grains  
262 include silicates, PGM (platinum group minerals), base metal sulfides, carbonates and others that  
263 are very similar to those found in Phanerozoic ophiolites (c.f. [Dobrzhinetskaya et al., 2009](#);  
264 [Robinson et al., 2015](#); [Yang et al., 2007, 2014](#); [Zhou et al., 2014](#)). The most common single-  
265 phase inclusions are Os-Ir PGM and silicates including olivine, enstatite, diopside and tremolite  
266 (Table 1). Multi-phase inclusions include various combinations of silicate minerals, along with  
267 apatite and rutile. Carbonate minerals, particularly dolomite, are surprisingly abundant, and some  
268 native elements, such as Os and C are present (Table 1). Thus, some of the inclusions appear to  
269 be derived from the host mantle (e.g., olivine and enstatite were likely derived from harzburgite),  
270 whereas others (tremolite, apatite, carbonates, etc.) were likely derived from crustal material,  
271 although some could possibly represent mantle-derived minerals as well. In younger ophiolites,  
272 similar suites of mineral inclusions are thought to have been included in chromite grains after  
273 partial assimilation of the remnants of subducted slabs and overlying sediments in the mantle  
274 ([Yang et al., 2007](#); [Zhou et al., 2014](#); [González-Jiménez et al., 2017](#); [Lian et al., 2020](#)).

275

276





277

278 **Fig.5.** Typical microtextures of chromite grains in the Zunhua podiform chromitite bodies all taken in reflected light  
 279 microscopy. The chromite grains are bright, and the faint brighter white lines in some of the grains are more-altered  
 280 ferrit-chromite. The dark tones are serpentized dunite. (a) cubic grain of disseminated chromite. Note the ol-filled  
 281 negative crystals defining the "happy face" in the center of the crystal; (b) nodular chromite grains with cracks, and  
 282 small inclusions of the host dunite filling the negative cubic crystals; (c) orbicular chromite grains, with smooth  
 283 outer rims, and irregular inner rims with areas showing negative crystal faces suggesting inward growth of chromite,  
 284 capturing the host dunite in the cores; (d) nodular and semi-cubic chromite grains, some with inclusions of the host  
 285 ol melt that became flattened to form apparent cumulate igneous layering; (e) nodular and orbicular chromite that  
 286 grew inward from smaller grains, leaving a semi-open core. Note how the chromite grains are "stuck" together,  
 287 suggesting melt bubbles coalescing in an immiscible fluid; (f) nodules and orbicules of chromite that accumulated in  
 288 layers, with the impingement of grains breaking apart the fragile orbicules; (g) strongly deformed chromite

289 mylonite; (h) long strings of chromite nodules partially merged together that coalesced into a layer; (i) two nodules  
 290 of chromite enclosed within an orbicular nest.

## Inclusions in Zunhua Podiform Chromitites

<b>Mineral species</b>	<b>Silicate</b>		Ol, Di, Tr, Ph
	<b>Base-metal mineral (BMM)</b>		CuS, FeS, Ni <sub>2</sub> S
	<b>Platinum group minerals (PGM)</b>		OsS <sub>2</sub> , OsIrS
	<b>Carbonates</b>		Dol, Cal, Mgs
	<b>Phosphates</b>		Ap
	<b>Oxide</b>		TiO <sub>2</sub>
<b>Occurrence</b>	<b>Single phase</b>		PGM, TiO <sub>2</sub> , Ol, Di, Tr, Dol
	<b>Multiphase</b>	<b>Two</b>	Ol+Talc, Ol+Srp, Ol+Tr, Tr+Bt, Tr+Dol, Dol+Bt
		<b>Three</b>	En+Di+Ph, Tr+Ol+Ph, En+Ap+Bt, Ol+Ap+Bt, Dol+En+Ap, Dol+Tr+Bt, Tr+Di+Bt
		<b>Four</b>	En+Tr+Ap+Bt, Tr+Ap+BMM+Bt, Tr+Di+BMM+Bt, Tr+Ap+Dol+Bt, Tr+Di+Dol+Bt, Tr+Dol+Srp+Bt, Tr+Dol+BMM+Bt, Tr+Dol+BMM+Rt, Di+Dol+Cal+Bt

291

292 **Table 1.** Array of inclusions in chromite grains documented from the Zunhua chromitites using SEM-EDS.

293 Abbreviations: Ap-Apatite; BMM-Base metal mineral; Bt-Biotite; Cal-Calcite; Di-Diopside; Dol-Dolomite; En-  
 294 Enstatite; Ol-Olivine; PGM-Platinum group minerals; Ph-Phlogopite; Rt-Rutile; Srp-Serpentine; Talc-Talc; Tr-  
 295 Tremolite.

296

297 *4.2. Identification and Verification of TiO<sub>2</sub> (II)*

298 There is a wide range of different inclusions preserved within the Zunhua chromitites as  
 299 summarized in [Table 1](#). We focused on a multi-phase inclusion composed of apatite, amphibole,  
 300 rutile and a UHP polymorph of titanium dioxide (TiO<sub>2</sub> (II)) ([Fig. 6](#)). Results of our EDX semi-  
 301 quantitative analyses are shown in [Fig. 7](#). [Table 2](#) shows a comparison of d-spacings measured  
 302 from diffraction patterns of two HREM images and one SEAD image from this study with  
 303 calculated data of TiO<sub>2</sub> (II) from literature ([El Gorsey et al., 2001](#)).

**Table 2**

Comparison of d-spacings measured from diffraction patterns of two HREM images and one SEAD image from this study with calculated data of TiO<sub>2</sub> (II) from literature ([El Gorsey et al., 2001](#)).  $a_0 = 4.535 \text{ \AA}$ ,  $b_0 = 5.499 \text{ \AA}$ ,  $c_0 = 4.900 \text{ \AA}$

HREM obs (Å)		SAED obs (Å)	Mean obs(Å)	Calc (Å)	indexed planes of TiO <sub>2</sub> (II)
5.55	5.58	5.32	5.483	5.50	(010)
2.74	-	2.66	2.70	2.75	(020)
4.33	4.64	4.48	4.48	4.54	(100)
3.58	3.63	3.43	3.46	3.49	(110)
2.14	-	2.24	2.19	2.26	(200)

Note: Obs is short for observation; Calc is short for calculation.

305  
 306  
 307 To verify the structure of a crystal, it is necessary to further measure the lengths of diffracting  
 308 vectors that represent the  $1/d_{hkl}$  and angles between adjacent diffracting lattice planes. We  
 309 measured the angles between planes from two different FFT diffraction patterns and compared  
 310 them to calculated angles from known mineral phases. The diffraction patterns display the  
 311 following set of angles between planes: the angle between (110)/(100) =  $39.8^\circ$  ( $39.47^\circ$   
 312 calculated); the angle between (110)/(010) =  $50.2^\circ$  ( $50.53^\circ$  calculated) (more data in [Table 3](#)).  
 313 Based on the excellent match between observed and calculated data, the TiO<sub>2</sub> phase is  
 314 confidently and unambiguously identified as TiO<sub>2</sub> (II) with an orthorhombic  $\alpha$ -PbO<sub>2</sub> structure,  
 315 the ultra-high pressure (UHP) polymorph of rutile (TiO<sub>2</sub>).

316

317

**Table 3**

Angles between lattice planes of the observed TiO<sub>2</sub> (II) from this study and calculated TiO<sub>2</sub> (II) from the literature (El Gorsey et al., 2001).

Planes	Calc (°)	HREM obs (°)	SAED obs (°)
(110)   (100)	39.47	40.50	39.80
(110)   (010)	50.52	52.80	50.20
(220)   (200)	39.47	38.75	
(220)   (020)	50.52	53.00	
(120)   (100)	58.74		59.30
(120)   (020)	31.26		30.90
(130)   (100)	67.96		68.10
(130)   (030)	22.04		21.70

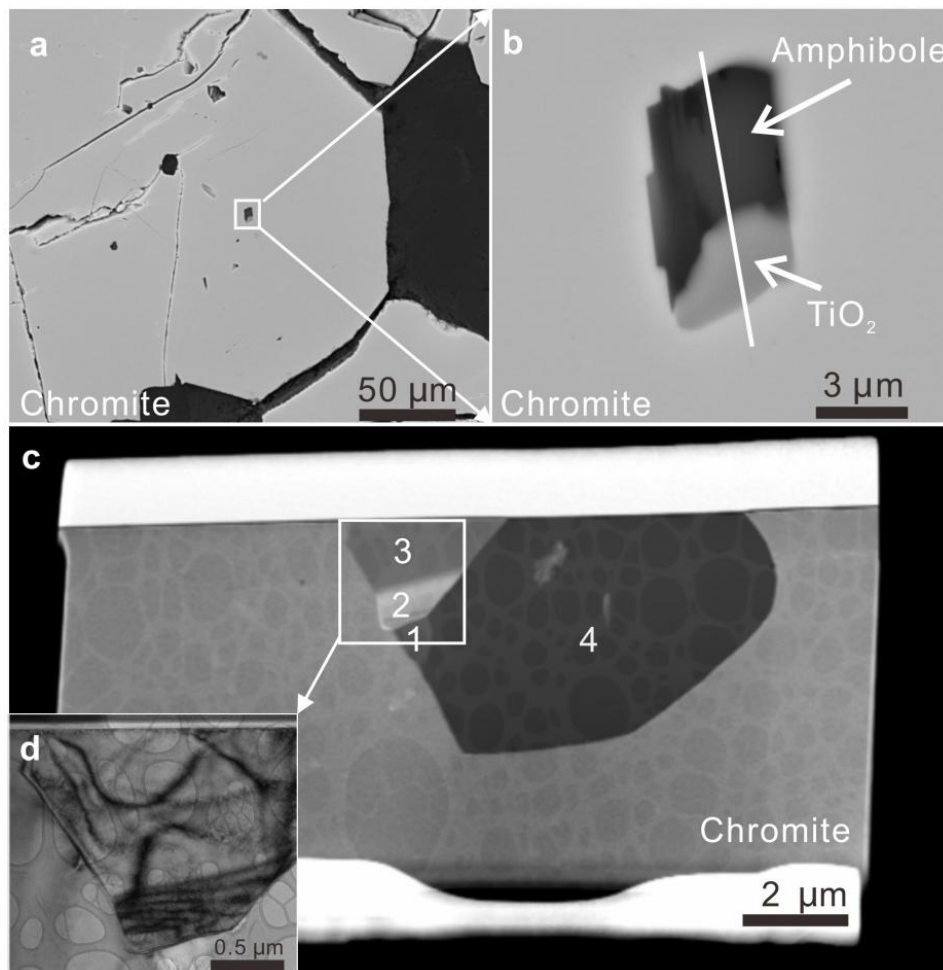
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319

### 320 4.3. Microstructural relationships of the multi-phase inclusion containing UHP TiO<sub>2</sub> (II)

321 We have identified the first Archean UHP inclusion in an orogenic podiform chromitite  
 322 (section 4.2). The host chromite grain for the TiO<sub>2</sub> (II) is in a chromitite sample from the  
 323 serpentinized dunite pod within harzburgite at the Zhuling location (Fig. 1 b,e). The host chromite  
 324 grain is 250 μm in diameter and is surrounded by altered fibrous minerals including serpentine  
 325 and talc (Fig. 3e,f). The multi-phase inclusion containing TiO<sub>2</sub> (II) is isolated, clearly separated  
 326 from any cracks, and thus is interpreted to be a primary feature (Fig. 6a). There is no evidence  
 327 for cracks in chromite even on a nanometer scale in the SEM images or in the TEM sample (Fig.  
 328 6). Therefore, the phases trapped inside the chromite pressure-vessel have been sealed and  
 329 isolated since they formed in the Archean, until we cut them open, so they were shielded from  
 330 fluids, and the pressure of the surrounding chromite grain did not allow decompression reactions  
 331 to take place; it has been a closed system for > 2.5 billion years. Most regional metamorphic  
 332 UHP rocks form in open systems, influenced by fluids and decompression of the host rocks  
 333 (Wang et al., 2014; Xia et al., 2018), whereas we are presenting data from a closed system within

334 the multi-phase inclusions encapsulated in the chromite. Our observations thus can be considered  
335 as a very rare occurrence of completely preserved inclusions in a host mineral.  
336



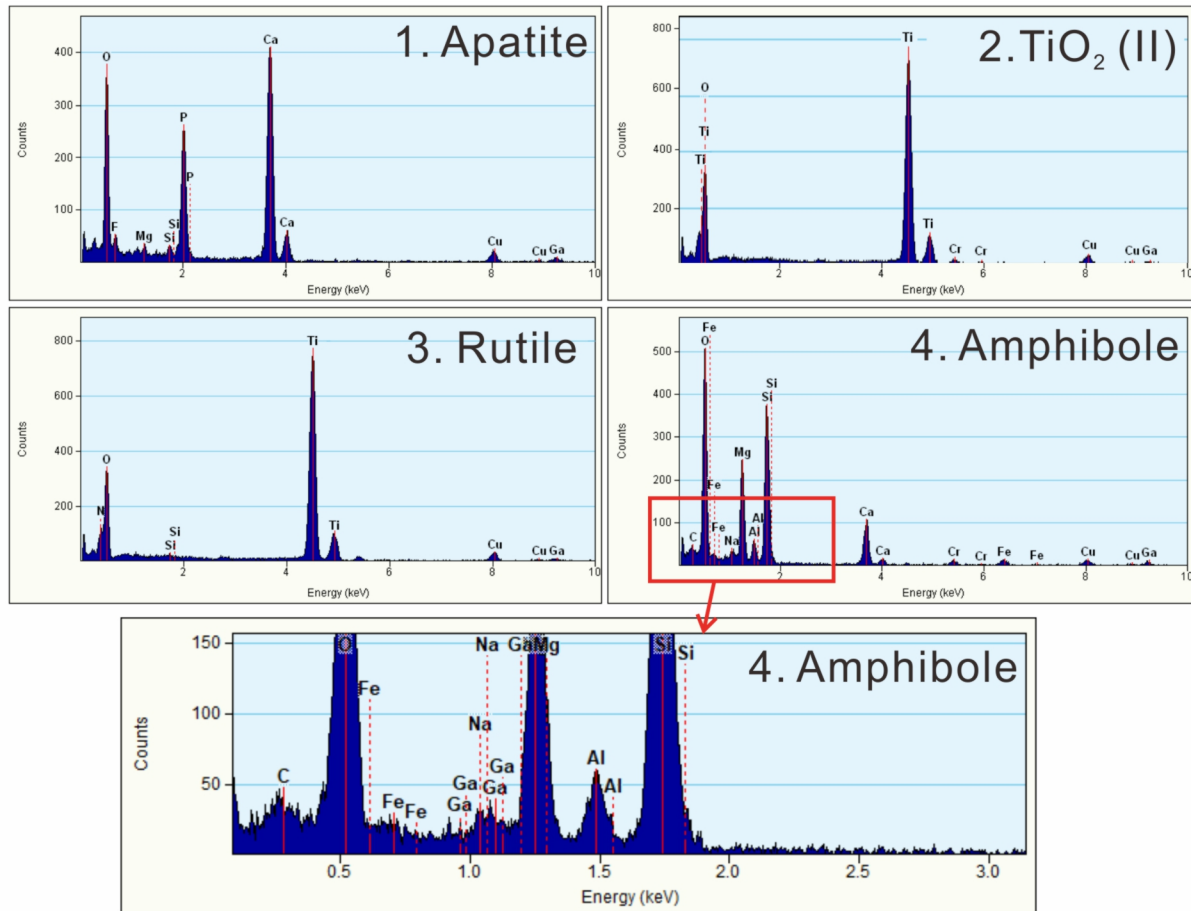
337  
338 **Fig. 6.** Back-scattered electron (BSE) image from SEM, high-angle annular dark field (HAADF) Z-contrast image,  
339 and EDX data from TEM of a chromite grain with a multi-phase inclusion, from sample location shown in **Fig. 1e**  
340 **and 1f.** (a) BSE image shows part of the chromite grain containing the inclusion. (b) BSE image of the multi-phase  
341 inclusion within the chromite grain. The white line shows the FIB cutting position. (c) HAADF Z-contrast image  
342 shows a multi-phase inclusion in the chromite. Note the perforated carbon support film in the background (making  
343 the leopard-skin appearance) on which the TEM foil is resting; the bright material at the top is a platinum protection  
344 layer and the bright layer at the bottom is re-deposited sputtered material. Four areas in the inclusion are defined by  
345 different grey scale contrasts; (1) apatite, (2) TiO<sub>2</sub> (II), (3) rutile, (4) amphibole. Note that the contrast of the TiO<sub>2</sub>

346 (II) grain is brighter than the rutile, because of its higher density. The apatite grain (1) is small and has a similar  
347 contrast to the adjacent amphibole.(d) Bright Field image of the selected area in (c). The rutile is characterized by  
348 curved dark diffraction contrast lines whereas the TiO<sub>2</sub> (II) polymorph shows 5 dark parallel diffraction contrast  
349 lines that are due to an inclined interface.

350

351 The multi-phase inclusion displays an irregular interface with chromite (Fig. 6b). Note the  
352 irregular shape of the interface between the inclusion and chromite has two rounded edges and  
353 an arrow-like appearance in the upper part. Overall, it is not the shape of a negative crystal in  
354 which the inclusions have nucleated and grown. The shape of the inclusion is affected by the size  
355 and arrangement of its component minerals, strongly suggesting an overgrowth of an existing  
356 mineral assemblage by chromite. The multi-phase inclusion (Fig. 6) is composed of TiO<sub>2</sub> and  
357 amphibole as determined by Energy Dispersive X-ray (EDX) analyses with SEM. After FIB  
358 cutting and preparation, apatite, amphibole, and two TiO<sub>2</sub> phases were identified using (EDX)  
359 analyses (Fig. 7).The EDX analysis on the apatite (F-rich) by TEM shows a composition of (in  
360 atomic percentage, simplified as in at. %): F = 7.71, P = 35.34, Ca = 56.93, and an analysis of  
361 amphibole by SEM shows a composition of (in atomic %): O = 68.90, Mg = 10.04, Si = 16.92,  
362 Ca=4.14. Rutile, F-apatite and amphibole are typical minerals in crustal rocks (F-apatite usually  
363 grows from silicate melt, not from magmatic-hydrothermal fluid) but TiO<sub>2</sub> (II) is a rare UHP  
364 phase found in both crustal and mantle rocks and meteorites (Dobrzhinetskaya et al., 2009;  
365 Hwang et al., 2000; Wu et al., 2005; Wirth et al., 2009; El Gorse et al., 2009).

366



367

368 **Fig.7.** EDX analyses of the multi-phase inclusion. The analysis positions are shown in [Fig. 3c](#).

369

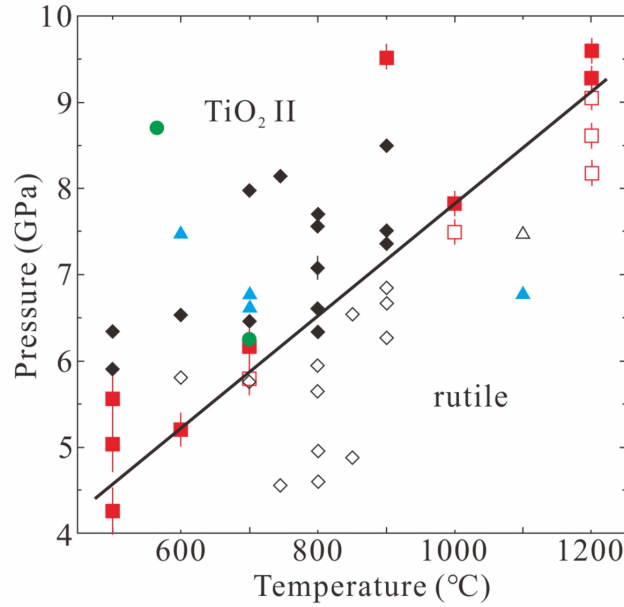
#### 370 4.4. Formation of UHP $\text{TiO}_2$ (II)

371 Although rare,  $\text{TiO}_2$  (II) has been previously found in several Phanerozoic UHP orogenic  
 372 settings, including as an inclusion in garnet of the Saxonian Erzgebirge diamondiferous quartzo-  
 373 feldspathic rocks ([Hacker and Gerya, 2013](#); [Gilotti, 2013](#)), in omphacite from coesite-bearing  
 374 eclogite ([Wu et al., 2005](#)), as an inclusion in coesite within a diamond ([Wirth et al., 2009](#)), and  
 375 associated with coesite within a podiform chromitite of the Luobusa ophiolite ([Dobrzhinetskaya](#)  
 376 [et al., 2009](#)). It is also known from shock-induced metamorphic rocks in meteorite impacts ([El](#)  
 377 [Gorsey et al., 2001](#)) and spherules in meteorite ejecta ([Smith et al., 2016](#)). However, we rule out  
 378 the possibility of a meteoritic spherule origin based on the well-documented mantle origin of the

379 Zunhua podiform chromites (Li et al. 2002; Huang et al., 2004; Kusky et al., 2004, 2007, 2016,  
380 2020; Polat et al., 2006; Wang et al., 2019; Ning et al., 2020), the lack of shock-induced minerals  
381 or microstructures in the samples, and the composition of the host chromite is totally different  
382 from that of meteoritic spherules, which are typically Fe-Ni alloys or silica formed by gas phase  
383 condensation of an impact-produced rock-vapor cloud (Johnson and Melosh, 2012), and which  
384 also lack volatile and hydrous phases such as amphibole and F-apatite that are present in the  
385 Zunhua samples.

386 Experimental constraints on the P-T conditions for the formation of  $\text{TiO}_2$  (II) (Withers et  
387 al., 2003) are shown in Fig. 8, with the pressure of the transition from rutile to  $\text{TiO}_2$  (II)  
388 increasing with increasing temperature. Based on the microstructures of the rutile, apatite and  
389 amphibole in SEM and TEM images (Fig. 6), these grains must have been overgrown by  
390 chromite during chromite crystallization. The encapsulating crystallization temperature was  
391 previously determined to be 1000-1200 °C (Li et al., 2002; Huang et al., 2004; Kusky et al.,  
392 2007) so we simply use the phase diagram (Withers et al., 2003) (Fig. 8) to estimate the pressure.  
393 This shows that the  $\text{TiO}_2$ - $\text{TiO}_2$  (II) phase transition in our samples should have happened at 7.5-  
394 9 GPa. Additional constraints on the pressure are obtained from phase relationships of the other  
395 phases in the inclusion.





396

397 **Fig. 8.** Phase diagram for rutile-TiO<sub>2</sub> (II), modified from Withers et al. (2003). Filled symbols represent TiO<sub>2</sub> (II),  
 398 and open symbols represent rutile. Squares are experimental results of Withers et al. (2003), diamonds are results of  
 399 Akaogiet al. (1992), triangles are results of Olsen et al. (1999), and circles are results of Tang and Endo (1994).

400

#### 401 4.5. Preservation and stability of UHP TiO<sub>2</sub> (II)

402 From the SEM and TEM images, we further note that there are no deformation features  
 403 or dislocations in any of the phases, and the shape of rutile continues into the α-PbO<sub>2</sub> structure  
 404 meaning that it was a single UHP crystal at the time of incorporation into the host chromite.  
 405 Furthermore, from the two diffraction patterns of the rutile and α-PbO<sub>2</sub> we obtain the lattice  
 406 plane spacing or hkl d-spacing ( $d_{hkl}$ ) and from that we determine which lattice planes are parallel  
 407 to each other yielding an orientation relationship of the lattice planes of the two phases:

408

$$\begin{aligned}
 409 \quad (100)_{Ru} &= 4.5937 \text{ (Å)} // (100)_{\alpha-PbO_2} = 4.535 \text{ (Å)} \\
 410 \quad (021)_{Ru} &= 1.8147 \text{ (Å)} // (030)_{\alpha-PbO_2} = 1.8330 \text{ (Å)} \\
 411 \quad (121)_{Ru} &= 1.6877 \text{ (Å)} // (130)_{\alpha-PbO_2} = 1.6978 \text{ (Å)}
 \end{aligned}$$

412

413 From these data it is obvious that the respective lattice plane spacings  $d_{hkl}$  are very similar,

414 showing that this particular orientation is energetically favorable. Additionally, we can determine  
415 parallel directions in the two crystals; for example, direction[0-12] in rutile is parallel to [001] in  
416  $\alpha$ -PbO<sub>2</sub>. Presenting the direction and the lattice planes fully describes the orientation relationship  
417 of rutile and  $\alpha$ -PbO<sub>2</sub>:

$$\begin{aligned} 418 & (100)_{\text{Ru}} // (100)_{\alpha\text{-PbO}_2} \\ 419 & (021)_{\text{Ru}} // (030)_{\alpha\text{-PbO}_2} \\ 420 & (121)_{\text{Ru}} // (130)_{\alpha\text{-PbO}_2} \\ 421 & [0-12]_{\text{Ru}} // [001]_{\alpha\text{-PbO}_2} \end{aligned}$$

422  
423 Thus, the rutile and  $\alpha$ -PbO<sub>2</sub> exhibit a good match of lattice planes. The phase transition rutile –  
424 TiO<sub>2</sub> in the  $\alpha$ -PbO<sub>2</sub> structure is a reconstructive phase transition, which is energetically  
425 facilitated by good matching lattices, as discussed below.

426 Chromite must have crystallized at ~1000–1250 °C, but temperatures above 1,200°C are  
427 not compatible with the presence of apatite. Apatite would have converted to tuite ( $\gamma$  -  
428 Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>), the high-pressure form of apatite) at 1200 °C based on experimental data on  
429 samples with a starting composition of MORB (Konzett and Frost, 2009). With experimental  
430 compositions of basalt and Mg-basalt (similar to that of the multi-phase inclusion), hydroxyl  
431 apatite was shown to be unstable above 7.5 GPa at 950°C (Konzett and Frost, 2009) (*In our case*  
432 *it is F-apatite, for which there are no experimental constraints, so we assume that it is similar to*  
433 *hydroxyl apatite*). Because what we observe is structurally apatite, not tuite, in the inclusion,  
434 with no deformation features or signs of a phase transformation from tuite to apatite, this  
435 provides an additional constraint on our previous temperature estimate of 1000–1250°C, putting  
436 us at the lower limit because of the presence of apatite. This temperature is also in good  
437 agreement with the PT stability of rutile and rutile in  $\alpha$ -PbO<sub>2</sub> (Withers et al., 2003) structure  
438 (TiO<sub>2</sub> (II)). We should observe apatite + rutile and/or  $\alpha$ -PbO<sub>2</sub>, in the inclusion at P-T conditions  
439 of 7.5GPa and 1000°C (Konzett and Frost, 2009), which is in agreement with the phases present,

440 and the temperature range estimated for the crystallization of the chromite around the inclusions.  
441 The amphibole (tremolite) is stable through the PT conditions mentioned, until it breaks down at  
442 1000–1100°C (Chernosky et al., 1998).

443 We therefore have excellent constraints for a temperature of approximately 1000°C to  
444 explain the presence of apatite and tremolite in the inclusions. That temperature yields a pressure  
445 of 7.5 GPa from both phase diagrams. These P-T conditions also explain why tremolite is still  
446 stable and did not transform into diopside +enstatite+quartz +water (Chernosky et al., 1998),  
447 providing yet an additional constraint for the P-T conditions being 7.5GPa and 1000°C.

448 Due to slow cooling from high temperature during exhumation we can assume a long  
449 thermal treatment (annealing) that removed any defects from the crystals. Accepting that TiO<sub>2</sub> –  
450 apatite and amphibole were overgrown by chromite at a pressure of 7.5–9 GPa (depth 270–330  
451 km) then TiO<sub>2</sub> was structurally  $\alpha$ -PbO<sub>2</sub> (chemically TiO<sub>2</sub> (II)). Thus, what we observe today in  
452 the multi-phase inclusion is a partial back-transformation of  $\alpha$ -PbO<sub>2</sub> into the rutile structure.  
453 That reconstructive phase transformation nucleated at the apatite/TiO<sub>2</sub> (II) phase boundary  
454 growing into the TiO<sub>2</sub> (II) phase. Back-transformation was stopped due to the volume increase  
455 during the back-transformation thus generating a pressure increase within the inclusion during  
456 uplift.

457

## 458 **5. Discussion**

### 459 *5.1. Deep subduction and recycling of crustal minerals in the Archean*

460 There are few direct ways to study Earth's deep interior, thus most inferences about the  
461 character of the deep mantle are based on studies of geophysics, meteorites, and high-pressure  
462 experiments. Only in rare cases can small samples such as high-pressure polymorphs of olivine

463 (ringwoodite) or  $\text{CaSiO}_3$  perovskite be brought from the deep mantle to the surface in kimberlitic  
464 diamonds (e.g., [Nestola et al., 2018](#)) or by mantle convection to be incorporated in podiform  
465 chromitites of ophiolites ([Dobrzhinetskaya et al., 2009](#); [Zhou et al., 2014](#); [González-Jiménez et](#)  
466 [al., 2017](#); [Lian et al., 2020](#)). Such minerals offer a rare direct way to investigate Earth's deep  
467 interior. Cubic chromite is an ideal micro-container of relict UHP mineral phases because it is  
468 mechanically strong, highly refractory, isotropic in thermal contraction and stable over a wide  
469 range of P-T conditions ([Dobrzhinetskaya et al., 2009](#); [Yang et al., 2014](#)).

470       Based on our microscopic, SEM, TEM, and Raman observations of the Zunhua samples,  
471 and their geological relationships, including thermal expansion coefficients and the bulk moduli  
472 of the concerned phases ([supplementary data file S2](#)), we propose the following hypothesis ([Fig.](#)  
473 [9](#)) for the formation and preservation of the inclusion and its UHP phase. Crustal material,  
474 including rutile, apatite, and amphibole (as well as the carbonates and other minerals in other  
475 inclusions) were part of a subducting oceanic slab that reached a depth of at least 270 km  
476 between 2.6 and 2.55 billion years ago (path a-b-c on [Fig. 9a](#)). At this stage the rutile converted  
477 to  $\text{TiO}_2$  (II) (with an  $\alpha\text{-PbO}_2$  structure) at approx. 7.5 GPa, but apatite and tremolite and  
478 chromite were still stable.

479       The crustal minerals (tremolite, apatite and UHP rutile or  $\text{TiO}_2$  II) were separated from the  
480 subducting slab and entrained in the circulating mantle as the slab rolled-back or in early stages  
481 of subduction, which both disrupt the normal flow of the mantle, inducing upward flow above  
482 the slab ([Kusky et al., 2014](#); [Stern and Gerya, 2018](#)) (path c-d on [Figs. 9a, 9b](#)). The exact process  
483 of transferring material from the lower plate to the overriding mantle wedge may have involved  
484 serpentinite diapirism of material from the subduction channel, intruding into the upper plate, as  
485 recently documented from American Samoa ([Jackson et al., 2007](#)), in the Calabrian arc ([Polonia](#)

486 [et al., 2017](#)), at Gaussberg, Antarctica (Murphy et al., 2002), and invoked as a general process  
487 that may even help explain the diversity of arc magmatism ([Codillio et al., 2018](#); [Cruz-Arbe et](#)  
488 [al., 2018](#); [Nielsen and Marschall, 2017](#)). [Zhang et al. \(2019\)](#) show experimentally that partial  
489 melting of mixed sedimentary/peridotite material from the subduction channel at conditions  
490 (~300 km) similar to ours (4-15 GPa, 1200-1800 C) is possible, producing melts with both  
491 melted peridotite characteristics and traces of the sedimentary material. We suggest the  
492 xenocrystic inclusions represent un-melted remnants of this material transferred from the  
493 subduction channel to the overriding mantle wedge. We estimate from the make-up of the multi-  
494 phase inclusion that it was originally a very small (unrepresentative) piece of mafic material  
495 from the slab that was entrained in the subduction channel and transferred to the overriding  
496 mantle wedge during subduction (path b-c-d on [Figs. 8a, 8b](#)). This was just one inclusion,  
497 probably transferred together with the other inclusions ([Table 1](#)) in a serpentinite-mélange diapir,  
498 characteristic of rocks we have mapped ([Fig. 1](#)) in the Zunhua mélange. The process is similar to  
499 that recently documented for serpentinite diapirs above subducting slabs that are shown to  
500 transfer material from the subducting to the overriding plate ([Polinia et al., 2017](#)), perhaps  
501 eventually forming schlieren in the harzburgite ([Fig. 1f](#) and [Fig. 8b](#), location d).

502 Numerical models ([Stern and Gerya, 2018](#)) show that slab material can be brought up to  
503 near the surface from > 200 km through the change in mantle circulation in the mantle wedge  
504 overlying the subducting plate during subduction initiation or slab rollback, and incorporated  
505 into fore-arc spreading systems. In models of [Stern and Gerya \(2018\)](#) both solid and molten parts  
506 of subducting oceanic lithosphere can be incorporated into the overlying rising mantle wedge,  
507 and the zone of melt generation in the mantle wedge above the slab extends below 200 km depth.  
508 Adiabatic upwelling of mantle to accommodate space created by slab rollback ([Kusky et al.,](#)

509 2014) (Fig. 9b) generates high-Mg melts, forming highly mafic dikes (likely boninitic as shown  
510 by Ning et al., 2020, and Huang et al. 2021), reacting with harzburgite to form remnant dunite +  
511 chromite pods that cut the harzburgite host with trapped xenocrysts from the mantle harzburgite,  
512 and crustal minerals derived from schlieren scraped off the down-going slab (location d on Fig.  
513 9b), at 7.5 GPa (270 km) and 1000°C. Thus, in addition to trapping crustal minerals such as the  
514 apatite, tremolite, rutile (TiO<sub>2</sub>) and carbonates (Table 1), small xenocrysts of ambient mantle  
515 were trapped as inclusions, including olivine, orthopyroxene (from a harzburgite host), sulfides,  
516 base metals and native elements (PGM) (Table 1) that may have come from deep mantle sources  
517 (c.f., Yang et al., 2014). The presence of F-apatite also implies that apatite, TiO<sub>2</sub> and amphibole  
518 were trapped at depths > 200 km, because F-apatite typically grows from a silicate melt whereas  
519 OH-apatite grows from magmatic hydrothermal fluids (Li and Costa, 2020).

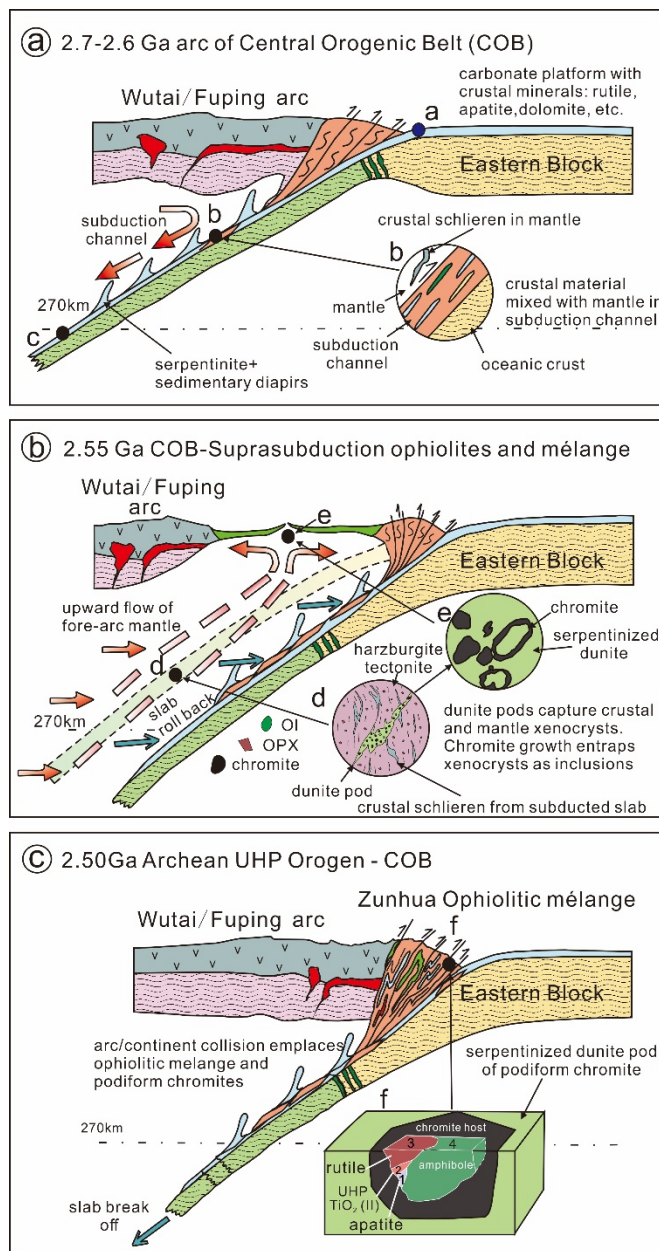
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522

523

524 **Fig.9.** Tectonic model for the formation and preservation of the UHP TiO<sub>2</sub> II inclusion in the ophiolitic podiform  
 525 chromite mélangé (vertical scale condensed). In panel (a), crustal material from the passive margin sequence and



from the accretionary prism is subducted to at least 270 km, where the rutile converts to the UHP phase TiO<sub>2</sub> II (path a-b-c). Flow in the mantle wedge parallels the subduction. Panel (b) shows slab rollback inducing a change in mantle flow to accommodate space created by slab rollback (Kusky et al., 2014) such that some of the deeply subducted material (plus xenocrysts of the ambient mantle, likely transferred to the upper plate in serpentinite diapirs (Polonia et al., 2017)) including the UHP-inclusion bearing chromites are encapsulated in the highly-mafic dikes and dunite pods. These are then entrained in upward flow (location d), and incorporated into the oceanic lithosphere (location e) at the crust/mantle interface in a fore arc spreading center (path c-d-e). Sketch in (b) for location d is based on outcrop data (Fig. 1e) from the field area, and for location e is based on thin section in Fig. 3d.

(c) Collision of the arc terrane with the continent (EB - East Block of the North China Craton) at 2.5 Ga emplaces the ophiolitic mélangé that preserves the

547 UHP inclusions in the podiform chromite blocks (position f), with accompanying late deformation and  
 548 metamorphism. Block diagram is sketch of thin section and surface of FIB foil through the inclusion with UHP TiO<sub>2</sub>  
 549 (II).

550

551 Numerical simulations of subduction processes (Stern and Gerya, 2018) show that soon  
552 after subduction initiation mantle flow is upward above the subducting slab (as in Fig. 9b), with  
553 temperatures above 1000°C, and a thin zone of melt brings mantle harzburgite with melts up into  
554 the fore-arc region to generate fore-arc ophiolites. In companion papers (Ning et al., 2020;  
555 Huang et al., 2021), we present evidence that the chromites from Zunhua interacted with a Mg-  
556 rich melt with boninitic affinity, which is considered characteristic of subduction initiation in  
557 forearc zones (Stern and Gerya, 2018; Rollinson, 2019). The mantle wedge at this stage is  
558 contaminated with serpentinite diapirs derived from the lower plate (Polinia et al., 2017),  
559 “schlieren,” and various small pieces of the subducted slab (location and sketch d in Fig. 9b), are  
560 incorporated into the magmas generated by partially melting the harzburgite (Fig. 9b). As the  
561 chromite grows within these highly mafic dikes and dunite pods it encapsulates the inclusions, at  
562 1000°C and 7.5 GPa. This is witnessed by the shape of the inclusion presented in Fig. 6b.  
563 Upwelling in the mantle circulates this segment of deep mantle upward to become the mantle  
564 section beneath a forearc spreading center at 2.55 Ga. At this stage our samples would have been  
565 transported from > 270 km to < 10 km depth, where they became part of the fore-arc oceanic  
566 crust of an intra-oceanic arc system (position e on Fig. 9b), that collided with the Eastern Block  
567 of the NCC (Fig. 9c), deforming the fore-arc ophiolite and forming the Zunhua mélange  
568 (position f on Fig. 9c), preserved in the upper plate of accretionary orogen on the surface today  
569 (Kusky et al., 2018; Wang et al., 2019; Ning et al., 2020).

570 During its rise to the surface the UHP TiO<sub>2</sub> (II) was preserved because it was sealed inside  
571 the impervious chromite pressure vessel (Fig. 9c). Thermal expansion of all phases, especially  
572 tremolite, allowed the UHP phase to stay at UHP conditions since entrapment. The extra pressure  
573 exerted by the expanding tremolite was greater than that of the smaller expansion of the host



574 chromite. This was countered by the opposite effect of the bulk moduli, as the external pressure  
575 decreased during exhumation ([supplementary file S1](#)). The thermal contraction of rutile and  
576 apatite and amphibole is 2X larger than that of chromite ([supplementary Table 1](#)). Consequently,  
577 rutile, apatite and amphibole shrink faster than chromite as the rock cools after crystallization,  
578 suggesting that no deformation will occur within the inclusions. If there had been defects in the  
579 rutile, apatite, and amphibole, they would have been healed by annealing during uplift thus  
580 moving the defects to the crystal surfaces or inclusion/host interfaces. However, there are no  
581 signs of damage or deformation in the rutile, apatite and amphibole. The above is consistent with  
582 all the phase relationships, mineral physics, microfabrics, and geological relationships. The  
583 combined field and P-T data on the inclusions provide unique constraints on the depth of  
584 subduction of both continental and oceanic material in the late Archean. In younger orogens,  
585 such as Sulu, the presence of intragranular coesite and inclusions in microdiamonds suggests  
586 subduction of continental material to at least 150 km ([Wang et al., 2014](#); [Xia et al., 2018](#)). In the  
587 case of Zunhua, inclusion of continental crustal material in the podiform chromitites is more  
588 complex, but indicates subduction to at least 270 km. The return flow was different from that in  
589 classical blueschist/eclogite terranes ([Hacker et al., 2013](#)), but may be applicable to  
590 understanding emplacement of UHP ophiolitic mélanges and chromitites in many Phanerozoic  
591 and Precambrian orogens ([Yang et al., 2014](#); [Kusky et al., 2018, 2020](#)).

592       There is still considerable controversy over the tectonic/dynamic processes that bring UHP  
593 inclusions in ophiolitic podiform chromitites to the surface in Phanerozoic ophiolites, but  
594 diamonds and suites of UHP and highly-reduced inclusions, very similar to those in the Zunhua  
595 podiform chromitites have now been identified in many ophiolites world-wide ([Dobrzhinetskaya](#)  
596 [et al., 2009](#); [Yang et al., 2007, 2014](#); [Lian et al., 2020](#)). The model we propose here may be

597 generally applicable to podiform chromites containing UHP inclusions of all ages throughout the  
598 world. Although our findings open many questions about the relationships between deep mantle  
599 dynamics, the deep carbon cycle, and lithospheric tectonics, our documentation of circa > 2.55  
600 Ga UHP inclusions in a 2.55 Ga ophiolitic podiform chromitite, along with a suite of inclusions  
601 similar to those in Phanerozoic podiform chromitites, shows clearly that the tectonic/dynamic  
602 processes that bring these deep mantle minerals back to the surface today, have been operating  
603 for at least the last 2.5–2.6 Ga, if not longer. Our results show that the structural and thermal  
604 conditions in Archean subduction zones and accretionary orogens were similar to those of the  
605 Phanerozoic.

606

#### 607 *5.2. Deeper implications for Archean tectonics.*

608 Our results document that the Central (Taihang) Orogenic Belt of the North China Craton  
609 is the world's first recognized UHP Archean orogen. The Central Orogenic Belt is also the  
610 world's first well-constrained spatially and temporally linked Archean paired metamorphic belt  
611 ([Huang et al, 2020](#)), preserving contemporaneous parallel belts of high dT/dP (720-1200  
612 °C/GPa) and intermediate dT/dP 425-600 °C/GPa) which is considered diagnostic of the  
613 asymmetric thermal structure of Phanerozoic subduction systems ([Brown et al., 2020](#)), showing  
614 that the Central Orogenic Belt of the NCC is the world's first-recognized Archean UHP paired  
615 metamorphic accretionary orogen. Together, this is the strongest evidence yet for the operation  
616 of asymmetric subduction to depths approaching the mantle transition zone, showing similar  
617 depth scales of Phanerozoic and Archean orogens.

618 The length scales of subduction in the Central (Taihang) Orogenic Belt are on a scale of  
619 thousands of kilometers (the orogen, or paleo-subduction zone, is 1600 km long) much like that

620 on present-day Earth. The time scales of the early subduction have been constrained in our  
621 related work to have been at least 2.6–2.55 Ga; [Ning et al., 2020](#)), or 2.68-2.52 Ga ([Kusky et al.,](#)  
622 [2020](#)). This was followed by fore-arc extension and arc evolution (2.55-2.52 Ga; [Ning et al.,](#)  
623 [2020](#); [Deng et al., 2018](#)), to collision, well constrained by the ages of metamorphic minerals in  
624 syn-collisional fabrics, and cross-cutting undeformed igneous dikes (2.50 Ga; [Wang et al., 2019](#);  
625 [Xiao et al., 2021](#)). Late stages of the end-Archean orogeny were marked by uplift and erosion of  
626 the orogen to produce a foreland basin (2.48 Ga; [Huang et al., 2019](#)) with associated high-grade  
627 metamorphism in the hinterland (2.48 Ga; [Kusky et al., 2016](#)), and arc-polarity reversal (2.50-  
628 2.48 Ga; [Kusky et al., 2016](#); [Deng et al., 2018](#)). The time scales of these events in the Central  
629 (Taihang) Orogenic Belt are all remarkably similar to the time scales of contemporary (i.e.,  
630 Banda arc/Australia collision) and Phanerozoic examples of arc continent collisions (Pliocene of  
631 Taiwan; Cretaceous for the Caribbean arc; Oligocene in the Appinides; Miocene across the  
632 Philippines; and Ordovician up in the Grampian), typically lasting only a few to tens of millions  
633 of years ([Brown et al., 2011](#)). Thus, we have shown that the length, depth, and time scales of late  
634 Archean convergent margin processes were all indistinguishable from those of the Earth in the  
635 past 500 million years.

636           Because of the higher heat production in the Archean ([Korenaga, 2013](#); [Herzberg et al.,](#)  
637 [2010](#)) there has been much modeling and speculation that mantle temperatures may have been  
638 significantly higher, thus preventing subduction (c.f. [van Hunen and Moyen, 2012](#)), or may have  
639 been significantly different from the modern style of subduction (c.f. [Zheng and Zhao, 2020](#);  
640 [Kusky, 2020](#)). However, [Aulbach and Arndt \(2019\)](#) have argued that mantle temperatures have  
641 not exceeded present values by more than 100°C over the past 3.0 Ga, and [Agrusta et al. \(2018\)](#)

642 use numerical modeling to suggest that warmer mantle temperatures set up conditions that  
643 enhanced deep subduction and facilitated recycling of volatiles to the deep mantle.

644 Another line of argument against modern-style plate tectonics in the Archean is based on  
645 the now-disproven lack of documentation of UHP phases in Archean orogens, lack of  
646 documented spatially and temporally linked paired metamorphic belts, and lack of ophiolites and  
647 mélanges in the Archean record (e.g., [Stern, 2008](#)). In this work we document that all such  
648 features are present in the Central (Taihang) Orogenic Belt of the NCC. In other works, we have  
649 documented Archean ophiolitic fragments (ophirags), and ophiolitic mélanges, showing clear  
650 evidence of seafloor alteration, throughout the 1600 km long COB (summarized in [Kusky, 2004](#);  
651 [Kusky et al., 2020](#); [Jiang et al., 2020](#)). With the clear documentation now of all of the above key  
652 tectonic indicators for convergent margins and deep subduction in the Archean, we argue with  
653 confidence plate tectonics has been operating at least since the late Archean, and possibly longer  
654 (e.g., [Kusky et al., 2018](#); [Windley et al., 2021](#)).

655 It has been argued that plate tectonics can not be proven to have operated on Earth until it  
656 can be proven that there was an established globally linked network of weak plate boundaries  
657 ([Lenardic, 2018](#); [Brown et al., 2020](#)). However, as pointed out by [Windley et al. \(2021\)](#) and  
658 [Kusky et al. \(2021\)](#) this is a non-testable proposition, since the size of the preserved Archean  
659 regions (generally cratons or fragments within them) is remarkably small, with many regions  
660 (such as Isua, or Nulliak, or Nuvaguttiuk) only tens of km in area, and others such as the eastern  
661 Pilbara only measuring 200 x 200 km. The Central (Taihang) Orogenic Belt is 1600 km long,  
662 making it one of the largest well-studied Archean orogens, and having established geological  
663 signatures of subduction along the length of this orogen for tens to hundreds of Ma, to depths  
664 exceeding 270 km is more than a “local” effect. Through comparative tectonic analysis using the

665 geologic indicators of plate interactions, we suggest that the Central (Taihang) orogen is but one  
666 of many preserved plate boundaries from the late Archean, with others in the Superior Province  
667 (Percival et al., 2012; Kusky and Hudleston, 1999), Yilgarn (Kusky et al., 2018), and most other  
668 cratons, as summarized in Windley et al. (2021).

669

## 670 **6. Conclusions**

671 The presence of both crustal and mantle mineral phases in an Archaean orogenic  
672 ophiolitic podiform chromite mélange demonstrates that deep subduction of shallow crustal  
673 materials and return flow took place in the Neoproterozoic. Our discovery of the UHP phase, TiO<sub>2</sub>  
674 (II), provides direct physical evidence for plate tectonics in the Archean and direct mineralogical  
675 evidence for deep subduction on early Earth. The Central (Taihang) Orogenic Belt of the North  
676 China Craton is the world's first-recognized ultra-high pressure (UHP) paired metamorphic  
677 orogen.

678

679

## 680 **Author Contributions**

681 The project was conceptualized by T.K., designed by T.K., L.W., R.W., P.T.R., and A.P., and  
682 was administered by T.K. and L.W. Funding was obtained by T.K., L.W., Y.H., A.P., and  
683 P.T.R.. Field work was conducted by T.K., W.L., W.B.N., A.P., Y.H. and Y.Z. Analytical results  
684 were obtained by Y.H., R.W., L.W, and P.T.R.. Assessment of data, construction of models and  
685 regional results, writing and drafting were done by all authors.

686

## 687 **Declaration of Competing Interests**

688 The authors declare no competing financial or other interests.

689

## 690 **Data Availability**

691 All data used in this manuscript are included in the text, Supplementary Data, or in the cited  
692 published manuscripts.

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702

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980 **Contents of Supplementary Text**

981

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990 the UHP phase

991

992 **1. Analytical methods**

993 **1.1. Scanning electron microscopy (CUGW)**

994 Scanning electron microscopy (SEM) with secondary electron imaging and back-scattered  
995 electron modes was used to observe the morphology of chromite grains. The composition of  
996 selected targets was acquired by SEM with energy-dispersive X-ray spectroscopy (EDS). SEM  
997 analyses were carried out in the State Key Laboratory of Geological Processes and Mineral  
998 Resources, China University of Geosciences, Wuhan using a FEI Quanta 450 field emission SEM.  
999 Standard operating conditions for SEM imaging and EDS analyses were 10–20 kV accelerating  
1000 voltage, working distance of 10–13 mm, and electron beam current of 1 nA. Thin section samples  
1001 were coated with a few nanometre of carbon prior to analysis.

1002

1003 **1.2. Mineral phase identification by TEM. 1. FIB Sample Preparation (GFZ)**

1004 Analysis by transmission electron microscopy (TEM) requires electron transparent  
1005 samples, obtained by milling thin foils with a thickness less than 0.15  $\mu\text{m}$  (Wirth, 2009). Typical  
1006 electron transparent foils made by FIB have dimensions of  $15 \times 10 \times 0.15 \mu\text{m}$ . The FIB instrument  
1007 (FEI FIB 200 TEM) operated at the GeoForschungsZentrum (GFZ), Potsdam was used for  
1008 sample preparation. This single beam instrument allows lift-out of samples as ex-situ lift out  
1009 only, following the procedures of Wirth (2004, 2009). Selected samples and locations where the  
1010 foils were obtained were first studied under a petrographic microscope and SEM. For preparation  
1011 of the foils, a Ga-ion beam (30 keV acceleration voltage) was focused onto selected locations of  
1012 thin section surfaces to sputter material from the chromite grains.

1013

1014 **1.3. Mineral phase identification by TEM. 2. Transmission Electron Microscopy (GFZ)**

1015 TEM investigations were performed using a TECNAI F20 XTWIN TEM operated at 200  
1016 kV with a field emission gun (FEG) as the electron source at the GFZ in Potsdam, Germany. The

1017 TEM is equipped with a GatanTridiem™ energy filter, an EDAX Genesis™ X-ray analyzer with  
1018 an ultra-thin window, and a Fishione high-angle annular dark field detector. A Tridiem energy  
1019 filter was used for acquisition of bright and dark field images as well as high-resolution images  
1020 applying a 20-eV window to the zero-loss peak. EDX spectra were acquired using the TIA  
1021 software package in the scanning transmission mode of the TEM. To minimize mass loss due to  
1022 electron sputtering during data acquisition the electron beam was scanned within a preselected  
1023 area. The acquisition time of EDX spectra was 60 s.

1024

#### 1025 **1.4. Mineral phase identification by TEM. 3. Mineral Phase Confirmation**

1026 In situ EDX analysis was carried out on the target inclusion sample as the first step to  
1027 obtain its semi-quantitative chemical components. This reduces the possible range of the  
1028 unknown phase into a few choices of minerals that have similar chemical components but  
1029 different crystal structures (polymorphs). Then, electron diffraction patterns from mineral phases  
1030 were recorded on image plates by selected area electron diffraction (SAED) from which the  
1031 observed  $d_{hkl}$  lattice plane distances of the mineral phases were measured. One image typically  
1032 contains several diffraction spots that can be assigned to different lattice planes in the crystal.  
1033 From the diffraction spots (diffraction vectors), we calculated spacings between the lattice  
1034 planes and the angles between adjacent vectors. Based on the chemical composition measured  
1035 with EDX we deduce the presence of a particular phase. If the observed d-spacing and angles  
1036 between adjacent planes match the calculated d-spacings and angles from a known structure, an  
1037 unambiguous identification of the phase is possible. Error of measurement angles is  $< 0.5^\circ$  in  
1038 electron diffraction patterns. Based on measured different  $d_{hkl}$  values the unit cell parameters can  
1039 be calculated.

1040

1041 **S.2.Supplementary File 2.**

1042

1043 **2.Evaluation of the possible role of overpressure on the formation and preservation of the**  
1044 **UHP phase**

1045

1046 To test if volume and pressure changes were important for the inclusions during  
1047 increasing P-T conditions (subduction) or decreasing conditions (exhumation), we used data  
1048 from the thermal expansion/contraction tables (Ahrens, 1995) for our estimates of possible  
1049 overpressure within the inclusion vessel. Using the textural phase relationships described above,  
1050 and considering the bulk moduli and thermal expansion coefficients for the phases concerned  
1051 (Supplementary Table 1), it is possible that the phase transition occurred at the lower end of our  
1052 pressure window (7.5 GPa), rather than the high-end estimate of 9 GPa. This suggestion is based  
1053 on experimental data for the transition of apatite to its UHP equivalent, known as tuite (Konzett  
1054 and Frost, 2009). Because we do not observe any signs of a phase transformation in our apatite  
1055 grain, the phase diagram of Konzett and Frost (2009) suggests that our **maximum** estimate of 9  
1056 GPa (330 km) at 1,200°C is still within the stability field of apatite. On the other hand,  
1057 estimating the **minimum** P-T conditions possible for our sample, it appears that both apatite and  
1058 TiO<sub>2</sub> (II) could be stable at a temperature of 1,000°C and a pressure of 7–8 GPa. However, we  
1059 need to consider a possible internal overpressure in the inclusion, because the thermal expansion  
1060 coefficients ( $\alpha_0$  ( $10^{-6}$ )) of the phases in the inclusion are larger than that of the confining chromite  
1061 grain (Supplementary Table 1). Thus, if the crustal minerals that form the inclusion were trapped  
1062 by a chromite grain growing from a highly mafic (boninitic) melt at 7–8 GPa (as shown by the

1063 textural relationships described above), their temperature would suddenly rise from that of the  
 1064 cooler ambient slab that brought them to the appropriate depth, but would then cool slowly as  
 1065 they rose to the surface. Because the inclusions have a larger thermal expansion coefficient than  
 1066 the host chromite ([Supplementary Table 1](#)), they may have originally expanded slightly, thus  
 1067 increasing internal pressure. Thus, it may not have been necessary to subduct the sample as far as  
 1068 our maximum pressure boundary (9 GPa or 330–430 km) to initiate the transition of rutile to  
 1069 TiO<sub>2</sub> (II). For example, such an overpressure may allow the sample to achieve an internal  
 1070 pressure >7.5 GPa at depths as shallow as 270 km. So, in total our observations are in accordance  
 1071 with experimental data, and all indicators are consistent with formation of the TiO<sub>2</sub> (II) at 7.5  
 1072 GPa and 1,000°C.

1073 We used the following thermal expansion/contraction values ([Ahrens, 1995](#)) for our  
 1074 estimates of possible overpressure within the inclusion vessel:

**Supplementary Table 1**

Thermal Expansion and Bulk Modulus Coefficients. (from Mineral Physics and Crystallography  
 AGU Reference Shelf 2: [Ahrens, 1995](#)).

Mineral	T range (K)	$\alpha_0$ ( $10^{-6}$ )
Picochromite	293-1473	16.5
Rutile	298-1883	28.9
Tremolite	297-973	31
Apatite	297-	34

<b>Bulk Modulus</b>	
Mineral	Ks = adiabatic bulk modulus (Gpa)
Apatite	212.3
Chromite	203.3
Rutile	215.5
Amphibole	87-93

1077  
 1078 *Bulk modulus describes which confining pressure is required to reduce the volume of a phase.*  
 1079



1080 Of course, we cannot completely exclude the possibility that the apatite was a UHP phase  
1081 (tuite) originally that also transformed back during cooling to simple apatite, and the pressures  
1082 were higher than we estimate. Although, we observed no features of back transformation in the  
1083 apatite, it is possible that such defects, if they existed, could have been healed completely by  
1084 annealing during the uplift phase.

1085 During exhumation, all phases would have slowly cooled with the thermal contraction of  
1086 the inclusions being greater than the host chromite by virtue of their larger  $\alpha_0$  (thermal expansion  
1087 coefficient) (Supplementary Table 1). However, this would be modulated during uplift by the  
1088 adiabatic bulk modulus ( $K_s$ ) which is large for all of the observed phases except amphibole,  
1089 which would expand slightly faster than the host, meaning that the internal pressure would have  
1090 been maintained within the pressure vessel during exhumation.

1091

## 1092 **Supplementary References**

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