Geo-material provenance and technological properties investigation in Copper Age menhirs production at Allai (central-western Sardinia, Italy)

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ABSTRACT

During the 2nd millennium BC anthropomorphic menhirs belonging to a 3rd millennium BC sanctuary were reused as building material in the Arasseda Nuraghe (Sardinia, Italy). To analyse the Arasseda menhirs and the local Monte Ironi geological samples (presenting similar visual features), chemical (pXRF, ICP-OES, ICP-MS), mineralogical-chemical (PXRD) and physical (Mohs hardness) measurements were performed. Through the experimental data, the menhirs source provenance and the technological properties (workability, durability) of the raw material chosen for sculptural purposes during Copper Age were investigated. To the authors' knowledge this is the first archaeometric study on the Arasseda menhirs (the third on Sardinian menhirs) and one between the few recently developed on European megaliths.

KEYWORDS

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Sardinia; Copper Age; menhir; provenance study; technological properties

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Introduction

Megalithism appeared in Europe during the 6th millennium BC, soon erecting a number of archaeological evidence related to cults of death and ancestors. Among the megalithic monuments it is worth mentioning menhirs, that are present in many geographical contexts between Spain and Ural mountains. The process of standing stones' spread across the Central-Western part of Europe and their artistic and symbolic evolution between 6th and 3rd millennia BC, is still not clear. However, today a good part of the archaeologists endorses the polygenic origin of the phenomenon (Guilaine 1980; 2004), rejecting the traditional diffusionist and migrationist theses (Arnal, Arnal, and Demurtas 1983).

As in other regions of Europe (Taçon 1999; Calado 2002) Sardinian menhirs were probably realised for the cult of death (Perra 1992; Cossu 1996) or used as territorial markers (Atzeni 1982; Saba 2000; Usai and Perra 2012). Today, over 100 anthropomorphic menhirs (made of a wide variety of sedimentary, metamorphic and volcanic stones) are known in Sardinia, specifically for the areas called Nurra, Marghine, Mandrolisai, Barbagia di Belvì, Barigadu, Marmilla, Sarcidano and Sulcis (Cicilloni 2008, 2013) (Figure 1).

In the 1990s some Eneolithic menhirs were found in Arasseda, near the small town of Allai (Barigadu zone, central-western Sardinia) (Figure 1). They were reused to build the Arasseda Nuraghe during the Sardinian Bronze Age (2nd millennium BC). These artefacts show an ogival frontal profile, a plano-convex section and are characterised by geometric representations (generally interpreted as human anatomical parts) made by the bas-relief technique (Atzeni 1992; Cossu 1996; Cicilloni 2008) (Figure 2).

An ancient quarry of ignimbrite blocks (Figure 3A) was discovered near the nuraghe and the sculptures' find area (Cossu 1996). The recent finding of Monte Claro culture polished pottery (cfr. Basoli, Doro, and Zedda 2012) during a survey performed in the quarry site (Figure 3B), suggested the probable use of the stone blocks in Eneolithic times (2900–2300 cal BC according to Melis 2009), perhaps for the production of the Arasseda menhirs.

So far, few studies investigated the source provenance and the technological properties of European megaliths (Bevins, and Ixer 2013; Bevins, Ixer, and Pearce 2014; Thorpe et al. 1991; Pirson, Toussaint, and Frèbutte 2002, 2003; Williams-Thorpe et al. 2006) and a small part of them concerns menhirs. Only two of the available research works addressed Italian standing stones characterised by anthropomorphic features (Di Battistini, Franzini, and Lezzerini 2008; Rubinetto et al. 2014) similar to those of Eneolithic Sardinia, probably produced from the first centuries of 3rd millennium BC (Melis 2009; Cocchi Genick 2012; Perra 2012).

During the last decades, some assumptions on stone working technology and provenance of geo-resources

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Figure 1. The island of Sardinia in the Central European geographic framework (A); generalised map of Sardinia showing the menhir find localities (B); territory of Allai showing the Arasseda hill with nuraghe, menhirs and quarry site locations (C).

employed in Neolithic and Eneolithic Sardinian megalithism were formulated (Atzeni 1982; Maoddi 1995; Castaldi, 2000; Marini et al. 2007; Murru et al. 2008). However, the research on this item, especially with reference to menhirs, has always been rare due to the failure to sistematically adopt a synergy between the traditional archaeological investigation and archeometry. The application of this joint approach to the study of prehistoric Sardinian standing stones is currently restricted to two cases, performed on the menhirs of



Figure 2. Menhirs from Allai: Arasseda II (1), Arasseda III (2) and Arasseda VII (3).



Figure 3. A) Arasseda quarry site: layout plan and cross sections; B) Eneolithic (Monte Claro culture) ceramic shards from the Arasseda quarry site.

Sarcidano (Figure 1), specifically of Nurallao (Serra, Mameli, and Cannas 2016a) and Laconi (Serra, Mameli, and Cannas 2016b).

In the frame of the few archaeometric examinations of the Eneolithic Sardinian standing stones, the authors present the results of a study concerning the three menhirs recently removed from Arasseda Nuraghe and displayed in the Sardinian archaeological museum of Laconi. In continuity with the European tradition of archaeometric studies on megaliths, the selected approach (visual inspection and chemical analysis) aims to detect the source provenance of the raw materials employed for the production of the Arasseda menhirs. In addition, through the definition of mineralogy and hardness of the stone, the research investigates the workability and durability of the georesource employed in the Arasseda menhirs' manufacture.

Materials and methods

Geological setting

In the Barigadu region, on the paleozoic metamorphic basement, the appearance of rhyolitic lava domes and flows took place during the Cenozoic Era (Figure 4). In the northern part of Allai the small ignimbritic outcrop of Monte Ironi (produced by a dacitic glowing cloud developed during the Oligo-Miocene period) closed the volcanic stratigraphy (Figure 5A). This pyroclastic outcrop, composed of idiochromatic rocks always reddish-brown in colour because of the oxidation of iron in the contained iron-minerals (Assorgia et al. 1995, 1998), forms the Arasseda hill, today surrounded by a wide quaternary alluvial soil deposit (Figure 4).

Sampling

The strong layering of paleozoic shales and cenozoic rhyolites, rules out their use to make megaliths as menhirs. Conversely, the Monte Ironi ignimbrites show a structure without close cleavage planes (Argiolas et al. 2006) that certainly could allow the production of megalithic sculptural works. In addition, among the geo-resources present in Allai, the Monte Ironi volcanic rock is the only one surely compatible, from a visual point of view, with the stone employed for the Arasseda menhir production. Therefore, it was selected by the authors on the basis of its structural properties and because of its similarities with the artefacts.

Eight geological samples were collected within a sampling area of 0.5 km² (1×0.5 km), along the North-East/South-West axis of the Monte Ironi outcrop (Figures 4, 5A) (on the sampling method see Ruge, Barber, and Magee 2007; Shotton and Hendry 1979). Each sample had a surface area of at least 50 cm^2 and a thickness of the fresh rock of about 3 cm. The wide surface area ensured the possibility of carrying out independent measurements and avoiding any overlap of the analysed zones (Potts et al. 2006). The thickness was sufficient to exclude X-ray scattering phenomena that may compromise the analyses based on these photons (Serra, Mameli, and Cannas 2016a, 2016b). The samples were labelled as MIA (Monte Ironi Allai). In order to obtain the best representativeness of the geological unit, the Monte Ironi specimens were sampled to cover the entire extension of the outcrop (Beardsley and Goles 2001; Janssens 2003; Orton

2000; Piccioli 1998; Potts et al. 2006; Ruge, Barber, and Magee 2007), including samples from the unmodified bedrock (MIA_C1-6) and from the quarry site of Arasseda (MIA_C7-8) (Figure 5). The topographic position of each sample was recorded by means of a GPS system (Fornaseri, Malpieri, and Tolomeo 1975) and shown in the map of Figure 4.

The archaeometric study was performed on the three Arasseda Eneolithic menhirs hosted in the "Museo della Statuaria Preistorica in Sardegna" of Laconi (Figure 2). The discoverers labelled these menhirs as Arasseda II, III and VII respectively (Atzeni 1992; Cossu 1996).

Analyses on the geological samples

The visual inspection of the geological samples was performed by naked eye and 2.5X magnifying lens (Hermes and Ritchie 1997b; Ricq de Bouard 2002; Garrison 2003) and allowed to describe their macroscopic features.

All the eight intact samples were first analysed with a portable X-Ray Fluorescence spectrophotometer (pXRF) to determine (qualitatively and semi-quantitatively) their elemental composition. The pXRF analyses were carried out through an ASSING LITHOS 3000 spectrophotometer. The experimental conditions were defined as follows: acquisition time = 600 s; voltage = 25 kV; electric current = 150 μ Å; collimator diameter of the incident X-rays beam = 5 mm; distance between the source and the sample = 10 mm. On each sample, the analysis was performed on three different areas of the fresh rock core, detectable at 2 cm in depth under the cortical surface (Figure 6A), which was supposed not to be altered by weathering and lithospheric diagenesis (Siegesmund, Weiss, and Vollbrecht 2002; Shackley 2011; Garrison 2003; Lundblad, Mills, and Hon 2008). ASSING LITHOS software was used for the qualitative assignment of the peaks to the related chemical elements. In semi-quantitative analysis the curve-fitting procedure was performed by means of the PyMCA 4.6.0 software (Solé et al. 2007) to calculate the integrated area associated to each fluorescence Ka line (De Francesco, Crisci, and Bocci 2008). Due to the matrix effects typical of X-Ray Fluorescence, the integrated areas associated with each Ka line are not directly linked to the concentration of the element within the sample. Therefore, to evaluate possible differences in the chemical composition of the samples, analytical intensity ratios were calculated (Banning, 2000; Baxter, 1994; Dello-Russo, 2004, Hermes and Ritchie, 1997a). Over the three measurements, the average of the intensity ratios and the corresponding standard deviations were computed. The values thus obtained were used to investigate samples clustering through binary scatter plots.



Figure 4. Geological map of Allai (modified after Assorgia et al. 1995; Cherchi and Montadert 1982) showing the sampling locations of the Monte Ironi outcrop constituting the Arasseda hill.

Four samples, two coming from the Arasseda quarry and two other ones randomly selected (Orton 2000; Beardsley and Goles 2001) among those collected on the unmodified bedrock, were powdered with a shaker miller using tungsten carbide jar and balls, to avoid iron contamination due to the common steel jar and balls that could lead to overestimated iron content values (Jones, Bailey, and Back 1997; Djindjian 2002).

pXRF measurements were performed on both intact and powdered samples to evaluate the possibility to get reliable data by collecting the measurements directly on the intact specimens in a non-destructive mode and to verify potential sources of error arising from morphological or compositional inhomogeneities that can affect intact specimens.

Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) were applied on the powdered specimens in order to quantify the chemical elements which resulted effective in samples clustering by pXRF data, and to validate (by more sensitive techniques) the non-destructive approach. ICP-OES and ICP-MS analyses were carried out through the Perkin Elmer OPTIMA 5300 DV (WinLab32 software) and Perkin Elmer ELAN DRC-e (Elan software) spectrometers respectively. Powder X-Ray Diffraction (PXRD) measurements were carried out on two powdered geological samples to determine their mineralogical composition and to investigate the linked technological properties. The Mohs test performed on the minerals visually detectable on the intact samples (corresponding to the ones revealed by PXRD) and their matrix, was useful to empirically evaluate hardness (Goffer 2007). PXRD patterns were collected by a θ - θ Seifert X 3000 diffractometer equipped with a Cu K α source (λ =1.54056 Å). The experimental parameters were the following: voltage = 40 kV; electric current = 40 mÅ; angular range from 0° to 80° 2 θ ; goniometer step = 0.05 θ /s; acquisition time = 1 h. Phase identification was carried out by means of the Analyze software.

Analyses on the menhirs

Material removal from the menhirs for archaeometric analyses was not approved by the ministerial authorities. Therefore, a visual examination was performed, followed by non-destructive pXRF measurements, which reliability was first validated by destructive techniques (ICP-OES, ICP-MS) on the geological samples. The comparison between the analytical intensity ratios of the menhirs and geological samples was used to



Figure 5. Northern view of Monte Ironi ignimbrites outcropping in the Arasseda hill (A); scars (B) and tool marks (C) left by quarrying in the Arasseda quarry site.

determine the menhirs' source provenance. The adopted instrument and set-up were the same used for the geological specimens. The use of a portable equipment allowed chemical analyses of the menhirs inside the museum, avoiding their transport. To prevent the risk of surface analyses on layers potentially affected by depletion or enrichment of chemical elements (Potts et al. 2006) due to the post-depositional processes previously cited for the geological material, pXRF measurements were performed onto the recent scrapes and fractures accidentally generated during the agricultural activities leading to the menhirs finding. The X-ray irradiated area was circular-shaped with a diameter of about 1 cm while the scrapes and fractures involve far wider areas. Their depth was always higher than 2 cm, highlighting the fresh rock core of the menhirs (Figure 6B).

Results

Geological samples

Visual examination

The visual inspection of the samples coming from the Monte Ironi unit outcropping in Allai, allowed to

observe a porphyritic texture with several macroscopic (2–5 mm in diameter) lithic inclusions. The geological specimens, always grayish orange pink in colour (5YR 7/2 according to Munsell rock colour charts), also highlighted more femic phenocrysts then sialic ones (Figure 7A). These crystals were generally from 2 to 5 mm in diameter.

pXRF analysis

Qualitatively, pXRF measurements revealed a compositional set formed by K, Ca, Ti, Mn, Fe, Rb, Sr and Zr for all the intact geological samples (e.g., Figure 8A). The same elemental suite was found on the powdered samples (MIA_C2, C5, C7, C8), in which W arising from the tungsten carbide jar used for the milling process were also present (e.g., Figure 8B).

The scatter plots of the K/Fe vs Sr/Fe, Ca/Fe vs Sr/Fe and Rb/Fe vs Sr/Fe intensity ratios (Table 1) grouping the eight geological samples in a single cluster (Figure 9A).

ICP analysis

ICP-OES and ICP-MS measurements were performed on the four milled samples (labelled as MIA_C2, C5,





Figure 6. Weathered layer and fresh rock core on a Monte Ironi geological sample (A) and the Arasseda III upper fracture (B).

C7, C8). Through the K vs Sr, Ca vs Sr and Rb vs Sr scatter plots (Figure 9B) (reporting the chemical concentrations presented in Table 2), the ICP techniques confirmed the clustering shown by the pXRF intensity ratios computed for the intact geological specimens.

PXRD and Mohs mineral hardness

The PXRD analyses performed on two milled geological samples (MIA_C2, C5) revealed the presence of orthoclase (K-feldspar) as main crystalline phase, associated with quartz (SiO₂ polymorph) and orthoferrosilite (Fe-Mg silicate) (Figure 10A). All these



Figure 7. Macroscopic features of the MIA_C2 geological sample (A) and the Arasedda VII menhir (B). LI = lithic inclusion; FM = femic mineral; SM = sialic mineral.



Figure 8. Comparison between pXRF spectra collected on the intact (A) and milled (B) MIA_C5 geological sample, and on the Arasseda III menhir (C).

Table 1. Geological samples from Monte Ironi-Allai: pXRF analytical intensity ratios.

Sample	K(Ka)/Fe(Ka)	Ca(Kɑ)/Fe(Kɑ)	Rb(Ka)/Fe(Ka)	Sr(Ka)/Fe(Ka)
MIA_C1	0.126 ± 0.003	0.040 ± 0.001	0.013 ± 0.001	0.012 ± 0.001
MIA_C2	0.107 ± 0.010	0.052 ± 0.009	0.015 ± 0.002	0.013 ± 0.001
MIA_C3	0.134 ± 0.004	0.038 ± 0.002	0.015 ± 0.001	0.012 ± 0.002
MIA_C4	0.142 ± 0.013	0.049 ± 0.006	0.015 ± 0.003	0.013 ± 0.002
MIA_C5	0.142 ± 0.001	0.058 ± 0.002	0.016 ± 0.001	0.013 ± 0.001
MIA_C6	0.143 ± 0.005	0.051 ± 0.002	0.017 ± 0.001	0.013 ± 0.001
MIA_C7	0.125 ± 0.007	0.043 ± 0.004	0.013 ± 0.001	0.011 ± 0.001
MIA_C8	0.156 ± 0.007	0.047 ± 0.003	0.015 ± 0.001	0.013 ± 0.001

minerals (in the same way as the stone matrix) could be scratched with a steel tip, in accordance with the 6th and 7th levels of the Mohs index about rocks and minerals hardness (Figure 10B) (Goffer 2007).

Archaeological samples

Visual examination

Similarly to the geological samples, a porphyritic fabric with a number of lithic shards (up to 5 mm in diameter), several femic crystals and few sialic ones (from 2 to 5 mm in diameter) were found on the menhirs (e.g., Figure 7B). In the same way as the geological material, the artefacts showed a grayish orange pink colour, in agreement with 5YR 7/2 of Munsell rock colour charts.

pXRF analysis

The non-destructive pXRF measurements performed on the menhirs revealed the same chemical elements recognised on the geological material: K, Ca, Ti, Mn, Fe, Rb, Sr and Zr (e.g., Figure 8C).

The menhirs analytical intensities were used to calculate K/Fe vs Sr/Fe, Ca/Fe vs Sr/Fe and Rb/Fe vs Sr/Fe intensity ratios (Table 3).

Discussion

Due to the conservative limits imposed on the menhirs, the provenance study was based on non-destructive pXRF analyses on both the geological and archaeological samples. The non-destructive method was first



Figure 9. Scatter plots of pXRF intensity ratios (A) and ICP chemical content (B) of the Monte Ironi geological samples.

validated in terms of reliability and effectiveness in the samples clustering by pXRF, ICP-OES and ICP-MS on the powdered geological samples (for a specific study on the comparison between pXRF and ICP data, although on archaeological non-lithic materials, check Mitchell et al. 2012). The coherence of results collected on the geological material applying different techniques on powdered and intact samples, proved the possibility to obtain effective chemical information even using only the non-destructive approach. Therefore, non-destructive pXRF measurements were also carried out on the Arasseda menhirs.

The comparison between the compositional data nondestructively collected by pXRF on geological specimens and menhirs highlighted a good chemical affinity between Arasseda II-III and the Monte Ironi geomaterial, in agreement with the visually detected similarities. The third menhir (Arasseda VII) did not show a reliable link with the local volcanic source (Figure 11).

Table 2. Geological samples from Monte Ironi-Allai: ICP-OES and ICP-MS absolute concentrations.

ICP-OES (ppm)			ICP-MS (ppm)	
Sample	К	Ca	Rb	Sr
MIA_C2	14.58 ± 0.03	40,859.40 ± 0.18	304 ± 0.05	87.76 ± 0.05
MIA_C5	15.09 ± 0.01	41,395.42 ± 0.11	307 ± 0.08	85.65 ± 0.04
MIA_C7	15.20 ± 0.01	42,924.88 ± 0.20	314 ± 0.12	83.71 ± 0.02
MIA_C8	15.10 ± 0.02	49,092.74 ± 0.15	291 ± 0.24	82.11 ± 0.01

Chemical comparison between geological and archaeological samples showed the exploitation of the Monte Ironi outcrop to produce two (Arasseda II and III) out of three menhirs analysed for the Arasseda megalithic context. Because of Arasseda II-III analytical data are very close to the ones obtained for all the Monte Ironi geological samples (including those from the quarry), it was not possible to confirm the provenance of these artefacts from the quarry site. Although Arasseda VII showed a great macroscopic affinity with the Monte Ironi raw material, its pXRF data revealed Sr/Fe and Ca/Fe values higher than the geological samples' ones. It is possible that Arasseda VII comes from an unknown stone source visually similar to that of Monte Ironi or, more probably, from a Ca and Sr rich Monte Ironi zone but today not detectable due to the local quaternary soil coverage (Assorgia et al. 1995, 1998). The supposed intra-source variability was not revealed by all analyses performed on the collected geological samples but it cannot be excluded because a finer sampling grid might intercept localised variation of the bedrock.

Being Monte Ironi ignimbrite the raw material constituting two out of three Arasseda menhirs, it was characterised by PXRD and Mohs hardness measurements to provide information on its technological properties.

PXRD measurements revealed a mineralogical suite composed of minerals with Mohs hardness ranging



Figure 10. PXRD patterns recognised on the MIA_C2 and MIA_C5 geological samples (A). List of the minerals and corresponding Mohs hardness (B).

Table 3. Archaeological samples: pXRF analytical intensity ratios.

pXRF						
Sample	K(Ka)/Fe(Ka)	Ca(Ka)/Fe(Ka)	Rb(Ka)/Fe(Ka)	Sr(Ka)/Fe(Ka)		
Arasseda II	0.111 ± 0.023	0.045 ± 0.006	0.014 ± 0.002	0.011 ± 0.001		
Arasseda III	0.114 ± 0.006	0.052 ± 0.003	0.014 ± 0.002	0.013 ± 0.002		
Arasseda VII	0.129 ± 0.005	0.131 ± 0.003	0.016 ± 0.002	0.017 ± 0.001		

from 6 to 7 (Goffer 2007; Rapp 2009): orthoclase, quartz and orthoferrosilite. A similar hardness was recognised on the rock matrix in which the minerals are embedded, confirming the status of hard-stone of the Monte Ironi ignimbrite. In Arasseda the use of a stone raw material as the one of Monte Ironi, surely not easy to work by means of prehistoric technologies, is related to the absence of viable options among the alternative sources present in the vicinity of the site selected for the menhirs'erection. In fact, the others geo-sources of Allai are totally useless for sculptural purposes due to the above mentioned structural defects. However, the forced choice resulted to be a high-performing raw material characterised by technological properties as durability (Zaykov et al. 1999) capable of ensuring the long life of the menhirs iconography and its related significances, as confirmed by the good preservation of the Arasseda menhirs basreliefs (Figure 2) in agreement with the results previously obtained for Laconi Menhirs (Serra, Mameli, and Cannas 2016b). In the frame outlined, the employment of the local ignimbrite also avoided the difficult long-distance transportation of megaliths from fairly far distances (Pirson, Toussaint, and Frèbutte 2003).

Conclusions

In agreement with Mitchell et al. (2012), the results of non-destructive pXRF analysis, although concerning a limited number of major, minor and trace elements, are sufficient to reveal chemical differences/similarities useful for a samples clustering. This claim is proved by the chemical features of the geological samples of Allai that are grouped in a unique cluster by non-destructive pXRF, ICP-OES and ICP-MS data. Because of this result, the non-destructive pXRF technique can be considered an effective tool of analysis, especially to characterise highly valuable archaeological artefacts inaccessible to invasive approaches, as in the case of the anthropomorphic menhirs herein studied.

On the basis of the assumptions previously reported, a provenance study of the Allai menhirs were carried out by means of non-destructive pXRF, enabling to identify the stone source of two of them in the ignimbrites of Monte Ironi outcropping near the erection site of the artefact, and highlighting a small sourcing area closed to the megalithic context of Arasseda. In the Sardinian framework, a similar case is known for the Eneolithic human-shaped menhirs of Laconi, probably dating back to the first stages of the III millennium BC (Serra, Mameli, and Cannas 2016b). Outside the island, a short-distance raw material transportation was demonstrated for the Italian anthropomorphic "stelae" of Groppoli (Di Battistini, Franzini, and Lezzerini 2008) and Saint Martin de Corléans (Rubinetto et al. 2014) dating back from Copper Age to Bronze Age (3rd - 2nd millennia BC), and for a number of Neolithic Belgian menhirs alignments (Pirson, Toussaint, and Frèbutte 2003).

The preventive designation of the Arasseda hill as the place to host megaliths, could be the first reason that led to the adoption of the Monte Ironi geo-resouce



Figure 11. Comparative plots of pXRF intensity ratios computed for the Allai menhirs and the Monte Ironi geological samples.

to make the artefacts studied in this paper. The lack, among the stone raw materials present in Allai, of suitable alternative to the Monte Ironi ignimbrite (from a structural standpoint), imposed the employment of this volcanic rock in menhir manufacture. Although the choice was mainly regulated by the local reduced availability of stone sources (rather than intrinsic technological properties of stone), the exploitation of this rock achieved positive side-effects on menhirs production. In facts, the localisation of its outcrop in Arasseda itself, allowed to avoid long-distance raw material procurement and favoured the workforce cost saving in megaliths' transportation. Finally, an important implication probably unexpected by prehistoric Sardinian craftsmen of Allai, was the durability of this hardly workable stone (depending on hardness revealed by PXRD and Mohs patterns) which ensured a long-lasting preservation of the menhirs symbolic bas-relief, keeping alive their religious and political value (cfr. Serra, Mameli, and Cannas 2016a, 2016b).

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