

High-resolution petrographic evidence confirming detrital and biogenic magnetites as remanence carriers in Zongpu carbonates, South Tibet

1 Qian Zhao¹, Baochun Huang^{2*}, Zhiyu Yi², Pengfei¹

2 ¹ Key Laboratory of Orogenic Belt and Crustal Evolution, Ministry of Education, School of Earth
3 and Space Sciences, Peking University, Beijing, China

4 ² Planetary Environmental and Astrobiological Research Laboratory (PEARL), School of
5 Atmospheric Sciences, Sun Yat-sen University, Zhuhai, China

6 * Correspondence:

7 Baochun Huang

8 bchuang@pku.edu.cn

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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. The
13 characteristic remanences (ChRMs) obtained from this unit were, however, argued for a chemical
14 remagnetization via orogenic fluids. This study carries out a high-resolution petrographic study on
15 the 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 biogenic magnetite in nanoscale. 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 pigment hematite and goethite that are incapable of carrying a stable remanence. We therefore
22 argue that the ChRMs of the limestones from the Zongpu Formation in the Gamba area are carried by
23 detrital and biogenic magnetites rather than authigenic magnetite. The paleomagnetic data from the
24 Gamba area are interpreted as primary origin and can thus be used for tectonic reconstructions. We
25 emphasize that magnetic extraction, integrated with advanced mineralogic studies (e.g., electron
26 backscatter diffraction and electron diffraction) are effective approaches for investigating the origin
27 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 provide a unique
55 opportunity for constraining the locus of the Tethyan Himalaya covering a critical stage of the India-
56 Asia collision. Detailed lithological and biostratigraphic (Willems and Zhang, 1993; Wan et al.,
57 2002a,b), sedimentological (Li et al., 2015), and geochemical (Wang et al., 2008; Q. Zhang et al.,
58 2019) investigations provide a solid foundation for paleomagnetic studies.

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

69 On the basis of rock magnetism and SEM observations incorporating EDS analysis, Huang et al.,
70 (2017a,b, 2019) argued for the presence of abundant authigenic magnetites in carbonates preserved
71 within the Tethyan domain of Tibet. These authigenic magnetites were suggested to result from a
72 partial or complete replacement of pyrite crystals/framboids by secondary magnetites that were
73 responsible for a widespread chemical remagnetization in the Gamba and Tring area, Tethyan
74 Himalaya (Huang et al., 2017a,b). However, authigenic magnetic minerals are common for marine
75 sediments due to the diagenesis during the burial process that may alter the combination of magnetic
76 components (Roberts, 2015 and references therein) and complicate the discrimination of rock
77 magnetic parameters. The authigenic magnetic spherules cannot be directly related to a chemical
78 remagnetization (Saffer and McCabe, 1992; Suk et al., 1992), although the ability of carrying stable
79 remanence of these magnetic spherules remains elusive (Xu et al., 1994; Suk and Halgedahl, 1996).

80 Moreover, as EDS analyses cannot distinguish magnetic particles among magnetite, hematite, and
81 goethite due to the imprecise measurement of Fe/O ratios (Sun and Jackson, 1994; Xu et al., 1998;
82 Weil and Van der Voo, 2002; Franke et al., 2007), the arguments by Huang et al., (2017a,b, 2019)
83 needs to be further studied.

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

89 **2 Sampling sites and experimental methods**

90 Figure 1 illustrates the structure of the Indus-Yarlung Zangbo suture zone, in which
91 paleomagnetic sampling localities and lithostratigraphic units are indicated. Detailed geological
92 background is available in many previous studies (e.g., Wan et al., 2002a,b; Yi et al., 2011; Li et al.,
93 2015; Huang et al., 2017a). The Paleocene carbonate rocks of the Zongpu Formation were deposited
94 in a shallow-marine carbonate ramp on the northern Indian passive margin (Li et al., 2015). The
95 Zongpu Formation is divided into four members by lithology; massive limestone (Member I), marls
96 (Member II), nodular limestones (Member III), and well-bedded limestones (Member IV) (Willems
97 and Zhang, 1993). Polished thin sections were processed on samples collected by Yi et al. (2011). In
98 addition, block limestone samples of ~1 kilogram in weight was collected from the top of the Zongpu
99 Formation for a magnetic extraction and SEM/TEM observations (GPS: 28°16'45.28"N,
100 88°32'46.97" E, Section A of Yi et al. (2011), Figure 1C).

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

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

116 Magnetic extracts of pilot samples were prepared for SEM observation as thin sections using
117 resin as an adhesive (Figure S1B). An alternative and highly recommended procedure to prepare
118 SEM samples was to drop the solutions with magnetic extracts on a monocrystalline silicon wafer
119 (Figure S1C). To prepare TEM specimens, distilled water with magnetic extracts was moved to a
120 small container. A rare-earth magnet hovered ~1 cm above the TEM grid which was floated on the
121 surface of the solutions, to attract magnetic extracts for ~5 min (Figure S1D). EDS, electron
122 backscatter diffraction (EBSD), and photographs were performed with SEM/ESEM system at SESS

123 and Electron Microscopy Laboratory (EML) in the School of Physics, Peking University. The TEM
124 was performed using a JEOL 2100 TEM (200kV) at the Institute of Geology and Geophysics,
125 Chinese Academy of Sciences (IGGCAS).

126 **3 Results**

127 **3.1 Optical petrography and Raman spectroscopy analysis**

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

142 Furthermore, the Raman spectrum investigations indicate the presence of hematite, goethite, and
143 pyrite (Figure 3). These results are consistent with our observations under the optical microscope. As
144 compared with the corresponding spectra of minerals shown in Hanesch (2009) and the RRUFF
145 database (<https://rruff.info>), the offset peaks might well be caused by different crystallinities of the
146 natural minerals.

147 **3.2 SEM observations of magnetic extracts**

148 Abundant pure iron oxides were observed from magnetic extracts by SEM observation. These
149 submicron iron oxide grains are presented in various morphology, consisting of broken-octahedral,
150 subangular, irregular, and well-rounded crystals (Figures 4A-4J), suggestive of a detrital origin. The
151 acquired Electron Back-scattering Patterns (EBSPs) for these grains show a spinel pattern (Figures
152 4M-4P) that confirm a detrital origin for magnetites, although there may be hematite in some cases.
153 Interestingly, we also found several euhedral magnetic crystals with clear particle boundaries, about
154 50-100 nm in size (Figures 4K and 4L). Accordingly, we suggest that these submicron and nanoscale
155 magnetite particles fit the size range of SD and PSD (Dunlop and Özdemir, 1997) and are the
156 possible remanence carrier in the limestones of the Zongpu Formation in the Gamba area.

157 Despite the frequent occurrence of detrital magnetite, iron oxide spherules were also founded in
158 the magnetic extracts (Figure 4A). EDS line scanning and mapping show that the iron oxide
159 assemblage contains S in addition to Fe and O in a form of pseudoframboid (Figure 5). Given that
160 cosmic spherules usually contain a low content of Ni (Brownlow et al., 1966) which was not detected
161 by the EDS analysis, we exclude the possibility of cosmogenesis. Along with our observations in thin
162 sections, we argue that these pseudoframboids are iron (hydr)oxides (hematite and/or goethite) that
163 are replaced by framboidal pyrite (Suk et al., 1990) or perhaps framboidal greigite (Roberts et al.,
164 2011).

165 3.3 TEM observations of magnetic extracts

166 The TEM observations reveal that magnetic grains with variable grain sizes are commonly
167 presented in magnetic extracts from the Zongpu Formation (Figures 6A-6D). Further high-resolution
168 TEM (HRTEM) indicates that the observed *d*-spacing values (Figure 6F), as well as diffraction
169 patterns (Figure 6G) for the magnetic particles, match well with the crystal structure of
170 titanomagnetite. All analyzed magnetic minerals, including submicron and nanosized particles, have
171 clear lattice fringes (Figures 6E and 6F) and sharp diffraction patterns (Figures 6G-6I) which indicate
172 good crystallinity.

173 Nanosized and euhedral magnetic crystals were also observed under TEM imaging for the
174 studied samples (Figures 6C, 6I, and 6L). The grain size of magnetite and titanomagnetite ranges
175 from tens of nm to several μm , which is consistent with the SD to MD size of magnetite (Dunlop and
176 Özdemir, 1997). Non-spheroidal iron oxides are observed in TEM. Together with the EDS spectra
177 (Figures 6J-6L) and mineral morphologies, we believe that the remanence magnetic carrier should be
178 detrital magnetite and/or euhedral magnetic particles from the Zongpu Formation in the Gamba area.

179 4 Discussion

180 4.1 Origin of the euhedral magnetite in nanoscale

181 SD euhedral magnetites were observed in the magnetic extracts (Figures 4K, 4L, and 6C). There
182 are two possible origins for such magnetic particles in sediments: (1) the magnetic inclusion as
183 erosional detritus from igneous and metamorphic rocks (e.g., Chang et al., 2016) transported by
184 rivers and ocean currents; (2) biogenic magnetite (Kopp and Kirschvink, 2008). Both types of
185 magnetic particles are able to carry stable paleomagnetic signals over billions of years (Kirschvink
186 and Lowenstam, 1979; Tarduno et al., 2006; Tarduno et al., 2010). Usually, most of the magnetic
187 nanoparticle inclusions hosted within silicate crystals show high content of Si and low content of Ti
188 that can be identified by EDS analyses (Chang et al., 2016). In this study, however, only very low
189 content of Si and no Ti were detected from the euhedral magnetic crystals (Figure 6L). Furthermore,
190 silicate minerals (e.g., plagioclase and clinopyroxene) were not observed in thin sections (Figures 2
191 and S4), probably due to the low clastic influx and high carbonate saturation during deposition of the
192 Zongpu Formation (Li et al., 2015). In this case, the origin of euhedral magnetite in nanoscale from
193 silicate-hosted magnetic mineral inclusions is highly unlikely. We suggest the nanosized and
194 euhedral magnetic particles are biogenic magnetite that is capable of carrying stable remanences in
195 limestones (Chang et al., 1987).

196 4.2 The possible origin of iron oxide spherules

197 In addition to the detrital and biogenic magnetites observed in magnetic extracts, iron oxide
198 spherules were also identified from the Zongpu Formation in the Gamba area (Figures 4, 5, and S2).
199 Several previous studies attribute the remagnetization of carbonates to the replacement of framboidal
200 pyrite by oxidation that is related to orogenic fluids (see review by McCabe and Elmore, 1989).
201 However, the photomicrographs of limestone samples in Huang et al. (2017a) present well-preserved
202 fossils (benthic foraminifer, echinoderm, ostracod, and green algae) with particles/matrix support and
203 show no sign of orogenic-type fluids (Figure S4, Li and Hu, 2020). Besides, the variations of carbon
204 and oxygen isotope of bulk carbonate cover the key interval of the Paleocene-Eocene thermal
205 maximum (PETM) (Q. Zhang et al., 2019). The strontium isotopic ratios ($^{87}\text{Sr}/^{86}\text{Sr}$) of calcite are
206 comparable with the global oceanic strontium isotope record (Wang et al., 2008) which indicates that

207 the carbonates in the Gamba area have not been altered by orogenic fluids. The origin of iron oxide
208 spherules should thus be explained in other mechanisms.

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

219 **4.3 Primary versus secondary origin of the ChRMs**

220 The new-formed Superparamagnetic (SP) to stable single domain (SSD) grain-sized magnetite is
221 a general indicator for chemical remagnetization in carbonates which could well explain the
222 remagnetization that occurred in the Paleozoic carbonates of North America (Channell and McCabe,
223 1994; Suk and Halgedahl, 1996; Xu et al., 1998; Elmore et al., 2006). A mix of SP and SD particles
224 yields wasp-waisted hysteresis loops and distribution of Day plot along the SP-SD mixing line
225 (Jackson and Swanson-Hysell, 2012). Nevertheless, magnetic minerals in different assemblage and
226 shape anisotropy can also present contrasting coercivity distributions, resulting in wasp-waisted
227 hysteresis loops (Jackson, 1990; Roberts et al., 1995; Newell and Merrill, 2000; Zwing et al., 2005).
228 It is generally difficult to interpret the magnetic grain size and mineralogy by wasp-waisted
229 hysteresis loops or Day-plot only (Tauxe et al., 1996; Roberts et al., 2018). It also should be cautious
230 when using a Day diagram to diagnose remagnetization as occasionally ‘*false positives*’ and ‘*false*
231 *negatives*’ results may present (Jackson and Swanson-Hysell, 2012; Roberts et al., 2018). Moreover,
232 the validity of application of Day-plot in shallow-water carbonates, which are isolated from aqueous
233 detrital input, remains unverified (Jackson and Swanson-Hysell, 2012). On the other hand, our
234 SEM/TEM observations indicate the content of abundant detrital and biogenic magnetites in the
235 investigated carbonates (Figures 4 and 6). The optical petrography and Raman spectra analyses
236 present robust evidence that iron (hydr)oxides, i.e., goethite and hematite (Figures 2, 3, and S2),
237 rather than magnetite, as substitutes of pyrite framboids. The imaginable detrital/biogenic magnetite,
238 along with goethite and hematite, would yield wasp-waisted hysteresis loops and distribution of Day
239 plot along the SP-SD mixing line which leads to an incorrect interpretation of remagnetization from
240 Day plot locations (Huang et al., 2017a,b, 2019).

241 The argument of previous paleomagnetic investigation for a chemical remagnetization of the
242 carbonate rocks in the Gamba area was mainly based on SEM and EDS interpretation (Huang et al.,
243 2017a). Whereafter, the same authors performed analogous analytical processes on the Upper
244 Cretaceous to Paleocene carbonates from the Tingri area in the Tethyan Himalaya and the Upper
245 Triassic limestones in the eastern Qiangtang block, argued for a widespread remagnetization in the
246 Tibetan Tethyan domain (Huang et al., 2017b, 2019). However, the critical “authigenic magnetite”,
247 along with the “orogenic fluids” are only speculated by the authors, regardless that conventional EDS
248 techniques only have a semi-quantitative character which cannot directly distinguish the exact iron
249 oxides. On the contrary, the geochemical evidence from the Zongpu Formation precludes the
250 existence of widespread orogenic fluids as discussed above. Consequently, the remagnetization
251 mechanism of chemical alteration suggested by Huang et al. (2017a) is problematic.

252 The presence of abundant detrital and biogenic magnetites in the Zongpu limestones precludes
253 widespread chemical remagnetization in the Gamba area. On the other hand, the occurrence of anatase
254 in the underlying Jidula Formation suggests that the overlying Zongpu limestones were never heated
255 over 260°C (Patzelt et al., 1996) and hence exclude a thermal remagnetization in the Gamba area.
256 Moreover, the ChRMs from Gamba carbonates yielded positive fold and reversal tests (Patzelt et al.,
257 1996; Yi et al., 2011, 2017), and the paleomagnetic pole from the Zongpu Formation hence meets all
258 the criteria for a paleomagnetic study ($R = 7$) (Meert et al., 2020). We therefore concluded that
259 detrital and biogenic magnetites are the main magnetic carriers of primary remanence and the
260 paleomagnetic results reported by Yi et al. (2011) from the Gamba area can still be used for
261 paleogeographic reconstruction.

262 **5 Conclusion and perspective**

263 The high-resolution petrographic study was carried out on Paleocene carbonates (the Zongpu
264 Formation) from Gamba, South Tibet. Electron microscopic observation of magnetic extracts
265 identified abundant detrital and biogenic magnetites. Minor framboidal iron oxides were also
266 identified using SEM, optical microscope, and Raman spectrum investigations. However, the
267 magnetic minerals in these framboids are pigment hematite and/or goethite rather than authigenic
268 magnetite. Therefore, the ChRMs of the limestones from the Zongpu Formation in the Gamba area
269 are carried by detrital and biogenic magnetites. The arguments of chemical remagnetization, based on
270 oversimplified semiquantitative EDS analyses and incomplete rock magnetic measurements in
271 previous studies, should be rejected. Instead, the paleomagnetic data obtained from the Paleocene
272 carbonates in the Gamba area can be used for tectonic reconstructions. We suggest that
273 comprehensive analyses of magnetic extracts with advanced EBSD and TEM are extremely
274 important and favorable to diagnose the substantial magnetization carriers in carbonate rocks. The
275 remagnetization hypotheses in Paleocene carbonates from the Tingri area, Tethyan Himalaya, and the
276 Late Triassic carbonates from the Qiangtang terrane require further study based on the thorough
277 petrographic and mineralogical investigations to determine the origin of the magnetization.

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512 2 Figure captions

513 **Figure 1.** (A) Schematic structural map of the India-Asia collision zone. (B) Geologic map of the Gamba area
 514 with the sampling locations of Yi et al. (2011) (red star) and Huang et al., (2017a) (green star). (C)
 515 Lithostratigraphy of section A of Yi et al. (2011). Red and blue dots indicate the sampling levels for samples
 516 collected for thin sections observation and magnetic extraction, respectively.

517 **Figure 2.** Photomicrographs illustrating the iron oxide mineralogical features of limestones from the Zongpu
 518 Formation in the Gamba area under reflected light (A-D, I-L) and plane-polarized light (E-H, M-P) images.
 519 Cal = calcite; Gt = goethite; pHem = pigment hematite; Py = pyrite.

520 **Figure 3.** Raman spectrum of the limestones from the Zongpu Formation. Three types of Fe-O-S minerals,
521 i.e., hematite (222 cm^{-1} , 297 cm^{-1} , and 390 cm^{-1}) (A), goethite (208 cm^{-1} , 270 cm^{-1} and 380 cm^{-1}) (B) and pyrite
522 (344 cm^{-1} and 379 cm^{-1}) (C) can be identified in the Raman spectra.

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

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

532 **Figure 6.** High-resolution TEM and SAED analyses of magnetic minerals for magnetic extracts from
533 limestone in the Zongpu Formation. (A-D) Bright-field TEM images at progressively higher magnifications
534 reveal characteristics of mixed magnetic particles with different sizes. (E-F) Clear lattice fringes for the
535 magnetic minerals are observed. (G-I) Ring-like and spot-like SAED patterns and (J-L) EDS spectra of the
536 magnetic particles in B-D. The particles in different sizes are magnetite (B, C) and titanomagnetite (D).

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Figure 6.JPEG

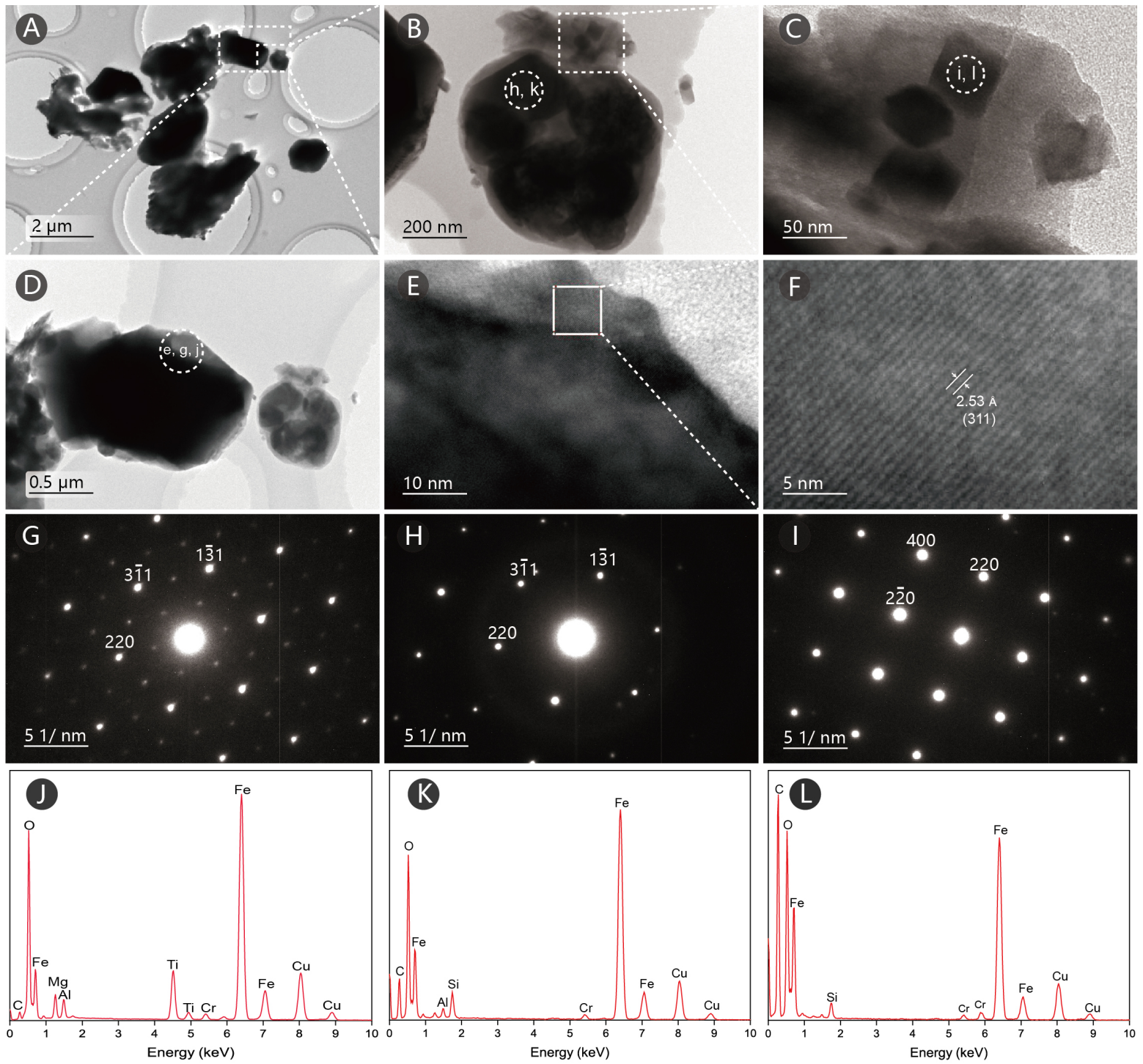


Figure 5.JPEG

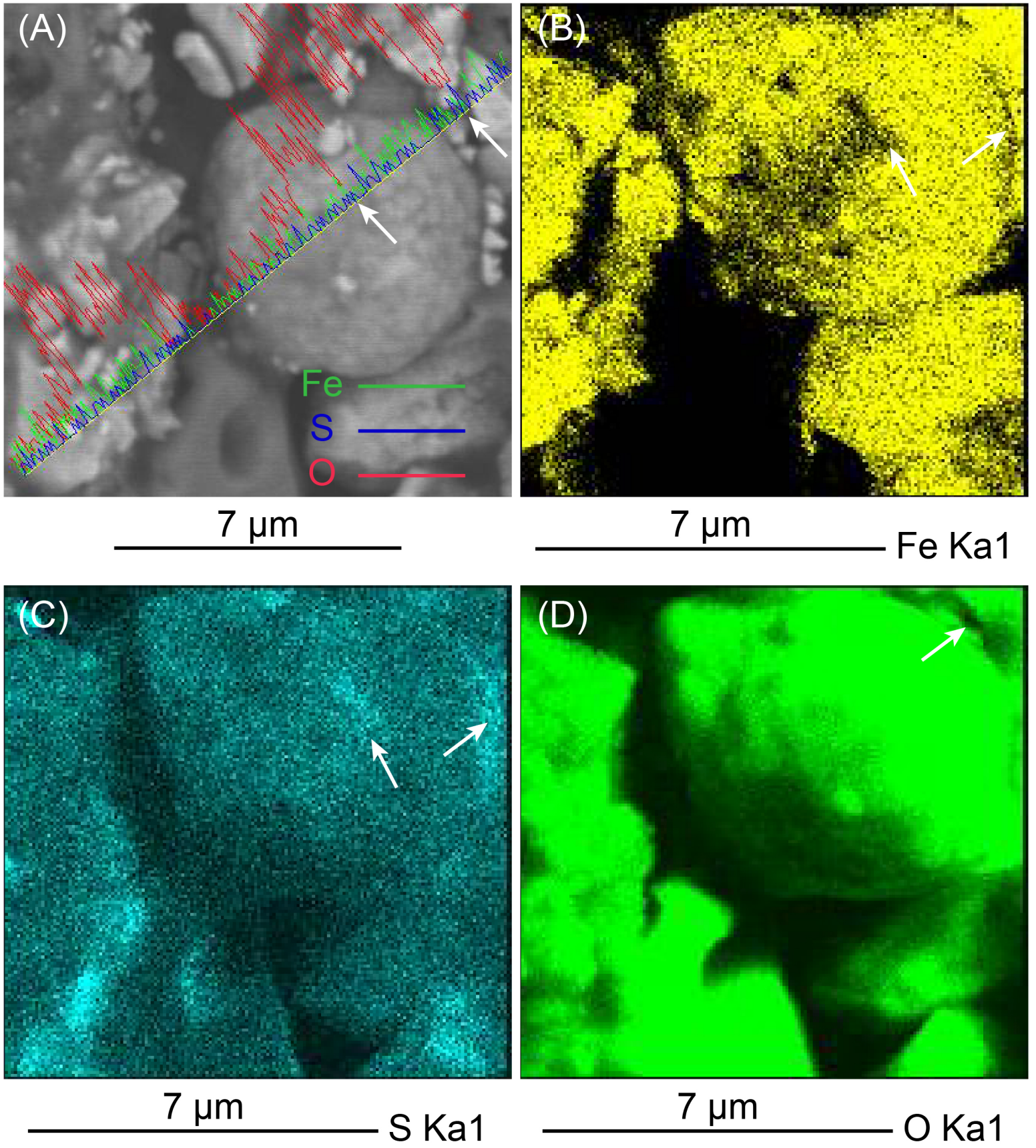


Figure 4.JPEG

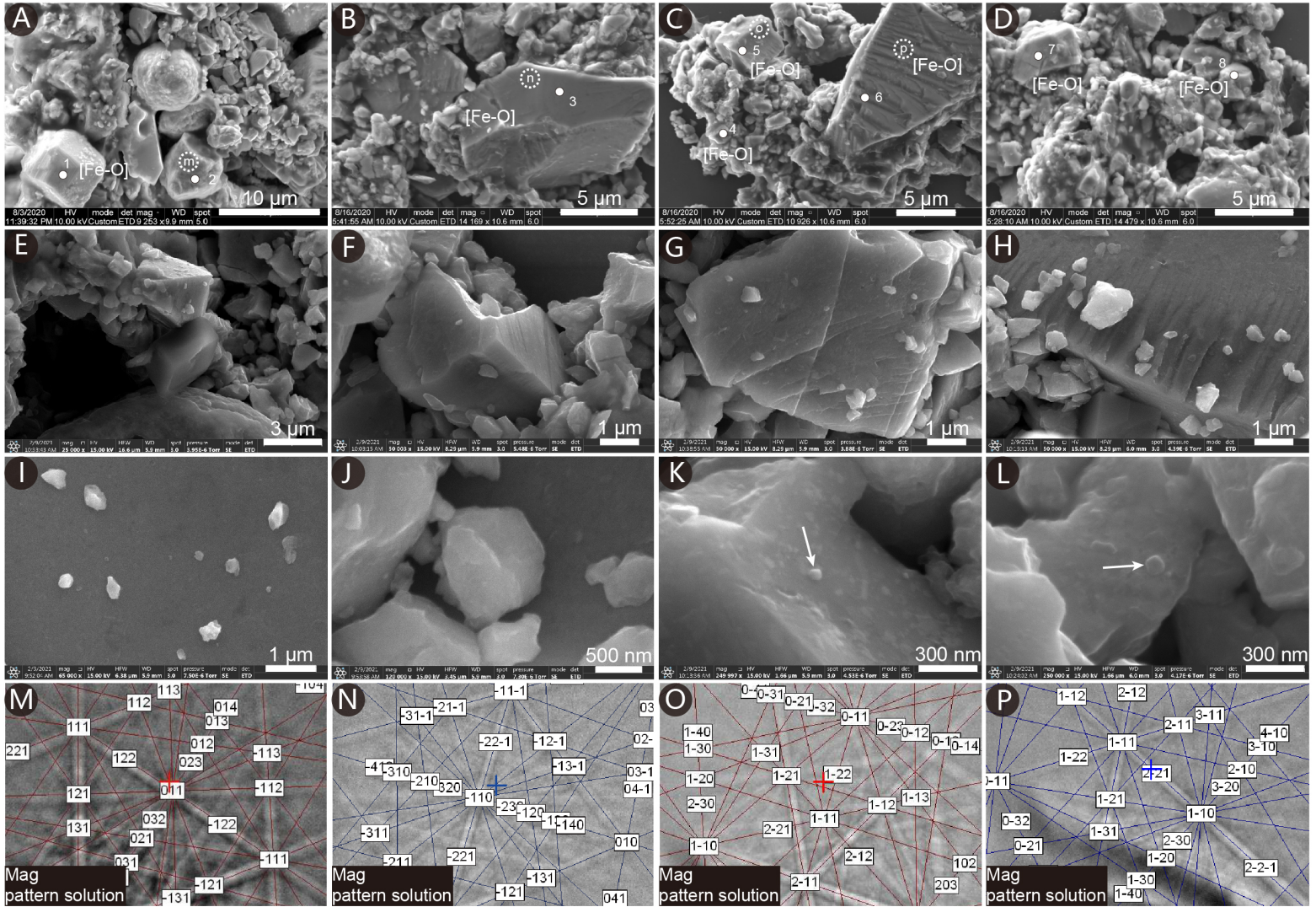


Figure 3.JPEG

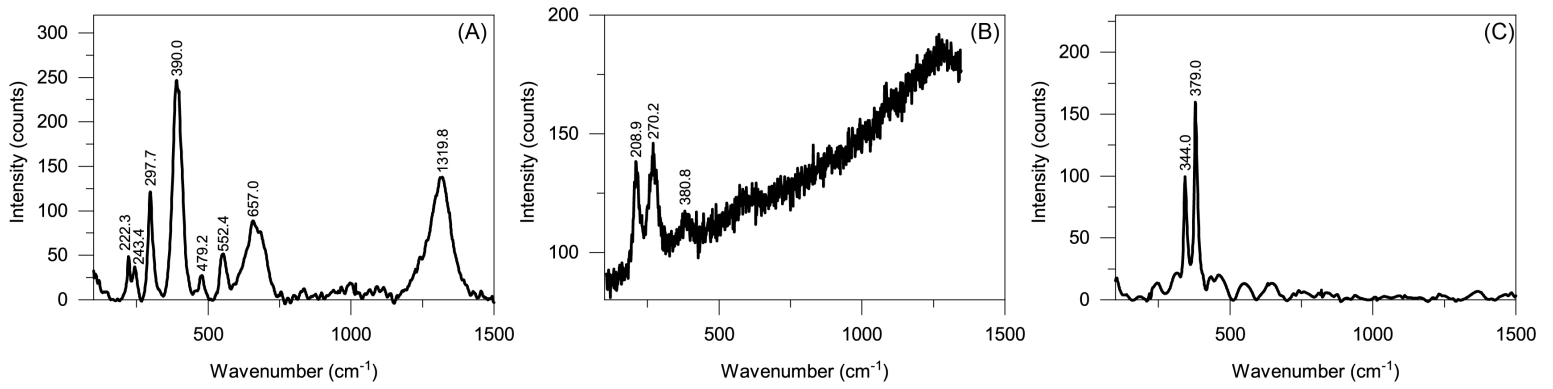


Figure 2.JPEG

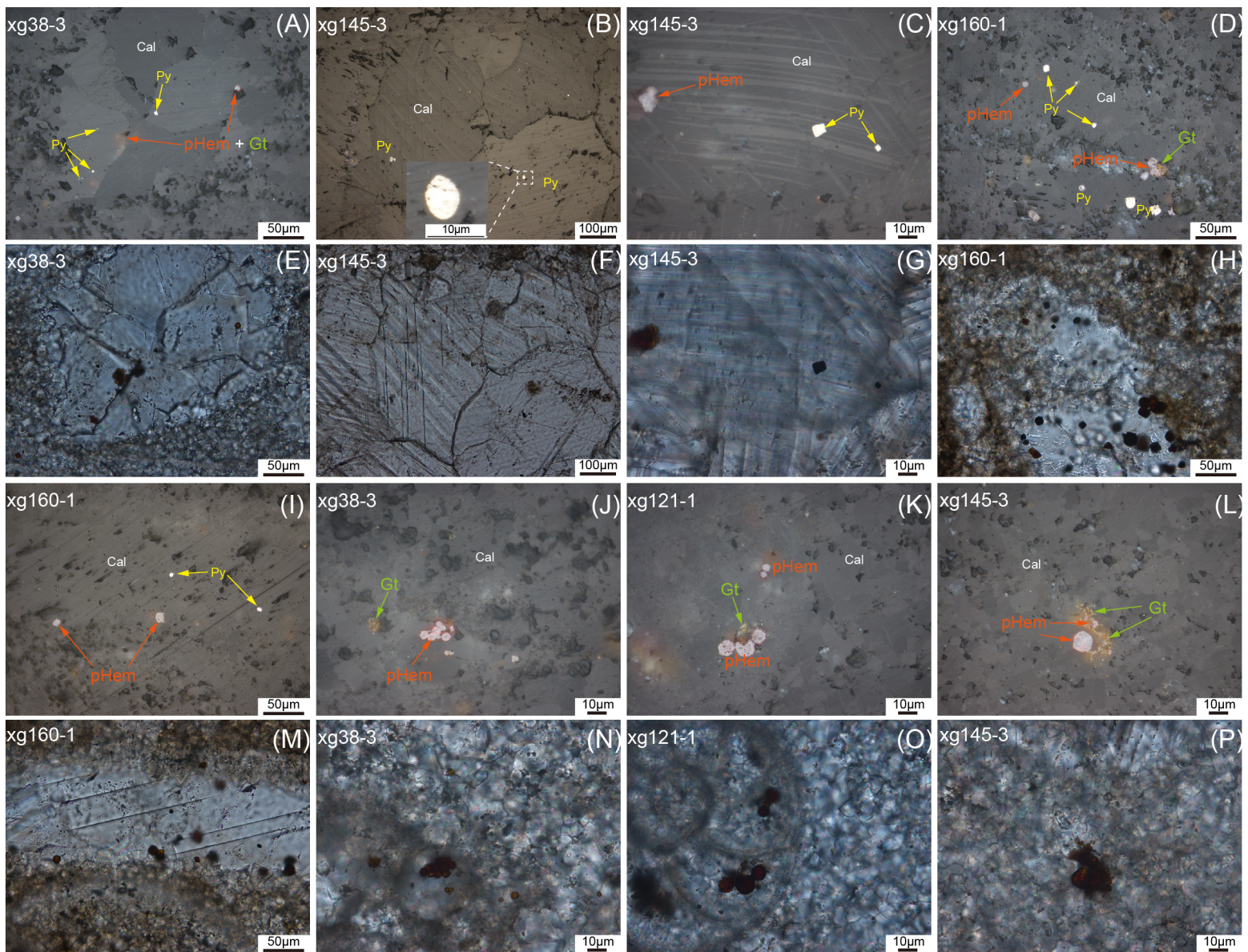
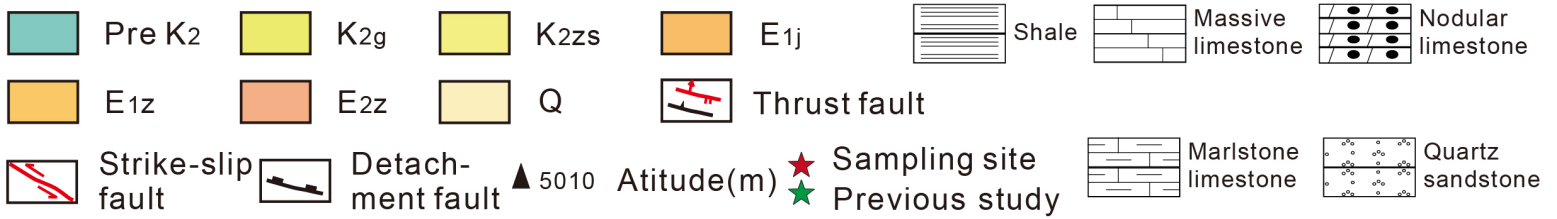
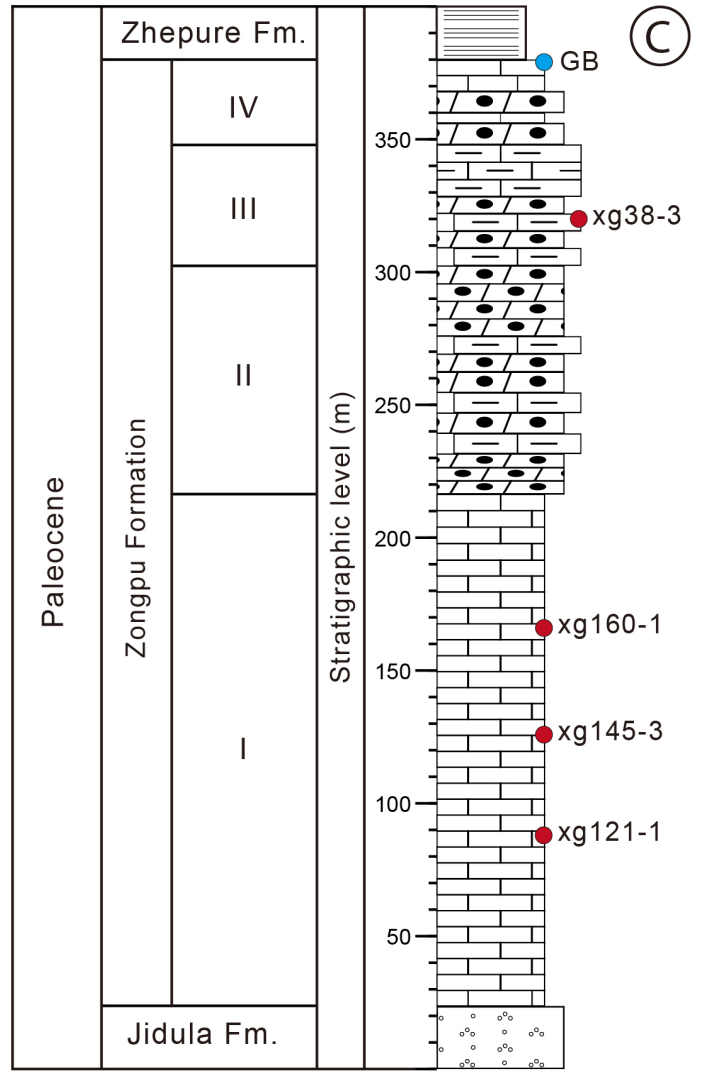
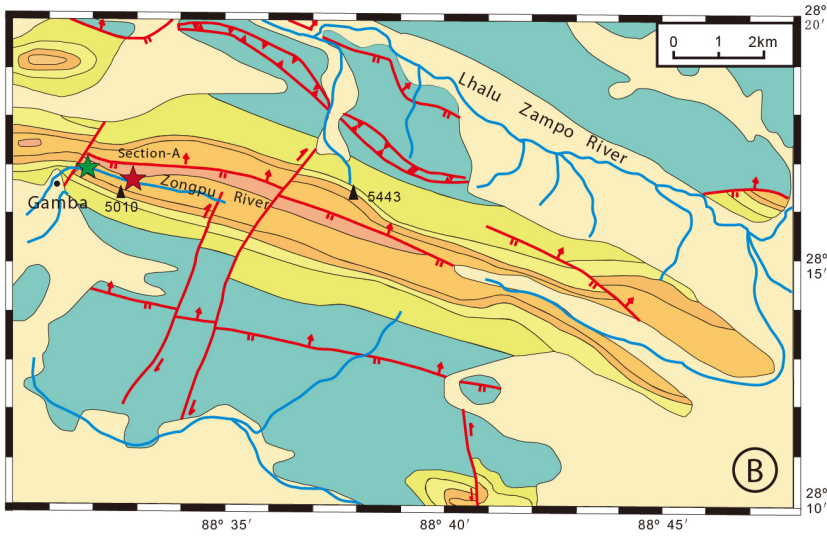
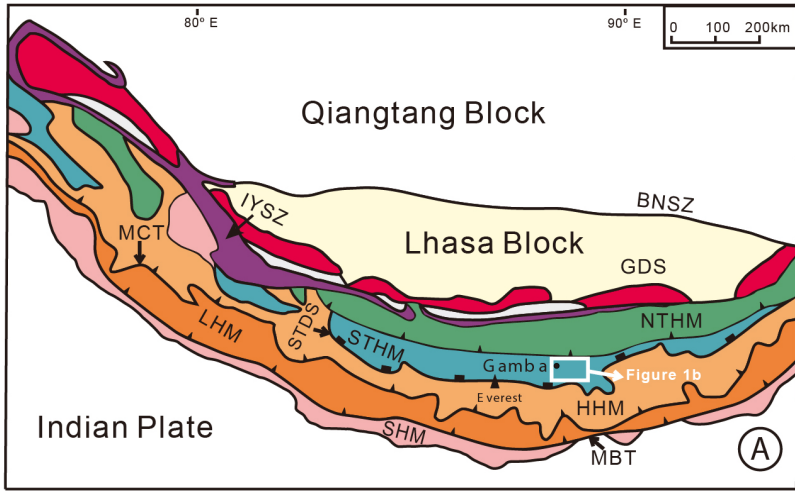


Figure 1.JPEG



Supplementary Material

1 Supplementary Figures

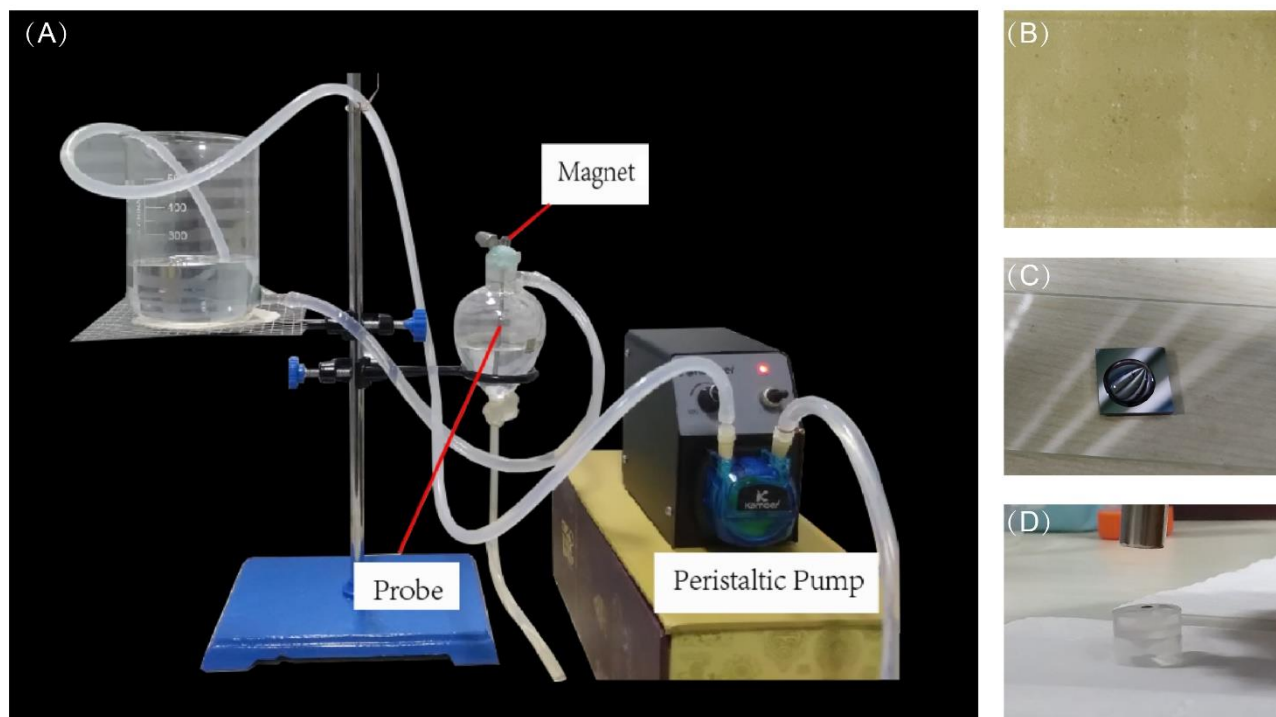


Figure S1. (a) Magnetic extraction apparatus. (b) Samples preparation of thin sections using resin as an adhesive; Magnetic extracts dropping on a monocrystalline silicon wafer (c), and TEM grid (d).

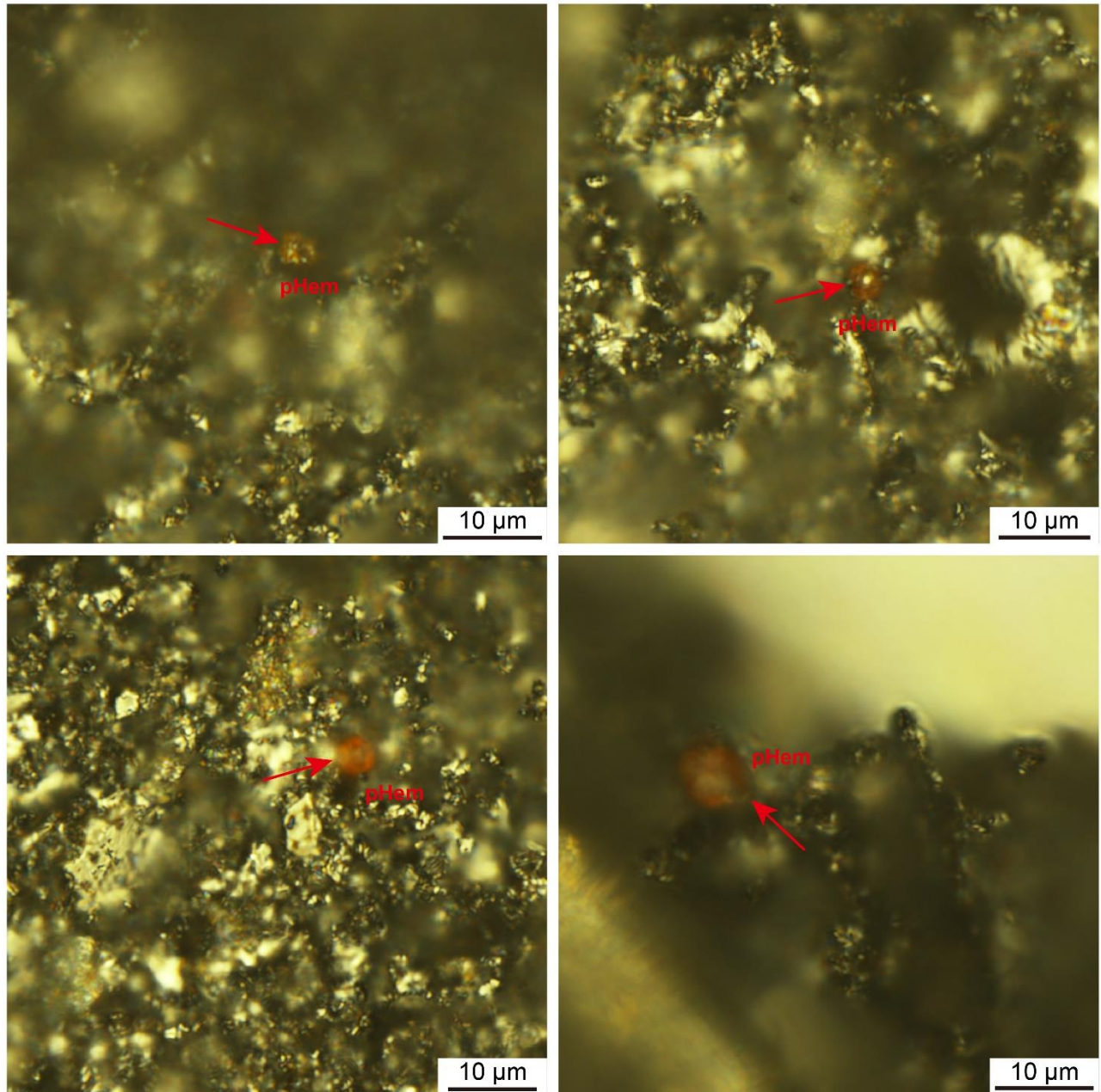


Figure S2. Photomicrographs illustrating the iron oxide spherules of magnetic extracts from the Zongpu carbonate rocks in the Gamba area under reflected light. pHem = pigment hematite.

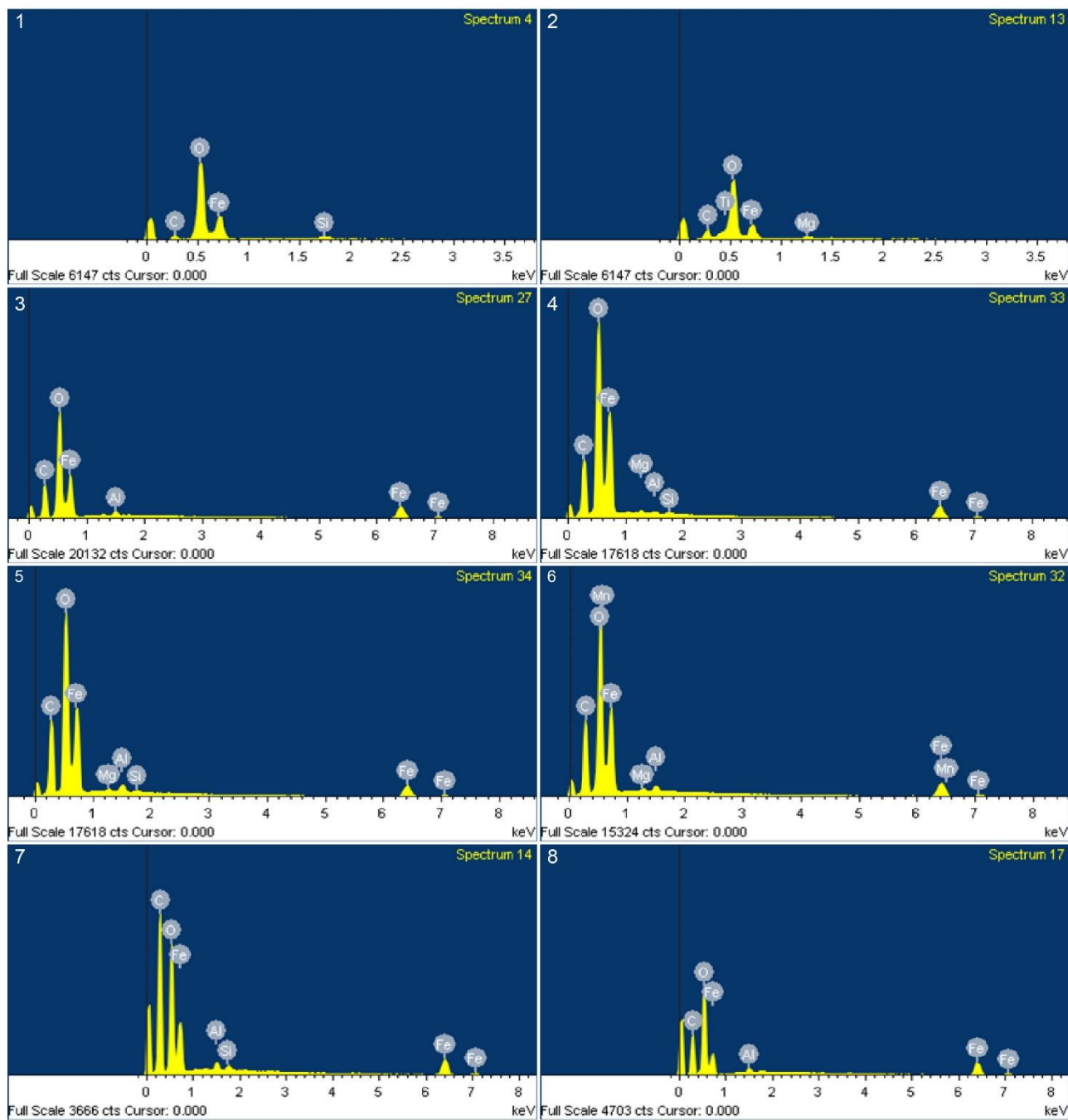


Figure S3. EDS analysis results of the spots shown in Figure 4.

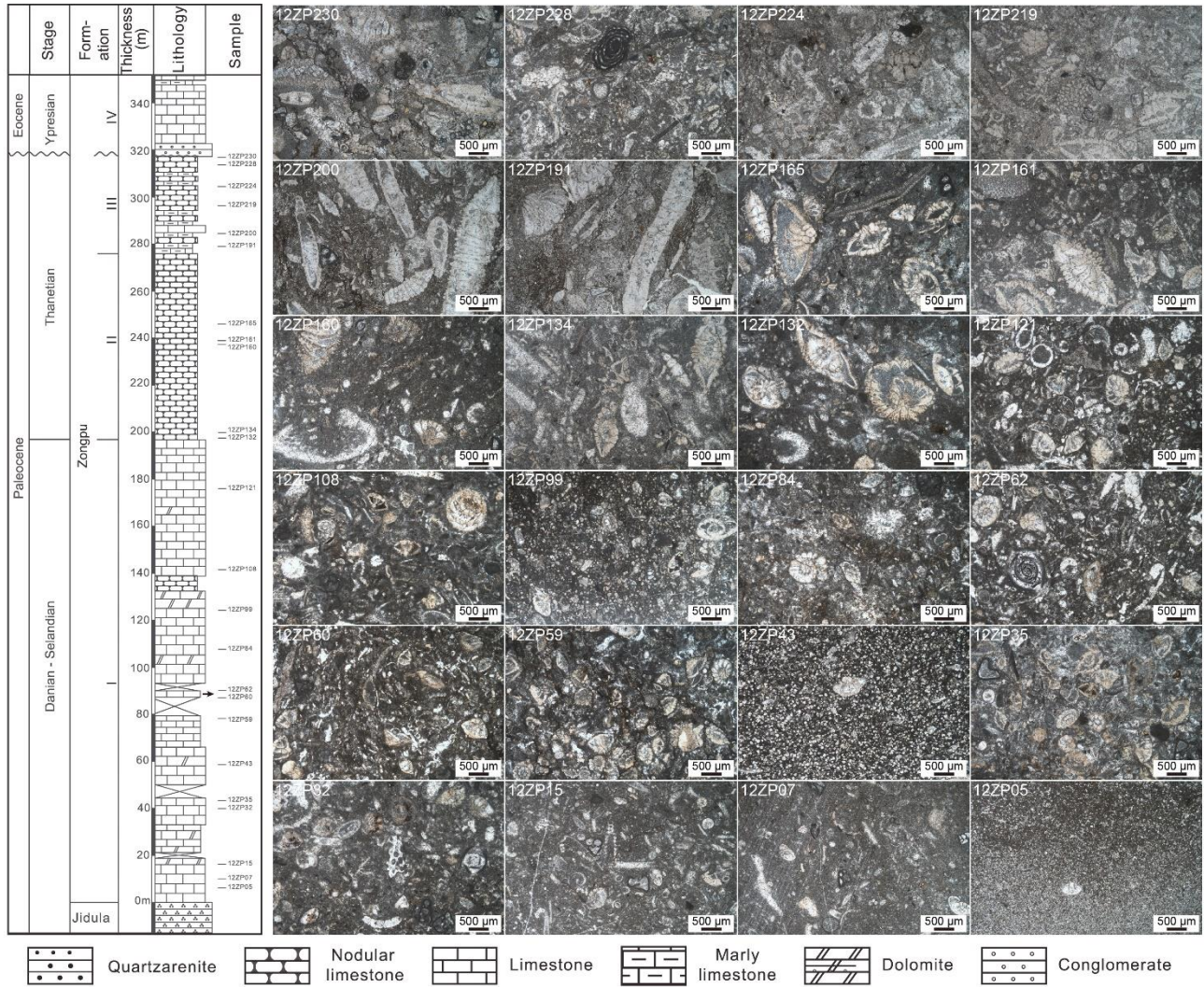


Figure S4. Left: Lithostratigraphy of the Zongpu Formation. Partial sampling localities of rock magnetic measurements in Huang et al. (2017a) were indicated, modified after Li et al. (2015) and Huang et al. (2017a). Right: Corresponding photomicrographs, collected from Li and Hu (2020). Please see www.csdata.org for more micrographs and detailed descriptions.

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