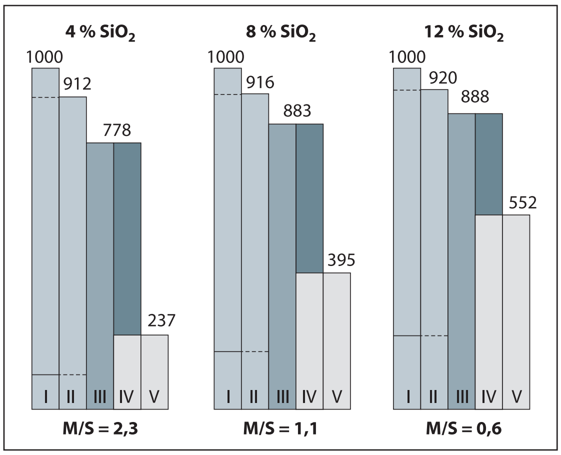
A new look at experimental ironmaking

The author discusses current experiments in reconstructing ancient ironmaking and suggests that serious experiments and more complex methods, which take proper slag formation into account, are needed.

Arne ESPELUND
(NO)

The modern blacksmith can buy his or her iron and steel as a cheap commodity in any hardware store. Still it is there: the dream of making one's own iron, well supported by the fact that ancient bloomery iron had a very good reputation. In my country Norway it was said by blacksmiths that it flowed on the anvil. They would gladly trade finished objects, such as horse shoes, in exchange for an ancient bloom (known from Fyresdal about 1930).

The remains of bloomery furnaces tell you that the construction was simple. A medieval furnace in my country consisted of a shaft made from clay with an inner diameter of about 30 cm. It was supported on three sides with vertical slabs of stone with an insulating layer of sand in between. The fourth side was "open", with a tap hole for slag and a hole for the mouthpiece of the bellows. The height is assumed to have been some 80 cm. The slag was tapped at intervals while the solid bloom was taken up with some sort of tongs. The Roman age furnace were different, and so was the type of furnace used between AD 1400-1800. However, most experiments aim at a re-enacting of the Medieval process based upon the type described above, which was common in Europe.



■ **Fig. 1** Column I initial weight 1000 g, showing in the lower part a total of some 10 – 20 % of oxides other than Fe_2O_3. Column II weight loss when Fe_2O_3 is reduced to FeO. Column III metallic iron is produced, with a further weight loss. In column IV the iron is consolidated. Column V shows the remaining slag. M/S expresses the ratio metal/slag. SiO_2 "steals" FeO and has a marked effect on output. However, a minimum of silica is required for slag formation, necessary as a solvent for FeO. With some 25% SiO_2 in the ore no metal can be expected!

Most of such experiments are happenings, performed on special occasions at archaeological open air museums. Many are not reported at all, but is is claimed by some that good iron is obtained. However, for this author they often miss two points: a comparison with ancient, well defined bloomery iron, and also some words about the output, relative to the amount of ore and the slag produced. In the following I shall present a few points, which should be relevant for experimenters. Admittedly, my information stems mainly from my home country Norway. However, with some 10 000 slag heaps representing three or more specific bloomery methods, spread in time and space, it is fair to say that my country has very rich material for such archaeo-metallurgical studies, which should involve archaeologists, metallurgists as well as blacksmiths. I am eager to reach experimenters with my ideas.

The combustion in a bloomery furnace can be represented by the equation.

$$2\,C + O_2 + 4\,N_2 = 2\,CO + 4\,N_2$$

This expression represents the fact that air consists of about four parts of nitrogen and one part of oxygen, and that the nitrogen just passes through the furnace. Enthalpy calculations express that the maximum (adiabatic) temperature of this combustion taking place in a shaft furnace with an excess of charcoal lies at around 1400 °C (*Espelund 1996, 2009*). Due to heat loss it is fair to assume that 1200 °C is the maximal operating temperature for a normal size bloomery furnace (while in large blast furnaces with cold blast, temperatures above 1300 °C are achieved). There is hardly any danger of running the bloomery furnace at a too high temperature, which might lead to liquid cast iron.

The reduction of the iron ore, assumed to consist of Fe_2O_3, is stepwise via Fe_3O_4 and FeO to metallic iron Fe. The equilibrium gas pressure at the final reduction step is represented by a gas ratio of p_{CO}/p_{CO2} near 1 : 1, while the gas emerging from the surface of glowing charcoal hardly contains any CO_2. When a normal ore is applied, one can only expect a reduction of the two elements iron Fe and phosphorus P, the latter dissolved in to the metallic phase. Elements such as silicon, aluminium and manganese are retained as oxides and end up in the slag.

The chemical analysis of bloomery slags from sites expressing a large and successful smelting show about 25 w % SiO_2, while the individual values for FeO and MnO present a wide scatter. However, as these two oxides behave in the same way in a slag, it is permissible to add them. This gives values around 62 w% for the sum. The solidified slag shows above all the presence of the compound fayalite $(Fe, Mn)_2SiO_2$, for which the value FeO+MnO is about 70%. As will be shown, a high amount of FeO in the slag is required to secure fluidity and a low content of carbon in the metal, required for good, malleable iron.

The morphology of Norwegian bloomery slags can broadly speaking be divide into three types: large pieces with cavities expressing the decay of organic material (from the slag pit furnaces of the Roman iron Age), flat and dense pieces from the Medieval side-tapped furnaces and porous slag from the most recent period (with the Evenstad type of furnace: dug-in with no tapping of slag).

It follows from this that only ores with a sum of FeO + MnO well above the value for a good slag can give any metal. In **fig. 1** the output of iron and the amount of slag produced are shown for three samples of roasted ore with alternative values 4, 8 and 12% of SiO_2 and a minor amount of MnO and Al_2O_3. Initial weight of ore 1000 g. Resulting slag assumed to be of the fayalite type.

This expresses that the ore provided for the experimental smelting in Eindhoven, the Netherlands, 2006, containing some 73% Fe_2O_3 and 23% SiO_2 could hardly give any iron in a bloomery process. If this iron ore upon smelting resulted in iron, there is reason to question the quality of this metal. Notice that an analysis is useless unless it includes silica, if possible also MnO and Al_2O_3, which at times are significant.

In Norway there are many museum pieces of bloomery iron. They are of two types:

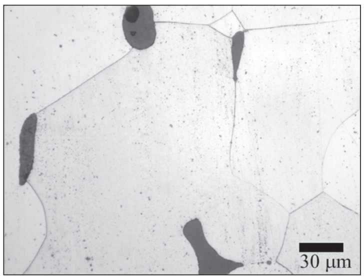
Rather flat, weighing some 2 - 12 kg, found in particular in the county of Telemark, and rounded, weighing some 18 kg, found mainly in Trøndelag. Both types have been cut halfway through with an axe, clearly in the red-hot state, probably in order to test the quality.

This author has had a chance to study some such blooms. Their densities have been measured by immersion in water, found to lie between 5 and 6.5, in contrast to 7.8 for pure iron, expressing that the blooms have an evenly distributed porosity. This also tells that the blooms are primary, a fact that is supported by the external dimensions.

Drillings from the bloom from Skeibrok were analysed chemically, giving the following

Element	% by weight
Si	0.01
Mn	< 0.01
C	< 0.01
S	0.008
P	0.006

The figures tell us that this is an extremely pure and slag-free iron (Si and Mn reflect slag inclusions). This is confirmed by metalloghraphy, as shown in **fig. 4**. A micrograph of a sample taken from the bloom from Skeibrok. Etching in 2% nital. It shows ferritic iron with very small amounts of slag at the boundaries where three grains meet.



■ **Fig. 4.** Micrograph of a sample taken from the bloom from Skeibrok. Etching in 2% nital. It shows ferritic iron with very small amounts of slag at the boundaries where three grains meet.

The analysis of the bloom from Li shows the following values:

Element	% by weight
Si	0.09
Mn	0.16
C	0.43
S	0.011
P	0.015

Microscopy revealed large differences in composition, in the range 0.7 - 0.1 %C. The value for carbon therefore must be regarded as a compromise. Still the content of slag is very low.

The two blooms described are representative for Medieval ironmaking in Norway. No secondary slag removal was required, therefore the output during the processing to finished objects must have been above 90 %. This is in great contrast to the results of experimental ironmaking in Sweden and Denmark (*Englund 2002, Lyngstrøm 2008*)

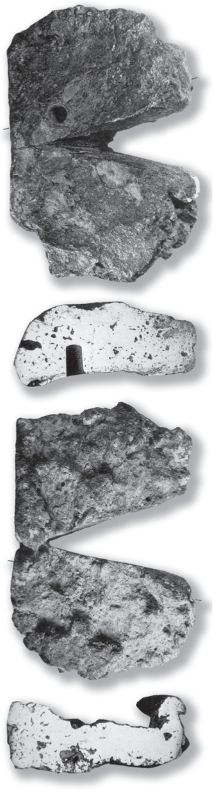
The Fe-C diagram expresses the equilibrium phase relations as a function of temperature. As seen, the iron created in a bloomery furnace in the presence of solid carbon, runs at a temperature of about 1100 °C should be of the austenite type, containing some 2% carbon. On the anvil this iron is hard and brittle, very different from the malleable iron, desired by blacksmiths. A majority of the primary blooms depicted in **fig. 2** contain little or no carbon. The result of such successful smelting can only be explained by assuming a slag control, represented by the chemical equilibrium

$$\mathrm{FeO} + \underline{\mathrm{C}} \longleftrightarrow \mathrm{Fe} + \mathrm{CO}$$

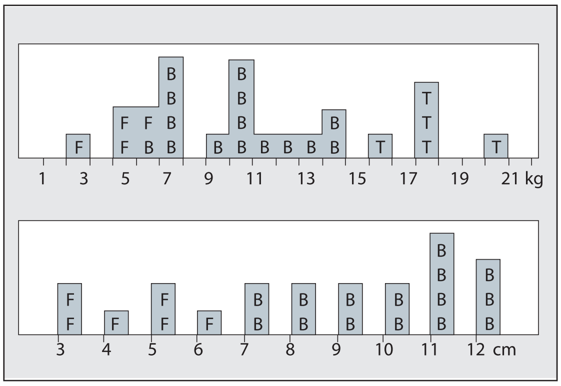
When liquid slag is present, near saturation with wustite FeO, the carbon content of the corresponding metal will be negligible (*Espelund 2008b*). The successful smelting is represented by a replacement of **carbon control** by **slag control**. In the English-speaking world it was said: *"Make your slag and the metal looks after itself."* The slag must be a silicate slag, commonly named fayalite slag.

It now only remains to achieve proper slag formation, in addition to a reduction from oxides to metallic iron. In a properly run bloomery furnace, using a rich ore, it is likely that iron oxide is reduced all the way to metal, without allowing enough FeO for the reaction with silica in the ore and a refining.

In the Evenstad process, fully described in contemporary writing from the year 1782, the primary product is a carbon-rich metal. This is refined in the furnace by an addition of 4 l of ore towards the end of a smelting cycle, lasting some 5 hours (*Evenstad 1968*). The other process described in contemporary writing: the Catalan process from the Pyr-



■ **Fig. 3** Blooms from Skeibrok a-b (weight 4.02 kg) and Li c-d (weight 2.83 kg). Shown are also the cut surfaces. From Martens (1979).



■ **Fig. 2** Histograms according to weight and thickness of ancient blooms, found in Norway, now mostly in museums. F denotes fellujern, B blåsterjern, in agreement with two kinds mentioned with prices in the medieval law book Jonsbok from AD 1281. Values from Martens (1979). Blåsterjern is a primary metal, while fellujarn has been compacted by moderate smithing. Blooms marked T, added in the present figure, have been found mainly in Trøndelag and stem from the Roman Iron Age type of furnace. As the latter blooms are rounded, thickness has no meaning.

enees – Basque country, uses a shaft which is divided vertically into two parts: In the one part normal reduction takes place while fine grained ore is supplied continuously to the slag bath from the other part. Both processes are very specific and at the same time express sound metallurgy, which clearly is based upon the principle of **slag control**. It follows that processes such as the two mentioned in contemporary writing would never have been developed if iron could be made in the simplest possible way.

In a lecture held in 2001 I presented alternate ways to produce good iron by proper slag control (*Espelund 2008a*). During recent years I have come across many samples of slag containing 5 – 10% SiO_2 while a normal "end" slag should contain some 25%. Admittedly at most places only slag of the latter kind has been found. However, at sites such as Møsstrond in Telemark (*Espelund 2004*), Fagstad near Lillehammer and also near Uppsala (*Hjärthner-Holdar 1993*) slags of two kinds have been reported (*Espelund 2009*).

It is therefore claimed that one wide-spread way to make good iron involved a **two-step process**. In the first smelting the reduction was moderate. The oxide Fe_2O_3 in the ore was reduced to FeO, which reacted with silica present to form fayalite according to

$$2\,FeO + SiO_2 = Fe_2SiO_4$$

This transition is expressed by the columns I –II in **fig. 1**. During the smelting step that followed therefore fayalite would be present, thus securing good iron.

As the intermediate product was very valuable, it is not likely there will be much to find. However, at a site visited in September this year (Røst in Dalsbygda, Os) , only rich ore and a few pieces of slag containing some 9 % SiO_2 were found. It appears that only the semi product was produced at this place and smelted to good iron somewhere else.

It is tempting to compare a two-step process with a cow chewing of the cud! We know of this behaviour because we have seen it, not from finds of the intermediate product. Preliminary studies of find material indicate that the Medieval type of furnace could be used for both process No. 1 and 2.

Concluding remarks

It is most likely that blacksmiths will continue their attempts to make good iron in a simple, direct process by charging the furnace with roasted ore and charcoal. I hope that with this contribution I can convince them that an ore with a high ratio of iron oxide to silica is a primary requirement. It is remarkable that the good ironmakers in Tranemo, Sweden went some 300 km north to fetch their good ore in Härjedalen (*Espelund 2008b*).

It is likely that some iron will be created in experiments of this kind. Such was the situation also for our forefathers. The development of very specific and complex methods can only be explained by a moderate success in standard experiments of the kind seen in Eindhoven. The reproducibility of the high quality of iron and also the output expressed by material finds are remarkable. No modern bloomery ironmaker has presented results that can be compared with the best results of our forefathers.

It now remains to make serious experiments with a two-step process, or some other method, which takes proper **slag formation** into account. Perhaps a much reduced ratio of charcoal to ore will give the right conditions for the pre-reduction. I hope that some of the arduous blacksmiths will try the proposed two-step process by systematic experiments!

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Summary

Ein neuer Blick auf die experimentelle Herstellung von Eisen

Eine große Zahl experimentalarchäologischer Aktivitäten zur Eisenherstellung werden nicht publiziert, und wenn, dann werden oft zwei Punkte vergessen: Der Vergleich mit bekannten Funden archäologischer Eisenobjekte, aber auch das quantitative Resultat des Schmelzprozesses im Vergleich zur Menge des eingesetzten Erzes und der anfallenden Schlacke. Eine gute Publikation sollte alle Schritte des Herstellungsprozesses beschreiben: Die Verbrennung in einem Schmelzofen, die Eisenreduzierung und die chemische Analyse des gewonnenen Eisens als auch der Schlacke. Es ist in jedem Fall notwendig, die Ergebnisse der Experimente mit archäologischen Objekten zu vergleichen. Weiterhin ist es wichtig, seriöse Experimente mit solchen Methoden durchzuführen, die zur Bildung entsprechender Schlacken führen, um Ergebnisse zu erzielen, die auch tatsächlich mit den Produkten unserer Vorfahren vergleichbar sind.

Une nouvelle approche du travail expérimental du fer

Les expériences liées au travail du métal sont aujourd'hui nombreuses, mais n'apportent pas toujours leur contribution à la réflexion scientifique à leur juste valeur. La plupart ont lieu lors d'événements particuliers, et nombreuses sont celles qui, malgré des résultats très encourageants, ne donnent pas lieu à des publication de résultats ou à des comparaisons avec des vestiges archéologiques.

Pourtant, il est nécessaire pour une bonne expérience de prendre en compte toutes les étapes du processus : la combustion dans le fourneau, la réduction du fer, l'analyse chimique du métal obtenu ainsi que des scories. Il est indispensable également de confronter ces résultats avec les observations de terrain et les vestiges conservés... Ce n'est que par là que le travail expérimental des forgerons d'aujourd'hui permettra de retrouver les techniques d'hier.

■ **Arne Espelund** is Professor Emeritus at the Department of Materials Science and Engineering - NTNU in Trondheim, Norway.