

The hearth of the matter: A high-resolution magnetic microarchaeological study of a Neanderthal combustion feature utilizing Quantum Diamond Microscopy

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ABSTRACT

This study presents an integrative geoarchaeological and microarchaeological case study of combustion features, employing a multi-method analytical approach incorporating archaeomagnetic, rock magnetic, and soil micromorphological methods, alongside Quantum Diamond Microscopy (QDM). The case study focuses on a combustion feature from the Middle Palaeolithic site of El Salt (80 to 45.2ka). The primary aim is to reconstruct the depositional and post-depositional history of this combustion feature, identifying human activities and environmental processes using high-resolution magnetic and micromorphological data.

The combustion feature H89/90 was studied as part of the research into a large palimpsest of combustion features located within Unit XI. Archaeomagnetic and rock magnetic microarchaeological analyses of the combustion feature demonstrates that it is relatively well-preserved and in situ. This integrative approach reveals clear evidence of anthropogenic behaviours, including hearth maintenance, the use of grass-based fuels, and potentially the active sustaining of fire through the successive addition of these fuels. Furthermore, the study highlights maghemite as the primary magnetic carrier within the combustion feature. This mineral is not commonly reported as a dominant magnetic phase in archaeological hearths. Its presence suggests either previously unrecognized burning conditions, microenvironmental factors unique to this feature, or a broader need to improve the resolution at which hearth mineralogy is studied.

These interpretations are possible through high-resolution rock magnetic analysis, enhanced by QDM's micrometer-scale magnetic field mapping. Representing the first known application of QDM in archaeological science, this method allows researchers to visualize internal magnetic fabrics directly within thin sections, enabling the identification of specific microfacies and magnetic structures that are typically invisible to traditional micromorphological analysis. As a result, this method not only deepens our understanding of the magnetic architecture of the hearth but also strengthens behavioural interpretations of its formation and use. Overall, the study demonstrates the value of a deeply integrated magnetic microarchaeological approach for investigating human behaviour associated with fire use.

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1. Introduction and research aims

Geoarchaeological analyses, especially those involving micromorphology and microcontextual thin section analysis, are integral to interpreting a wide array of data points, including geochemical, geophysical, and morphological data obtained from anthropogenic sedimentary features, which are direct manifestations of human activities. Building upon the foundations laid by previous studies, such as Dinçkal et al. (2024a; 2024b), our research has demonstrated the advantages of integrating geoarchaeological soil micromorphology with archaeomagnetic (directional and rock magnetic) analysis of combustion features.

This case study presents a comprehensive magnetic microarchaeological analysis of the combustion feature H89/90 from the Middle Palaeolithic site of El Salt (Spain, Fig. 1A–C). Hearth H89/90 is a seemingly single combustion event within a complex palimpsest of combustion material potentially providing a single example of Neanderthal combustion practices within this area of frequent combustion. The study of this combustion feature employed an integrative methodology combining archaeomagnetic, rock magnetic, and soil micromorphological analyses, now enhanced by the application of the Quantum Diamond Microscope (QDM). The QDM targets the properties of fluorescent nitrogen-vacancy (NV) colour centres in diamonds to create spatially resolved maps of local magnetic fields. This method utilizes NV electronic spins, which are optically initialized and read out, across a diamond sensor surface where the sample of interest is placed (Glenn et al., 2017; Levine et al., 2019). As a relatively new form of technology, the use of the QDM in the geosciences has been limited but is developing. It has been used in the analysis of meteorites for insights into the early solar system (Volk et al., 2021), in the study of modern soils to understand the magnetic mineralogy influenced by precipitation

(Fu et al., 2023), and in environmental magnetism through high-resolution studies of speleothems (cave formations) to reconstruct palaeoclimate conditions (Fu et al., 2021).

The primary aim is to use the combined potential of these methods to reconstruct the hearth's life history, focusing on both the anthropogenic behaviours that shaped its structure and the environmental, geogenic, and anthropogenic post-depositional processes that may have altered it. This study serves as an introduction to the application of QDM in archaeological and soil micromorphology, focusing on the analysis of a single thin section from a hearth from the Middle Palaeolithic site of El Salt. To our knowledge, the application of the Quantum Diamond Microscope in archaeological science is unprecedented, particularly when used alongside soil micromorphology. This study thus introduces a significant methodological innovation by demonstrating the QDM's potential to improve the resolution and accuracy of rock magnetic and archaeomagnetic interpretation in direct connection with micromorphological analysis. Focusing on a single thin section allows for controlled integration of QDM results with other magnetic microarchaeological data, facilitating precise interpretation of anthropogenic and geogenic features within the hearth.

1.1. El Salt and combustion feature H89/90

El Salt, Alcoi, Spain, is a Middle Palaeolithic site with a stratified sequence spanning approximately 80 to 45.2 thousand years ago (Galván et al., 2014). The site contains well-preserved sedimentary deposits with abundant evidence of human activity, including hearths (Mallol et al., 2013; Leierer et al., 2020; Dinçkal et al., 2024a; 2024b; Herrejón-Lagunilla et al., 2024), lithic assemblages (Mayor et al., 2020, 2022, 2025), and faunal and botanical remains that provide insights into Neanderthal subsistence strategies and environmental adaptations

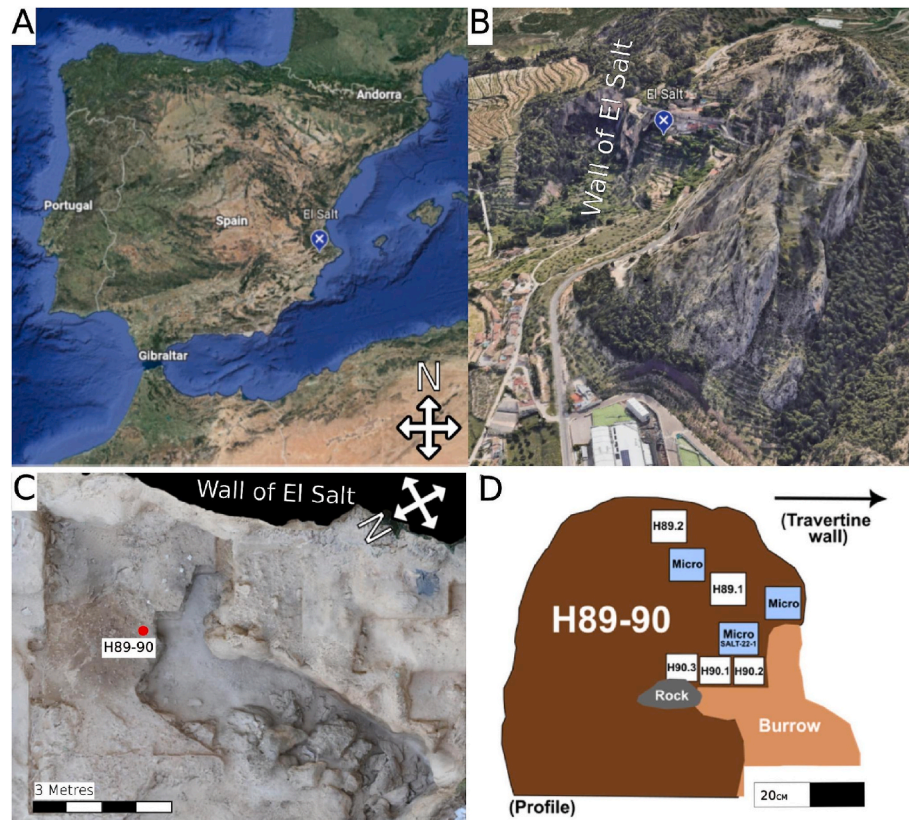


Fig. 1. A) Location of Site in Spain, B) Location of Site on Landscape. A) and B) source from Google Earth. C) Location of the combustion feature H89-90 on site, D) Scheme of the samples location within H89-90 (blue squares = micromorphology samples; light red circles = H89 archaeomagnetic samples; dark red circles = H90 archaeomagnetic samples). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

(Pérez et al., 2019; Marquina et al., 2020; Marquina et al., 2021; Marin-Monfort et al., 2021; Fagoaga et al., 2018, 2019). A defining feature of El Salt is the presence of extensive deposits with palimpsest nature that involve combustion features, reflecting repeated episodes of fire use and occupation over time (Mayor et al., 2020, 2022, 2025). These overlapping combustion features reveal patterns of site maintenance, spatial organization, and the reoccupation of living spaces by Neanderthal groups. In this context, H89-90 stands out as a focal point for further investigation.

Hearth H89 is located within stratigraphic unit XI. This unit, which is brown, silty clayey and ~8 to 10 cm in thickness, is found towards the base of the site's stratified deposit and is currently undergoing large-surface excavation. The hearth is covered by patches of gray silty clay (field facies LG14) and brown silty clay (field facies LM1). It lies within a large archaeological palimpsest deposit that includes other combustion features of differing morphology and is found adjacent to the hypothetical hearth H90. However, due to the nature of the palimpsest, it was difficult to distinguish between H89 and H90. It is even unclear if they represent distinct hearths. In advance, when we refer to H89, we specifically mean components identified solely within the area hypothetically designated as hearth H89. And when mentioning H90, we specifically refer to the area of the alleged hearth H90. When we refer to H89/90, we mean the entire complex, including samples found across what might be the combined extent of the two hearths or singular features.

During excavation, H89 was found to consist of two layers: a white layer (~2 cm-thick) on top and a black layer (0.5-2 cm-thick) just beneath, which is typical for El Salt combustion features (e.g. Mallol et al., 2013). The white layer exhibited a greenish hue and reduced thickness in certain areas, particularly in the northeast section of the hearth. The black layer partially overlaid another hearth, H91. Its thickness ranged from 0.5 cm when situated over the ashes of Hearth H91 to approximately 2 cm when resting on the silt of an underlying stratigraphic unit beneath Hearth H89.

2. Materials and methods

2.1. Sampling strategy

Prior to excavation, 15 Eppendorf vials of white layer sediment (approximately 2 to 3 g per vial) were collected from H89 in transects of ~5 cm across the surface for Fourier Transform Infrared Spectroscopy (FTIR) analysis. FTIR spectroscopy provides sedimentary profiles of archaeological samples and supports other microarchaeological analyses by efficiently identifying the mineral composition of the sedimentary matrix (Berna et al., 2007; Poduska et al., 2011; Regev et al., 2010; Rodríguez Cintas and Cabanes, 2017). While minerals like calcite, clay, and quartz are expected in Paleolithic sediments, others such as dahlite or apatite may indicate faunal remains or ash diagenesis (Berna et al., 2007; Rodríguez-Cintas and Cabanes, 2017).

Two magnetically oriented blocks from H89 (H89.1 and H89.2), and three from H90 (H90.1, H90.2 and H90.3) were collected for archaeomagnetic directional analyses (see Section 2.5 Archaeomagnetic Directions and Rock Magnetic Analysis). H89.1, H89.2 and H90.3 only included black layers, while H90.1 and H90.2 included white and black layers. Additionally, bulk samples from H89-90 white layer sediment were collected for additional rock magnetic analyses.

Three micromorphological samples were taken from H89 (Salt-22-1, Salt-22-4 and Salt-22-5; Fig. 1D, Blue boxes). Salt-22-1 was from an area of the hearth near a burrow and contained the facies above the hearth as well as the white layer. Due to the presence of a tufa rock fragment, the sample broke while being gathered and only the ash layer was recovered. Salt-22-4 was taken from a location that seemed to only contain the black layer, while Salt-22-5 included the white layer, the thermally altered combustion surface, and carbonized substrata (i.e. it contains the complete combustion facies sequence). For this reason, Salt-22-5 was

chosen for QDM analysis.

FTIR samples were collected during the 2021 field season, Archaeomagnetic and micromorphological samples were collected in 2022. Fig. 1 and D and 1E highlight the surrounding context and the positioning of the samples within the H89-90 complex.

2.2. Archaeological soil micromorphology

Micromorphological samples were gathered by delimiting the sampling location during excavation and carefully excavating around it to save a small block (~10 × 10 cm) as the excavation deepened. In the field, samples were wrapped with plaster of paris to maintain their integrity. Total station coordinates were recorded both above and below each sample. Once removed, the samples were transported to the Archaeological Micromorphology and Biomarkers Laboratory (AMBI-LAB) at the Universidad de La Laguna (Spain) for processing.

The samples were dried for 48 h at 40 degrees Celsius, then impregnated with a 70/30 polystyrene solution with an acetone thinner and less than 1% hardener. No accelerant was used, as it could induce artificial heating during hardening, potentially affecting the sample's inherent magnetic signature (Dinçkal et al., 2024a). Once cured, samples were cut into 6 cm × 8 cm × ~4 cm-thick chips with a radial saw. The chips were sent to Wagner Petrographic Laboratory, Ltd., in Utah (USA) to be manufactured into 7.62 cm × 5.06 cm × 30micron-thick thin sections. Thin sections were analyzed at the AMBILAB micromorphology facilities utilizing transmitting light microscopes for Plane Polarized Light (PPL) and Cross Polarized Light (XPL) analysis.

2.3. Microfacies rock magnetic analysis

Microfacies rock magnetic analysis was performed upon micromorphological slab samples following a similar preparation for rock magnetic analysis outlined in Dinçkal et al. (2024a). Samples were taken from slabs once the microstratigraphy was understood and then divided into four subsamples, each representing a distinct component of the hearth's microstratigraphic structure. Rock magnetic analysis on microfacies samples included room-temperature measurements using a Lake Shore VSM 8600 vibrating sample magnetometer to examine hysteresis loops under an applied field of ±1.8T, backfield coercivity fields measured at a saturation field of 2.5T, and first-order reversal curves (FORC). Microfacies analysis was conducted using a MPMS3 'Squid-VSM' magnetic properties measurement system (MPMS) for low-temperature analyses, which encompassed AC (Alternating Current) susceptibility, room-temperature saturation of isothermal remanent magnetization (RT-SIRM), low-field warming and cooling curves, as well as zero-field (ZFC) and field cooling (FC) at low temperatures. All analyses were conducted at the University of Minnesota's Institute for Rock Magnetism. Backfield coercivity curve component analysis was conducted with the Max UnMix application (Maxbauer et al., 2016). First Order Reversal Curves (FORC) diagrams were produced from 231 measurements, utilizing a smoothing factor of 8 with 100 colors (see Supp. Figs. 1–4). FORCs were processed with the use of the FORCinel application (Harrison and Feinberg, 2008). Fig. and graphs were organized with the use of R with the ggPlot2 package (R Core Team, 2021; Wickham, 2016). Rock magnetic analyses in microfacies were conducted on four samples from the Salt-22-5 micromorphological sequence.

2.4. Quantum Diamond Microscopy

For a detailed analysis of the methodology and science behind QDM application, see Glenn et al. (2017) and Fu et al. (2021). From a single QDM run, three images are obtained: a LED light image, a Laser Reflective Image, and the QDM Image itself. The thin section, approximately ~30 µm, had been polished previously and provided a suitable surface to retrieve magnetic results from. The addition of the LED and

Laser images allowed for the sample locations to be directly identified in thin section scans. The diamond lens was pressed onto the surface of the sample, with the lens and laser attuned to the diamond's surface of contact.

Optically Detected Magnetic Resonance (ODMR) parameters were identified using the QDM_Lab program, with measurements taken under an instantaneous bias magnetic field of 0.9 mT. The field was consistently reversed during the course of measurement to provide a near-zero net bias field (Glenn et al., 2017). Results were obtained as an average of approximately 40 sweeps, with the raw results processed using the QDMlab software package in MATLAB (Volk et al., 2022).

Results were processed by retrieving both the magnitude of the Positive Current and the Negative Current of the magnetic field. When the two results are added together (Positive Current + Negative Current), a map of ferromagnetism (s.l.) and ferrimagnetism (s.s.) is identified. Alternatively, to produce a map of paramagnetism, the negative current is subtracted from the positive current (Positive Current – Negative Current).

Maps were corrected for background using QDM_Lab. During this process, different maps were produced with different scales to highlight the influence and strength of magnetic fields. Using Adobe Photoshop, final QDM maps for ferromagnetism and paramagnetism were then overlaid onto the LED images.

2.5. Archaeomagnetic directions and rock magnetic analysis

Archaeomagnetic hand blocks were carefully delimited prior to the excavation of the H89-H90 hearth complex, allowing stable blocks to form during the process. Plaster was poured over the isolated materials. Once the plaster was set, it was leveled using a plastic plate and a spirit level. A mark indicating magnetic North was then inscribed on the surface with a magnetic compass (Thellier, 1936). The oriented samples were subsequently removed and sent to the Paleomagnetic Laboratory at the Universidad de Burgos (Spain) for further processing.

In the laboratory, the blocks were first consolidated by spraying and immersion in a sodium silicate and water solution. Following consolidation, they were cast in additional plaster and cut into cubic specimens of approximately 8–10 cm³. From these, 18 specimens were selected for thermal demagnetization of the natural remanent magnetization (NRM) to determine the mean archaeomagnetic directions associated with the last heating events of the hearths. Of these, 8 specimens originated from hearth H89 (4 from H89.1 and 4 from H89.2), and 10 from hearth H90 (2 from H90.1, 4 from H90.2, and 4 from H90.3). All specimens were taken from the uppermost ~2 cm of the oriented blocks, with the exception of one that was sourced from ~2 to 4 cm in depth.

Thermal demagnetization was conducted in 14 steps, including measurement of the NRM, up to a maximum temperature of 585 °C. This process was carried out using an ASC TD48-DC thermal demagnetizer. Remanent magnetization was measured with a 2G-755 Superconducting Rock Magnetometer, utilizing the 2G Enterprises Data Acquisition software (v. 2.99.5). All measurements took place at the Paleomagnetic Laboratory of the Universidad de Burgos.

During the demagnetization process, one specimen from H90.1 (H901C4A) broke at a relatively low temperature and was excluded from directional analyses. Another specimen from H89 (H892C3A) also broke, but this occurred at a higher temperature step (500 °C), allowing directional data to still be recovered. Mean archaeomagnetic directions were calculated using Remasoft software (Chadima and Hrouda, 2006).

Additional rock magnetic analyses on macroscopically identified facies (two black layer samples from archaeomagnetic oriented blocks and a single white layer from an unoriented bulk sample) were carried out. This included progressive IRM acquisition, hysteresis parameters (± 1 T), backfield coercivity curves and thermomagnetic curves up to 700 °C in air. Measurements were conducted using a Variable Field Translation Balance at the University of Burgos.

2.6. Fourier Transform Infrared Spectroscopy

FTIR analysis was carried out using an iS5 FT-IR Nicolet Thermo Scientific Spectrometer at the Laboratory for MicroArchaeology (ALMA), University of Rutgers. Infrared spectra were obtained by grinding a small sample and KBr in an agate mill. The mix was compressed into a pellet and inserted into the sample holder of the spectrometer. Each sample was scanned 32 times at a range of 4000–400 cm⁻¹ with a 4 cm⁻¹ resolution. The resulting spectra were interpreted with the assistance of a database of standards, while the origin of geogenic or anthropogenic calcite was determined following Regev et al. (2010) and Poduska et al. (2011). The main mineral components of each sample were determined using the standards from the Kimmel Center for Archaeological Science, Weizmann Institute of Science. FTIR is an important methodological tool that provides important information and is especially effective when used as an initial analysis to determine the general composition of archaeological samples.

3. Results

3.1. Archaeological soil micromorphology

The spatial arrangement of these microfacies is illustrated in Fig. 2 as seen in samples Salt-22-4 and Salt-22-5. Although Salt-22-1 also contains the White Layer, it lacks the Black Layer. Instead, it includes two additional stratigraphic elements: brown silty sediment (field facies LM1), and fragments of a small animal burrow adjacent to H89-90. Table 1 details all units along with their micromorphological characteristics.

Soil micromorphological analysis of H89/90 reveals a characteristic two-tiered structure typical of El Salt hearths (see Fig. 2 for all thin sections as well as the location of the microfacies), formed by combustion on an organic-rich substrate (Mallol et al., 2013). The lower Black Layer comprises two microfacies: a carbonized surface (Carbonized Organic Unit, Fig. 3E and F) underlain by thermally altered sediment (Thermally Altered Humified Organic Unit, Fig. 3G and H). Above this lies the White Layer, composed of two ash-dominated microfacies: a Decalcified Ash Unit at the base (Fig. 3C and D) overlain by a Calcite-rich Ash Unit (Fig. 3A and B). The boundary between these two groups of microfacies is interpreted as the original ignition surface, based on comparisons with previously studied hearths at El Salt and experimental combustion features on local soil, which demonstrate that burning occurred atop a humifying organic-rich layer (Mallol et al., 2013; Herrejón Lagunilla et al., 2019).

A notable feature is the decalcified ash (Fig. 3C) occurring just above this boundary, suggesting post-depositional chemical alteration from below, as no sedimentary process accounts for its separation from the overlying calcite-rich ash. The calcite-rich ash exhibits minor recrystallization, likely resulting from recent water percolation and does not appear to have compromised its structural integrity. Despite these limited diagenetic effects, the H89-90 complex remains well preserved. Red aggregates composed of a clayey matrix with microcharcoal fragments (Figs. 3B and 5E) are present in the ash units with sharp contact with the surrounding ash matrix.

3.2. Microfacies rock magnetic analysis

Hysteresis loops (Fig. 4A) display pot-bellied morphologies (Tauxe et al., 1996; 2002) typical of low-coercivity minerals (e.g., magnetite/maghemite), with the strongest magnetization in samples near the H89 combustion interface (Salt-22-5-1B and 2) and the weakest in the lower black layer (Salt-22-5-3). Backfield analysis confirms the dominance of low-coercivity magnetic components across the sampled interval. Most samples required two to three components to adequately model the magnetization, though the presence of a third component or higher in several cases may represent model overfitting rather than a

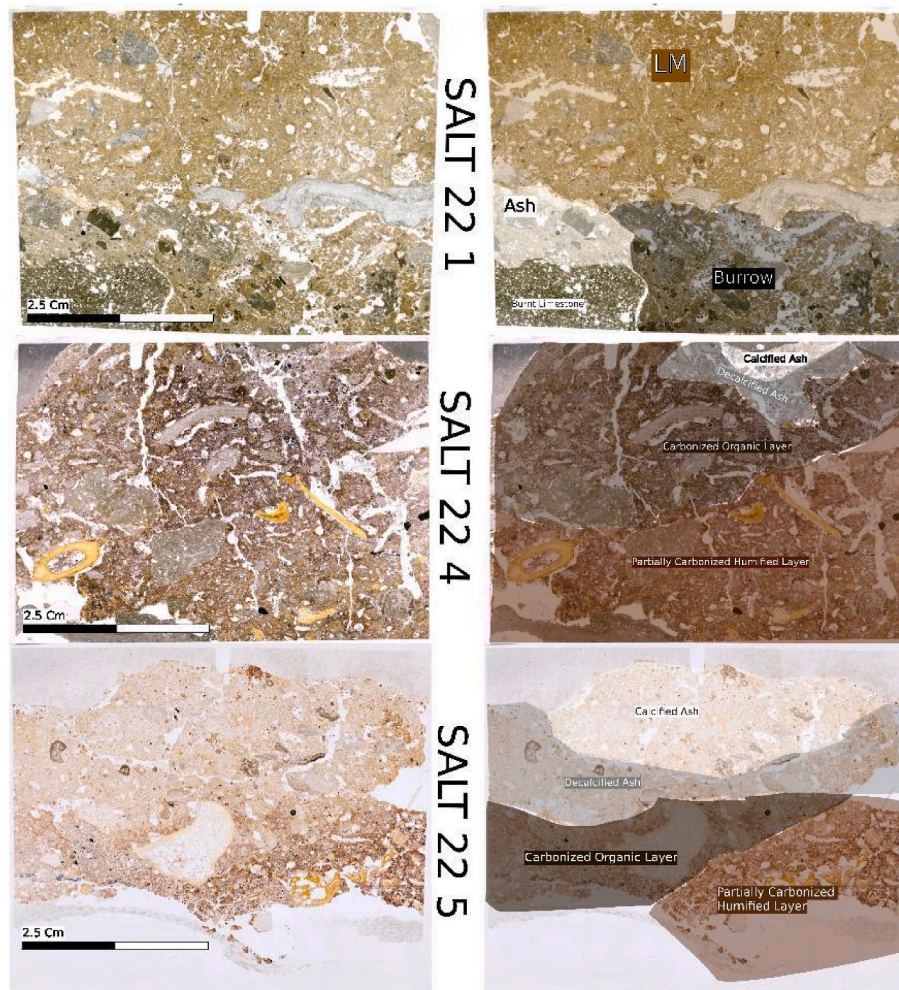


Fig. 2. Thin Sections used in Micromorphology Analysis. Left, clean the plate of each thin section. Right, Separation of Stratigraphic Units in each thin section. Note, due to the thinness of the ash unit above the burnt limestone in Salt 22-1, the separation between Calcified and Decalcified ash hasn't been marked.

distinct magnetic population (Kruiver et al., 2001; Maxbauer et al., 2016). Unmixing results for Salt-22-5-1a (from the top of the white layer) indicate the strongest dominance by a single component, with the strongest component contributing a proportional value of 0.83 (see Supp. Fig. 14 and Supp. Table 1). In contrast, Salt-22-5-3 (from the black layer) exhibited the least dominance by any single component. In this sample, the most dominant component contributed only 0.65 in the two-component model and 0.58 in the three-component model (see Supp. Fig. 19 and Supp. Table 4). FORC diagrams (see Supp. Figs. 1–4) reveal consistent patterns of strong superparamagnetic contributions, with vortex-domain particles most dominant in the ash units (Roberts et al., 2000, 2017). Low-temperature MPMS data suggest maghemite as a primary mineral carrying the magnetic signature. ZFC/FC curves show unblocking above 250 K with no Verwey transition (Fig. 4B), and room-temperature remanence (Fig. 4C) data confirm oxidation and a low-coercivity magnetic signature (Özdemir and Dunlop, 2010). Paramagnetic correction of hysteresis loops shows significant variation between the degree of correction: Sample 1A (upper ash) required the least amount of correction (see Supp. Fig. 6), indicating preserved ash with low paramagnetic influence and Fig. 4A II & III for correction trends). Samples 1B (Supp. Fig. 8), 2 (Supp. Fig. 10), and especially 3 (Supp. Fig. 12) required greater correction, suggesting increased influence paramagnetic content, in relative terms. Further research comparing combustion sediment with non-anthropogenic local sediment can help identify whether this pattern reflects that lower paramagnetic signals can be a proxy for the degree of combustion and organic matter loss.

3.3. Quantum Diamond Microscope results

3.3.1. The carbonized black layers from H89

QDM images of the Partially Carbonized Humified Layer (Fig. 5A.II and Fig. 5A.III) reveal a sparse distribution of ferromagnetic components, with only occasional and variably intense magnetic field formations across the groundmass. Their low frequency makes it difficult to identify clear patterns for ferrimagnetic minerals. In contrast, paramagnetic signals are more frequent and provide clearer insight into fabric features. For instance, the coprolite in Fig. 5A shows little visible fabric differentiation in ferromagnetic maps compared to the surrounding groundmass yet exhibits a distinct paramagnetic pattern, nearly absent within the coprolite but densely concentrated around it, allowing clearer identification of magnetic fabrics (Fig. 5A.III).

In the Carbonized Organic Layer, Fig. 5B shows an example of the fabric contrast being reversed due to the presence of intensively ferromagnetic (s.l.) nodule (Fig. 5B), which dominates the surrounding sediment's magnetic signature (both para- and ferromagnetic). This nodule is located near the interface between this layer and the overlying Ash units. Rock magnetic results indicate it has the strongest magnetic signature across all layers. However, the extreme magnetic intensity observed in QDM images is not matched by a comparable magnetic intensity in the rock magnetic data from the stratigraphically associated sample, suggesting this nodule is not skewing results. Thin section analysis also reveals no other formations resembling it.

Beyond this nodule, the paramagnetic signal in the Carbonized

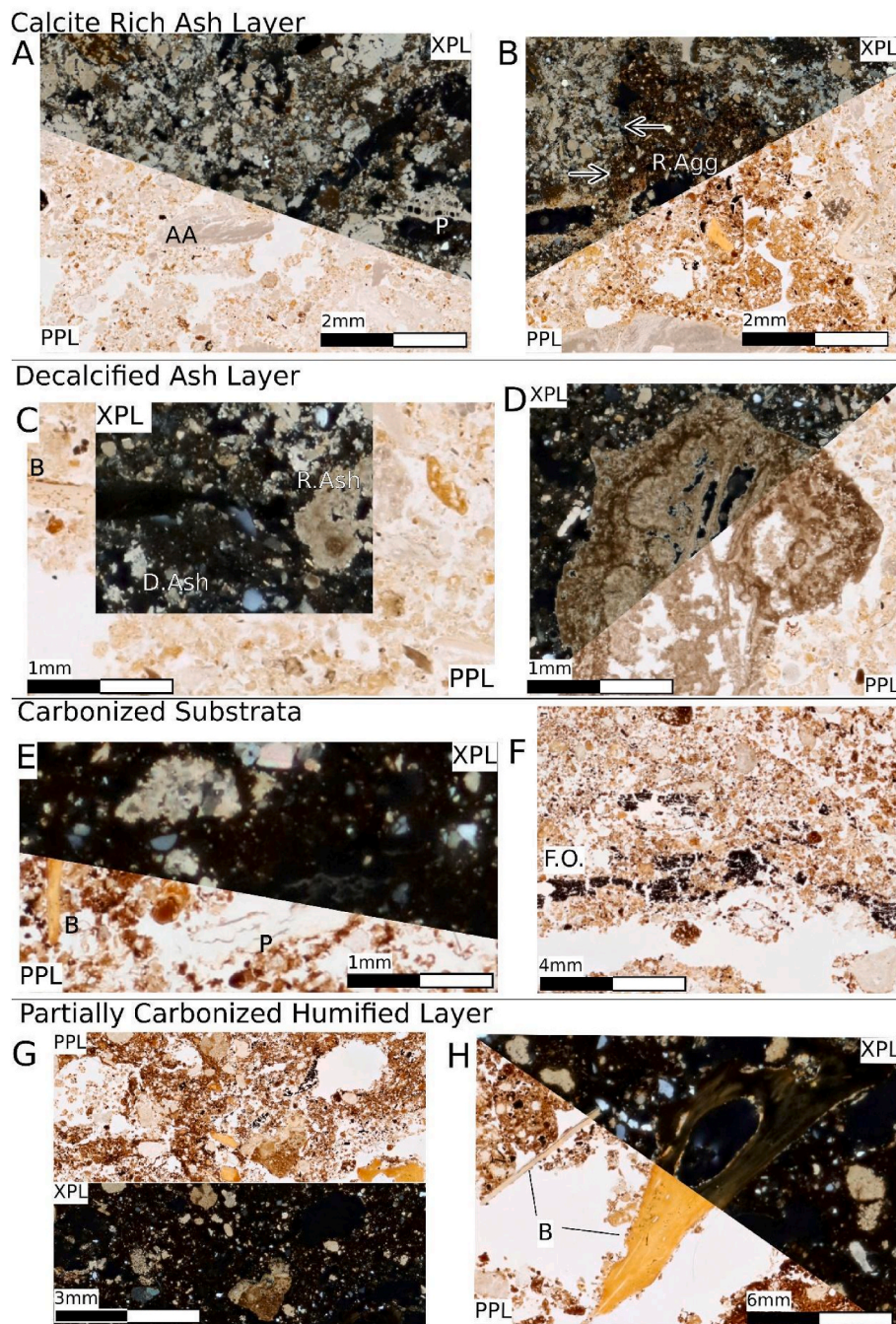


Fig. 3. Thin Section Micrograph from Salt-22-5. **A-B)** Calcite-rich ash layer. From the calcite-rich ash layer, the micrograph shows the calcite-rich ash primarily visible through the strongly birefringent round mass in XPL. AA refers to ash that appears to preserve the morphology of plant matter prior to combustion (articulate ashes', see [Mentzer, 2014](#); [Berna et al., 2012](#)). This is also visible in P, which denotes the location of phytoliths present with the same plant matter. **B)** Denotes the red aggregates (R.Agg) present within the unit. While seemingly allochthonous in appearance, the red aggregates appear to predate the cooling of the hearth ashes, as indicated by the close, unbroken contact between the birefringent ash and the red aggregates within the XPL components of the image (see arrows). **C)** Decalcified ash (D.Ash) component and groundmass. Note the lack of birefringence within the D.Ash, compared to the birefringence within the R.Ash component of the hearth. **(B)** one color indicates calcined thermal alteration. **D)** Burnt tufa present within the decalcified ash unit. **E)** Phytolith (P) and low thermally altered bone (B) present within the carbonized unit. **F)** Carbonized fibrous organics (F.O) within the same unit. **G)** PPL and XPL reference for the groundmass of the Thermally Altered Humified Unit; **H)** Both unburnt and partially burnt micro bone (B) present. The thin bone indicates the calcined bone. (PPL = Plane Polarized Light; XPL = Cross Polarized Light). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Organic Layer is significantly reduced compared to the Partially Carbonized Humified Layer, consistent with the paramagnetic decline observed in rock magnetic analysis. Meanwhile, the ferromagnetic component shows a moderate increase (compare [Fig. 5C.II](#) to [5A.II](#)).

3.3.2. The ashy white layers from H89

Analysis of both the decalcified and calcite-rich ash layers shows an overall increase in both the concentration of the ferro and paramagnetic minerals, a trend consistent across both units (see surrounding groundmass in [Fig. 5D](#)). Interestingly, while rock magnetic analysis indicates a reduction in paramagnetic signals, QDM imaging reveals a

Table 1

Summary of Micromorph and Rock Magnetic Parameters for Combustion feature microfacies.

Microfacies	Thin Section Samples	Rock Magnetic Samples	Micromorphological Description	Micromorphological Interpretation	Magnetic Mineralogy
Burrow	Salt-22-1	NA	The burrow contains internal fabrics similar to the LM but with a higher concentration of material from the adjacent burnt unit, including burnt limestone and tufa. Its microfabric features pellet-like circular inclusions and large, gravel-sized secondary calcite growths. A network of channel and chamber voids is present, with a microstructure ranging from closely aggregated pellets to more open configurations. Evidence of water movement includes iron aggregates, orthogenic iron oxides, and staining of limestone and bone with iron, black organic residues, or manganese.	Burrows are common in the XB and XI units at El Salt. Their contact with the LM is uncertain; they likely predate the LM but definitely postdate the ash units.	NA
LM	Salt-22-1	NA	The unit consists of reddish-brown sediment with unburnt limestone and calcite fragments. The groundmass comprises clayey silts with dispersed micritic calcite. Pores include vesicular voids, channels, and microfissures. Infrequent black specks, likely organic, and some fibrous organic matter are present. Some areas show secondary porous calcite formations with black hypercoatings. Iron oxidation is minimal, and bone material appears unburnt.	Standard sedimentary material found throughout El Salt.	NA
Transition Calcite Rich Ash	Very sharp contact with clear Salt-22-1 Salt-22-4 Salt-22-5	Salt-22-5-1A. Salt-22-5-1B	change in composition material. The microstructure is granular with a single-spaced enaulic distribution, featuring packing, planar, channel, and chamber voids that enhance its pedogenic character. The fine fraction contains abundant calcite, ash pseudomorphs, articulated ashes, burnt micro-bones, micro-charcoal, and melted phytoliths. Coarse components include thermally altered tufa and limestone fragments. Higher in the unit, red aggregates with pellet-like morphologies and organic inclusions appear. Whether these were introduced pre- or post-burning is unclear, though they resemble humified sedimentary material from El Salt. The contact between red aggregates and ash is sharp yet interfaced, suggesting the aggregates are likely not post-depositional.	The presence of articulated ash (Fig. 3A), micro-bone, and a calcite-rich B-fabric suggests the ash layer is largely in situ, with minimal evidence of post-depositional alteration.	Strongly Magnetic, Magnetic Mineralogy dominated by vortex domain, SP particles and SD grains. Very minor Paramagnetic contribution.
Transition Decalcified Ash	This transition is most evident Salt-22-1 Salt-22-4 Salt-22-5	Salt-22-5-1B Salt-22-5-2	under XPL, where the dull B-fabric of the lower units becomes distinctly crystallitic in this The microstructure displays a double-spaced fine enaulic pattern with a moderately separated granular texture. Voids are primarily packing. The fine fraction includes light beige clays, calcitic material, and infrequent burnt fibrous organics, with moderate quartz and calcite presence under XPL. Coarse components include calcined bone fragments, a calcined tooth, phosphatic coprolite remnants, and heavily thermally altered limestone exhibiting decalcification and recalcification. The B-fabric resembles the lower unit, showing speckled and crystallitic zones. Thermal alteration is evident in large limestone, bone, and tufa aggregates. Organic matter and pedogenic features are unevenly distributed, with isolated iron and manganese-stained zones distinguishing this layer from the one below.	This unit represents a significant shift in material composition and thermal influence compared to what is below. Localized evidence of burning and recalcification processes in the limestone or organic component, and the absence of calcite ash indicates that there was likely a component of calcite ash that was decalcified.	Calcite Rich Ash. Strongly Magnetic Magnetic Mineralogy dominated by vortex domain, SP particles and SD grains. Very minor Paramagnetic contribution but still requires more paramagnetic correction than Calcite Rich Ash deposits.
Transition	Defined by a diffuse, undulating yet sharp boundary between the Decalcified Ash and Carbonized Unit, marked by reduced red clay content and an overall decline in carbonized and organic material within the ash layer.				

(continued on next page)

Table 1 (continued)

Microfacies	Thin Section Samples	Rock Magnetic Samples	Micromorphological Description	Micromorphological Interpretation	Magnetic Mineralogy
Carbonized Organic Unit	Salt-22-4 Salt-22-5	Salt-22-5-2	This unit is characterized by a light brown, double-spaced fine enaulic structure dominated by packing voids, with some vughs and planar voids. The microstructure is moderately granular. Fine material consists of silt to fine sand-sized clay aggregates, stained silt, opaque iron/manganese clay aggregates, melted phytoliths, frequent micro charcoal, and infrequent micro bone fragments. Coarse fragments include burnt bones and limestone, along with frequent charcoal and iron/manganese pedofeatures. Large bone fragments are articulated. Increased carbonized organic material and significant iron staining suggest heightened thermal alteration. Burnt material is more concentrated and articulated in lower layers, becoming more chaotically distributed near the contact with the ash layer. The B-fabric appears speckled, reflecting compositional and thermal variation from underlying deposits.	This layer is a strongly thermoaltered variation on the Layer XI type sedimentary deposit at El Salt, with a significant reduction in the humified organic components (which are now becoming more carbonized and burnt to a higher degree).	Strongly Magnetic, Magnetic Mineralogy dominated by SP particles and SD grains. Moderata Paramagnetic contribution to magnetic signature.
Transition	The transition from the lower units is distinct but heavily undulating, primarily identified by a reduction in humified organic and red clay components, and accompanied by an increase in carbonate or burnt material, which is only apparent under PPL (Plane Polarized Light), as under XPL (Cross Polarized Light) no major shift in the groundmass can be observed.				
Partially Carbonized Humified Organic Unit	Salt-22-4 Salt-22-5	Salt-22-5-3	Reddish-brown, clay- and organic-rich heterogeneously distributed materials. C/F (coarse-to-fine) ratio ranges from close fine enaulic to single-spaced fine enaulic. The microstructure features finely to moderately separated granular forms. Coarse inclusions, limestone, bone fragments, and sedimentary aggregates, alongside organic material such as rhombic black specks, fibrous charcoal, and micro-bone fragments. B-fabric is undifferentiated with occasional speckling from coarse inclusions. Voids are variable, often linked to bioturbation. Sedimentary aggregates include micritic calcite, burnt bone, and coprolites (both omnivorous, herbivorous and carnivorous). Bone ranges from gravel to fine sand sized, infrequent evidence of thermal alteration, some are partially articulated. Manganese and iron oxide staining is common, forming dark red or black accumulations on bones and in the groundmass.	This unit appears to be a moderately thermoaltered variation of the standard layer XI sedimentary deposit from El Salt, characterized by rich organic matter, humified deposits with some moderate carbonization.	Weakest magnetic signature. Magnetic Mineralogy driven primarily by SP particles and SD grains. Coercivity curve unmixing analysis identifies 2, maybe 3 components contributing to the overall magnetic signature, no component is entirely dominant in its contribution. Very strong paramagnetic contribution

paradoxical increase in paramagnetic response (Fig. 5D.III). This discrepancy is explained by the fact that even though SP particles are ferric in structure, because they are so fine in their domain structures they behave in a way similar to paramagnetic grains in the QDM and are thus mapped in the paramagnetic maps (Chen et al., 2022; Mosavian et al., 2024).

Superparamagnetism is evident in all rock magnetic samples but appears more pronounced in the ash layers. The heightened intensity in both ferrimagnetic and paramagnetic QDM maps likely reflects an increase in overall ferrimagnetic mineral concentrations, especially above the combustion zone. Without the context provided by rock magnetic data, the dense paramagnetic signals in QDM images would not be interpreted as superparamagnetic and could be mistaken for a paramagnetic contribution. The observed magnetic enhancement likely stems from an increase in ultrafine (i.e SP) particles which correlate with an overall increase within the ferric content of the white layers compared to the black layers. Although the frequency of larger domain ferrimagnetic minerals does not show a marked increase in the QDM maps (compare Ferrimagnetic maps of Fig. 4A–D), the magnetic

intensity is notably greater than in the underlying black layers (see Fig. 5D.II and Fig. 4A–D). The increase of SP grains seen in the ‘paramagnetic’ map most likely reinforces this clear indication the magnetic enhancement is being driven by ultra-fine particles.

As previously noted, red aggregates (Figs. 3B and 5E.I) appear within the ash layers, raising questions about whether they were deposited post-hearth formation or incorporated during combustion. QDM imaging was used to assess their fabric alignment. In both ferrimagnetic and paramagnetic maps, the red aggregates show no substantial magnetic contrast with the surrounding ash groundmass (Fig. 5E.II and 5E.III). In contrast to the clearer magnetic distinctions seen in features like the coprolite (Fig. 5A.III) or the burnt limestone fragment (which are primarily low to negative magnetic -or diamagnetic-) in Fig. 5D.II and 5D.III, the red aggregates lack discernible boundaries. This suggests they were not introduced post-depositionally, which would have led to them appearing with greater relief in the magnetic maps. Instead, the magnetic mineralogy of the red aggregates appears to have developed in tandem with the surrounding ash, indicating a co-developed origin prior to its final cooling.

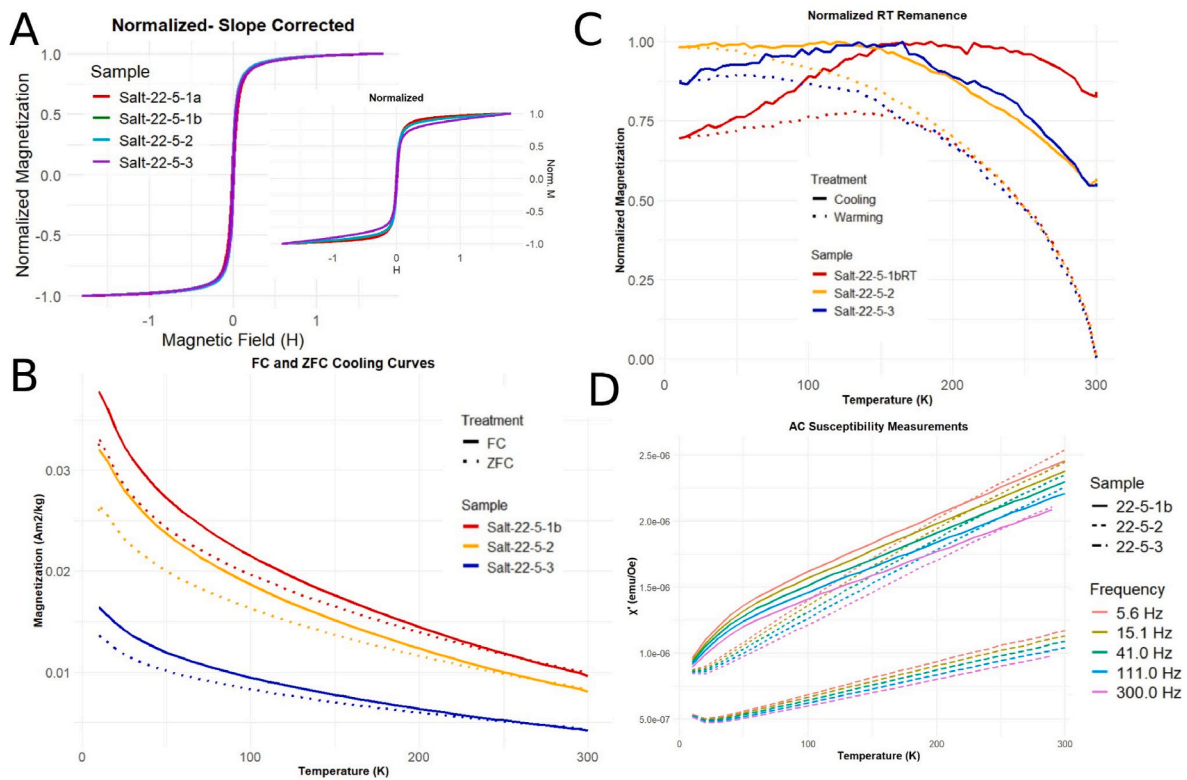


Fig. 4. Rock Magnetic Results: A) Normalized Hysteresis loops. All loops are pot-bellied, with samples 2 and 1B showing the combustion area being magnetically the strongest and sample 3 (black layer) having the weakest magnetic signature, representing the area that is the least thermally affected. Normalized hysteresis loops corrected for paramagnetic influence show the variation in paramagnetism influencing the magnetic signature (For further reference see [Supp. Figs. 7–12](#)). B) FC and ZFC cooling curves, there is no evidence for either the Verwey transition at ~ 120 K to indicate magnetite, nor the Morin transition at ~ 260 K to indicate hematite. C) RT remanence shows, similar to the FC and ZFC, no Verwey transition or Morin transition for magnetite or hematite respectively. Samples Salt-22-5-1B and Salt-22-5-3 show a hump forming during the cooling phase, which is often diagnostic of maghemite. This hump is heavily minimized in Salt-22-5-2. D) AC susceptibility shows a high amount of superparamagnetism at play within each of the samples, which is visible in the change in magnetic susceptibility between each frequency of the field. The uptake in magnetism occurring at ~ 50 K for samples Salt-22-5-2 and Salt-22-5-3 especially, is a product of the paramagnetism at play, which remagnetizes below ~ 50 K.

3.4. Archaeomagnetic directions and rock magnetic analysis

NRM oscillates between 8.0×10^{-6} and $4.2 \times 10^{-5} \text{ Am}^2\text{kg}^{-1}$ in H89, and between 2.4×10^{-6} and $7.8 \times 10^{-5} \text{ Am}^2\text{kg}^{-1}$ in H90. The component related to the last heating (Comp. A in [Fig. 6](#)) of H89 was isolated between 200°C and $330\text{--}500^\circ\text{C}$ ([Fig. 6a-b](#)). In the case of H90, Comp. A was isolated between $200\text{--}250^\circ\text{C}$ and $330\text{--}450^\circ\text{C}$ ([Fig. 6c](#)). This component, interpreted to be the Characteristic Remanent Magnetization (ChRM) direction, is of normal polarity in both hearths. Higher temperature steps are interpreted as the record prior to the last burning. Steps below $200\text{--}250^\circ\text{C}$ were excluded to avoid possible viscous influence. The main mechanism of magnetization of Comp. A is interpreted as a partial thermoremanence.

Mean archaeomagnetic directions and their main associated parameters are summarized in [Table 2](#) and represented in [Fig. 6d-f](#). To exclude potential outliers, two filters were applied when calculating the mean archaeomagnetic directions: (1) specimens with maximum angular deviation or MAD $> 5^\circ$ and (2) specimens with an angular distance from the mean (after filtering by MAD) $> 25^\circ$ are excluded.

The rock magnetic analysis conducted on samples of facies recognized macroscopically (white and black layers within the H89-90 hearth complex, see [Supp. Fig. 20](#) for location) reinforces the earlier magnetic microarchaeological findings. Hysteresis loops, progressive IRM acquisition, and backfield coercivity curves align well with prior results indicating the dominance of low-coercivity ferromagnetic minerals (see Supplementary Material). Of particular interest are the thermomagnetic curves. The white layer exhibits a fully reversible thermomagnetic

curve, pointing to temperatures of at least 700°C in these facies ([Supp. Fig. 21](#)). Its Curie temperature, approximately 610°C , suggests partial maghemitization (grain-surface oxidation of magnetite particles), consistent with previous analyses. In contrast, the black layers' thermomagnetic curves are not reversible. They display Curie temperatures near 580°C , characteristic of magnetite, and show secondary magnetite formation upon cooling ([Supp. Fig. 22](#) and [Supp. Fig. 23](#)). This indicates lower heating in this facies, aligning with micromorphological observations of partial burning in the black layer. The black layer of both hearths reached in their last heating maximum temperatures of about $450\text{--}500^\circ\text{C}$. This is the maximum unblocking temperature ($\text{max } T_{\text{UB}}$) observed in the component A ([Fig. 6a-c](#)). This result agrees well with the behavior described in their thermomagnetic curves (see [Supp. Figs. 22–23](#)).

By comparison, comparative Rock Mag analyses on loose samples of unburnt substrate reveal the predominance of low coercivity minerals and the presence of magnetite, with Curie temperatures close to 585°C (see [Supp. Fig. 24](#)).

3.5. Fourier Transform Infrared Spectroscopy

FTIR analysis shows that the H89 White Layer sediment is mainly composed of calcite, along with recrystallised calcium-based minerals such as dahllite, as well as clays that show peaks indicative of burning ([Fig. 7](#)). At El Salt, dahllite appears frequently in trace amounts, where it can be diagnostic of in-situ mild alteration of bone, tufa and limestone fragments, and in larger concentrations, it signals diagenetic alteration

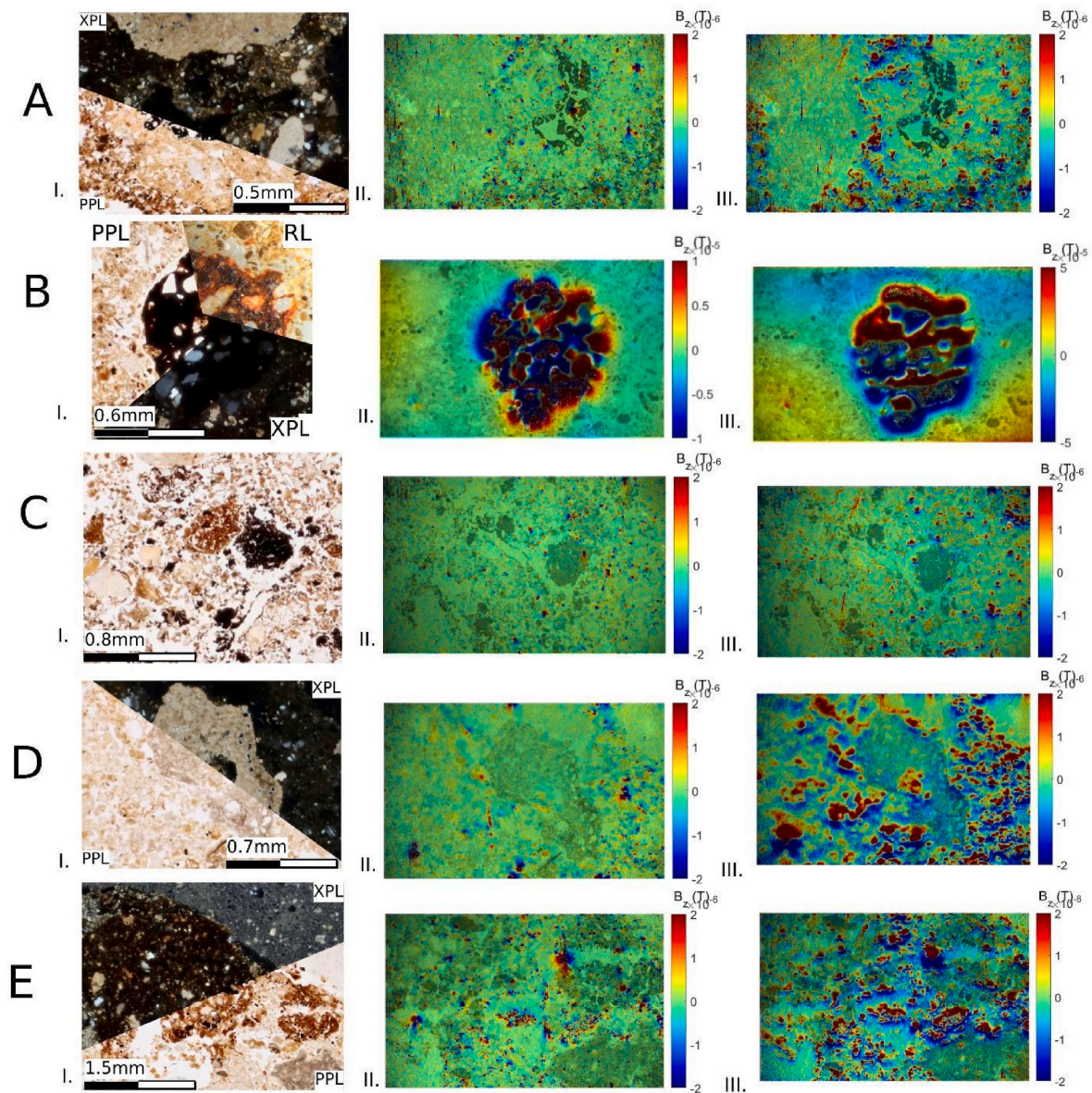


Fig. 5. QDM Results. **A)** Micrographs in QDM results for coprolite samples within thermally altered substrata. **I.** Coprolite sample shown in XPL and PPL. **II and III.** Ferromagnetic and paramagnetic results, respectively. Note; the difference in the distribution of magnetic fields identified within the general groundmass as well as the internal groundmass of the coprolite, indicating different authigenic processes. **B)** **I.** Microphotograph of the iron nodule in PPL, Reflective Light (RL), and XPL. Note; the sharp contact with the boundary of the sediment. **II and III.** Ferromagnetic and paramagnetic QDM maps, respectively. Compared to the other magnetic QDM maps presented here, note the order-of-magnitude increase in the scales for both paramagnetic and ferromagnetic intensities. **C)** Organic material within the groundmass of the carbonized layer. **I.** Microphotograph. Note; that the QDM orientations are offset by 90° compared to the microphotograph. **II and III.** Ferromagnetic and paramagnetic maps, respectively. Note; that the general distribution of ferromagnetic and paramagnetic components appears relatively aligned, and that the frequency of these magnetic fields is relatively low in comparison to the ash layers. **D)** Photographs and QDM results for the decalcified ash layer with an interbedded limestone fragment. **I.** Microphotograph in PPL and XPL. **II and III.** Ferromagnetic and paramagnetic maps, respectively. Note the fabric contrast between the diamagnetic burnt limestone and the substantially more magnetic surrounding sediment present within the decalcified ash. **E)** **I.** Microphotograph of red aggregate component within the calcified ash unit. **II and III.** Ferromagnetic and paramagnetic maps, respectively. Note that, while the limestone fragment in the bottom right corner shows substantial differences in frequency and distribution of magnetic components, the red aggregates do not show any significant difference in distribution frequency or position compared to the surrounding ash. Of final note, paramagnetic maps for ash layers show a higher frequency and dominance of the paramagnetic component, which appears counter to the rock magnetic results that indicated a reduction in the paramagnetic component in ash layers. This is interpreted as a result of the increased frequency and concentration of superparamagnetic domain-size ferrimagnetic minerals present within the ash layer compared to those in the lower levels. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

of the ash itself (Mallol et al., 2013). Either way, its presence provides a handy fingerprint for post-depositional change (Karkanis et al., 2000).

4. Discussion

This study integrates a broad range of soil and magnetic techniques,

including archaeomagnetism (directional analyses), micromorphology, FTIR, rock magnetism, and the novel application of Quantum Diamond Microscopy (QDM). In the discussion that follows, we address a series of interrelated questions concerning the structural coherence of the hearth (s), the extent of mechanical and chemical alterations affecting the feature, and the differentiation between anthropogenic signals and post-

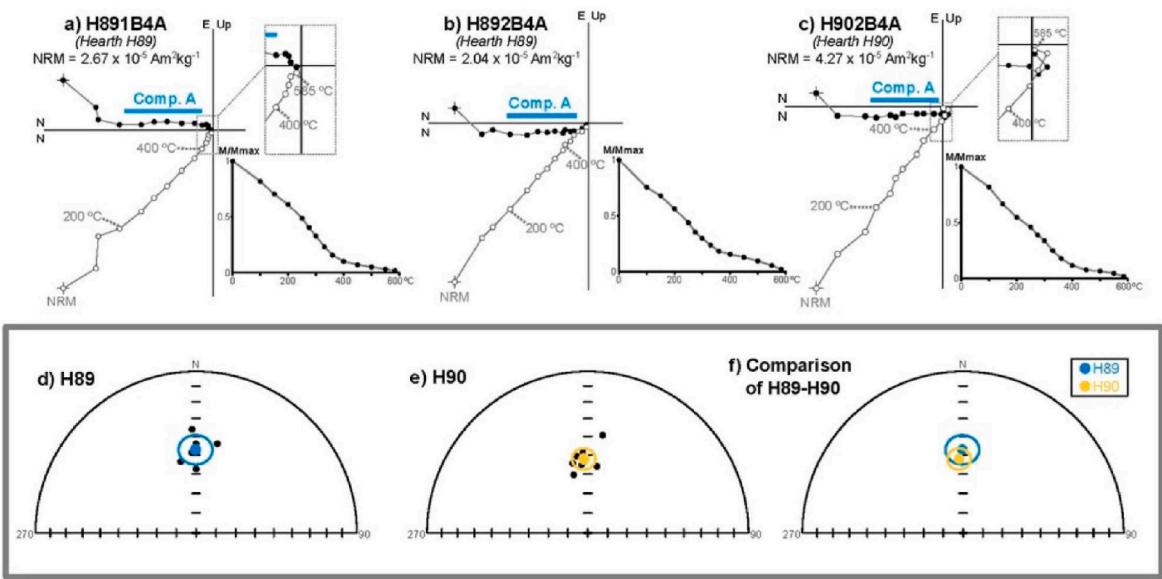


Fig. 6. Archaeomagnetic results (a-c) Examples of orthogonal NRM demagnetization diagrams from H89 and H90. Open (closed) symbols represent the vertical (horizontal) projections of vector endpoints. Sample code, intensity (NRM_0), and the intensity decay are indicated for each sample; (d-e) stereograms with all ChRM directions along with the mean archaeomagnetic directions and α_{95} of H89 and H90 hearths. (f) comparison of the mean archaeomagnetic directions of H89-H90. See Table 2.

Table 2

Summary of the main statistics parameters of the mean archaeomagnetic directions of H89 and H90 (calculated according to Fisher's 1953 statistic).

Hearth	N	N'	Dec. (°)	Inc. (°)	k	α_{95}
H89 (MAD & cut-off filter)	6	8	359.5	47.2	72.2	7.9
H90 (MAD & cut-off filter)	8	9	356.9	52.1	98.8	5.6

(N = number of specimens considered for the calculation of the mean direction; N' = total number of demagnetized specimens; Dec. = declination; Inc. = Inclination; k = precision parameter; α_{95} = semi angle of confidence at 95% probability.

depositional or environmental processes.

4.1. Hearth or hearths

Based on the archaeomagnetic results and the similarity of micromorphological components, there is no compelling evidence to support the interpretation that H89 and H90 represent two separate hearths. Archaeomagnetic results alone are not conclusive. Mean archaeomagnetic directions exhibit overlapping α_{95} (Fig. 6f). This does not prove the existence of a single hearth, since it could be two different fires burnt within a time frame too short for the direction of the Earth's

Magnetic Field (EMF) to have significantly changed or at different times in which the EMF's direction was the same. But neither do they prove that these are two different fires.

Stratigraphic features associated with ash layers in micromorphological samples 22-4 and 22-5 are also present in the fragment analyzed from sample 22-1 (Fig. 2 see Salt-22-1). The micromorphological evidence therefore does not support the presence of multiple, discrete combustion structures. If we consider this evidence along with the overlapping α_{95} of the mean archaeomagnetic directions, there is no compelling evidence to support the interpretation that H89 and H90 represent two separate hearths. Nevertheless, the hypothesis of two hearths cannot be completely ruled out either.

To further investigate this issue would require a detailed archaeostratigraphic and lithic refitting study (e.g. Mayor et al., 2025), in conjunction with the present dataset. Nevertheless, this is beyond the scope of the present work.

4.1.1. Mechanical integrity of the hearth

Micromorphological evidence indicates that the hearth represented by the H89-90 complex is well preserved. It closely resembles in situ combustion features documented at El Salt (Mallol et al., 2013; Leierer et al., 2020; Herrejón Lagunilla et al., 2024; Dinçkal et al., 2024b), exhibiting the characteristic stratigraphic sequence of a basal black layer

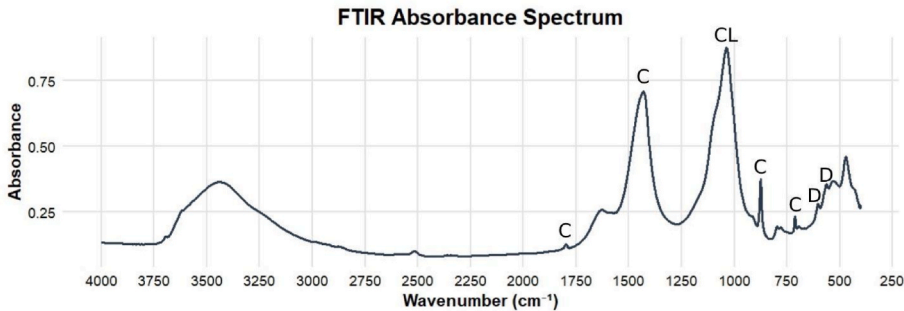


Fig. 7. Typical FTIR spectra from Hearth 89/90 White Layer at El Salt. C denoted Calcite diagnostic peaks, identified at 1798 cm^{-1} , $\sim 1425\text{ cm}^{-1}$, $\sim 875\text{ cm}^{-1}$, $\sim 718\text{ cm}^{-1}$. CL denoted diagnostic of clays at peak position of $\sim 1036\text{ cm}^{-1}$, and D denoted peaks diagnostic of Dahllite at $\sim 603\text{ cm}^{-1}$, and $\sim 562\text{ cm}^{-1}$.

rich in burned organic matter overlain by a white ash layer.

There is no micromorphological indication of mechanical disturbance. The substratum shows no signs of trampling, such as crushed or disarticulated bone, or mixing between the combustion and ash horizons (Miller et al., 2010). Most bone fragments appear articulated in the Black Layers (Table 1), particularly the largest bone fragment which shows no evidence of crushing. The contact between the Black and White layers remains intact, with no evidence of ash deformation or sediment mixing. The preservation of articulated ash (Table 1) in the White Layers further supports this interpretation (Fig. 3A; Mentzer, 2014; Berna et al., 2012).

The clayey red aggregates (Figs. 3B and 5E) are present in the upper portions of the ash layer. While their irregular morphology could suggest bioturbation (e.g. by insects or roots), no direct micromorphological evidence supports this interpretation, only the aggregates themselves are visible. Quantum Diamond Microscopy (QDM) data indicate that the distribution of magnetic fields within these red aggregates is indistinguishable from the surrounding ash in both ferromagnetic and paramagnetic components (Fig. 5E II and III). In contrast, areas with known material differences such as; coprolites in the black layer (Fig. 5A. II and III), highly magnetic iron nodules (Fig. 5B. II and III), or non-magnetic limestone (Fig. 5D II and III), exhibit visual distinctive distributions and frequency of magnetic fields. This uniformity suggests that the red aggregates entered the hearth prior to or during combustion, rather than as post-depositional intrusions.

Paramagnetic analysis provides further support. Hysteresis loop data and AC susceptibility show that samples with higher ash content show less influence of paramagnetic material in relative terms (Fig. 4A and D, Supp. Figs. 5–13). If bioturbation or post-depositional mixing had introduced external material into the ash, a higher correction could be expected. That this correction remains low supports minimal external input. Given the ash layer's inherent vulnerability to disturbance (Karkanas, 2021), its integrity, thickness and lateral continuity strongly suggests that the underlying black layer is also well preserved. Paramagnetic correction may therefore serve as a useful proxy for assessing mechanical integrity in combustion features, however further experimental investigations can clarify this point.

Archaeomagnetic (directional) results reinforce these conclusions. The low scatter and high values of precision parameter k in both H89 and H90 mean directions (Fig. 6d-e; Table 2) support the interpretation that the hearth remained structurally stable and undisturbed.

4.1.2. Diagenetic and chemical integrity

Rock magnetic analyses indicate that maghemite is a major primary carrier of the magnetic signature along with magnetite. Although maghemite is observed in all facies, it is particularly evident in the ashes. The black layer sample collected as H89-90 (Supp. Figs. 22–23) has Curie temperatures close to 580 °C, indicative of magnetite. This finding diverges from previous observations at El Salt and other archaeological sites, where magnetite has typically been considered the primary carrier (Leierer et al., 2020; Dinçkal et al., 2024a; Herrejón Lagunilla et al., 2024), although certain maghemitization and/or slightly oxidized magnetite has previously been noted within combustion features (Dinçkal et al., 2024b; Herrejón Lagunilla et al., 2024).

This raises several broader questions: Is maghemite specific to this hearth, reflecting unique depositional or post-depositional processes? Does it reflect broader site-wide conditions at El Salt that have previously gone unobserved at this level of detail due to differences in observation scales and methodology employed between this and prior studies? While our current dataset cannot fully resolve these site-wide or methodological issues, it does allow us to evaluate the diagenetic history of the hearth itself.

A key issue is whether the maghemite formed syn-depositionally, during combustion, cooling, or shortly after, when iron minerals stabilize. Rapid formation within ten days has been demonstrated as a direct result of thermokinetic reactions during combustion (Jordanova et al.,

2019). Alternatively, the formation of maghemite could be entirely post-depositional, and not be reflective of the time or process of combustion, occurring after the deposit has formed and stabilized. This could be an environmental and chemical changes within the soil leading to post-depositional inputs such as chemical changes via water movement, or oxygen infiltration from subaerial exposition which can occur months or years after the deposit has occurred (Thompson and Oldfield, 1986; Herries, 2006; Jordanova et al., 2019). This distinction is crucial because it has implications for when magnetic remanence was recorded in prehistoric fires as well as our understanding of their possible post-depositional alteration. If maghemite formed post-depositionally, it could be assumed that magnetic signatures and micromorphological evidence of redoximorphic processes would occur unevenly through the combustion feature. But micromorphological redox features are largely absent in this feature and the archaeomagnetic directions are well-grouped indicating a snapshot record of the Earth's magnetic field direction.

Some iron nodulation is visible in the black layers, primarily the discrete iron nodule seen in Fig. 5B. However, with its sharp contact with the surrounding sediment, no evidence for additional iron impregnation of the surrounding sediment, and abrupt magnetic contrast seen in the QDM, this nodule is likely formed prior to the formation of the hearth and does not indicate post-depositional iron nodule formation. We also observe no substantial ferric hypocoatings or fabric impregnation features consistent with redoximorphic processes originating from this nodule or elsewhere. This strongly indicates that redox processes did not play a significant role in altering the magnetic mineralogy.

Support for a syn-depositional formation of maghemite comes from archaeomagnetic directional analysis, which reveals strong directional consistency across most studied specimens. The thermal demagnetization diagrams of the NRM obtained in the black layers show partial thermoremanences up to 450 – 500 °C (e.g. Fig. 6a-c). We cannot completely exclude that the identified maghemite, a type of oxidized magnetite, was formed time after burning, particularly within the ashes. However, in terms of the archaeomagnetic record, this is irrelevant since the ancient Earth's magnetic field direction was determined from the BL facies, with magnetite as main carrier. Nevertheless, the fact that well-clustered mean directions of the Earth's magnetic field were recorded in the burned facies (black layers) of both hearths, strongly suggests that magnetite and/or maghemite, formed during the cooling process. Regardless of whether the magnetization mechanism is of thermal (TRM or a p-TRM) or thermochemical (TCRM) origin, the directional fidelity is out of doubt as all these processes take place during cooling (Dunlop and Özdemir, 1997: 410).

Further evidence for the maghemitization occurring during the cooling of the combustion feature can be found in the variation of the maghemite signature between measurements of the RT magnetic analysis (see Fig. 4C). The diagnostic maghemite hump is the most moderate in Salt-22-5-3 and most prominent in Salt-22-5-1B. Salt-22-5-3 (black layer) represents carbonized material not fully combusted at the same temperatures that would have formed the calcined ash layers. In addition, this unit would have been insulated from the atmosphere with the build of ash, minimizing the available oxygen which would have been consumed by the hotter white layer microfacies above it. By comparison Salt-22-5-1B represents ash formation with great access to oxygen during combustion and cooling, promoting stronger maghemitization of the ferric component as seen by the more pronounced diagnostic hump within the RT curves (Fig. 4C).

While magnetite and/or maghemite are the dominant carriers of this remanence, it should be noted that the applied field limits of the IRM acquisition and backfield measurements (1.8 and 2.5 T respectively) are insufficient to saturate high-coercivity phases, meaning that a minor antiferromagnetic contribution such as hematite cannot be excluded. Indications of such a component are visible in the coercivity unmixing results (for example in Supp. Fig. 14) as a potential minor component,

though its mineralogical contribution is effectively masked by the strong magnetization capacity of the dominant ferrimagnetic phases. It is important to note that the addition of a third component in the unmixing models may be a product of overfitting (Kruiver et al., 2001; Maxbauer et al., 2016), making it difficult to verify whether this component is real. Nevertheless, antiferromagnetic minerals such as hematite are a consistent feature of combustion features, occurring primarily in rubified layers but also present in black and white layers (Dinçkal et al., 2022, 2024a).

This location does show evidence of diagenesis. Localized calcite dissolution (Fig. 3C) is visible micromorphologically at the base of the white ash layer, indicating mild bottom-up diagenesis where the ash interfaces with the underlying organic-rich combustion layer. This appears to be a uniform decalcification occurring in all thin sections at the base of the ash deposit, however this decalcification appears limited to contact interfaces. Calcite remains intact in the upper white layer across the entire higher portion of the combustion feature, without evidence of redox features, calcite recrystallization, or severe bone and limestone dissolution to explain the decalcification occurring at contact.

A potential explanation for the localized decalcification is by water percolation solely into the lower levels of the combustion feature through the presence of animal burrows. Animal burrows (Fig. 2, Salt-22-1) are present consistently across which show evidence for water movement in the form of authigenic crystal coatings within the voids of the burrow (as in Salt-22-1, see Supp. Fig. 25). FTIR identification of dahllite throughout the ash sequence somewhat reinforces that diagenesis is occurring, as dahllite is often cited as a by-product of the diagenetic pathway of calcite (Karkanis et al., 2000). However, dahllite is also ubiquitous at El Salt, reported repeatedly (Mallol et al., 2013) and present in our own control samples (FTIR spectra provided with the Supplementary Material), which likely indicates that its formation processes are site-wide and not unique to this hearth. Either way, in this situation the dissolution of the ash calcite would be post-depositional to the fire, occurring syn-depositionally with the burrow formation as water was able to penetrate into the deposit. Dating the calcite diagenesis is difficult because it depends on the formation of a burrow that is hard to place chronologically. The burrow likely cuts through much of the unit, increasing water percolation and material deposition over time, which further obscures when the diagenesis occurred relative to the combustion feature. While the burrow may have facilitated calcite diagenesis by increasing moisture and material movement, an alternative explanation is the interaction between organic matter in the upper black layer and unstable calcite, particularly under gradual moisture intrusion. This would suggest that diagenesis occurred closer in time to the combustion event, before deeper burial within the deposit.

Regardless of its cause, the diagenesis has not altered the top of the ash layer, nor does not appear strong enough to dissolve calcium-rich materials like bone, tufa, or limestone throughout the combustion feature. This suggests it is largely restricted to the hearth's unstable ash-derived calcite and has not significantly affected other key components such as iron compounds and oxides relevant to this study. These chemical changes have not disrupted the magnetic properties of the combustion feature, as evidenced by three key points: (1) the persistent dominance of the magnetic signature throughout the ash layer, (2) QDM maps showing no significant shifts in magnetic fabric localization between ash units, and (3) the rock magnetic analysis indicating a strong superparamagnetic domain (SP) signature. SP ferrimagnetic grains (0.01 – 0.03 μm) are the finest and most sensitive particles to alteration; if water movement had significantly impacted the magnetic signal, the SP assemblage would be the first to show degradation or they would have simply disappeared.

Despite the magnetic mineralogical differences between the ashes (maghemite) and the BL (magnetite), which likely reflect distinct authigenic pathways during combustion, there is no evidence that localized chemical changes have disrupted the magnetic mineralogy, nor have they compromised the archaeomagnetic directional record.

Together, these lines of evidence highlight that, although some diagenetic alteration is present within the calcitic ash material, the magnetic mineralogy and overall structure of H89–90 exhibit good physical preservation.

4.2. Anthropogenic interpretations

The field morphology and micromorphology of the H89-90 combustion feature suggest a single-use fire, or possibly a brief sequence of uses without significant intervening deposition. This is supported by the thinness of the deposit and the absence of re-lighting indicators or carbonized fuel remnants within the ash (Leierer et al., 2020; Karkanis, 2021). As with other El Salt combustion features (Mallol et al., 2013; Leierer et al., 2020; Dinçkal et al., 2024a; 2024b; Herrejón-Lagunilla et al., 2024), this fire appears to have been lit directly on an organic-rich substrate, with woody fuel (evidenced by the calcitic wood ash layer) placed directly onto it. There is no evidence of post-use human action: no micromorphological signs of trampling, raking, or cleaning appear, and the fragile ash matrix is exceptionally well preserved (Miller et al., 2010; Mentzer, 2014; Berna et al., 2012). Articulated ash and bones are present, with minimal fracturing, indicating that the hearth remained structurally undisturbed.

The Black Layers exhibit clear micromorphological evidence of grass and fibrous organic fuels being burned rather than wood. This aligns with previous observations at El Salt, which suggest that combustion events were conducted directly on the topsoil, igniting pre-existing surface materials (Mallol et al., 2013; Leierer et al., 2020; Dinçkal et al., 2024a; 2024b). Within the ashes there doesn't seem to be preserved evidence for the use of large wood in the construction of the fire, as woody charcoal is absent and microcharcoal is very fine. It is likely that once again grass or leaf fuels were utilized in the primary combustion events which is reinforced by the presence of articulated ash and phytoliths (Fig. 3A; Berna et al., 2012). The aforementioned red aggregates found within the ash layer could also be evidence of this. QDM analysis shows these aggregates do not seem to delimit from the ash in the distribution of magnetic field within the sedimentary fabric, suggesting they were introduced prior to or during combustion but ultimately underwent a similar level of thermal alteration as the surrounding ash. Their morphology and composition are similar to rubified soil present at El Salt (Leierer et al., 2020; Dinçkal et al., 2024a), which could indicate their presence as soil fragments adhering to roots, grasses, or are trapped among leafy debris which may have been gathered from the immediate surroundings (Goldberg et al., 2009). The fact that these aggregates occur higher within the ash unit suggests that these grassy, leafy or root fuels sources were added after the fire had already begun, potentially as a means of maintaining combustion by gathering material lying around. This might imply opportunistic fuel use, possibly from nearby vegetation, rather than reliance on stockpiled fuel resources.

4.3. Applying quantum diamond microscopy

One of the key strengths of QDM applied to Geoarchaeology lies in its usefulness to distinguish between anthropogenic and environmental features, as well as pre-depositional, syn-depositional, and post-depositional characteristics, through integration with micromorphological analysis. It is particularly effective in resolving complex combustion structures, where the different sources of a microfacies' magnetic signature can be identified. For example, QDM can differentiate between a magnetic signal originating from a pre-depositional iron nodule, in situ variations in the distribution of magnetic fields which vary between material with distinct sedimentary fabrics (also helping us to establish whether those fabrics are related or not), and an allochthonous soil fragment formed prior to deposition. QDM also aids in microfacies characterization by informing of magnetic mineral composition within the sediment. In the ash layers studied here, it revealed

evenly dispersed superparamagnetic components throughout the sediment, indicating a consistent, combustion-derived origin even in a context where the ash was altered. In contrast, thermally altered combustion substrate layers (black layers) display more heterogeneous and clustered magnetic fields, reflecting a higher degree of variability in the spatial distribution. This heterogeneity, when reinforced with the rock magnetic study, indicates that the magnetic mineralogy and associated underlying formation processes were more complex than those observed in the overlying ash layers, which are characterized by more uniform high-temperature alteration and formation. When interpreted with the use of micromorphology, these subtle features become more legible within their context, deepening our understanding of both behavioural and depositional dynamics.

Perhaps most importantly, the integration of QDM and micromorphology enhances process-oriented interpretation of how magnetic minerals integrate into the sedimentary fabric. QDM resolves internal magnetic contrasts that distinguish sedimentary inclusions: for instance, coprolites and limestone fragments found within the black layers are magnetically distinct from their surrounding matrix. In the case of diamagnetic in nature limestone (see Fig. 4D for an example from the Ash layer), the magnetic mineral content is notably low, as evidenced by the low frequency of magnetic fields observed in the QDM maps. Consequently, thermal alteration does not significantly enhance its magnetic properties, rendering the limestone magnetically distinct from the surrounding sedimentary magnetic fabric. The coprolite (Fig. 4A), which compositionally contains clays, organic matter, and minor ferric components, exhibits weak and infrequent magnetic fields in the QDM maps. While some magnetic activity is present, it is markedly less intense and less frequent than that of the surrounding sediment. This pattern suggests that the magnetic signature of the coprolite was not as affected by the combustion event, likely due to uneven heating and limited heat penetration deeper into the black layer. By comparison, red soil aggregates within the ash layers exhibit no notable relief in the density or distribution of magnetic fields, as shown in the QDM maps. Any spatial variation of these fields does not correspond to the sedimentary contact between the red clayey aggregates and the surrounding ashy sedimentary structures, as it does in limestone or the coprolites in the black layers. This suggests that the mineralogy forming the magnetic fields within these red soils along with the ash were either similarly altered or formed during the combustion event responsible for the ash layer. These contrasts provide valuable insight into sediment formation, deposition, and post-depositional alteration, directly linking the locations in which magnetic fields are generated to both human activity and environmental context.

Despite its strengths, QDM faces limitations. Chief among them are high cost and limited accessibility. As a relatively new method in geoarchaeology, few institutions possess the equipment or expertise needed. However, the development of QDMs as a laboratory tool has advanced considerably in recent years (Shaji et al., 2025a, Shaji et al., 2025b). These advances are expected to make the equipment more compact and user-friendly within standard soil and geoscientific settings, which in turn will reduce both costs and exclusivity. As a result, QDM is likely to become an increasingly viable option for targeted micromorphological research, particularly in contexts involving combustion features or iron-rich sediments.

Another challenge is the absence of established reference datasets for archaeological contexts. Without clear precedents for how various sediment types or microfeatures appear under QDM, interpretations remain cautious and are most robust when supported by complementary analytical methods. In its current state of development, QDM in archaeological science should be understood as a tool that derives its interpretive value primarily through integration with rock magnetic and micromorphological analysis, rather than as a standalone method. The magnetic field maps it produces are most meaningful when grounded in an established understanding of the magnetic mineralogy and sediment microstructure of the material being studied. For example, paramagnetic

maps cannot distinguish between paramagnetic grains and minerals within the superparamagnetic particle size range. This ambiguity can significantly complicate interpretations of the underlying processes forming or altering different microfacies. This underscores the importance of complementary rock magnetic analysis at the microfacies level to clarify which mineralogical components are the primary drivers of map signal.

To fully realise QDM's potential within geoarchaeology and soil micromorphology, substantial future research is required. This includes the development of comprehensive reference databases covering a range of archaeological sediment types derived from a wide variety of human behaviours and processes, its integration with as broad a range of rock magnetic methods as possible, and a clear intention to sample material in a standardised manner for QDM use from the outset of any study. Establishing how QDM signatures correspond to specific mineralogical and sedimentological processes across a range of site types and depositional contexts is important as the method is applied with greater interpretive confidence. These steps are essential for QDM to become a reliable, cross-disciplinary tool in archaeology and soil science, and the present study should be understood as an early contribution toward that longer-term goal.

4.4. Summary and conclusions

This study presents a high-resolution, multi-method reconstruction of a Middle Palaeolithic combustion feature, showing how integrated micromorphology, chemistry, archaeomagnetism, rock-magnetism, and magnetic imaging advance archaeological interpretation and geoarchaeological methodology.

The analysis provides evidence for the types of anthropogenic processes that played a role in the hearth 89/90's construction, use, and preservation. The consistent magnetic mineralogy, magnetic direction, intact stratigraphy, and micromorphological coherence point to it being a single hearth formed with deliberate human fuel management, and potential use of opportunistic fuel gathering from around the site. However, further evidence is needed to definitively confirm that this is a single structure. The presence of articulated ash, fuel-related soil aggregates, and minimal evidence of post-depositional disturbance suggest purposeful fire maintenance and limited post-use modification.

Maghemite has been observed mainly in the ashes, while magnetite is the main remanence carrier in the BL facies of the H89-90 complex. The evidence strongly supports a syn-depositional origin of maghemite, likely formed during the hearth's cooling phase rather than through later diagenetic transformation. This raises questions for future research into the mechanisms of maghemite formation during and immediately following combustion, which may provide valuable information about the anthropocentric processes and environmental conditions underlying the hearth use. In any case, the magnetic mineralogy and the stability of the archaeomagnetic direction are reliable, and together with the micromorphological evidence, reinforce the high preservation quality of the structure.

Quantum Diamond Microscopy (QDM) played a critical role in identifying these patterns. This technique facilitates a direct integration of micromorphology and rock magnetism, enabling the precise identification of interactions between magnetic fields, minerals, and sedimentary fabrics. As a result, it enhances the interpretation of syn-depositional components by distinguishing them from potential contaminants, clarifying the timing of sedimentary formation associated with magnetic minerals, and isolating unique anthropogenic signatures and features. However, it should be emphasized that the interpretive value of QDM in this context is contingent upon its integration with rock magnetic and micromorphological analysis. The present study represents an early step in the longer-term development of QDM as a tool with geoarchaeology and archaeological sciences.

CRediT authorship contribution statement

Ada Dinçkal: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Visualization, Writing – original draft, Writing – review & editing. **Joshua Feinberg:** Formal analysis, Methodology, Supervision. **Ángela Herrejón Lagunilla:** Formal analysis, Methodology, Writing – original draft, Writing – review & editing. **Dimitri Papavasiliou:** Formal analysis, Investigation, Writing – review & editing. **Angel Carrancho:** Conceptualization, Formal analysis, Investigation, Methodology, Writing – review & editing. **Cristo M. Hernández Gomez:** Funding acquisition, Investigation, Project administration, Supervision, Writing – review & editing. **Carolina Mallol:** Conceptualization, Formal analysis, Funding acquisition, Investigation, Methodology, Supervision, Writing – review & editing.

Declaration of competing interest

We declare that there are **no conflicts of interest** associated with this research or the preparation of this manuscript.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jas.2026.106612>.

Data availability

All data utilized in this study is present within the published text and figures, or provided in the supplementary material.

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