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# Aurignacian grinding stone from Surein I (Crimea): "trace-ing" the roots of starch-based diet

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#### Aurignacian Grinding Stone from Surein I (Crimea): "trace-ing" the 1 roots of starch-based diet 2

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#### 25 Abstract

26 This study is applied to the unique grinding stone from Surein I retrieved in the Aurignacian 27 layers of the rockshelter located in the south of the Crimea Peninsula. Our research enables 28 us (i) to make reliable inference on the agency establishing the functional modification on the 29 surface of the Surein I grinding stone, (ii) to demonstrate this grinding stone served as steady 30 surface (Face A) to mechanically process plant material including roots and tubers (under 31 surface storage organs, USOs) and (iii) to set a chrono-cultural framework for starchy plant 32 tenderization, also responding to key issues relating to the dietary breadth of early waves of

- 33 Homo sapiens at the northern latitudes. We present a pilot research design which integrates
- 34 data derived from macro and micro-scales techniques, by coupling use-wear traces analysis
- 35 and use-related starch granules observation. The multi-scale approach allows distinctive
- 36 resolutions for surface texture analysis thanks to the combined use of stereo, metallographic
- 37 and digital microscopes; whereas transmitted and polarized light microscopes were used to
- 38 observe use-related biogenic residues (U-RBR), namely starch granules, and SEM provided
- 39 resolution down to the nano-scale. Our data suggest that Homo sapiens was exploiting the
- 40 rich environment of the Pontic steppe-grassland since its earliest presence in south-eastern
- 41 Europe by processing starchy plants to obtain calorific food. Moreover, this study brings fresh
- 42 lines of evidence to the broadening of dietary strategies during late MIS 3 (40-25 ka calBP) by

analysing ground stones from the poorly investigated non-flaked industry, and opens new
 scenarios for the reasoning on *Homo sapiens* successful colonization of Eurasia.

45 Keywords: Wear-traces, Microscopy, Resolution, Methodological refinement, Use-Related
46 Biogenic Residues (U-RBR), Starch

# 47 Highlights

- Methodological refinement for functional analysis of Palaeolithic ground stones
- Optical light and scanning electron microscopy applied for surface texture and starch analysis
- Mapping of the functional related features
- Surein I (Crimea) is the oldest direct evidence of grinding stone used to process USOs
- *Homo sapiens* dietary flexibility and adaptation to Pontic steppe plant resources
- 54 **1. Introduction**

55 Within the broad assemblage of non-flaked industry, Palaeolithic ground stones 56 represent an under investigated source of data on different materials' transformation among 57 which plants, listed in the perishable edibles, are highly informative to reconstruct dietary 58 strategies. The term ground stones (macrolithic tools) is comprehensive of a multitude of stone 59 objects 'which were manufactured and/or used according to motions' (Dubreuil et al. 2015 p. 60 106). In the case of Early Upper Palaeolithic items, we are referring to those 'not altered from 61 their natural rock shape' (Adams, 2014 p. 15) used in both passive and active motions (Ebeling 62 and Rowan 2004, p. 108), such as lower steady grinding stone (i.e. Surein I presented here) 63 and movable items as pestles as we already applied in our previous work on Gravettian: the 64 terms grinding stone and pestles were used with reference to grinding and pounding activities 65 before crop domestication (Longo et al., 2018; Revedin et al., 2010; 2015). The present 66 research focuses on a task-specific tool used to mechanically transform plant starch-rich 67 storage organs (PSRSO) during early Upper Palaeolithic, when seedy cereals - to which 68 processing is referred most of the literature - were unlikely to be available. We evaluated that 69 wear-traces and starch granules extracted from ground stones may provide reliable 70 information about plant foraging and the mechanical processing aimed specifically at starch 71 extraction from the under and above surface storage organs (USOs and ASOs, Longo, 2016; 72 Longo et al., 2018; Revedin et al., 2010; 2015). We are fully aware that PSRSO processing 73 cannot be considered only as a technological/cultural phenomenon as it is intimately 74 connected with other biological and behavioural adaptations (Butterworth et al., 2016; Perry 75 et al., 2015; Longo, 2016), determining interactions that require complex yet careful analysis: 76 a challenge that goes beyond the scope of this work (Longo, 2016; Longo et al., 2020a). Here, 77 our main objective is to present the methodological refinements implemented in order to 78 investigate a large slab from Surein I (Crimea), aiming to locate and directly relate the active 79 surface of the ground stone with the adhering starch granules, by ensuring the provenience of 80 the U-RBR from the intentionally used areas across the large steady stone. This was obtained 81 by sampling the residues directly from the stone and also from the mold that revealed the deep 82 peel-off effect (see 3.5, 3.6 Fig. 9), a procedure that, to the authors knowledge, represent an original contribution to both functional and residues analysis. An attempt to identify the 83 84 functionally active areas was already performed by roughness analysis on the 3D model (Next 85 Engine, Longo et al. 2018). Moreover, the mapping of the wear-traces provided insights for 86 the spatial distribution of the utilized areas and the main direction of use.

The project "Unfolding the complexity of nutrition at the dawn of modern humans in Eurasia" started in 2016 (L.L. SUG, Singapore), already surveyed and sampled twenty sites dating back to Late Pleistocene - Initial/Early Upper Palaeolithic (EUP) and Upper Palaeolithic (UP), spanning western Eurasia to Central Asia (from Moldova to Crimea, to the Don river reaching the Altai Mountains, in Siberia). Our data disclosed the processing of PSRSO by means of ground stones since around 40 ka, possibly starting with the very early waves of *Homo sapiens* venturing out of Africa towards boreal western Eurasia.

94 The experience acquired during our previous studies on palaeolithic ground stones supports 95 the development of a multistep analysis at macro and micro scale for the surface texture 96 modifications of the limestone slab, and down to the nanoscale for the starch granules (1-100 97 µm). Although surface texture is difficult to define, among tribological studies there is a general 98 agreement in considering it as the "the features of surface relief" (Myshkin and Grigoriev, 99 2013). Texture analysis of the grinding stone from Surein I displayed evidence of man-induced 100 surface modification, allowing to interpret its possible uses, amongst which the very likely 101 processing of PSRSO.

102 Our new procedures have proved to be useful to acquire relevant data supporting strong 103 evidence of plant processing in the Pontic steppe since the Aurignacian. Here, we present the 104 pilot study on the grinding stone from the Aurignacian layer 3, the lowermost according to 105 1926-29 excavation carried out by G. A. Bonch-Osmolovsky at Surein I rockshelter (Crimea) 106 (Fig. 1a-b). The Crimea peninsula is recognized for the richness of its Late Middle/and Early 107 Upper Palaeolithic record represented by both behavioural and biological remains referable to 108 Neandertal man (Kiik Koba, Ak-Kaya, Zaskalnaya VI, Stepanchuk et al., 2017) and to Homo 109 sapiens (Buran Kaya III burials, Pratt et al., 2011, and a molar tooth from Surein I unfortunately 110 lost as reported in Chabai et al., 2004, p. 56). The two species lived in the same environment, 111 therefore were exposed to the same range of available food sources, although isotopic data 112 support a diversified access to food sources by the two species (Drucker et al., 2017).





114 Fig. 1. a) The Surein I rockshelter is located on the southern rim of the Crimea Peninsula (Pontic Steppe); b) Map

of Surein I rockshelter (modified from Bataille, 2016, Fig. 3: 53). In red, square 9B where the grinding stone was found; c) Drawing of the "structure" retrieved in square 9B, where the grinding stone is reported according to E.A. 117 Vekilova (1957) in Fig. 34: 307; d) the restaging at MAE RAS, St. Petersburg (photo L. Longo, June 1991). The 118 exhibit is based on the information published by Vekilova. Surein I grinding stone is clearly visible with the water-119 weathered dorsal face exposed, surrounded by fauna remains still in anatomic connection. In plain black, flint 120 artefacts; e) Surein I grinding stone: left, the unused surface (Face B); middle, the side; right, the coarse used 121 surface (Face A).

Until recently, the study of the lithic assemblages mainly revolved around very detailed analysis of the flaked industry. Despite the numerous efforts based on this approach, the study of the material culture failed to clarify the conundrum of the so-called "transitional industry" and to pinpoint the actual dwellers of the EUP layers that have not, as yet, been identified unequivocally (Bataille et al., 2016, p. 53; Demidenko, 2014a).

127 The study of the Palaeolithic non-flaked industry might bring new and unexplored 128 information regarding the dietary habits of the hominins roaming in the refugia of Crimean Peninsula around 40,000 years ago. The analysis of the grinding stone involved a 129 130 methodological refinement which includes the use of different resolution microscopes such as 131 stereomicroscope, reflected and transmitted light (IHMC, RAS), digital microscopy (hereon 132 DM) and electron scanning microscope, SEM, covering wear-traces and U-RBR, namely 133 starch grains. Bulky macro tools are difficult to examine under the standard structural designs 134 of most conventional light microscopes. We decided to use digital microscopes (Hirox and 135 Keyence) to scan large experimental stones, molds and araldite positives. Starch analysis was 136 then carried out according to established procedures at IHAE FEBRAS (Vladivostok) using 137 transmitted and polarized light and under SEM. We are confident that the methodological and 138 technological refinements applied to the study of Surein I grinding stone have enabled optimal 139 conditions for functional interpretation, which involves contextual different scales of resolution, 140 impacting on our research outcomes. The investigation, aiming at identifying functional 141 features supported by fine-grained data on the non-flaked stone tool from Surein I finally 142 demonstrates this is a grinding stone that serves as a steady surface to mechanically process 143 storage organs for modern human consumption.

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# 2. The archaeological material

The large rockshelter of Surein I in the Crimea Peninsula (alias Siuren or Suiren or 145 146 Surein, according to different transliterations, Demidenko et al., 2012; Demidenko, 2014a; 147 Bataille et al., 2018; Vekilova, 1957) is situated in the eastern slope of Bel'bek gorge, not far 148 from Sebastopol, on the second ridge of the Crimean Mountains (Fig 1a). The rockshelter is 149 43 m long, 15 m deep and 9-10 m high (Fig 1b), and is located 110 m above the sea level and 150 15-17 m above a small creek flowing nearby. K.S. Merezhkovski (alias Merejkowski and Ruev, 151 2018) in 1879-1880 excavated a first trench in the central part of the rockshelter and retrieved 152 1150 flint artefacts, not thoroughly published, conserved in part at the Historical Geology Chair 153 in St. Petersburg University, and in part at the Department of Archaeology of the Peter the Great Museum of Anthropology and Ethnography (Kunstkamera) of the Russian Academy ofSciences, St. Petersburg, Russia (hereinafter MAE RAS).

156 In 1926-1929 G.A. Bonch-Osmolovsky, continued excavations at Surein I and 157 identified three cultural layers by pioneering cutting-edge methodologies as he carefully 158 collected all the materials related to human activities and applied complex statistical analysis 159 for the interpretation of the finds (Vasil'ev, 2008, pp. 25-27), not yet in use by contemporary 160 western archaeologists. The excavated surface was divided into a grid (2 x 2 m) and Surein I 161 stratigraphy was presented during the Second (proto-INQUA) Congress held in 1932 162 (Leningrad, Soviet Union). Describing the flint inventory from Surein I rockshelter, the Russian 163 scholar attributed the findings to the western European Aurignacian complexes - the only 164 comparisons available at the time - and dated Surein I horizons to the second half of the last 165 Würm glaciation (Bonch-Osmolovsky, 1926; 1932). During 1926-29 excavation about 1000 166 tools were discovered in the bottom level together with a single molar tooth attributed to Homo 167 sapiens (Chabai et al., 2004, p. 56; unfortunately, the molar is lost, at present). The blade 168 industry includes scrapers, Busquet burins, small size hand axes, retouched blades, 169 Mousterian scrapers. 30 years later, E.A. Vekilova (Vekilova, 1957) re-addressed the study of 170 Surein I, comparing the materials with Trans-Caucasian assemblages (the Upper Palaeolithic 171 of Georgia) at that time finally available for comparisons to the Russian scholars. More recently 172 (1994-1997), a Ukrainian-Belgian team excavated the rockshelter intercepting the 1926-1929 173 trench and layers (F-G-H) obtaining the oldest absolute radiocarbon dating around 30 ka BP 174 which are considered "too young [... and] not represent the true age" Demidenko, 2014a; 175 2014b, p. 6720; Demidenko et al., 2012). The directly dated human bones at Buran Kaya III 176 recently retrieved in layer C demonstrate the presence of *Homo sapiens* in Crimea as early 177 as 31,900+240/-222 BP (Pratt et al., 2011) and further consolidate evidence for modern 178 humans as the actual users of the Surein I grinding stone.

Whatever demic or emic model can be called into the play, the attribution of the lower layers of Surein I to the Aurignacian is still undisputed as demonstrated by the new excavation and the radiocarbon dating obtained in 1994-1997, endorsing the previous stratigraphy, although detailing 8 successive dwelling episodes (Bataille, 2016, Fig 2, p. 52). The oldest phases are confirmed as the Krems/Dufour type and attributed to (proto) Aurignacian, and the faunal composition is also similar to that retrieved in Bonch-Osmolovsky trench (Demidenko, 2014b).

The present paper focused on the large, oval-shaped, biogenic limestone grinding stone from Surein I (236x122x68 mm; 3477 g; Fig. 1e) unearthed during the 1926–29 excavations in the lowermost layer 3 of the rockshelter (corresponding to layer G of 1994-97 excavation, Bataille, 2016, Fig. 2, p. 52), and dated back to >31 ka uncal BP. The large

190 grinding stone was retrieved in an intriguing close relationship with horse remains, still in 191 anatomical connection (Fig. 1c-d) and was immediately noticed during the excavation, 192 photographed, mapped and drawn (Fig. 1b-c, adapted from Vekilova, 1957; Bataille, 2016, 193 Fig. 3). It is worth mentioning the cutting-edge methodology applied by Bonch-Osmolovsky, 194 who paid careful attention to the documentation with notes, photographs and drawings 195 devoted to this exceptional context, unusual at the time the excavation was carried out. On 196 the basis of the forerunning scientific approach applied by Bonch-Osmolovsky, a real innovator 197 in those times, the whole area (about 1 m<sup>2</sup>) was philologically reconstructed in the museum 198 (MAE RAS, St. Petersburg), where the structure is restaged under a glass cage (Fig. 1d), 199 which avoid dust and other putative contaminations, and it is available for study. Such detailed 200 documentation of the artefact biography represents a strong case to clearly assess the 201 contextual association of the grinding stone with the flaked industry and the horse remains still 202 in anatomical connection, all referred to the layer 3 (the layer G of 1994-1997 excavation) and 203 attributed to the Aurignacian. In 1957 E.A. Vekilova duly published such well documented 204 material and also reported functional remarks derived from S.A. Semenov observations on the 205 flint assemblages.

206 The stone shows two very different faces (Fig 1e): one strongly weathered by water 207 when immersed in the river water (Face B, which faced upwards when retrieved during the 208 excavation), whereas the opposite (Face A, leaning on the soil) is still rough and it is the one 209 that was certainly used, after its collection from the river. The research design applied a fresh 210 multi-scale approach to investigate both wear-traces and starch granules extracted from the 211 functionally active areas of the large grinding stone. Shape, size and general morphology were 212 acquired by means of 3D scanning and photogrammetry, while the comparative surface 213 texture analysis of the used and unused areas of both faces, was integrated with the 214 morphological characterization of ancient "starch granules candidate" still adhering to the 215 active areas, and carried out by means of microscopes with different resolutions (Birarda et 216 al., 2020; Longo et al., 2020a).

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# 3. The multi-scale contextual approach

Even 219 For this study several microscopes with different resolution and magnification were 220 involved (Tab. 1): Optical microscope (OM), Digital microscope (DM), Scanning Electron 221 Microscope (SEM).

Model	Microscope	Magnification	Location
MBC-10	OM, Stereomicroscope	8.4-98 x	IHMC, St. Petersburg (Russia)

OLYMPUS BHMJ	OM Metallographic (DIC lenses) Canon EOS 400D camera Helicon Focus software	50-500 x	IHMC, St. Petersburg (Russia)
AXIO Scope A1	OM Transmitted/Polarized light	200-800 x	IHAE FEBRAS, Vladivostok (Russia)
Hirox KH-8700	3D Digital Microscope (DM)	35-2500 x (2D and 3D modality)	STARC, The Cyprus Institute, Nicosia, Cyprus
Keyence VHX 7000	3D Digital Microscope (DM)	35-2000 x (2D and 3D modality)	VCH Lab, ADM School, NTU, Singapore
Jeol JSM-6700F	Field Emission Gun (FEG) SEM	200-2000 x acceleration voltage (5 kV)	NTU, Singapore
Zeiss EVO 40	SEM with EDS OXFORD INCA energy	200-2000 x acceleration voltage (5 kV)	UniFi, Florence, Italy
Zeiss Supra 40	Field Emission Gun (FEG) SEM with Gemini column	800-25000 x low acceleration voltage (2 kV)	IOM-CNR, Elettra Sincrotrone, Basovizza, Italy

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Tab. 1: microscopes involved in the study. Surein I grinding stone was directly investigated in St. Petersburg by 223 means of optical microscopes (OM), and sonicated to extract the starch. The molds (imprints) and the araldite 224 positives were analysed by means of DM in Cyprus and Singapore. Both molds and starch grains were analysed 225 with SEM.

226 The study for attributing the function of Surein I grinding stone was carried out at 227 different scales of resolution and magnification and with various methodologies based on 228 tribological principles (Longo et al., 2018; 2020a; Birarda et al., 2020). Among them, surface 229 texture can be considered as "the features of surface relief" (Myshkin and Grigoriev, 2013), 230 and texture analysis is intended as the study of the deviation of the surface from a plane. This 231 can be applied to both macro and microscale as texture reflects the appearance of distinctive 232 surface patterns that can be analysed according to two main approaches: comparative and 233 parametric. Comparative wear-traces detection is based on qualitative analysis which reflects 234 analyst's experience and it is buffered by the resolution power and magnification of the 235 inspection technology. The comparative approach is applied in this analysis based on "the 236 expert visual evaluation of the similarities" between the observed tool and the experimental 237 reference (Myshkin and Grigoriev, 2013) by applying conventional Optical Microscopy (stereo 238 and reflected/metallographic microscopes), enhanced by the increasing resolution and 239 magnification lended by Digital Microscope and SEM. The resolution power based on visual 240 light, led and electron sources is influenced by the wavelength of the source beams and it 241 affects the discrimination between two adjacent points, hence the detailing capacity of the 242 obtained image. Our strategy is to analyse the functionality of grinding stone involving different 243 resolutions with the aim to increase the reliability of the comparative observation and to

244 understand the possible functions of the lower steady tool under scrutiny. The direct 245 observation of the archaeological grinding stone was carried out at MAE RAS, St. Petersburg, 246 by N.S. and V.T. using both low power and high-power approaches (optical microscopy). 247 Replicative experiments were carried out by N.S. to detail the reproduction of wear-traces and 248 worked materials transformation, and by L.L. and I.P. to build a suitable reference collection 249 for plants residues calibrated on the Pontic steppe coenosis (Longo et al., 2020a; Skakun et 250 al., 2019). In order to investigate the tool function, all "classical" approaches to wear-traces 251 analysis were applied (stereomicroscope and metallographic microscope, pioneered by S.A. 252 Semenov (1964) and L.H. Keeley (1980) and the following development, and the microscopes 253 involved are reported in Tab. 1.

254 During the survey (2015) the stone was 3D scanned to obtain a digital model by means 255 of Next Engine (Longo and Skakun, 2017; Longo et al., 2018). The putative used areas were 256 molded (impressions taken) using high-definition polyvinyl siloxane (PVS, Provil® novo light, 257 Heraeus Kulzer GmbH), selected after preliminary direct inspection by means of stereo and 258 metallographic scopes. The surface texture nanoscale replication was ensured by polyvinyl 259 siloxane impressions, a long-tested procedure for the authors (Longo, 2003) and the molds 260 were mapped on the actual surface of the stones. Repeated molding proved to be a very 261 useful practice during the experimental reproduction of the gestures and the kinematics of the 262 pounding activities. Also, molding disclosed an unexpected positive outcome due to its peel-263 off effect, since the sequential peeling (up to three per used-area) was at first cleaning the 264 surface (from dust and putative contaminants adhering the surface) and, in second instance, 265 it dislodged unpredictable micro to nanoscale residues (i.e. starch, raphides and fibers 266 adhering to the molds) out of the inner pits, the crevices and the unevenness of the surface. 267 The following sonication (standard ultrasonic tank) of peeled/molded areas were still testifying 268 the presence of starch grains and other microremains (see paragraph 3.5)

In laboratory conditions, the molds were thus photographed by G.S. Digital microscopy and SEM investigation was carried out on those obtained during 2015 and 2016 sampling campaigns by L.L. and G.S. who worked independently by using digital microscopes: Keyence VHX 7000 at VCH Lab at NTU (Singapore) and Hirox KH-8700 at Cyl-STARC (Cyprus). Starch granules were observed at both light microscope and SEM (I.P. and L.L.). Finally, the observations were cross-checked and the results discussed within the three groups of weartraces analysts working in St. Petersburg, Cyprus and Singapore.

Use-Related biogenic residue analysis (i.e. starch granules) was carried out by I.P., using a Zeiss AXIO Scope A1 and L.L. using several SEM, at low vacuum and with no coating, to be furtherly chemoprofiled by means of FTIR spectrometry and ToF-Sims (Birarda et al., 2020; Longo et al., 2020a). By using low and high-powered microscopes we feel confident that our functional interpretation is supported by a solid methodological and technological refinement which involves contextual different scales of resolution impacting on our researchoutcomes.

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## 3.1 Traceology with direct optical observation

284 The traceological analysis of the stone was carried out at low and medium 285 magnification (8.4–98x) with MBC-10 binocular microscope and oblique illumination. When 286 studying at micro level (50–500x), an Olympus BHMJ metallographic microscope was used, 287 equipped with a reflected light illuminator and differential-interference contrast (DIC) lenses. 288 The DIC produces a bias retardation which enhances the perception of the micro 289 topographical variations of the surface, and of the overall contrast, an effect that is obvious in 290 direct observation (Plisson, 2015, p. 221) and reduces chromatic aberration, a phenomenon 291 to which digital sensors are particularly sensitive. In fact, even when the sample texture or 292 micro-relief does not require this kind of vertical resolution enhancement, the DIC still improves 293 the quality of the photography (Plisson, 2015, p. 221). Micro-level shooting was carried out 294 using a Canon EOS 400D camera mounted on the metallographic microscope. To obtain high-295 quality photographs of the stone surface by focusing over the entire area in one frame, the 296 Helicon Focus software was used. The surface of the tool is usually embossed, but with the 297 help of this focus stacking software, we are able to quickly and correctly combine several (5 298 to 20 or more) source images, at different focusing distances, into a fully sharp image (Fig. 299 2a-b). During this study, special attention was paid to the following diagnostic features: macro 300 changes of the relief, linear traces, gloss and polishing.

301 When describing flint and bone inventory from the Surein I rockshelter, G.A. Bonch-302 Osmolovsky made remarks about the characteristics of putative working traces. However, this 303 is the first time that the functional study of the grinding stone is reported. The large pebble 304 belongs to the structure in the area 9B from the lowermost layer G as reported in E.A. Vekilova 305 (1957). The grinding stone shows an irregular-oval shape with a flat-convex cross section (Fig. 306 1e: center). The large oval stone shows two very different surfaces: Face A (Fig. 1e: right) is 307 quite coarse and still showing evidence of microfossils composing the biogenic original rock 308 (Fig. 9a), while Face B (Fig. 1e: left) is highly smoothed by water weathering (typical of stone 309 surface long exposed to water running in a river, which actually flows not far from the shelter). 310 Face A was downwards and embedded in the sediment, as carefully reported during the 311 excavation and in the philological restaging in the museum (Fig. 1c-d), and it is that on which 312 we concentrate our analysis: use-wear traces and adhering starch granules. Macro and micro 313 traces of utilization were concentrated in the central part of Face A, which is fairly flat with 314 negligible relief elevations. Significant macroscopic features are recorded only in certain areas 315 of the surface like polishing and flattening of the central area of the stone; no modern damage 316 was observed. Microscopic examination reveals spotty micropolishing with blurred edges,

317 situated on the highest points of the relief; it looks as dense, smooth, with varying degrees of 318 brightness, fading towards the periphery. In some areas we observed craters formed as a 319 result of light blows. The boundaries of the smoothed areas, when compared with the natural 320 surface of the stone, are slightly worn out and even, the protruding tops are polished. Linear 321 traces in polished areas look like short and long shallow lines with blurred, softly outlined 322 edges, less often as parallel scratches directed along the long axis of the object (Fig. 2 a-b). 323 However, the kinematic shows a general trend of the motion along the main axis of the 324 implement (Fig. 6). Well-defined wear traces are concentrated in the central part of the stone, 325 where molds 3 and 5 were taken, and to a lesser extent on peripheral areas. The features of 326 wear (striations, spotted polishes and the clear general abrasion of the central area which is 327 slightly lowered) drove us to identify this tool as a lower steady grinding stone. The direction 328 of linear traces, sub parallel to the long axis of the tool indicates that when working, the upper 329 mobile stone moved back and forth along one main direction.



Fig. 2. Use-wear traces observed with different microscopy; a-b) optical microscope (OM) images of use-wear traces, flattened areas, groups of striations and rubbed areas are visible (b); c-d) DM images of the use-wear traces on the molds 3 and 5: c, 3D elaboration of the wear features at 1000x; d, bunches of striations visible at 400x) as observed with HIrox KH-8700. The length of parallel striae ranges from 70 to 200 µm and are visible on flattened areas; e-f) SEM images of striation and rubbed areas as observed on the mold (mold 3; e, 600x; f, 800x).

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# 3.2 Replicative experiment to reproduce the use of the grinding stone

To verify the results of use-wear analysis and to obtain a sufficient number of standards, we undertook a series of experiments that further the work begun by S.A. Semenov (1974), G.F. Korobkova (1972, 1987, 1999) and other researchers (Hamon and Plisson 2008; Revedin et al. 2010; Skakun and Plisson 2014; Skakun et al. 2020). This was necessary since the Palaeolithic ground stones are unmodified pebbles and slabs contrary to those used for 342 processing plant materials from later periods (Neolithic) which are shaped to fit their utilization. 343 hence their morphology became highly diagnostic. Firstly, they were used to process wild 344 plants, and not cultivated cereals, which are qualitatively different in structure. Secondly, their 345 working parts, as a rule, preserve the stone's natural surface and are not processed, like the 346 Neolithic ones, with special picketage for better adhesion of the processed material (mostly hard seeds) to the working surface of the tool, and therefore show evident shaping and other 347 348 signs of wear. Our experiments were carried out according to a specifically developed 349 protocol, which included the selection of blanks for tools similar to archaeological samples in 350 shape and raw materials, selection of plant materials, identification of macro and micro use-351 wear traces at different stages of work, detailed recording of the experiment. Large 352 pebbles/slabs with a flat surface were selected for the lower, steady grinding stones, and small 353 flat-convex in profile pebbles as the upper active tools (Fig. 3a). A total of six experiments 354 were carried out. For the main experiment on grinding vegetation, Typha sp. (USO) roots were 355 used as modified material (Fig 3b-c). This plant was chosen because recent studies of the 356 lower cultural layer (Layer G) of Surein I (AMS OxA-5154 28450 ± 600), from where the studied 357 stone originates, show that the Crimean foothills of this time were characterized by a forest-358 steppe landscape (Demidenko et al., 2012; Chabai, 2000). The proximity to the water of the 359 settlement is also confirmed by the remains of water vole and beaver (Vekilova 1971, p. 124, 360 Table 3 and p. 126, Table 4), indirectly suggesting the likely presence of Typha sp. (USO) 361 along the reservoir. When preparing the experiments, the roots of Typha sp. (USO) were 362 preliminarily dried by a fire and peeled manually. Light crushing of the roots, kneading and 363 grinding were carried out in two ways: in a circular and reciprocating motion, with increased 364 pressure of the upper active tools on the processed material (Fig. 3b).



Fig. 3. The replicative experiment enacted to reproduce plant processing a) The raw material is consistent with the archaeological ones (limestone); b) N.N.S. during the replicative experiment; c) the stones are used for mechanical

368 processing of the cattail dried rhizomes (USO, Typha sp.) to produce a fine flour; d-e) optical microscopy images 369 of wear traces created during the experimental work of the pebbles used as grinding stones and upper active 370 stones.

- 371 The first macro-traces of use in the form of a slight sheen on the protruding parts of the surface, 372 a slight deformation in the form of flattened asperities of the rock original surface arising from 373 light impacts, were recorded after two hours of operation (Fig. 3d). Clearer wear-traces, similar 374 to those found on archaeological objects: slight grinding of the protruding sections of the relief 375 of the working part, spots of a rather bright shine with uneven boundaries, fading towards the 376 periphery of the working part, weakly expressed micro-traces in the form of thin shallow lines 377 on the lower stone and upper stone were produced after 5 hours of intensive work (Fig. 3e). 378 As a result of rubbing the roots of Typha sp. (USO), a thin, light substance (Fig. 3d) was 379 obtained, similar in appearance to the product acquired during the experimental work with 380 replicas of instruments from the Palaeolithic site of Bilancino (Italy) and Pavlov VI (Revedin et 381 al., 2010).
- 382 It should be noted that there were no traces of friction between the upper and lower grinding
- 383 stones on both experimental and original tools. Apparently, this was hindered by the fibrous
- 384 layer of the processed plant material.



385386Fig. 4. a) Ethnographic tool (lower stone and mano) for grinding berries and grains (MAE RAS No. 958-6 / b-c)

Cheyenne, Wyoming, USA, Late 19th century; b-c) microphoto of the working surfaces of the lower grinding stone
(b, 100x, scale bar = 1 mm) and mano (c, 100x, scale bar = 1 mm).

389 According to the palynological data obtained from the study of the cultural layer G of Surein I, 390 it is possible to reconstruct that near the rockshelter grew mountain ash - Sorbus aucuparia 391 L., buckthorn - Rhamnus cathartica I., and juniper - Juniperus sp. (Vekilova, 1957, p. 251). 392 However, we could not report on starches referable to these plants. Nonetheless, we tested 393 wear traces on a mortar used for grinding berries and grains belonging to the ethnographic 394 collection of the MAE RAS (No. 958-6, Chevenne, Wyoming, USA, Late 19th century). The 395 microscopic features of utilization on the ethnographic mortar are close to the traces we 396 described on the original grinding stone from Suren I and its experimental replica. The most 397 significant differences are expressed by the absence of macro deformation of the working 398 surface of the bottom of the mortar, that shows, instead, more even polishing covering the 399 bottom and partly the walls of the mortar, together with randomly located linear microscopic 400 features were found on its bottom (Fig. 4, modified after Skakun et al., 2020).

Further experimental work will be devoted to the study of use-wear features occurring on different types of rocks (sandstone, diabase, quartzite, granite) used for processing plant materials, aiming at identifying the dependence of the degree of wear development according to the duration of use of the tools, the type of processed plant materials and the tribological performance of the different rocks.

406

## 3.3 Digital microscopy and SEM

407 Direct investigation of large and bulky tools like ground stones by means of 408 conventional OMs is constrained by their structural design, as the stand and head are 409 designed for thin samples (e.g. slides), making it difficult to accommodate bulky shaped tools. 410 Similarly, the design of the SEM chamber is not suitable for oversized specimens. Therefore, 411 the design of DM is a relevant aspect in the selection of the most suitable microscope for 412 grinding stone wear-traces analysis. The DM stand is tall and adjustable, making it possible 413 to orient the optical head with respect to the surface of the stone, hence facilitating the 414 observation of large implements from multi-angle observation, as demonstrated during the 415 study of experimental grinding stone (VCH lab, Singapore). Moreover, the manual mode 416 allows for the tilting of the lens up to 90° and rotating the stage of 180°, facilitating observation 417 of the stone from various angles without handling. During inspection, all adjustments were 418 made without stone manipulation, using a small motorized console that greatly facilitates the 419 stitching operation (mosaicking pictures), enhancing precision and saving time for precisely 420 locating/mapping of the relevant spots on the tool surface.

421 Besides the issue raised by the dimension of the macrolithic tools, DM and SEM can 422 also overcome the depth-of-field and the focal distance constraints when inspecting highly 423 uneven surface texture and facilitate the scanning at different resolutions and higher 424 magnifications. Visual light microscopy is limited in the resolution by the classical Abbe 425 diffraction limit ( $d = \lambda/2.8$ ) and the magnification of metallographic microscopy is generally 426 capped at a spatial resolution of hundreds of nanometers due to the wavelength range of 427 visual light. In the case of the ground stone's uneven surface, the overall capacity of DM 428 revealed to be highly informative when observing raw materials with marked roughness, 429 crevices, holes, contours and, in this case, microfossils. The DM technology can greatly 430 influence image quality because of its combined lenses, video-camera and graphic software 431 which are developed to optimize the relationship between depth-of-field, resolution and 432 brightness, providing images that appear to have higher resolution than allowed by the 433 diffraction-limited optics. The combination of the images - taken by the digital video camera at 434 several focal planes (up to 120) - allows to visualize the fine topography of a very large area 435 (highly improving the image processing obtained with Helicon Focus, already mentioned in 436 3.1) and to build a 3D imaging. The field emission gun characterizing FESEM allows for direct 437 investigation at very low potentials (we used maximum 5 kV) without coating, nevertheless 438 providing extremely focused high and low-energy electron beams with a spatial resolution < 439 2nm (that means 3 or 6 times better than conventional SEM) (Borrel et al., 2014). However, 440 during scanning some samples may overcharge, ending with white striations on the images 441 as happened with our starch granules (e.g. Fig. 8a' and Fig. 9d). The option "no coating" 442 revealed crucial when carrying out further chemoprofiling of the U-RBR by applying FTIR 443 spectroscopy and ToF-SIMS (Longo et al., 2020a). In the case of rough surfaces (like those 444 of the ground stones, very different from the fine and smooth surface texture of rocks used in 445 flaked industries) the wide depth of field available in DM and SEM reveals itself as crucial.

446 In order to overcome both structural and optical constraints, replicas of the surface (i.e. 447 molding compound polyvinyl siloxane) were taken to allow the higher scale observation, being 448 a familiar and long-tested procedure for the authors (Longo, 2003; Longo et al., 2020a; 2020b; 449 Macdonald et al., 2018; Pedergnana et al., 2016). In the case of macrolithic tools the position 450 of the impressions on the grinding stone was documented to map the observed traces (Fig. 451 6). Molds are suitable for direct investigation under any optical microscope and prove to be 452 highly reliable even when scanned with DM (Fig. 2c-d) and SEM (Fig. 2e-f). The molds of the 453 putative used areas were analysed with two different 3D digital microscopes: Hirox KH-8700 454 (at CyI-STARC in Cyprus, by G.S. and L.L.) and Keyence VHX-7000 (at VCH Lab, NTU, 455 Singapore, by L.L.) which cover a magnification range from 35 to 2500x (times). The DM 456 higher resolution details the features already observed under lower resolution microscopy (Fig. 457 2 c-d: striations polish) and adds evidence of lesser-developed traces, decidedly increasing 458 the ultimate functional attribution. In Fig. 5 we exemplify the key features observed with DM 459 on the 4 molds (out of 5) taken out of Face A. Fig. 5a shows the mapping, on mold 3, of the

460 bunches of striae on the flattened area - surrounded by the unmodified original surface -461 outlined in red (5b and 5c). The following microphotos are imaging polished areas and 462 striations on molds 2, 4 and 5. It is noticeable that molds 3 and 5 are covering the central area 463 of the grinding stone, where the most intense and lasting pressure was exercised, therefore 464 flattening and lowering the area causing a slight shadowing of the surface that can recall the 465 look of tribochemical wear, observed as spotted polish with different degrees of brightness. 466 Moreover, bunches of shorter and lighter striae testify a coupled motion such as pounding and 467 dragging as the alignment of the striae is suggesting (Fig. 5a-c), and accordingly their 468 orientation is both parallel and perpendicular to the main axis of the grinding stone. On the 469 molds we observed U-RBR such as starch granules and fibres (Fig. 5e and h), that were then 470 observed under the SEM (Fig. 10). Molds 2 and 4, taken in peripheral areas affected by a 471 lesser prolonged activity, show more defined and sharp striae and polished areas (Fig. 5e and 472 5f). The unprecedented results of our study demonstrate DMs as the most feasible equipment 473 for the study of large implements with rough surfaces as ground stones enabling easy 474 inspection at both high resolution and magnification.



Fig. 5. a) Mold 3 with flattened areas highlighted in red and striation in yellow; b) smashed biogenic formations visible around a flattened area where bunches of parallel striations (indicated by the red arrow) are visible and they are crossed by a couple of longer striae parallel to the main axis of the stone (35x and c at 100x); d) striations on a rubbed area on mold 5 (200x); e) series of sharp striae visible already at low magnification (35x) on mold 4; f) bunches of parallel micro-striations on a polished area on mold 2 (600x); g) plant fiber on a rubbed area, next to a striation and to parallel striations on mold 5 (100x); h) the same residue at higher magnification (2500x), part of the striation is still clearly visible next to it.

483

# 3.4 Mapping of the wear-traces

The identified use-wear traces on the stone (shapeless rubbed-down traces, weak and light linear traces, spotted polished areas, other wear features), detailed by scanning the molds with the DMs, were photographed at increasing magnification (within a consistent standard: 200X, 500X, 1000X, 1500X, 2000X), and one or more details were identified as reference to map the same trait/s when imaged at higher magnification (Fig. 5a-c). Moreover, the contact side of the molds (impressions) were photographed and orthorectified.

490 In addition to molding, Surein I stone tool was 3D scanned (Next Engine, at maximum 491 intensity) for digital reproduction and for off-site inspection (Longo et al., 2018; McCartney and 492 Sorrentino, 2019, pp. 69-73). In order to elaborate the 3D model and the digital elevation model 493 (DEM) (McCarty, 2014), the stone tool was also photographed and referred to a coordinate 494 system. The orthophotos of the used surface (Face A), were matched into a final orthomosaic. 495 This procedure allowed us to import in AutoCAD the orthorectified images of the molds of Face 496 A, together with the images acquired with the different microscopes. The wear-traces 497 microphotos were scaled (according to the magnification) and co-registered through the 498 recognition of three common points. When entered in AutoCAD each magnification was 499 treated as a different layer. The final goal was to relocate the microscopic features in each 500 relative position on the molds, and the impressions were finally mirrored and aligned to the 501 stone surface. This procedure aims to create a schematic map of the featured traces of use 502 on the grinding stone's original surface, assigning the utilized areas and the main direction of 503 use (e.g. see the direction of the striations and the areas of their higher concentration, Fig. 6).



504

505 Fig.6. Schematic map of the molded areas: flattened areas (outlined in blue) and striations alignment (in red) are 506 highlighted, revealing their actual location on the central part of Face A and the striations can be even oblique to 507 the main axes of the stone (evidence for a widened motion).

508

# 3.5 The use-related starch granules

509 Use-related starch granules were extracted through standard ultrasonic tank cleaner 510 at room temperature (double-frequency ultrasonic power 180 W, 28 kHz is used for overall 511 clean, 40 kHz for precise clean) by soaking part of the tool. The operation occurred at the MAE 512 RAS in St. Petersburg (2017), after both the preliminary inspection through stereomicroscope 513 and metallographic microscope and the molding carried out as described in 3.1. Surein I liquid 514 sample was then processed by I.P. at the Institute of History, Archaeology and Ethnology, Far 515 East Branch, RAS, (IHAE FEB, Vladivostok, Russia). The preparation followed the methods 516 in use by scholars (Torrence and Barton, 2006; Therin and Lentifer, 2006; Yang et al., 2012). 517 Bleaching and careful cleaning of the lab surfaces and consumables is routine prior to starch 518 granule extraction. Cesium chloride (CsCl) was the salt added to prepare the heavy liquid to 519 segregate the starch granules from the accompanying sediment. The addition of this salt calls 520 for multiple rinsing episodes in order to be carefully washed-off the final solution and to ensure 521 the effectiveness of the further nanoscale analysis (Birarda et al., 2020; Longo et al. 2020a).

A Zeiss AXIO Scope A1 was used to scan the isolated residues. The solution (10% glycerol and 90% distilled water) with the isolated starch granules was mounted on a slide and observed at 200-800x under unpolarized (Fig. 7 a-e) and polarized transmitted light using DICcontrast modalities (Fig. 7 a'-c'). Micrographs and measurements were taken using the microscope software. All starch granules showed the typical Maltese cross (extinction cross

- 527 under polarized light) and other diagnostic features (Gott et al., 2006) (Fig. 7 a'-c'). The starch 528 granules are classified according to criteria proposed in previous studies (Piperno et al., 2004; 529 Torrence and Barton, 2006): the shape of the granules in various projections, surface features, 530 position and shape of the hilum, the presence and features of facets, the type of polarization-531 cross and characteristics of rays, the presence of lamellae and damage allowing for a
- 532 confident identification of genuine starch granules.



533 534

Fig. 7. Surein I: Starch granules and U-RBR isolated from the utilized areas on face A, bar 20 µm, e) - 50 µm; a-535 b) Polyhedral granules, black arrows - surface craters after enzymatic attack; c) Ellipsoidal granule; a'-c') the same 536 starches granules under polarised light. The extinction crosses are visible; d) Plant vascular tissue; e) Plant tissue 537 or fiber.

538 Seven granules of starch and fragments of plant tissues were obtained out of the 539 sonicated sample of Surein I. None of them has a complex of attributes reliably comparable 540 with any sample of the reference collection in Vladivostok lab, which includes 120 species 541 from 24 families encompassing wild and cultivated cereals, pulses, plants with USO and ferns. 542 This collection is mainly composed and constantly replenished with plants present in the 543 Russian Far East that do not correspond to the studied region, either geographically and 544 paleoecologically. At DAIS (Ca' Foscari, Venice, Italy) we are building a reference collection 545 with plants consistent with the western and south-eastern coenosis of boreal Eurasia, selected 546 among the list reported in recent reviews (Hardy, 2010; Kovárník and Beneš, 2018; Shipley 547 and Kindscher, 2016). As well, SEM imaging and FTIR spectrometry are applied to 548 characterize the starch granules in order to build a physical-chemical reference of starches 549 from the PSRSO record coherent with the Pontic steppe coenosis during Late MIS 3.

550 The seven starch granules can be divided into two groups. The first includes 4 granules 551 of a polyhedral shape. Size range is  $15-23 \mu m$ . The extinction cross varies from (X) to (+) type 552 and the rays are straight or curved. Hilum is located in the center, rounded. Some granules 553 exhibit radial cracks. Two starch granules have surface damage in the form of craters, similar 554 to the result of an enzymatic attack (Fig. 7 a-b).

555 The other 3 grains have oval-like forms. Another section-view was not available. Size 556 range is  $18-19 \mu m$ . The polarization cross varies from (X) to (+) type and the rays are straight 557 and curved. Hilum is located in the center, with a rounded outline. Two of them have lamellae 558 and one show transverse crack.

In addition to starch granules, the sample contained other residues. There are fragments of plant tissues such as vascular and epithelial tissues and plant fibers (Fig. 7 d-e). Therefore, the contextual presence of different plants-related remains and other use-related biogenic residues (as already observed by Hardy et al., 2001; Pearsall, 2015) are supportive of the genuine origin of the starch granules observed on Surein I grinding stone.

564 The limited amount of starch granules in the liquid sample can be explained by the 565 history of the artifact (surely washed and most probably brushed since the discovery), its 566 curation and in the last instance by the starch sampling strategy. It is relevant to recall that 567 polyvinyl siloxane imprints removed the main residues from the working surface and, due to 568 the geometry and dimensions of the ground stone, only a small part of the tool original area 569 was fitting in the ultrasonic tank, hence the sonication interested just a small peripheral area 570 of the tool (Skakun et al., 2019). Because of this constraint, the sonicated area is not one 571 referable to the most active areas. Such a small amount of starch granules extracted from the 572 sonication, in comparison with the more consistent number extracted from the molds, is 573 considered a good evidence of the absence of modern contamination and the localization of 574 residues in areas with use-wear traces is conducive to their authenticity. Therefore, we can 575 confidently say that the detected residues are mainly associated with the ground stone 576 utilization, and not due to soil deposits or other biasing conditions.

577 When comparing images of the starch granules we evaluate that the set of starch types 578 observed under OM and SEM did not differ significantly. There are distinguishable types and 579 shapes in the SEM samples: flattened round-oval granules corresponding to the Triticeae tribe 580 (Fig. 8), which is the most common group composing the Pontic steppe grassland. The first 581 group of OM starch granules finds analogies with another type of starch granules in SEM. 582 They have a multifaceted elongated shape. The presence, in the OM samples, of such 583 characteristics as the shape of the hilum, lamellae, and cracks are the projection of the internal 584 structure of the grain which can be detailed with the resolution of the SEM (Fig. 11). This is 585 the very first report on the coupled observation (OM and SEM) of ancient starch granules (Fig. 586 8).



587

588 Fig. 8. Surein I grinding stone. Correlated starch granules form as observed with SEM and OM microscopy; a) 589 Polyhedral shaped grains on the mold, bar 10  $\mu$ m; b) Polyhedral shaped grains from the sonicated sample, bar 20 590  $\mu$ m.

591

# 3.6 Starch granules under SEM

592 During the sampling, we developed a standardized procedure for the analysis of both 593 wear-traces and U-RBR (i.e. starch). It included wearing powder-free gloves, careful dusting 594 of the stone surface with a clean soft brush and running a first macroscopic inspection of the 595 putative used areas, taking close-up macro photos (Longo et al., 2020b). Once the suitable 596 areas are identified (see above 3.1), the further steps include deep cleaning of the areas by 597 means of the molding peel-off effect exerted by the thixotropic property of the polyvinyl 598 siloxane that enters in the unevenness, holes and crevices of the stone surface and extract 599 actual ancient starch granules (Fig. 9). The sonication was performed at MAE RAS, St. 600 Petersburg, to extract putative "genuine" use-related biogenic residues (Collins and Copeland, 601 2011; Copeland and Hardy, 2018). Molding obtained prior to sonication can peel-off putative 602 contaminants (a case still under analysis, since contamination cannot be ruled out in any 603 archaeological condition). However, the starch granules extracted do not look modern and 604 both DM and SEM inspections proved that starch granules and other U-RBR, adhering to the 605 molds, are associated with wear-traces (Fig. 9d and Fig. 8a: SEM images of starch adhering 606 to mold 3; Fig. 5g-h and Fig. 10: DM and SEM images of other U-RBR adhering to molds 1, 3 607 and 5). Molding generates peels from plant remains still entrapped into the crevices of the 608 archaeological grinding stone: this demonstrates the direct relationship of the starch granules 609 with the functionally active areas (as exemplified by the wear-traces on molds 5, pictured in 610 Fig. 5g-h), a case never reported before, to our knowledge. This unexpected discovery proved 611 to be fruitful for extracting starch granules out of the molds during the chemo-profiling of the 612 granules by means of SR-FTIR spectroscopy. A selection of starch granules isolated in 613 Vladivostok underwent the nanoscale analysis with FEG-SEM (Fig. 11; Longo et al., 2020a). 614 To perform the very first systematic observation of Palaeolithic starch granules under scanning 615 electron microscopy a Zeiss SUPRA 40 high resolution FEG-SEM, based on the 3rd 616 generation GEMINI column (available at ION CNR-Elettra Sincrotrone, Basovizza, Italy) was 617 used. The optimal structural characteristic of this FEG-SEM enables for direct observation of 618 the starches (and other residues) with no coating, thus enabling further analysis (e.g. SR-FTIR 619 and TOF-SIMS, Longo et al., 2020a; Birarda et al., 2020).



620

621 Fig. 9. a) The univen surface of the active side (Face A) of Surein I grinding stone. Limestone microfossils are 622 detailed with the function "digital microscope mode" provided by the Ricoh WG-30 camera (7,5x); b) DM false 623 colour 3D model of the mold microtopography: the deeper areas are in green and blue, while the higher are in 624 yellow and red (Keyence VCH 7000); c) we propose an ideal model of the crevices according to b). This 625 unevenness can serve to entrap both the contaminants and the U-RBR. In our hypothesis 1: the first mold extracts 626 putative contaminants, together with sediment, dust, and other biases and can be used as cross-check reference; 627 2-3, second and third mold progressively clear the microtopography and increase the extraction of genuine ancient 628 starches as demonstrated in d) where a starch granule is entrapped in the crevices of the 3rd mold (view under 629 SEM).

As mentioned above, the limits imposed by the resolution of conventional light
 microscopy to detect starch granules, mostly when dealing with those falling in the lowest size
 range (<50 μm) can be overcome by SEM: this allows a further level of analysis of the U-RBR</li>

morphological features, and has proved useful in examining the small starches extracted from Surein I grinding stone. Hence, SEM did help to identify new features appreciable at higher magnification, and, compared to visible light microscopy, has enabled us to count more starch granules (ten in a single 0,5 µL droplet) and to observe other plant remains (i.e. fibres and raphides, Fig. 10 and Fig. 11 d, f). SEM inspection renders the surface sculpturing and the morphological features highly evident, highlighting attributes never featured before for such ancient remains (Fig. 11).



640 641

Fig. 10. Surein I, molds 1, 3 and 5: adhering plant remains. a, d, g) DM direct observation of the molds at different
magnifications showing adhering plants remains; a, b, c) putative plant remains under DM and SEM with EDAX
analysis evidencing CI, C, Ca, O, Na, Si; d, e, f) plant tissues at DM and SEM; g, h, i) DM and SEM images of
candidate phytolith.

645 In the case of Surein I, starch granules are clustering in two types with evident hilum, 646 distal or central, and the shape looks as "roundish with a stretch" in the central portion of the 647 grain and also lamellae were clearly visible (Fig. 11). During the scanning, several granules evidently crushed or broken were observed. Accompanying plants remains were also 648 649 evidenced such as raphides, parenchyma, and phytoliths (Fig. 11 c-d and f: raphides, in light 650 blue), that are thoroughly discussed elsewhere (Birarda et al., 2020; Longo et al., 2020a; 651 2020b). As mentioned above, the lack of tailored reference collection for plants used for 652 economic purposes during the Late MIS 3, when Homo sapiens was living in the Pontic steppe,

653 makes it challenging to speculate on the taxonomic attribution of the identified starch granules 654 (Hardy et al., 2001). Therefore, we are implementing the reference collection with plants 655 available in the Pontic steppe today. The list includes those present during late MIS 3, i.e. 656 Betula sp., Rhamnus cathartica I., Sorbus aucuparia I., Juniperus sp., willow Salix sp, Populus 657 tremula as already reported by Vekilova (1957; 1971) and shrubs and grasses with storage 658 organs (Hardy, 2010) suitable to be collected and processed for the extraction of dietary 659 carbohydrates like Stipa sp. (among ASO) and Typha sp., Arundo donax, and Phragmites sp. 660 (among USO), already reported in pollen lists of the area available in the literature (Hammerman, 1929; 1934; Gerasimenko, 2004; Demidenko et al., 2012). Modern plants are 661 662 collected with the dual purpose (i) to extract starch, under lab-controlled conditions, for 663 physical-chemical and morpho-structural characterization (including ageing under controlled 664 parameters) and (ii) to be used in experiments of mechanical tenderization and traces 665 development (G.S. PhD project, ongoing).

Another positive mark of SEM observation is the "3D like" effect which makes the observation of the U-RBR much detailed and allows for the identification of features at the nanoscale like lamellae and exfoliation, interpreted as due to mechanical processing as well as to other physical or chemical events occurring during the tenderization, hence man-made (Birarda et al., 2020, Fig. 4). However, the phenomenon might have occurred during diagenesis, and the on-going ageing experiments in a climatic chamber (at DAIS, Venice) will possibly shed light on the mechanism of the exfoliation and other features observed.



Fig. 11. SEM: Starch granules extracted from the sonication of molds 3 and 5, usually they are distributed along the outline of the droplet. Starch granules in a-d and g-i are imaged at different magnifications with the lower range resembling those obtained under OM. Starches (on purpose coloured in light orange in 11d) and raphides (calcium oxalate crystals) on purpose coloured in light blue 11c-f) are still adhering to different granules, testifying their consistency with USO attribution of the granules; e-f; h-i) Starch different morphology, surface structure and central hilum.

## **680 4. Discussion**

681 Our research aimed at: (i) collecting evidence supporting Surein I grinding stone was 682 used to mechanically transform PSRSO into food, (ii) implementing the investigative 683 procedures with ad hoc methodological refinements for both wear-traces and U-RBR analysis. 684 Finally, the results obtained with this innovative research design are supporting fine-grained 685 data to unfold the complexity of nutrition at the dawn of modern human colonization of the 686 south-eastern Eurasian steppe during late MIS 3 (40-25 ka calBP). By that time, the boreal 687 continent was already inhabited by Eurasian archaic humans, namely Neandertals and 688 Denisovans further to the east, well adapted to their ancestral boreal environments, who were 689 thriving on a highly carnivorous diet (Jaouen et al., 2019). Even for the ancient settlers of 690 Crimea, prompted to be one of the south-eastern refugia for late Neandertals, the bulk 691 collagen  $\delta$ 15N high values are suggesting they fed on a typical terrestrial-based diet (Drucker 692 et al., 2017). The early occurrence of Homo sapiens in the peninsula is represented by the 693 remains of Buran Kaya III, layer C, which direct dating supports for modern humans presence

694 before the harsh HS 4 conditions (Heinrich Stadial) (Pean et al., 2013). Together with the 695 recent radiocarbon dating of Bacho Kiro cave (Bulgaria) it is possible to set back the early 696 presence of anatomically modern humans in the Pontic Steppe around 40 ka calBP (Fewlass 697 et al., 2020), extending the length for the overlapping of the newcomers with the late 698 Neandertals in the area. Vekilova (1957) anticipated that Surein I oldest assemblages recall 699 features comparable with the Transcaucasian Upper Palaeolithic (Zamiatnin, 1957, and lately 700 Korkia, 1998), among which some instruments show similarities with the Levantine industries, 701 and with the EUP assemblage of Mezmaiskaya cave (Golovanova et al., 2006). However, in 702 this mixed peopling scenario, with late Neandertals' resilient presence and early Homo 703 sapiens appearance, there is much debate over who actually may have developed the so 704 called "transitional industries" reported for the Pontic Steppe sites. These industries include 705 innovative elements - and are listed with different names i.e. Late Micoquian, Streletskayan, 706 or eastern Szeletian, Proto-Aurignacian, Aurignacian, etc. (Demidenko, 2014b; Bataille et al., 707 2016, just to name a few within a vast literature). Therefore, it is crucial to explore other lines 708 of evidence to disentangle the long chain of circumstances that allow us to identify who actually 709 developed the transitional/Initial/EUP cultures: the resilient Neandertals or modern humans 710 newcomers. Although flaked industries represent - along with bones - the most frequent 711 physical remains left by hunter-gatherer activities, they cannot be considered as the only 712 source of behavioural information and to reconstruct nutritional strategies.

713 Off the beaten track, we addressed the broad assemblage of non-flaked industry 714 specifically focusing on ground stones retrieved in boreal latitude EUP sites associated with 715 Homo sapiens, in order to elucidate their high potential as reliable source of information for 716 dietary strategies (Birarda et al., 2020; Longo, 2016; Longo et al., 2020a). The investigation 717 of Surein I task-specific tool - the large biogenic limestone slab retrieved in the lowermost 718 Layer 3 of G.A. Bonch-Osmolovsky 1926-29 excavation (layer G, Bataille, 2016) referred to 719 Aurignacian – provided data to demonstrate its use to transform PSRSO by mechanical 720 tenderization such as grinding, pounding and threshing.

721 The methodological refinement applied to the study of Surein I grinding stone 722 consistently integrates the research design with different stages: from the sampling strategy -723 to avoid further potential contamination - to the concerns arisen during the complex integration 724 of the probes in use, and analysis from the macroscale down to the nanoscale. We developed 725 a coupled macro to nanoscale investigation of the Surein I grinding stone, considering the 726 contextual occurrence of wear-traces and U-RBR, namely starch granules. The application of 727 different microscopes with increasing resolution and magnifying capability (Optical, Digital and 728 Scanning Electron Microscopy) made it possible to couple the different methodologies and to 729 overcome the limits due to the resolution power of the applied technologies. Our procedures

730 combine the acquisition of 3D geometry, the direct observation of wear-traces (in the 731 museums) and the further detailing of the features on the stone tool molds (imprints). Mapping 732 and relocating the functional features (the wear-traces, the flattened areas, and the orientation 733 of the striations) from the molds to the original stone is a step forward in functional analysis of 734 ground stones when investigated at high resolution. We are aware that due to methodological 735 constraints. e.g. heuristic requirements, equipment availability, and time restraints, these 736 techniques are applied at present to a limited number of samples and to address specific 737 questions (i.e. wear-traces on different raw materials pebbles and starch granules, Longo et 738 al., 2020a; Birarda et al., 2020).

739 Furthermore, we identify a best practice to extract U-RBR correctly from the used areas 740 of the stones with reference to the internal and external constraints of the available facilities. 741 The starch granules adhering to the grinding stone are tiny (in the case of Surein I < 50  $\mu$ m 742 average size, within a range of 1-100µm), thus, requires micro and nanoscale analysis to be 743 appreciated. One of the main constraints of use-related residues analysis is that it can be 744 affected by the unpredictable effects of contamination (Crowther et al., 2014; Dozier, 2016; 745 Mercader et al., 2018 for review), which is an even greater concern when sampling from 746 museum collections and old excavations. The topic is widely treated by several scholars 747 (Pearsall, 2015; Ma et al., 2017) who also report on experiments carried out to verify the 748 feasibility of starch migrating from the soil or from the surrounding weeds, to the surface of the 749 stone tools. Hart (2011) has demonstrated that is not the case, and if contamination occurred 750 it is limited to the very external surface of the tool, since he did not find any contaminants 751 starch granules in the sonication of the control experiments. Hart's results can be taken as 752 reference for the reliability of U-RBR entrapped at the bottom of crevices, holes, etc. of the 753 coarse ground stone surface. We actually paid great attention, through all the analytical steps 754 to control putative pollution and other biases that may hinder actual U-RBR and specifically 755 those related to starchy plants intentional processing. The practice of taking three successive 756 molds, progressively clearing possible biases, made the ultrasonic extraction suitable for the 757 identification of ancient use-related biogenic residues, although it can limit the number of 758 remains extracted as experienced in our study (only seven starch grains were extracted from 759 the sonication). The contextual extraction of U-RBR signifies the presence of different plant-760 related remains (starch, raphides, parenchyma, phytoliths) on the areas bearing clear wear-761 traces and proved to be supportive of the genuine origin of the starches observed on Surein I 762 grinding stone (Hardy et al., 2001; Pearsall, 2015). The standardized procedure we developed 763 to extract starch granules has enabled us to correlate residues and wear-traces - intended as 764 an evidence of PSRSO intentional processing - as well as to confirm the presence of genuine 765 ancient starches. Considering the long history of the grinding stone examined, any trace of modern starch on it would not display the dilapidated appearance that all our starch granulesdisplayed.

768 Plant remains are perishable and difficult to track. For the southern hemisphere, J. 769 Mercader was the first reporting on sorghum seeds from Ngalaue cave (Mozambigue), a site 770 dated to 105-55 ka (Mercader, 2009). Further to the south, parenchyma remains, retrieved in 771 hearths and ash-rich layers, were attributed to charred roots and rhizomes at Border cave 772 (177 ka), and at Klasies River cave since 65 ka (Larbey et al., 2019; Wadley et al., 2020). 773 Although no ground stones have been retrieved in the mentioned sites, therefore no direct 774 evidence for intentional processing is documented, the findings are consistent with the 775 presence of Homo sapiens. Phytoliths and starch granules were recovered from two slabs 776 retrieved in the Middle Sangoan occupations of Sai Island (Sudan) and interpreted as 777 evidence of plants processing dating back to 200 ka ago (Van Peer et al., 2003). One can 778 speculate that Homo sapiens in Africa was already including in his dietary breadth starch-rich 779 foods, mostly rhizomes, the veritable plant storage organs. Therefore, it is possible that plant 780 processing was a technological practice that Homo sapiens exported with its northward 781 explorations.

782 At boreal latitudes, putative plant foods have been reported throughout the late 783 Pleistocene (Hardy, 2010; Kovárník and Beneš, 2018; Shipley, 2016); however, the intentional 784 plant processing by means of ground stones is not very frequent until the Holocene. Hence, 785 ours is a challenging hypothesis for the considered time period (late MIS 3). In the Levant 786 phytolith from grass husks were associated with Neandertal (70-55 ka) at Amud cave (Madella 787 et al., 2002), whereas charred legumes and nuts (i.e. acorns and pistachio) were recognized 788 at Kebara (48-59 ka; Lev et al., 2005). Pulses (i.e. lentil, chickpea, pea, vetchling), fruit and 789 nuts are reported at Theopetra Cave in Greece (Mangafa, 2000). Starch granules are reported 790 on 3 artefacts from the Swabia Aurignacian sites by Hardy B.L. et al. 2008, although they 791 caution that it is unlikely that flaked tools were used in the starchy plants processing. Plant 792 remains correlated with prevailing shrub tundra have been reported from the Aurignacian site 793 of Hohle Fels (Swabia, Germany), where few seeds attributed to Asteraceae and to the broad 794 group of Poaceae are referred to be used by humans around 44,2 ka calBP, together with 795 bark fragments interpreted as bearberry (Riehl et al., 2015). In Crimea, the very first 796 publication reporting intentional plant processing dates back to 2001, thanks to the pivotal and 797 inspiring study on 50 flaked flint tools from the Late Middle Palaeolithic layers of Starosele and 798 from Buran Kaya III layer C, attributed to the Early Upper Palaeolithic (Streletskaya) (Hardy et 799 al., 2001). The coupled investigation of wear-traces and residues analysis highlighted the 800 retrieval of soft plant parenchyma and wood tissues, the latter interpreted as hafting traces for 801 spear heads and, more in general, woodwork undertaken in both the sites. What is particularly 802 relevant for the present study is the data derived from the analysis of the trapezoidal microlith 803 from Buran Kaya III. The OM compelling analysis of the residues revealed the presence of 804 starch granules and raphides (calcium oxalate crystals) and the wear-traces suggest the tool 805 "was hafted and used to plane or to scrape a starchy substance" (Hardy et al., 2001, p. 10976). 806 As said, for late Pleistocene starches, it is very difficult to "identify the plant material to taxon" 807 (ibid.), however, the presence of raphides suggests they originated from starch-rich storage 808 organs like rhizomes (USOs, Hardy et al., 2001). Intriguingly enough, our analysis of Surein I 809 grinding stone revealed not only wear-traces compatible with plant processing, but OM and 810 SEM inspection clarify the presence of both starches and raphides, that are conducive to 811 interpret starch granules as pertaining to three to four different geophytes (Fig. 11). Our 812 previous work on ground stones from Gravettian hunter-gatherer settlements clearly 813 demonstrated the intentional tenderization of geophytes (USOs) such as Typha rhizomes, 814 Botrichym ternatum, Lactuca tuberosa and Arctium lappa which were processed in the Italian 815 peninsula and in Central Europe, therefore throughout the cold boreal territories (Longo et al., 816 2020a; Longo et al., 2018; Revedin et al., 2010; Skakun et al., 2019).

817 Ethnographic sources report starch-rich water-lilies rhizomes are foraged from spring 818 to autumn across Eurasia northern latitudes (Gubanov et al., 2002). The use of baking or 819 boiling USOs is well reported in the Far East to prepare a sort of porridge or soup, and kasha 820 is still the traditional meal in Russia (Podmaskin, 2007). Also, it is acknowledged that 821 mechanical processing, leaching and even roasting are necessary steps that make plant food 822 bioavailable for the further metabolization occurring once plant foods are ingested. The 823 practice of mechanical processing into flour and then drying the crumble, which is therefore 824 losing most of the mass and the weight, reduces the bulkiness and concentrates the nutritional 825 power and can readily be consumed or stored for a delayed utilization, making the staple 826 starchy-food easy to store and to transport, i.e. a highly suitable reservoir for Palaeolithic 827 mobile hunter-gatherers.

828 Plants are predictable and their perennating undersurface storage organs - meaning 829 available all year round - are rich in highly nutritious carbohydrates and short-chain fatty acids, 830 which enable plants to survive ecological and climatic downturns. This turns out to be vital 831 even for Homo sapiens who was the hominin who could efficiently transform PSRSO into 832 calorific food due to the ptialin, the salivary enzyme regulated by the AMY 1 gene that is 833 duplicated in Homo sapiens but not in the archaic humans (Perry et al., 2015; Butterworth et 834 al. 2016; Longo, forthcoming). It is our opinion that the step-change capacity of efficiently 835 metabolizing highly calorific starch-rich food revealed a crucial adaptation to survive the 836 adverse climatic conditions Homo sapiens faced throughout late MIS 3 "volcanic winters" 837 (Golovanova et al., 2010), enabling Homo sapiens to access different nutrients in the course 838 of climatic downturns occurring at the northern latitudes. The Surein I grinding stone was

839 possibly used to process geophytes (USOs) surviving in cold and more arid steppe, where 840 mostly lean animals such as horses, deers and steppe bisons were supplying fats and meat. 841 Direct isotopic evidence from the Homo sapiens burial retrieved Buran Kava III supports that 842 plant food consumption in EUP modern humans was significantly higher compared to the 843 Neandertals diet, and the two species most probably co-existed in Crimea (Drucker et al., 844 2017), supporting they accessed different foods. Although still speculative, it is worth 845 considering that starches might have supplied Homo sapiens with those nutrients that spared 846 him from the constraints of a strictly carnivore diet (i.e. rabbit starvation; Speth and Spielmann, 1983; Speth, 2018). 847

## 848 Final remarks

849 Altogether, our study enabled us to identify Surein I macrolithic tool as a grinding stone, 850 used by *Homo sapiens* to mechanically process starch-rich storage organs into more easily 851 bioaccessible chunks and gross-grained flour. Our approach represents a due methodological 852 refinement, highly relevant towards establishing the ground stone's function within the elusive 853 processing strategies of plant originated food since the very beginning of Early Upper 854 Palaeolithic. We trust that our multi-scale contextual approach supports fine-grained data that 855 Surein I task-specific macrolithic tool was used to process under surface starch-rich organs 856 (USOs). Relying on the results of wear-traces and starch granules (including fibers and 857 raphides) directly associated to the used areas, we speculate that Homo sapiens, while 858 roaming across the Pontic Steppe, was foraging plants devoted to starch extraction from under 859 surface storage organs (USO, such as cattail, cane, reed) by mechanically processing them 860 into raw food. The chronology for modern humans' presence in south-eastern Europe, makes 861 Surein I grinding stone the oldest direct evidence of intentional geophyte processing during 862 Aurignacian settling, providing proof for a breakthrough in the dietary strategies of modern 863 humanity.

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