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1 **Evidence from burial sediments for prehistoric burial practice and ritual in**
2 **Monte Claro chambered tombs: micromorphology, mineralogy and**
3 **geochemistry**

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19 **Highlights**

- 20 • Signatures present in burial sediments can aid interpretation of human burials
21 • Ca depletion in sediments underlying disarticulated remains indicative of primary burial
22 • Reddish deposit identified as being ochre applied to the grave
23 • The silicified remains of a sedge torch reveal how the burial chamber was illuminated

¹ Deceased 13 May 2018 (MRU) and 26 September 2016 (DRB)

24 **Abstract**

25 The burial matrix in archaeological graves is seldom subject to detailed examination for evidence of
26 its potential interaction with the interred remains. Sediments adjacent to skeletal remains resting on
27 a marl platform in a subterranean chambered burial dated to the third millennium BC were
28 investigated using micromorphological, archaeobotanical, and chemical methods.
29 Micromorphological features in the tombs, typical of CaCO₃ depletion, appeared more abundant in
30 positions close to the skeletal remains than in positions further away, suggesting that the carbonate
31 dissolution was caused by acidification resulting from the body decay as well as from environmental
32 factors. Organic signatures around the remains were dominated by background sedimentary organic
33 matter of the marl, attesting to the exposed style of burial being unfavourable for the preservation of
34 organic remains. Red concentrates present in a distinct region of the resting platform were
35 distinguishable from similar coloured deposits formed by debris from the vault. They were identified
36 as haematite (ochre) deposits, matching features observed in other prehistoric sites, including those
37 of the same culture, and attesting to ritual associated with the treatment of the dead. Greyish fibrous
38 contexts found on the burial resting platforms were identified as silicified sedge, probably remnants
39 of the combustible parts of torches. The position and nature of the sedge-derived material suggests
40 their use for illuminating the graves at depth and/or as a possible votive ritual. The detailed scientific
41 examination of the burial matrix can reveal unique perspectives and add interpretative value to the
42 archaeological investigation of graves.

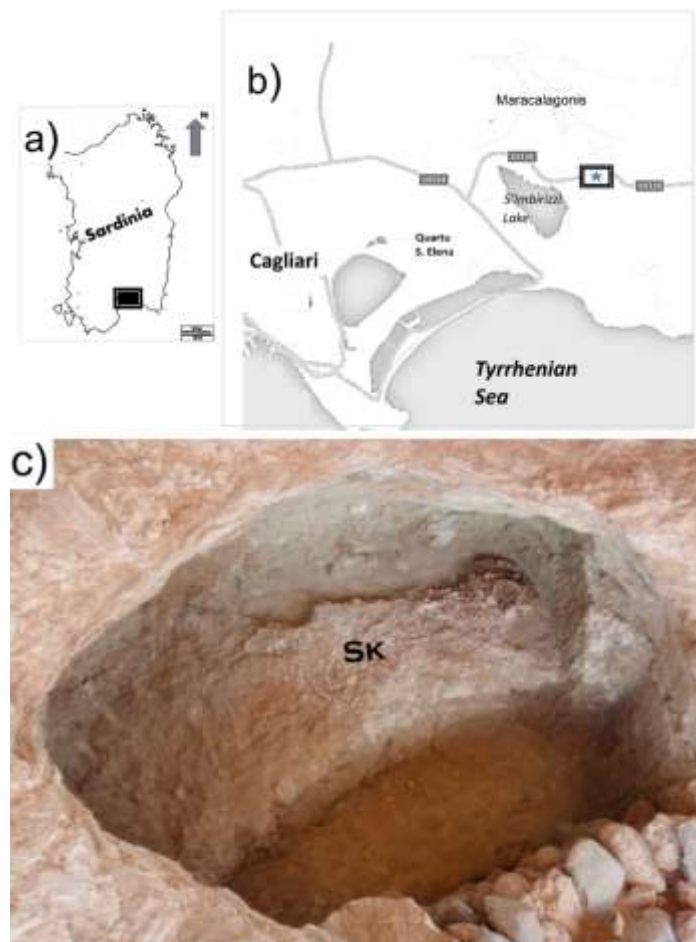
43 **Keywords:** Eneolithic chambered burials; geoarchaeology and micromorphology; organic
44 geochemistry; ochre; ritual burning.

45 **1 Introduction and site description**

46 The InterArChive project considered the extent to which the burial matrix preserves evidence that is
47 currently unexploited within the archaeological investigation of human burials (Usai et al. 2014).
48 Soils/sediments originating from a diverse range of temporal and geographical contexts were
49 examined using systematic sampling and a multidisciplinary analytical approach (Burns et al. 2017;
50 Pickering et al. 2014, 2018; Usai et al. 2014). One of the sites examined, Ganni, Sardinia, belongs to
51 the Eneolithic Culture of Monte Claro, a culture that displays intriguing signs of ritual burial practices
52 (Manunza 2010a, 2010b; Ugas 1993). The culture was named after discovery, in 1906, of an Eneolithic
53 burial in the Monte Claro area within the city of Cagliari. The burial contained a stone bench as a
54 resting platform for the dead, and various grave goods including a situlate vessel and a *monoansato*
55 (single handled) bowl. In this, and other Monte Claro tombs discovered later, the primary deposition
56 of the dead was in a crouched position on their left side with features ascribed to ochre in some of

57 the graves. The practice of adorning ancient graves with ochre has been described for archaeological
58 sites as far back as Palaeolithic age (Roebroeks et al. 2012), the deep red colouration in burials from
59 various sites and periods being attributed to haematite (Gialanella et al. 2011; Roebroeks et al. 2012).

60 Two underground chambered Copper Age burials of the Eneolithic Culture of Monte Claro, at the
61 archaeological site of Ganni (Fig. 1a, b), presented a unique opportunity to examine the evidence for
62 ritual burial practices within this culture. The vaults, carved into the massive Miocene bedrock
63 (Carmignani et al. 2012) using prehistoric implements (Fig. 2a), extend to c. 3 m underground. Tomb
64 I contained two burial chambers (T1 and T2) whereas Tomb II was constructed with a small entrance
65 vestibule connected to a single burial chamber (Fig. 2b). Tomb II contained human skeletal remains
66 radiocarbon dated to 2469-2293 BC (Manunza 2013). Tomb II and chamber T2 of Tomb I were found
67 fully sealed and unviolated, and hence with all architectural elements in place. The archaeological
68 features of the tombs and their cultural implications have been described by Manunza (2013).



69

70 Fig. 1. a) Location of the site in Sardinia (black rectangle); b) inset corresponding to the black rectangle
71 in a): site location is indicated by the star; c) close-up of the burial chamber of Tomb II with position
72 of skeletal remains indicated by the letters Sk.

73 The Gannì graves feature sub-vertical access tunnels leading from the land surface to underground
74 burial chambers delimited by small stone walls and stone benches employed as resting platforms for
75 the dead (Fig. 1c). Such architecture was frequent in Monte Claro burials in Sardinia and also among
76 coeval cultural groups of Aegean-Anatolian background in central-southern Italy. The former include
77 sites some distance from Gannì as well as others that are closer to the site (Table S1).

78 At the time of sampling, the skeletal remains in Chamber T1 of Tomb I at Gannì were disarticulated,
79 having been disturbed by thieves. Prior to the disturbance, Chamber T1 contained disarticulated and
80 damaged bone as well as the skeletons of a male and a female aged between 18 and 25 years and two
81 males aged 30-45 and 20-30 years resting on stone benches. In the second, unviolated chamber, T2,
82 the remains of two children rested in a niche in the wall and, directly on the floor below, were the
83 skeletons of a male and female couple (aged 18-25 years) next to a child aged 9-10 years. Tomb II,
84 fully sealed and unviolated, contained poorly-preserved skeletal remains of an adult female aged 35-
85 50 years in anatomical position, lying crouched on the left side and resting on a bench carved in the
86 marl (Fig. 3). Archaeological interpretation suggested that the burials date to the Monte Claro culture
87 (Manunza 2013). The absence of bone lesions excluded interpersonal violence as causes of death,
88 suggesting a local epidemic or, more likely given the patterns of articulation, that these were sites of
89 collective burials (Manunza 2013).

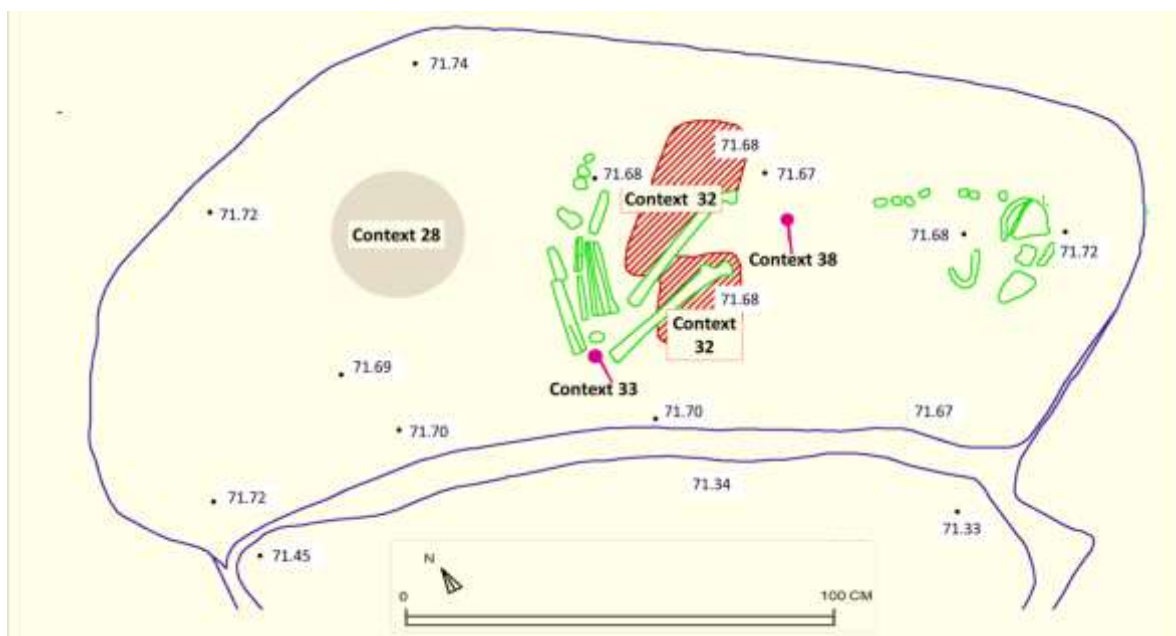


90

91 Fig. 2. a) Internal vault of Tomb II carved with prehistoric implements during the Copper Age; b) front
92 vestibule of Tomb II connecting to burial chamber through a small stone wall (see Fig. 1c for close-up).

93 The positioning of individuals either on stone-carved benches or in direct contact with the floor at
94 Gannì, and also in the Cagliari, Selargius, Settimo and Serdiana sites, has been interpreted to relate to
95 social status – tombs where additional efforts were expended to fashion stone benches were
96 postulated to have been destined for people of higher status (Manunza 2013). An emergent group of
97 high status within the local community was also suggested by the exceptional quality of some of the

108 pottery present in Tomb I (Manunza 2013). This included bowls decorated by incisions, plates, vases
109 with polished surfaces, cups, beakers, situlate vessels and fragments of vases. The pottery was similar
110 to that found at many other Monte Claro sites, the *situlae* being very similar to those found in sites
111 South of the S'Imbirizzi Lake and in Cagliari. Vessels and *monoansato* bowls from Gannì were also very
112 similar to those of the first Monte Claro burial excavated in Cagliari, particularly in the shape of the
113 rims and identical decoration of the bowls. Other fragments of ceramics had identical decorations to
114 those of a site near Villasor. A stone axe, three-pointed stone tool and a pointed copper implement
115 were also found. The grave goods were all from Tomb I, no grave goods were present in Tomb II, and
116 their features were not sufficient to determine whether they were part of any particular burial practice
117 or ritual at the site.



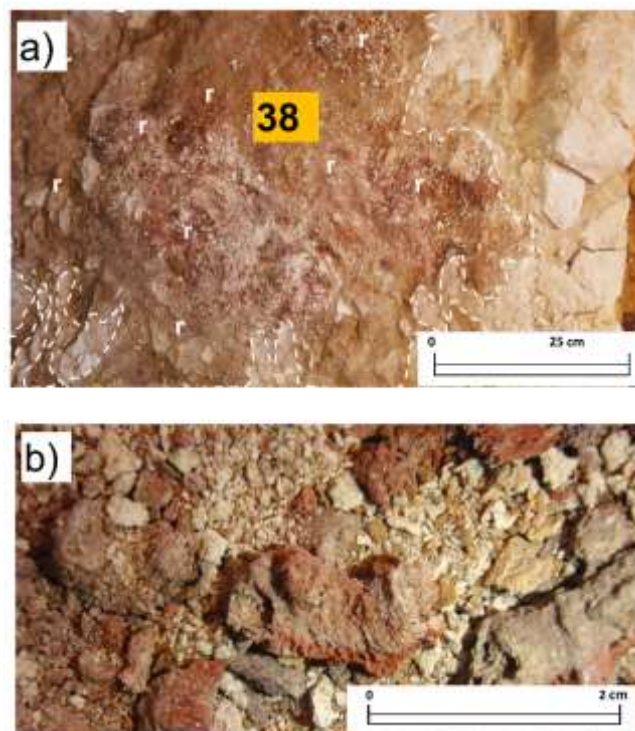
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109 Fig. 3. Plan of Tomb II resting platform, with context and sample distribution (notations in Table S2;
110 height expressed in metres above sea level). The skeletal remains (green in colour image) were
111 orientated NW-SE with the skull to the south east.

112 The vault marl, of dominant colour in the yellow-red range (2.5YR8-7/1-2) (Munsell 2009), contained
113 variable amounts of orange-coloured deposition nodules. Much of the bone in Tomb II had been lost
114 due to decay and the skeletal remains and the platform on which they rested (Fig. 3) were covered in
115 places by sparse sediment deposits, tentatively interpreted on site as debris fallen from the vault. The
116 debris displayed several colours in the yellow-red range (2.5YR7-6/3-4) (Munsell 2009) and rare fine
117 bio-voids and cracks were visible to the naked eye. Fine rock fragments and poorly developed soil
118 aggregates (peds), barely visible at field scale, suggested a low degree of pedogenesis. Two visually

119 distinct areas of the resting platform, one red-coloured (Context 32) and the other grey (Context 28),
120 were evident (Fig. 3).

121 *1.1 Deep red concentrates*

122 Concentrates of very deep red colour, varying in the range 2.5YR 8-5/3-4 (Munsell 2009), were situated
123 in the vicinity of the pelvis and thighs of the skeleton in Tomb II (Context 32; Fig. 3). The material
124 appeared as a covering over part of the resting platform and as deposits with sharp or diffuse
125 boundaries over bone fragments and the local rock/sediment components (Fig. 4). Preliminarily on-
126 site interpretation was that the material could be ochre (Manunza 2013), as observed in other ancient
127 burials (Roebroeks et al. 2012).



128
129 Fig. 4. a) Photograph of Context 32, comprising a deep red material (area marked by 'r' and enclosed
130 by white outline); b) detail of the red material showing the intense red feature indicated with 'r' in a).

131 *1.2 Fibrous grey contexts*

132 Loose grey material was found near the presumed position of the feet in Tomb II (Context 28 in Fig.
133 3), forming a thin, irregular, sub-circular lens over the resting platform (Fig. S1). A visually similar
134 material was present in a clump to the side of and between the two burial platforms in Chamber T1
135 of Tomb I (Context 13). Visual inspection revealed the material to include fine fibrous filaments; its
136 nature and possible ritual implications was unclear from field and macro-scale observations.

137 The nature, chemical and mineralogical compositions of two coloured pedofeatures in Context 32 and
138 of the grey fibrous material of Contexts 13 and 28 were examined to determine their compositions
139 and significance in relation to the grave sediments, burial practices and effects of body decomposition
140 on the burial matrices that had been adjacent to and beneath the skeletal remains.

141 **2 Methods**

142 *2.1 Sampling*

143 The sampling protocols for the InterArChive project (Usai et al. 2014) were followed as closely as
144 possible given the limitations of the incomplete nature of the skeletal remains (Fig. 3, Table S2).
145 Undisturbed sediment samples were collected with Kubiena tins or hand lifting, each with a loose
146 replicate. Site controls were collected from the marl surrounding the grave: from the sides, C1(α), and
147 from a marl bed equivalent to the resting platform from the vault in Tomb II, C1(β) (Carmignani et al.
148 2012). Control C4 was collected from the thin layer deposited on top of the skeletal remains in Tomb
149 II. The sediments from the skull and pelvic regions were collected from the upper part of the resting
150 platform under the skeletal remains. Loose sediment samples for organic chemical analysis were
151 collected from numerous positions around the skeletal remains (Table S2) and from the red and grey
152 contexts. Thin sections were analysed from the block-lifted undisturbed samples. They were
153 examined by light microscopy and scanning electron microscopy-energy dispersive X-ray spectroscopy
154 (SEM-EDS). Loose sediment samples were dried, sieved and aliquots of selected samples subjected to
155 X-ray diffraction (XRD). Aliquots were also subjected to total organic carbon analysis and extraction
156 with organic solvent to recover organic residues. The organic residues were derivatised and analysed
157 by gas chromatography-mass spectrometry (GC-MS). Full experimental details are given in SI.

158 **3 Results and discussion**

159 *3.1 Micromorphology and inorganic chemistry*

160 *3.1.1 Grave and site controls*

161 The material overlying the skeleton (Control C4) was visually and compositionally similar to the marl
162 of the vault, though with some evidence of pedogenesis, consistent with debris originating from the
163 chamber vault rather than being from allochthonous material that was intentionally deposited on the
164 skeleton. Thus, the C1 controls, representing the marl from which the vault was excavated, exhibited
165 largely uniform groundmass with c. 50% grey and crystallitic fine material ($\leq 2 \mu\text{m}$) and a coarser
166 fraction (mainly $\leq 50 \mu\text{m}$ grains but occasionally as grains and composite silt agglomerates of up to
167 $200 \mu\text{m}$). The orange-coloured nodules observed on-site in the grave vault were not observed in the
168 thin sections from C1(β), probably as a result of local variability. Voids in the controls were mostly
169 planar, randomly oriented and typical of rock fracturing. Quartz and calcite were both very well

170 represented in XRD analysis of the vault control C1(β) (Fig. S2). Control C4 showed evidence of
171 pedogenesis through the presence of 2 - 200 μm granular peds (80% of slide area), sub-angular blocky
172 peds and small randomly orientated biovoids. Orange-coloured nodules observed in thin section
173 reflect material derived from the vault.

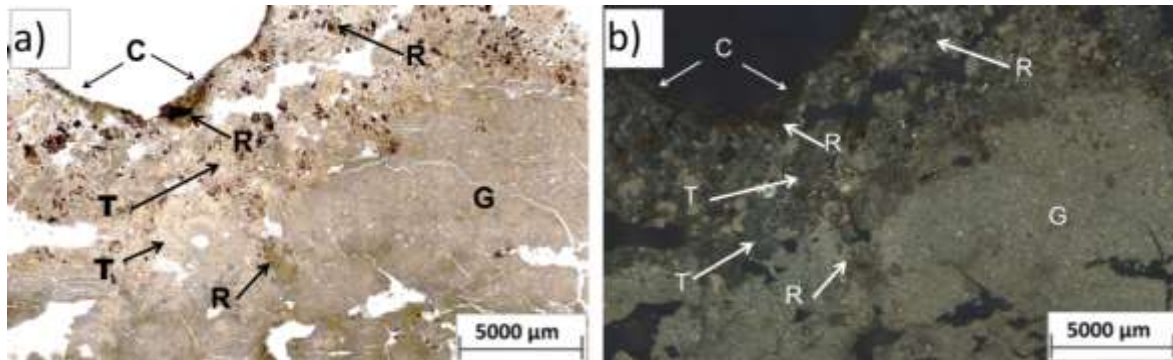
174 3.1.2 Debris on resting plane (Contexts 32, 37 and 38)

175 The samples from the resting platform, while exhibiting compositional features consistent with
176 derivation from the marl, showed limited evidence of pedogenesis and alteration. Thus, within the
177 upper *c.* 10 mm the deposits under the pelvis (Context 38) and the red area adjacent to it (Context
178 32), rare, very small and poorly developed peds were observed (Fig. S3). Voids with sub-parallel
179 orientation to the resting platform and cloudy crystallitic *b*-fabric only in the regions under the skull
180 (Context 37) and pelvis (Context 38) reflect pedogenic processes in areas contiguous to the skeletal
181 remains, *i.e.* in the upper *c.* 10 mm of areas of the resting platform below the skeleton.

182 3.1.2.1 Pedofeatures T and R

183 The debris coating the upper 10 mm of the surface of the resting platform contained two distinct
184 pedofeatures, designated T and R. The debris was disturbed beneath the thigh (Context 32) and pelvic
185 regions (Context 38). Pedofeature T was compositionally similar in both areas and dominated the
186 samples. Its chemical composition and anatomical location are consistent with decalcified marl
187 formed by leaching due to generation of acidic fluids during decomposition of the body. Specifically,
188 pedofeature T, beige (PPL) and frequently isotropic or dark grey in XPL exhibited lower crystallinity
189 than the groundmass (Fig. 5). Abundance levels were $\geq 50\%$ of the thin section areas throughout the
190 upper 8 mm of the resting platform, with a sharp decrease to almost 0% at 8 mm depth but
191 occasionally extending further down to 20 mm depth (Fig. 5). SEM-EDS revealed that pedofeature T
192 in the thin sections from Contexts 32 and 38 showed no significant difference in elemental
193 composition, suggesting similar origins or processes of formation in both contexts. Pedofeature T was
194 dominated by Si and Al together with Fe, K, and trace amounts of Na, Mg, P and S (Table S3). The Fe
195 contents were higher, and Ca substantially lower, than in the adjacent grey matrix in the thin section
196 and in the C1(β) control (Table S3). XRD analysis indicated that Context 38 exhibited significantly
197 depleted calcite levels relative to the marl of the vault. Given that the micromorphology and depth
198 profiles of pedofeature T suggest that it formed as an alteration product rather than as a surface
199 deposit, the chemical composition was compared with that of the unaltered grey fabric of the resting
200 platform (Table S3). As with the vault control, the grey fabric was dominated by Ca, the levels being
201 much higher and Si, Al and Fe being lower than in pedofeature T (Table S3). To account for
202 decalcification, the data were normalised to the Si content, enabling direct comparison of pedofeature

203 T and the grey matrix (Fig. 6). The significantly lower Ca and Mg contents and slightly higher, but
204 statistically significant, Fe content is consistent with pedofeature T being formed by depletion of the
205 grey matrix. The prevalence of pedofeature T in the deposit directly under the pelvis hints at the
206 depletion being caused by generation of acidic fluids during decomposition of the body, consistent
207 also with the lower prevalence under the skull.



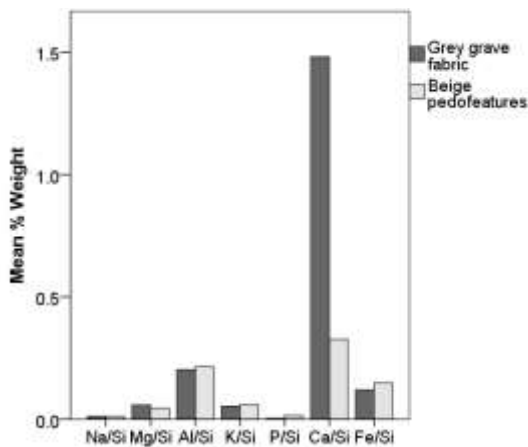
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209 Fig. 5. Mosaic image comprising all adjacent fields of view from the thin section from Context 32: a)
210 PPL, pedofeature R comprises deep to very light red opaque material and b) XPL. Pedofeature T
211 appears black or very dark. The bulk of pedofeature T appears as a disturbed zone of lower crystallinity
212 situated under the sub-horizontal resting plane (toward the upper part of the image). A small cavity
213 within the sub-horizontal resting plane is denoted C. The grey matrix (G) appears mostly in the
214 lowermost part of the section (as also observed in Context 38).

215 Pedofeature R, deep to very light red opaque material (Figs 3 and S4), was generally adjacent to
216 pedofeature T (Fig. 5). The abundance levels dropped sharply from c. 10% area in the uppermost 7-
217 11 mm of the resting plane in Context 32 to only occasional occurrences below 7-11 mm depth.
218 Context 38 contained a few fragments of pedofeature R below the pelvic bone. The material differs
219 chemically from the marl of the vault and was identified as haematite, consistent with ochre deposits
220 added to the surface of the resting plane. Specifically, the material of the reddened area of the resting
221 platform (Context 32) contained isolated micro-patches of Fe oxides of variable colour intensity in thin
222 section (Fig. S4). The accumulations (ranging from 10 to 150 µm) were interpreted as weakly to
223 strongly impregnated nodules and occasionally as fragmented typic- or hypo-coatings.

224 Pedofeature R in Context 32 exhibited an appreciable content of silica as revealed by SEM-EDS analysis
225 (Si = 10.2%) and the Fe contents of the red particles were highly variable (Table S3). The low
226 concentrations of Al, Ca and Mg suggest relatively low levels of aluminosilicate and carbonate
227 components by comparison with the vault control and grey matrix in the undisturbed samples from

228 the resting platform (Table S3). XRD analysis indicated these deep red components to be composed
 229 mainly of haematite, consistent with ochre deposits: goethite was not detected in any of the samples
 230 (Fig. S2). The similar levels of quartz and calcite in the host materials (Fig. S2) suggest similar mineral
 231 types, sizes and orientations. Hence, it is likely that the intensity of the secondary XRD radiation
 232 correlates with differences in amounts of mineral species present. Thus, the depth distribution,
 233 disturbed nature, chemistry and morphology of the pedofeature is consistent with an allochthonous
 234 material deposited on the surface of the resting platform and incorporated into the disturbed/debris
 235 layer to a depth of c. 11 mm.



	<i>p</i> -value (ANOVA) ²³⁷
Ca/Si	< 0.000*
Fe/Si	0.037*
P/Si	0.348
K/Si	0.218
Al/Si	0.396
Mg/Si	0.008*

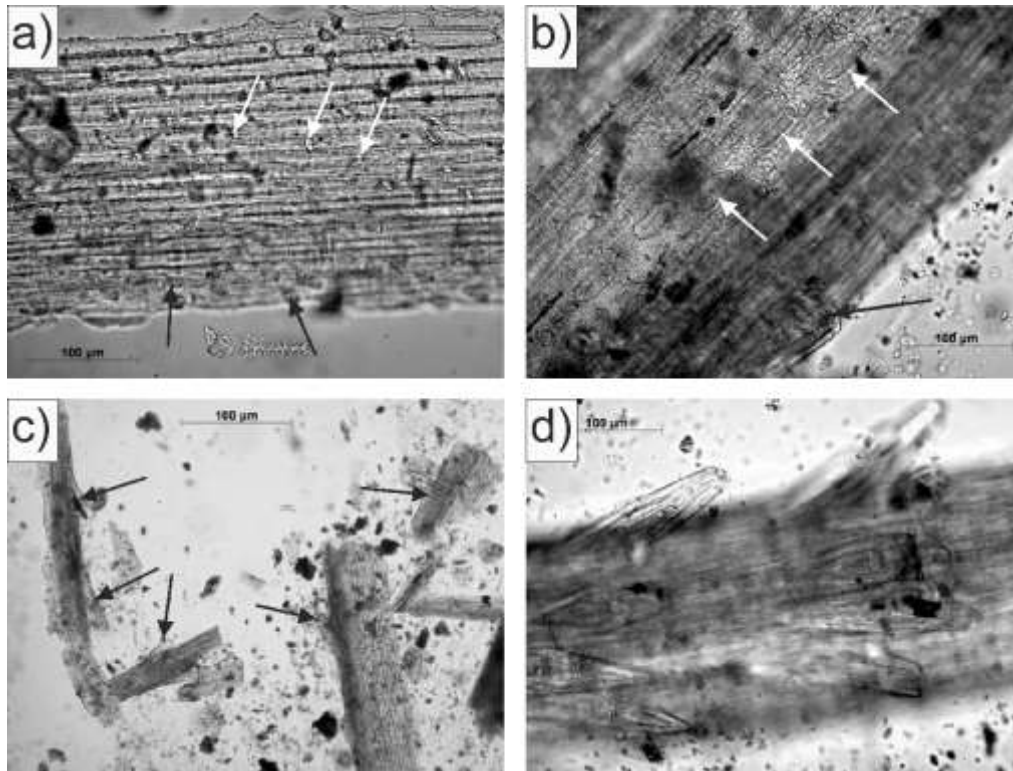
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238 Fig. 6: Comparison of relative element abundances in beige pedofeature T and grey grave *b*-fabric.
 239 Significance threshold = 0.05.

240 3.2 Grey fibrous material (Contexts 13 and 28)

241 Microscopy revealed the dominant material of the grey fibrous deposit of Context 13 to contain
 242 recognisable remains of plant material. When concentrated by acid dissolution of carbonate,
 243 characteristic features of Poales (grasses and sedges) could be recognised. The plant remains were
 244 preserved mainly as silica, consistent with the material having been burnt *in situ*. Specifically, under
 245 low-power microscopy, the grey fibrous material of Context 13 appeared to include two components:
 246 a minor one comprising tubular structures (c. 15 mm × 2 mm), possibly parts of recent roots, and a
 247 major component comprising finely divided amorphous granules and powdery material. The latter,
 248 also present in Context 28, contained bright white spicules with maximum dimension up to about 4 ×
 249 0.5 mm but mostly about 2 × 0.25 mm. Treatment of unconsolidated replicates of Context 13 with
 250 hydrochloric acid revealed that the granules and powder were highly calcareous whereas the spicules
 251 remained undissolved. At ×400 magnification the spicules were recognisable as fragments of plant
 252 epidermis in the form of silica skeletons, where the original cell walls had been replaced by silica. Most
 253 specimens bore typical epidermal features of Poales (grasses and sedges) in the form of long and short
 254 cells, the former with characteristic finely sinuous walls (Fig. 7a). Many fragments bore small ‘prickles’

255 (micro-hairs) on their surfaces (Fig. 7a) and some bore more obvious and quite substantial marginal
256 teeth (Fig. 7b, c). Some fragments bore rows of stomata exhibiting the morphological characteristics
257 (elongated dumbbell shaped structures) associated with grasses and sedges (Fig. 7b). In other cases
258 the fragments appeared to be more or less cylindrical, with prominent teeth (Fig. 7d), and were
259 presumably from a bristle or awn from the tip of a leaf or from a spikelet within a flowering head.



260

261 Fig. 7. Optical microscope ($\times 400$ magnification) images of silicified plant remains from Context 13.
262 Epidermis with a) long cells, short cells (white arrows) and prickles (black arrows). The single 'spiny'
263 structure at the bottom centre is a detached epidermal cell; b) marginal tooth (black arrow) and a row
264 of stomata (white arrows); c) sharp marginal teeth indicated by arrows; d) awn fragment with whorls
265 of teeth.

266 Further identification of the grey fibrous material was not possible in the absence of relevant
267 reference material though, on the basis of the tissue anatomy observed, only one or two taxa are likely
268 candidates. The size is rather small for one of the major cultivated grasses (*i.e.* cereals). Preservation
269 by silicification² has been observed occasionally in archaeological occupation deposits where it was
270 inferred that large quantities of silica-rich plant material had been burnt under strongly oxidizing

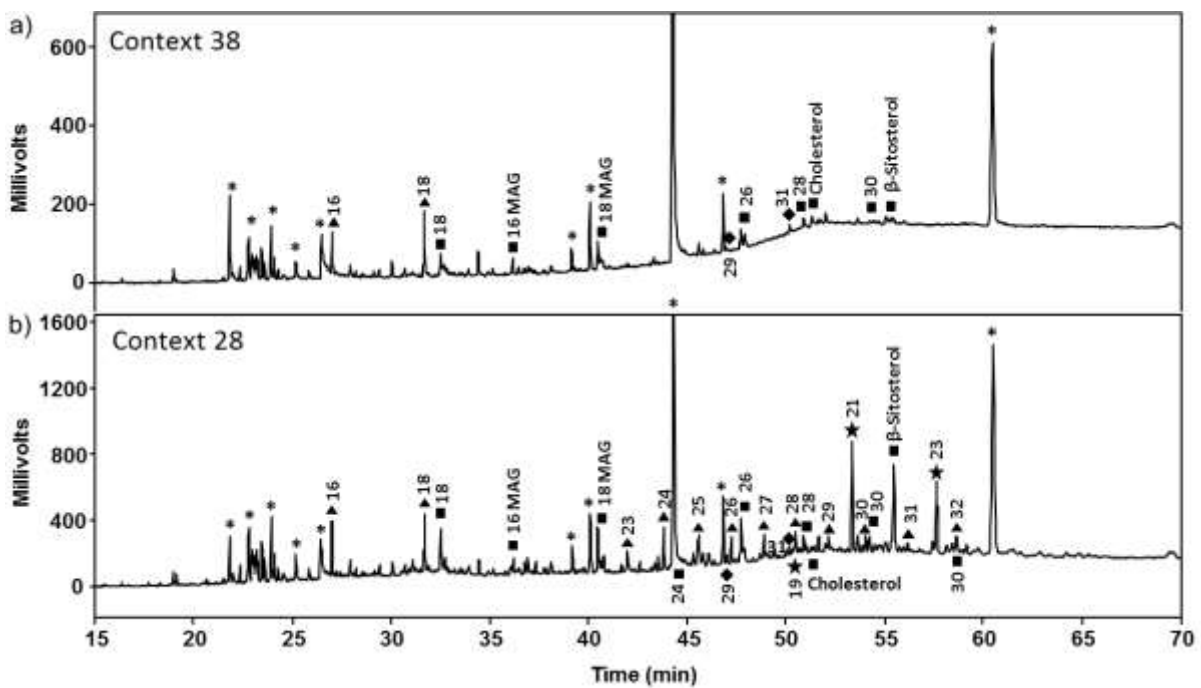
² Silicification is a term used in palaeobotany to denote permineralisation of tissues by silica within an aqueous burial environment, and this differs as a mechanism from what is presumed to have occurred in these archaeological specimens.

271 conditions, generating temperatures sufficient for the silica in the tissues to be fused, but low enough
272 to prevent loss of cell structure (Robinson and Straker 1991). Such silicified material was attributed
273 to rapid accumulation or burial of plant materials, protecting the delicate cellular structure from
274 mechanical damage (Hall and Huntley 2007). Material of this kind in several prehistoric and medieval
275 sites in England represented concentrations of cereal chaff (Robinson and Straker 1991) and, in several
276 sites in Northern England, material other than cereal chaff was sometimes recorded (Hall and Huntley
277 2007). SEM-EDS analysis of the fibrous material in Context 28 confirms its siliceous nature (Table S3),
278 consistent with silicified plant epidermis as suggested from palaeobotany. The XRD analysis indicated
279 that calcite and quartz were scarce in the grey fibrous material of Context 28, contrasting with the
280 high level of Si detected by SEM-EDS (Table S3) and calcareous component indicated by dissolution
281 with acid. The apparent discrepancies are consistent with dominance of poorly ordered carbonate
282 and silica (Drees et al. 1992) formed by a process, such as burning, that did not induce crystallisation.
283 Hence, the components of Contexts 13 and 28 represent remains of leaf and flower parts from non-
284 cultivated herbaceous materials that underwent silicification induced by fire. Given the delicate
285 nature of the material and its form, the mineralisation must have occurred *in situ*.

286 3.3 Organic chemical analysis

287 With the exception of the grey material (Context 28) the organic carbon contents of the sediment
288 samples from around the skeletal remains were not distinguishable from those of the controls
289 suggesting there to be no significant contribution from the human remains. The absence of organic
290 material associated with the coloured pedofeatures in Context 32 is consistent with its colour being
291 due to haematite rather than to an organic dye. By contrast, Context 28 contained molecular
292 signatures consistent with an origin from sedge. Specifically, total organic carbon (TOC) analysis
293 revealed the majority of sediment samples from around the skeletal remains to be very similar to the
294 controls, containing low levels of organic carbon (TOC values \ll 1%; Table S4) and with the majority
295 of carbon being in the form of inorganic carbonate. By contrast, the grey material (Context 28)
296 exhibited the highest TOC content (0.77%). GC-MS analysis of the total solvent extractable organic
297 matter revealed the samples to contain organic compounds including C_{16:0} and C_{18:0} fatty acids,
298 monoacylglycerols (MAGs), *n*-alkanols (C_{18:0} and C₂₄-C₃₀), *n*-alkanes (C₂₉ and C₃₁), β -sitosterol and
299 cholesterol (Fig. 8), all present in low concentrations, consistent with the low organic matter contents
300 indicated by elemental analysis. For the most part, little variation was evident between the lipid
301 profiles of the controls (e.g. C1(β)) and samples taken from around the skeletal remains (e.g. Fig. 8a)
302 suggesting they contain predominantly background sediment organic matter with little contribution
303 from the human remains.

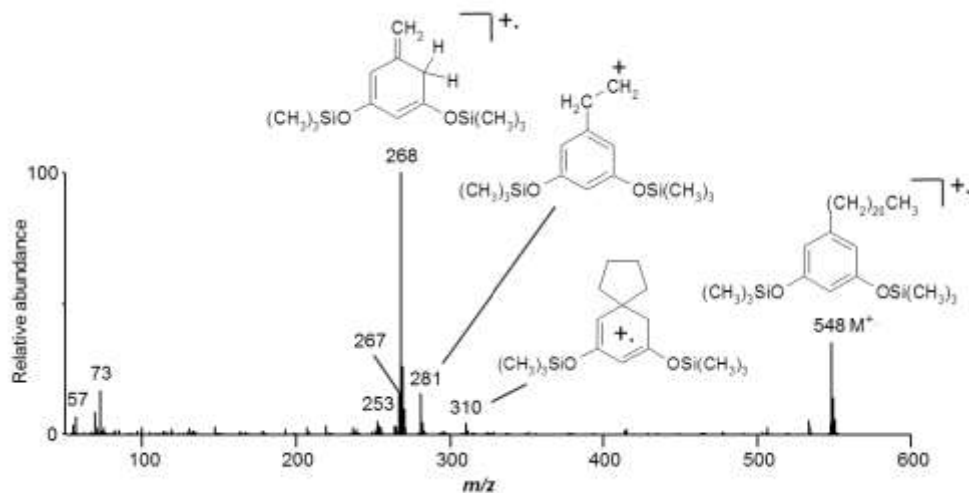
304 The extract from the grey deposit (Context 28) contained appreciable amounts of C₂₂-C₃₂ *n*-alkanoic
 305 acids, C₂₄-C₃₀ *n*-alkanols and β-sitosterol (Fig. 8b) together representing 6136 ng/g sediment, *cf.* 383
 306 ng/g sediment for C1(β). The higher levels of those components in the grey deposit is indicative of a
 307 significant higher plant input (Eglinton and Hamilton 1967; Jambu et al. 1993). In addition, the profile
 308 includes two prominent peaks at retention times (*t_R*) 53.5 and 57.5 min (Fig. 8b), which do not feature
 309 in the other extracts and are comparable in abundance to sitosterol. GC-MS revealed their molecular
 310 ions (M⁺) at *m/z* 548 and 576 and the same principal fragment ions: *m/z* 268 (base peak) accompanied
 311 by low abundance ions at *m/z* 267, 281 and 310 (e.g. Fig. 9). The EI mass spectra are characteristic of
 312 long chain alkylresorcinols (Avsejs et al. 2002). The ratio of *m/z* 268:267 of approximately 5:1 is
 313 consistent with the attachment of the alkyl chain at position 5 on the aromatic ring (Zarnowski and
 314 Kozubek 1999). Accordingly, the two components were assigned as 5-*n*-alkylresorcinols having C₂₁
 315 and C₂₃ alkyl chains, respectively. The presence of a minor C₁₉ alkyl homologue (M⁺ = *m/z* 520) at *t_R* =
 316 50.5 min (Fig. 8b) was revealed in the *m/z* 268 extracted ion chromatogram.



317

318 Fig. 8. Partial FID gas chromatograms of the total lipid extracts from a) the abdomen, Context 38, and
 319 b) the grey material, Context 28. Triangles = *n*-alkanoic acids, squares = alcohols (mainly *n*-alkanols
 320 unless labelled otherwise), diamonds = *n*-alkanes, stars = 5-*n*-alkylresorcinols, MAG =
 321 monoacylglycerols. Numbers denote carbon chain length. * = plasticisers arising from plastic bags
 322 used for sample storage.

323 Previous reports of 5-*n*-alkylresorcinols possessing alkyl chains in the range C₁₃-C₂₇ and exhibiting a
 324 prevalence of odd-chain members indicate a range of natural sources including higher plants, algae,
 325 fungi and bacteria (Kozubek and Tyman 1999). Their biological function is largely uncertain although
 326 they are believed to fulfil roles including microbial resistance, growth regulation and membrane
 327 modulation (Kozubek and Tyman 1999). Cereals such as wheat and rye contain substantial amounts
 328 of alkylresorcinols. Previous studies, involving pollen analysis or examination of the contents of burial
 329 pots, have indicated foodstuffs, including cereals, as occasional items placed in human burials (Lagerås
 330 2000). The 5-*n*-alkylresorcinol distributions of cereals, typically C₁₇-C₂₅ and dominated by the C₁₉ and
 331 C₂₁ components (Chen et al. 2004; Mattila et al. 2005; Ross et al. 2003), do not match that of the grey
 332 context, whereas dominance of the C₂₁ and C₂₃ 5-*n*-alkylresorcinol homologues for the sedge
 333 *Rhynchospora alba* (Avsejs et al. 2002) represents a close match. The resorcinols and silicified remains
 334 indicate that the grey material associated with the burial is plant-derived, consistent with the visual
 335 description of a fibrous plant like material overlying the area from which the sample was collected.
 336 The survival of such clear indicators of plant remains after 4500 years of burial is remarkable, especially
 337 given their exposure to air and to combustion. The apparent recalcitrance of the 5-*n*-alkylresorcinols
 338 in that material could be due in part to their antimicrobial properties and may have extended
 339 protection to other biomarkers such as sitosterol.



340
 341 Fig. 9. Electron ionisation mass spectrum (70 eV) of C₂₁ 5-*n*-alkylresorcinol.

342 As with the samples in close association with the skeletal remains, the total lipid extract from the red
 343 material (Context 32) is extremely similar to those of the controls. The solvent extract itself was
 344 colourless and no components which would explain the red appearance of the sediment sample could
 345 be identified by GC-MS. In order to rule out the possibility of the substance responsible for the colour

346 of the sample being physically or chemically bound to sediment matrix, for example via ester linkages
347 between acid residues and free hydroxyl groups present on mineral surfaces, the sediment sample
348 was re-extracted with methanolic sulfuric acid which can disrupt such associations and release the
349 bound molecules as methyl esters (Huseby and Ocampo 1997). The acid extraction did not produce a
350 coloured extract. Thus, on the basis of its inconspicuous extractable lipid profile it appears that the
351 red material owes its colour to an inorganic mineral colourant rather than an organic chromophore.

352 **4 Summary and conclusions**

353 Analysis of the sediments from the tomb provided intriguing information relating to the burial practice
354 and ritual of the Monte Claro culture burial at Gannì. Furthermore, the impact of body decay on the
355 matrix adjacent to the human remains was evident through alteration of the sediments.

356 *4.1 Pedogenesis associated with the human remains*

357 Consistent with the tentative on-site archaeological interpretation, the lithified debris lying on the
358 bone surface is attributed to material that had fallen from the vault of the chamber rather than being
359 brought from elsewhere and lain on the skeleton for ritual or other purposes. Pedogenesis was
360 pronounced only in the material that directly interacted with the organic matter from the body, both
361 in the debris covering the remains and within the upper c. 10 mm of the resting plane under the
362 skeletal remains. The similar organic carbon contents and compositions of these samples and the marl
363 indicates that the organic components of the body tissues have degraded to below detectable limits
364 within the burial chamber where the remains were exposed to the air. Thus, despite the complete
365 degradation of the organic remains the pedogenic alteration of the sediment adjacent to the remains
366 provides evidence of *in situ* decomposition of the body.

367 *4.2 Calcium carbonate depletion from body decomposition*

368 Pedofeature T, in the thigh and pelvic regions and in Context 32 containing the red concentrates, is a
369 depletion pedofeature characterized by a significant loss of CaCO₃, small decrease in Mg and
370 consequent relative increase in Fe levels compared with the surrounding grey matrix. Although
371 sporadic patches of carbonate depletion occur in most samples, depletion features were considerably
372 more abundant beneath the thigh and pelvic regions reaching ≥ 50% area within the uppermost 10
373 mm of the resting platform. The decalcification is attributed to localised acidification resulting from
374 the generation of organic acids during body decomposition (Dent et al. 2004). Subject to the sediment
375 matrix containing carbonates, such evidence could be of value in recognising primary burials in cases
376 where skeletal articulation can not be established.

377 4.3 *Ochre coloured context (Context 32)*

378 The red haematite concentrates in Context 32, representing c. 10% by area of the disturbed/debris
379 layer at the surface of the resting platform, were mixed with deposits that otherwise have very similar
380 chemical compositions (with the exception of P and S) to the marl of the vault. The low organic matter
381 content confirms the inorganic nature of Context 32. The extreme Fe concentrations in some regions,
382 and high variability, reflect the heterogeneity observed by micromorphology. The Fe concentrations
383 are well within the range recorded elsewhere for ochre type deposits (Attard Montalto et al. 2012;
384 Gialanella et al. 2011; Iriarte et al. 2009; Popelka-Filcoff et al. 2008; Ramos et al. 2008). The
385 heterogeneous mixture of haematite, silicates, clays and carbonates is also consistent with the
386 previous analyses of archaeological ochre pigments (Darchuk et al. 2010; Gialanella et al. 2011). At
387 other sites in Sardinia (Monte Claro culture: Sibiola, Su Fraigu (S. Sperate) and Sa Costa) and a Neolithic
388 site at Ozieri (Manunza 2010a, 2010b) ochre was used for grave paintings and to adorn the dead. At
389 Sibiola (16 km N of Ganni), ochre was used for single inhumations of elderly women, in tombs of
390 identical architecture to those at Ganni. Thus, the identification of ochre deposits at Ganni, applied
391 to a specific area of the grave as part of the burial ritual, complements and extends previous
392 interpretations.

393 4.4 *Grey fibrous material: torches from sedge*

394 The grey fibrous material of the thin sub-circular lens of Tomb II and the clump to the side of the
395 skeleton in Tomb I contained plant remains from the leaf and flower parts of non-cultivated
396 herbaceous materials of the order Poales. Though almost entirely silicified, the material showed
397 poorly ordered crystal form. The grey material from Tomb II (Context 28) contained plant-derived
398 lipid components including a series of 5-*n*-alkylresorcinols. This supports the evidence from
399 archaeobotanical analysis that the deposit relates to degraded plant material and suggests that it was
400 deliberately placed in the tomb, probably as part of the burial ritual. In combination with the
401 archaeobotanical evidence, the 5-*n*-alkylresorcinols provide compelling evidence for the material
402 being from sedge. Analogous to elsewhere, it is suggested that silicification was induced during
403 combustion. The rapid accumulation of the material *in situ* strongly suggests it was burnt in the burial
404 chamber, though combustion was incomplete enabling some organic signatures to preserve. Given
405 that the only access to the tombs at the time of burial was through a c. 3 m long vertical tunnel, it is
406 likely that the grey fibrous materials derive from remains of torches fabricated from sedge, incomplete
407 combustion possibly being associated with a tightly bound area. No other comparable contexts or
408 other evidence for the use of torches exist in burials of the Monte Claro culture, nor is it known how
409 these people illuminated their dwellings and caves.

410 The use of torches within the tombs could have been either for practical purposes or as part of a burial
411 ritual. There is scant evidence of votive or ritual burning at other sites of the Monte Claro culture.
412 Hypotheses of ritual burning for the dead were proposed on the basis of the presence of pieces of
413 burnt wood found among grave goods adjacent to the dead in a cist (Gemiliano site) and the presence
414 of some 5-8 cm vessels, considered to be either ointment holders or lamps, found near cremated or
415 semi-combusted human bones in a disturbed cave (Tani site). No information or further evidence to
416 support the hypotheses is available (Lilliu 1998). In both cases, it is likely that those who sculpted the
417 vaults and buried the dead needed light to illuminate the tombs during their passage, to carry the
418 dead into the deep (3 m) and dark underground chambers and to prepare the burials with ochre and
419 grave offerings for the dead. It is reasonable to assume that as the living withdrew from the tombs
420 they left the torches in place, most likely as a votive offering to provide light for the dead.

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