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African Iron Production and Iron-Working Technologies: Methods

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Abstract and Keywords

Iron production was a particularly important precolonial African technology, with iron becoming a central component of socioeconomic life in many societies across the continent. Iron-bearing ores are much more abundant in the earth’s crust than those of copper, and in Africa, iron was recovered from these ores using the bloomery process, until the importation of European iron in the later second millennium eventually undermined local production. Although smelting was most intensively focused in regions where all the necessary components of a smelt were plentiful—iron ore, ceramic, fuel, and water—frequent occurrences of small-scale, local iron production mean that iron slag and associated remains are common finds on archaeological sites across Africa.

The archaeological remains found on iron production and iron-working sites can provide detailed information about the past processes that were undertaken at these sites, as well as the people involved with the technologies both as practitioners and consumers. A variety of analytical approaches are commonly used by archaeometallurgists to learn more about past iron technologies, particularly those methods that explore the chemistry and mineralogy of archaeological samples. By interpreting the results of these analyses in conjunction with ethnographic, historical, and experimental data, it is possible to reconstruct the techniques and ingredients that past smelters and smiths employed in their crafts, and address important questions concerning the organization of production, the acquisition of raw materials, innovations and changes in technological approach, and the environmental and social changes that accompanied these technologies.

Keywords: iron, technology analysis, microscopy, chemistry, archaeometry

A Brief Introduction to the Role of Iron in Africa

Iron has played a significant role in the sociocultural, economic, and environmental spheres of many African communities, past and present, not only for utilitarian items, but also in the creation of symbolic, artistic, and ornamental objects. Prior to the development of smelting technologies that could extract iron metal from an ore, iron was available only in isolated naturally occurring forms, predominantly nickel-rich meteoritic iron in areas of the Nile Valley and the Red Sea coast. The rarity of this metal, perhaps in conjunction with its hard and brittle qualities, meant that it was predominantly used to make ceremonial or decorative items. This “iron from the sky” was highly prized, appearing in the archaeological record almost exclusively in burial contexts.[1]

At some point in the first millennium BCE,[2] evidence for iron-smelting technologies began to appear in several core regions (including—but not limited to—the Great Lakes region of eastern Africa, and the northwestern Sahara). In a continent where copper minerals were spatially limited yet where iron ores were near ubiquitous, the ability to obtain iron metal from an ore was a development of profound consequence, and in time these technologies spread to all corners of Africa.[3] The adoption of iron-smelting technologies, although gradual, was revolutionary.

Iron became and remained a highly valuable material in those communities that produced and used it. The malleability and strength of iron—transformed into tools, weapons, jewelry, or something else entirely—meant that the control of iron production became embedded in negotiations of power and prestige, and iron became a cornerstone of social, economic, and political life. Certainly, by the end of the first millennium AD, iron had become an everyday material across most of the continent, important not only for its physical properties and applications in agriculture and domestic life, but also for the symbolic role it played in the rituals and customs of many African societies.[4]

Making and Working with Iron

Iron-working technologies broadly include both the winning of iron from an ore (smelting) and the processing and shaping of smelted iron metal into a finished object (smithing). The process begins with the sourcing and extraction of raw materials, including clay, water, and tempering materials to make the furnace structure and the tuyères; charcoal and iron-rich ore minerals to charge the furnace; and also—as needed—sand, fluxes, or traditional medicines to encourage a successful and productive smelt.

Different techniques for mining were used across the continent, dependent on the nature of the ore deposit itself.[5] Surface collection of ore outcrops—as documented in modern and historical examples—was no doubt also widespread in the past, as was the collection of alluvial ores (such as magnetite-rich ore sands, see Figure 1) from the beds or banks of rivers. However, neither of these methods would leave much evidence for ore collection in the archaeological record.

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Figure 1. A dry river bed in Pare, Tanzania: a source of magnetite-rich ore sands.

Photo by the author.

The extraction of subterranean iron ores at either open faces or through the digging of pits is more likely to leave a recognizable trace, though it is also possible that more recent mining for the same or associated minerals might have obliterated the marks of earlier mining episodes.

Once ore has been procured, charcoal prepared, and clays obtained and shaped into furnaces and tuyères, smelting can begin. “Bloomery” or “solid-state” smelting is the process of iron production most commonly encountered in the archaeological record (so-called because the iron remains solid throughout the process, growing as a “bloom” of iron rather than melting as in a blast furnace), and it was practiced across much of precolonial Africa. The principals of bloomery iron smelting are generally well understood, and have been discussed at length elsewhere.[6]

Iron smelting comprises two fundamental chemical and physical events that need to occur together in order to separate iron metal from a host rock. Iron oxides contained within an ore have to be physically removed from the surrounding rock (or gangue) minerals. These iron oxides must then also be chemically reduced to elemental iron, by removing the oxygen atoms that are combined with the iron in its oxidized state. Bloomery smelting thus operates at a temperature below the melting point of iron (1540°C), requiring a temperature high enough only to melt the gangue minerals to enable them to separate from the unmolten iron oxides. This generally requires sustained temperatures of around 1200°C, though this is dependent on the composition of the gangue.

In order for these chemical and physical events to successfully take place, three conditions have to be met: (1) a high enough temperature to melt the gangue minerals and facilitate their separation from the iron oxides, (2) a means by which to physically separate the molten gangue from the residual iron, and (3) a carbon-rich reducing environment to separate the oxygen and iron atoms. The smelting operation takes place within a furnace designed to meet these criteria, but there is much variation possible in furnace configuration beyond these essential requirements.[7]

Air, and thus oxygen, is introduced to the furnace either through forced draft using bellows and tuyères (ceramic pipes that channel air into a furnace or smithing hearth), or by natural draft taking advantage of prevailing winds or utilizing the chimney effect, that is, the updraft movement of hot air. This enables the fuel (usually charcoal) to burn, with carbon monoxide produced as a byproduct of the incomplete combustion of the carbon-rich fuel. This oxygen-hungry carbon monoxide reacts with the iron oxide, combining with its oxygen atoms to form carbon dioxide and reducing the iron oxide to the successively lower oxides of magnetite and wüstite, which are in turn reduced to metallic iron (see Figure 2).

Not all of the iron oxide in the ore ends up as iron metal. Some of the unreduced iron oxide combines with the molten gangue minerals, as do varying proportions of technical ceramics, fuel ash, and any fluxes that have melted into the smelt. Together this ends up as the waste product of the process: slag. However, while the smelt is still progressing, the forming liquid slag plays an important role by encasing the solid particles of iron and preventing them from reoxidizing as they move within the furnace. This protective slag coating also limits the diffusion of carbon into the forming bloom, avoiding the inadvertent production of brittle cast iron.

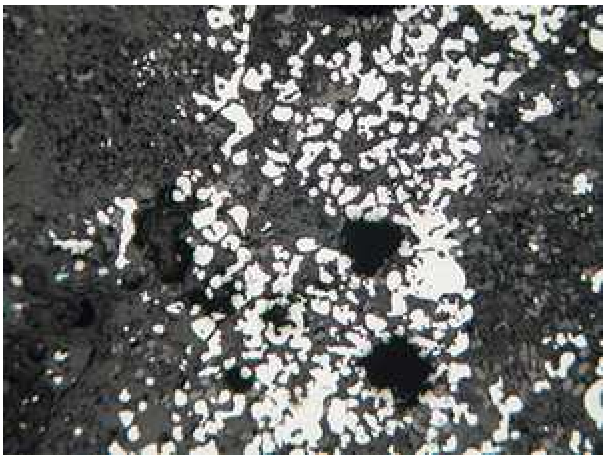

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Figure 2. Foils of reduced iron (white) in a sample of slag from Uganda. Reflected light, image width *c*. 2 mm.

Source: Louise Iles, “Reconstructing the Iron Production Technologies of Western Uganda: Reconciling Archaeometallurgical and Ethnoarchaeological Approaches,” unpublished PhD thesis, Institute of Archaeology, University College London (2011), 290.

Furthermore, the molten slag also provides a medium within which these iron particles can coalesce, allowing them to gradually combine into a spongy mass of iron bloom.

The furnace, in addition to containing the reaction process described above, must also provide a way to physically separate the slag from the iron bloom. In the case of pit furnaces, the molten slag drips into a pit dug beneath the furnace, which is often packed with plant material in order to provide a support for the charge. Alternatively, the slag can be periodically drained from the furnace (termed “slag tapping”), resulting in slag with a characteristic flow-like structure.

The timescale of this process may extend over one day, or several days, and may even require a period of re-smelting (such as the two-stage primary and secondary smelting technologies documented in Malawi and Tanzania[8]). The end result is a solid and malleable “bloom” of iron metal. This impure and heterogeneous bloom of iron initially contains a considerable amount of slag and charcoal inclusions. In order for this material to be forged, it first needs to be consolidated by repeatedly heating the bloom to a yellow heat in order for the slag to re-melt, and hammering it on an anvil to expel the trapped slag, air, and other inclusions. This is known as bloom refining.[9] Once this has taken place—in itself a considerable process, with appreciable losses in weight and size—the resultant iron billet will have become workable and is ready for smithing (to be worked into an iron object).

Smithing hearths are generally—though not always—smaller than smelting furnaces, and are often devoid of the superstructures that many smelting furnaces have.[10] Air is usually introduced into a hearth using a tuyère and bellows, and a blacksmith will use various hammers, tongs, quenching bowls, and anvils to work the iron (see Figure 3).

Click to view larger

Figure 3. Inside a traditional smithy, Pare, Tanzania, 2014. Note the traditional medicines wrapped in banana leaves (circled).

Photo by the author.

Fluxes may be used to encourage welds to join, and like smelting processes, amulets and medicines are often also present in or around the hearth. The process of shaping iron into a usable object combines many smithing techniques. Exposing the iron metal to repeated episodes of heating, cooling, and hammering alters its microstructure and thus mechanical properties, and allows the blacksmith to exert control over the hardness, brittleness, and toughness of the object as required.

Investigating Past Iron Technologies

Walking survey forms the basis of most archaeological fieldwork and serves both to indicate the distribution of sites across a landscape and to identify locations that hold particularly high potential for excavation. The robust nature of slag and the sometimes vast scale of production remains mean that iron production sites are generally easy to locate.[11] Geophysical survey techniques such as gradiometry or resistivity can additionally be used to guide excavation strategies, to explore the depth and distribution of sub-surface remains—for example, to estimate the volume of slag present at a site, as at Meroe, Sudan[12]—or to locate buried furnace structures. This last aim presents certain challenges in that it can be difficult to distinguish the signals of iron-rich slag and furnace remains from those related to natural iron-bearing deposits.

Establishing the extent of archaeological remains is key for estimating the scale of production of an iron-smelting site. Sites range from very large industrial production activity (such as the extensive iron production sites in Bassar, Togo, Meroe, Sudan, or the regions of Mema and Dogon, Mali[13]) to localized smelting on a much smaller scale (such as smelting within settlement sites of northern Zimbabwe, or pastoralist smelting on the Laikipia Plateau, Kenya[14]). Establishing a secure chronological framework is also critical for understanding the intensity and duration of production activity at these sites, and can contribute toward understanding the impact of smelting on local resources and environments, and the relationship between estimated outputs of metal, local populations, and trade.[15]

In the course of investigating the remains of iron production and iron working, it is likely that a very broad range of archaeological materials will be encountered. The most common artifact present on all iron-processing sites—whether smithing or smelting—is likely to be slag (Figure 4), followed closely by the remains of ceramic items, including furnace structures and tuyères, as well as fragments of unreduced ore and iron objects.

Click to view larger

Figure 4. Small slag heap at a smelting site in western Uganda.

Source: Iles, *Reconstructing the Iron Production*, 114.

Charcoal will often be present, and sometimes other plant remains and traces might also be found.[16]

The remains of smelting and smithing activity can appear similar, both macroscopically and microscopically. Both processes would likely result in tuyères, furnace bases, and slag in evidence at archaeological sites, and thus attributing remains to either of these processes can require judgment and experience, especially if the remains are fragmentary.[17] Observations about the scale of the remains, the size of slag blocks, the nature of furnaces or hearths if present, and the presence of plano-convex hearth bottom slag or hammerscale—both diagnostic of smithing activity—contribute to these interpretations of site function. Sites where smithing has been carried out would also potentially contain large immobile anvil-stones.

Materials on which to undertake an analysis of iron production and iron-working technologies might include discarded raw materials, technological debris and waste products, and artifacts in various states of completion. The archaeometallurgist has a suite of tools drawn from the physical sciences at their disposal to study this range of artifacts, specifically from the fields of chemistry and mineralogy. These methods provide data that can build a reconstruction of the technical aspects of the processes: the ingredients that were used, the working temperatures, the tools that were used, and so on. However, a holistic study of iron production and working technologies also requires a consideration of the sociocultural contexts that framed these actions (e.g., the choices of techniques or ingredients, the symbolic or cultural meanings embedded within the materials selected for use, or their methods of application). As much as there are technical constraints as outlined above, there is always room for cultural choice and variability.

Slag is a particularly valuable asset for archaeometallurgists, and importantly, it is one that tends to be very well preserved archaeologically. Encased within slag—in its chemistry and microstructure—is information regarding the raw materials that were introduced into the smelt (the compositions and contributions of the ore charge, fuel, fluxes, and any ceramics that melted into the slag) and how the slag formed (temperatures, cooling rates, atmospheres).

Encased upon and throughout the slag is further macroscopic and microscopic information about the structure and operation of the furnace (e.g., slag tapping or non-slag tapping, the shape of the furnace structure that the slag solidified against (see Figures 5 AND 6), or the use of plant materials in the furnace structure or as fuel (see Figure 7). Together, these strands allow the reconstruction of the chemical and physical environments of the furnace.

Click to view larger

Figures 5 and 6. Slag block in-situ, in furnace (top), and after removal (bottom, upside down), showing how clearly some slag blocks can reflect the shape of the furnace they formed within.

Source: Iles, *Reconstructing the Iron Production*, 139, 152.

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Figure 7. Slag fragment from western Uganda. White circles indicate the position of reed impressions preserved on the slag surface.

Source: Louise Iles, *Reconstructing the Iron Production*, 179.

Ceramics, in the form of tuyère fragments and furnace wall fragments, are also common at metallurgical sites. These ceramics would have had to be able to function within the challenging high-temperature environment of the furnace. Analysis of these ceramics provides information on the techniques used to form them, how they were fired and used, and the types of clay and tempering materials that they were made from. Analysis of the techniques used in their manufacture and compositional groups can give an idea about the organization of production and the spread of technological ideas, and can be fed back into the slag analyses to better understand the ceramic component of the slag melt.

Fragments of unreduced ore found at smelting sites can be useful for corroborating data derived from analyses of slag remains as to which ore or ores were used within a smelt.[18] By undertaking compositional analyses, it is possible to understand what minerals may have contributed to iron and slag formation from the ore. However, ore pieces found on a smelting site may not necessarily be representative of that used in past smelts, so these interpretations must be carefully considered.

Iron objects are a final category of material that can provide information about smelting and smithing technologies. The working methods of the blacksmith, the carbon content of the metal, and the chemical-physical conditions that the iron was exposed to as it was worked are preserved in the microstructure of the item. The hardness of the iron can also be estimated by using a Vickers microhardness tester, which applies a specific force to a very small area of the surface of a mounted and polished sample. The width of the indentation that is left behind can be measured under the microscope and used to calculate the hardness of the metal. Analysis of inclusions of slag remaining within the microstructure of objects made from bloomery iron also has the potential to link artifacts back to specific smelting technologies, such as those that used unusual ores with distinctive chemical signatures.[19]

Before undertaking any kind of analysis, it is first necessary to define the purpose of that analysis and the questions it hopes to address. Analysis can be a destructive and costly endeavour (both in terms of price and time), so it is important to tailor the analytical approach to the desired research outcome. There are many and varied questions that a researcher might want to ask of iron production remains. They might be as basic as determining what process an artifact derived from or how it had been used, or they might be as complex as assessing variation in technological practice within and between multiple metallurgical sites. However, sometimes it is impossible to address a desired research question with a given dataset. Equally, although it is preferable to ascertain research direction before data collection begins, it can in some circumstances be necessary to undertake research that is instead driven by the availability of a specific assemblage of archaeological material. This can be a significant constraint on a research agenda, as can be the availability and accessibility of analytical equipment, personnel, and funding, or curatorial restrictions on destructive sampling or the portability of archaeological items.

In general, there are three main categories of archaeometric techniques relevant to archaeometallurgy:

- Optical/imaging analysis: qualitative microstructural analysis, undertaking a visual identification of mineral phases present within a sample and their different proportions. These methods are experience based, in that a researcher’s interpretation of a given sample is based on prior knowledge of similar samples. These methods include optical microscopy (both petrographic [thin-section] and metallographic [reflected light]) and scanning electron microscopy (SEM).
- Elemental analysis: identifying and quantifying the elemental composition of a sample. These methods include SEM spectroscopy, X-ray fluorescence spectroscopy (XRF), neutron activation analysis (NAA), inductively coupled plasma mass spectrometry or optical/atomic emission spectroscopy (ICP-MS/ICP-AES/OES), atomic absorption spectrometry (AAS), and particle-induced X-ray emission (PIXE).
- Structural analysis: exploring the compounds present in a sample by analyzing the crystalline structures within a sample. These methods include Raman spectroscopy and X-ray diffraction (XRD).

However, investigations of iron production generally use these techniques in two ways: 1) to understand what a sample is formed of (i.e., its bulk chemistry or overall composition), and 2) to understand how it formed (i.e., its microstructure).[20]

Bulk Chemical Analysis

X-ray Fluorescence Spectroscopy

XRF analysis operates on the principle that primary X-rays can be used to irradiate and excite the atoms of a prepared sample, which consequently emit X-ray photons (termed secondary or "characteristic" X-rays), the energies of which are specific to the atoms from which they originated. A spectrometer—either energy dispersive (EDS) or wavelength dispersive (WDS), with the latter providing lower limits of detection and higher precision—measures the energy of the photons and counts how many there are of each energy, thereby not only identifying the elements present within a sample but also estimating how much of a particular element is present.

XRF analysis is suitable for the analysis of tuyère,[21] slag, furnace wall, and ore. It is relatively simple and inexpensive compared to other techniques, and thus is widely used. It provides reasonable major and minor element analysis, but it is not very sensitive so tends to have poor detection limits for trace elements. Light elements, those lighter than sodium, cannot be detected. This includes carbon—iron's primary alloying element. The lighter the element, the worse the detection limit.

This method requires the removal of material from the sample to be analyzed.[22] The material is then milled into a powder and made either into a pressed pellet (which is better suited to major element analysis) or a fused bead (which is best suited to minor and trace element analysis). Ideally both methods would be used on each sample. In the case of slag, which is inherently heterogeneous, the larger the sample the better, to account for internal variability. A few hundred grams might even be required, depending on the nature of the material.

Particle-Induced X-ray Emission

In contrast to XRF, where X-rays are fired at a sample to result in emitted secondary X-rays, PIXE instead uses a beam of protons to excite the sample and emit characteristic X-rays. Detection limits are low, and the beam can be used outside of a vacuum, which gives more flexibility of use (although this necessarily impacts upon sensitivity).

Neutron Activation Analysis

During NAA, samples are irradiated with neutrons, and the intensity of radioactivity characteristic for each element is measured. It is better suited than XRF for trace elements, as it is sensitive, precise, and accurate. It can be used on samples removed from archaeological items, or on small objects themselves: it is a nondestructive process, and no preparation is required. However, this method is steadily declining in popularity despite these advantages. The process itself is time consuming and expensive, as there are few suitable facilities where NAA can be conducted. A further consideration is that the samples—or the objects themselves—remain irradiated for some time after analysis, which can demand the implementation of inconvenient handling protocols.

Inductively Coupled Plasma Spectrometry

ICP spectrometry is a versatile technique, specifically targeted toward the analysis of trace elements, with good detection limits, sensitivity, and precision. One particular strength is the large number of elements it can detect. ICP-MS has a much higher sensitivity than ICP-OES, and although analyses traditionally cost more, this is becoming the most prevalent form of ICP analysis. Samples for both techniques are dissolved, usually in an acid, and this solution is sprayed into a chamber of argon gas and heated to between 8,000 and 10,000˚C. The characteristic wavelengths of the photons that are emitted are quantified using either an optical emission spectrometer (ICP-OES, which converts the measurement into elemental concentrations) or a mass spectrometer (ICP-MS, which detects the positively charged ions that are also emitted and can therefore also provide measurements of isotopic ratios of some elements).

ICP spectrometry measures many elements (between twenty and fifty) simultaneously, and thus it is a very fast analytical technique. Nevertheless, the protocol for sample preparation introduces some drawbacks, as the sample needs to be dissolved in acid. A balance needs to be struck between the dissolution of as many relevant elements as is possible, with minimal contamination and as few safety concerns as possible. For some elements, a particularly high dilution is necessary, which results in higher detection limits. Other elements might be lost completely in the sample decomposition. For these elements, it is wise to use a further analytical technique (e.g., AAS) to corroborate the ICP results.

Atomic Absorption Spectroscopy

AAS is also a solution-based, destructive method that requires solid samples to be dissolved, but it can measure only one element at a time. The sample solution is vaporized and passed through a flame, which heats the solution to a temperature that forces the molecules to decompose to individual elements, approximately 2,000–3,000˚C.

A beam of light—at a wavelength characteristic of the element to be analyzed—is passed through the vapor, some of which is absorbed. The intensity of the light after it has passed through the vapor is measured and compared to its initial intensity; this can then be used to estimate the concentration of that element in the original sample. The process needs to be repeated for each individual element that is to be analyzed.

Each of these techniques provide elemental analyses of the sample being studied. In the case of metals, this is sufficient, but for silicate materials (e.g., ceramics, slag), the elements have to be converted to oxides by stoichiometry post-analysis. In many cases, this is not problematic. For elements with several valences (and thus oxidation states, for example, iron), this means that the oxidation state(s) must be either estimated based on prior knowledge of that sample type, or investigated further, using XRD or microstructural analysis to understand the mineralogy of the sample.

X-ray Diffraction

XRD analysis requires a powdered sample to be irradiated, from which the resultant diffracted X-rays are detected and plotted as peaks that can be compared with known diffraction patterns of a reference database of mineral species. This enables the identification of crystalline structures, and thus the identification of mineral phases. This is especially useful when researchers are seeking to identify mineral phases that cannot be recognized microscopically, or to understand the proportions of, for example, the different oxide states of iron occurring within a sample. However, the sensitivity of XRD analysis depends on the complexity of overlapping diffraction patterns, or if a sample, for example, includes phases with poor crystallinity. Iron-rich phases in particular—common in slag—can be difficult to interpret as they generate many overlapping peaks.

Microstructural Analysis

Optical Microscopy

A primary means by which to examine almost all of the materials associated with iron production and iron-working technologies is microscopy. Standard optical microscopes can generally provide up to 1000x magnification, and camera attachments mean that high-quality digital images can be easily produced. By studying samples of slag, tuyère, ore, and iron metal under the microscope, it is possible to observe and identify the mineralogical compositions of those samples and their internal microstructures (i.e., the sizes of the crystal structures and how they are distributed across a sample). By comparing this with published examples of different minerals, and building on the experience of the person undertaking the analyses, it becomes possible to estimate the conditions under which those samples formed. For ceramics, this means understanding the type and size of any tempering materials, and the extent of vitrification, and the clay composition itself, and how the clay had been worked (see Figure 8).

For slag, the size and distribution of the various phases that make up the slag can provide detailed information about how the samples were formed and what conditions they had been subjected to (e.g., heat, pressure, cooling rates), as well as preliminary estimations of the chemical composition (see Figure 9).

For iron objects, an examination of patterns of grain growth, texture, and grain deformation across the sample gives an idea of the alloying components of the metal, the temperatures it had been exposed to, and the ways in which it had been mechanically deformed (see Figure 10)[23].

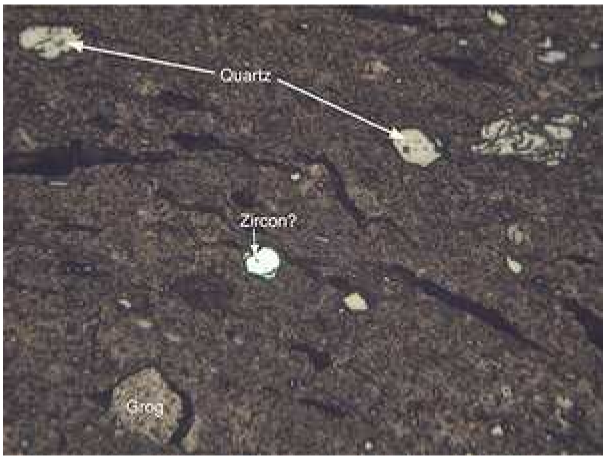

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Figure 8. A ceramic sample from an iron production site in Uganda, showing the clay matrix and a variety of different inclusions. Reflected light, image width *c.* 2 mm.

Source: Iles, *Reconstructing the Iron Production*, 243.

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Figure 9. Slag sample, showing feathery lathes of fayalite (light-grey) in a glassy matrix (mid-grey). Porosity and cracks in the sample appear black. Reflected light, image width *c.* 0.5 mm.

Source: Iles, *Reconstructing the Iron Production*, 158.

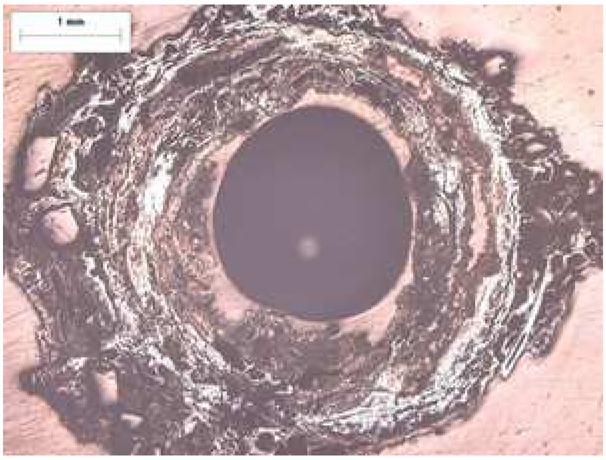

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Figure 10 here. Cross-section through a highly corroded iron bead from Tanzania. Reflected light.

Photo by the author.

Reflected light microscopy is used for samples known to be composed primarily of opaque minerals: metals, many ores, major constituents of slag. The sample surface is illuminated from above, and light reflects back up to the eyepiece of the microscope, revealing the mineralogical microstructure of the sample (see Figure 12). Samples are prepared by being cut from an artifact and set in a resin block. Then the surface of the mounted sample is ground and polished until it is very smooth and highly reflective (see Figure 11).

Click to view larger

Figure 11 here. Samples of ore prepared for reflected light microscopy.

Source: Iles, *Reconstructing the Iron Production*, 224.

In comparison, transmitted light microscopy is used for samples that are non-opaque once ground to a thickness of thirty micrometers (as prepared as a thin-section).

Light is shone through the sample, and in doing so interacts in characteristic way with different minerals depending on their color, refractive index, and optical symmetry (see Figures 13 and 14). Observations of the optical properties of these minerals enable their identification.

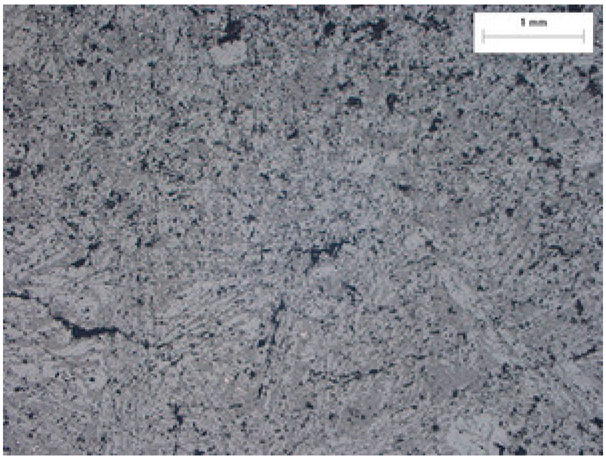

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Figure 12. Slag sample from Tanzania showing olivine phases. Reflected light.

Photo by the author.

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Figure 13. Same sample area as Figure 12, showing olivine phases. Transmitted light, plane polarized light.

Photo by the author.

Click to view larger

Figure 14. Same sample, showing olivine and orthpyroxene phases. Transmitted light, cross polarized light.

Photo by the author.

Scanning Electron Microscopy

Electron microscopy is able to produce images of samples prepared for optical microscopy at much higher magnification. It operates on the principle that an electron beam directed at a prepared sample under vacuum will excite the surface of the sample and cause electrons and X-ray photons to be emitted from it. Detection of the emitted electrons is used to generate optical images of the sample surface. Secondary electrons (SEs) are low energy, and so only those emitted from atoms nearest to the surface will reach the detector. As such, raised areas of the sample are more likely to produce SEs that can be detected, and therefore an SE image will generally reflect the topography of the sample. This can be useful, for example, in assessments of the vitrification of ceramics used in high-temperature processes.[24] Backscattered electrons (BSE), of higher energy, are a result of the interaction of the incident electron beam with the nucleus of the atoms, and their intensity is related to the average atomic number of the different phases of the exposed sample area.

BSE imaging thereby provides a visual representation of the chemical make-up of the sample, which can be useful in determining the distribution of phases across a sample (see Figures 15 and 16). However, it is most useful when used in conjunction with electron probe microanalysis.

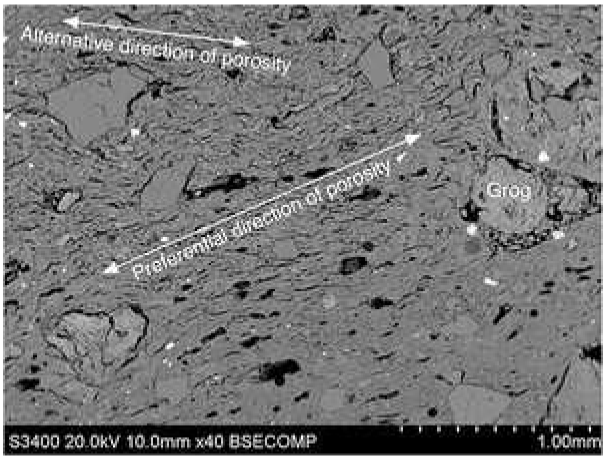

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Figure 15. BSE image of tuyère sample from Uganda, illustrating porosity and inclusions.

Source: Iles, *Reconstructing the Iron Production*, 210.

Electron Probe Microanalysis

The high-resolution imaging capabilities of electron microscopy and the qualitative data it produces can be supplemented with electron beam microanalysis to determine elemental compositions of specific areas of the samples. This produces a more detailed understanding of the internal chemical make-up of the samples and the distribution of elements across different phases. Detection of the emitted X-ray photons, as measured and counted by a spectrometer, provides a chemical analysis of selected areas of the sample, as each element produces photons of a characteristic energy. Again, there are several options for how the photons are measured, with implications for the accuracy and sensitivity of the analysis. Wavelength dispersive spectroscopy (WDS) is much more sensitive, particularly for trace element analysis, than energy dispersive spectroscopy (EDS).

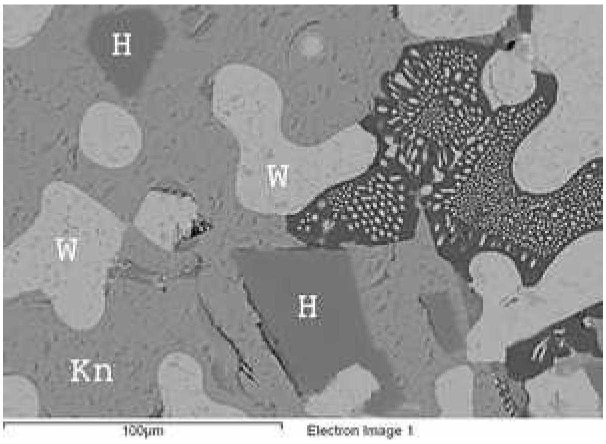

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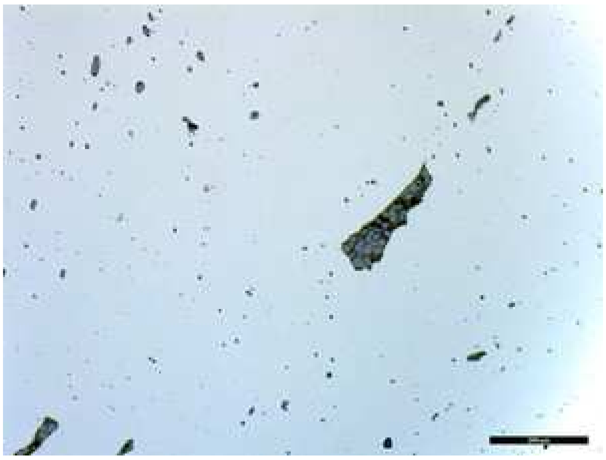
Figure 16. BSE image of slag sample from Uganda, showing wüstite (W), knebelite (Kn), and hercynite (H) phases in a glassy matrix (dark grey).

Source: Iles, *Reconstructing the Iron Production*, 290.

This technique is particularly useful when dealing with heterogeneous materials such as slag, as specific areas of interest within the sample can be pinpointed and analyzed for elemental composition. As well as being used to confirm the chemical composition of crystal structures identified through optical microscopy, this technique can also characterize those that are otherwise unidentifiable. With regards to samples of ceramic material, this method also allows for the generation of a more accurate chemical analysis of clay matrices. This is important as a bulk chemical analysis will include the compositions of any inclusions or temper (such as quartz or slag); a targeted chemical analysis of the matrix alone will facilitate an understanding of the properties of the clay itself.



Click to view larger

Figure 17. Multi-phase slag inclusions present in iron object, viewed in reflected light.

Photo by the author.

Similarly, this kind of microanalysis is able to provide chemical signatures of slag inclusions trapped within iron objects (see Figure 17), which can be useful if trying to link iron objects to slag systems.

Laser-Ablation ICP-MS

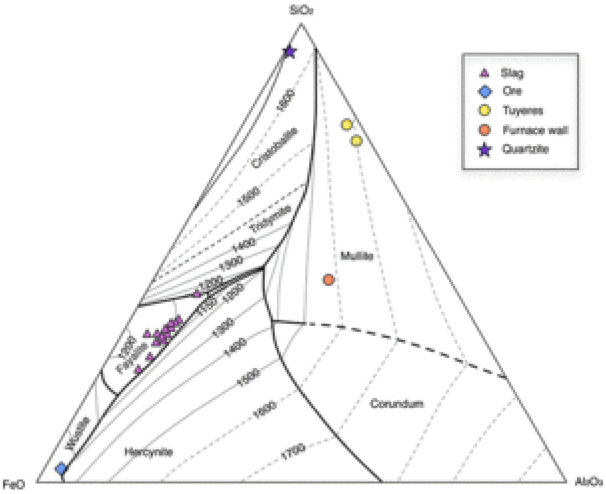
As a microprobe application of ICP-MS, laser ablation negates the need for time-consuming sample digestion in acid, and removes any risk of contamination associated with such methods (although samples still have to be flat and small enough to fit in a sample chamber—less than four cubic centimeters). A laser beam is directed at the surface of a sample, targeting and vaporizing an area of interest. Although this method does not account for sample heterogeneity (unless LA-ICP-MS is undertaken on a powder pellet or fused bead), it does allow for the elemental analysis to reflect spatial differentiations across the sample itself. Limits of detection are, however, worse than for traditional ICP-MS, and it is not possible to generate fully quantitative data using this technique.

Making Sense of the Data

Interpreting Chemical Data

For all of these analytical methods, an important factor to be considered prior to any interpretations of the data is an assessment of how reliable the analytical instrument is. The operating parameters should be checked by calibration, that is, compared to standards of known composition, preferably prepared for analysis in the same way as the analyzed samples have been. Normalization of the results to 100 percent permits easy comparisons to be drawn within the dataset and with other datasets, but can mask problems with the analyses, so it is important to consider results in both normalized and un-normalized forms.

Once reliable data has been collected on the chemical compositions of samples, the more meaningful work—the interpretation—can begin. Various methods of multivariate analyses can be used to explore the relationships between chemical and mineralogical variables and to reveal patterns of compositional variation within a dataset.[25] In order to gain a basic understanding of how a smelting system might have operated, one can plot the chemical compositions of slag samples in a ternary diagram to facilitate comparison with equilibrium phase diagrams (see Figure 18).[26]



Click to view larger

Figure 18. Ternary phase diagram showing system FeO-SiO_2-Al_2O_3 (after Slag Atlas 1995). Plotted are compositions of all analyzed archaeological samples from an iron production site in western Uganda, using PED-XRF data normalized to 100 percent.

Source: Iles, *Reconstructing the Iron Production*, 160.

These can be used to determine estimated temperatures and conditions under which the slag formed, providing a guide to the minimum operating temperatures of a furnace firing episode.

These estimations can feed back into interpretations of the associated ceramic materials, in terms of the temperatures that they would have needed to withstand, and the contribution of these ceramics to the slag melt dependent on their estimated refractory qualities. Furthermore, it is also possible to correlate information from these diagrams with the results of any microstructural analysis.

Experimental Archaeology and Ethnoarchaeology

Experimental archaeology and reconstructions of iron technologies have played a significant role in the development of researchers’ understanding of how iron production and working technologies operated in the real world. These encounters—either through undertaking experiments themselves, or observing traditional metalworking activities—have encouraged archaeologists and archaeometallurgists to go beyond a theoretical appreciation of chemical and physical processes, allowing them to experience the material, operational, and socioeconomic constraints—often unexpected—that acted upon past technologies. Both approaches have tended to focus primarily on iron smelting, but some studies have also explored mining and smithing technologies.[27]

It is certainly true that “our ability to imagine what people in the past thought was worthwhile is always limited by what we think is worthwhile.”[28] It is not possible to completely overcome this limitation—people are products of what they already know, in the same way that past metalworkers were also a product of what they knew and believed—but both experimental archaeology and ethnoarchaeology go some way to addressing the issue by providing a challenge to prevailing ideas.

Experimental archaeology offers an opportunity to test new hypotheses on production processes or the behavior of different materials, in the lab or in the field. It allows researchers to apply a more hands-on approach to problem solving, putting themselves in the role of the metalworkers that they are trying to understand. It also provides a platform through which different variables can be explored in order to produce results that best match excavated archaeological materials. However, iron technologies are complex undertakings, and before meaningful experiments can be undertaken it is necessary to build the experience and know-how of the processes in question: a path that requires a considerable investment of time, energy, and dedication. Nevertheless, this learning process and the development of a series of experiments can prove invaluable in itself by encouraging new perspectives on how technologies may have been carried out in the past—particularly, perhaps, when working outside, using "authentic" tools and methods, and dealing with an unpredictable environment.

Both experiments and reconstructions carry with them the risk of failure, and it is in this failure that new and novel ideas are often generated, stimulating fresh ideas of how past technologies operated, and constructing new hypotheses that can be tested.[29] For example, Schmidt's series of reconstructions of iron smelting in Tanzania[30]—some of which failed, some of which succeeded—documented the disagreements and negotiations between the master smelter and his team, demonstrating the role that personal interactions play in shaping the acquisition of material resources and how smelting activity is carried out.

Ethnoarchaeological studies of iron production in Africa over the past decades, and historical accounts of smithing and smelting activity recorded in the 19th and 20th centuries have been instrumental in informing archaeometallurgists working within the continent, as well as those working elsewhere, of the extensive social and symbolic frameworks that surrounded metalworking practices. These intangible aspects formed a fundamental part of past metal working technologies, but can be difficult to address archaeologically. By documenting such symbolic behaviors through ethnoarchaeological methods (interviews, reconstructions, and so on) it can become possible to provide further detail to archaeological interpretations.

As methods by which to more fully understand past iron production processes, experimental archaeology and ethnoarchaeology can address a wide scope of questions, ranging from aspects of furnace design and construction, slag formation, operational parameters, social influences on production, sequences of activity, raw material selection and preparation, and the symbolic and performative aspects of production processes. However, their limitations and reliability have to be carefully considered.[31] Ethnoarchaeological data depends on the reliability of individual sources or historical accounts, and the inherent biases that they contain. These methods are heavily influenced not only by the context in which the observation or reconstruction was undertaken, but also by the personal relationship between the researcher and the subject—factors such as trust, representation, mutual understanding, and respect. How representative a single reconstruction or interview (or set of interviews or reconstructions) can be in terms of understanding the technology of a region should also be considered, as should the fact that reconstructions today are generally far removed from being undertaken in earnest to produce iron. Since the last smelting industries in Africa drew to a close in the mid-20th century, there are very few individuals surviving that have first-hand experience of mining or smelting technologies. Smithing technologies, on the other hand, are still being undertaken in some places, or have ceased relatively recently. An ability to sufficiently assess the reliability of ethnoarchaeological data relies on an honest and open presentation of the methods of data collection by the researcher. The need for detailed reporting of experimental archaeology is similarly important and allows for the results to be relatable to archaeological data. The authenticity of "traditional" methods and tools should be scrutinized, and there should be explicit discussion of the levels of scientific control maintained over variables, especially if researchers are working in a nonlaboratory environment.

Materials generated through both ethnoarchaeological reconstructions and experimental archaeology are often examined by the chemical and microstructural methods described below, allowing easy comparisons with archaeological materials. Furthermore, measurements of the mass of inputs (e.g., ore, clay, charcoal, bloomery iron) versus outputs (e.g., slag, bloomery iron, worked iron) can be used to infer the consumption rates of raw materials in relation to archaeological sites.

Understanding African Iron Technologies: Final Thoughts

The intention of any archaeological analysis is to try and understand the people that left traces of their past actions and behaviors. Explorations of iron production and iron-working technologies are well suited to understanding the material demands and needs of past populations, as chemical and microstructural analyses can provide a secure framework for the reconstruction of the physical actions and material choices of past craftspeople. Understanding *why* they made these choices is a more difficult endeavour. Analytical investigation of iron production remains can explore how something was made and used, and what raw materials may have been involved, yet it is the interpretation of this data which allows archaeometallurgists and archaeologists to consider more nuanced questions as to the technologies and material culture as a whole, and why processes were undertaken in the way that they were: What knowledge or experience was required for different processes? Can changes be seen over time? Were there constraints on raw material selection? What sociocultural influences were there on technological choices? Only by striving to understand the wider contexts of the production and consumption of iron objects can more detailed questions about the societies that drove these production activities and the individuals who implemented them be explored.

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Notes:

(1.) Daniela Comelli et al., “The Meteoritic Origin of Tutankhamun’s Iron Dagger Blade,” *Meteoritics and Planetary Science* 51 (2016): 1301–1309.

(2.) The precise timing of the inception of iron production in different regions of Africa is much debated. See discussion in Stanley Alpern, “Did They or Didn’t They Invent It? Iron in Sub-Saharan Africa,” *History in Africa* 32 (2005): 41–94; and David Killick, “What Do We Know about African Iron Working?” *Journal of African Archaeology* 2 (2004): 97–112.

(3.) However, not all communities practiced iron-working technologies. David Killick, “Cairo to Cape: The Spread of Metallurgy through Eastern and Southern Africa,” *Journal of World Prehistory* 22 (2009): 399–414; and Augustin Holl, “Early West African Metallurgies: New Data and Old Orthodoxy,” *Journal of World Prehistory* 22 (2009): 415–438.

(4.) Eugenia Herbert, *Iron, Gender, and Power: Rituals of Transformation in African Societies* (Bloomington: Indiana University Press, 1993); S. Terry Childs and William Dewey, “Forging Symbolic Meaning in Zaire and Zimbabwe,” in *The Culture and Technology of African Iron Production*, ed. P. Schmidt (Gainsville: University Press of Florida, 1996), 145–171; and Pierre de Maret, “The Smith’s Myth and the Origin of Leadership in Central Africa,” in Randi Haaland and Peter Shinnie, *African Iron Working: Ancient and Traditional*, eds. R Haaland and P. Shinnie (Oslo: Norwegian University Press, 1985), 73–87.

(5.) See Walter Cline, *Mining and Metallurgy in Negro Africa* (Menasha, WI: George Banta, 1937); Jean Brown, *Traditional Metalworking in Kenya* (Oxford: Oxbow, 1995); and Shadreck Chirikure, *Metals in Past Societies: A Global Perspective on Indigenous African Metallurgy* (New York: Springer, 2015).

(6.) Radomír Pleiner, *Iron in Archaeology: The European Bloomery Smelters* (Prague: Archeologigky Ustav Avcr, 2000); and William Rostoker and Bennett Bronson, *Pre-Industrial Iron: Its Technology and Ethnology* (Philadelphia: Pennsylvania University Press, 1990).

(7.) See examples in Cline, *Mining and Metallurgy*; Brown, *Traditional Metalworking*.

(8.) Nikolaas van der Merwe and Donald Avery, “Science and Magic in African Technology: Traditional Iron Smelting in Malawi,” *Africa* 57 (1987): 143–172; and S. Davison and P. N. Mosley, “Iron-smelting in the Upper North Rukuru Basin of Northern Malawi,” *Azania* 23 (1988): 57–99.

(9.) Peter Crew, “Bloom Refining and Smithing Slags and Other Residues,” Historical Metallurgy Society: Archaeology Datasheet No. 6, March 1996.

(10.) See Brown, *Traditional Metalworking*.

(11.) See discussion of survey method in Louise Iles, “Iron Production in Uganda: Memories of a Near-Forgotten Industry,” in *Reanimating Industrial Spaces: Conducting Memory Work in Post-Industrial Societies*, ed. H.Orange (Walnut Creek, CA: Left Coast Press, 2015), 158–175.

(12.) Jane Humphris and Chris Carey, “New Methods for Investigating Slag Heaps: Integrating Geoprospection, Excavation and Quantitative Methods at Meroe, Sudan,” *Journal of Archaeological Science* 70 (2016): 132–144.

(13.) Philip de Barros, “A Quantified, Chronologically Controlled, Regional Approach to a Traditional Iron Production Centre in West Africa,” *Africa* 56 (1986): 148–174; Humphris and Carey, “New Methods”; Randi Haaland, “Man’s Role in the Changing Habitat of Mema during the Old Kingdom of Ghana,” *Norwegian Archaeological Review* 13 (1980): 31–46; and Caroline Robion-Brunner, *Forgerons et Sidérurgie en Pays Dogon, Vers une Histoire de la Production du Fer sur le Plateau de Bandiagara (Mali) Durant les Empires Précoloniaux* (Frankfurt: Africa Magna Verlag, 2010).

(14.) Shadreck Chirikure and Thilo Rehren, “Iron Smelting in Pre-Colonial Zimbabwe: Evidence for Diachronic Change from Swart Village and Baranda, Northern Zimbabwe,” *Journal of African Archaeology* 4 (2006): 37–54; and Louise Iles and Paul Lane, “Iron Production in Second Millennium AD Pastoralist Contexts on the Laikipia Plateau, Kenya,” *Azania* 50 (2015): 372–401.

(15.) See ORE article by Shadreck Chirikure, “Documenting Precolonial Trade in Africa.”

(16.) For example, see Louise Iles, “The Use of Plants in Iron Production: Insights from Smelting Remains from Buganda,” in *The Archaeology of African Plant Use*, eds. C. Stevens, S. Nixon, M. Murray, and D. Fuller (San Francisco: Left Coast Press, 2014), 267–274.

(17.) Hans-Gurt Bachmann, *The Identification of Slags from Archaeological Sites* (London: Institute of Archaeology, 1982); and Vincent Serneels, “Quantification of Smithing Activities Based on the Investigation of Slag and Other Material Remains,” in *Archaeometallurgy in Europe* (Milan: Associazione Italiana di Metallurgia, 2003), 469–478.

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(20.) See A. Mark Pollard et al., *Analytical Chemistry in Archaeology* (Cambridge, U.K.: Cambridge University Press, 2007); and A. Mark Pollard and Carl Heron, *Archaeological*

Chemistry (Cambridge, U.K.: Royal Society of Chemistry, 2008), for an in-depth discussion of these methods.

(21.) In the case of ceramic material, it is important to note that this method provides a bulk chemistry of the clay including any inclusions or tempering material. This bulk chemistry is important for understanding the ceramic contribution to the slag melt, but in order to fully characterize the fabric of the ceramic, any bulk chemical analysis should be carried out in conjunction with petrographic and/or microprobe/SEM-EDS analysis.

(22.) Portable XRF—though nondestructive—has limited use in iron metallurgy as it is poorly suited to the analysis of heterogeneous slags and ores, in addition to being insensitive to iron’s primary alloys. For further discussion, see Ellery Frahm and Roger Doonan, “The Technological versus Methodological Revolution of Portable XRF in Archaeology,” *Journal of Archaeological Science* 40 (2013): 1425–1434.

(23.) David Scott, *Metallography and Microstructure of Ancient and Historic Metals* (Los Angeles: J. Paul Getty Trust, 1991).

(24.) Marcos Martinón-Torres and Thilo Rehren, “Technical Ceramics,” in *Archaeometallurgy in Global Perspective: Methods and Syntheses*, eds. Ben Roberts and Chris Thornton (New York: Springer, 2014), 107–132.

(25.) See, for example, Michael Charlton et al., “Measuring Variation in Iron Smelting Slags: An Empirical Evaluation of Group-Identification Procedures,” in *The World of Iron*, eds. Jane Humphris and Thilo Rehren (London: Archetype, 2013), 421–430.

(26.) Andreas Hauptmann, “The Investigation of Archaeological Slag,” in *Archaeometallurgy in Global Perspective: Methods and Syntheses*, eds. Ben Roberts and Chris Thornton (New York: Springer, 2014), 91–106.

(27.) See, for example, S. Terry Childs, “Social Identity and Craft Specialization among Toro Iron Workers in Western Uganda,” *Archaeological Papers of the American Anthropological Association* 8 (1998): 109–121.

(28.) David Dungworth, “Experimental Archaeometallurgy: Hypothesis Testing, Happy Accidents and Theatrical Performances,” in *Accidental and Experimental Archaeometallurgy*, eds. David Dungworth and Roger Doonan (London: Historical Metallurgical Society, 2013), 11–16.

(29.) Dungworth, “Experimental Archaeometallurgy,” 15, describes an early bronze casting experience in which he repeatedly checked whether the bronze was molten prior to casting. It was only later that he realized that his inexperience and the frequent checking behavior that it resulted in had led to cassiterite (tin oxide) forming in the microstructure of the final objects. Serendipitously, this led to a re-evaluation of explanations of cassiterite in archaeological bronze artefacts.

(30.) Peter Schmidt, *Iron Technology in East Africa: Symbolism, Science, and Archaeology* (Bloomington: Indiana University Press, 1997).

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