A pioneer of experimental metallurgy: Monsieur de Réaumur

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Introduction

At the death of Louis XIV in 1715, the regent Philippe d'Orléans requested the "Académie des Science" founded by Colbert in 1666, to prepare a "Description des Arts et des Métiers". As an active member of the Academy, René Antoine Ferchault de Réaumur was asked to make a report on the manufacture of iron and steel.

Born in 1683 in a family of recent nobility, he was very much attached to its traditions and privileges. At the age of 20, however, he abandoned his study of law to devote himself to mathematics and physics. Réaumur, in spite of his aristocratic background, and for his time, had an open mind and a great scientific curiosity. He considered that only carefully conducted experiments could give reliable answers. He also believed that rules must be deduced from experiments, rather than using experiments to prove already-made propositions.

In 1722 the Academy published Réamur's twelve Memoirs on "L'Art de Convertir le Fer Forgé en Acier" (the art of converting iron into steel)*.

Carburization of Bar Iron:

Réaumur's experiments concerned essentially the manufacture of steel by cementation of wrought iron. He made that choice because, although plain steel was made in France, high quality grades were imported either from Germany or England. Cementation was used most often in their manufacture, by carburization at high temperature of wrought iron bars packed with suitable carbon sources in sealed boxes. This process known since antiquity. The failure to use it successfully in France aroused Réaumur's curiosity.

Several attempts to introduce the process had been unsuccessful. Réaumur wrote: "The Court has been oppressed (accablée) especially during the last three or four years, by Frenchmen and foreigners of all countries, who, in the hope of making their fortune, have presented themselves as having the true secret of converting iron into steel. But no fruit of their labour has been seen . . . those who promised to change the irons of the Kingdom into steel, have almost been regarded as the searchers after the philosopher's stone".[1] Thus, the challenging aspect of this study stimulated Réaumur's interest.

Scientific knowledge in Réaumur's time

The elementary scientific knowledge at the beginning of the 18th century must not have been of great help to Réaumur. One must remember that natural substances like air, water and even ores were considered pure elements. Metals like gold, copper and iron were seen as alloys, and amalgams as mixed elements. Artificial phenomena were either "diacresis" i.e. decompositions, corruptions, dilutions; or "syncresis" i.e. mixions, combinations, coagulations. To monitor these artificial phenomena, the scientist had two main elements at his disposal, namely: Phlogiston, or fire, and Menstrua, or dissolving agents. The real nature of fire was unknown but it was considered a pure element called Phlogiston by Stahl in 1697. Furthermore, there was still confusion between the concepts of heat and temperature. It was accepted that to make pig iron one had to add phlogiston to the iron ore (a pure element). To obtain wrought iron, more phlogiston must be added, and steel is iron saturated with phlogiston. In spite of Réaumur's work, these views were still published in the "Encyclopédie" in 1751–1765.[2]

Réaumur's approach to a technical problem

As implied previously, when surveying the French iron and steel industry, Réaumur was impressed by the poor quality of French steel compared to imported ones manufactured by cementation. He decided therefore to study this process by conducting experiments designed to answer the following questions:

1—What is the best source of carbon?
2—How to recognise suitable wrought irons?
3—What are the best operating conditions?
4—What differentiates wrought iron from steel?
5—How to assess, on small samples, the quality of steels?
6—What is the best quenching practice?

At the beginning of the 18th century this was probably the first scientific approach to solve a metallurgical problem.

The twelve Memoirs of Monsieur de Réaumur

To answer the two first questions, Monsieur de Réaumur devised a suitable experimental procedure. To compare various carbon sources tests had to be

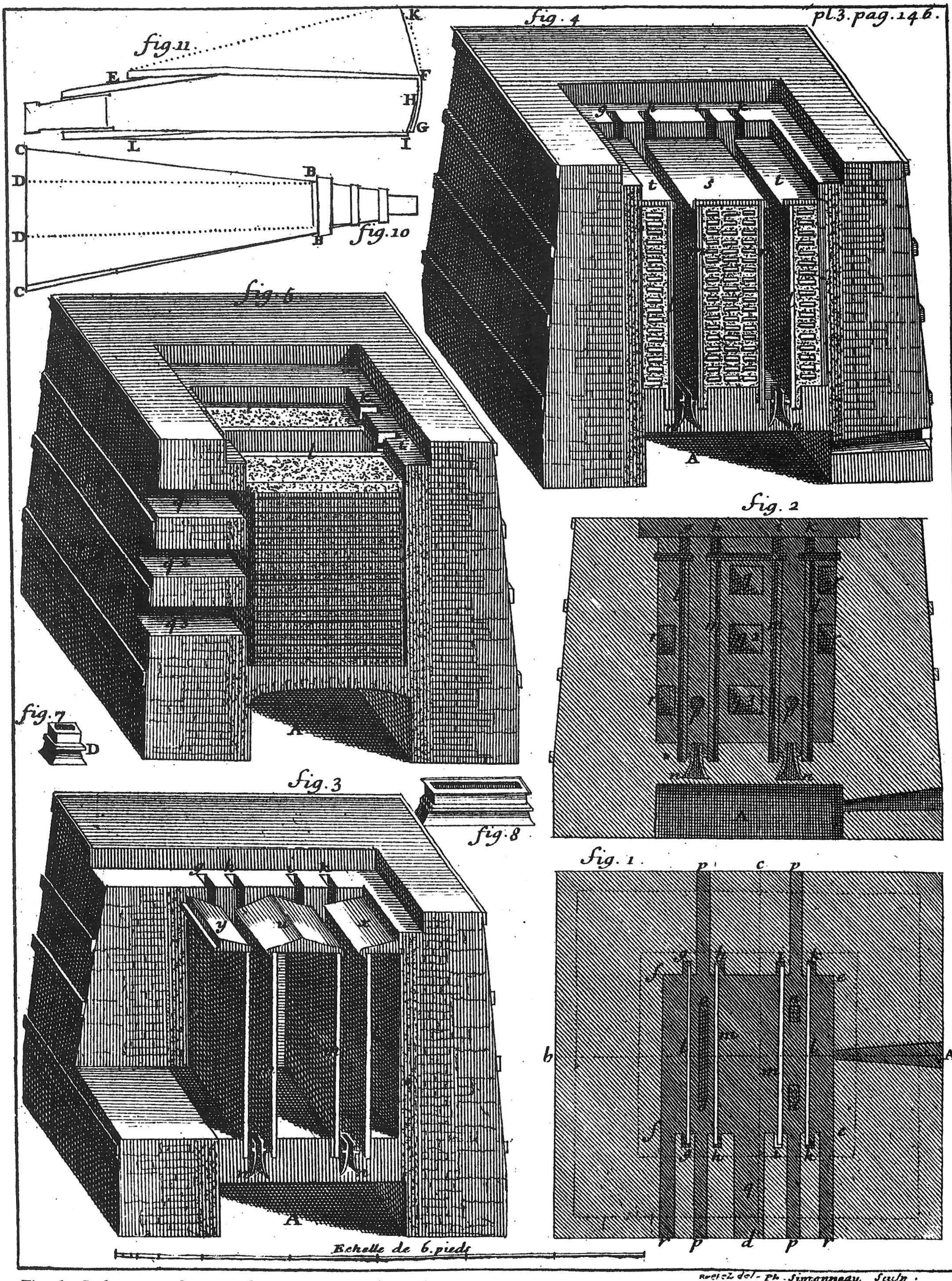

Fig. 1. Laboratory furnace showing section through.

made with identical iron samples. Therefore the specimens had to be cut from the same bar. In those days, bars were short, and, therefore, the samples were small. To carburize them, Réaumur designed small cementation boxes (*Mem. 3*)*. Because Réaumur could neither measure nor control the required temperatures, he had to heat simultaneously several samples at a temperature as uniform as possible. For this purpose, Réaumur developed a special small "laboratory" furnace shown on Fig. 1 (*Mem. 4*).

From his first tests he concluded that:

1—The most efficient carbon sources are charcoal, soot and carbonized leather (*Mem. 1*).
2—Certain salts which alone have no effects can still reinforce the action of ordinary carbon sources (*Mem. 1*).
3.—Fire alone cannot convert iron into steel. (*Mem. 2*).
4—The conversion occurs only when "the iron becomes soft" i.e. above a certain temperature (*Mem. 2*).
5—Some irons give better steels than others (*Mem. 5*).

This last conclusion prompted Réaumur to seek how to identify suitable irons. He found that good indications were given by the aspect of the fracture of a piece of iron, mostly if viewed through a magnifying glass. He proposed seven classes of iron starting with those showing large or thick flakes (1 on Fig. 2), followed by coarse and then fine grains (10 & 11) and finally a fibrous structure (15) (*Mem. 5*). He said that irons showing small grains produced the best steels. About this, R. F. Tylecote wrote: Réaumur, in 1722, seems to have been the first to discuss the properties of ferrous metal in terms of grain structure.[3]

Having observed that the conversion progresses from the outside towards the centre of the sample, Réaumur thought that fine grain irons were better because they contain more voids through which foreign matters could penetrate (*Mem. 6*). In spite of this oversimplfication, Réaumur gave a remarkable description of diffusion, a still unknown phenomena at that time (*Mem. 6 & 7*).

Then Réaumur tried to understand what has been introduced into the iron to make steel. Was it phlogiston as generally professed? Weighing very carefully a piece of iron before and after conversion, he noticed a weight increase of nearly 0.40% and he concluded that this extra weight must have a cause (*Mem. 6*).

The next logical step was to try to determine the reason for this extra weight. This was not easy to answer without chemical analysis and when carbon, as an element, was still unknown. From his earlier experiments, Réaumur had found that it must be sulphurous matters and salts. At that time the name sulphur was given to all matters which burn.[4] Réaumur's explanation was that fire, which comprises only sulphurous matters, penetrates the iron through the dilated voids, leaving some unburnt products. He likened this to the soot deposited inside a chimney that "has been mixed with the flame and nevertheless remains highly combustible". To confirm his views, he showed that though soot is a powerful carbon source, the addition of 1/3 of flowers of sulphur seems to stop its effect (*Mem. 12*).

Thus, through rigorous experimentation, Réaumur got an inkling of the role of carbon in the conversion of iron into steel, a fact confirmed 66 years later by Vandermonde, Bertholet and Monge.[5]

To check his views, Réaumur reconverted steel into iron by packing it with matters able to absorb the same sulphurs and salts which is a modern and very scientific approach (*Mem. 8*).

Quality Control

In his 10th memoir, Réaumur introduced the concept of quality control. He was impressed by the fact that the craftsmen could only assess the quality of different steels by manufacturing and testing tools made with them. Hence, their reluctance to change their source of supply. Réaumur was of the view that testing the quality on small samples was possible and would be very useful to both the tool and the steel makers.

Analysing what makes the difference between good and poor steels, he concluded that three characteristics should be taken into consideration, namely:

—grain structure ("la grainure").
—hardness after quenching.
—ductility ("le corps").

To estimate hardness, Réaumur recommended a test based on the use of standard scratching materials, thus anticipating the scratch test of Mohs in 1822.[6]

To compare ductility, he designed a special device (Fig. 3) to measure how much a quenched piece of wire could be bent before rupture. To make a valid comparison two conditions were necessary:

—The specimens had to have identical dimensions. This was achieved by drawing them through the same die.
—The samples had to be quenched from the same temperature. This was realised by heating them together in a small bath of molten lead, an anticipation of the salt-bath furnaces.

Originality of Réaumur's study

Monsieur de Réaumur was one of the first to undertake a rigorous experimental study of a metallurgical problem, to develop ingenious testing devices and procedures and to present comprehensive reports of his findings. His twelve memoirs cover 382 pages with 10 plates. His experimental setup is shown on Fig. 4.

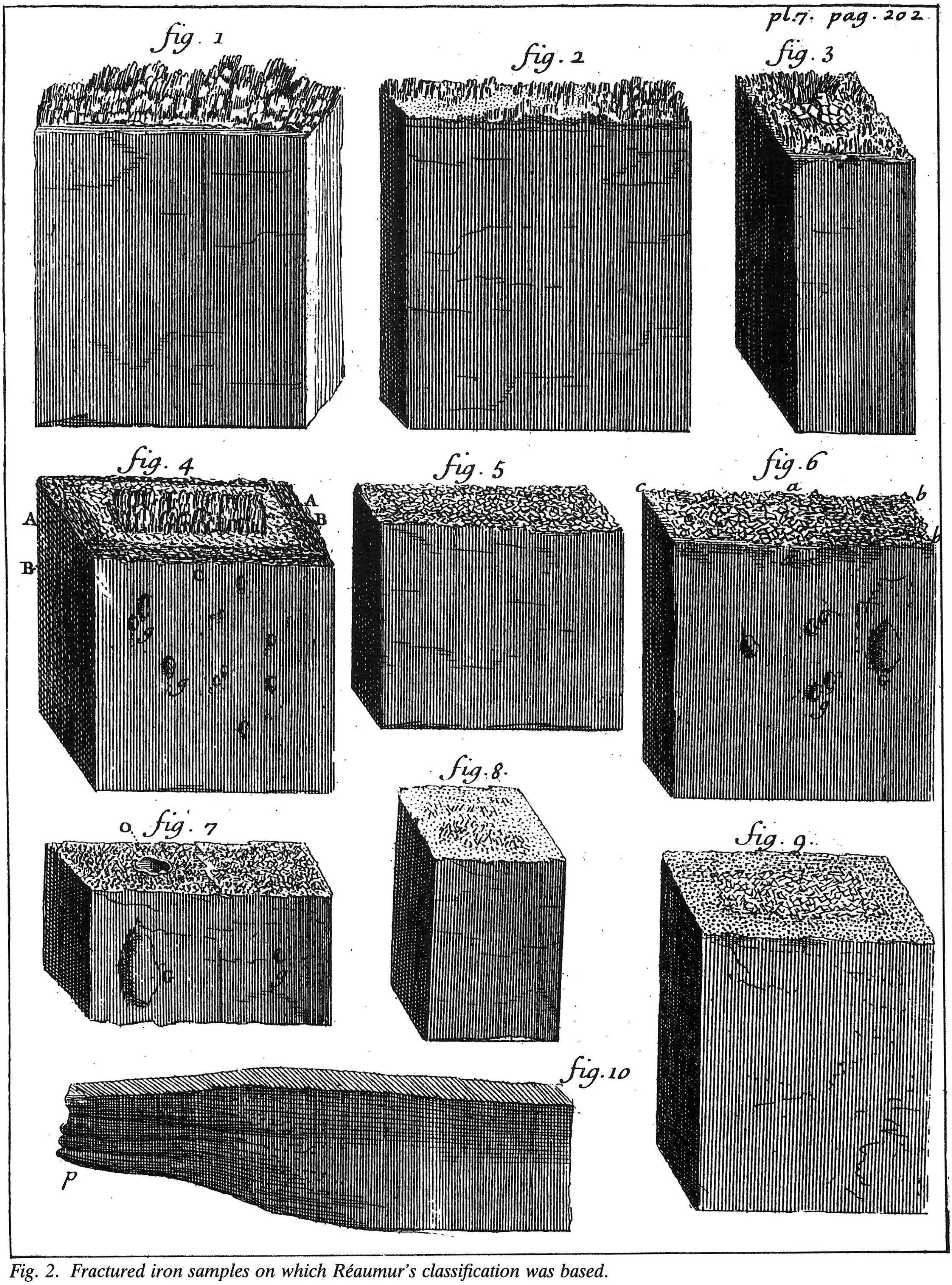

Fig. 2. Fractured iron samples on which Réaumur's classification was based.

If in his Memoirs on the conversion of wrought iron into steel, Réaumur did not invent a new process, he succeeded in making a systematic study of the parameters controlling an existing technology. His objectives were to improve a known method of making steel by scientific monitoring of the process and the introduction of quality control of the raw materials and finished products. By this he innovated and was a pioneer of applied science in the modern sense.

On the other hand, in the six Memoirs on "L'Art d'adoucir le Fer Fondu" Réaumur actually invented the malleabilisation of cast iron by scientific experiments.

Comments on Réaumur's works by some contemporary scientists

During the 18th century, several renowned metallurgists recognized the value of Réaumur's work. Already in 1734, **Swedenborg** in his *De Ferro* reproduced *in extenso* Réaumur's Memoirs on the conversion of iron into steel and on the trompes used in Southern France.[7]

In the first volume of the Encyclopédie, published in 1751, **Diderot** himself wrote the article 'Acier'. In spite of his dislike for Réaumur,[8] he mentioned his work extensively, praising his method and originality.[9]

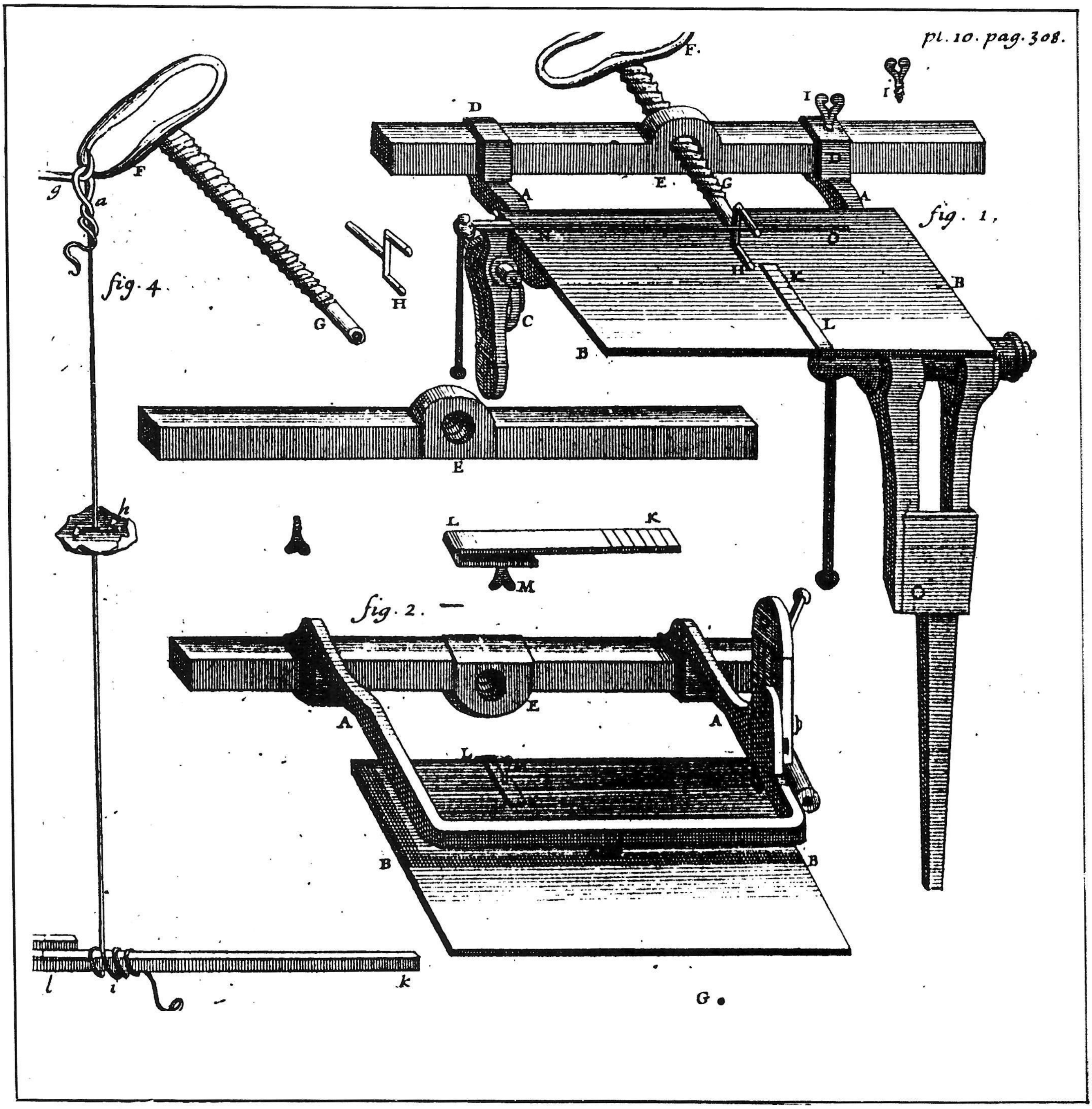

Fig. 3. Laboratory bending test machine.

However, this opinion was not shared by all scientists and in the 6th volume of the same Encyclopédie, in the article 'Fer', the **Baron d'Holbach**, well acquainted with German science, still wrote in 1761 that steel was iron saturated with phlogiston.[10]

Jars (1732–1769) in his *Dissertation sur le Fer et l'Acier*,[11] while admiring the work of Réaumur (who he considered his Master), is surprised that it had no apparent practical impact in France.

In 1761, **Courtivron** and **Bouchu**[12] praised Réaumur's achievements and reproduced in extenso some of his Memoirs.

Even in 1864, when dealing with carburization of bar-iron in Volume II of his *Metallurgy*, **John Percy** praised Réaumur's experiments "of which the records are worthy of being perused even at the present day".[13]

However, Réaumur's ideas were not accepted unanimously. In 1775, **Grignon**, Maître de Forge, was still in favour of the phlogiston theory.[14] He criticized Réaumur, saying that "working on small samples in metallurgy may often take shadow for the truth.[15] Grignon made very accurate descriptions of metallurgical processes. However, when describing the tin plating works at Bain he did not even mention Réaumur who had been working there.[16]

Also in 1775, **du Coudray**[17] considered Réaumur's theories to be completely wrong. Even in 1804, **J. C. Manson** refused the idea that something had to be introduced into the iron to make the conversion,[18] in spite of the fact that the role of carbon had already been established in 1788.[5]

In 1786, **Vandermonde** and **Bertholet** were requested mentioned. de Dietrich wrote that in 1789 the Royal

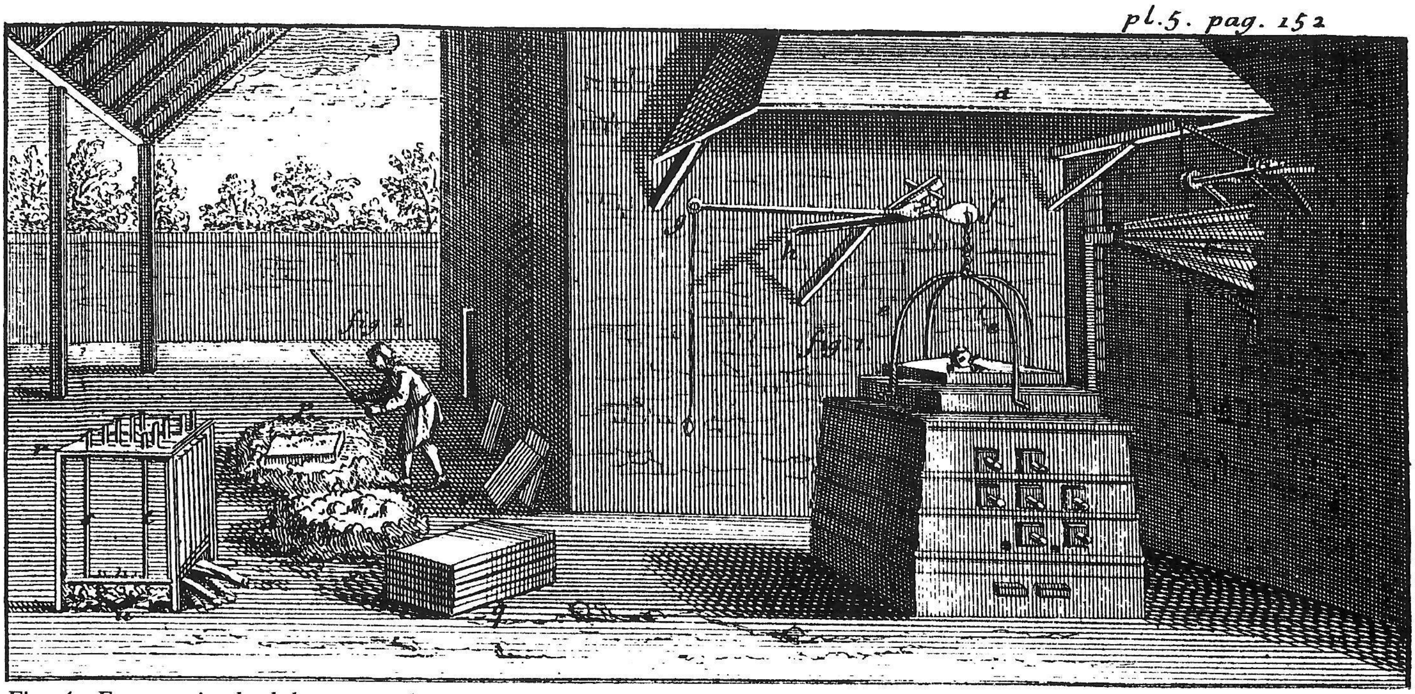

Fig. 4. Furnace in the laboratory showing bellows, peephole and cover lifting device.

by the Académie des Sciences to assess the quality of cemented steel made at Remmelsdorff. They were certainly aware of Réaumur's recommendations on the scientific testing of steel samples. Nevertheless they used the old method of making tools and having them tested by craftsmen.[19]

Between 1786 and his execution in 1793, the **Baron de Dietrich**, Maître de Forge and Maire of Strasbourg, made a survey of the mines and iron mills in the Pyrenees, Alsace and Lorraine, at the request of the government. He was familiar with Réaumur's Memoirs on gold mining[20, 21] and on pickling before tin plating[22]. However Réaumur's Memoirs on iron and steel are not "Manufacture d'Armes Blanches" at Klingenthal were still using German steel[23] and that the very good steel made at La Hutte[24] had to bear the Styrian Mark to be accepted by the trade.

In the early 19th century, full credit was given to Réaumur by **J. H. Hassenfratz**, in his *Siderotechnic*, for his perception of the role of carbon and his classification of irons based on the aspect of fractures.[25] However, by then, what Réaumur had earlier foreseen had been scientifically established by chemical analysis.

Impact on French steel production:

Although Réaumur's Memoirs on iron and steel are mentioned by several authors, some admiring his work, others refuting his conclusions, they appear not to have had a positive impact on the French steel industry.

Apparently Réaumur, a wealthy aristocrat and somewhat of a dilletante, was much more interested in solving a scientific problem and finding his own explanations than in developing a "trivial" industrial application.

In two other instances related to the iron and steel industry, Réaumur established the scientific basis but failed to capture full technological implications. In 1723 he studied with **Trésaguet**, Ingénieur des Ponts et Chaussées, how to improve the manufacture of marine anchors at Cosne.[26] The conclusions of his study do not seem to have been decisive, as as final report was presented by Trésaguet alone in 1737.[27] In 1725, at Beaumont-la-Ferrière (Nivernais), Réaumur made a study of pickling prior to tin plating.[28] He recommended use of diluted cheap acids instead of the usual fermented rye water. This was not implemented because of the difficulty of controlling pickling by diluted oil of vitriol (H_2SO_4).[29]

During the 18th century the French economy was still based mostly on agriculture. Iron mills were erected mainly to increase the returns of the forests. Most of the mill owners were landlords, interested more in the financial aspects of their business than in the technical operation that was usually left in the hands of uneducated foremen working by tradition and reluctant to make any change. There were some exceptions like **Bouchu**, Maître de forge at Vauxheulle, who praised Réaumur's work but did not put his recommendations into practice.

In France the difficulties procuring charcoal were not sufficient to promote new technologies as they were in England. They were, however, such that it was difficult to increase the production of iron and to develop the cementation process that consumes so much charcoal.

The production of low grade steels or "aciers à terre" was sufficient to meet the demand of agriculture. Enough cast and wrought iron was produced to meet the needs of both the military and domestic markets. Iron and steel production was yielding very good profits. The demand for quality steels was restricted to cutting tools, cutlery and "armes blanches". Such steels were very costly and, therefore, used sparingly.[30]

Furthermore two economic factors discouraged any increase in steel production. The rate of interest was high (more than twice what it was in England), and this restricted new investment. Exchange rates were such that imported quality steels were relatively cheap. Therefore their local production was less rewarding than the sale of common iron products.

Probably there was also a strong reluctance from the crasftsmen, who were using steel, to change their sources of supply. They believed strongly that the steel they were using was the main secret of their reputation. To change supplier would have been taking a big risk, and also would have required many technical adjustments. There was no incentive to adopt an indigenous product that was not cheaper than the best imported steel. As already mentioned, the steel mill of La Hutte had to put the Styrian mark on its products to have them accepted by the tool makers.

At the beginning of the 19th century French iron and steel production was still much less than that of England. When demand increased, the cementation process had been superseded by more efficient methods imported from England.

Summary

In 1722, the Académie des Sciences published Réaumur's experiments on the cementation of wrought iron. He reached remarkable conclusions through a rigorous approach and cleverly designed experiments. Although the process was not new, Réaumur was the first to introduce systematic testing on small samples, to design the specific "laboratory" equipment required, to make metallographic studies based on grain structure and to develop a method to compare steel properties using small samples.

His work was praised by several scientists of the 18th century: Swedenborg, Diderot, Jars, Courtivron, Bouchu. However, other authors, more practitioners than scientists, were not accepting his views and remained strongly in favour of the phlogiston theory: Baron d'Holbach, Grignon, du Coudray, Manson.

Réaumur's work had practically no impact on the manufacture of steel in France. This for several reasons:

Réaumur was more interested in finding explanations to natural phenomena than in practical applications.

The Maîtres de Forge had no incentives to produce quality steels that were in very small demand and, owing to the relative cheapness of imported products, would not increase their already large profits.

The crasftsmen using quality steels firmly believed that their reputation was dependent mainly on the steel they were purchasing, and, therefore, were very reluctant to change suppliers.

When the French demand for steel and the need for indigenous production increased, the cementation process was obsolete, with the exception of some very special product.

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*–Monsieur de Réaumur: *L'Art de convertir le Fer en Acier* (Paris 1722).
Also published in translation as: A. G. Sisco & C. S. Smith: *Réaumur's memoirs on steel and iron.* (Chicago 1956).
Citations thus: (*Mem.'s*) refer to the Memoir dealing with the subject. All figures are reproduced from the Memoirs.

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