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Innovative ochre processing and tool use in China ~40,000 years ago

Fa-Gang Wang¹[†], Shi-Xia Yang^{2,3,4,5}[†]* Jun-Yi Ge^{2,3}, Andreu Ollé^{6,7}, Ke-Liang Zhao^{2,3}, Jian-6 Ping Yue⁸, Daniela Eugenia Rosso^{9,10}, Katerina Douka^{4,11}, Ying Guan^{2,3}, Wen-Yan Li¹, Hai-7 Yong Yang¹², Lian-Qiang Liu¹, Fei Xie¹, Zheng-Tang Guo¹³, Ri-Xiang Zhu¹⁴, Cheng-Long 8 Deng^{14,15}*, Francesco d'Errico^{16,17}*, Michael Petraglia^{4,18,19,20}* 9 10 ¹Hebei Provincial Institute of Cultural Relics and Archeology, Shijiazhuang 050031, China. ²Key Laboratory of Vertebrate Evolution and Human Origins, Institute of Vertebrate 11 Palaeontology and Palaeoanthropology, Chinese Academy of Sciences, Beijing 100044, China. 12 13 ³Center for Excellence in Life and Palaeoenvironment, Chinese Academy of Sciences, Beijing 14 100044, China. ⁴Department of Archaeology, Max Planck Institute for the Science of Human History, Jena 15 07745, Germany. 16 ⁵State Key Laboratory of Loess and Quaternary Geology, Institute of Earth Environment, 17 18 Chinese Academy of Sciences, Xi'an 710061, China. 19 ⁶Institut Català de Palaeoecologia Humana i Evolució Social (IPHES-CERCA), Zona Educacional 4, Campus Sescelades URV (Edifici W3), 43007, Tarragona, Spain. 20 ⁷Universitat Rovira i Virgili, Dept. d'Història i Història de l'Art, Av. Catalunya 35, 43002, 21 22 Tarragona, Spain. ⁸Department of History, Anhui University, Hefei 230039, China. 23 ⁹Université Côte d'Azur, CNRS, CEPAM, Nice, France. 24 ¹⁰Departament de Prehistòria, Arqueologia i Història Antiga, Grupo de Investigación Prehistoria 25 del Mediterráneo Occidental (PREMEDOC), Universitat de València, Valencia, Spain. 26 ¹¹Oxford Radiocarbon Accelerator Unit, Research Laboratory for Archaeology and the History 27 28 of Art, University of Oxford, Oxford, OX1 3TG, UK. ¹²Museum of Yuzhou, Yuxian 075700, China. 29 ¹³Key Laboratory of Cenozoic Geology and Environment, Institute of Geology and Geophysics, 30 Chinese Academy of Sciences, Beijing 100029, China. 31 ¹⁴State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese 32 Academy of Sciences, Beijing 100029, China. 33 ¹⁵College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, Beijing 34 35 100049, China.

- ¹⁶PACEA UMR 5199, Université de Bordeaux, CNRS, Pessac, France.
- ¹⁷SFF Centre for Early Sapiens Behaviour (SapienCE), University of Bergen, Bergen, Norway.
- ¹⁸Human Origins Program, National Museum of Natural History, Smithsonian Institution,
 Washington, D.C. 20560, USA.
- 40 ¹⁹School of Social Science, The University of Queensland, Brisbane, Queensland 4072, Australia.
- 41 ²⁰Australian Research Centre for Human Evolution (ARCHE), Griffith University, Brisbane,
- 42 Australia.

- 43 [†]These authors contributed equally to this work.
- * Corresponding authors. yangshixia@ivpp.ac.cn (S.X.Y.), cldeng@mail.iggcas.ac.cn (C.L.D.),
 francesco.derrico@u-bordeaux.fr (F.D.), petraglia@shh.mpg.de (M.P.).
- 46

47 Homo sapiens was present in northern Asia by ~40,000 years ago, having ultimately replaced 48 49 archaic populations in Eurasia after episodes of earlier population expansions and interbreeding¹⁻⁴. Cultural adaptations associated with the last Neanderthals, Denisovans and the 50 incoming populations of *H. sapiens* into Asia remain elusive^{1,5-7}. Here, we describe Xiamabei, a 51 well-preserved, ~40,000-year-old archaeological site in northern China, containing the earliest 52 53 known ochre processing feature in Eastern Asia together with a distinctive miniaturized lithic 54 assemblage, with bladelet-like tools bearing traces of hafting, and a bone tool. The cultural 55 assembly of traits at Xiamabei is unique for Eastern Asia and does not correspond with those found at other archaeological site assemblages inhabited by archaic populations or to those 56 generally associated with the expansion of *H. sapiens*, such as the Initial Upper Palaeolithic^{8–10}. 57 The record of northern Asia supports a process of cultural diversification and innovations 58 emerging in a period of hominin hybridization and admixture^{2,6,7,11}. 59

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62 Main Text

63 One of the most profound events in human evolution was the worldwide expansion of *Homo* sapiens^{1,5,12}. Fossil, genetic and archaeological evidence indicates that *H. sapiens* dispersed out 64 of Africa multiple times over the last ~200 thousand years $(ka)^{1,13}$, interbreeding with archaic 65 hominins, such as the Neanderthals and Denisovans as they migrated across Eurasia^{6,8,14}. Current 66 palaeoanthropological and archaeological evidence demonstrates that H. sapiens was present in 67 northern Asia by at least 40 ka ago^{2-4} . It is often argued that the terrestrial expansion of modern 68 humans was facilitated by the use of advanced economic, social and symbolic adaptations 69 allowing occupation of a variety of ecosystems¹⁴. Pigment use, in particular, is seen as a key 70 indicator of symbolically mediated behaviour^{15–17}, and technological innovations, such as the use 71 of miniturised technology, is considered to have had adaptive and economic advantages^{18,19}. 72 Archaeological data, however, remain ambiguous as to the arrival of *H. sapiens* populations in 73 74 China (Supplementary Information A). With the exception of Initial Upper Palaeolithic (IUP) and Mousterian toolkits in the Altai and Siberia, and a few sites in northern China^{20–22}, little is 75

76 known about stone tool industries in Eastern Asia until microblade assemblages become the dominant technology after 29 ka^{9,23,24}. With respect to possible evidence for early symbolism, 77 78 only two engraved bones are known, one bearing residues of ochre, from northern China (ca. 125–105 ka), interpreted as the product of archaic hominins²⁵. Most artefacts signaling symbolic 79 practices are much younger, and likely linked to *H. sapiens*, such as pendants and ochre at 80 Zhoukoudian Upper Cave (ca. 35.1-33.5 ka)⁴ and beads at Shuidonggou 2 (SDG 2) (ca. 31 ka)²³. 81 Beads, pendants, and figurines become common after ca. 29 ka, when microblade technology 82 began to spread across northern China²⁶⁻²⁸. 83

Here we report the results of archaeological findings at Xiamabei, a newly excavated and 84 well-preserved site in the Nihewan Basin of northern China (Fig. 1). Xiamabei contains the 85 86 earliest evidence of ochre processing, a novel miniaturized lithic technology, with bladelet-like forms and hafted items, and a bone tool, dating to \sim 41–39 ka. Xiamabei stands apart from any 87 other known archaeological site in China as it possesses a novel set of cultural characteristics at 88 an early date. Located at the transition zone between the Inner Mongolian Plateau and North 89 90 China Plain, the site offers important new insights in the expansion of *H. sapiens* along the northern route¹⁰. 91

Xiamabei is on the southern bank of the Huliu River, where excavations were initiated in 92 2013 in the form of a trench exposing a 12 m^2 area (Supplementary Information B). The 93 94 stratigraphy, extending to a depth of 290 cm, encompasses six main layers in a floodplain environment (Fig. 2). Layer 6, the main cultural horizon, 10-20 cm in thickness, is composed of 95 dark brown silty sediment, with occasional clay and sand aggregates. Layer 6 revealed evidence 96 for ochre use and processing, a charcoal-rich hearth, 382 miniaturized lithics, a single bone tool 97 (Extended Data Fig. 1) and 437 mammal bones (Fig. 1b-d). Characterization methods were 98 applied to sediment samples, ochre items, the bone tool, and a sample of the miniaturized lithics. 99 Accelerator mass spectrometry (AMS) ¹⁴C and optically stimulated luminescence (OSL) dating 100 yielded ages ranging between ca. 43 ka to 28 ka for the sequence, with the Layer 6 cultural 101 horizon dating to ca. 41–39 ka (Fig. 2, Supplementary Information C). 102

Pollen from Layer 6 indicates a steppe landscape with patches of coniferous forest
dominated by *Pinus*. The pollen assemblages and high *Artemisia*/Chenopodiaceae (A/C) ratio in
Layer 6 indicate a relatively cool and semi-arid climate during the site occupation
(Supplementary Information D). Faunal remains were highly fragmentary, with only 7.19%

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107 being taxonomically identifiable. The presence of horse, deer and zokor is consistent with the 108 pollen evidence, reflecting a steppe landscape with patches of forest (Supplementary Information 109 D). The bulk of the mammalian fossils were less than 20 mm in length and most were burnt, some even highly carbonized, suggesting their possible use as fuel (Supplementary Information 110 D2). Cutmarks were identified on two faunal fragments indicating that lithics were used to 111 process carcasses. A single bone tool bearing traces of use wear in the form of microflake scars 112 and polishing at one end, and regularization by scraping at the other end, possibly to facilitate 113 prehension or hafting, was recovered from Layer 6. The tool represents one of the earliest 114 examples in northern China for the working of bone with techniques proper to this raw material 115 (Extended Data Fig. 1). 116

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118 Ochre processing evidence

Evidence for ochre processing at Xiamabei consists of three artefacts lying in close spatial 119 120 association and on a spot of red stained sediment whose colouration reduces in intensity on the objects (Fig. 3, Extended Data Figs. 2–5, Supplementary Information E and F). The three 121 artefacts (Figs. 3a, b) consist of an ochre piece (OP1) consisting of an allochthonous hard iron-122 123 rich nodule bearing clear traces of having been repeatedly abraded to produce a bright dark red ochre powder (Fig. 3c), a smaller and more friable ochre piece (OP2) of a different composition 124 (Fig. 3d), resulting from crushing an original larger piece to produce ochre powder, and an 125 elongated limestone slab (LS) with smoothed areas stained with ochre (Fig. 3e) and preserving 126 residues of hematite-rich ochre (Fig. 3f) similar to that composing the smaller ochre piece. No 127 ochre residues were identified on an associated quartzite cobble (QC) bearing evidence of use as 128 a pestle (Fig. 3g). 129

X-ray diffraction (XRD), Micro Raman spectroscopy (MR), Micro-X-ray fluorescence
(Micro-XRF), mineral magnetism (MM), scanning electron microscopy coupled with energy
dispersive spectroscopy (SEM-EDS) were applied to 12 sediment samples from inside and
outside the red stained area on which the ochre pieces and associated artefacts were lying
(Extended Data Fig. 6, Supplementary Information G). In contrast to samples located at various
distances from the red stained area, two samples from inside the area are rich in FeO and contain
abundant microfragments of hematite-rich rocks. This indicates that the reddish colour of the

sediment (on which the stone artefacts and the ochre fragments were laying) is due to thepresence of diffuse ochre particles.

139 Taken together, the evidence indicates that different types of ochre were brought to the site and processed using abrasion and pounding to produce ochre powder of different colour and 140 granulometry. Although the purpose of such an activity cannot be established (e.g., the 141 production of paint for colouring objects or decorating bodies, tanning of hides, using ochre as a 142 loading agent for adhesives), the quantity of ochre powder produced was large enough for the 143 leftover to permanently impregnate the sediment of the area on which tasks took place. This 144 work area, which represents the earliest known instance of ochre processing in Eastern Asi a^{28} , 145 indicates that the use of this material was part of the behavioural repertoire of regional 146 147 populations by ~40 ka, preceding the origin of microblade technologies in China by 10,000 years.

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149 Novel miniaturized lithic assemblage with hafting evidence

The Xiamabei lithic assemblage, composed of 382 artefacts, is a novel technology for northern 150 China, especially in consideration of its age. Most of the lithic artefacts (94%) are smaller than 151 152 40 mm and 58.37% (n=209) are smaller than 20 mm. Locally available small chert nodules and quartz pebbles dominate the lithic assemblage, though porphyry pebbles were also sometimes 153 154 used. Freehand Hard Hammer Percussion (FHHP) and Bipolar Percussion (BP) methods were the main reduction techniques, as shown by the coexistence of flake-cores with pronounced 155 156 Hertzian initiations and bipolar pieces with evidence of crushing on opposed platforms and diffuse bulbs of percussion (Supplementary Information H). BP is the predominant reduction 157 158 technique, contributing to 70.11% of the identifiable specimens. The maximum length and width of BP cores were not significantly smaller than the FHHP cores, indicating that although hand-159 160 held flaking of small cores was possible, preference was given to bipolar reduction. Bipolar splinters had an average width of 13.59 mm (29.5% smaller than 10 mm), falling within the 161 width range of formal microblades from younger sites in northern China²⁹. BP reduction 162 strategies therefore resulted in the efficient production of bladelet-like blanks that could be used 163 as tools, with lengths frequently measuring 20 mm or less (Fig. 4)³⁰. 164

In contrast to both earlier and contemporary lithic assemblages from the Nihewan
 Basin^{31,32} retouched tools are exceedingly rare at Xiamabei. Only three pieces in the Xiamabei
 assemblage show formal edge modification: two denticulates and one slightly retouched end-

168 scraper. Functional analysis conducted on a selected sample of lithics, including 13 bipolar and 4 169 FHHP products, identified use-wear on most of them (Supplementary Information I). Seven 170 pieces show clear evidence of hafting based on the arrangement of micro-scarring, the absence of use-wear on areas covered by the hafting material, and the frequent presence of plant fiber 171 imprints, pointing to the use of binding elements (Fig. 4). Bone hafts have been identified in two 172 cases and exceptionally illustrated by a bladelet-like piece recovered with a portion of the haft 173 still in place (Fig. 4a, Extended Data Figs. 7–9). Wear patterns indicate the hafted pieces were 174 175 used for a variety of purposes including hide scraping, boring and scraping against hard matter (likely wood), whittling soft plant material, and probably cutting soft animal matter. Two pieces 176 bearing an end-scraper outline were used in a manner consistent with classic Upper Palaeolithic 177 hide working tools, although only one of the implements showed formal distal retouch (Fig. 4b). 178 179 Use-wear on unhafted pieces complete the range of identified activities - mainly represented by 180 cutting actions on different materials, boring on a hard material, and in two cases, as wedges on quartz flakes. Overall, the microscopic analyses indicated that the Xiamabei artefacts, including 181 bladelet-like tools manufactured by the BP technique, were used in a variety of activities 182 (Extended Data Fig. 10). In addition, ten artefacts with ochre residues, mainly in the form of 183 184 dispersed particles, were identified. On four pieces, residues were associated with the hafted area, and on two pieces hide working was identified as residues were located on the active edge. 185 Ochre may have been used as an additive for hide working or as a loading agent for a hafting 186 adhesive. 187

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189 Implications for cultural adaptations in Eastern Asia

The Xiamabei site excavations and analyses identifies the appearance, ~40,000 years ago, of new 190 cultural features that were either unknown or exceedingly rare in this region and in the 191 neighboring regions. The combination of two knapping techniques for the production of small 192 blanks, used for a variety of tasks and sometimes hafted, indicates the existence of a complex 193 194 technical system involving the use and transformation of different raw materials. Such a 195 technical system, not identified at older and penecontemporaneous sites, gives the Xiamabei 196 assemblage an original character. A workshop for the production and use of mineral pigments at 197 Xiamabei constitutes a second new cultural element in comparison with earlier and contemporary sites. The use of ochre is not, however, associated with ostrich egg beads and 198

pierced teeth and shells found in younger sites, nor with the arrival, attested at younger sites, of
microblade technology. Although the taxonomic affiliation of the human group that occupied
Xiamabei ~40,000 years ago is not known, and an occupation by late Denisovans or even
Neanderthals cannot be excluded, the most parsimonious hypothesis, considering the presence of
contemporary fossils of modern humans at Tianyuandong², and somewhat younger at Salkhit³
and Zhoukoudian Upper Cave⁴, is that the visitors to Xiamabei were *H. sapiens*.

205 The way in which key cultural innovations emerged in different regions of the world remains unclear. For some regions, such as Africa, a discontinuous and regionally variable 206 accretion model has been proposed, indirectly linked to the long and complex process that has 207 led to the emergence of our species in that $continent^{33}$. It has been argued that packages of 208 209 innovations originally developed in Africa or elsewhere may have been introduced by the spread of modern humans into Eurasia. This is the model most often favoured for Asia, a region in 210 which, following European trends, blade and microblade technology, personal ornaments, ochre 211 use and complex bone technologies are seen as the signature of incoming *H. sapiens* populations. 212

The record emerging from northern China challenges dominant paradigms by showing 213 that during a critical time window, at ca. 40 ka, a variety of cultural adaptations existed. The 214 occurrence of a varied, though more simple lithic technology when compared to bladelet 215 216 production, associated with previously unrecorded hafting techniques, and the presence of some innovations (ochre use, an expedient bone tool), and not others (formal bone tools, ornaments), 217 218 suggest that the cultural adaptations at Xiamabei may reflect a first colonisation by modern humans, potentially involving cultural and genetic mixing with local Denisovans, and perhaps 219 220 replaced by a later second arrival. This supports the view that current evolutionary scenarios are simplistic and that we should expect repeated but differential episodes of genetic and cultural 221 222 exchange over large geographic areas. We should also expect to identify a mosaic pattern involving, in some instances, the spread of innovation packages, and in others, the persistence of 223 224 local traditions or the adoption of local invention of innovations with different rates of complexities in technological and symbolic practices. This more complex evolutionary scenario 225 226 fits better with current biological and cultural evidence in comparison to one that envisions a 227 spread of innovation associated with a single, rapid wave of *H. sapiens* populations across Eurasia. 228

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- 325 conducted field excavation and site sampling; J.Y.G., K.L.Z., K.D. and C.L.D. conducted
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- 330 contributions from the other authors.
- 331 **Competing interests** The authors declare no competing interests.
- **Data and materials availability** All relevant data are available in the main text or the
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- 334 Institute of Vertebrate Palaeontology and Palaeoanthropology, Chinese Academy of Sciences,
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- They are available for further research.

337 Supplementary Materials

- 338 Supplementary Information A–I
- 339 SI References



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Fig. 1 | Location of Xiamabei in the Nihewan Basin, China, and archaeological site

excavations. a, Location of Xiamabei in northern China, with regional map showing the

location of the site on the south bank of the Huliu River, Nihewan Basin. **b**, Distribution of

344 cultural remains on the Layer 6 surface. **c**, Ochre processing feature showing a quartzite cobble

- 345 (QC), an ochre piece (OP1) and a limestone slab (LS). **d**, View of the Layer 6 hearth, dated to
- **346** ~41–39 ka.

347





349 Fig. 2 | Stratigraphy, sedimentary sequence and the position of luminescence and

radiocarbon samples at Xiamabei. a, Main stratigraphic column. b, Layers identified in the
 field. c, Bayesian model of AMS ¹⁴C and OSL dates. The age probability distribution in blue, red
 and gray correspond to the OSL, AMS ¹⁴C and modelled ages, respectively.



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Fig. 3 | Artefacts laying on the red stained sediment patch. a, Quartzite cobble (QC),

limestone slab (LS) and ochre piece (OP1) identified during the excavation. **b**, LS laying on an

- intense red stained sediment patch. **c**, Ochre piece modified by grinding (OP1). **d**, Ochre
- 357 fragment probably resulting from crushing a larger ochre piece (OP2). **e**, LS showing ochre
- staining. **f**, Ochre microfragment (OMF) collected on the LS. **g**, Quartzite cobble (CG).



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Fig. 4 | Examples of use wear on lithics and residues on hafted pieces. a, Bladelet-like piece 360 361 with an adhering portion of the bone haft (no. 129); (1) detail of the bone; (2) imprints of plant 362 fibers on calcium carbonate concretions likely related to binding; (3) ventral view of the active edge; (4-7) several details of the grit-influenced plant polish produced by a whitling action. **b**, 363 End-scraper with punctual distal retouch (no. 385); (1) frontal view of the retouched area; (2) 364 spot with ochre particles on the active distal end; (3-5) scarring and slight adequation on the 365 hafted portion; (6-8) use wear produced by hide scraping. Images: a1-2 and b1, 3-6 3D Digital 366 Microscope; a3-6, b8 Scanning Electron Microscope; a7, b2, 7 Reflected Light Optical 367 Microscope. 368

369 Methods

370 Radiocarbon dating

Six animal bone fragments (all mammals) from Layers 2, 4 and 6 were collected and submitted to the Oxford Radiocarbon Accelerator Unit (ORAU) for radiocarbon dating. The bones were pretreated using routine methods for collagen extraction and purification, including an ultrafiltration step, as described in Ramsey et al.³⁴ and Brock et al.³⁴.

The dates are reported in radiocarbon years BP (Before Present - AD 1950) using the halflife of 5568 years. Isotopic fractionation was corrected for using the δ^{13} C values measured on the AMS. The quoted δ^{13} C values are measured independently on a stable isotope mass spectrometer (to ±0.3 per mil relative to VPDB). The new ¹⁴C determinations are calibrated using the INTCAL20 calibration curve³⁶ and the OxCal 4.4 platform³⁷, with the age ranges in Supplementary Information Table C1 expressed at the 95.4% confidence interval. The dating report is included in Supplementary Information C1.

382 Luminescence dating

Dating of sediments using optically stimulated luminescence (OSL) signals in mineral grains was 383 first introduced by Huntley et al.³⁸. The OSL dating technique mostly determines the time 384 elapsed since the last sunlight exposure of a deposit, i.e., its burial time. The technique has been 385 widely used to date geological and archaeological deposits³⁹⁻⁴² in recent decades. The 386 luminescence emitted from minerals (e.g., quartz and feldspar) under artificial light exposure is 387 proportional to the absorbed energy accumulated within the crystal lattice of minerals by ionizing 388 radiation (e.g., alpha, beta or gamma radiation) from radioactive elements such as uranium (U), 389 thorium (Th), and potassium (K) in the environment, as well as cosmic rays^{40,43}. The total 390 luminescence of a sample which is often calibrated as a radiation dose (termed the equivalent 391 dose, De), is determined by comparing natural luminescence signals with signals vielded after a 392 393 known laboratory irradiation dose. Calculating the rate of natural irradiation (dose rate) at which the sample absorbs energy from radiation in the environment during burial involves assessing the 394 395 radioactivity of the sample and its surroundings using chemical and radiometric methods, and estimating the radiation contributed by cosmic rays. The luminescence age of sediments is then 396

achieved by subdividing the equivalent dose (Gy) by the dose rate $(Gy/ka)^{44}$.

398 A total of 10 sediment samples (Supplementary Information Table C3) was collected in the 399 Xiamabei sequence for OSL dating. All OSL samples were obtained by hammering steel tubes (20 cm-long cylinders with a diameter of 5 cm) into a freshly dug vertical section. The tubes 400 401 were then covered and sealed with aluminum foil, and wrapped in black plastic bags and taped to avoid light exposure and moisture loss. The sediment from the sunlight-exposed end of the 402 403 cylinder was separated and used for dose rate measurement. The material from the middle of the cylinder was used for De measurement. Sample preparation, measurement and data analysis 404 procedures are described in Supplementary Information C2. 405

406 Bayesian modelling and age of the Xiamabei section

To establish the precise chronological framework of individual depositional units in Xiamabei, we conducted Bayesian analysis that includes both the optical and ¹⁴C ages, using OxCal v4.4 $^{45-49}$. All ¹⁴C ages were calibrated using the INTCAL 20 calibration curve. For each OSL age, we used C date in calendar years before 2020 when the samples were collected with associated 1 σ errors as likelihood estimates. In order to harmonise the time the OSL was obtained and the radiocarbon convention "BP=1950", we subtracted 70 years from the OSL ages (2020–1950) prior to them being modeled.

In order to estimate the posterior distributions (i.e., the modelled ages), the stratigraphic 414 order of each sample was input using the Sequence function (Supplementary Information Table 415 416 C4), based on the prior assumption that a sample stratigraphically lower is older than those above. Samples that are from the same stratigraphic layer with similar depth, such as in Layer 6, were 417 modelled as a Phase, in which the measured ages are assumed to belong to same period. For the 418 layers with abrupt and clear boundaries between them, such as Layers 6 and 5, double (upper or 419 420 lower) boundaries were placed for them to constrain their start or end ages. Otherwise, for layers which gradual stratigraphic changes were identified, assuming continuous sediment 421 422 accumulation, single transitional boundaries were placed between them. The samples, phases and 423 sequences were arranged according to their relative stratigraphic order.

To check for outliers, we applied the general t-type outlier model to detect outlier ages by assessing the likelihood of each age being consistent with the modelled ages. A prior outlier probability of 5% was assigned for the other samples with their posterior outlier probability

calculated during the modelling process. The CQL codes used to run the Bayesian model and 427 428 generate posterior age estimates are listed in Supplementary Information Table C5. The 429 generated probability distribution functions (PDF) for each of the samples are shown in Fig. 2 430 and their corresponding 95.4% probability ranges are summarized in Supplementary Information 431 Table C5. As demonstrated in Fig. 2, the Bayesian model results in a significant improvement of the precision in age estimates for all the samples. None of the samples was flagged as an outlier, 432 433 as indicated by the posterior outlier probabilities, which are less than 4% for all samples. Using a Date command we calculated the total span of each phase of interest, in particular, Layer 6 and 434 Layer 4. The model is shown in Supplementary Information C3. 435

436 **Ochre analysis**

Four artefacts were submitted to microscopic and geochemical analyses (Fig. 3): two ochre pieces (OP1 and OP2, Extended Data Figs. 2,3), a quartzite cobble (QC, Extended Data Fig. 4) and an elongated limestone slab (LS, Extended Data Fig. 5). During excavation, these four artefacts were found in close spatial association (Fig. 3a) and lying on a large red stained sediment spot (Fig. 3b). The cobble was cleaned under the microscope with a wet toothpick and a soft brush. The recovered sediment was analysed to check the presence of phytoliths (Supplementary Information F).

The four artefacts were examined with a motorised Leica Z6APOA microscope equipped 444 445 with a DFC420 digital camera and Leica Application Suite software (v.4.13, Wetzlar, Germany), equipped with the Multifocus module and Leica Map DCM 3D computer software (6.2, Wetzlar, 446 Germany). The Multifocus module allows the acquisition of extended depth of field images by 447 relying on adapted algorithms that combine digital images collected at different heights into a 448 single, sharp, composite image. The obtained microscopic images were digitized and edited in 449 the Adobe® Photoshop® CS5.1 Extended software. The Leica Map DCM 3D allowed 450 451 production of 3D reconstructions of areas of interest on OP1.

Anthropogenic modifications on grindstones and ochre pieces were identified macro- and microscopically. On grindstones, we recorded the presence of pits (depressions produced by a pounding action), and smoothed areas (surfaces that have lost, comparatively to neighbouring areas, irregularities and projections through abrasive action)^{50–52}. On ochre pieces, we identified polished areas (shiny surfaces that may have been produced by use or by rubbing against a soft 457 surface), striations (linear parallel marks arranged in groups produced by grinding the piece 458 against an abrasive surface), and facets (flat areas covered with striations produced by grinding 459 an object on a flat, hard abrasive surface)⁵³⁻⁵⁵.

An ochre microfragment (OMF, Fig. 3f; Extended Data Fig. 5) adhering to the surface of the limestone slab LS (zone 1 in Extended Data Fig. 5a) was removed with a scalpel under the microscope without producing any damage to the artefact and put on a carbon adhesive tab adhering to a SEM stub.

464 Scanning Electron Microscopy (SEM) observations and Energy Dispersive X-Ray spectroscopy (EDS) analyses on OP1 and OP2 were conducted with a JEOL IT 500 HR 465 instrument equipped with two EDS Oxford Instruments Ultimax 100 spectrometers. Both 466 samples were observed under the same magnifications ($\times 60, \times 1000, \times 3000, \times 4000$ and in a few 467 cases x10,000) (Supplementary Information E). EDS analyses and backscattered electron images 468 469 (BSE) were obtained under low vacuum mode at a pressure of 30 Pa with a 20 kV accelerating voltage. SEM observations and EDS analyses on sample OMF were conducted with a Tescan 470 471 Vega3 XMU scanning electron microscope (TESCAN FRANCE, Fuveau, France) equipped with an Oxford X-MaxN 50 EDS detector (Oxford Instruments, Abingdon, U.K). A 10 kV 472 473 accelerating voltage was used for imaging using secondary and backscattered electrons, and 20 474 kV for EDS analyses. All samples were observed with no prior metal or carbon coating. All EDS spectra were processed with the Aztec software (version 3.1, Oxford Instruments, UK). 475

μ-Raman (μ-RS) analyses were conducted to determine the mineralogical composition of 476 477 OP1, OP2 and sample OMF, using a SENTERRA Dispersive Raman Microscope (Bruker) equipped with an internal calibration system. The working area was examined using an 478 479 integrated colour camera. Spectra were acquired with a 785 nm laser and a laser power of 1 to 10 mW. The spectra were recorded with an integration time varying from 5 to 10 s, in a spectral 480 range from 100 to 2200 cm⁻¹, with a number of co-additions varying between 5 to 10 depending 481 482 on the presence of fluorescence radiation and signal-to-noise ratio. Data was collected with the 483 OPUS 7.2 software package (Bruker, Billerica, USA). Mineral identification was based on the comparison of the recorded spectra with those of available in the RRUFF spectra library, 484 University of Arizona⁵⁶. 485

The elemental composition of OP1 and OP2 was established by energy dispersive X-ray fluorescence (EDXRF) (Extended Data Fig. 6). Measurements were acquired using a positioning 488 device consisting of a metallic receptacle to which the spectrometer was fixed. Sample OMF was not analysed because its size is smaller than the spectrometer aperture $(3x4 \text{ mm in diameter})^{57}$. 489 490 EDXRF measurements were performed with a portable SPECTRO xSORT X-ray fluorescence spectrometer from Ametek, equipped with a silicon drift detector (SDD), a W X-ray tube with an 491 492 excitation source set at 40 kV, 0.1 mA. Spectra acquisition times were set to 120 s. The spectrometer is internally calibrated by an automated measure of the elemental composition of a 493 standard metal shutter. A supplementary calibration^{58,59}, based on the Lucas-Tooth and Price 494 methodology, was applied⁶⁰. This calibration, developed with the X-labpro software (Ametek, 495 Berwyn, USA) adjusts the mass attenuation coefficient and calibration slopes for major and trace 496 elements by using certified standards and reference samples analysed by ICP-OES and ICP-MS. 497 The Ochre analysis results in details can be found in Supplementary Information E. 498

499 Sediment analysis

Twelve sediment samples, as listed in Supplementary Information Table G1, were selected for mineralogical, elemental and magnetic analysis. Two samples (X1 and X2) come from the red stained area on which the ochre fragments OP1 and OPI2, limestone slab LS and cobble QS were found (Extended Data Fig. 10), two samples (X3 and X6) were retrieved in the same layer but at ca. 2 m far from the stained area, eight other (X4, X5, XMB2101–XMB2106) were collected between 20 and 200 m far from the excavated area (Supplementary Information Fig. G1). All the samples consist of floodplain silt.

XRD analysis⁶¹ was carried out on the twelve samples (Supplementary Information Table 507 G1), using a Netherland PANalytical X'PerPRO diffractometer with the following parameters: 508 Ni-filtered Cu-K α /40kV/40mA, scattering slit of 1/16°, receiving slit of 5 mm, continuous scan 509 510 mode, scanning speed of 0.049884°/s, and a scanning step of 0.0167113°. Bulk minerals were identified based on the following peaks: quartz, 4.26 Å; anorthoclase, 3.21 Å; albite, 3.18 Å; 511 rutile, 3.189 Å; calcite, 3.03 Å; dolomite, 2.89 Å; Hematite, 2.70 Å; Koninckite, 8.42 Å; and clay 512 minerals smectite/chlorite, 14.1 Å; illite, 10.0 Å. The relative proportions of the identified 513 minerals were roughly determined using their peak intensities by measuring the heights of the 514 main reflections with PANalytical X'pert HighScore software (Version 2.2e). 515

Raman spectra^{61,62} were obtained with a Witec alpha300R confocal-Raman spectrometer
equipped with a solid-state continuous-wave laser emitting at 532 nm, and diffraction gratings of

518 300 grooves mm⁻¹. A piece of single-crystal silicon was used to calibrate the wavenumbers of 519 the shifts. Laser focusing and sample viewing are performed through a Zeiss microscope fitted 520 with an EC Epiplan 50× objective lens (NA=0.75). The spot size is less than 1 μ m and the 521 resolution is 4.8 cm⁻¹. The laser power impinging on the hematite single crystal was 0.40 mW. A 522 spectra acquisition time of 30 s and total spectra with 20 accumulations were collected for each 523 measurement.

Non-destructive Micro-XRF analyses were performed on the twelve sediment samples 524 (Supplementary Information Table G1) following the procedures of Li et al.⁶³. The Micro-XRF 525 measurements were carried out on the M4 TORADO PLUS Micro-XRF analyzer. One area of 526 about $0.5-1 \text{ cm}^2$ for each sample was randomly selected for collecting the XRF signals with the 527 same measuring parameters (e.g., 50 kV of high voltage, 600 µA of anode current, 20 µm of spot 528 size, 10 µm of pixel size, and 20 ms/pixel of pixel time). For semi-quantitative comparisons, 529 530 each XRF spectrum was normalized with the signal of the Rh-La peak, which is generated by a Rh X-ray source. The data were analyzed with the M4 TORNADO Bruker Microanalysis 531 Software. 532

Mineral magnetic measurements were made on the twelve sediment samples 533 (Supplementary Information Table G1) in order to determine the magnetic mineralogy^{64–71}. γ -T 534 curves were obtained by continuous exposure of samples through temperature cycles from room 535 temperature to 700°C and back to room temperature with a ramping rate of 2°C/min, using an 536 537 AGICO MFK1-FA equipped with CS-3 temperature control system. To minimize the possibility of oxidation, the samples were heated and cooled in an argon atmosphere. For each sample, we 538 subtracted the contribution of the sample holder and thermocouple to the magnetic susceptibility. 539 Hysteresis loops were measured using a MicroMag 3900 Vibrating Sample Magnetometer (VSM) 540 (Princeton Measurements Corp., USA). The magnetic field was cycled between ±1.5 T for each 541 542 sample. Saturation magnetization (M_s) , saturation remanence (M_r) , and coercivity (B_c) were determined after the correction for the paramagnetic contribution identified from the slope at 543 high fields. Samples were then demagnetized in alternating fields up to 1.5 T, and an isothermal 544 remanent magnetization (IRM) was imparted from 0 to 1.5 T also using the MicroMag 3900 545 546 VSM. Subsequently the IRM at 1.5 T was demagnetized in a stepwise backfield from 0 to -1.5 T to obtain coercivity of remanence (B_{cr}) . Magnetic component analyses of coercivity distributions 547 were analyzed using the IRM-CLG program of Kruiver et al.⁶⁶. 548

To better constrain the origin and microstructure of the sediment, selected samples (X1, X2, X4, X6) were observed using optical microscopy and analysed using Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-Ray spectroscopy (EDS). Backscattered electron microscopy analyses were conducted using a scanning electron microscope with an energy dispersive spectrometer (JSM-IT500).

Detailed results of the sediment analyses can be found in Supplementary Information G.

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556 Stone tool functional and residue analysis

The stone tool functional analysis is based on the fundamentals of traceology^{72–74}, which has been subsequently consolidated, systematized^{75–79} and strengthened to improve interpretion^{76,77}. The study applied criteria established experimentally^{80–84}, incorporating a multi-scalar approach and a multi-technique microscopic analysis^{75–88}. Potential residues were characterized microscopically, including study of their elementary chemical composition, without removing adhering materials from tools^{89–92}.

563 Use-wear and residues were documented with the combined use of Optical Microscopy 564 (OM), Scanning Electron Microscopy (SEM) and 3D Digital Microscopy (3D DM). Four main 565 procedures (I-IV) were used for analysis.

566 I) A preliminary assessment of the samples and the location of features of special interest 567 was conducted. This was conducted at low magnification, using a stereomicroscope (Euromex 568 DZ.1105), with a magnification range of $8 \times -80 \times (0.8 \times -8 \times \text{ zoom}, 10 \times \text{ oculars})$, and equipped 569 with a 20MP 1" Scemex camera (Horizontal Field of View 16.6 mm -1.67 mm).

570 II) A systematic screening at different magnifications using 3D DM (Hirox KH-8700) was conducted. This microscope is equipped with a dual illumination revolver zoom lens (MXG-571 572 5000REZ) which together with the high intensity LED light source allows for the observation of samples with a 5700 K color temperature. This lens consists of a triple objective turret with a 573 different zoom range per objective, allowing for magnifications ranging from 35 to 5000× 574 (HFOV 8.6 mm-60 μ). The microscope is equipped with a high-sensitivity compact CCD camera 575 that enabled the capture of 24 frames per second at high resolution (1600×1200 pixels). The dual 576 illumination system allows for the observation of sample topography with ring and coaxial light. 577

The former is suitable for the observation of topographical irregularities with a uniform even illumination at low magnifications, while coaxial light serves to highlight the topography of flat surfaces at high magnifications. Ring and coaxial lights were used individually and even combined, according to the observation conditions required by the samples. The interest of this microscope lies on its integrated stacking and stitching technology, capable of performing realtime 2D and 3D tiltings and generating wide field of view images of large surface areas which, in turn, create quantifiable 3D models.

III) More conventional observations were conducted with a reflected light microscope. We used a Zeiss Axio Scope A.1 metallographic microscope, with differential interference contrast (DIC) system involving a Nomarsky interference contrast filter. The microscope was equipped with 10× oculars and objectives EC Epiplan ranging from $5\times/0.13$ to $50\times/0.5$ HD DIC, resulting in nominal magnifications ranging from 50 to 500 times. Pictures were taken with a 6.3 MP 1.8" FLIR camera, and fully focused images were obtained through a motorized stage and Winkoms Encoder Z 1.0 and Helicon Focus 5.3 software. Resulting HFOV ranges from 2.9mm to 295µm.

IV) An environmental SEM (ESEM FEI Quanta 600 model) with an energy dispersive X-592 593 ray spectrometer (EDX-EXL II System analytical oxford) for microanalysis, equipped with an INCA software (v 4.01) from Oxford Instruments for digital image acquisition, was conducted. 594 Most observations were carried out between $60 \times$ and $2000 \times$ (HFOV 6.9 mm to 207 μ , 24" 595 display). Observations were entirely done in low vacuum mode (LV), so the coating of the 596 597 samples with conductive materials (gold or carbon) was not required. Large field (LFD) and 598 back-scattered electron detectors (DualBSD) were used in combination to observe residues and 599 use-wear features.

Sample processing based on protocols tested in previous studies^{79,87} were adapted in our 600 601 microscopic workflow. No cleaning was applied during first observations; a very soft cleaning was performed prior to the characterization of residues by OM, 3D DM or SEM analysis; and, a 602 603 more thorough cleaning was subsequently applied when necessary, to document use-wear features in detail. The main cleaning steps were: 1) Removal of the ink and varnish used to mark 604 605 the archaeological tools with acetone; 2) 5 to 15 min ultrasonic baths in a 2% neutral phosphatefree detergent solution (Derquim[®]); 3) 5 min baths in hydrogen peroxide (H_2O_2 , 130vol) to 606 607 remove modern organic matter, when spotted; 4) Removal of residues from cleaning products under running water; 5) 2-5 min ultrasonic baths in pure acetone to remove any residues from 608

handling. When necessary, 5 min baths in a 10% HCl solution were applied to remove carbonated residues. Before each observation, tools were dried with compressed air to avoid contact with the laboratory paper and to reduce the formation of liquid spots on the surface, which may lead to misleading interpretations. During the entire process, samples were handled using powder-free gloves and plastic tweezers.

Experimental reference collections available at the IPHES Lithic Technology Laboratory, derived from published and ongoing programs, were used to compare to the Xiamabei results, thereby assisting in interpretations. The experimental programs involved actions on a range of common contact materials, and artefacts made on a variety of raw materials, including chert, quartzite, quartz, limestone, basalt, and obsidian^{83–85,88,90,91,93–97}. Interpretations were also supported by information supplied in the wider literature. Detailed reporting on the Xiamabei samples is provided in Supplementary Information I.

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