Reconstitution of the operating chain in Paleo-iron and steel metallurgy from the archaeological remains: comparative studies with the African ethno-archaeology

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Reference

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Reconstitution of the operating chain in Paleo-iron and steel metallurgy from the archaeological remains: comparative studies with the African ethno-archaeology

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Abstract. The reconstitution of the operating chain in paleo-iron and steel metallurgy from the ore up to the piece based on archaeological European remains is particularly complicated. The archaeometric methodological means are numerous (metallography, mineralogy, petrography, chemical analysis and so on). However, it is from the data collected with these means that we can define some values allowing us to approach the distinction between iron and steel metallurgy processes. This article intends to illustrate this aspect using studies made on African ethnoarchaeological remains (Burkina Faso and Mali). Indeed, such comparative studies show at least two points of first importance:
- age-long existence of the African iron and steel metallurgy as authenticated by many archaeological remains;
- everlastingness of the know-how associated with direct reduction. Our research works show that these remains are used as a capital value in archaeology and archaeometry. On the one hand they allow us to refine the significance of some discriminative values in terms of functional specialization (reduction, refining forging, working out forging) and on the other hand to define the technico-social organization inherent to the breaking up of the various iron and steel metallurgy activities in the operating chain.

Introduction

The reconstitution of the operating chain in paleo-iron and steel metallurgy (from the ore up to the object) from the archaeological remains is particularly complicated (intrinsic heterogeneity to the physico-chemical aspect of reduction in a solid phase, multiplicity and simultaneity of some operations, representative quality of the archaeological sampling...).

Thanks to a well established experience of many years of multibranche’s research work in the UMR 5060 CNRS (archaeologists, historians, geologists, metallurgists, physical chemists and so on) we have improved our problematic and our methodology linking archaeology to archeometry in order to better emphasize the archaeological remains in terms of discriminative process.

Without schedule our research results (Mangin et al., 2000) we try through this article to compare our knowledge with the African ethnoarchaeology. Indeed if we have on the one hand a better understanding of the ore extraction and ore direct reducing process (analytical correlating, chemical heritage, metallographical correlations, applied archaeology) and on the other hand a better knowledge of the working out of the piece (thermomechanical setting), we are still missing some information on the post-reduction process (refining smithing).
It seems particularly important to consider that the activity in the operating chain is divided according to work specializations (fig. 1) (reduction, refining smithing of half product: spongy iron, working out of the piece) interacting with a technico social organization more accurate in the roman age (characteristic of the archaeological remains, particular skills, geographical situation of the activity areas, exchanges and bonds between different workshops, macro and micro production).

Quite a lot of African archaeological sites are interesting for the following points:

- Existence of an African autochthonous home of the old days of the iron metallurgy - some dating (Termit, Niger) (Paris et al., 1992) showing the existence of an iron metallurgy in this country in the second millennium BC. Those elements allow us to confirm the “diffusionist” thesis of the iron metallurgy (south Caucasus 1700-1500 BC).

- The everlastingness of the tradition and know-how of the full operating chain in direct

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Fig. 1. Operating chain of direct iron and steel metallurgy.
siderurgy at some rare sites (Toungaré: Burkina Faso, Aridjou: Mali...).

This allows us to observe in situ and in vivo the reality of the work organization and of the specific functions that we actually try to analyze and to restore in Europe from the incomplete archaeological remains we have at our disposal.

<table>
<thead>
<tr>
<th>Operating</th>
<th>Extraction</th>
<th>Preparation</th>
<th>Smelting</th>
<th>Refining</th>
<th>Smithing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Process</td>
<td>boring, extracting, timbering, ventilation, pumping</td>
<td>washing, breaking, roasting, calcining</td>
<td>Low Furnace</td>
<td>refining, smithing</td>
<td>working out smithing</td>
</tr>
<tr>
<td>Products</td>
<td>pig ore</td>
<td>ore extration and sizing, charcoal, fluidizer, wood</td>
<td>iron sponge, bloom</td>
<td>semi product, ingot</td>
<td>object</td>
</tr>
<tr>
<td>Residuals</td>
<td>barren, gangue, debris</td>
<td>reducing slags, vitrified clay ore</td>
<td>smithing slags, reducing slags</td>
<td>scale, &quot;cake&quot;</td>
<td>iron pieces</td>
</tr>
</tbody>
</table>

**ARCHEOLOGICAL INDEX**
- Furnaces typology: wall thickness, vitrified lining, tuyere...
- Spatial and temporal organization of the operations.
- Slags deposits...

**ARCHEOMETRIC INDEX**
A. Typology of residues
- Surface morphology
  - Inside (internal) morphology
    - inclusion, defect, bubble, porosity, cavities, crack, stratification, texture...

B. Metrology
- Relative proportion (quantity)
- Dimension
- Weight
- Density

C. Metallography
  Cf. detail

D. Mineralogy - Petrography

E. Chemical analysis
- Analytical correlations, index of basicity
- Chemical heritage, tracer, oxidation degree...

F. Charcoal analysis
- Wood species
- Dating

**MACROGRAPHIC EXAMINATION**
- Image analysis
  - distribution of porosities
  - distribution and nature of inclusions
  - distribution of phases
  - quantification
- Stratification
- Impurities
- Precipitation
- Kneading
- Welding
- Cracks
- Texture

**MICROGRAPHIC EXAMINATION**
- Structural typology (ferrite, pearlite, bainite, martensite...)
- Phases analysis
- Structural morphology (heterogeneity, anisotropy)
  - solidification structure (directional, dendrite, equiaxed, globular, filaments, rosettes, monocristalline, polycrystalline, columnar, grain size, overheated structure, recrystallization...)
  - deformation texture (thermomechanical treatment, preferred orientation, deformation band, twinning, cold working, kneading...)
  - thermal treatment
  - thermomechanical treatment
  - precipitation, segregation, point defect, dislocation
- Structural context
  - carbonizing, decarburization,
  - oxidation, reduction (oxidation degree)
  - cohesion, bond,
  - diffusion
- Inclusions analysis
  - identification
  - formation and morphology (extraneous, endogeneous)
  - distribution, quantification, size,
  - mechanical properties (malleability)
- Hardness test - microhardness

**Fig. 2. Main discriminatory index of the iron production operating chain.**

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Likewise some African sites also present some structures of well preserved "low furnace" (Martinelli, 1992). If there is no doubt as to the major interest gained in the study of such sites for our work research in the context of our systemic approach of the siderurgy, we must take care not to make a hasty diachronical transposition.

**Discriminatory metallographical values**

First of all we must remind you that the results stemming from the archeometry investigations have a limited validity because of intrinsic and extrinsic factors connected to the studied materials whatever the methodological means employed (Mangin et al., 2000).

The choice and therefore the representative value of the selection of samples represent a preponderant factor. Furthermore one must not lose sight of the scale ratio (mini- maxi-production, relative proportion, macro and microscopic tests...). Besides the experimental archaeology, a non exhaustive summary of the main discriminative index is represented on figure 2.

In any case, only by confronting globally the values stemming from the different methodological means used could we reduce our doubts and complete the assessment of coherent measures (criterion) enabling evidence as regards the hypothesis relative to the preponderance of a specific iron and steel making activity (process).

Let’s now detail some metallographical values illustrated by some African experiences:
- Toungaré and Gooden in Burkina Faso.
- Aridingi in Mali.

**Ethnoarchaeological comparaisons**

**(Case study)**

**Burkina-Faso**

**Toungare site** (Sourou country fig. 3)

It is a contemporary site corresponding to a blacksmith village, Samo, which masters the full operating chain.
collected there (in September 1994) reducing processes are taking place only when need arises (a process lasts for 24 hours) in order to accumulate enough blooms for the village needs.

The blooms (fig. 5) are stored and used according to the needs. The last reduction process dates back from 1989.

The refining forge has a forced ventilation (draught) (fig. 6) and is settled in the periphery of the village which has otherwise a small workshop forge.

The bloom is broken up, then refined and the pieces are stored in baskets before being used to make objects. Besides the study of slags we show the metallographic study of a piece of refining bloom.

The image analysis of macrographical pictures on a representative part shows a porosity ratio above 30%. A few non-metallic inclusions are identified at this scale. The micrographical analysis shows a quite homogeneous structure of quite thin lamellar perlite corresponding to a steel showing close properties with eutectoid (that is to say 0.8 per cent of carbon) (fig. 7).

We can observe a slight superficial decarburizing combined with the presence of acicular Ferrite linked to a local overheating. The medium-hardness is 250 HV (vickers of 500 g).

In that case it is important to consider, on the one hand, the spatial and temporal organization of the operations (reducing - refining - working out) and, on the other hand, the carbon content of the bloom, quite high and homogeneous, which allows for a wide field of applications in the forge (thermics treatments of possible quenching, long decarburizing). This observation is probably due to a good control of the process of reducing combined with a natural ventilation (draught) allowing a constant reducing atmosphere suitable for the circulation of carbon. In that context the production of hypereutectoid steels was possible.
The reducing site revealed the presence of furnace foundations (Ø 1 m), of tuyere, ash and of quite a lot of big blocks of slag. Otherwise, searching close to the site allowed us to find some slag, some charcoal and some vitrified structures (small ones) linked to the base of a small furnace containing some pieces of slag and some charcoal (Ø 33 cm) called by archaeologist "under furnace".

The borings in these dwelling sites have revealed an important metallic furniture and some slag. The studied slag come from the reducing site (Libouré district, slags I5 and surface) and from the dwelling sites (Taagoogo district, slags III 132.III147.III16).

- Slags from the reducing site
  They have been taken near a furnace designed for reducing.

The metallographic analysis reveals that the structure of the matrix is quite homogeneous with a lot of wustite dendrites (Feo). The remaining metallic elements are very uncommon and are generally localized near the porous parts (diffusion shaft of reducing gas Co/Co2).

The link with the matrix is close and there is no peripheral "re-oxidation". Otherwise, we can distinctly observe a process of agglomeration of the metallic globules to make a grain (speck) of a biggest size (germination - development) (fig. 8).

The chemical etching shows a structure containing mainly ferrite.

In that case we are faced with a slag which shows all the typical metallographic characteristics (typical features) of the reducing process. The outlines of the porous parts although ovoid are quite irregular, the poor ratio of steel in the slag could indicate a particularly efficient reducing process.

- Slags in the dwelling site
  Slags (III 132 III 147)

The metallic elements are rare and scattered in a "silicated" and quite homogeneous matrix. This one shows quite a lot of porous parts with the outlines shred (light angle) and has big cracks. Some rare dendrites of wustite have been found. The steel has some inclusions of a bony morphology which has no systematic strong bond with the matrix. Reoxidation that can be observed in

Gooden area (Bulkiemé country fig. 3)

This area represents a basic interest for the research on "old" metallurgy in the country. Indeed, there is no other area, as far as we know now, which has such remains of the ore-working linked to the living conditions (Kiethega, 1986).

It is an well-known fact that the iron and steel metallurgy has been practised before the first half of the sixteenth century, period when the Nakomsé have settled there.

We assume the metallurgy remains they found in that area at the time dated back to the Ninsi period. This people, settled on the central plateau Mossi, is characterized by an oral culture like a warrior nation, who specialized in the iron metallurgy (Izard, 1970).

Our archaeological research concerns a reducing site and two dwelling sites 2 km away from each other.
periphery has remained partial. The outstanding thing is the presence of what we call fragments in comparison to the archaeological blooms, which are situated in the inclusions as in some fissures (fig. 9).

It can be a preponderant element characterizing the process of a refining forge.

- Slags III

The less compact slag compared to the first ones show a lot of metallic elements scattered on the whole surface of the samples. These are particularly situated inside the matrix without having any close bond with the porous parts and are automatically in a state of more or less complete reoxidation (fig. 10). A wustite ring encircles the whole of the metallic island and some corrosion pits can be observed.

The contact between the matrix and the metallic element is very strong. These observations constitute some characteristic values of an oxidizing process close to the physico-chemical conditions of a working out forge.

Mali

During the year 1995, É. Huyscom has organized a traditional smelting operation at the site of Aridingi in the Dogon region of Mali. The whole process has been recorded and sample provided for further examination (Serneels, 1997; Huyscom et al., 1996).

**The smelting process**

The furnace has been rebuilt on the remains of a similar ruined structure. Walls are made with slags and clay with a carefully smoothen internal lining. The height of the shaft is about 200 cm and it is standing on a small pit of 50 cm deep. The internal diameter is 30 cm at the top and 120 cm at the tuyeres level. 13 long tuyeres, made of unfired clay tempered with straw, are placed in six large openings at the ground level. They are tilted down and penetrating to the centre of the furnace. Air draught is naturally induced.

The furnace was put lit with straw. Then the tuyeres were introduced, the openings were closed and the charcoal was loaded. The smelt went on during 40 hours. Loads of 30 to 40 kg of ore of charcoal were added every 3 or 4 hours. The temperature measured at the nose of one tuyere, remained stable and low, about 800 °C, when the furnace was loaded. Several hours after the last load had been put in, the temperature rose for 2 hours and reached 1 050 °C. A second increase of the temperature took place when the inner level reached the enlargement of the shaft of the furnace. When the temperature increased for the third time, the furnace had to be opened immediately. Tuyeres and earth were removed from the openings and the bloom was taken out.

**Yield and production**

The furnace was loaded with about 250 kg of charcoal of which 45 kg were left unburnt at the
end of the smelt. About 210 kg local iron ore recently extracted was added. It was crushed to centimetre long pieces but not roasted. The analyzed sample still contains 8% of water but it is probable that the loaded ore was more humid. A load of 25 kg of old iron ore, recycled from a previous smelt, was added.

After the smelt, the internal lining was not damaged and no slag was adhering to the walls. Tuyeres and materials which were closing the openings were damaged during opening and it is impossible to estimate the fusion during the smelt.

A thick layer of fayalitic slag was formed at the bottom of the furnace and there were some other smaller pieces of slag in the remaining charcoal. Altogether, the weight of free slag amounted to about 47 kg. The bloom lay at the tuyere level in the centre of the furnace, on top of the main slag block. It was broken in 4 main pieces and several smaller ones during the extraction. The central part of the bloom, weighing 8.6 kg was made up of pure metal. The other fragments, 60.65 kg altogether, were made of metal embedded in slag. An estimated content of 50% seems reasonable from the microscopic examination of the sample.

From these field and laboratory data, a ratio between ore, slag and iron of 2:12:1:0.5 could be given. The evidence is that there is an important loss of weight during the smelt, which can only be explained assuming that the iron ore were very wet.

The iron bloom

The metallic product formed during this smelt is very heterogeneous. It is an irregular lump of about 50 cm in diameter. It has been broken in several pieces during the removal. Its total weight, including small lumps that were perhaps dispersed in the furnace, is 69.25 kg. The central part is a compact block of 8.6 kg containing mainly consolidated metal. The remainder, forming the bulk of the product, is a mixed material containing slag, metal and charcoal, having a spongy texture with many large and irregular voids.

- The central part of the bloom
  The outer surface is very irregular with entering holes and indented edges. Some charcoal is trapped. The external surface is coated with a very thin layer of slag. Many fragments of a thin rim of ferrite are also visible in places along the surface and inside the largest voids.

  The carbon contents of the metal can vary from nearly 0.02% to 1.2% or more. Most of the pieces, about 70%, are constituted of eutectoid steel with a pearlitic structure with 0.8% C. On one side of the bloom some areas with a lower contents of carbon are visible. The amount of ferrite varies. Transition between eutectoid steel to ferritic iron, even gradual, could occur very rapidly (less than 0.5 cm).

  A very small amount of slag is trapped inside the metal in smooth rounded inclusions. The total quantity of slag is far less than one percent. Big pieces of charcoal are present outside of metallic area but not really inside it. Voids cover about 20% of the surface. The larger voids could be connected with the exterior and show similar features to the outer surface (ferritic rim, slag).

  Small holes are widespread and appear to be a very typical feature of the bloom. They are irregular in shape and size and show straight, jagged or convex boundaries. Frequently, folded lamellae of metal are trapped inside. Voids seem to tend to close (due to the compaction of the metal?) as shown by irregular scars of more or less soldered voids.

- The peripheral part of the bloom
  The studied sample of the peripheral bloom has a spongy texture made of about 30 small domains (0.5 to 5 cm²), corresponding to the former fragments of ore. Each of them has its own internal structure and different situations can be observed.

  One domain only is chiefly made of metal with a texture similar to the central part of the bloom. The carbon content is gradually increasing from 0.02 to 1.0% from one side to the other. A few circular slag inclusions and many empty and irregular voids are also visible. A thin external layer of slag is more or less continuous.

  Most other domains are made of slag containing a variable amount of ferritic iron, always with
a very low carbon content. Mineralogical components of the slag are glass, fayalite and iron oxides (wüstite and magnetite). In many places iron oxides develop on the surface of iron particles.

Iron takes the form of round particles or vermicular aggregates filling from a few to 90% percent of the volume. On the edges of the domains, metal is always denser than internally, showing a more complete reduction of the outer surface.

One area has only a very thin outer layer of iron and inside it, the entire volume is occupied by a slag which is very rich in iron oxides. It is considered to be an unreduced lump of ore.

The peripheral bloom shows a variety of structures, reflecting an incomplete process at various stages.

**General comment**

The smelting of iron ore by the direct method was carried out by the Dogon smiths in a large furnace using natural induced draught. The process took a long time and temperature seemed very low.

The yield of the smelt is between 45 and 55%. The product is very heterogeneous. A piece of compact steel, free of slag weighing about 8.6 kg, has been produced together with 60.65 kg of a mixed material containing about 50% of slag and mainly ferritic iron spread as small particles or vermicular aggregates.

Both kinds of raw product are worked in the smithing hearth to gain as much metal as possible. The central compact part is used for special purposes, especially for the making of massive tools like anvils and hammers. During the smithing of several parts of the bloom into various objects, the measured loss of weight was of 65%.

**Conclusions**

These examples stemming from the study of different African sites (Burkina Faso and Mali) show the ethnoarchaeology contribution to our problematic, that is to say to the characterization of the operating chain in direct iron and steel metallurgy using archaeological remains. Indeed in addition to the experimental archaeology, we have some actual and historical references allowing us to authenticate the relevance of the disminiative values established from the study of European archaeological sites both on the process and on the shared work.

If the metallographical values seem to be suitable in that respect (reduction, refining forge, fining forge...), we must however compare them to the data coming from other methodological means indicated in the first figure. We must say on that subject (I) that a metallographical value of oxidation and/or of reducing can be found as well on a reduction or forge area (particularly in the case of the refining forge).

However the study of a statistically representative sampling allows us to moderate, even edge out, the chances of interpretation. Moreover if the refining forge and the working out forge are undoubtedly located on distinguished sites they can be totally made simultaneously on other sites and moreover they can have an oxidising or reducing (carburizing) reaction. This last process requires specialized skills to control the different parameters involved (atmosphere, temperature, timing...) and shows how difficult it is.

Moreover we must be wary of any simple and pure transposition. Indeed if the physicochemical aspect of the process (oxidoreduction) can be considered as an incontestable process, the wide know-how and the different country behaviours (the typology of the furnaces (4), the kind of ores...), and particularly in Europe, show that we must moderate our conclusions. In any case we must take these results into account in our research as well as at the others all the more so because they have the advantage of being verifiable (Échard, 1968-1994).
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