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Archaeometallurgical Investigation of Iron Production at Megalithic Site of Malli, Vidarbha, Maharashtra

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Abstract: *The Indian subcontinent has garnered significant attention for early ferrous-metal manufacturing and the production of high-carbon wootz steel. Excavations at Malli, an Iron Age site in Gondia district, Maharashtra, revealed the presence of furnaces, iron antiquities, besides abundant slags datable to the mid-second millennium BCE. The current paper deals with two aspects of early Iron Age metallurgy at Malli. First, the results of chemical analyses and microstructural observations of iron slags, using energy-dispersive X-ray spectroscopy (SEM-EDS), X-ray diffraction (XRD), and X-ray fluorescence (XRF), are presented. Secondly, it attempts to offer archaeo-cultural insights from the results of a scientific survey.*

Keywords: *Megaliths, Vidarbha, smelting, iron furnace, slag compositions, early ferrous manufacturing*

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Introduction

The early Iron Age in Vidarbha, Maharashtra, is associated with the Megalithic culture, which first witnessed the widespread use of iron. This era was characterised by master iron smelters and foragers who created a culture that bore testimony to their rich social, religious, economic, and technological traditions. One notices an overwhelming presence of Megalithic burial sites in Vidarbha as compared to habitation sites. This disparity also extends across the geography of Vidarbha, as the number of sites in eastern Vidarbha significantly outnumbers those in its western part. Malli is not only the first megalithic burial-cum habitation site reported from western Vidarbha but also the first megalithic site excavated there. Malli is also significant due to its early evidence of ferrous-metal manufacturing. Iron and slag samples from this site were taken up for scientific studies to understand the technical aspects of iron metallurgy dating back to the mid-second millennium BCE. The results of the same are detailed in this paper.

Regional and archaeological setting

Central India, particularly the eastern part of Maharashtra, i.e., Vidarbha, features numerous megalithic burials. Archaeologically, this group of burials is distinctly identified as representing a specific megalithic culture, namely that of Vidarbha. The easy access to natural resources, especially rocks needed for burial preparation, favourable environmental conditions, fertile soil, and fluvial resources, as well as other essential resources for settlement and sustenance, collectively made this region attractive to megalithic peoples. This supportive ecological environment played a crucial role not only in the initial stages of human habitation in the area but also in its subsequent growth and survival. The spread of megaliths in the eastern part of Vidarbha is primarily observed along the tributaries of the Wainganga and Wardha rivers. The Malli site is located in the Gondia district within the same drainage system (Figure 1).

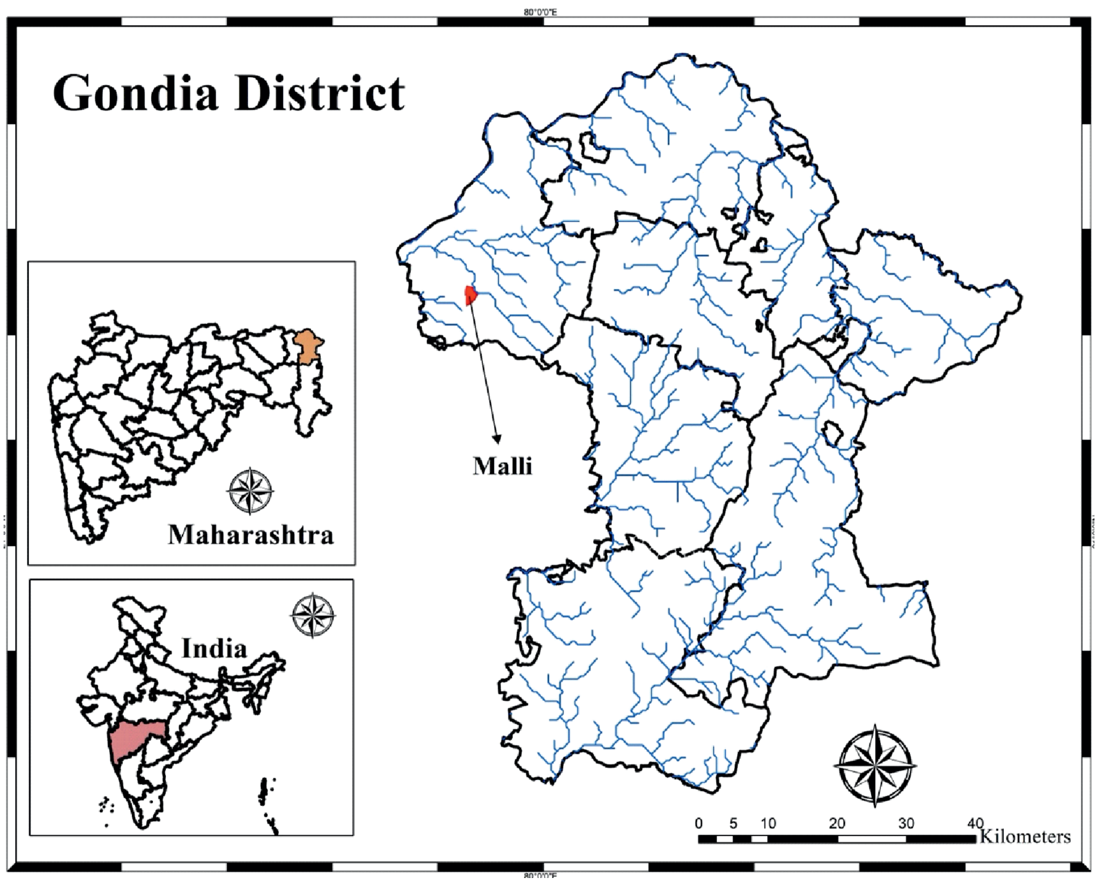

Figure 1: Location of Malli in the bank of Chorkhambara river district Gondia, Maharashtra (Prepared by Virag Sontakke)

The excavations at the site, conducted by the Directorate of Archaeology and Museums, Government of Maharashtra, during the 2010-11 and 2012-13 seasons under the direction of the first author, uncovered habitation deposits, megalithic burials and several remains related to iron manufacturing (Sontakke 2015:43-45). The habitation deposit of Malli was situated on the bank of the Chorkhambara River and was accumulated in the form of five mounds nearby. The survey conducted at Malli revealed 396 megalithic burials of various types. Main megalith types include stone circles, cairns, cists, menhirs, dolmens and capstones. In addition to numerous typological variations, the megaliths of Malli are also significant for their variations in inner architecture, as such types are not commonly found in other parts of Vidarbha. The habitation deposits were found nearly 1 km away from the cemetery. Although the top deposit of the mounds had washed away, surface surveys and trial test pits at all the mounds suggested their association with the early Iron Age period. The excavations were conducted on Mound 2 and Mound 5 due to their intact nature. Excavations at Malli revealed a twofold culture sequence. Period I is associated with a microlithic assemblage, which belongs to a non-ceramic deposit. The main deposit of the excavation at Malli was identified as belonging to the Early Iron Age. A wood charcoal sample from the Early Iron Age phase was sent for radiocarbon dating to the Birbal Sahni Institute of Palaeosciences, Lucknow (BS-4001), and a date of 3400 ± 100 (1947–1494 Cal BC) was returned (Naik & Sontakke, 2022: 908). As far as material culture is concerned, besides ceramics and stone objects, Malli has given reasonably good evidence regarding iron objects. This paper will focus on the evidence and its examination of iron production at Malli.

Archaeological evidence of iron from Malli

The excavations at Malli revealed numerous metal objects that shed light on the ancient inhabitants' technological progress and cultural practices. These artefacts offer valuable insights into the period's material culture and tool types. The copper objects suggest a link to the Chalcolithic period, while the iron implements mark an important shift from the Bronze Age to the Iron Age. The discovery of iron artefacts from the megalithic period indicates the widespread use of iron. These iron objects can be categorised into weaponry, such as spearheads, dagger blades, spikes, and arrowheads, and tools vital for daily life like sickles, knives, adzes, axes, and nails. Iron tools, such as axes, chisels, knives, and other implements, at the site suggest a variety of daily activities (Figure 2).

During the excavations at the habitation deposit in Malli, scattered evidence related to iron production was uncovered. At a depth of 135 cm in trench A2 on the southern side of the trench, a large hearth-like structure was discovered. It was pear-shaped and surrounded by dark reddish-black silty soil. The extreme redness of the soil indicated high-temperature burning activity in this area. The opening of the hearth was positioned in the north, where it narrowed, while it widened towards the south. This hearth was embedded in approximately 8 to 10 cm of habitation floor, which was only partially preserved in trench A2. The hearth also yielded two circular pits near it. The smaller one was adjacent to the hearth and fully circular in plan, while the larger was located over a metre away in the section of trench A. Both pits were empty. Considering all the evidence, these pits were likely used for water storage, and the pear-shaped hearth was probably an iron smelting furnace (Figure 3). The complete absence of bones and potsherds in this activity area points to the non-residential nature of this habitation floor. Iron ore (magnetite and hematite), smelting slag from furnace remnants, tapping slag, liquid slag, bowl-shaped forging slag, small forging slag, and other materials amounted to 70 bags and 500 individual pieces, indicating large-scale and repeated iron working at the site (Figure 4). Although no tuyere remains were recovered during the excavations, the abundance and variety of slags, along with numerous complete iron implements, suggest that iron working was conducted on-site during the early Iron Age.

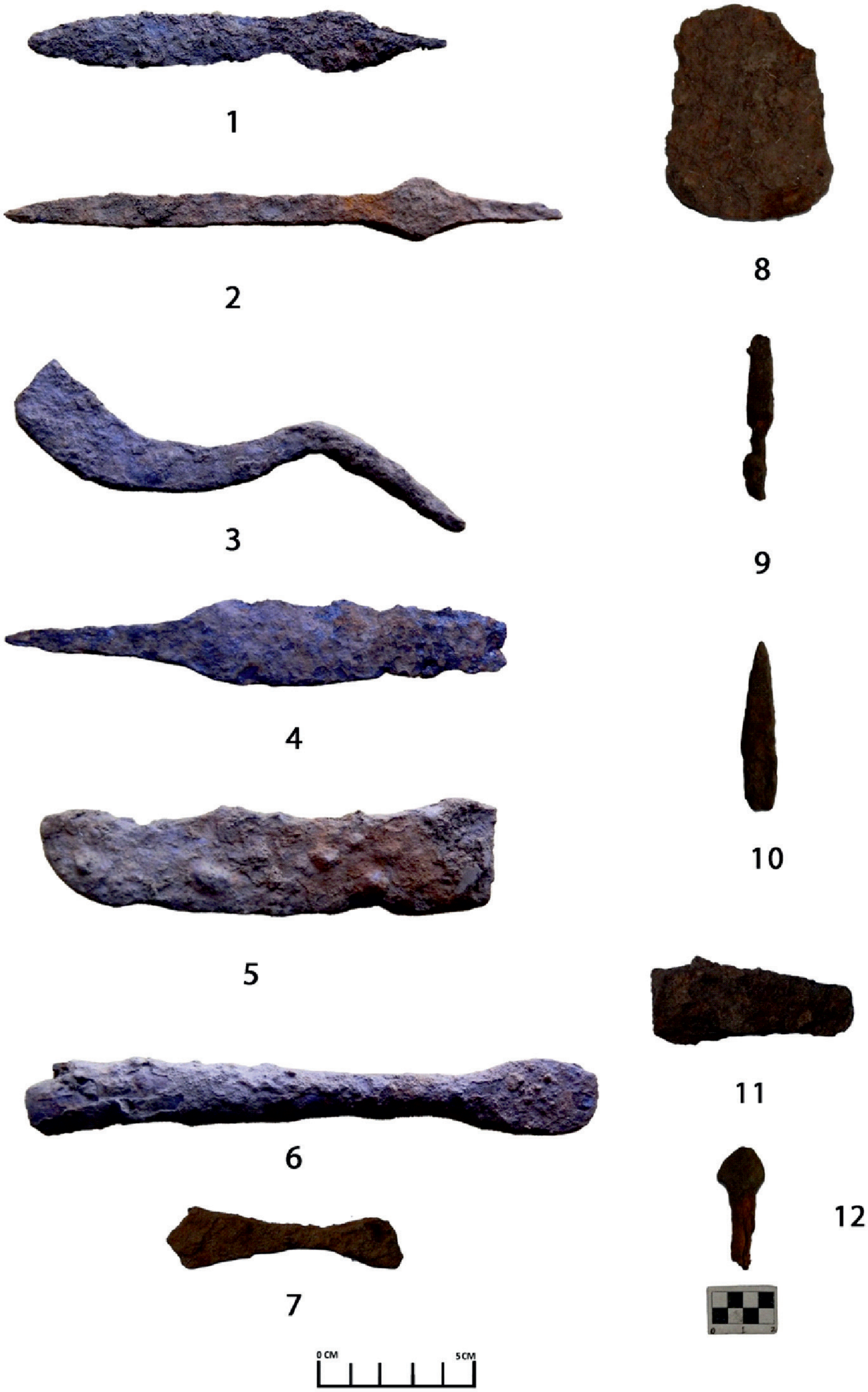

Figure 2: Iron objects found in habitation deposit at Malli. Photo: Virag Sontakke

Figure 3: The iron smelting floor at trench A2. Photo: Virag Sontakke

Figure 4: Iron slags and ore found in the excavations of trench A & B. Photo prepared by: Akinori Uesugi

In trench A1, at a depth of 165 cm, a concentration of iron waste was observed (Figure 5). The ongoing firing activity in this area caused the soil to turn dark red to blackish. Further excavations here revealed a large amount of blackish, gritty iron waste material emerging from the mouth of a circular furnace (Figure 6). This furnace appears to have been destroyed and reused multiple times, as indicated by changes in the slag flow, remnants of which can still be seen near the furnace. About a metre away from this structure, another small circular iron furnace was discovered in the same deposit. Several pieces of iron ore and iron slag nodules were also found near this second furnace. Since only a few Iron Age sites in India have provided evidence of on-site smelting, the material discovered at Malli offers a unique window into understanding iron technology during the early Iron Age in South Asia (Deo and Jamkhedkar 1982). The site is therefore of great importance, as it has revealed evidence of iron ore, iron-smelting slag, smithery slag, and an active and long-lived settlement.

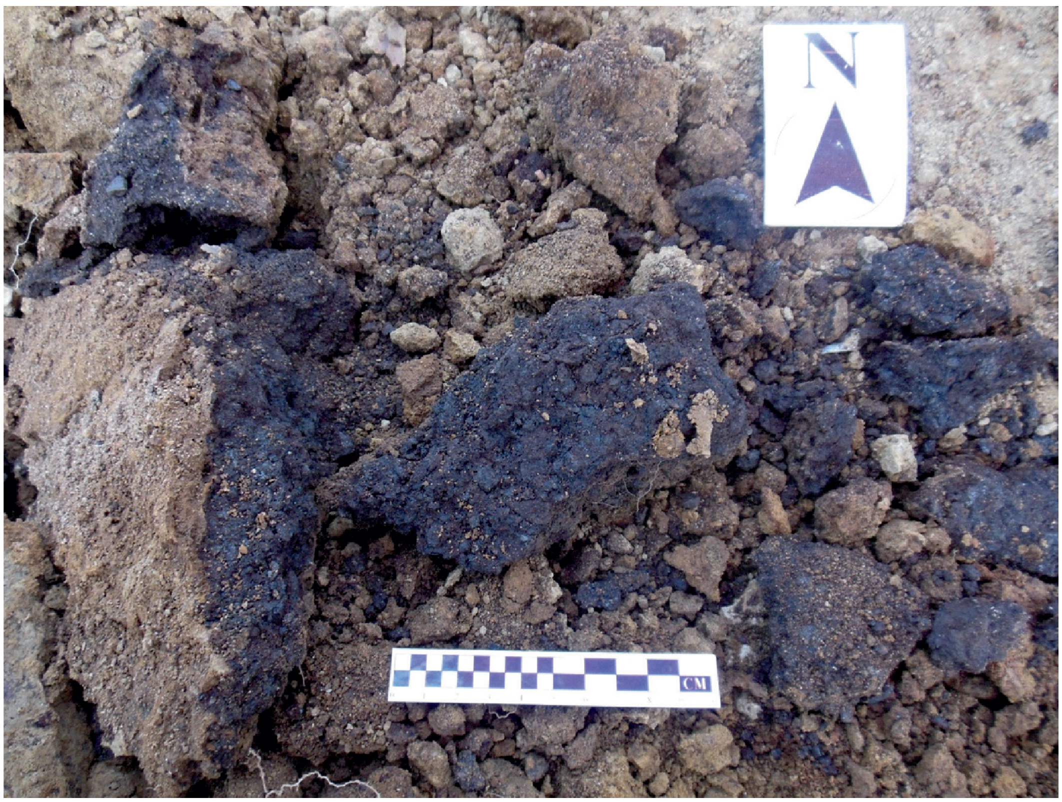

Figure 5: Lumps of iron waste recovered from the trench A. Photo: Virag Sontakke

Metallurgical Analysis of Iron Slag

Materials and Methods

Three slag specimens were selected by Tomotaka Sasada from an archaeological point of view, based on excavation context and macroscopic characteristics recorded during post-excavation examination (Fig. 7).

During primary handling, each selected specimen was assigned a lot number and stored separately in a labelled bag. These three specimens correspond to Lot nos. 023, 007, and 008 in the field observation record. For laboratory analysis in Japan, two separate analytical points were examined on each specimen, resulting in six analytical labels (Slags 1–6) used in the present study. Thus, Slags 1–6 do not represent six independent slag specimens, but six analytical points taken from three archaeologically selected slag specimens (See table 1).

Figure 6: Circular iron kiln with the iron slags and waste found in Malli at trench A. Photo: Virag Sontakke

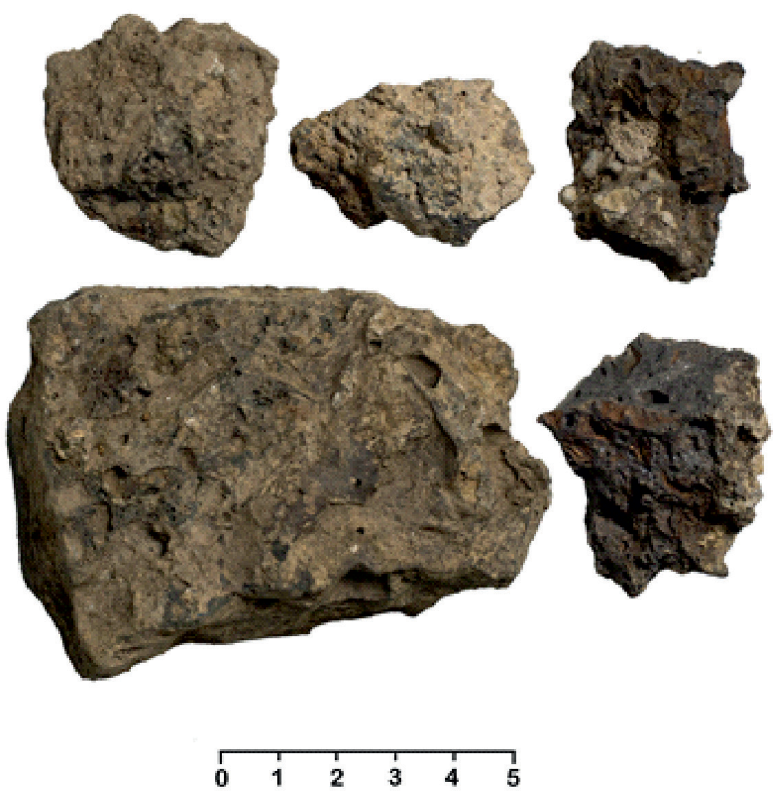

Fig. 7: Slag samples for analysis

Table 1: Correspondence between analytical slag numbers and archaeologically selected slag specimens from Malli

Analytical slag no.	*Lot no.*	*Context*	*Depth*	*Macroscopic category*	*Macroscopic features / remarks*	*Note*
Slag 1	023	Mound 2	Not recorded	Smithing slag	A relatively large fragment of smithing slag with abundant residual iron.	First analytical point examined on Lot no. 023
Slag 2	007	Mound 1 (B4)	-96	Smithing slag	A fragment of small smithing slag. The upper surface is rough and irregular.	First analytical point examined on Lot no. 007
Slag 3	008	Mound 1	Not recorded	Smithing slag	A bowl-shaped smithing slag fragment with a cracked, uneven upper surface; its weight suggests relatively high-temperature smithing.	First analytical point examined on Lot no. 008
Slag 4	007	Mound 1 (B4)	-96	Smithing slag	Same as Slag 2.	Second analytical point examined on Lot no. 007
Slag 5	023	Mound 2	Not recorded	Smithing slag	Same as Slag 1.	Second analytical point examined on Lot no. 023
Slag 6	008	Mound 1	Not recorded	Smithing slag	Same as Slag 3.	Second analytical point examined on Lot no. 008

Note: Slags 1–6 do not represent six independent slag specimens; rather, they correspond to six analytical points taken from three archaeologically selected slag specimens. The correspondence is as follows: Slags 1 and 5 = Lot no. 023; Slags 2 and 4 = Lot no. 007; Slags 3 and 6 = Lot no. 008.)

In the analysis of the slags, energy-dispersive X-ray spectroscopy (SEM-EDS), X-ray diffraction (XRD), X-ray fluorescence (XRF), and Mössbauer spectroscopy were employed. X-ray diffraction provided structural and phase information, XRF and SEM, composition, and the Mössbauer analyses of the Fe2+: Fe3+ ratios in the samples. The slags were analyzed using a Hitachi High Technologies SU-6600 scanning electron microscope with energy-dispersion (SEM-EDS) at an accelerating voltage of 15 kV; RINT-Ultima III Rigaku X-ray diffractometer (XRD) with a wavelength of 0.15418 nm produced by a Cu-ka filament; and Supermini Rigaku X-ray fluorescence (XRF) analyzer. For the SEM analyses, the slags were cut and polished to obtain a flat surface; the polished samples were also used for the XRD analyses. The XRD data were collected over a 2q range of 10–80°, with a step size of 0.01° and a dwell time of 1 s per step. As for the EDS analyses, standardless quantification was employed for the elemental mapping and spot analysis to determine the chemical distribution within the polished slag cross-sections. For compositional analyses, XRF analyses were carried out on pulverized samples. By combining the results, it became possible to retroactively determine the operating temperatures and atmospheres during the iron-making processes.

Additionally, two iron objects from the habitation deposit of the early Iron Age were chosen for analysis. One was an unspecified iron object (MLI 42), and the other was a knife (MLI 66). Both were covered with a thick layer of corrosion; however, they had areas of uncorroded metal inside. A small sample was cut from the interior to examine the uncorroded metal region. These samples were mounted in resin, and their surfaces were polished to remove all scratches. The resulting mirror-like surfaces were etched with 5 % nital etchant. Additionally, samples for metallographic observation were examined using optical microscopy (OM) and electron probe microanalysis (EPMA). Vickers hardness tests were conducted on both samples.

Results

XRD analysis identified two types of slags originating from the smelting and smithery processes. The results of selected samples are shown in fig. 8. The results confirm the presence of iron-rich phases, predominantly goethite (FeOOH), alongside silicate phases such as fayalite (Fe_2SiO_4). Notably, sharp diffraction peaks for goethite indicates the high crystallinity, and it suggests that the slags underwent a relatively slow cooling process or were maintained at high temperatures just below their melting point for an extended period, allowing for significant grain growth.

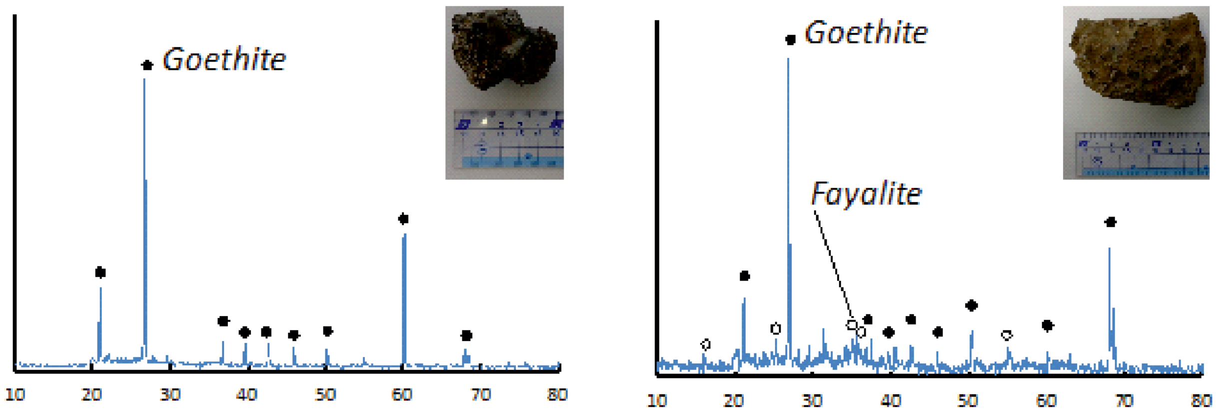

Figure 8: XRD analysis of Malli slags in the smelting (left) and smithery process (right). The x-axis is in 2 (deg). Photo: Eiji Yamasue, Benjamin Sabatini, Tomotaka Sasada

SEM-EDS images and compositions from the slag analyses conducted on samples from Malli are shown in figures 8 and 9. It was observed that the slags could be roughly identified as of two types based on high and low Fe content in the form of FeO+Fe_2O_3. The analytical labels Slags 1–6 correspond to six analytical points measured on three archaeologically selected slag specimens (Lot nos. 0023, 007,

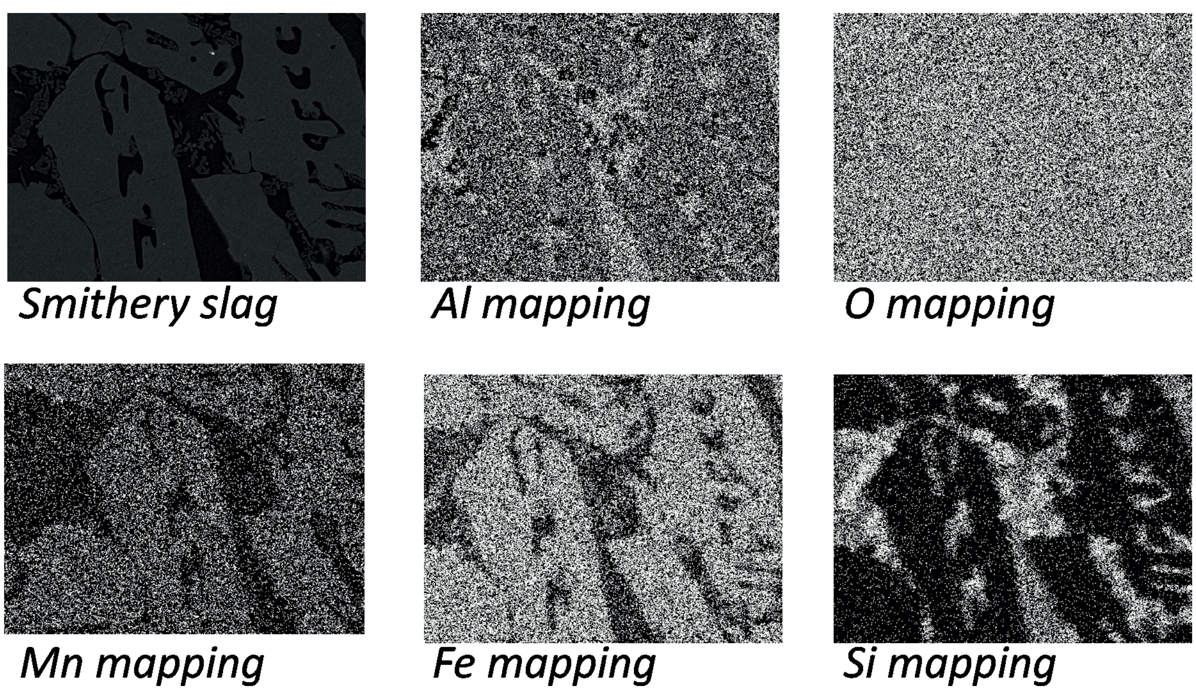

Figure 8: SEM-EDS images for smithing slags from Malli. Photo: Eiji Yamasue, Benjamin Sabatini, Tomotaka Sasada

and 008; see Table X). Macroscopically, these three specimens were classified as smithing slag in the field observation record. This aligns with the results of XRD analysis. Because two analytical points were examined on each specimen, the paired labels (Slags 1/5, 2/4, and 3/6) represent intra-specimen variation within the same archaeological specimen. The results revealed that slag samples 1, 2 and 3 had high iron content going up to 90% of the total composition**.** It must be mentioned here that these samples with higher Fe content could not strictly be classified as slag and may have resulted from the presence of an oxide scale. Conversely, samples 4, 5 and 6 with lower Fe content, appears to be a typical silicate-based slag.

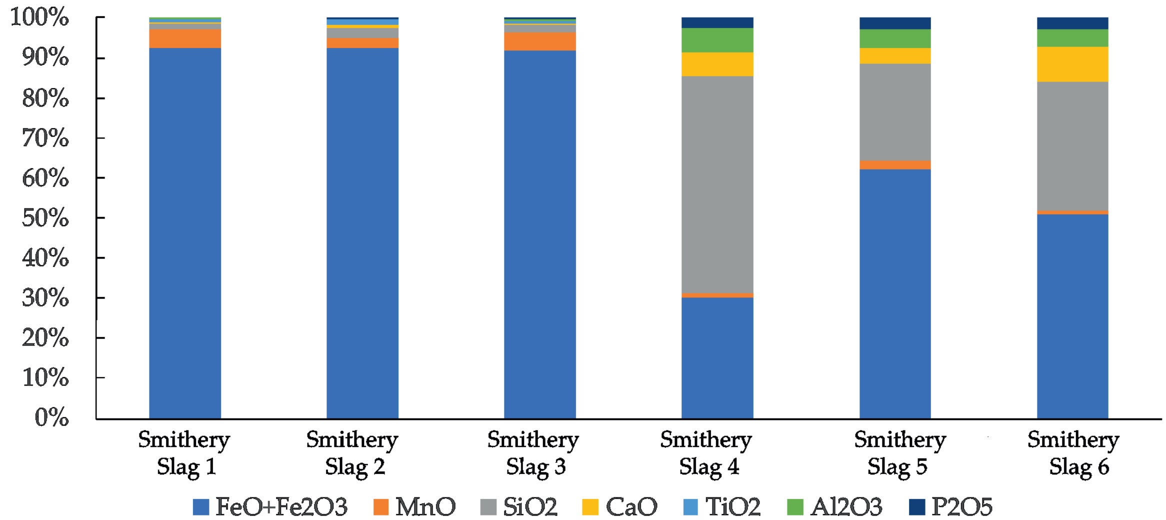

Figure 9: Composition of several smithery slags from Malli. Prepared by: Eiji Yamasue, Benjamin Sabatini, Tomotaka Sasada

The compositional and basicity details pertaining to six slag samples from Malli as revealed from geochemical study are shown below in figure 10.

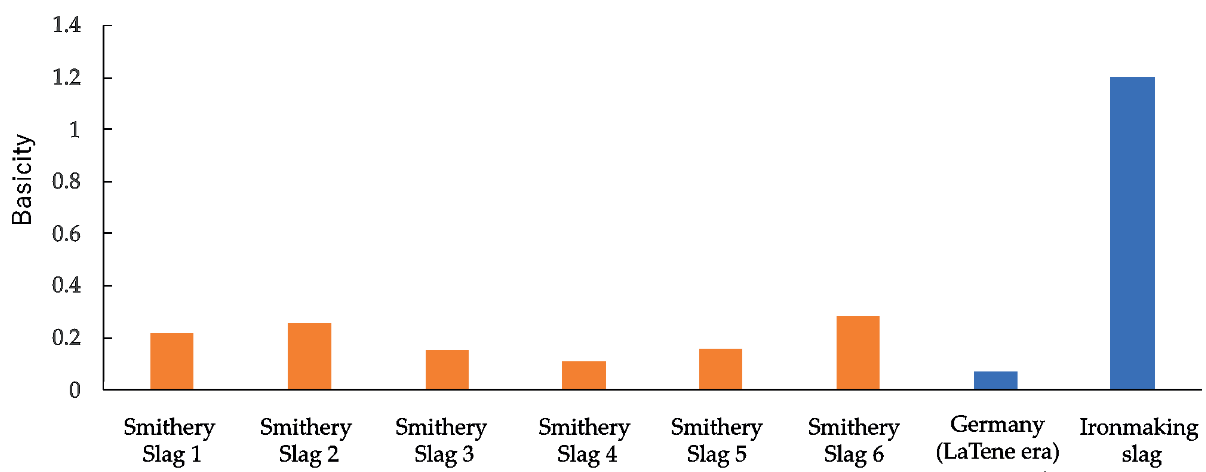

Figure 10: Basicity of several smithery slags from Malli. Prepared by: Eiji Yamasue, Benjamin Sabatini, Tomotaka Sasada

As per SEM mappings, elemental Fe, Mn, and Al are present throughout, with Fe and Mn showing some coexistence and segregation. The results indicate that Si occurs in lower amounts of Fe, alongside a small quantity of fayalite. It also became clear that the basicity of each slag (molar ratio of CaO to SiO_2) in this study averages around 0.2. This means that all of the slags were produced under similar conditions at Malli.

SEM imaging and EPMA analysis was performed for the two iron objects from Malli-MLI 42 and MLI 66. The high magnification optical image revealed that the upper part of the iron object MLI 42 was distinguished from the lower part by its microstructure. The microstructure of the upper part was ferrite with perlite, while that of the lower part was a single ferrite phase. EPMA analysis revealed that the upper part (ferrite with perlite phase) contained about 0.16 wt% carbon, whereas the lower part contained nearly 0 wt% carbon. The Vickers hardness of each part was HV 196 and 155, respectively. The lower ferrite part was probably formed by decarburising low-carbon steel, that was heated at a high temperature for an extended period in an oxidising atmosphere (Figure 11).

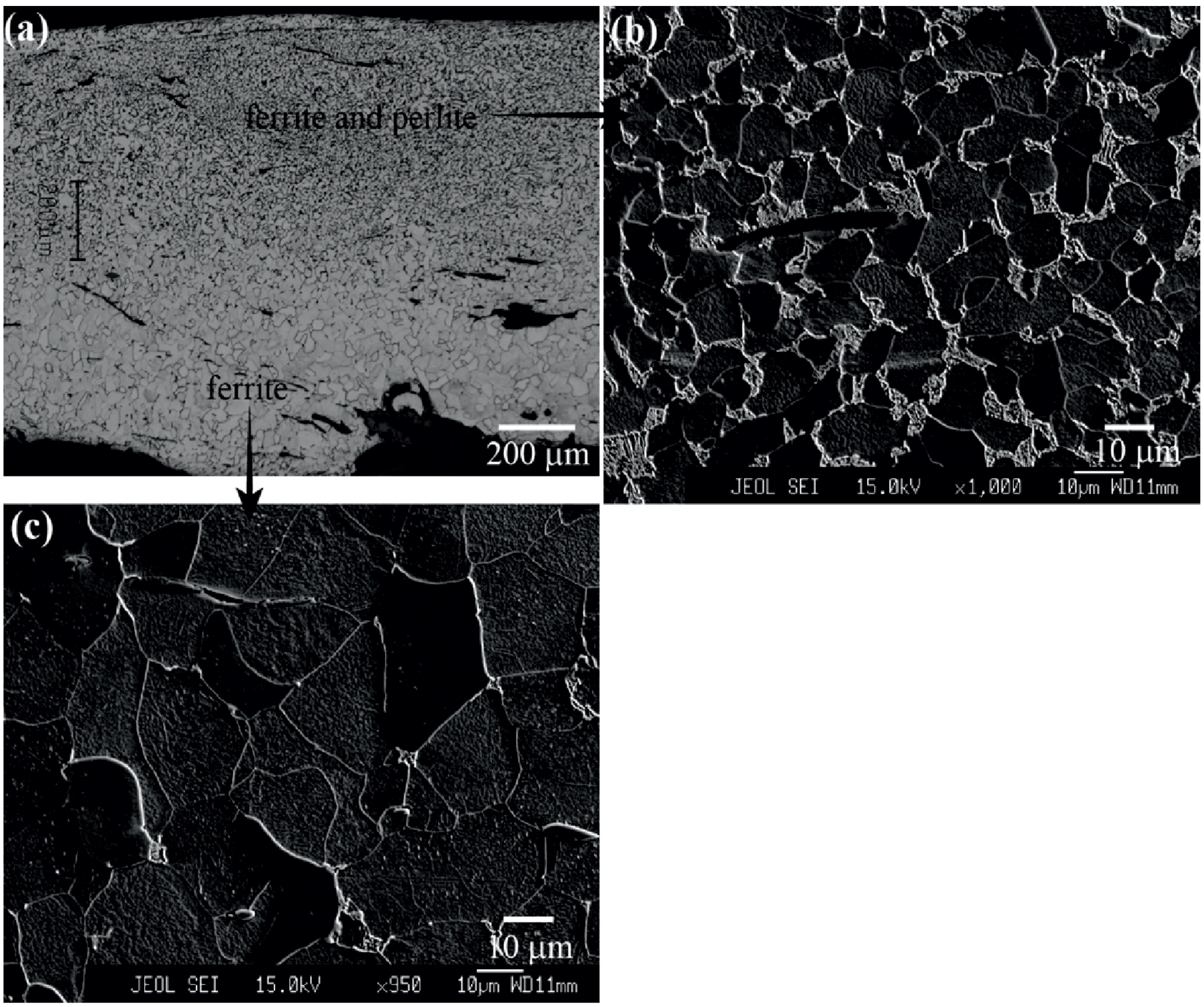

Figure 11: Microstructure of unspecified iron object from Malli (MLI 42). Prepared by: Eiji Yamasue, Benjamin Sabatini, Tomotaka Sasada
(a) Optical Image (Low magnification) (b) SEM Image (High magnification) at the upper part.
(c) SEM Image (High magnification) at the lower part.

Optical image of the sample MLI 66 (a knife), generated by SEM, showed that the microstructure of the cross-section was also composed of two parts. Unlike unspecified iron object (sample MLI 42), both parts of MLI 66 revealed the presence of ferrite with perlite. The difference between two parts in MLI 66 was carbon content. Whereas the upper part of MLI 66 contained about 0.14 wt% carbon, the lower part contained about 0.36 wt% carbon. Therefore, the perlite ratio in the lower part was more than that of the upper part. Vickers hardness of the upper and lower parts was HV 156 and 224, respectively (Figure 12).

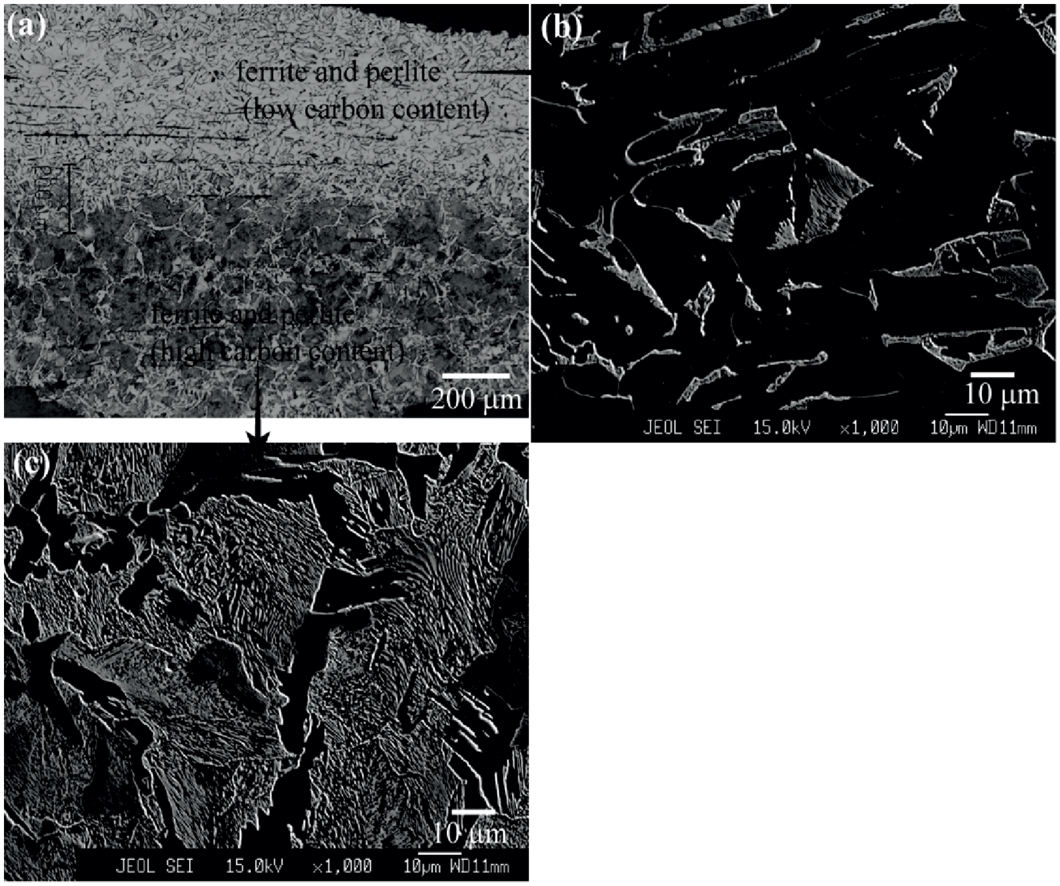

Figure 12: Microstructure of an iron knife from Malli. (MLI 66). Prepared by: Eiji Yamasue, Benjamin Sabatini, Tomotaka Sasada
(a) Optical Image (Low magnification) (b) SEM Image (High magnification) at upper part.
(c) SEM Image (High magnification) at the lower part.

Observations

- XRD analysis of six slag samples from Malli revealed that the two of the slag samples, have noticeably sharp peaks of goethite (FeOOH), and a small continuous halo between q = 20~35. These results are noticeably different from common iron-making and smithing slags, which generally consist of a halo with several sharp peaks that are typical of a glassy structure.
- The SEM images indicated a well-formed crystalline phase in slag samples which is different from the usual slags. One possible reason for this crystallinity could be that the slag was subjected to high temperatures just below melting point for an extended period, promoting the growth of larger crystals. This suggests that the smithing at Malli was likely continuously performed without removing the waste products, leading to unusually large crystals. Alternatively, the slags may have been exposed to other high-temperature sources during their history, independent of their original processing.
- Considering that the phosphorous content in iron ores is only 0.17%, there is a high likelihood that the iron products at Malli contained lower concentrations of phosphorous and, therefore, were of higher quality. The chemical composition of the slags revealed that P 2 O 5 concentrations ranged from approximately 0.2 to 3.0 wt% (Figure 9). Given that the phosphorus content in the local iron ores is measured to be only 0.17 wt%, this represents a significant enrichment in the slag phase—up to 17 times the concentration of the ore. This high enrichment factor suggests that the

smelting and refining processes at Malli were highly effective in dephosphorizing the metal. The partitioning of phosphorus into the slag, likely facilitated by the consistent basicity (CaO/SiO 2 ~ 0.2), ensured the production of high-quality iron with low phosphorus content, thereby minimizing the risk of "cold shortness" (brittleness) in the final implements.

- Basicity is a key indicator used to evaluate slag properties such as its dephosphorisation ability. The high CaO: SiO_2 ratios of the slags from Malli, despite being low in the ore, may suggest that a calcium charge agent was added or that a refractory material containing more calcium was used during furnace construction (Yamasue 2018:526). The slags at Malli were also exposed to calcium additives, either from the charge or from furnace building materials, as evidenced by remains of whitish lime coatings in some of the furnaces from Malli. High CaO: SiO_2 ratios in Malli slags, despite being low in the ore, can also be explained by the use of calcium additives. The deliberate application of charge additives, such as lime coating in furnaces, indicates that the inhabitants of Malli were aware of the benefits of calcium additives in dephosphorisation in iron.
- SEM imaging and EPMA analysis performed on two iron objects demonstrated a difference in composition with regard to their microstructure, hardness and proportions of carbon content in their upper and lower part. This disparity as seen in both the specimens could be realtedwith the intended purpose associated with each part.

Concluding Remarks

The megalithic communities of Vidarbha possessed a developed and standardised iron technology that was notably adaptable. Numerous sites have furnished the evidence of iron woking and iron smelting (Deshpande *etal.,* 2011: 4; Deshpande & Mohanty, 2015: 421; Mohanty & Vaidya, 2017:287; Gogte *etal*. 1982:68).Sites like Naikund, Shirkanda and Bhagimohari provide valuable insights relating to direct evidence of iron production in Early iron age Vidarbha(Deo and Jamkhedkar 1982, Mohanty & Vaidya, 2017:289). Prior research on various items from megalithic graves indicates an advanced metallurgical tradition (Deglukar & Lad, 1992: 40; Deshpande et. *al.* 2023: 1310-1315; Gogte, 1982a & b: 52,56; Park and Shinde 2013: 3822-3833; Roy, 2019: 600). Iron tools recovered from sites such as Mahurjhari (Deo 1970), Takalghat Khapa (Deo 1973), and Naikund (Deo and Jamkhedkar 1982) contain pure iron, with purity levels ranging from 98.2% to 99.6%, indicating a high level of technological expertise and a long tradition of practice. A similar tradition of iron manufacturing was observed at Malli. Presence of abundant slag and charcoal pieces, furnace along with patches of heavily burnt, reddish soil near the kilns indicate that iron smelting was thriving at Malli. The effective production of iron products necessitates the complete separation of metallic iron and slag. Consequently, forming low-melting slag phases is crucial for efficiently separating the slag from the metal. The Megalithic people at Malli added limestone to assist this process, as evidenced by the discovery of limestone alongside slag and charcoal thereby establishing high end knowledge and skill of the local people for such an operation.

With nearly 500 samples, the 70 bags of iron slags confirm a large-scale smelting activity ongoing at Malli. In addition to iron slag, chunks of iron ore were also discovered near the furnace. The presence of iron ore, smelted slags left behind in the furnace, tapping slags, liquid slags, and bowl-shaped forging slags, all indicate that a series of iron smelting processes, including smelting, refining, and forging were carried out at Malli. This evidence is unique, as Malli is the only site after Naikund to furnish such a large-scale evidence of iron manufacturing. Unlike Naikund, which used clay bricks to build a circular furnace, Malli's circular furnace structure was constructed with only mud and clay

(Deo & Jamkhedkar, 1982: 54). The evidence of kilns and 14C dates could be indicative of an earlier indigenous origin of iron production at Malli than at Naikund.

This work contributes to the continued study of iron-making in ancient India by providing data and insight into the resultant slag compositions and structures. Based on the types of iron remains, it appears that a series of iron production processes like smelting, refining, and forging were carried out at Malli. Though neither crucible slag nor the by-products of crucible processes were found but given the abundance of numerous iron artefacts, smithing and smelting slags, kilns, charcoal, activity areas with excessive burning, it becomes clear that the megalithic people at Malli were actively engaged in iron working. Also, while the quantity is small, finding of malachite and verdigrised slag suggests that copper production too took place at the site. Based on the above analysis Malli can be proposed as an iron manufacturing center during early iron age in Central India.

The authors have no competing interests to declare.

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