

■ **Fig. 1** Smelting according to Evenstad's manuscript from year 1782, printed in 1790. The 60 pages long manuscript covers every aspect of contemporary ironmaking. Drawing H. Ness

## Experimental ironmaking once more: combining theory and find material

**In the previous paper the excellent quality of Norwegian Roman Age and Medieval blooms was documented. The number of sites tells us that the method was reproducible, and that the blacksmith could make finished items from the blooms with a high output, very different from many reported experiments.**

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### Introduction

In the previous paper *A new look at experimental ironmaking* (Espelund, in *EuroREA 2009*) I presented the excellent quality of Roman age and Medieval Norwegian blooms, most of which are now kept and preserved in Norwegian archaeological museums. Because they are, so to speak, slag-free, no smithing in order to remove slag was necessary. Their densities are around 6, in contrast to 7.8, which indicate that they are in a primary state and were not influenced by heavy smithing. This viewpoint is also supported by the general lack of hearths and anvil stones at Roman and Medieval bloomery sites in Norway. They number some 500 and 10,000 respectively. The lack of entrained slag is expressed chemically by the very low contents of silicon (Si) and manganese (Mn) (representing oxides) found by conventional analysis of the metal, and their low densities due to empty micropores. They appear to be evenly distributed and present no problem upon smithing.

The large number of sites, the older ones very large with several furnaces and up to 50 tons of slag, express that the process was reproducible. In this paper I will suggest that the processes were *complex*, but not *complicated*.

The quality is remarkable. A basic question is how the sintering of assumed primary small fragments to large lumps of solid, slag-free iron was achieved. The carbon control can be attributed to human efforts while the sintering must be a purely physical phenomenon. A driving force for the sintering process is the lowering of the specific surface area, as is also known for snow crystals, growing in time when left undisturbed. In the year 1794 the Swedish metallurgist Carl Rinman clearly expressed that iron oxide must remain in the slag in order to flux it and also combine *förena*, the ductile grains of iron (Rinman 1794:17). He inserted a rod of iron into the tuyere of his furnace in order to test the quality of the slag, a measure also known from the Catalan process.

Because of their quality, the output upon smithing of the primary bloom into finished objects is assumed to represent some 90 %. Accordingly, the blooms indicate a process that is different from the standard experiments of modern bloomery ironmakers with their yield of some 15 to 20%. In particular I refer to reported experiments in a medieval type of furnace in Sweden and Denmark. In Norway such experiments have been carried out at Kittilsbu in Oppland and at

Borre in Vestfold, however not reported in the same thorough way as those first mentioned.

Smelting by the Evenstad technique by following his cookbook-like advice from the year 1782 is another, but successful story. By <sup>14</sup>C-dating we know that it was introduced around year 1400, probably less than 50 years after the Black Death. Pictures, redrawn from experimental smelting are shown in **Fig. 1**.

As shown, it is a step-wise process. Wood is at first transformed into charcoal in the conical-shaped furnace. It should be noted that a primary over-reduction to a carburised metal is followed by an oxidising refining by adding roasted ore at a late stage, when only a little remains in the furnace. This is a modern interpretation of his very practical advice. The product of the smelting is finally removed with a shovel. Some of it represents a bloom, while the rest is thrown onto the slag heap. The process therefore is direct only in the sense that it was performed in the same furnace. It consists of reduction followed by refining.

As an example of a misleading experiment, I would like to present the following case study: The Norwegian TV NRK I on January 2nd 2009 showed smelting which was claimed to be successful. The tapping of slag went well while the iron produced appeared to be brittle. However, when asked for details I was told that bones had been added to roasted bog iron ore. Bones contain calcium oxide (CaO) and also

phosphorus (P), detrimental for the quality of the iron produced. By replacing iron oxide (FeO) in the slag, this (CaO) can increase the output as well as the fluidity of the slag, however, at the cost of metal quality. The archaeologist T. Gansum has also added bones to his charge – in his case deliberately (*Gansum & et al 2002*). However, analysis of hundreds of ancient slag samples have only rarely revealed contents of CaO above 1% (*Espelund 2009a*).

In this paper I shall go into theoretical details, show some analyses, express something about the ore situation and present my ideas about how a two-step process can be carried out.

### Basics of the iron-carbon -oxygen system

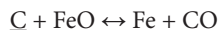
The iron-carbon diagram is, from a metallurgist's point of view, indispensable when the formation and the quality of iron and steel are discussed. It is shown in **Fig. 2**.

Noteworthy is the line Acm, representing saturation with iron carbide Fe<sub>3</sub>C (or elemental carbon C), which reaches 2% at 1100 °C. Carbon atoms are accommodated in the face-centred crystalline cubic structure of γ-Fe, also named austenite. A metal with 2 % carbon cannot be forged.

The Baur-Glässner diagram, shown in **Fig. 3** expresses both the relations between gas mixtures, such as those created in a bloomery furnace, and the phases α- and γ-Fe, carbon and wüstite (FeO).

The lower curve shows the gas composition in chemical equilibrium with solid carbon. Notice that the content of carbon monoxide (CO) rises drastically between 650 and 800 °C. As seen, no reduction of FeO to metallic Fe can take place below a temperature of about 660 °C, which is a red-glow heat. The right curve expresses the gas ratio for coexistence of the lowest oxide FeO and the metal. This metal is practically carbon-free, well suited for forging, while the values for γ-iron in contact with carbon, shown by means of the left curve, are high – the same as those in the Fe-C-diagram. This value is much too high for malleable iron.

The removal, or exclusion, of carbon in γ-iron can be expressed by the equation:



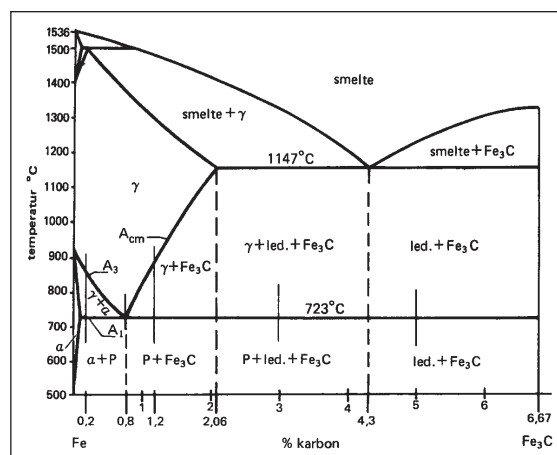
This is a chemical equilibrium, where  $\underline{C}$  expresses carbon dissolved in the metal. A high content of FeO corresponds to a low value for dissolved carbon.

The idea should therefore be to maintain iron oxide (FeO) together with the metal produced, if possible “at birth”, in the *statum nascendi*. However, at the operating temperature of a bloomery furnace – some 1100 °C – FeO is solid. Its chemical function can be taken over by FeO dissolved in a liquid iron silicate, so-called fayalite slag. This slag is fluid at normal operating temperatures and is therefore separated from the solid iron. The remaining question now is how we can secure a proper slag. Over-reduction to a carburized metal without the beneficial effect of FeO dissolved in a silicate slag seems to be the problem.

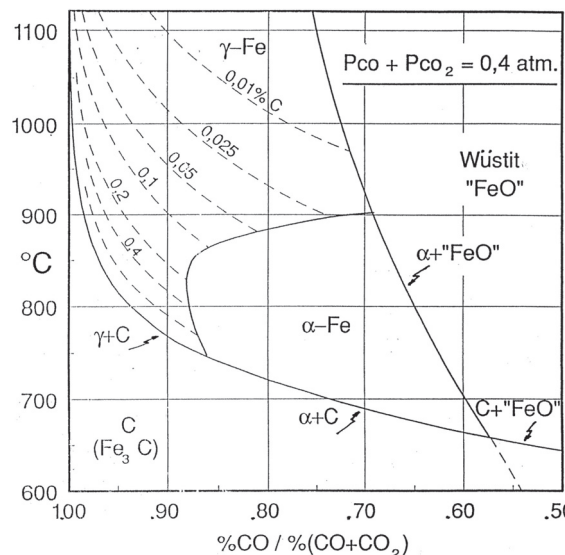
### From finds to theory and back again

Among archaeometallurgists in Norway the character of the slag now is regarded as very significant. The Roman type is vesicular, present also as large lumps, expressing that it solidified in a slag pit. Organic material (wood) in the pit was imbedded in liquid slag and later decayed. The Medieval slag is mostly dense and flat with a smooth upper and a rough lower side, expressing tapping sideways at ground level, while the “Evenstad” type is porous, and that gas evolution, due to an addition of ore at a late stage, took place during solidification. Because of its specific character, <sup>14</sup>C-dating of charcoal from a slag heap will rarely be a surprise to the experienced archaeometallurgist. The three categories normally represent periods of maximum production around year 200, 1100 and 1600 AD, respectively.

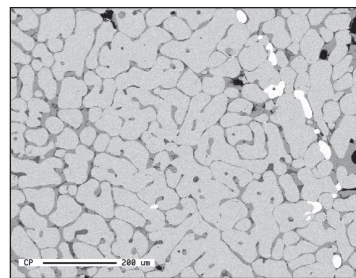
Wherever slag is present in large amounts, it expresses a large and successful production. Irrespective of site, period, type of furnace, process and ore, the “end” slag contains 63 ± 3% (FeO and MnO) and 23 ± 3% SiO<sub>2</sub>. The individual values for FeO and MnO show a wide scatter, but



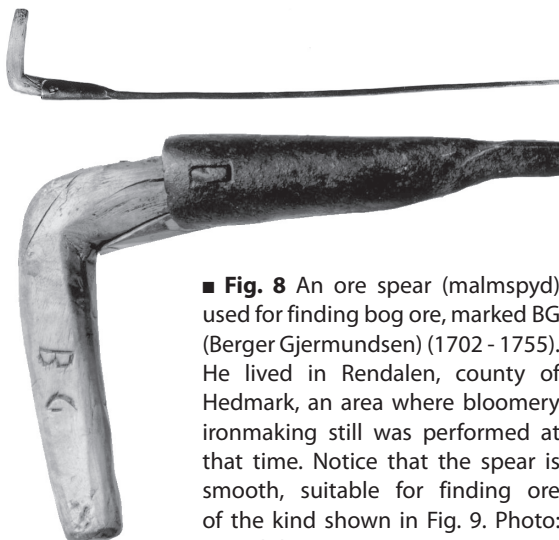
■ **Fig. 2** The Fe-C-diagram expressing stability ranges for the different phases. Abscissa %C, ordinate temperature. A vertical line is drawn at %C = 0.2, a material of the best possible quality for general purposes in the smithy. This iron must be produced as a solid in the γ-state around 1100 °C, which is the operating temperature of a bloomery furnace



■ **Fig. 3** The Baur-Glässner diagram for a combined pressure of CO + CO<sub>2</sub> = 0.4 atm., balance nitrogen N<sub>2</sub>. This is the typical gas created by combustion of charcoal in a bloomery furnace. Abscissa ratio CO/(CO + CO<sub>2</sub>), ordinate temperature. Dotted lines in the γ-field express the carbon content at different temperatures and gas mixtures.



■ **Fig. 4** Slag from Nystaul NIKU No. 99/31. Sample No. 2 in the table 1. White metallic iron, rounded particles FeO, and dark matrix fayalite-like, probably glass



■ **Fig. 8** An ore spear (malmspyd) used for finding bog ore, marked BG (Berger Gjermundsen) (1702 - 1755). He lived in Rendalen, county of Hedmark, an area where bloomery ironmaking still was performed at that time. Notice that the spear is smooth, suitable for finding ore of the kind shown in Fig. 9. Photo: P. Fredriksen

they can safely be added because of similarity as slag components. Samples with such analyses have been gathered from Roman age, Medieval and newer sites in Norway, Medieval sites from Iceland, a Roman age site in Austria, as well as Catalan sites in the Pyrenees (*Espelund 2009a*).

For chemical analysis, representative of pieces that express solidification from a homogeneous liquid, should be selected. A few samples of slag containing around 10% SiO<sub>2</sub> seem to express a story different from the one above. Was it a failure? Or was it produced on purpose as a semi-product? It seems to be necessary to cast a look on the ore and to follow it all the way to a good metal. In Fig. 1 in the previous article bog iron ore containing alternatively 4, 8 and 12 % SiO<sub>2</sub> was followed through four steps via FeO as

an intermediate oxide. It is possible that 4% SiO<sub>2</sub> may be the minimum required for the desired slag formation (*Espelund 2009b*), while 20 % or more of SiO<sub>2</sub> in the ore will give fayalite slag and little or no metal.

At a few sites two kinds of slag have been found, as shown in the **table 1** (R represents the ratio [%FeO + % MnO] / % SiO<sub>2</sub> with molar values. For fayalite the value of R = 2. The slags marked 1, 3 and 6 therefore contain fayalite with excess FeO.)

The slag No. 2 has been analysed with SEM. **Fig. 4** shows back-scattered electrons.

A thorough search for bloomery sites by Mr. Asbjørn Ryen in the community Os, county of Hedmark, led to a site at Røst in Dalsbygda, shown in **Fig. 5**.

The only visible structures are two rings of stones, reminiscent of places for an open fire, and also two pieces of burnt clay. However, in front of Mr. Skare we found roasted ore and charcoal, and behind the junipers to the left a few pieces of slag. <sup>14</sup>C-dating AD 1165-1225 (charcoal from birch). No charcoal pit or large slag heap was found in the vicinity.

A sample of roasted ore was acquired by magnetic separation. Chemical analyses of this ore and of three pieces of slag are given in **table 2**. Notice the similarity for S 1-3 with slags nos. 2, 4 and 7 in **table 1**. Could it be that the slags represent a pre-treatment

of ore, a partial reduction of Fe<sub>2</sub>O<sub>3</sub> to FeO, followed by reaction of FeO and SiO<sub>2</sub> to fayalite Fe<sub>2</sub>SiO<sub>4</sub>?

The slag marked S2 was also studied by SEM-analysis, resulting in a picture very much like that in **Fig. 4**. The site is regarded as a place where only step 1 of a two-step process was carried out.

A site near Granhaugsetra in Sollia, municipality of Stor-Elvdal, county Hedmark similar to the site Røst is now being studied.

### How was step 1 performed?

**Fig. 4** is dominated by the lowest oxide of iron FeO, while specks of metal also occur. The formation of this mixture must take place in an environment where the combustion of carbon (charcoal) delivers heat, while a chemical equilibrium between the solid or liquid slag with carbon or the equivalent CO-rich gas must be avoided.

In **Fig. 6** the standard medieval bloomery furnace has been modified by making holes for admission of air in the shaft, which is only half full.

It is conceivable that the wanted ratios of CO/(CO + CO<sub>2</sub>) near 0.75 – see **Fig. 3** – can be created in this way. Such secondary combustion by admission of air is essential for the puddling process for the refining of pig iron invented by Cort in England. The heat of combustion

	1	2	3	4	5	6	7
	Niku99/31 End slag* Medieval Møsstrand	Niku 99/31* Hellegryte slag	Fagstad Slag D**	Fagstad Slag E**	Fagstad E SEM**	AMI 2 Lyckebo***	AMI 1 Lyckebo***
MnO	1.280	0.250	12.610	1.990	1.969	0.250	0.110
FeO	64.460	90.50	54.10	81.010	76.101	61.940	76.400
<b>SiO<sub>2</sub></b>	<b>24.080</b>	<b>6.330</b>	<b>21.610</b>	<b>8.510</b>	<b>10.481</b>	<b>25.500</b>	<b>9.200</b>
Al <sub>2</sub> O <sub>3</sub>	4.910	0.630	6.790	2.000	2.556	5.900	2.800
P <sub>2</sub> O <sub>5</sub>	0.322	0.138	2.410	4.186	6.278	0.190	0.230
<b>Sum of 10 oxides</b>	<b>98.062</b>	<b>98.268</b>	<b>100.120</b>	<b>99.696</b>	<b>100.000</b>	<b>*99.360</b>	<b>*92.490</b>
FeO+MnO	65.740	-	66.710	-	-	62.190	-
R	2.280	11.950	2.580	8.130	-	2.050	6.930

■ **Table 1** Analyses from sites with two kinds of slag. Notice the differences for % SiO<sub>2</sub> and R, both clearly at two markedly different levels. As seen, the parallel values for FeO are not so distinctly different as the contents of silica SiO<sub>2</sub>

	M	S1	S2	S3
MnO	1.71	3.84	2.8	3.53
FeO		81.4	85.1	81.25
Fe <sub>2</sub> O <sub>3</sub>	93.92			
Al <sub>2</sub> O <sub>3</sub>	1.31	2.58	2.19	2.47
SiO <sub>2</sub>	2.82	9.52	8.05	9.97
CaO	0.17	1.64	1.02	1.40
MgO	0.01	0.39	0.23	0.41
K <sub>2</sub> O	0.08	0.38	0.33	0.29
P <sub>2</sub> O <sub>5</sub>	0.058	0.088	0.045	0.061
TiO <sub>2</sub>	0.01	0.05	0.06	0.08
BaO	0.17	0.23	0.20	0.21
Sum	100.3	100.1	100.0	99.7
<b>R</b>	<b>25.5</b>	<b>7.46</b>	<b>9.11</b>	<b>7.09</b>

■ **Table 2** Chemical analyses of roasted ore M and of three pieces of slag S1 – S3 from the site Røst

of carbon to CO<sub>2</sub> is almost 3 times larger than the primary combustion to CO, so that the charcoal required in this case is much less than in a true reduction furnace.

The problem of tapping or of some other method for retrieving slags of the type shown in Fig. 4 from the furnace remains to be solved. In Fig. 6 tapping of such a mixture is indicated. But it seems unlikely that liquid, FeO-rich slag can pass through a layer of incandescent charcoal. However if the temperature can be maintained, such tapping is conceivable after the charcoal has been consumed. The present author is eager to discuss such a process with experienced smelters.

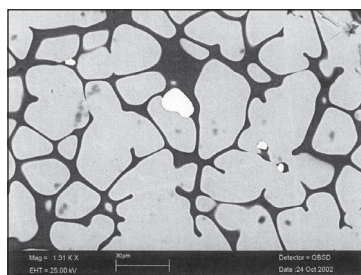
One question is whether the proposed practice was performed only in Norway. A micrograph of slag from England, presented without any further description, shows exactly the same pattern as intermediate slag in Norway, such as shown in Fig. 4 above.

### The ore situation

Attempts to make experiments and produce bloomery iron have been met with difficulties in finding a good ore. Broadly speaking, in my country we now hardly find bog iron



■ Fig. 5 The archaeologist K. Skare at the site Røst



■ Fig. 7 Micrograph of slag from England (Bayley & al., 2008). Notice FeO as a dominant phase. A nearly similar sample is shown by Pleiner (2000), as in picture 2 on Plate XXIII

ore of a satisfactory quality. This pattern was also expressed in 2007 by Swedish ironmakers from Tranemo near Göteborg, who went 300 km north to find good ore. I claim that good ore of the kind described by Ole Evenstad in the year 1782 has been consumed and not renewed. He used a metallic stick in order to locate good ore, mostly found as lumps in a layer some 20 cm below the topsoil. Such a stick is shown in Fig. 8.

The development of bog ore formation can be explained in the following way: The Scandinavian Peninsula was largely covered by ice until about 10 000 years ago. The slow movement of this ice removed the soil and the vegetation, leaving in places moraine material. Barren rocks on exposed surfaces dominated. A humus layer was gradually built up during the millennia to follow. Where sloping mires (*bakkemyrer*) were created the conditions for ore formation were favourable, provided a reasonable thickness and precipitation. The formation took place underground over decades or centuries and resulted in lump-size limonite. The botanists name such mires *minerotrophic*. A rare find of such ore is documented in Soknedalen, county of Sør-Trøndelag, shown in Fig. 9.

The analysis of a lump of this ore after roasting at 650 °C ran as follows:

Sørstuv	
MnO	0.45
FeO	
Fe <sub>2</sub> O <sub>3</sub>	84.07
Al <sub>2</sub> O <sub>3</sub>	1.63
SiO <sub>2</sub>	12.62
CaO	0.36
MgO	0.07
K <sub>2</sub> O	0.09
P <sub>2</sub> O <sub>5</sub>	0.001
TiO <sub>2</sub>	0.04
BaO	0.12
<b>Sum</b>	<b>99.4</b>
<b>R</b>	<b>5.0</b>

The value for R = 5.0 (%FeO+%MnO) / %SiO<sub>2</sub> (molar) expresses that it is acceptable as a raw material. Notice the low content of phosphorus.

During recent centuries or millennia these mires have grown in thickness so that their character

has changed to *ombrotrophic*, i.e. affected by rainwater only. Accordingly there is no longer any contact between dissolving minerals in the bottom and oxygen in the air.

It can be mentioned that the consumption of this type of bog ore during the Roman Iron Age in Trøndelag has been assessed to at least some 25 000 tons (Espelund 2009c).

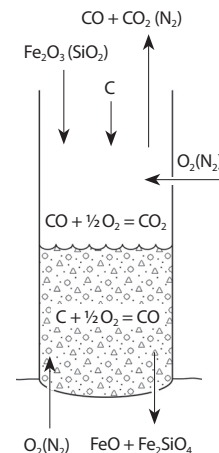
However, visible ore formation takes place also nowadays in ochre wells (*raudveller*). Ground water at some 4 °C containing dissolved divalent ions of iron Fe<sup>2+</sup> when emerging and meeting oxygen in the air will precipitate ochre as a running process. Chemically it can be represented by FeOOH. In contrast to the sloping mires, a well is created below the bog. When dried this ochre is extremely fine-grained and fluffy, suitable as a pigment. It appears to have been avoided as a raw material for iron production in the past, due either to fineness, too much silica or phosphorus (introduced by bacteria which thrive in such an environment). I have found some twenty such ore wells in my country. In Fig. 10 the ochre well at Namlåmyra in the community Midtre Gauldal, county of Sør-Trøndelag is shown.

An analysis of the fresh precipitate after roasting at 600 °C gave the following result:

Namlå	
Fe <sub>2</sub> O <sub>3</sub>	76.86
MnO	3.36
SiO <sub>2</sub>	15.9
Al <sub>2</sub> O <sub>3</sub>	0.01
P <sub>2</sub> O <sub>5</sub>	0.055
CaO	3.1
MgO	0.01
BaO	0.2
TiO <sub>2</sub>	0.01
K <sub>2</sub> O	0.01
<b>Sum</b>	<b>99.5</b>
<b>R</b>	<b>3.8</b>

The temperature of the emerging water was found to be + 4 °C, while pH was 6.84.

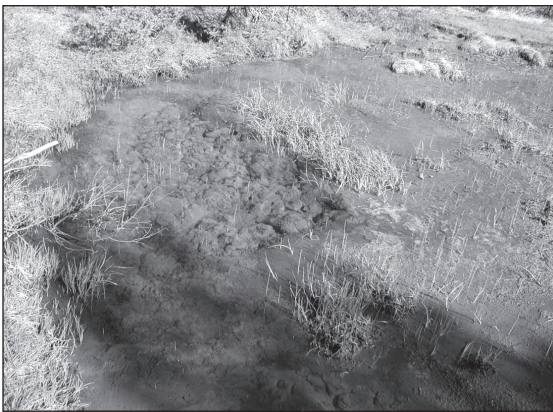
This type of current ochre formation does not appear to occur on the Continent. On the other hand,



■ Fig. 6 Modified medieval furnace, in which air is admitted in the shaft, allowing secondary combustion of CO to CO<sub>2</sub>. Such a process might transform the silica in iron ore into fayalite. Concept A. E.



■ **Fig. 9** Limonite ore in a ditch near Sørstuvollen in Soknedal, community of Midtre Gauldal, county Sør-Trøndelag. On the picture the local informant Arnt O. Solberg and Magne Rokne. Due to road building, drainage of water has taken place at this spot. The overall impression is that of a sloping mire (bakkemyr)



■ **Fig. 10.** The ore well at Namlåmyra in Singsås county of Sør-Trøndelag

large lumps of ore used formerly for house construction in Denmark and Lower Saxonia in Germany are not available in Norway.

### Conclusion

In this and the previous paper, an emphasis is put on a presentation of representative finds and also on a connection to metallurgical theory. One should reckon that it took some 1000 years for ironmaking to reach Scandinavia from a beginning in the Near East around year 1500 BC. It is very naïve to believe that the simplest, possible modern experiment should lead to a metal of comparative quality to that created by the early ironmakers during the Middle Ages.

The manuscript of Ole Evenstad tells about a complex, discontinuous practice, which is revealed to us in

his remarkable book on contemporary ironmaking. His method is satisfactory from a modern metallurgical point of view. It would not have been revealed by the finds only.

It is conceivable that a pre-treatment of ore, rendering a semi-product composed of mainly iron oxide and iron silicate, would be an excellent raw material to feed into a normal reduction furnace as an alternative to a material composed of iron oxide and silica. The presence of fayalite should warrant a metal that met the demands of the blacksmith.

The author is looking forward to a practical cooperation with blacksmiths who are willing to incorporate a serious interpretation of finds material, as well as modern metallurgical thinking in their experiments. It is a pity that facilities for analysis of ores, slag and metal hardly can be said to be available as a service to the many experimenters.

### Acknowledgement

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### Summary

#### ■ **Noch einmal experimentelle Eisenherstellung – Zur Kombination von Theorie und Fundmaterial**

Im vorigen Artikel wurde die gute Qualität der norwegischen Luppen aus der Römerzeit und dem Mittelalter beschrieben. Die Anzahl der Fundstellen zeigt, dass die Herstellung reproduzierbar war, und auch, dass der Schmied Fertigprodukte mit hoher Ausbeute herstellen konnte, sehr unterschiedlich von vielen beschriebenen Experimenten.

Das Fe-C-Diagramm zeigt, dass ein Kohlenstoff-Gehalt von 2 % bei angemessener Arbeitstemperatur im Rennofen zu erwarten ist. Der erwünschte niedrige Inhalt kann nur durch Schlackenkontrolle erzeugt werden. Im Evenstad-Verfahren wird das Primär-Eisen durch einen Zusatz von extra Eisenerz gefrischt. Im Mittelalter-Verfahren schlägt der Verfasser vor, dass die Herstellung zwei Stufen umfasst. In der ersten Stufe wurde ein Gemisch von FeO und Fayalit hergestellt. Das Zwischenprodukt wurde dann reduziert, wobei man gutes Eisen und Schlacke erhielt. Der Verfasser schlägt zuletzt vor, wie das Zwischenprodukt hergestellt werden konnte.

#### ■ **Nouvelles expérimentations sur le travail du fer: études théoriques et matières premières**

L'article précédent a documenté l'excellente qualité des lumps Norvégien de l'époque Romane et Médiévale. Le nombre de sites trouvés prouvent la reproductibilité de la méthode, ainsi que l'habilité du forgeron à faire des produits avec un grand débit des lumps, ce qui est très différent des récents résultats d'expériences.

Le diagramme Fe-C montre que 2% C doit être envisagé pour un acier produit à la température de fonctionnement du four. L'acier à basse teneur en carbone désiré peut seulement être obtenu par control du laitier. Dans le procédé d'Evenstad, le métal primaire carburé est raffiné par ajout de minerai en fin de processus. Pour le procédé Médiéval, l'auteur suggère une première étape consistant à la réduction partielle en FeO et fayalite, suivie d'une réduction de ce semi produit. De petites quantités de laitier trouvées contenant 8-10% de SiO<sub>2</sub> semblent représenter ce semi produit. L'auteur suggère une méthode pratique pour la première étape – celle d'une réduction partielle.

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