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1 **Complex mixture of organic matter in the xenolithic clast from the Zag meteorite revealed**
2 **by coordinated analyses using AFM-IR, NanoSIMS and STXM/XANES**

3
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32 **Abstract**

33 Primitive carbonaceous xenolithic clasts found in sturdy metamorphosed meteorites often
34 provide opportunities to reach labile volatile-rich materials which are easily destroyed during
35 atmospheric entry and materials which we do not have sampled as individual meteorites. Among
36 them, a xenolithic carbonaceous clast in the Zag ordinary chondrite has been providing us with
37 the opportunity to analyze a possible sample from D/P-type asteroids. Here we performed a new
38 suite of coordinated analyses of organic matter in the Zag clast using the state-of-the-art AFM-IR
39 combined with NanoSIMS, STXM/XANES, Raman, and (S)TEM on adjacent ultramicrotomed
40 thin sections from a single sample grain. We successfully demonstrated the practicality of
41 coordinated analyses using AFM-IR, Raman and NanoSIMS on the same sample area, as well as
42 STXM/XANES on adjacent (and nearly identical) thin sections to those used for AFM-IR. The
43 AFM-IR map and STXM maps provided consistent and complementary results. We found that at
44 least two types of organics were closely mixed in this specimen. One was deuterium-rich, C=O
45 rich organics with likely smaller aromatic domains, possibly originating in relatively oxidized
46 environments from D-rich precursors. The other type was less deuterium-rich, but aromatic-rich
47 organics, possibly produced in relatively reduced and higher temperature environments with less
48 deuterium-rich precursors. These characteristics point to complex mixtures of materials with
49 different origins and sampling a wide heliocentric range of the Solar System before accretion in
50 the parent body of the clast.

51

52 **Keywords**

53 meteorite, chondrite, asteroid, organic matter

54

55

56 **1. Introduction**

57

58 Primitive planetary materials are key to understanding the origin and evolution of our Solar
59 System. A wide variety of organic compounds are often found in these materials, dominated in
60 mass by a complex macromolecular organic matter, also known as insoluble organic matter
61 (IOM). The organic matter has formed and evolved at various stages of the solar system
62 evolution, including the precursor molecular clouds, protoplanetary disk, and subsequent small
63 bodies — some of them probably becoming raw materials for the origin of life.

64

65 However, the primitive and organic-rich small bodies are also usually volatile-rich and are easily
66 destroyed when they enter the Earth's atmosphere, and thus it is rare to collect these as
67 meteorites. Such fragile bodies are also easily pulverized when they collide with other small
68 bodies. However, if a primitive and fragile body collides with a firm body and fragments are
69 preserved in it, the fragile material may be able to reach the Earth as a xenolithic clast protected
70 inside a sturdy meteorite which is mechanically robust against atmospheric-entry alteration.

71 Carbonaceous chondrite-like clasts are often found in various meteorites including chondrites,
72 ureilites, howardites (e.g., Brearley, 1990; Brearley and Prinz, 1992; Zolensky et al., 1992;
73 Zolensky et al., 1996; Gounelle et al., 2003; Briani et al., 2009; Rubin and Bottke, 2009; Bonal et
74 al., 2010a; Bonal et al., 2010b; Briani et al., 2012; Kebukawa et al., 2017; Patzek et al., 2018;
75 Nittler et al., 2019; Patzek et al., 2020). A primitive xenolithic clast in the Zag H3-6 ordinary
76 chondrite is significantly rich in carbon, and shows molecular structure and isotopic
77 characteristics of organic matter different from the typical carbonaceous chondrite groups (Rubin
78 et al., 2002; Zolensky et al., 2017; Kebukawa et al., 2019a; Kebukawa et al., 2020). We have
79 proposed that the Zag clast originated from the outer solar system and was subsequently
80 incorporated into the ordinary chondrite parent body located in the inner solar system
81 (Kebukawa et al. 2019a, 2020). However, the detailed history of organic matter in the clast is not
82 well understood.

83

84 Classical methods to analyze trace organics in meteorites are solvent extraction and acid
85 demineralization. The spatial and textural contexts, however, are lost by these methods. To
86 understand chemical and physical processes that may have affected organic matter, fine-scale

87 observations of planetary materials are mandatory, including characterization of morphology and
88 spatial relationship between organics and minerals. Scanning transmission X-ray microscopy/X-
89 ray absorption near-edge structure (STXM/XANES) is a well-established technique for the
90 microscopic characterization of organic matter in meteorites (Le Guillou et al., 2014; e.g.,
91 Kebukawa et al., 2017; Vinogradoff et al., 2017) and other astromaterials including cometary
92 particles and micrometeorites (Cody et al., 2008; De Gregorio et al., 2011; Noguchi et al., 2017;
93 Yabuta et al., 2017). Alternatively, atomic force microscope-based infrared spectroscopy based
94 techniques such as AFM-IR (Kebukawa et al., 2019b; Mathurin et al., 2019; Phan et al., 2022)
95 and s-SNOM (Dominguez et al., 2014; Yesiltas et al., 2021; Young et al., 2022) have recently
96 begun to be employed to characterize astromaterials. These methods provide similar spatial
97 resolution at the scale of tens of nanometers, both detect organic functional groups although
98 STXM/XANES also allows the determination of oxidation states of e.g., Fe, and AFM-IR
99 provides mineral signatures. In addition, nanoscale secondary ion mass spectrometry
100 (NanoSIMS) produces isotopic signatures at comparable spatial resolution. These microscopic
101 methods provide complementary information of functional groups and isotopic ratios to elucidate
102 the origins and evolutionary history of organic matter which is heterogeneously mixed in the fine-
103 scale matrix of astromaterials. Thus, the same sample areas are often analyzed by both
104 STXM/XANES and NanoSIMS (De Gregorio et al., 2010; De Gregorio et al., 2013; Kebukawa
105 et al., 2019a; Kebukawa et al., 2020; Kebukawa et al., 2021b), and such coordinated analysis,
106 including AFM-IR, is also desired.

107

108 Here we report coordinated analysis using AFM-IR and NanoSIMS on the same area of the
109 unique organic-rich clast in the Zag meteorite. In addition, STXM/XANES, Raman, synchrotron-
110 based FTIR microspectroscopy, and (scanning) transmission electron microscopy ((S)TEM)
111 provide insight into the origins and evolutionary history of the organic matter in the Zag clast.

112

113 **2. Methods**

114 **2.1. Sample preparation with sulfur-embedded ultramicrotomy**

115

116 The ultramicrotomy method allows us to prepare several nearly-identical sections for different
117 analytical techniques, e.g., AFM-IR and STXM/XANES. The sulfur-embedding method was

118 employed to avoid epoxy resin which causes severe contamination for organic analysis. A small
119 particle of the Zag clast was embedded in molten sulfur for ultramicrotomed thin sections
120 (Noguchi et al., 2020). About 100 nm-thick sections were prepared with a LEICA
121 ultramicrotome using a DIATOME diamond knife, then transferred to copper TEM grids with
122 silicon monoxide supporting films (Pelco® Cu 300 mesh grid) for STXM/XANES and to a CVD
123 diamond window for AFM-IR and NanoSIMS analyses. After ultramicrotomy, the sections were
124 immediately heated gently at 80°C for 12 hours to sublimate the sulfur. We also prepared
125 approximately 100 nm-thick ultrathin sections in Embed 812 epoxy. Both sulfur-embedded and
126 epoxy-embedded ultrathin sections were placed on copper grids with carbon supporting film
127 (LAAD® Cu 100 mesh grid), and were investigated by (S)TEM.

128

129 **2.2. STXM/XANES**

130

131 Carbon-edge X-ray absorption near-edge structure (C-XANES) analyses were performed using
132 the scanning transmission X-ray microscope (STXM) at BL19A, Photon Factory, High-Energy
133 Accelerator Research Organization (KEK) (Takeichi et al., 2016). The elemental maps were
134 obtained by acquiring pairs of images with 0.1 μm steps per pixel below (I_b) and on the
135 absorption edges (I_a), namely at 280 and 292 eV for C K -edge, 525 eV and 539 eV for O K -edge,
136 and 705 eV and 709 eV for Fe L_3 -edge, and then taking the $-\ln(I_a/I_b)$ for each pixel. Spectral
137 image stacks at the C K -edge (280-320 eV) were acquired with energy step sizes of 0.1 eV in the
138 fine structure region and 0.5-1.0 eV in the pre- and post-fine structure regions, with a dwell time
139 of 3 ms, and with 0.1 μm steps per pixel. C-XANES spectra from the region-of-interests (ROIs)
140 were extracted and submitted to three-point smoothing to reduce noise, subtracted baselines were
141 fit by linear approximations at 280-283 eV, then normalized with the optical density at 295.5 eV.
142 The spectral component maps were obtained from images at 285.3 eV, 288.7 eV, and 290.5 eV
143 by subtracting an image at 283.0 eV, 289.2 eV, and 289.7 eV, respectively, after converting $-\ln(I/I_0)$,
144 where I is the spectral signal from ROI and I_0 is the spectral signal from blank area
145 adjacent to the ROI, for each image. We used the software aXis2000
146 (<http://unicorn.mcmaster.ca/aXis2000.html>) for STXM/XANES data analysis, and Adobe
147 Photoshop for generating RGB images.

148

149 2.3. Nanoscale IR spectroscopy (AFM-IR)

150

151 IR maps and spectra at the tens of nanometer scale were obtained using AFM-IR, a technique
152 combining an Atomic Force Microscope (AFM) to a tunable Infrared (IR) laser. An AFM probe
153 detects the photo-thermal expansion that occurs in the sample after IR absorption of the material
154 located just under the AFM tip (Dazzi et al., 2005). AFM-IR was used in tapping mode: in this
155 mode, the photo-thermal expansion is detected using a non-linear interaction (Mathurin et al.,
156 2019; Mathurin et al., 2020). The spatial resolution depends on the nanometer-sized AFM tip
157 radius, overcoming by orders of magnitude the diffraction limit of conventional IR microscopy.

158

159 The AFM-IR system was a NanoIR2 from Anasys Instrument (Bruker). The system was coupled
160 to a multi-chip Quantum Cascade Laser (QCL, Daylight Solutions) source covering the mid-IR
161 range from 900 cm^{-1} up to 1960 cm^{-1} . An Au-coated silicon AFM tip was used (Budget sensors,
162 tap 300). IR maps (AFM images of $50\text{ }\mu\text{m} \times 50\text{ }\mu\text{m}$, 1000×1000 pixels) were acquired at fixed
163 tuned laser wavenumbers: 1020 , 1500 , 1600 and 1740 cm^{-1} , corresponding to the absorptions of
164 silicates, carbonates, aromatic carbon and carbonyl chemical groups, respectively. The IR
165 mapping acquisitions were made using a 0.05 Hz scan rate with a laser power of 10.33% of the
166 QCL chip, a repetition rate of 1355 kHz and a pulse width of 160 ns .

167

168 These maps were combined in a composite three color RGB image (1020 cm^{-1} , green; 1500 cm^{-1} ,
169 blue; 1740 cm^{-1} red). Before combining the maps, they were corrected for minor AFM stripping
170 by interpolation, realigned on the 1020 cm^{-1} map using their topographic AFM maps, and
171 slightly smoothed using a 5 pixels kernel. To maximize the contrast between bands with different
172 intrinsic intensities in the composite, the intensity of each image recorded at a given
173 wavenumber was first normalized to its maximum intensity over the entire map before
174 combining them.

175

176 Spectra spanning the full QCL multi-chip spectral range were also recorded along the line shown
177 in **Fig. 2c**. Spectra presented in this study were acquired at a 1 cm^{-1} spectral resolution using the
178 same laser parameter as IR mappings spectra and are averages of 8 individual spectra taking on
179 the same position.

180

181 **2.4. Synchrotron Radiation Infrared microspectroscopy (SR- μ FTIR)**

182

183 Infrared measurements were performed on the SMIS beam line at the SOLEIL synchrotron. The
184 synchrotron IR beam was coupled to a Continuum infrared microscope (ThermoFisher
185 Scientific) to record hyperspectral maps from 750 cm^{-1} to 5000 cm^{-1} . The infrared spot size was
186 optimized close to the diffraction limit, with an IR beam $8\text{ }\mu\text{m} \times 8\text{ }\mu\text{m}$ confocal aperture. We
187 used a spatial sampling measured with a $4\text{ }\mu\text{m}$ step allowing the map to cover the entire
188 fragment. Each spectrum in the map was recorded at 8 cm^{-1} spectral resolution and is the average
189 of 64 individual scans.

190

191 **2.5. Raman**

192

193 A Raman spectral map was acquired with a Thermo Fisher DXR spectrometer, using a laser
194 source at 532 nm at a power of $\sim 300\text{ }\mu\text{W}$ on the sample to prevent sample alteration. The $11\text{ }\mu\text{m}$
195 $\times 26\text{ }\mu\text{m}$ map located on the central region of the sample was recorded with a $\times 100/0.9$ numerical
196 aperture objective at a sampling step of $1\text{ }\mu\text{m}$. Two scans with integration times of 10 s each
197 were co-added for each spectrum. The spot size of the Raman spectra was of the order of $\sim 1\text{ }\mu\text{m}$,
198 i.e. lower than that of conventional IR spectra.

199

200 The Raman spectra were fitted as described in Dartois et al. (2018). The Raman spectra are
201 analysed using a classical Raman band fitting procedure decomposition contributing to the D and
202 G bands, consisting of the deconvolution of five sub-bands (e.g., Sadezky et al. 2005; Kouketsu
203 et al. 2014), then grouped into two main contributions (“D” = $D1 + D2 + D4$, in green and “G” =
204 $D3 + G$ in red, shown in **Fig. 3**).

205

206 **2.6. NanoSIMS**

207

208 The hydrogen isotopic measurements were performed by scanning ion imaging with the
209 NanoSIMS instrument at MNHN in Paris. The NanoSIMS isotopic images were acquired with a
210 16 keV Cs^+ primary ion beam with a probe dwell time of 1 ms per pixel. The Cs^+ primary beam

211 was set at ~ 12 pA, resulting in a probe diameter of ~ 250 nm. The H^- and D^- secondary ions were
212 filtered in the mass spectrometer with a mass resolving power of 3000. Images of H^- , D^- were
213 simultaneously recorded on 2 individual electron multipliers (EM) as a stack of about 80 frames
214 over $20 \mu\text{m} \times 20 \mu\text{m}$ areas with 256×256 pixels per frame. We imaged 2 zones located on upper
215 and middle part of the sample (see **Fig. 2b**).

216
217 In order to avoid charging effects, the sample was coated with 20 nm of gold prior to
218 introduction into the NanoSIMS instrument. In order to reduce the transition time necessary to
219 reach the sputtering equilibrium regime, each imaged area was implanted for about 10 minutes
220 with a primary ion beam of 250 pA over $30 \mu\text{m} \times 30 \mu\text{m}$ areas centered on the region analyzed.

221
222 The instrumental mass fractionation was measured before and after sample analyses using
223 terrestrial standards (Type III Kerogen, Orgueil IOM and dedicated organic polymer thin films
224 with a large range of D/H ratios (Bardin et al., 2015)).

225
226 The NanoSIMS images were processed using the LIMAGE software developed by Larry Nittler
227 (Carnegie Institution in Washington DC, currently Arizona State University), ImageJ
228 (<https://imagej.nih.gov/ij/>) and IDL software. We performed dead time correction (44 ns) on all
229 data and frame alignment following the usual procedures. The frames were subsequently
230 summed and a mask was applied with a minimum threshold set at 1500 cts of H^- ions.

231
232 The H^- images were aligned to the AFM topographic images using the structures located on the
233 right border of the sample. The same alignment was then used for the D^- image and resulting
234 D/H images were produced using a smoothing kernel of 5×5 pixels to take account of the size
235 of the Cs^+ probe and reduce statistical fluctuations.

236 237 **2.7. TEM**

238
239 A field emission (S)TEM JEOL JEM-3200FSK at Kyushu University operating at 300 kV
240 equipped with an Ω energy filter was used for observation of phases in the ultrathin sections.
241 Another field emission (S)TEM JEOL JEM-2100F operating at 200 kV at Kyoto University was

242 used for additional (S)TEM observation and quantitative energy dispersive spectrometer (EDS)
243 analysis of minerals in the ultrathin sections. The ζ -factor method was used for quantitative
244 analysis (Watanabe and Williams, 2006). We used the ζ -factors determined by JEOL Co. Ltd.
245 We confirmed that their ζ -factors were applicable to obtain the chemical compositions of
246 minerals. EDS analysis of minerals in an ultramicrotomed section of Zag was also performed
247 using TEM JEOL JEM-2000FXII operating at 200 kV at Ibaraki University. The Cliff-Lorimer
248 method was used for quantitative analysis. K factors were determined experimentally using
249 many mineral standards.

250

251 **3. Results**

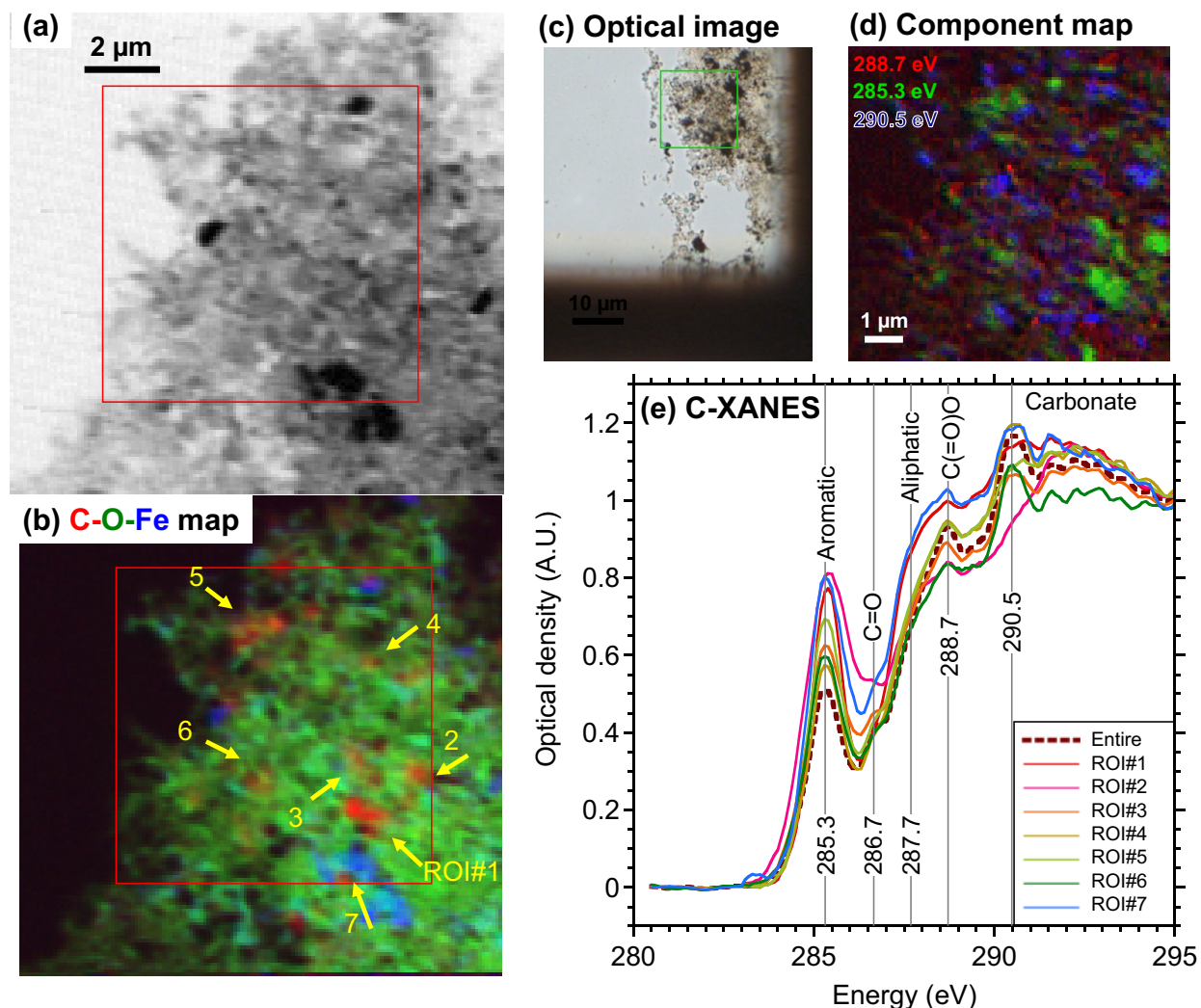
252 **3.1. STXM/C-XANES**

253

254 The STXM carbon map shows that sub-micrometer organic particles are spread over the section
255 of the Zag clast (**Fig. 1b**). Some particles show a well-defined carbon absorption (e.g., ROI#1
256 and #2 in **Fig. 1b**), and some are not well distinguished and rather diffuse into the mineral matrix
257 (e.g., ROI#6 in **Fig. 1b**). The molecular structures revealed by C-XANES show common features
258 with large variations in their relative intensities (**Fig. 1e**). Common features are a dominant peak
259 at 285.3 eV due to aromatic carbon, peaks at 288.7 eV due to C(=O)O (carboxy/ester) and at
260 290.5 eV due to carbonate (that could be both organic and inorganic), a small peak at 286.7 eV
261 due to C=O (ketone), and a shoulder at \sim 287.7 eV due to aliphatic carbon. The component map
262 (**Fig. 1d**) shows heterogeneities in the intensities of aromatic (285.3 eV) and C(=O)O (288.7 eV).
263 The well-defined particles, e.g., ROI#1 and #2 in **Fig. 1b**, tend to have a larger aromatic peak
264 (285.3 eV) and a smaller carbonate peak (290.5 eV) compared to the average spectra of the entire
265 analyzed area (the red square in **Fig. 1b**). While, ROIs#3, #4, and #6 showed less-aromatic
266 nature. These features generally agree with that of unheated carbonaceous chondrites (e.g., Le
267 Guillou et al., 2014). The spectral component map of the Zag clast (**Fig. 1d**) show that aromatic
268 carbon (285.3 eV) and carbonate (290.5 eV) are distributed separately as $< \sim 1 \mu\text{m}$ spots, while
269 C(=O)O (288.7 eV) is distributed as smaller spots or spread all over within a diffuse organic
270 matter component. The component map is consistent with C-XANES spectra extracted from
271 ROI#1 and #2 which are dominated by aromatic carbon (285.3 eV). It should be noted that
272 287.5-289 eV region is easily affected by contamination and other artifacts, particularly with

273 regions containing small amounts of carbon. Thus, the spreading of C(=O)O could be
 274 contamination/artifacts. However, C-rich regions as shown by arrows in **Fig. 1b** are likely less
 275 affected by these artifacts.

276
 277



278
 279 **Fig. 1.** (a) X-ray absorption image at 525 eV of the ultramicrotomed slice of the Zag clast. (b)
 280 carbon (in red) – oxygen (in green) – iron (in blue) map generated from STXM/XANES
 281 elemental maps, with ROI numbers indicated in yellow. (c) Optical microscopic image. (d)
 282 Compositional map generated by the stack data from the red squared region, at energies of 288.7
 283 eV (C(=O)O, in red), 285.3 eV (aromatic C=C, in green) and 290.5 eV (CO₃, in blue). (e) C-
 284 XANES spectra from the red square entire region, and from the ROIs indicated by arrows in b).
 285

286 3.2. SR- μ FTIR, AFM-IR, Raman, and NanoSIMS

287

288 Conventional synchrotron μ FTIR spectra of the ultramicrotomed slice shows peaks at $\sim 1740\text{ cm}^{-1}$, $\sim 1500\text{ cm}^{-1}$, and 1020 cm^{-1} corresponding to carbonyl (C=O), carbonates (CO₃ mode), and
289 silicate Si-O, respectively (**Fig. 2f, lower curve**). We display in **Fig. 2** AFM-IR maps recorded at
290 these fixed wavenumbers and combined to obtain a composite three-color image at high spatial
291 resolution (**Fig. 2c**). In addition, a line of spectra crossing the ultramicrotomed sample, covering
292 the full AFM-IR laser spectral span, was recorded. The integrated AFM-IR signal for the
293 different bands retrieved from the analysis of this spectral line map is shown in **Fig. 2e**. Three
294 individual AFM-IR spectra extracted in regions of high contrast at small spatial scale are also
295 shown in **Fig. 2f**. These spectra show two important features: (i) AFM-IR data are in agreement
296 with the conventional FTIR spectrum for the spatially widespread and intense bands, (ii) They
297 sample variations of the individual bands at much higher spatial resolution, allowing
298 measurement, for example, of the extent of at least four individual carbonates grains with
299 (sub-)micron sizes, which would be inaccessible with conventional μ FTIR. As shown in the
300 organics composite image in **Fig. 2g**, this spectral line crosses a region of interest with high C=O
301 and less C=C contribution than the lower part of the slice. The intrinsically lower band strength
302 in the FTIR of the C=C absorption band can be however seen locally in the AFM-IR spectrum of
303 #2 in **Fig. f**. At the global scale recorded FTIR spectrum, this FTIR C=C contribution appears as
304 minor because the spectrum of the entire slice is largely dominated by the intense carbonyl,
305 carbonate and silicates bands. The AFM-IR map is consistent to the STXM map in **Fig. 1d**, but
306 the large organic particles were not observed in the STXM. The large organic particle could be
307 lost in the section for STXM (there is a large void in the section as shown in **Fig. 1c** lower right),
308 or the large particle was not hit in the slice used for STXM due to up to several hundreds of nm
309 differences (equivalent to the thickness of several ultramicrotomed slices) of the sampling
310 positions between the slices for STXM and AFM-IR.

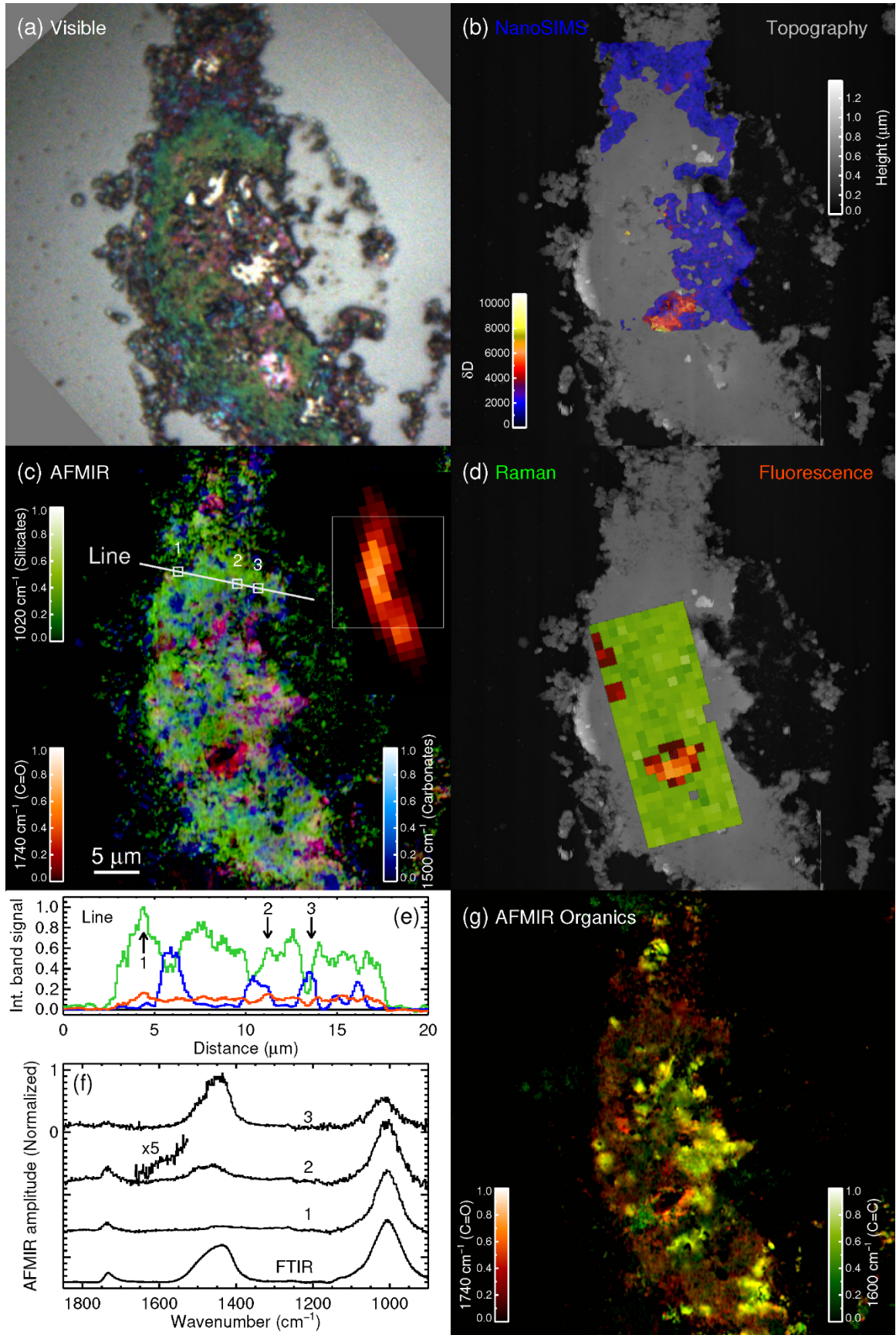
311

312
313 The Raman fluorescence map is indicative of a change of nature of the underlying organics. The
314 absolute level of fluorescence will strongly depend on the numerical aperture on the sample. The
315 map of **Fig. 2d** was obtained by integrating the recorded Raman signal in a 500 cm^{-1} wide
316 spectral window ($3500\text{-}3000\text{ cm}^{-1}$), i.e., above the D and G Raman bands, and corresponding to

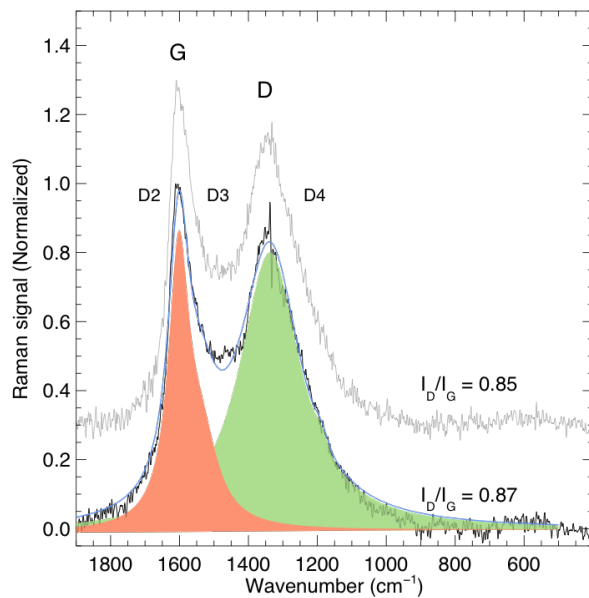
317 a window centered at about 540 nm. Raman composite image **Fig. 2d** shows an aromatic
318 component is widespread even outside the much more concentrated and intense aromatic local C
319 spots such as ROI#1 and 2 identified in **Fig. 1b** STXM/XANES elemental maps. Raman spectra
320 of the Zag clast show typical carbonaceous features represented by a G-band at around 1600
321 cm^{-1} and a D-band at around 1350 cm^{-1} (**Fig. 3**). The intensity ratios (I_D/I_G) of the G and D
322 bands are indicated in **Fig. 3**. The composite map generated with the AFM-IR data shows that
323 the submicrometer distribution of organic matter in Zag clast is consistent with STXM/XANES
324 maps (**Fig. 1b,d**). Note that the STXM/XANES analysis was done with ultramicrotomed sections
325 from the same sample grain and adjacent to the ones used for the AFM-IR and NanoSIMS
326 analyses.

327
328 For the isotopic data, as a result of the applied threshold on the H⁻ counts, only areas with
329 sufficiently high H concentrations, i.e. with substantial hydrogen-rich organic matter, are
330 indicated on the composite image displayed in **Fig. 2b**. The NanoSIMS D/H image reveals a
331 large hot-spot zone of a few tens μm^2 with high D/H ratios, located in the lower middle of the
332 grain. The AFM-IR map permits identification of a large organic grain with an intense carbonyl
333 signature that precisely overlaps the D-rich hot-spot and visible fluorescence (lower middle in
334 **Fig. 2b,c,d**). This large D-rich organic grain has a δD value of (4200-4500)‰ while the δD of
335 the surroundings and the main part of the sample is \sim (1000-1100)‰. The D/H ratios of both the
336 hot-spot and bulk are in agreement with the isotopic analysis reported in previous studies on the
337 same clast from Zag (Kebukawa et al., 2019a; Kebukawa et al., 2020). The D-rich organic grain
338 is associated with high Raman fluorescence clearly visible on **Fig. 2d**. In this zone the carbonyl
339 signal is high, whereas the C=C is low (**Fig. 2g**), suggesting abundant aliphatic organic moieties
340 more susceptible to fluorescence. The high fluorescence nature shown by Raman typically
341 indicates enrichment of double bonds but each aromatic domain must be small (Bertrand et al.,
342 1986). There is a C=O rich particle on the middle of **Fig. 2**, which also shows high fluorescence.
343 The area on the left of the D-rich zone is dominated by aromatics with less C=O groups (**Fig.**
344 **2g**). The area on the right of the D-rich zone is dominated by diffuse organic matter together with
345 a local concentration in form of organic particles (**Fig. 2c,g**).

346



348 **Fig. 2.** Panel of images showing: (a) visible optical reflection image; (b) NanoSIMS δD image
 349 (in colors) overlaid on AFM topography (grey scale); (c) AFM-IR composite RGB map
 350 combining normalized AFM-IR maps recorded at 1740 cm^{-1} (carbonyl), 1500 cm^{-1} (carbonates),
 351 1020 cm^{-1} (silicates), position of a spectra line map, and insert of the optimized μFTIR
 352 synchrotron map in the 1740 cm^{-1} carbonyl band; (d) Raman I_D/I_G map (green scale) and
 353 fluorescence (red scale) overlaid over the AFM topography map (grey scale); (e) AFM-IR line
 354 map obtained by integrating the three main band amplitudes (C=O in red at 1740 cm^{-1} ,
 355 carbonates in blue at 1500 cm^{-1} , silicates in green at 1020 cm^{-1}) and (f) three spectra extracted
 356 from the line (e) showing the compositional diversity at the sub μm scale. The lowest spectrum is
 357 a conventional IR spectrum of the entire ultramicrotomed slice; (g) AFM-IR two color composite
 358 image focusing on the vibrational groups of organics at 1740 cm^{-1} (C=O, red) and 1600 cm^{-1}
 359 (C=C, green).
 360



361
 362 **Fig 3.** (lower curve) Average Raman spectrum over the entire map of **Fig. 2d**, excluding the
 363 fluorescence dominated regions (normalized to the maximum), deconvolved in D and G bands.
 364 (upper curve) The best S/N individual spectrum.

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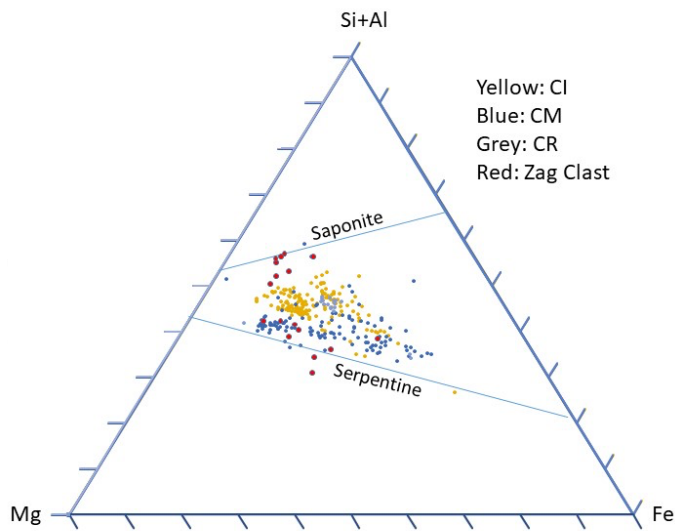
367 3.3. Mineralogy of the Zag clast

368

369 The mineralogy of the Zag clast was previously investigated by SEM-EDS, EPMA and TEM
370 analyses (Zolensky et al., 2003; Fries et al., 2013). The clast is predominantly a fine-grained
371 matrix, consisting of a mixture of serpentine, saponite, magnetite, Ca phosphates, organic-
372 dominated grains, and pyrrhotite with exsolved pentlandite. Set within the matrix are larger
373 aggregates of saponite, serpentine phases, magnetite, pyrrhotite/pentlandite, dolomite and
374 halite/sylvite crystals. The largest observed dolomite aggregate has very interesting zoning: Mn-
375 rich cores, carbonate, and very thin Na-Mg-rich rims. The Na-rich rims of these dolomites
376 suggest a link between this clast and the scattered halite/sylvite xenoliths in Zag. EDS and
377 EPMA measurements of phyllosilicate composition (**Fig. 4**) indicate intergrown serpentine and
378 saponite, with a composition typical of CI, CM and CR carbonaceous chondrites. These
379 identifications were verified by TEM observations. The clast appears to be homogeneous in
380 mineralogy, as far as can be observed by SEM-BSE observation.

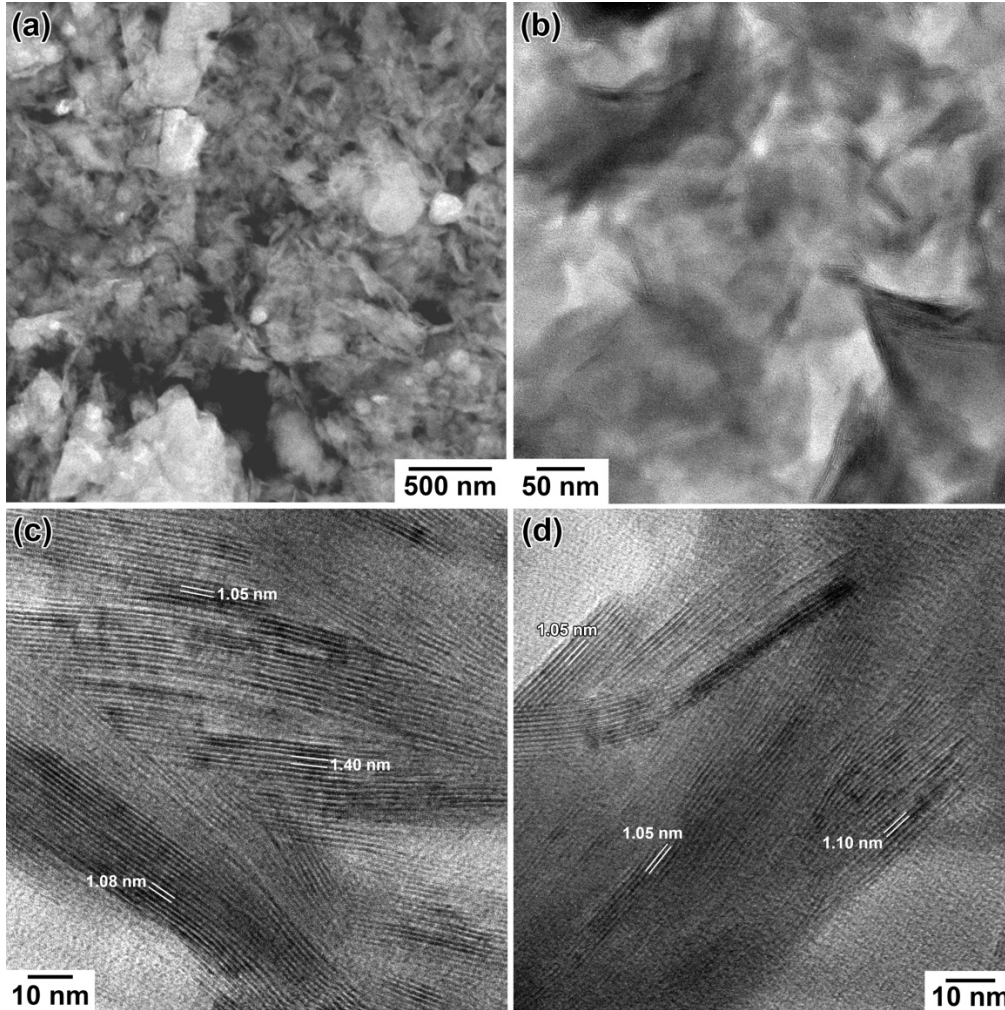
381
382 TEM observation of the ultrathin sections of four fragments from the clast shows that they are
383 composed mainly of phyllosilicates containing abundant Fe-Ni sulfides (pyrrhotite Fe_{1-x}S and
384 pentlandite $(\text{Fe},\text{Ni})_9\text{S}_8$), magnetite Fe_3O_4 , Mg-Fe carbonate $(\text{Mg},\text{Fe})\text{CO}_3$, and dolomite
385 $\text{CaMg}(\text{CO}_3)_2$ (**Figs. 5 and 6a-d**). High-resolution TEM images of phyllosilicates indicate that the
386 phyllosilicates are predominantly composed of saponite (**Figs. 5c, d**), which means that the four
387 fragments were obtained from the areas that contained mainly saponite as phyllosilicates.
388 Therefore, major chemical compositions of the phyllosilicates in the two fragments are plotted
389 along the saponite solid solution line (**Fig. 7a**), which is consistent with the TEM observation
390 (**Figs. 5c, d**). Their predominantly saponite-rich compositions are similar to those of
391 phyllosilicates in hydrated fine-grained Antarctic micrometeorites (AMMs) and chondritic
392 smooth interplanetary dust particles (CS IDPs) (Noguchi et al., 2002; Nakamura et al., 2004;
393 Sakamoto et al., 2010; Dobrică et al., 2019), as well as coarse-grained phyllosilicates in the
394 carbonate-rich lithology of the Tagish Lake ungrouped C2 chondrite (Nakamura et al., 2003). In
395 addition, we found magnesiochromite MgCr_2O_4 (**Fig. 6e**), which was reported from CI
396 chondrites (Rubin and Ma, 2021).

397



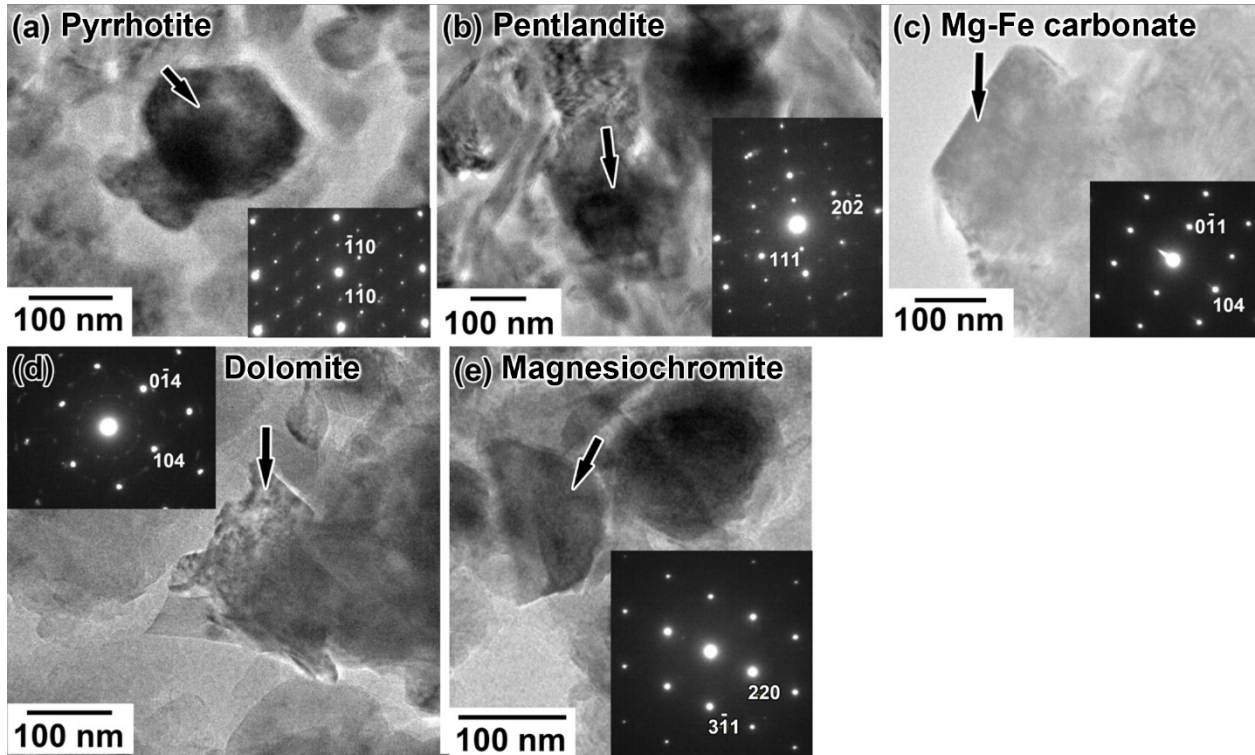
398

399 **Fig. 4.** Atomic % triangular diagram comparing bulk composition of phyllosilicates (essentially
 400 mixtures of serpentine family phases and saponite) in the Zag clast with matrix of CI, CM and
 401 CR chondrites. Note that there are some measurements of essentially pure aggregates of saponite
 402 and some of serpentine. Because the solid solution lines of saponite and serpentine shown in this
 403 diagram are actual mineral compositions rather than ideal ones, the lines are slanted rather than
 404 horizontal.



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Fig. 5. TEM images of mineral phases in the Zag clast. (a) Low magnification high-angle annular dark-field – scanning transmission electron microscope (HAADF–STEM) image of the clast. It is composed mainly of fibrous phyllosilicates embedding Fe-Ni sulfides and Mg-Fe carbonate. (b) Bright-field (BF) TEM image of phyllosilicates. (c and d) High-resolution BF-TEM images show that the phyllosilicates are composed of saponite.



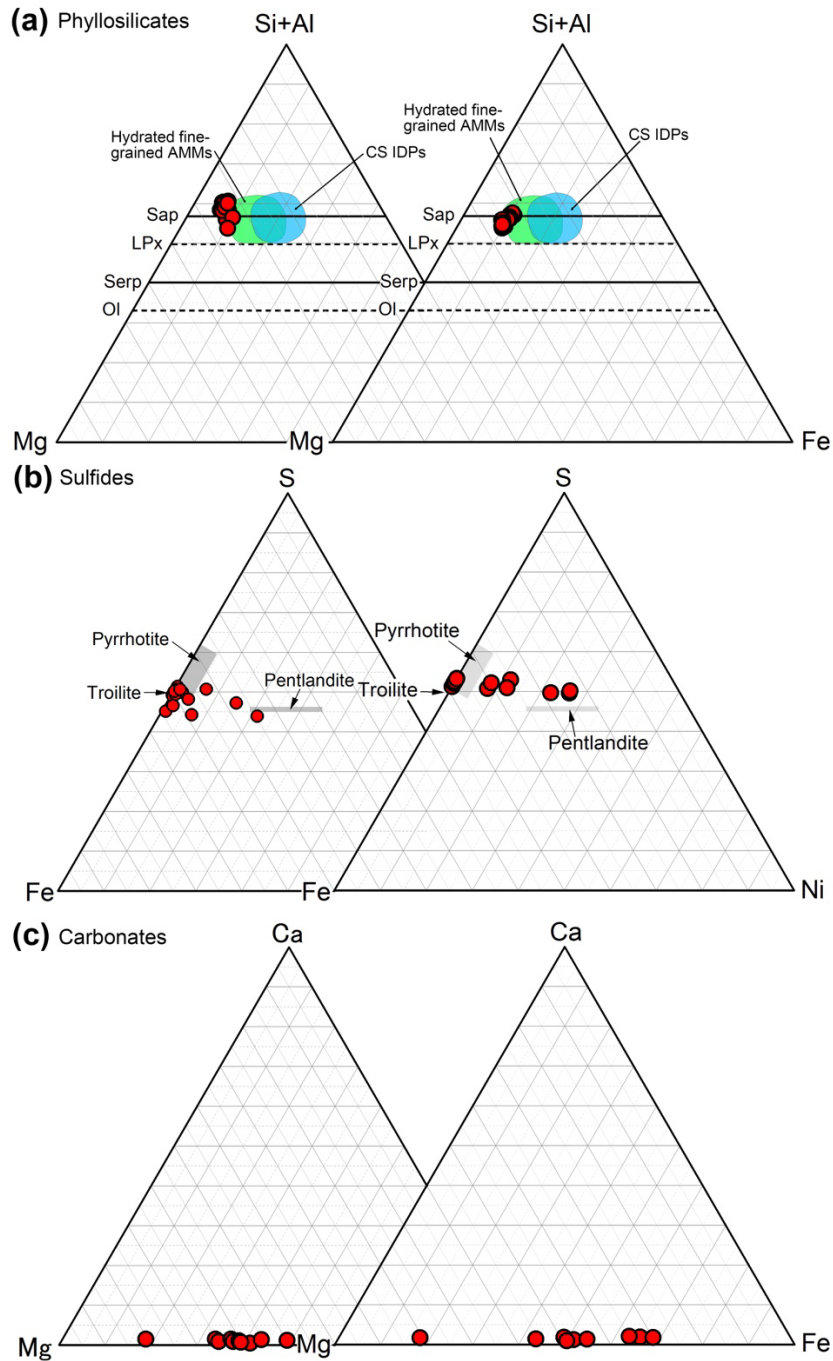
412

413 **Fig. 6.** TEM images of minerals and their selected area electron diffraction (SAED) patterns in

414 the clast. (a) Pyrrhotite. (b) Pentlandite. (c) Mg-Fe carbonate. (d) Dolomite. (e)

415 Magnesiochromite.

416



417

418 **Fig. 7.** Major chemical compositions of phyllosilicates, sulfides, and carbonates in the two
 419 fragments from the Zag clast. (a) [Si+Al]-Mg-Fe atomic ratios of phyllosilicates. Compositional
 420 fields of phyllosilicates in hydrated fine-grained AMMs and CS IDPs are from Noguchi et al.
 421 (2002), Nakamura et al. (2004), Sakamoto et al. (2010), and Dobrică et al. (2019). Because the
 422 solid solution lines of saponite and serpentine in these diagrams are ideal stoichiometric ones,
 423 they are parallel rather than slanted. (b) S-Fe-Ni atomic ratios of Fe-Ni sulfides in the clast. (c)

424 Ca-Mg-Fe atomic ratios of carbonates in the clast. Sap: saponite, LPx: low-Ca pyroxene, Serp:
425 serpentine, and Ol: olivine.

426

427 **4. Discussion: Origins of organic matter in the Zag clast**

428

429 The organics in the Zag clast appear in two different forms as shown by STXM/XANES, AFM-
430 IR, Raman and NanoSIMS: i) deuterium-rich, C=O rich with likely smaller aromatic domains,
431 and ii) less deuterium-rich, relatively aromatic-rich with likely larger aromatic domains. Both
432 types of organics have various shapes and size ranges from sub- μm up to a few μm . The latter
433 would correspond to the “individual particles” in carbonaceous chondrites reported by Le
434 Guillou et al. (2014) using STXM. These two forms should have different origins and/or
435 alteration processes since their molecular structures and D/H ratios are different. The former was
436 likely produced in relatively oxidized environments from D-rich precursors. Such organic
437 particles could be formed during aqueous processing by hydrophobic aggregation (Cody et al.,
438 2011; Kebukawa et al., 2013), from D-rich precursors from cold interstellar/outer solar nebulae
439 (e.g., Mumma and Charnley, 2011) or in the ionizing region of the Solar System (Remusat et al.,
440 2006; Gourier et al., 2008; Laurent et al., 2015). The other possibility is photosynthesis of the icy
441 dust particles in interstellar/outer solar nebulae (e.g., Muñoz Caro and Dartois, 2013). Such icy
442 dust could retain organic refractory materials after warming up in the solar nebula or
443 planetesimals.

444

445 The latter, less deuterium-rich and aromatic-rich organics were likely produced in relatively
446 reduced and higher temperature environments with less deuterium-enriched precursors, possibly
447 in the inner Solar System. Fischer-Tropsch type (FTT) synthesis could have occurred at higher
448 temperature regions in the nebula or in larger bodies (e.g., Anders et al., 1973; Kress and Tielens,
449 2001; Sekine et al., 2006; Nuth et al., 2008). Fischer-Tropsch synthesis intrinsically produces
450 hydrocarbons from CO and H₂ gases with a catalyst, but further processing could form aromatic
451 materials. Also, one cannot exclude the possibility of the contribution of carbonaceous materials
452 from plasmatic environments around evolved stars (e.g., Sakata et al., 1983; Kwok, 2004; Endo
453 et al., 2021). However, the FTT products are expected to have lower D/H ratios than the
454 observed values ($\sim 1000\%$), since the D/H ratio of H₂ in nebular gas was $\sim 2.1 \times 10^{-5}$ (-865%)

455 (Geiss and Gloeckler, 1998). Such high D/H of aromatic-rich organics can be explained by the
456 following two possibilities; (1) D-H exchange with H_2D^+ in a highly ionized region of the
457 protoplanetary disk (Remusat et al., 2006; Gourier et al., 2008), and/or (2) D-H exchange with
458 D-rich water in the parent body of the clast (Kebukawa et al. 2019a), whereas D-H exchange is
459 slow at low temperatures enough to keep heterogeneities in D/H ratios (Kebukawa et al. 2021a).

460

461 Such mixtures of organics with various origins imply a complex mixing history in the
462 protoplanetary disk before accretion of the original parent body of the Zag clast, involving
463 materials from various heliocentric locations (Remusat et al., 2010). In any cases, aqueous
464 alteration in the clast parent body overwrote isotopic and molecular structure information of the
465 original materials and thus it is difficult to precisely constrain the formation scenarios.

466

467 Large condensed organic particles around 5 micrometers in diameter (such as shown in **Fig. 2c**)
468 are rare in chondrites. Such large condensed organic particles were only reported in QUE 99177
469 (CR2) (Alexander et al., 2017), other than from the previous studies of the Zag clast (Kebukawa
470 et al. 2019, 2020). Some micrometeorites recovered from Antarctic snow exhibit large organic
471 matter concentrations, so called ultracarbonaceous Antarctic micrometeorites (UCAMMs)
472 (Duprat et al., 2010; Dobrică et al., 2012; Dartois et al., 2013; Yabuta et al., 2017; Dartois et al.,
473 2018). In addition, an UCAMM-like clast was found in the LAP 02342 CR2 chondrite (Nittler et
474 al., 2019). The UCAMMs are often rich in nitrogen and deuterium (Duprat et al. 2010; Dartois et
475 al. 2013, 2018), although it is not always the case (Yabuta et al. 2017) as there must exist a
476 distribution in organic rich meteorite compositions. Unfortunately, the nitrogen abundance is not
477 known in our sample. A notable difference between UCAMMs and the Zag clast are hydrated
478 minerals. Hydrated minerals such as phyllosilicates and carbonates were not found in UCAMMs
479 (Dobrică et al., 2012; Yabuta et al., 2017). Meanwhile, the Zag clast contains abundant
480 phyllosilicates and carbonates (**Figs. 4, 5**) which require aqueous alteration. Organic matter and
481 carbonates are likely embedded in the phyllosilicate matrix (**Figs. 1, 2**). Close association of
482 organic matter and carbonate minerals implies water-organic-mineral interactions (Chan et al.,
483 2017). In the case of the Tagish Lake meteorite, which is similar to the Zag clast, carbon isotopes
484 indicate that the main carbon source of carbonates was not organic matter (Fujiya et al., 2019).
485 Thus, the carbonates and organic matter in the Zag clast could have precipitated simultaneously,

486 but from different precursors; an aqueous fluid rich in CO₂ (Fujiya et al., 2019) and some other
487 purely organic precursors (Cody et al., 2011), respectively. In this scenario, the D/H ratios of
488 water and organic precursor must have been high, i.e., close to the current δD values of the
489 diffused organic matter (~1000-1100 ‰), due to the significant D/H exchange between water
490 and organic precursors (Kebukawa et al., 2021a).

491
492 The mineralogy of the clast has mineralogical heterogeneity. Some areas have mineralogy most
493 similar to CIs. However, in some areas its mineralogy is more similar to CS IDPs and hydrated
494 fine-grained AMMs. The chemistry and isotopic signatures of the clast suggest that the
495 mineralogy of the clast is intermediate between these meteorites, IDPs, and AMMs. Usually,
496 such fragile materials cannot reach the Earth as meteorites, e.g., CIs and Tagish Lake can be
497 easily disassembled with a light poking. There are only 9 CI chondrites recorded to date, and the
498 Tagish Lake meteorite is a very rare case—the only other similar one is the Tarda meteorite
499 (Marrocchi et al., 2021). In the case of the clast, such fragile materials were gently incorporated
500 into the regolith of the ordinary chondrite parent body and protected during atmospheric entry to
501 the Earth. The observed mixture of the clast parent body within the host parent body required
502 some degree of induration of the implanted projectile. However, the fact that halite survived in
503 this clast (Rubin et al., 2002; Zolensky et al., 2017), along with organic nanoglobules, hydrous
504 phyllosilicates, and other fragile materials, requires that it was not significantly processed by
505 shock or heating during incorporation into the ordinary chondrite host. There may well have
506 originally been much more of the clast material that was significantly processed during this
507 incorporation event, and which has not survived in an intact form. Impact shock among asteroids
508 is an extremely heterogeneous process (Stöffler et al., 1991), and thus it is not surprising that
509 some material escaped significant processing.

510
511 The population of CI-like clasts is smaller than CM-like clasts in chondrite meteorite breccias
512 which may be attributable to the friable nature of CI-like clasts, as well as CI-like material is
513 destroyed preferentially during regolith gardening (Brearley and Prinz, 1992). Meanwhile,
514 Patzek et al. (2018) showed that CI-like clasts occur all the studied brecciated meteorites
515 including polymict ureilites, HEDs, CR, CB, CH, and ordinary chondrites, whereas the CM-like
516 clasts have only been identified in HEDs and ordinary chondrites. There are many hydrated,

517 relatively unshocked clasts in H-chondrite regolith breccias (Rubin and Bottke, 2009). Raman
518 spectroscopic thermometer indicated that CI-like and CM-like clasts in polymict ureilites,
519 polymict eucrites, and howardite showed that the peak temperatures experienced by most of
520 these clasts were among the unheated CI and CM chondrites, but some clasts experienced
521 slightly higher temperatures > 100 °C. Nakashima et al. (2003) reported that the peripheries of
522 the CI-like clasts in the Tsukuba H chondrite were heated to 700 °C, while the interior of the
523 clasts has never experienced heating above 500 °C, indicating frictional heating between the
524 clasts and the host minerals. The Zag clast in our study showed no evidence of shock nor
525 heating, it is probably due to the large size of the clast where the inside could be protected from
526 heating. Rubin et al. (2002) suggested that the dark clasts in Zag formed from light-colored clasts
527 during shock events that melted and mobilized a significant fraction of their metallic Fe-Ni and
528 troilite grains. However, it is not the case of the clast studied here.

529

530 Rubin and Bottke (2009) implied that a moderate amount of ejecta was produced by the shock
531 events affected H chondrites 250–325 Ma ago and 3.4–4.1 Ga ago, the earlier event was
532 consistent with Nice Model (Levison et al. 2009). One can speculate that the Zag clast was
533 incorporated to regolith of a thermal-metamorphosed planetesimal with a low velocity impact,
534 perhaps 3.4–4.1 Ga ago allowing enough time to cool down the planetesimal from ^{26}Al -induced
535 heating. Then after lithification, and possibly after destruction of the original planetesimal and
536 reaccretion, the Zag meteorite was ejected from the H chondrite parent body, perhaps 250–325
537 Ma ago. Briani et al. (2012) also suggested that xenoliths and microxenoliths could have been
538 embedded in the H chondrite parent body earlier than 3 Ga ago, from the zodiacal cloud. Large
539 size (a few cm) of the Zag clast is not consistent with the dust size range of 10-100 μm in the
540 zodiacal cloud (Grogan et al. 2001), but the size cannot completely exclude the possibility of the
541 zodiacal cloud origin.

542

543 **5. Conclusions**

544

545 We performed AFM-IR and NanoSIMS coordinated analyses in addition to STXM/C-XANES,
546 Raman, TEM, and synchrotron μFTIR on a xenolithic carbonaceous clast from the Zag ordinary
547 chondrite. The mineralogy of this clast shown by (S)TEM shares more features more in common

548 with hydrated fine-grained AMMs and CS IDPs, rather than with CI chondrites and the Tagish
549 Lake C2 ungrouped meteorite. We found two types of organics; i) deuterium-rich, C=O rich
550 likely with smaller aromatic domains, probably produced by relatively oxidized environments
551 from D-rich precursors, and ii) less deuterium-rich, aromatic-rich organics, maybe produced in
552 relatively reduced and higher temperature environments, i.e., suitable for FTT synthesis, with
553 less deuterium-enriched precursors, possibly the inner Solar System. Both types of organics have
554 various shapes and size ranges from sub- μm up to a few μm . Such mixtures of organics with
555 various origins imply a complex mixing history involving materials from various heliocentric
556 locations before accretion of the original parent body of the Zag clast. If this parent body was an
557 aqueously altered D/P-type asteroid (Kebukawa et al. 2019, 2020), our findings support the idea
558 of a wide heliocentric mixture of materials in the early Solar System (Bockelée–Morvan et al.,
559 2002; Ciesla and Dullemond, 2010; Remusat et al., 2010; Brownlee, 2014).

560

561 Notably, we demonstrated the feasibility of successful coordinated analyses with AFM-IR,
562 Raman and NanoSIMS on the very same area of the sample allowed by the sulfur-embedded
563 ultramicrotoming technique which enabled production of several consecutive thin sections from
564 the same particle. The capability to perform coupled AFM-IR and NanoSIMS analysis on the
565 very same subsample permits for the first time comparison of the isotopic signature of sub- μm
566 components identified by their IR signatures. Moreover, STXM/C-XANES was performed on
567 adjacent, the nearly-identical ultramicrotomed slices with the AFM-IR. We showed that the
568 distributions of the organic matter in AFM-IR map and STXM/XANES map were consistent, but
569 also complementary, since STXM/XANES is more sensitive to aromatics than AFM-IR but
570 cannot detect silicates. Such technical advantages are critical for the characterization of precious
571 astromaterials including asteroidal regolith samples returned to Earth by the Hayabusa2 (Yabuta
572 et al. 2022 submitted) and OSIRIS-REx spacecraft.

573

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575

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587

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