An archaeological and archaeometrical approach of ferrous semi-product : a diachronic qualitative typology (VIIth c. BC. - IInd c. A.D.)

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An archaeological and archaeometrical approach of ferrous semi-product: typology, quality and circulation

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Introduction

From the raw bloom to the final product, depending on the nature of the latter, several semi-products can be obtained along the *chaîne opératoire*. Some of them are directly transformed on the ironmaking site. Some other, on the contrary are marketed between several workshops, production centres or markets. On relation with the quantity of work to obtain them, or with the quality of the constituting material, they could have different values. For these reasons, understanding the way these semi-products were obtained and in which technico-economical context, but also the way they were comprehended by the ancient craftsmen and blacksmith is a fundamental aspect of ironmaking technical and economical history, whatever the period under interest is. The aim of the general overview presented here is to review, from the archaeological site to the laboratory, the different clues that allow to enlighten several aspects linked to the semi-product production and exchanges. Indeed, even if the archaeometric observations are scientific facts, they can never be considerate out of any archaeological and historical context. Thus, a constant come and go must be undertaken between the physico-chemical data and this context. For example, as far as the metal quality is concerned, added to the fact that numerous parameters will influence the mechanical behaviour of the material, it is important to know that it is not the only aspect that will determine the use of a given material. It is merely a more complex conjunction of its quality, the skill of the blacksmith, the availability of the metals in the technico-economical context under study, etc.

Depending on the place in the *chaîne opératoire*, semi products can be more or less elaborated and of different natures: bloom slightly hammered after a first compaction treatment, ingot obtained after a more important hammering and cleansing, currency and socket bars, with a thinner shape closer to the form of the final artefact, etc.[1].

Semi-products can question several aspects of archaeological and historical studies. At the archaeological site level, it is of great interest to understand the spatial and technical organisation of the site, and sometimes of different production sites in the same area and period. It is also important to evaluate the nature of the forging sequences, the type of activities and production and the quantities. Another crucial question is the nature of the metal that is produced and marketed, linked to his mechanical behaviour, and the value it has in the different technico-economical contexts. To study this point it is necessary to try to evaluate it by identifying the parameters that mainly influence this quality and trying to define reproducible study methodologies in order to compare the different artefacts. Then, another important point is to study the semi-product diffusion and exchange on more or less long distances. To this purpose, crucial chemical data can be found in the slag inclusions (SI) embedded in the metallic artefact, that offers the possibility to identify homogeneous or heterogeneous corpuses but also, in specific cases, to enlighten the production region of the artefacts.

Our research for 10 years concerns an important number of sites and artefacts covering periods from the 1st iron age to medieval times. Following the methodology of former works made in east of France[2] and in the Swiss Jura[3], in the frame of several PhD works[4], we significantly completed our

1) BERRANGER-FLUZIN 2011.

2) LEROY 1997, LEROY et Al. 1999, FLUZIN et Al. 2000.

3) SERNEELS 1993; ESCHENLOHR 2001; ESCHENLOHR et Al. 2007; SENN-BISCHOFBERGER 2005.

4) ORENGO 2003; BAUVAIS, 2007; PAGES 2008; PAGES 2010; DESAULTY 2008; BERRANGER 2009; LEROY 2010.

pluridisciplinary and diachronic investigations on the French territory for the following areas: Grande Limagne d'Auvergne, Nord du Bassin parisien, Narbonnaise (Languedoc-Roussillon, Provence-Alpes-Côte-d'Azur), Normandy, Pays de Bray, Pyrénées ariégeoises and an important part of central Burgundy. More than 600 ironmaking, sites, tens of historical buildings and several hundred artefacts were studied.

These works allowed us to precisely define the internal characteristics of the materials in order to evaluate on the one hand the real nature of the forging activities on given sites, on the other hand the qualitative criteria that could help to identify some specificities and follow exchanges and technical breaks.

Reading wastes on the archaeological sites

Working sequences in bloomery process, as far as post reduction is concerned, are more or less numerous and complex. The post reduction work can begin in the continuity of the smelting stage (compacting of the raw bloom) and strive towards final forming of the artefacts, including the semi-product stage. The *chaîne opératoire* can thus be divided in space and time[5] and present different stages corresponding to several qualitative nuances of the material.

Depending on periods and geographic areas, the technical system reflects a global organisation more or less sophisticated and structured, varying with the context (rural, urban, etc) and different interactive variables. These variables can be environmental (raw material resources), social (hierarchy, economy, circulation, exchanges, intensity of production …) and are linked with the nature of the production (skills, specialisation, …).

Even if, the physico-chemical and thermomechanical characteristics of the material transformation are, up to now, better understood by rigorously crossing analytical methods and observation scales[6], the restitution of the practice reality from archaeology, ethnoarchaeology and experimentations remains difficult and needs numerous methodological precautions.

Thus, to try to decipher and understand the activity of a given archaeological site, but also the characteristics of the ferrous materials that circulate, it is necessary in a first step, to proceed to an exhaustive study of the vestiges. These vestiges include blooms, metal pieces, fragments of crude metal, slag type (taped, cake, shapeless, …), slag proportion, lining, semi-products, worked metal fragment, forging scales, rejected fragments, artefacts, tools, etc. These studies must be performed on a significant number of archaeological sites trying to favour the most important corpuses (here archaeological studies of 600 sites, 105 of them archeaometrically studied i.e. 700 samples[7]).

Moreover, considering the heterogeneity caused by the smelting and forging at solid sate, if the morphological criteria can let to a first typological classification, this aspect is not intrinsically a sufficiently discriminating factor (particularly for quality distinction). Our works shows that there is not a systematic link between morphology and internal characteristics of wastes. This observation is true for both forging slag cakes and manufactured products.

A similarity of shape and aspect is not necessary linked to a same texture, structure or chemical composition. Thus, exclusive morphological approaches must be considered very carefully all the more since a same artefact can present a strong variability inside his matter (compositional or inclusionary cleanliness gradients for metal, more or less stratifications for slag cakes, …). For this reason, it is necessary to proceed to observation and study of the complete transverse section of artefacts whatever their nature or sizes are.

Toward an evaluation of the metal quality: different ferrous metals

The detailed protocol of metallic semi product analyse will not be presented here and can be found in literature[8]. It consists in classical metallographic preparation with adapted etching: nital

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5) PETREQUIN et Al. 2000; FLUZIN 1999 and 2004; FLUZIN et Al. 2004; MANGIN-FLUZIN 2008; BAUVAIS-FLUZIN 2009.

6) MANGIN et Al. 2000; FLUZIN et Al. 2000.

7) FLUZIN et Al. 2011.

8) FLUZIN 1983; FLUZIN et Al. 2000; FLUZIN et Al. 2004.

Type	% of the surface > 0.2w% C	Weighted carbon content
Ferritic	0 - 20	< 0.3
Heterogeneous	20 - 40	0.3 – 0.6
Composite steel	40 – 70	0.6 – 0.8
Steel	70 - 100	> 0.6

Fig. 1: Categories of ferrous alloys with carbon (PAGES 2010, p. 197).

and Oberhoffer reagent. A very important point is to take into account different observation scales from the macroscopic to the microscopic one. If possible, the best way is to make a cross section on the entire artefact to evaluate the different heterogeneities. If that is not possible, representative sampling should be performed at different places of the artefact.

Several parameters will influence the mechanical behaviour of ancient ferrous metals. The first one is of course the carbon content that will change hardness, elastic limit, etc. Nevertheless, as far as ancient artefacts are concerned it is necessary to take into account the eventual presence of phosphorus in the metal but also the one of slag inclusions and porosities. Lastly, it is also important to evaluate if the artefact is made by welding of different metal pieces and to evaluate the quality of the welding lines.

Contrary to contemporary steels, carbon is rarely homogeneously distributed in the metal. Indeed, in most of cases, depending on the local thermodynamic conditions in the furnace, the metal is more or less carburised. Even in the case of relatively carburised materials, the carbon content is heterogeneous.

For this reason, it is difficult to assess correctly the carbon content of a semi-product. To this purpose, a new metallographic approach was proposed to evaluate the iron qualities true two parameters[9]:

- a macroscopic observation under binocular after nital etching on the metallographic cross section for evaluate the proportion of the surface that present carbon content higher that 0.2w%.

- a microscopic observation under microscope which allows to define the surface proportion of different carbon classes was determined as follows:<0.02, 0.02-0.2, 0.2-0.4, 0.4-0.6, 0.6-0.8, 0.8-1, etc. A simple average measurement of the carbon content does not express the distribution of this element accurately. Therefore, we decided to define a weighted carbon content as follows:

$$\%C^* = \sum_{i=1}^{n} p_i \times x_i$$

where %C* is the weighted carbon content, xi is the mean C content for a given class and pi is the surface proportion corresponding to the given class (sum of pi ¼ 1).

Only these two parameters are able (fig. 1) to completely describe the carburisation degree of an artefact. Then considering it, artefacts can be classified into different categories: ferritic iron, heterogeneous metal made of a mix of iron and steel, composite steel, steel indicates the definition of these categories[10].

As said before, phosphorus is an element that must be taken into account. Several studies clearly demonstrate that the presence in the metal of relatively low quantities of phosphorus (more than 0.1%) can drastically change the mechanical behaviour of it and consequently his commercial or technical value. Moreover it seems that specific technical gestures must be used to manage to forge and weld it correctly[11]. Thus, it appears that phosphorus iron must be considered as a specific material. Moreover, it will be important in the future to try to evaluate the real place of this material in the technical landscape of each period. For this reason, any study about semi-finished products must evaluate on the one hand the amount of phosphorus present in the materials and, in the other hand its distribution. Specific ghost structures after nital etching appears

9) PAGES 2010; PAGES et. Al. 2011-a; PAGES et. Al. 2011-b.

10) PAGES 2010; PAGES et. Al. 2011-a; PAGES et. Al. 2011-b.

11) VEGA et Al. 2002: VEGA et Al. 2003.

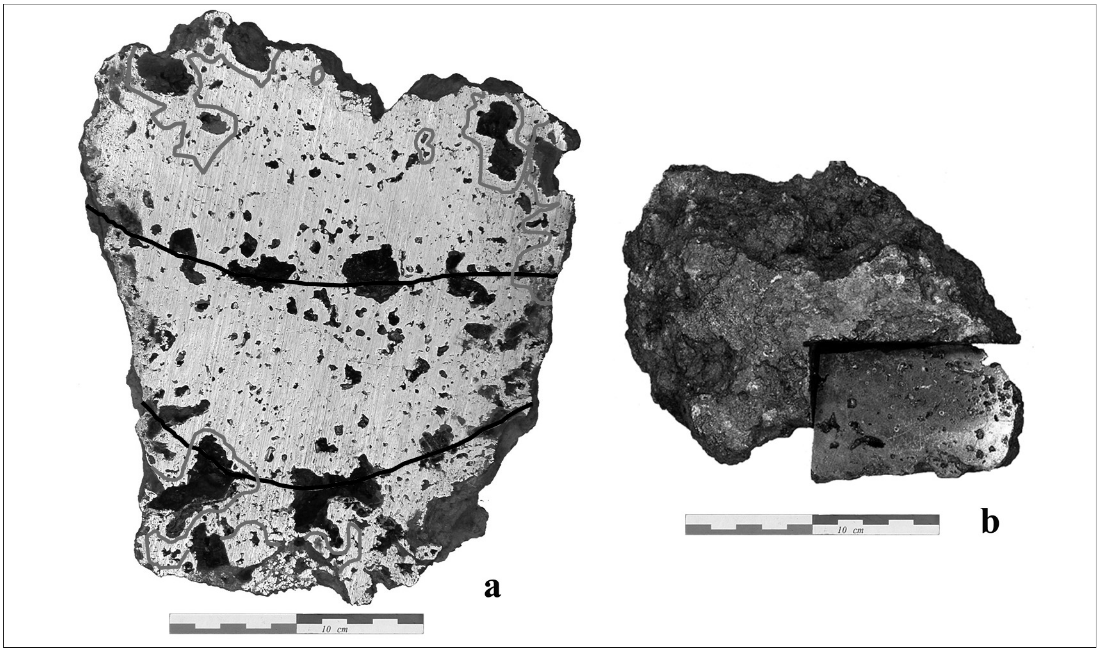

Fig. 2: a) Raw bloom from Piani d'Erna (Italy), 2nd century BC-1st century AD; weight 33 and 38 kg (FLUZIN 2006). b) Bloom from Selongey (Burgundy, France), Gallo-Roman, weight 2.37 kg.

on polished surfaces for P contents between 0.1 and 0.6 mass%. They can also be easily detected after an Oberhoffer etching. For higher contents, an Energy Dispersive Spectrometry coupled to a Scanning Electron Microscope analyse on the metallic matrix can indicate the presence of this element.

Depending on the smithing stage (cleansing), on the slag quantity initially present in the raw bloom, but also on the final shape of the artefact, more or less slag inclusions remain embedded in the semi and final products. We will show that they are a very useful mean to distinguish smelting processes and provenances[12]. However, as far as the mechanical behaviour of the metal is concerned, they can modify drastically the quality of the metal. Moreover, it is completely impossible to estimate ab inito the mechanical behaviour of a given artefact if it contains numerous inclusions. This effect was observed by performing uniaxial tensile tests on samples taken in bars reinforcing the Vincennes castle dungeon in France (XVIth c.)[13]. Different samples taken in bars presenting the same morphology, the same heterogeneous structure made of mixed carbon and steel and containing numerous slag inclusions have a completely different behaviour. This is also the case for samples taken in the same bar at different locations. Thus, to estimate the quality of the semi-finished product it is of great importance to evaluate the surface proportion of the slag inclusions but also porosities. It is then possible to express the cleanliness of the semi product.

Lastly, to evaluate the quality of an artefact or a semi product it is necessary to detect the welding lines and their quality but also the eventual voluntary thermo mechanical treatments (carburizing, quenching, tempering, etc). Practically, different operations must be distinguished: simple welding by hammering of porous or heterogeneous material, welding after folding and hammering, welding together separate metal pieces. Moreover, the most significant parameter to consider is the textural and structural homogeneity.

It appears after this short review that the quality of a ferrous semi-product is a very complex thing to evaluate. First, it is linked to the convolution of numerous parameters from the carbon content to the slag inclusion number and distribution. A first approach, after quantifying all these

12) DILLMANN-L'HERITIER 2007.

13) DILLMANN 2007; DILLMANN et Al. 2003; L'HÉRITIER et Al. 2005.

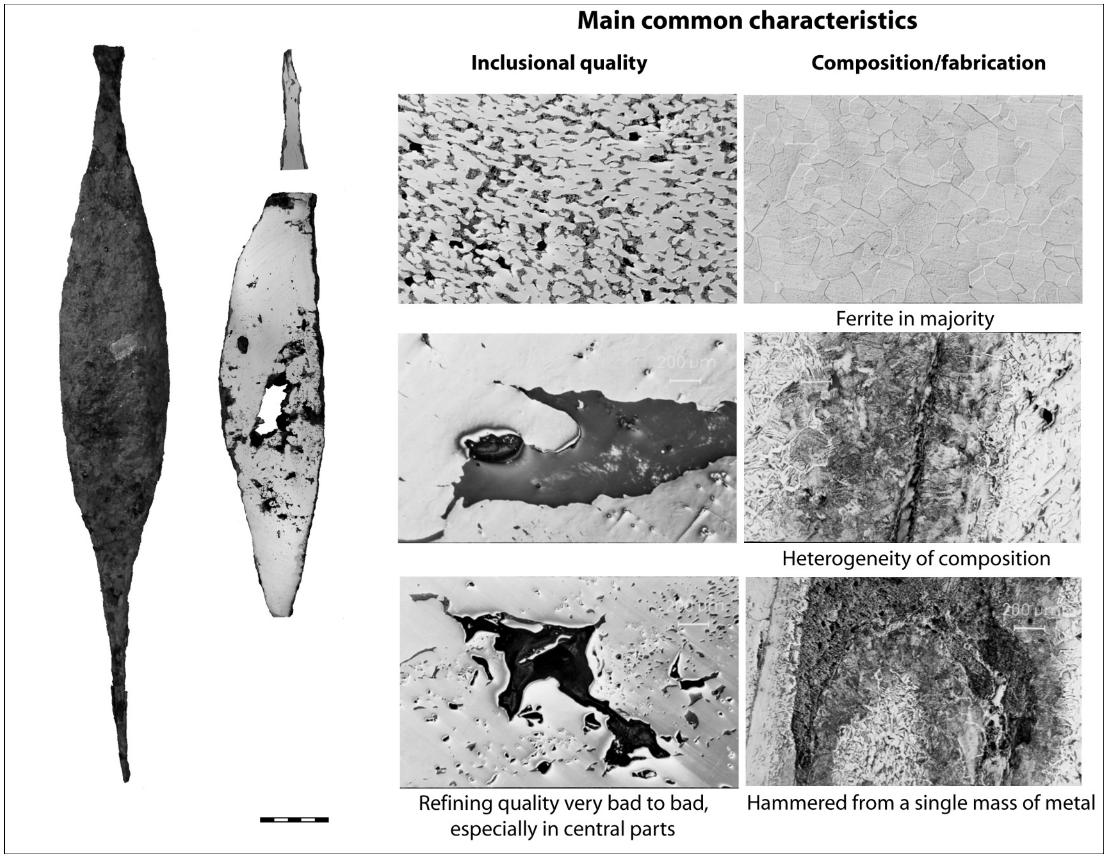

Fig. 3: Iron bipyramidal bar from Nottonville (Eure et Loir, France), 8th-5th century BC (^{14}C datation). Weight 4,9 kg (BERRANGER 2009).

parameters is to underline the ones that will have the main influence on the mechanical behaviour: a carburised steel without slag inclusion is completely different from a ferritic iron full of impurities. Nevertheless it is not so easy to make a difference between the numerous heterogeneous materials that are found in the archaeological sites: what about the comparison between a composite steel containing slag inclusions and a phosphoric iron? These points will be investigated in the future. Secondly, the notion of quality is, of course, relative and depends on the technico-economical context but also on the final use of the metal. At this stage, a necessary confrontation with the archaeological and historical data is necessary.

From raw metal to semi-products: examples of results

Nevertheless, considering all the information gathered in the metal, some interpretations of archaeological sites but also a global view of production and exchanges in given areas can be proposed from the study of semi-products. Following the sequences of the *chaîne opératoire*, several examples are given.

Because of their rarity in archaeological context, studies on raw blooms are not so numerous (fig. 2). The most of our observations (24 blooms morphologically studied including metallographic studies on 17 blooms or fragment of 2 to 38 kg) confirms the intrinsic compositional heterogeneity of blooms (numerous slag inclusions and porosities, high variability in carbon distribution, from 0.02 to 0.9%C and sometimes presenting locally cast iron structures). This latter material can sometimes be considered as waste but this assertion is not systematic. Moreover, some blooms can be very homogeneous (ferritic or low carburised steel) showing an excellent mastering of the

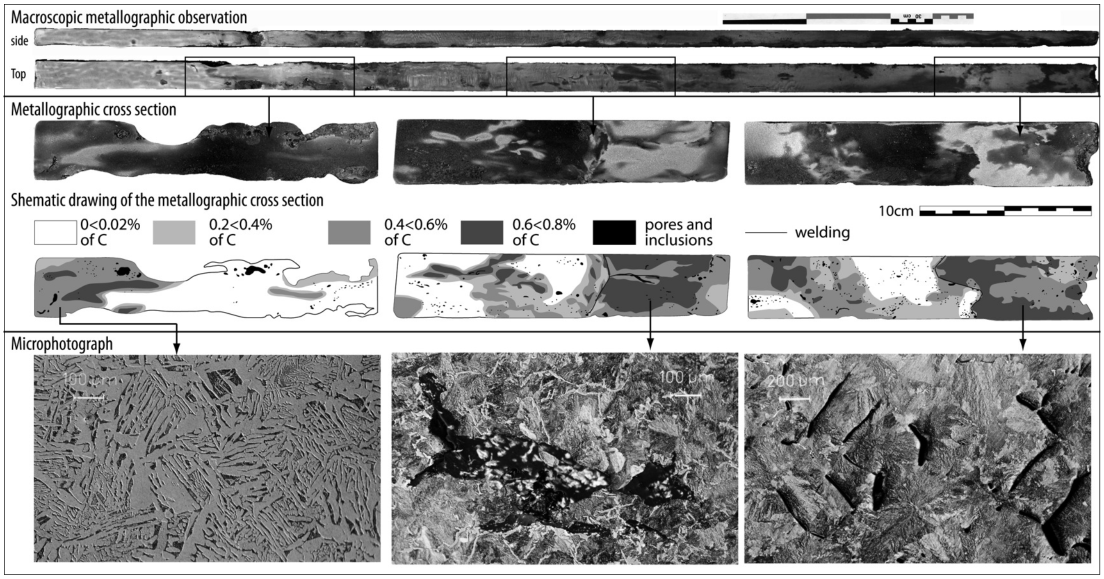

Fig. 4: An example of the metallographic observation of a bar of Saintes-Maries-de-la-Mer (1LSM10_1) at the macroscopic and the microscopic scale after Nital etching (PAGES et Al. 2011).

smelting stage. This kind of artefact is also easily forgeable[14]. Their presence in open urban areas and *oppida* shows that the raw materials (blooms) were exchanged as "raw semi product", probably on small distances and, that these areas unquestionably concentrated supplying and redistributed it[15].

As far as typology and morphology of semi product are concerned[16], it appears that in most of cases that we studied (730 artefact archaeologically examined including about 200 archaeometric studies), bipyramidal or salmon type ingots (more than 80 artefacts analysed, fig. 3) corresponds to a bloom or a bloom fragment very summarily compacted (sometimes folded down). We also confirm the hypothesis that the more or less strong elongation at their extremity was made in order to demonstrate the forgeability of the metal (qualitative test). In a general way, the thinner the semi product, the higher the cleanliness (compacting rate, and forming work).

It is important to mention the exception of the bars from the "Saintes-Maries-de-la-Mer", dated from 1c. AD (Eleven roman ships. Every ship contains between 20 to 150 tons of ferrous bars[17]). Indeed, despite of their significant weights (between 1.5 to 33 kg) and their crossections that can attempt 10x4 cm^2, the complete archaeometric study (54 bars) reveals a high inclusion cleanliness with a certain technological and morphological normalisation (fig. 4). Moreover, the longer bars (1.5 m) are made of 2 to 4 blooms welded together by a high quality work. These welding lines can be considered as true technological feats.

To sum up these results. Metallographic analyses show the semi-product morphology of the bars is correlated to the metal quality. It means that the standard seems to be linked to the metal quality: each specific morphology is linked to a particular metal. This association is not linked to the ship loading. It can be observed in all the studied ships.

At least six different reducing systems categories can be distinguished[18]:

- Category 1: High MnO contents.
- Category 2: High P2O5 content.
- Category 3: Higher MgO/Al2O3 ratios without significant MnO or P2O5 levels.

14) FLUZIN 2006.

15) BAUVAIS 2007; BERRANGER 2009.

16) PLEINER et Al. 2003; PLEINER 2006.

17) PAGES et Al. 2008, 2011; PAGES 2008 and 2010.

18) PAGES et Al. 2008, 2011-a and 2011-b.

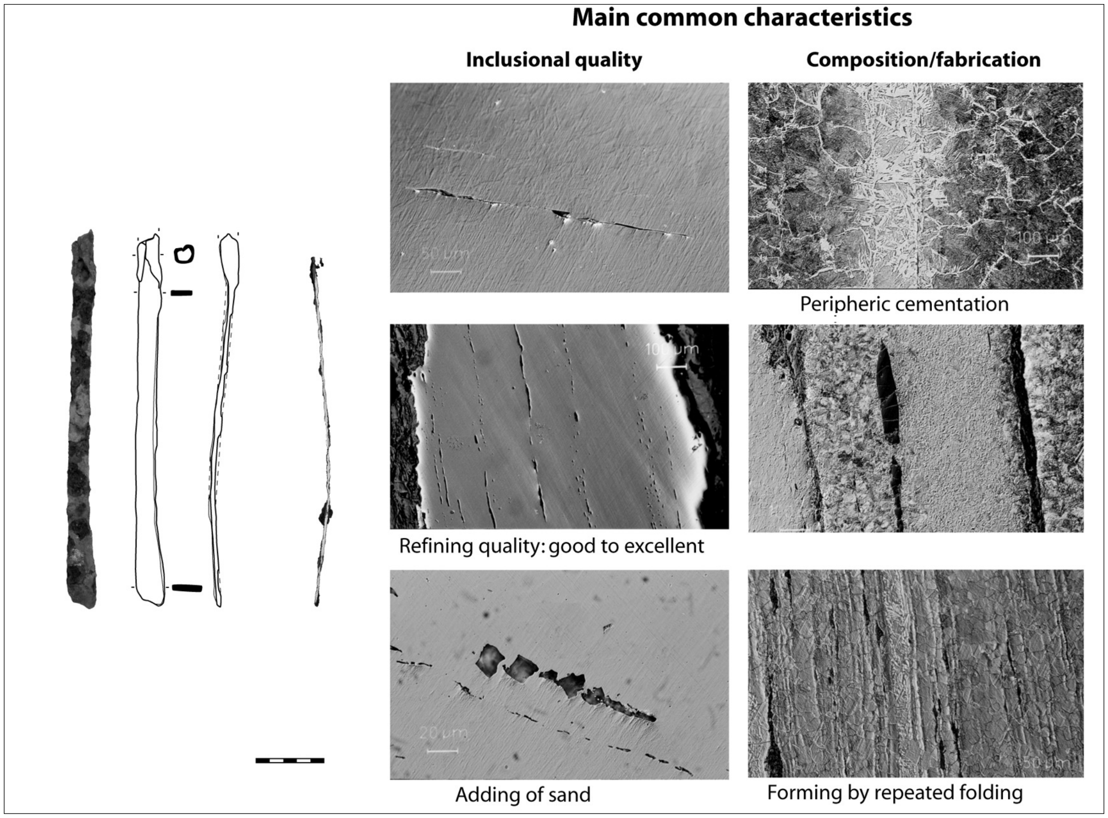

Fig. 5: Iron "socket bar" coming from St. Maur (Picardie, France), 2nd century BC. Weight 69 g (BERRANGER 2009).

- Category 4: Lower Al_2O_3/SiO_2 ratios without significant MnO or P_2O_5 levels.
- Category 5: Higher K_2O/CaO without significant MnO or P_2O_5 levels.
- Category 6: no particular composition.

The fact that some bars are constituted of blooms with different major chemical element signature could indicate that semi-products are not systematically forged on the reduction site as it is often asserted for this period. They could also be realised in different workshops more or less away from the reduction workshop and that import products from different workshops to manufacture the bars.

Another form of trade iron are "Currency bars"[19]. They present, mainly, a good to excellent quality of refining (depending of the heterogeneity of the initial bloom), and are constituted by a single bloom, sometimes hammered by repeated folding (more than 75 objects were examined by our team).

A new type of semi-product (300 specimens already studied), with the same morphology as the currency bars, but with a lower mass, was also evidenced: socket bars (fig. 5). They are small flat iron pieces with a socket (about 400x20x3 mm, average weight: about 100 g). These products seem to be mainly located in the French *Bassin Parisien* during the Iron Age[20]. Their studies reveal some qualitative particularities:

- Excellent inclusion cleanliness
- Intense hammering by successive folding that could conduct to very good mechanical properties
- Quasi-systematic cementation treatment. This cementation was probably made in order to facilitate welding by lowering the weld ability temperature.

19) CREW 1994.

20) BAUVAIS 2007; BAUVAIS-FLUZIN 2007; BERRANGER et Al. 2007; BERRANGER 2009; BERRANGER-FLUZIN 2011.

Considering these aspects, it seems that these latter semi-products show specific qualities indicating an elaborated functional role.

The chemical analysis of slag-inclusions contained in 19 socket-bars from 8 different sites of this sector permitted a more precise understanding of their circulation. The socket-bars, of local manufacturing and of local using, are composed of metals from various origins. This remark tends to confirm the massive importation of crude iron from distant sectors and a local transformation[21].

Conclusion

The aim of this paper was to present a review of the methodological aspect of our team for several years. In order to understand the production contexts of semi-products (technological, economical, historical), a pluridisciplinary approach has been attempted, from the understanding of the archaeological site to the one of the materials. Combining all sorts of information, gathered on wastes and products at different periods and on different areas, all these aspects can be enlightened.

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Riassunto

Un approccio archeologico e archeometrico dei semilavorati di ferro: tipologia, qualità e circolazione. In questo articolo viene presentato l'aspetto metodologico del lavoro multidisciplinare che il nostro gruppo conduce sui semilavorati. Esso combina le ricerche sugli scarti e sui prodotti, tenendo presente la stretta relazione che vi è tra l'archeologia e l'archeometria. In aggiunta all'analisi morfologia e tipologica esterna, vengono condotte sui manufatti differenti osservazioni metallografiche e analisi fisico-chimiche (particolarmente sugli inclusi nelle scorie) per ottenere una migliore comprensione dell'organizzazione dei siti e dell'economia tecnica in diverse aree durante determinati periodi.

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21) BERRANGER et Al. 2007.

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