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## High-resolution petrographic evidence confirming detrital and biogenic magnetites as remanence carriers for Zongpu carbonates in the Gamba area, South Tibet

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### 10 Abstract

11 Paleocene carbonates from the Gamba area of South Tibet provide the largest paleomagnetic  
12 dataset for constraining the paleogeography of the India-Asia collision in the early stage. Previous  
13 studies argued that the characteristic remanences (ChRMs) obtained from this unit were  
14 remagnetized via orogenic fluids. This study carries out a high-resolution petrographic study on the  
15 Paleocene carbonates from Gamba aiming to test the nature of the ChRMs. Electron microscopic  
16 observation on magnetic extracts identified a large amount of detrital magnetite that are multi- to  
17 single domain in sizes and nanoscale biogenic magnetite. Minor framboidal iron oxides were also  
18 identified, which were previously interpreted as authigenic magnetite that substitutes pyrite.  
19 However, our scanning and transmission electron microscopic (SEM/TEM) observations, along with  
20 optical microscope and Raman spectrum investigations further suggest that these magnetic minerals  
21 are pigmentary hematite and goethite that are incapable of carrying a stable primary magnetization.  
22 We therefore argue that the ChRMs of the limestones from the Zongpu Formation in the Gamba area  
23 are carried by detrital and biogenic magnetites rather than authigenic magnetite. The paleomagnetic  
24 data from the Gamba area are interpreted as primary origin and can thus be used for tectonic  
25 reconstructions. We emphasize that magnetic extraction, integrated with advanced mineralogic  
26 studies (e.g., electron backscatter diffraction and electron diffraction) are effective approaches for  
27 investigating the origin of magnetic carriers in carbonate rocks.

### 28 1 Introduction

29 Consecutive indentation of India into continental Asia resulted in a rapid uplift of the Tibetan  
30 Plateau that has profoundly changed the climatic pattern and topography of Asia since the Cenozoic  
31 era (Yin and Harrison, 2000; Jagoutz et al., 2016). The timing and position of the initial collision  
32 between India and Asia remain highly debated (e.g., Ding et al., 2005; Leech et al., 2005; Aitchison

33 et al., 2007; Ali and Aitchison, 2008; Najman et al., 2010; Yi et al., 2011; van Hinsbergen et al.,  
34 2012; Hu et al., 2016; An et al., 2021). On the paleolatitudinal comparison based on reliable  
35 paleomagnetic poles, paleomagnetism provides a direct constrain on timing and locus for the initial  
36 collision between India and Asia (e.g., Dupont-Nivet et al., 2010; Najman et al., 2010; Yi et al.,  
37 2011, 2021)

38 The Indian plate was subjected to rapid northward motion toward Asia during the Cretaceous  
39 and Paleocene (Patriat and Achache, 1984; Yin and Harrison, 2000; van Hinsbergen et al., 2011).  
40 The kinematics of the northern margin of India can be constrained by the Cretaceous and Paleogene  
41 paleomagnetic data obtained from the Tethyan Himalaya (Besse et al., 1984; Patzelt et al., 1996;  
42 Tong et al., 2008; Yi et al., 2011; Yang et al., 2015, 2019; Ma et al., 2016; Meng et al., 2019, 2020;  
43 Y. Zhang et al., 2019; Yuan et al., 2020). For the lack of contemporary volcanic rocks, the Late  
44 Cretaceous to Paleocene sedimentary rocks from the Tethyan Himalaya are especially crucial for  
45 reconstructing the overall process of the India-Asia collision. Several paleomagnetic poles were  
46 reported from the marine sediments of the Tethyan Himalaya with the Late Cretaceous to Paleocene  
47 in ages (Besse et al., 1984; Patzelt et al., 1996; Tong et al., 2008; Yi et al., 2011; Ma et al., 2016;  
48 Yang et al., 2019; Meng et al., 2020; Yuan et al., 2020). In the light of these poles, a variety of  
49 paleogeographic reconstructions were established with small (Besse et al., 1984; Tong et al., 2008),  
50 moderate (Yi et al., 2011), and enlarged (Meng et al., 2020) Greater India or hypothesized oceanic  
51 basins, namely, “Greater India Basin” (van Hinsbergen et al., 2012) or “North India Sea” (Yuan et  
52 al., 2020).

53 A continuously outcropped marine sedimentary sequence is well-preserved in the Gamba area.  
54 Among these units, the Zongshan (71-65 Ma) and Zongpu (62-56 Ma) formations (Willems and  
55 Zhang, 1993; Wan et al., 2002a, b) provide a unique opportunity for constraining the locus of the  
56 Tethyan Himalaya covering a critical stage of the India-Asia collision. Detailed lithological and  
57 biostratigraphic (Willems and Zhang, 1993; Wan et al., 2002a,b), sedimentological (Li et al., 2015),  
58 and geochemical (Wang et al., 2008; Q. Zhang et al., 2019) investigations provide a solid foundation  
59 for paleomagnetic studies.

60 Characteristic remanent magnetizations (ChRMs) reported from the Zongshan and Zongpu  
61 formations in the Gamba area passed positive fold and reversal tests, along with internally consistent  
62 magnetostratigraphy and biostratigraphic ages (71-56 Ma), permitting the original authors to interpret  
63 them as primary (Patzelt et al., 1996; Yi et al., 2011). However, based on the detailed rock magnetic  
64 and petrographic studies, along with a reanalysis of the fold test performed on the Zongpu Formation,  
65 Huang et al., (2017a,b) argued for a widespread remagnetization via orogenic fluids in the Gamba  
66 area, and thus the paleomagnetic poles obtained from Zongpu Formation limestones can no longer be  
67 used to constrain the geometry of the India-Asia collision. As a response, Yi et al. (2017) addressed  
68 the reliability of their fold tests performed on the Zongpu and Zongshan formations and  
69 acknowledged the new rock magnetic insight presented by Huang et al. (2017a), but argued for an  
70 acquisition of the ChRMs in the early diagenetic stage.

71 On the basis of rock magnetism and SEM observations incorporating EDS analysis, Huang et al.,  
72 (2017a,b, 2019) argued for the presence of abundant authigenic magnetites in carbonates preserved  
73 within the Tethyan domain of Tibet. These authigenic magnetites were suggested to result from a  
74 partial or complete replacement of pyrite crystals/framboids by secondary magnetites that were  
75 responsible for a widespread chemical remagnetization in the Gamba and Tring area, Tethyan  
76 Himalaya (Huang et al., 2017a,b). However, authigenic magnetic minerals are common for marine  
77 sediments due to the diagenesis during the burial process that may alter the combination of magnetic

78 components (Roberts, 2015 and references therein) and complicate the discrimination of rock  
79 magnetic parameters. The authigenic magnetic spherules cannot be directly related to a chemical  
80 remagnetization (Saffer and McCabe, 1992; Suk et al., 1992), although the ability of carrying stable  
81 remanence of these magnetic spherules remains elusive (Xu et al., 1994; Suk and Halgedahl, 1996).  
82 Moreover, as EDS analyses cannot distinguish magnetic particles among magnetite, hematite, and  
83 goethite due to the imprecise measurement of Fe/O ratios (Sun and Jackson, 1994; Xu et al., 1998;  
84 Weil and Van der Voo, 2002; Franke et al., 2007), the arguments by Huang et al., (2017a,b, 2019)  
85 needs to be further studied.

86 In an effort to clarify the type and origin of the magnetic carriers in the Zongpu carbonates, we  
87 carry out a combined study integrating optical microscopy, SEM/TEM observations, and Raman  
88 spectroscopy measurements on thin sections and magnetic extracts of pilot samples from the Zongpu  
89 Formation in the Gamab area, Tethyan Himalaya. By this way, we further evaluated the nature of  
90 ChRMs reported from the Zongpu Formation by previous studies.

## 91 2 Sampling sites and experimental methods

92 Figure 1 illustrates the structure of the Indus-Yarlung Zangbo suture zone, in which  
93 paleomagnetic sampling localities and lithostratigraphic units are indicated. Detailed geological  
94 background is available in many previous studies (e.g., Wan et al., 2002a,b; Yi et al., 2011; Li et al.,  
95 2015; Huang et al., 2017a). The Paleocene carbonate rocks of the Zongpu Formation were deposited  
96 in a shallow-marine carbonate ramp on the northern Indian passive margin (Li et al., 2015). The  
97 Zongpu Formation is divided into four members by lithology; massive limestone (Member I), marls  
98 (Member II), nodular limestones (Member III), and well-bedded limestones (Member IV) (Willems  
99 and Zhang, 1993). **Polished thin sections (xg38-3, xg160-1, xg145-3, and xg121-1) were processed  
100 on samples collected by Yi et al. (2011) (red dots in Figure 1C). In addition, block limestone  
101 samples (GB) of ~1 kilogram in weight was collected from the top of the Zongpu Formation for  
102 a magnetic extraction and SEM/TEM observations (Section A of Yi et al. (2011), Figure 1C).**

103 Raman spectra measurements were conducted using a Raman spectrometer (LabRAM HR  
104 Evolution) equipped with a laser (excitation wavelength of 532nm) in the School of Earth and Space  
105 Sciences (SESS), Peking University. Laser power was reduced by a filter to about 1 mW to avoid the  
106 transformation of magnetite, goethite, and pyrite (Hanesch, 2009). Data were obtained with a spectral  
107 resolution of  $1\text{cm}^{-1}$  across the  $100\text{-}1500\text{ cm}^{-1}$  wavenumber offset range. The experiment was carried  
108 out under an objective lens with 100 times magnification. Because of the low laser powers, more than  
109 ninety seconds integration time for individual measurements and 10 accumulations were set to  
110 improve the signal-to-noise ratio. In this study, Raman spectra were provided without smoothing or  
111 fitting to present the original results during the measurements.

112 To further examine the magnetic properties, the carbonate rock samples were first disaggregated  
113 and then put in buffered acetic acid to dissolve ( $\text{pH} = 4$ ) for several days. Magnetic extraction is  
114 performed using a self-designed magnetic probe extraction apparatus (Figure S1A). The slurry  
115 flowed through a tube with dispersed fine magnetic fractions and pumped continuously through the  
116 extraction equipment. Improved extraction-related procedures, following Hounslow et al. (1999),  
117 were used to avoid dissolution effects of ultrafine magnetic particles in samples (Sun and Jackson,  
118 1994).

119 Magnetic extracts of pilot samples were prepared for SEM observation as thin sections using  
120 resin as an adhesive (Figure S1B). An alternative and highly recommended procedure to prepare

121 SEM samples was to drop the solutions with magnetic extracts on a monocrystalline silicon wafer  
122 (Figure S1C). To prepare TEM specimens, distilled water with magnetic extracts was moved to a  
123 small container. A rare-earth magnet hovered ~1 cm above the TEM grid which was floated on the  
124 surface of the solutions, to attract magnetic extracts for ~5 min (Figure S1D). EDS, electron  
125 backscatter diffraction (EBSD), and photographs were performed with SEM/ESEM system at SESS  
126 and Electron Microscopy Laboratory (EML) in the School of Physics, Peking University. The TEM  
127 was performed using a JEOL 2100 TEM (200kV) at the Institute of Geology and Geophysics,  
128 Chinese Academy of Sciences (IGGCAS).

## 129 **3 Results**

### 130 **3.1 Optical petrography and Raman spectroscopy analysis**

131 An analysis of the iron oxide-sulfide assemblages in the thin sections (xg38-3, xg160-1, xg145-  
132 3, and xg121-1) and magnetic extracts (GB) under reflected white light shows that pyrite-substituted  
133 by iron oxides were the most abundant magnetic phase in the Zongpu Formation (Figures 2A-2D, 2I-  
134 2L, and S2). Based on the blood-red internal color under plane-polarized light (Figures 2E-2H, 2M-  
135 2P), we interpret the iron oxides with poor crystallinity as fine-grained pigmentary hematite. Goethite  
136 phases, displaying intense brownish yellow-orange internal reflections, are identified around hematite  
137 pseudoframboids (Figures 2D, 2J-2L). The iron sulfides, inferred as pyrite due to the bright-brassy  
138 colored reflections with a speckly appearance, yielded two morphologic groups: (1) framboid  
139 spherules (Figures 2A, 2B, and 2I) and (2) large euhedral grains (Figures 2C and 2D). The abundant  
140 occurrence of the pigmentary hematite and goethite along calcite boundaries and/or intergranular  
141 dissolved voids are noticeable. In contrast, the extensive presence of pyrite (framboids and euhedral  
142 grains) is well-preserved in calcite crystals as inclusions. Magnetite was not identified by optical  
143 microscope observation probably due to its low concentration, although it was supposed to be the  
144 main magnetic carrier in the limestones of the Zongpu Formation (Yi et al., 2011; Huang et al.,  
145 2017a).

146 In addition, the Raman spectrum investigations detected several characteristic peaks ( $222\text{ cm}^{-1}$ ,  
147  $297\text{ cm}^{-1}$ , and  $390\text{ cm}^{-1}$  in Figure 3A;  $208\text{ cm}^{-1}$ ,  $270\text{ cm}^{-1}$  and  $380\text{ cm}^{-1}$  in Figure 3B;  $344\text{ cm}^{-1}$  and  
148  $379\text{ cm}^{-1}$  in Figure 3C). Hematite, goethite and pyrite were identified by comparing the spectra of  
149 standard minerals in Hanesch (2009) and RRUFF database (<https://rruff.info>). For natural minerals,  
150 characteristic Ramana peaks are usually moderately offset due to differences in crystallinity and/or  
151 crystal defects (Hanesch, 2009). These results are consistent with the oxidation of pyrite framboids to  
152 iron (hydr)oxides observed under optical microscopic observations.

### 153 **3.2 SEM observations of magnetic extracts**

154 Abundant pure iron oxides were observed from magnetic extracts by SEM observation. These  
155 submicron iron oxide grains appear in various morphology and are composed of broken-octahedral,  
156 subangular, and irregular crystals (Figures 4A-4J), suggestive of a detrital origin. The acquired  
157 Electron Back-scattering Patterns (EBSPs) for these grains show a spinel pattern (Figures 4M-4P)  
158 that confirm a detrital origin for magnetites, although there may be hematite in some cases.  
159 Interestingly, we also found several euhedral magnetic crystals with clear particle boundaries, about  
160 50-100 nm in size (Figures 4K and 4L). Accordingly, we suggest that these submicron and nanoscale  
161 magnetite particles fit the size range of SD and PSD (Dunlop and Özdemir, 1997) and are the  
162 possible remanence carrier in the limestones of the Zongpu Formation in the Gamba area.



163 Despite the frequent occurrence of detrital magnetite, iron oxide spherules were also found in the  
164 magnetic extracts (Figure 4A). EDS line scanning and mapping show that the iron oxide assemblage  
165 contains S in addition to Fe and O in a form of pseudoframboid (Figure 5). Given that cosmic  
166 spherules usually contain a low content of Ni (Brownlow et al., 1966) which was not detected by the  
167 EDS analysis, we exclude the possibility of cosmogenesis. Along with our observations in thin  
168 sections, we argue that these pseudoframboids are iron (hydr)oxides (hematite and/or goethite) that  
169 substitute framboidal pyrite (Suk et al., 1990).

### 170 **3.3 TEM observations of magnetic extracts**

171 The TEM observations reveal that magnetic grains with variable grain sizes are commonly  
172 present in magnetic extracts from the Zongpu Formation (Figures 6A-6D). Further high-resolution  
173 TEM (HRTEM) and selected area electron diffraction (SAED) analyses were carried out to determine  
174 the crystal structure of the magnetic particles. All analyzed magnetic minerals, including submicron  
175 and nanosized particles, have clear lattice fringes (Figures 6E and 6F) and sharp diffraction patterns  
176 (Figures 6G-6I) which indicate that the analyzed magnetic minerals are titanomagnetite and  
177 magnetite with well-developed crystallinity.

178 The TEM images revealed presence of both nanosized and euhedral magnetic crystals for the  
179 studied samples (Figures 6C, 6I, and 6L). The grain size of magnetite and titanomagnetite ranges  
180 from tens of nm to several  $\mu\text{m}$ . Non-spheroidal iron oxides are observed in TEM. Together with the  
181 EDS spectra (Figures 6J-6L) and mineral morphologies, we believe that the remanence magnetic  
182 carrier should be detrital magnetite and/or euhedral magnetic particles from the Zongpu Formation in  
183 the Gamba area.

### 184 **3.4 Characteristics of demagnetization**

185 Previous rock magnetic investigations indicate the main magnetic carriers of remanence are  
186 magnetite from most Zongpu carbonates in the Gamba area, in addition, some of which detected  
187 goethite and hematite (Yi et al., 2011). All specimens were subjected to alternating field (AF)  
188 demagnetization up to 89 mT in the light of their relatively weak natural remnant magnetization  
189 (NRM). Two remnant magnetization components were isolated in the majority of specimens (Figure  
190 7A-F). After removal of a viscous component, xg0-3, xg38-3, and xg145-2 yield a stable  
191 characteristic remanence (ChRM) (Figure 7A, B, and D). Some specimens reveal an unstable  
192 demagnetization trajectory (xg121-2, and xg160-1; Figure 7C, E, and F) which were discarded for  
193 further discussion (Yi et al., 2011).

## 194 **4 Discussion**

### 195 **4.1 Origin of the nanoscale euhedral magnetite**

196 SD euhedral magnetites were observed in the magnetic extracts (Figures 4K, 4L, and 6C). There  
197 are two possible origins for such magnetic particles in sediments: (1) the magnetic inclusion as  
198 erosional detritus from igneous and metamorphic rocks (e.g., Chang et al., 2016); (2) biogenic  
199 magnetite (Kopp and Kirschvink, 2008). Both types of magnetic particles are able to carry stable  
200 paleomagnetic signals over billions of years (Kirschvink and Lowenstam, 1979; Tarduno et al., 2006;  
201 Tarduno et al., 2010). Usually, most of the magnetic nanoparticle inclusions hosted within silicate  
202 crystals show high content of Si and low content of Ti that can be identified by EDS analyses (Chang  
203 et al., 2016). In this study, however, only very low content of Si and no Ti were detected from the  
204 euhedral magnetic crystals (Figure 6L). Furthermore, silicate minerals (e.g., plagioclase and

205 clinopyroxene) were not observed in thin sections (Figures 2 and S4), probably due to the low clastic  
206 influx and high carbonate saturation during deposition of the Zongpu Formation (Li et al., 2015). In  
207 this case, the origin of nanoscale euhedral magnetite from silicate-hosted magnetic mineral inclusions  
208 is highly unlikely. We suggest the nanosized and euhedral magnetic particles are biogenic magnetite  
209 that is capable of carrying stable remanences in limestones (Chang et al., 1987). Further study should  
210 be required to detect robust evidence of biogenic magnetite based on a broader observation of  
211 magnetic extracts from Zongpu carbonates in the Gamba area.

## 212 4.2 The possible origin of iron oxide spherules

213 In addition to the detrital and biogenic magnetites observed in magnetic extracts, iron oxide  
214 spherules were also identified from the Zongpu Formation in the Gamba area (Figures 4, 5, and S2).  
215 Several previous studies attribute the remagnetization of carbonates to the replacement of framboidal  
216 pyrite by oxidation that is related to orogenic fluids (see review by McCabe and Elmore, 1989).  
217 However, the photomicrographs of limestone samples in Huang et al. (2017a) and section A of Yi et  
218 al. (2011) present well-preserved fossils (benthic foraminifer, echinoderm, ostracod, and green algae)  
219 with particles/matrix support and show no sign of orogenic-type fluids (Figure S4 and S5; Li and Hu,  
220 2020). Besides, the variations of carbon and oxygen isotope of bulk carbonate cover the key interval  
221 of the Paleocene-Eocene thermal maximum (PETM) (Q. Zhang et al., 2019). The strontium isotopic  
222 ratios ( $^{87}\text{Sr}/^{86}\text{Sr}$ ) of calcite are comparable with the global oceanic strontium isotope record (Figure  
223 S6; Wang et al., 2008) which indicates that the carbonates in the Gamba area have not been altered  
224 by orogenic fluids. The origin of iron oxide spherules should thus be explained in other mechanisms.

225 Suk et al. (1992) proposed different magnetic mineralogy for the primary and remagnetized  
226 carbonates. The iron sulfides (e.g., pyrite framboids) were moderately or completely oxidized to  
227 hematite in the former while a replacement of magnetite occurs in remagnetized carbonates.  
228 Moreover, oxidation of pyrite under modern atmosphere and groundwater conditions produces  
229 goethite and/or hematite (Todd et al., 2003; Sgavetti et al., 2009; Verron et al., 2019). Recently, a  
230 deep abiotic reaction mechanism of pyrite weathering in rocks was proposed which demonstrated that  
231 fracturing and erosion, in addition to atmospheric oxygen, control the reactivity of iron sulfide  
232 oxidation (Gu et al., 2020). Therefore, we suggest the large amounts of iron (hydr)oxides (e.g.,  
233 goethite and hematite) observed in carbonates from the Zongpu Formation in the Gamba area were  
234 more likely oxidized from pyrite under aqueous solutions in contact with the atmosphere.

## 235 4.3 Primary versus secondary origin of the ChRMs

236 The secondary superparamagnetic (SP) to stable single domain (SSD) grain-sized magnetite is a  
237 general indicator for chemical remagnetization in carbonates which could well explain the  
238 remagnetization that occurred in the Paleozoic carbonates of North America (Channell and McCabe,  
239 1994; Suk and Halgedahl, 1996; Xu et al., 1998; Elmore et al., 2006). A mix of SP and SD particles  
240 yields wasp-waisted hysteresis loops and distribution of Day plot along the SP-SD mixing line  
241 (Jackson and Swanson-Hysell, 2012). Nevertheless, magnetic minerals in different assemblage and  
242 shape anisotropy can also present contrasting coercivity distributions, resulting in wasp-waisted  
243 hysteresis loops (Jackson, 1990; Roberts et al., 1995; Newell and Merrill, 2000; Zwing et al., 2005;  
244 Jackson and Swanson-Hysell, 2012). It is generally difficult to interpret the magnetic grain size and  
245 mineralogy by wasp-waisted hysteresis loops or Day-plot only (Tauxe et al., 1996; Roberts et al.,  
246 2018). It also should be caution when using a Day diagram to diagnose remagnetization as  
247 occasionally 'false positives' and 'false negatives' results may present (Jackson and Swanson-Hysell,  
248 2012; Roberts et al., 2018). Moreover, the validity of application of Day-plot in shallow-water  
249 carbonates, which are isolated from aqueous detrital input, remains unverified (Jackson and

250 Swanson-Hysell, 2012). On the other hand, our SEM/TEM observations indicate the content of  
251 abundant detrital and biogenic magnetites in the investigated carbonates (Figures 4 and 6). The  
252 optical petrography and Raman spectra analyses present robust evidence that iron (hydr)oxides, i.e.,  
253 goethite and hematite (Figures 2, 3, and S2), rather than magnetite, as substitutes of pyrite framboids.  
254 The imaginable detrital and biogenic magnetite, along with goethite and hematite, would yield wasp-  
255 waisted hysteresis loops and distribution of Day plot along the SP-SD mixing line which leads to an  
256 incorrect interpretation of remagnetization from Day plot locations (Huang et al., 2017a,b, 2019).

257 The argument of previous paleomagnetic investigation for a chemical remagnetization of the  
258 carbonate rocks in the Gamba area was mainly based on SEM and EDS interpretation (Huang et al.,  
259 2017a). Whereafter, the same authors performed analogous analytical processes on the Upper  
260 Cretaceous to Paleocene carbonates from the Tingri area in the Tethyan Himalaya and the Upper  
261 Triassic limestones in the eastern Qiangtang block, argued for a widespread remagnetization in the  
262 Tibetan Tethyan domain (Huang et al., 2017b, 2019). However, the critical “authigenic magnetite”,  
263 along with the “orogenic fluids” are only speculated by the authors, regardless that conventional EDS  
264 techniques only have a semi-quantitative character which cannot directly distinguish the exact iron  
265 oxides. On the contrary, the geochemical evidence from the Zongpu Formation precludes the  
266 existence of widespread orogenic fluids as discussed above. Consequently, the remagnetization  
267 mechanism of chemical alteration suggested by Huang et al. (2017a) is problematic.

268 The acquisition of thermoviscous remanent magnetization (TVRM) may lead to widespread  
269 remagnetization (e.g., Kent, 1985). However, thermal demagnetization of limestones in the  
270 underlying Zongshan Formation reveals unblocking temperatures up to 500-550 °C (Patzelt et al.,  
271 1996). Moreover, the occurrence of anatase in the underlying Jidula Formation suggests that the  
272 overlying Zongpu limestones were never heated over 260°C (Patzelt et al., 1996). Middleton and  
273 Schmidt (1982) developed a relationship between relaxation and blocking temperature for magnetite  
274 with variable grain size, which is in better agreement with the laboratory-observed demagnetization  
275 temperature. Even assuming a TVRM acquisition for magnetite with grain size in a lognormal  
276 distribution, several billion years, at a heating temperature of ~300 °C, is still required to remove  
277 ChRMs locked at ~550 °C. Such temperatures are typical of low-grade regional metamorphism  
278 which have never been reported in a number of paleontological, stratigraphic and petrological studies  
279 (Willems and Zhang, 1993; Wan et al., 2002a, b; Li et al., 2015; Li and Hu et al., 2020).  
280 Furthermore, the folding of the Zongpu Formation initiated at ~56 Ma in the Gamba area (Zhang et  
281 al., 2012) when the overlying Zhepure Formation is mostly absent. As the fold tests have suggested a  
282 clear pre-folding origin of the Zongpu ChRMs, we argue that the burial depth of this unit is up to  
283 several hundred meters (i.e., no more than the thickness of the Zongpu Formation). Meanwhile, there  
284 was a close similarity of the reconstructed magnetostratigraphy from the Zongpu Formation (Yi et  
285 al., 2011) and the geomagnetic polarity time scale (GPTS, Gradstein et al., 2012) in the Paleocene,  
286 constraining on the biostratigraphic age (Willems and Zhang, 1993). In this case, the ChRMs in the  
287 Zongpu carbonates must be acquired during or shortly after the deposition (Yi et al., 2017). A  
288 thermal remagnetization via burial can therefore be ruled out at least for the Zongpu Formation.

289 In addition to thermoviscous remagnetization, the burial diagenetic alteration processes, i.e., clay  
290 diagenesis and maturation of organic matter, may also lead to a remagnetization in limestones  
291 (Elmore et al., 2012). The NRM intensity generally increases during the amount of alteration from  
292 smectite to illite by diagenesis (Katz et al., 2000). However, the magnetization of Zongpu carbonate  
293 rocks is as low as  $10^{-4}$ - $10^{-5}$  A/m (Figure 7; Yi et al., 2011) suggesting remagnetization via burial  
294 diagenetic is unlikely. Moreover, carbon isotope values of the Zongpu carbonate rocks prior to the  
295 carbon isotope excursion (CIE) during PETM is consistent with that of the planktonic foraminifera

296 from pelagic sections (Q. Zhang et al., 2019), indicative of limited diagenesis. It is important to  
297 consider the remanences in the Zongshan and Zongpu formations at Tingri area (~200 km west of  
298 Gamba) where no pre-folding magnetization was isolated so far (Besse et al., 1984; Liebke et al.,  
299 2013; Huang et al., 2015). Further paleomagnetic investigation is thus required to address the  
300 potential burial diagenetic remagnetization processes in the Tibetan Plateau.

301 The presence of abundant detrital and biogenic magnetites and the similar strontium isotopic  
302 ratios to coeval seawater in the Zongpu limestones precludes widespread chemical remagnetization in  
303 the Gamba area. The rock magnetic investigations (Yi et al., 2011; Huang et al., 2017a) and the  
304 characteristics of demagnetization (Figure 7) are consistent with high-resolution petrographic  
305 observations (Figures 2, 4, and 6). Moreover, the ChRMs from Gamba carbonates yielded positive  
306 fold and reversal tests (Patzelt et al., 1996; Yi et al., 2011, 2017), and the paleomagnetic pole from  
307 the Zongpu Formation hence meets all the criteria for a paleomagnetic study ( $R = 7$ ) (Meert et al.,  
308 2020). We therefore concluded that detrital and biogenic magnetites are the main magnetic carriers of  
309 primary remanence and the paleomagnetic results reported by Yi et al. (2011) from the Gamba area  
310 can still be used for paleogeographic reconstruction.

## 311 5 Conclusion and perspective

312 The high-resolution petrographic study was carried out on Paleocene carbonates (the Zongpu  
313 Formation) from Gamba, South Tibet. Electron microscopic observation of magnetic extracts  
314 identified abundant detrital and biogenic magnetites. Minor framboidal iron oxides were also  
315 identified using SEM, optical microscope, and Raman spectrum investigations. However, the  
316 magnetic minerals in these framboids are pigmentary hematite and/or goethite rather than authigenic  
317 magnetite. Therefore, the ChRMs of the limestones from the Zongpu Formation in the Gamba area  
318 are carried by detrital and biogenic magnetites. The arguments of chemical remagnetization, based on  
319 oversimplified semiquantitative EDS analyses and incomplete rock magnetic measurements in  
320 previous studies, should be rejected. Instead, the paleomagnetic data obtained from the Paleocene  
321 carbonates in the Gamba area can be used for tectonic reconstructions. We suggest that  
322 comprehensive analyses of magnetic extracts with advanced EBSD and TEM are extremely  
323 important and favorable to diagnose the substantial magnetization carriers in carbonate rocks. The  
324 remagnetization hypotheses in Paleocene carbonates from the Tingri area, Tethyan Himalaya, and the  
325 Late Triassic carbonates from the Qiangtang terrane require further study based on the thorough  
326 petrographic and mineralogical investigations to determine the origin of the magnetization.

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## 583 2 Figure captions

584 **Figure 1.** (A) Schematic structural map of the India-Asia collision zone. (B) Geologic map of the Gamba area  
585 with the paleomagnetic sampling locations of Yi et al. (2011) (section A-C) and Huang et al., (2017a)  
586 (green star). The sampling location of strontium isotopes study (Wang et al., 2008) was also marked. (C)  
587 Lithostratigraphy of section A of Yi et al. (2011). Red and blue dots indicate the sampling levels for samples  
588 collected for thin sections observation and magnetic extraction, respectively.

589 **Figure 2.** Photomicrographs illustrating the iron oxide mineralogical features of limestones from the Zongpu  
590 Formation in the Gamba area under reflected light (A-D, I-L) and plane-polarized light (E-H, M-P) images.  
591 White dots in Figures. 2C and 2L represent the spots of Raman spectroscopy analyses in Figure 3. Cal =  
592 calcite; Gt = goethite; pHem = pigmentary hematite; Py = pyrite.

593 **Figure 3.** Raman spectrum of the limestones from the Zongpu Formation. Three types of Fe-O-S minerals  
594 (hematite, goethite and pyrite) can be identified in the Raman spectra, respectively.

595 **Figure 4.** (A-L) Secondary-electron SEM images of magnetic extracts in limestones of the Zongpu Formation.  
596 (M and P) EBSPs solution of iron oxides corresponding to the white circles indicated in Figure 4 A-C. Zone  
597 axes are labeled using Miller indices. Note the white circle areas are not as accurate as that was shown in the  
598 images, because of the low resolution of SEM during EBSD analyses. White arrows in Figures 4K and 4L  
599 indicate the possible occurrence of biogenic magnetite. White dots represent the EDS spots as shown in Figure  
600 S3. [Fe-O] = iron oxides; Mag = magnetite.

601 **Figure 5.** Elemental mapping exhibits elemental compositions and distributions of an iron-oxidized framboid.  
602 (A) SEM image of a framboid with line scan by energy spectrum. (B-D) Fe, S, and O elements are scattered in  
603 most areas. White arrows show significant variation in the distributions of Fe, O, and S elements.

604 **Figure 6.** High-resolution TEM and SAED analyses of magnetic minerals for magnetic extracts from  
605 limestones in the Zongpu Formation. (A-D) Bright-field TEM images at progressively higher magnifications  
606 reveal characteristics of mixed magnetic particles with different sizes. (E-F) Clear lattice fringes for the  
607 magnetic minerals are observed. The lattice fringes in (F) correspond to  $\langle 311 \rangle$  plane of titanomagnetite. (G-I)  
608 Ring-like and spot-like SAED patterns indicate the particle aggregate and single particle, respectively. The  
609 corresponding Miller indices (hkl) are illustrated. (J-L) EDS spectra of the magnetic particles in B-D. The  
610 particles in different sizes are magnetite (B, C) and titanomagnetite (D).

611 **Figure 7.** Orthogonal (Zijderveld) vector plots of representative specimens from the Zongpu  
612 limestones in the Gamba area. All demagnetization data are from Yi et al. (2011). Polished thin  
613 sections were processed on the fresh end materials of the paleomagnetic specimens. Block limestone  
614 sample was collected for magnetic extraction from the top of the Section A in Yi et al. (2011).  
615 Directions are plotted in-situ; solid and open circles represent vector endpoints projected onto  
616 horizontal and vertical planes, respectively.

617

Figure 1.JPEG

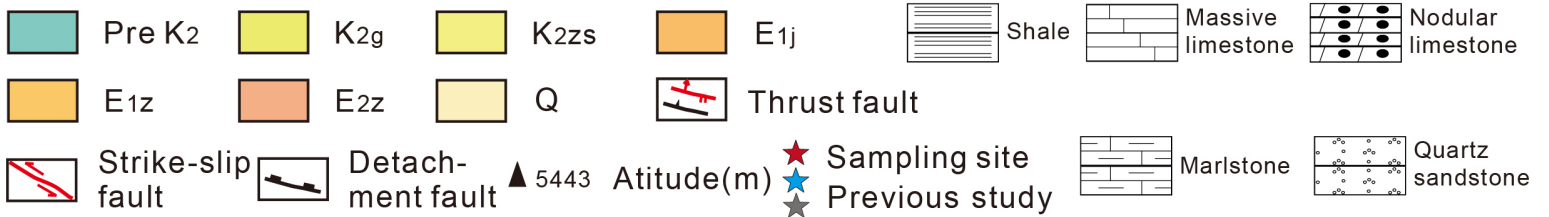
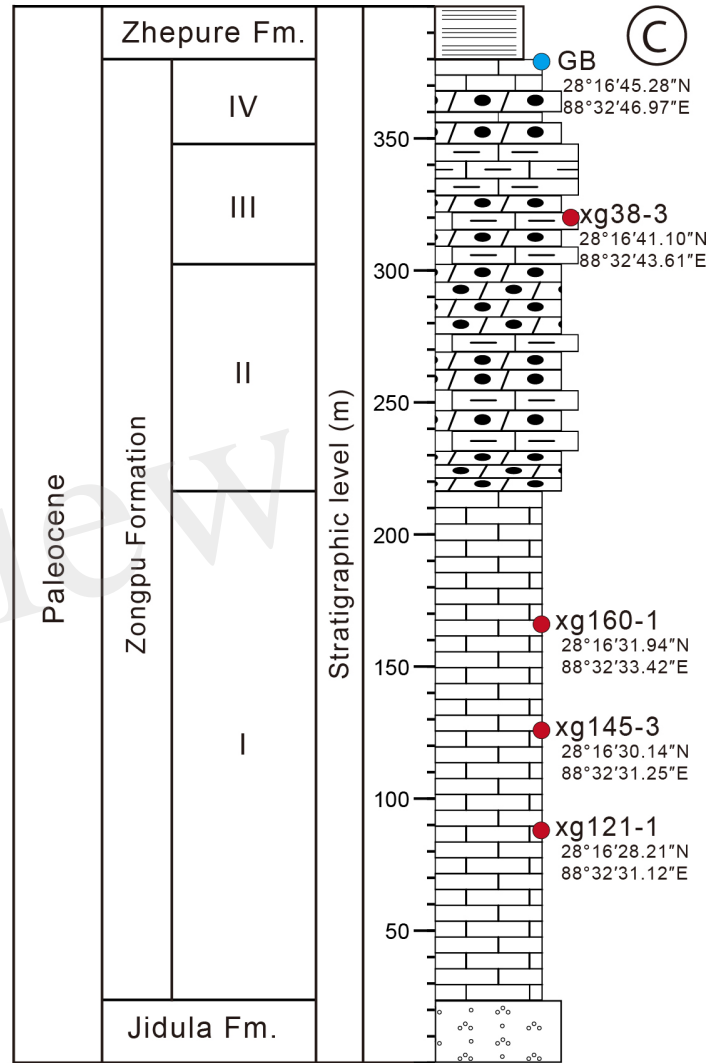
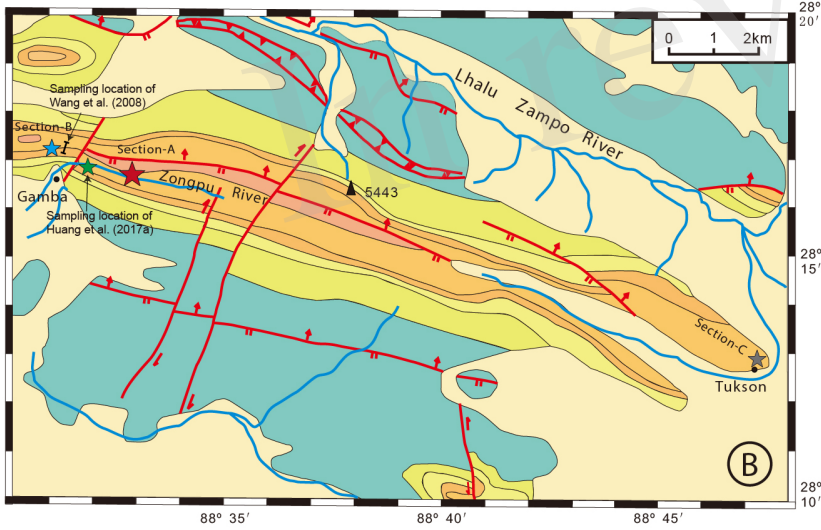
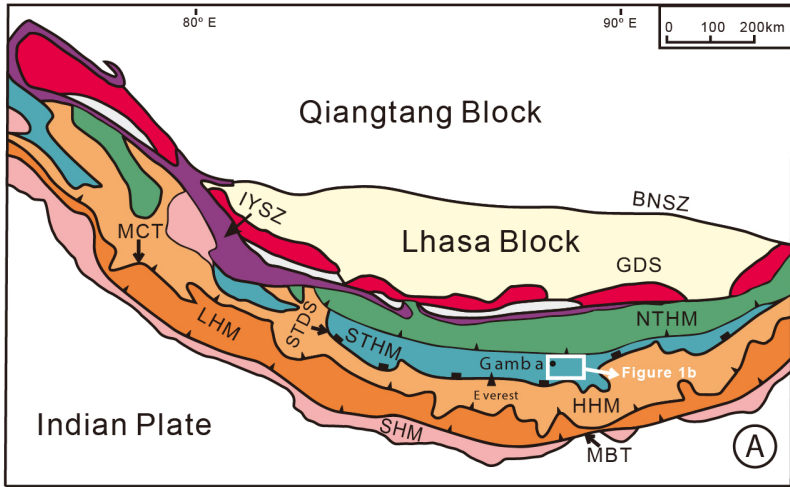




Figure 2.JPEG

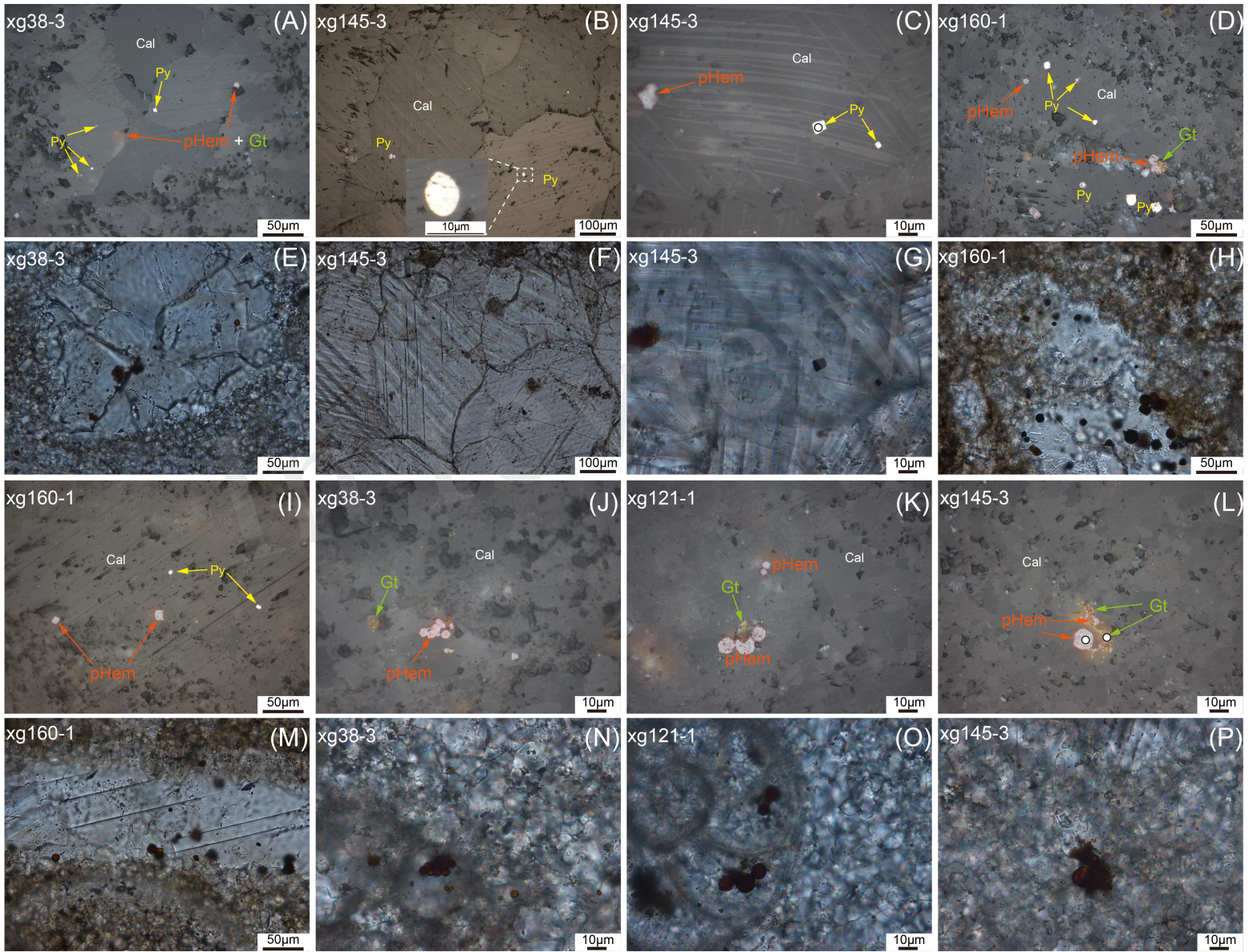


Figure 3.JPEG

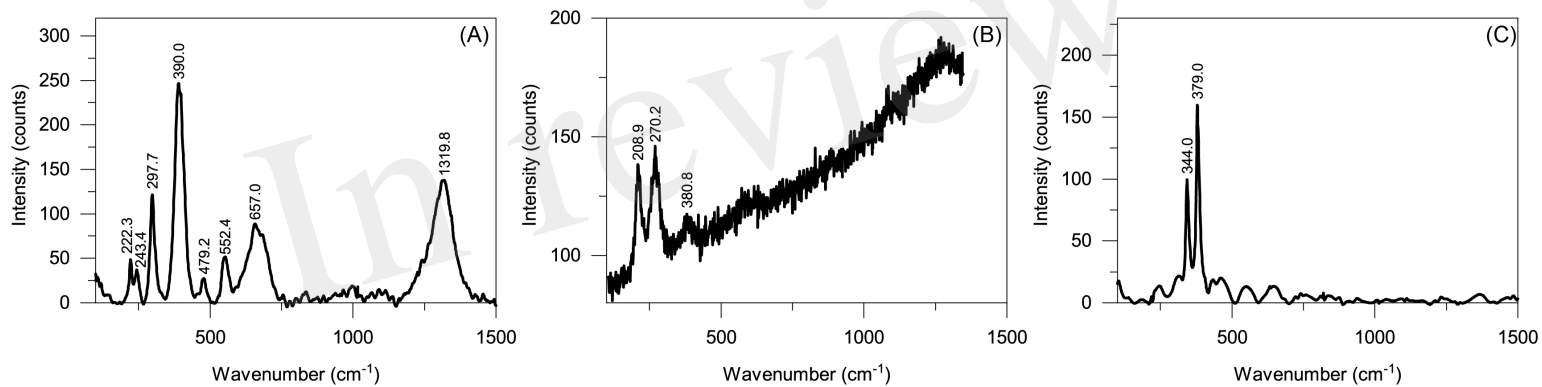




Figure 4.JPEG

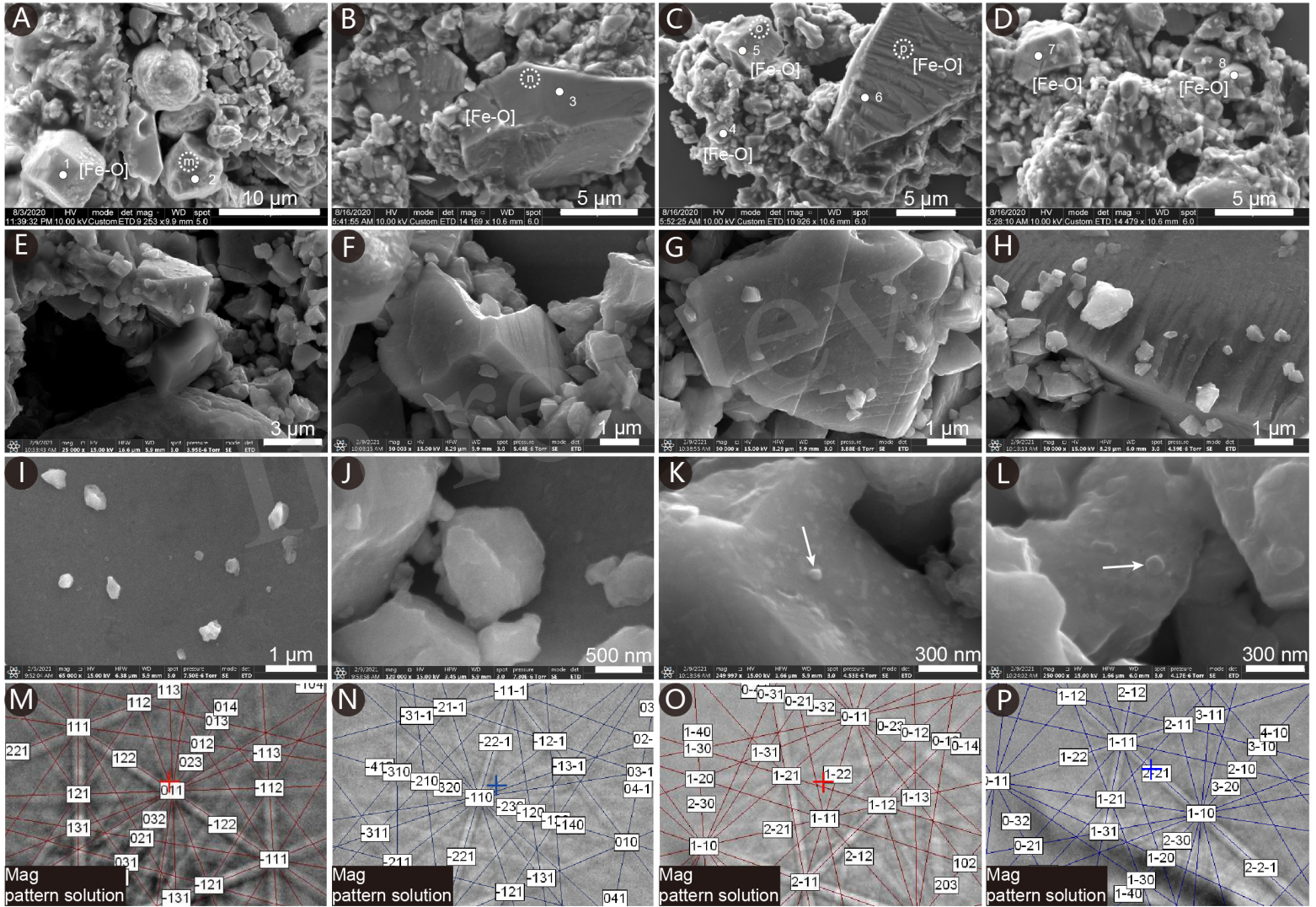




Figure 5.JPEG

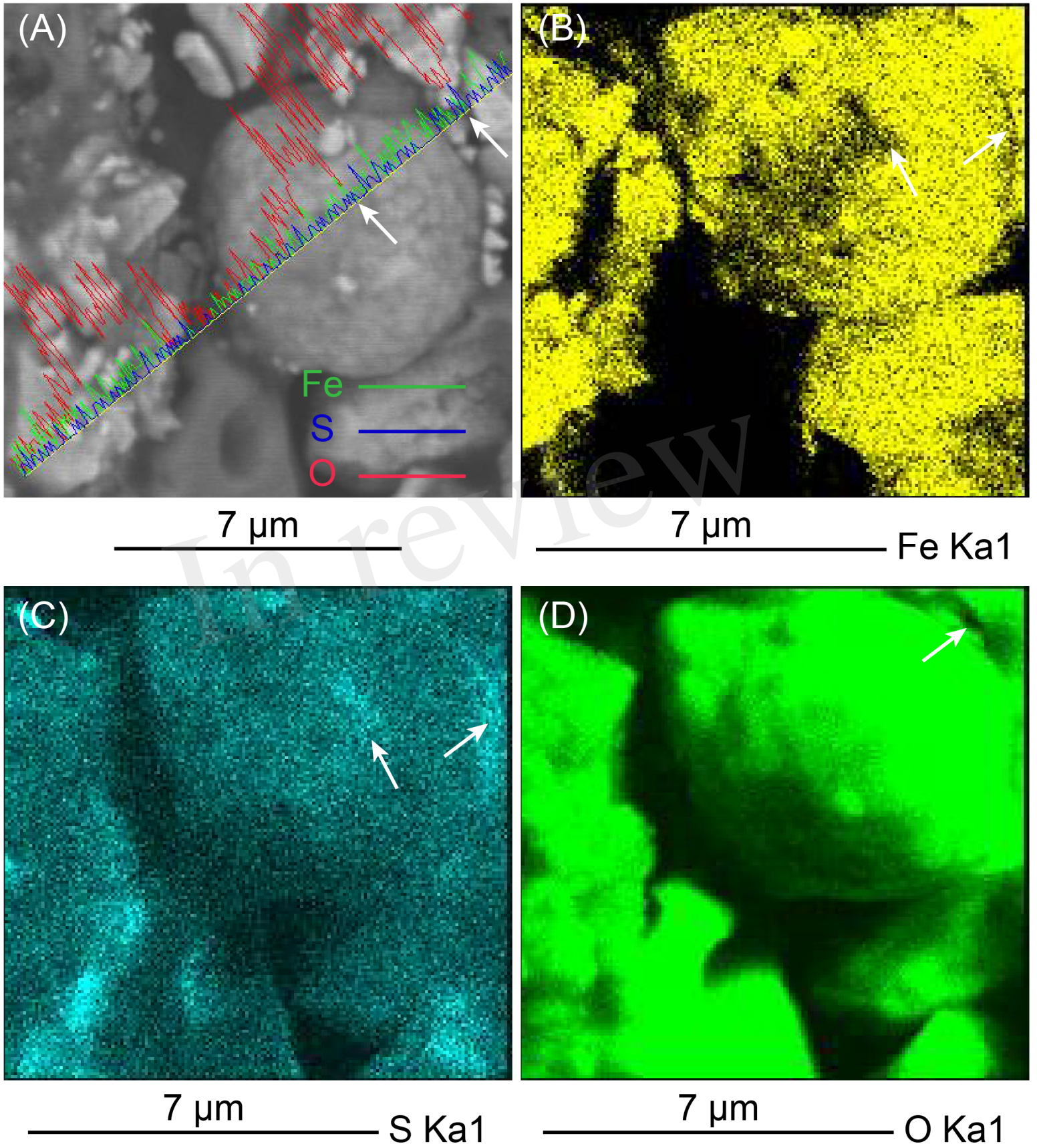


Figure 6.JPEG

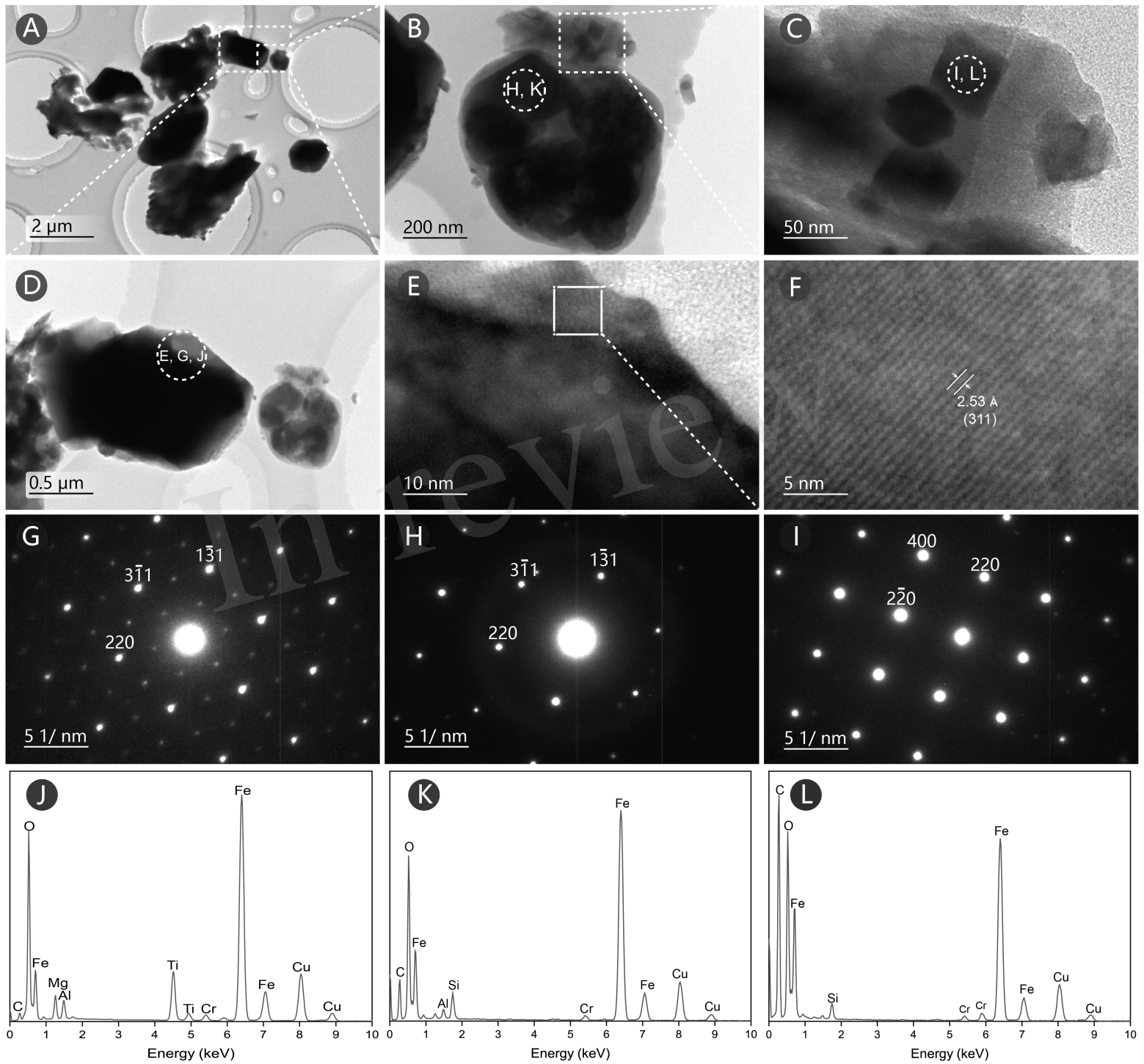
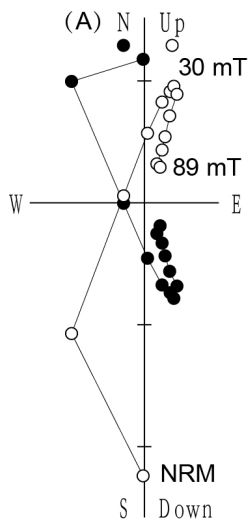
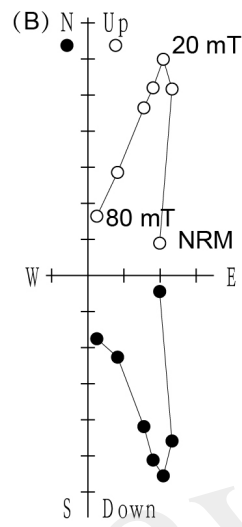


Figure 7.JPEG



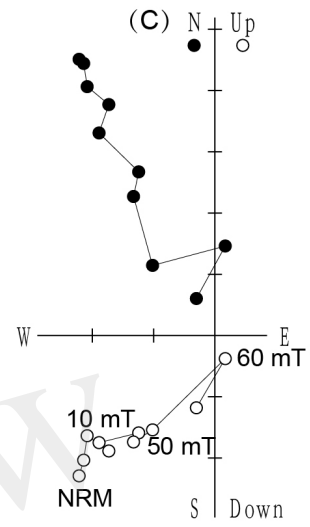
xg0-3-IS

Scale:  $1e-4$  A/m



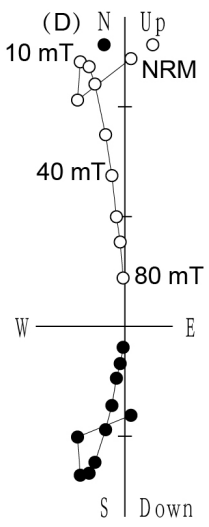
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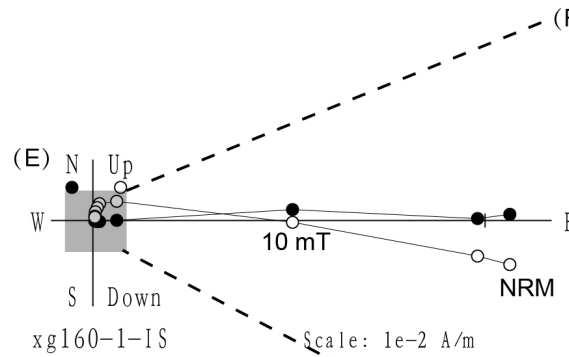
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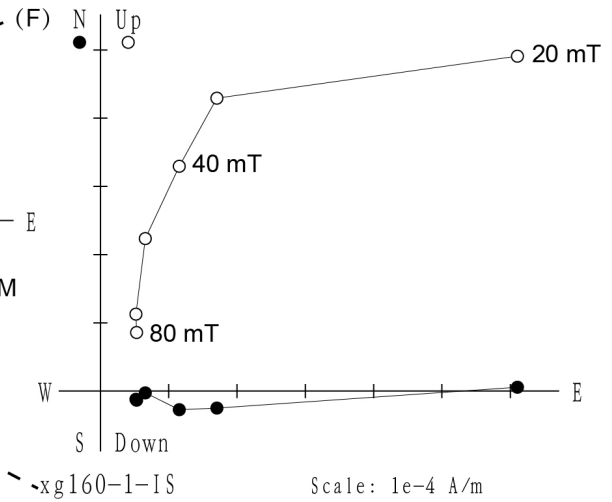
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xg160-1-IS

Scale:  $1e-2$  A/m



xg160-1-IS

Scale:  $1e-4$  A/m