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Dossier

Experimental research of crucible steel: a new insight and historical reflection

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Résumés

English Français

This paper describes experimental archaeo-metallurgical research on crucible steel, executed as a master project to obtain the master degree in conservation. After evaluating the results of different manufacturing techniques, the so-called Georgian crucible steel technique showed results that were remarkably similar to archaeological evidence from Merv, Turkmenistan. An objective historical reflection was made.

Cet article décrit une étude expérimentale archéo-métallurgique sur l'acier creuset, menée en tant que projet maîtrise pour l'obtention du grade de master en conservation et restauration. Après avoir évalué les résultats des techniques différentes de fabrication, la technique de l'acier au creuset géorgien a montré des résultats similaires aux preuves archéologiques de Merv, au Turkménistan. Une réflexion historique a été menée à ce sujet.

Entrées d'index

Mots-clés : archéologie, métallurgie, archéo-métallurgie expérimentale, acier au creuset, damas, métallography
Keywords : archeology, metallurgy, experimental archaeo-metallurgy, crucible steel, wootz, metallography

Notes de la rédaction

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Texte intégral

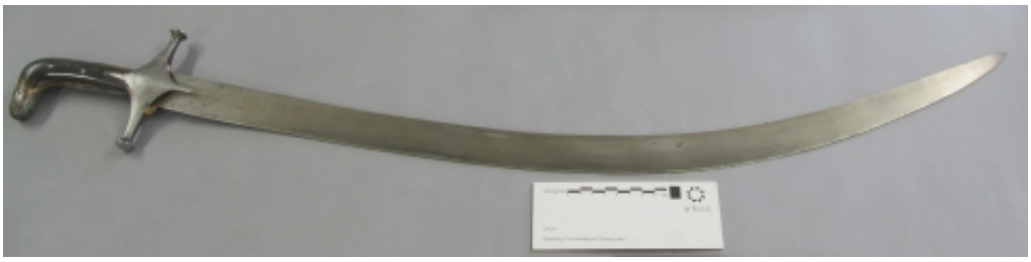
The author would like to thank Patrick Storme, professor and head of the Conservation and Restoration of Metals department at the Artesis university college; Seerp Visser; Dirk Anthierens; Gotscha Lagidse and Zaqro Nonikashvili for their valuable comments, ideas, practical knowledge and help in the research summarised here.

Introduction

1 Iron has played a very important role in the evolution of men since it was used for the first time. The development of metallurgical science and the knowledge our ancestors had about iron and steel were crucial for the rise and fall of nations. Among the most famous and intriguing historical sorts of this material, the Damask or Damascus steel is the best known. Damascus steel got famous through its features which were believed to be close to magical. The modern, more commonly accepted name for this steel is crucible steel, as the steel was molten in relatively small crucibles before it was forged into objects of the highest quality.

2 In short, crucible steel is hard and yet flexible and has a specific appearance. Its physics can be explained by its specific microstructures, which are described more in detail in 2.1 of this article. The appearance is characterized by a pattern that is formed on the surface of the polished and etched steel and is made up by these microstructures. Ill.2, *Damask of the crucible steel sabre* of ill.1 shows a typical pattern of crucible steel on the surface of the sabre.

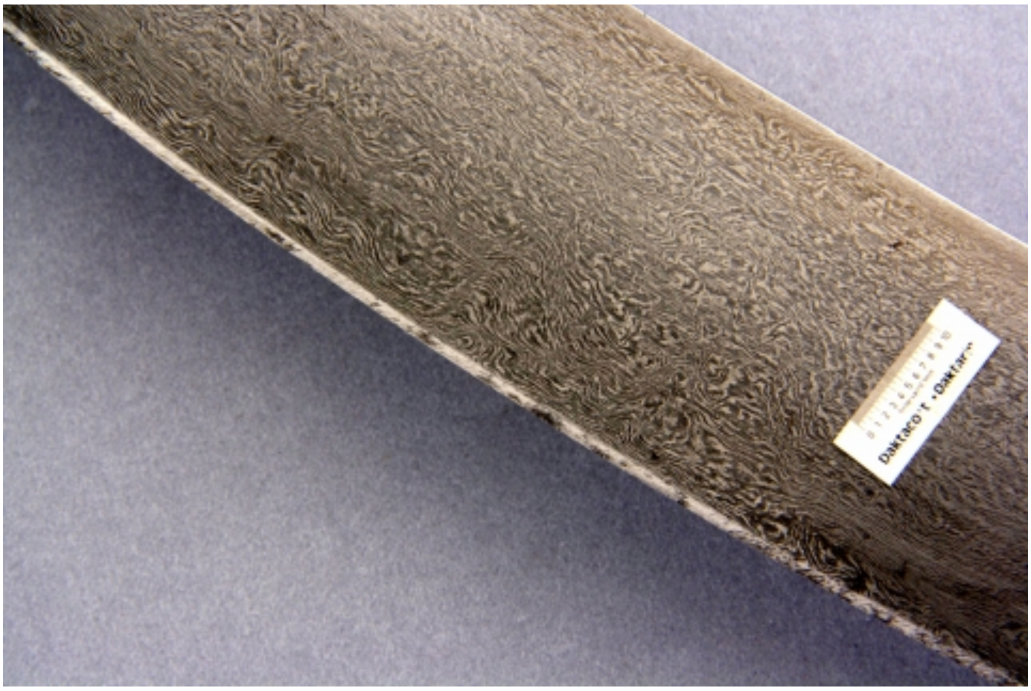
Fig. 1 Sabre from the collection of KLM-MRA made from crucible steel



The picture shows a Kozakian Shamshir, a typical weapon made of crucible steel from the collection of the KLM-MRA of Brussels.

Credits : Klaas Remmen

Fig. 2 Damask on the crucible steel



The wavy pattern on the polished and etched surface of the sabre shown in ill.1. This pattern is made out of groups of cementite (white lines) and the steel matrix (black lines)

Credits : Klaas Remmen

3 The history of crucible steel is long and interesting and is covered by a vast body of literature. It is commonly accepted that this legendary steel originates from India (Verhoeven, 2003) or Pakistan (TR Anantharamu, 1999) where it was first produced around the beginning of our era. In later times, it was also produced in some areas in Central Asia such as present Turkmenistan and Uzbekistan. It is commonly believed that the technique of making crucible steel was lost somewhere in the 20th century (Fedosov, 2007). According to the authors knowledge, the first persons who successfully recreated the steel after its technique was believed to be entirely lost, would have lived in the former USSR: V.I. Basov from Russia (Fedosov, 2007) and Prof. dr. Badri Amaglobeli from Georgia (Amaglobeli, 1984). The latter wrote a very detailed, but in western countries unknown PhD on the subject in 1984. Just a short while later dr. John Verhoeven and blacksmith Alfred Pendray, probably unaware of the research in the USSR did their own research on the subject, with great success (Verhoeven and Pendray, 1992; J.D. Verhoeven, 1996; J.D. Verhoeven, 1998).

4 Despite this body of evidence, there still remain a lot of questions. For example, there are uncertainties about how and to what extend production steps effect the wavy pattern on the etched steel[1], and there are deficient explanations for some archaeological findings. Some theories would be much more credible with strong empirical evidence. If we could, for example, get a better insight in the physics of pattern formation this could be beneficial for the determination of artefacts, since the type of pattern was a common way to judge the quality of crucible steel products (Panseri, 1965). This information could be of great value for historians and conservators.

5 By intensively studying the historical evidence of crucible steel, the conclusion could be made that a lot of information on the original production methods was either false, incomplete or unreliable. Certain facts we know today about production techniques originate mainly from a few historical accounts, made by 19th century travellers like Buchanan (Buchanan, 1829), Percy (Percy, 1864) and Voysey (Voysey, 1832), and by the enormous amount of research on the subject that was made by the western scientists

John Verhoeven and Alfred Pendray (J.D. Verhoeven, 1998; J.D. Verhoeven, 2001; John Verhoeven, 1998; Verhoeven, 2001; Verhoeven and Pendray, 1992). The former mentioned travellers all describe the processes how the steel was made in India, and to the authors knowledge, there is but one personal description of manufacturing methods used in Central Asia, that of Masalaski in 1841 (Khorasani, 2006). Other information is found in widespread metallographic (Piaskowski, 1978; Schastlivtsev, Gerasimov and Rodionov, 2008; Alan Williams, 2007; Williams, 2007) and archaeological (TR Anantharamu, 1999; Rehren and Papachristou, 2003; Srinivasan, 1994) studies.

6 The presented study explores some crucial steps in of the manufacturing process of crucible steel in depth. For this archaeo-metallurgic research historical, observational and experimental hands-on techniques were used.

Experimental archaeo-metallurgic research

7 In experimental archaeology, archaeo-metallurgic research is a relatively new discipline. It typically uses experiments to answer a research question. By conducting experiments, new information on ways our ancestors must have lived, worked and thought can be determined. It is important to stress that these techniques often do not give actual proof or hard data for a certain theory, but they can provide new insights. In other words, experiments may give a reflection on historical accounts.

8 Among the academic institutions using experimental archaeo-metallurgic techniques are the University of Hull and Exeter University in Great Britain, Universidad Autonoma de Madrid in Spain and the Royal University of Groningen in The Netherlands.

Crucible steel and experimental research

9 Steel in its purest form is nothing more than a combination of iron and carbon, and is therefore called a 'carbonsteel'. Carbon has a special effect on the hard- and toughness of steel and, in general we could state that the more carbon, the harder (and more brittle) the steel, up until the limit at 6,76% carbon. Beyond this point, the alloy would crack and pulverize; it has no more consistency (Budinsky and Budinsky, 2005). Crucible steel is a remarkably pure high-carbon steel with a percentage of 1-2% of carbon by weight, and was preferably used for highly valued weapons, armour and tools. On ill.1: Sabre from the collection of the KLM-MRA made from crucible steel (Klaas Remmen) shows a 'Shamshir', a weapon that was often made from crucible steel. This typical hypereutectoid composition is able to form specific microstructures, which are responsible for both the outstanding cutting abilities of the steel, as well as the aesthetic appearance of the etched surface.

10 The high carbon content forms very hard and brittle structures in steel, called cementite. The high carbon content in crucible steel gives way to the formation of a lot of cementite, which would not be so useful for edged weapons or tools, because the edge would chip off when a force was applied. By using complicated forging cycles, the ancient blacksmiths were able to get the hard cementite structures to ball up and group in lines in the finished product, while the matrix of softer steel around this hard cementite became tougher. The finished product was tough and able to resist blows, while the aligned cementite particles acted like a micro saw on the cutting edge of the

object. The groups of cementite particles, visible on the polished and etched surface of ancient crucible steel objects make up white, meandering lines. These lines are sometimes called the 'Damask' of the steel (J.D. Verhoeven, 2001) and are illustrated on ill.2 Damask of the crucible steel sabre of ill.1.

11 By starting off a series of experiments in which crucible steel was made using different techniques, different steps and aspects were studied. One of the techniques was the so-called 'Georgian' crucible steel technique. This technique was developed by dr. Zaqro Nonikashvili, a Georgian mastersmith who has been experimenting with crucible steel for over a decade. In November 2010 he kindly showed his technique on a crucible steel symposium which was held in Antwerp, Belgium. Like the other known mechanisms to obtain crucible steel, this technique uses low carbon iron and a carbon source to unite under pyrochemical conditions and form steel in a closed crucible. The way the Georgian technique works is nevertheless distinctly different than other known crucible steel productions. This paper presents the technique and makes some historical reflections on archaeological finds. It is followed by a description of the methodology, which was used in the experimental research.

Georgian crucible steel technique

12 In the Georgian crucible steel technique a certain amount of low carbon iron is divided in two equal parts. In the experiments pure iron was used. One half of the iron pieces was put into a clay crucible, and covered with sand or glass with an average melting point of around 1200°C. On this, a layer of charcoal is added. The other half of the iron is now also charged into the crucible, in alternating layers of iron and charcoal, where the final or top layer had to be charcoal. The crucible is then sealed with a lid that had a small hole in the middle. The way of charging the crucible is visible ill.3: Section of crucible and its charge (Seerp Visser). The black parts on the illustration represent the charcoal, the red parts represent the iron parts, while the green parts show the glass or sand.

Fig. 3 Section of crucible and its charge

The scheme shows the cross section of the crucible charge of the Georgian technique. the black parts represent charcoal, the red parts iron and the green part sand or glass.

Credits : Seerp Visser

13 When the crucible is fired in a coal or gas furnace, the temperatures in the crucible reaches 1200°C, and the sand or glass starts to melt and forms a sticky mass on top of the lowest half of the iron charge. This sticky mass, essentially just molten glass, protects the bottom half of the iron from getting carburised by the charcoal above. The

upper half of the iron charge, above the glass, starts to pick up carbon from the surrounding charcoal faster as the temperature rises, according to Fick's law (Ashby, Shercliff and Cebon, 2007).

14 During the process, the iron in the crucible picks up more and more carbon and the overall temperature rises up to +-1500°C. Eventually the original low carbon iron pieces will become a high carbon alloy in the cast iron range. Because the melting point of the iron-carbon alloy drops as the alloy consists of more carbon, these pieces of cast iron will melt. Once molten, the iron alloy starts to drip down through the pieces of charcoal and glass and further down to the bottom of the crucible. Unlike the lighter charcoal, this molten cast iron is able to go through the 'filter' of molten glass and sets around the lower part of the iron charge. Because of the high temperature, the bottom part of the charge now starts to pick up carbon from the molten cast iron surrounding it, and will eventually melt itself.

15 After an hour and a half this firing process is complete and all of the upper part of the iron charge is located at the bottom of the crucible. The crucible charge now consists of a molten charge of steel at the bottom of the crucible, on top of which there is a layer of molten glass, which still acts as a cover protecting the molten steel from reacting with the leftover charcoal. After cooling down, the steel forms the typical crucible steel structures, and consists of a hypereutectoid carbon percentage above 0,8% in weight. On ill.4: Cross-section of crucible steel after cooling (Klaas Remmen)

Fig. 4 Crossection of crucible after cooling

The picture shows a crucible after firing. The different parts are visible, steel on the bottom, a layer of dark blackish glass and pieces of charcoal on top.

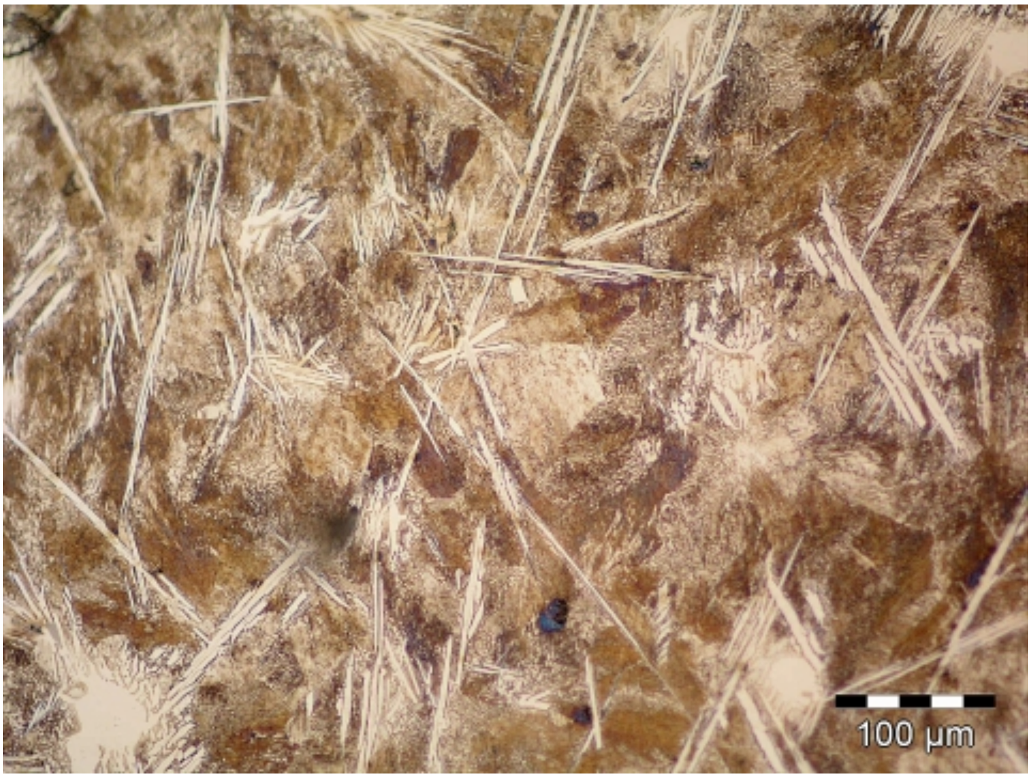
Credits: Klaas Remmen

Prills and metallographic studies

16 After the initial experiments, Nonikashvili's technique was found to be relatively easy in use. The metallurgical residue from the steel manufacturing consisted out of five different parts; leftovers from the crucible, leftover charcoal, a solid steel ingot and a slag layer, in which tiny droplets of an iron alloy were present. The crucible and charcoal were discarded, as the focus mainly lied on the other parts. The obtained steel ingot and the iron prills were examined for structures and consistency with metallography.

17 The specimens were cut using a cooled diamond saw and embedded in a common mounting resin. They were polished down to a 1µm grade using standard metallographic techniques, followed by etching with Nital. The samples from ingots distinctively showed a hypereutectoid composition with a pearlite matrix and needle-formed Widmanstätten cementite, as seen on ill.5: Structure of raw crucible steel.

Fig. 5 Structure of raw crucible steel



Structure of raw crucible steel. The white lines are grainboundry cementite and needle like Widmanstätten cementite. The fine fingerprint-like zones are pearlite grains.

Credits: Klaas Remmen

18 These microstructures are typical for raw crucible steel, and can easily be compared with metallographic studies of ancient crucible steel (M L Wayman, 1999), as seen ill.6: SEM image showing ancient Sri Lankan crucible steel (M L Wayman).

Fig. 6 SEM photograph from ancient sri lankan crucible steel

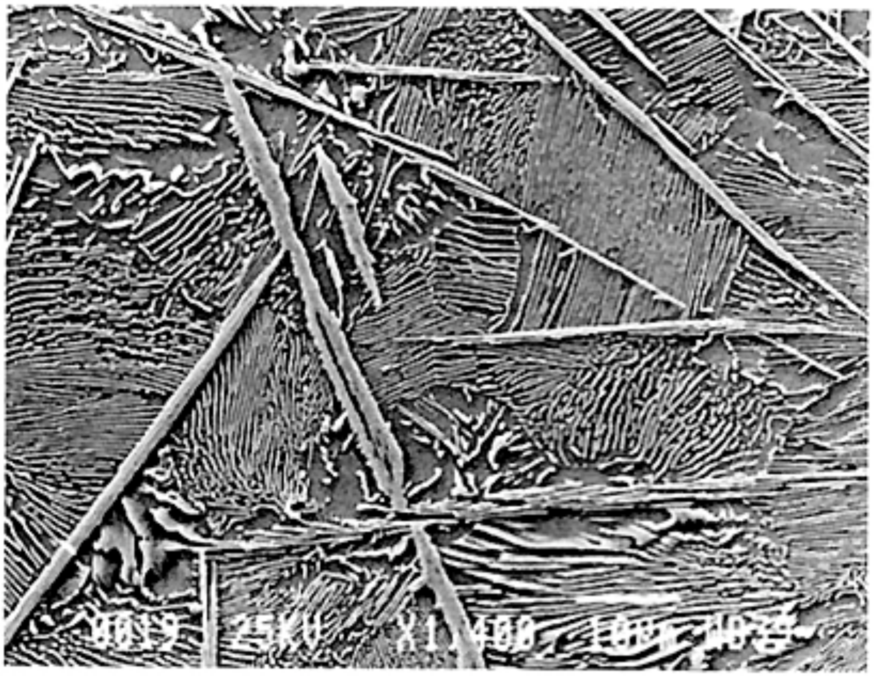


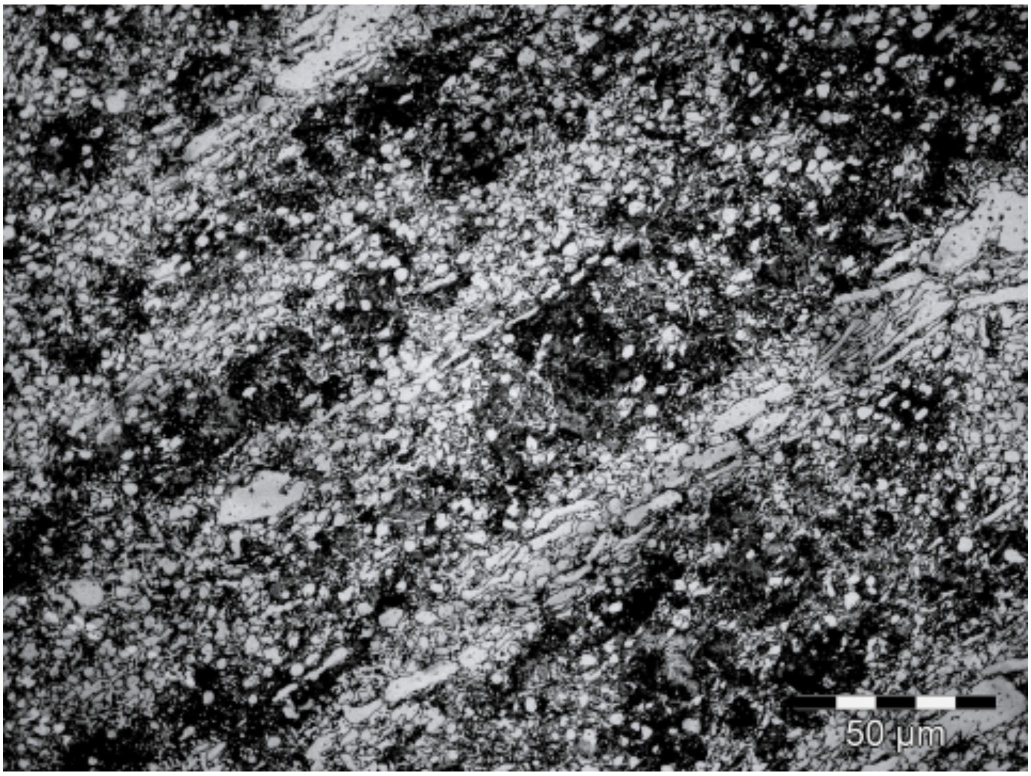
Figure 8: Ingot A hypereutectoid region, Widmanstatten cementite in pearlite. SEM micrograph. Nital etch. Magnification 1050x.

Structure of ancient Sri Lankan crucible steel. The microstructure is very similar to that of ill.5, and is consisting out of Widmanstätten cementite in a pearlite matrix. (G. J. M L Wayman, 'Crucible steelmaking in Sri Lanka', Historical Metallurgy 33 (1999), 26-42.)

Credits: M. L. Wayman

19 After forging some of the specimens with this structure, the steel showed a banding of cementite, (as seen on Fig. 7) which is responsible for the white markings or Damask on the etched surface (as seen on Fig. 8).

Fig. 7 Banding of cementite in forged crucible steel



Structure of forged crucible steel after minor reductions. The cementite is broken up into smaller particles and starts to "ball up" to round edged pieces and small spherical structures. These cementite particles are beginning to group into lines.

Credits: Klaas Remmen

Fig. 8 Polished and etched surface of a forged piece of crucible steel

Polished and etched surface of forged crucible steel. The groups of balled up cementite make out the white spots and lines.

Credits: Klaas Remmen

20 On top of the solidified ingot, there is systematically a layer of glass found in the crucible, which was deliberately added while loading the materials. This glass layer often sticks to the ingot, while the upper part is covered by ash and pieces of charcoal that were not consumed by neither the air in the crucible nor was it used for carburising the steel. Also, in every experiment using dr. Nonikashvili's technique, balled up prills of iron trapped in the glass were found, as seen on Fig. 9.

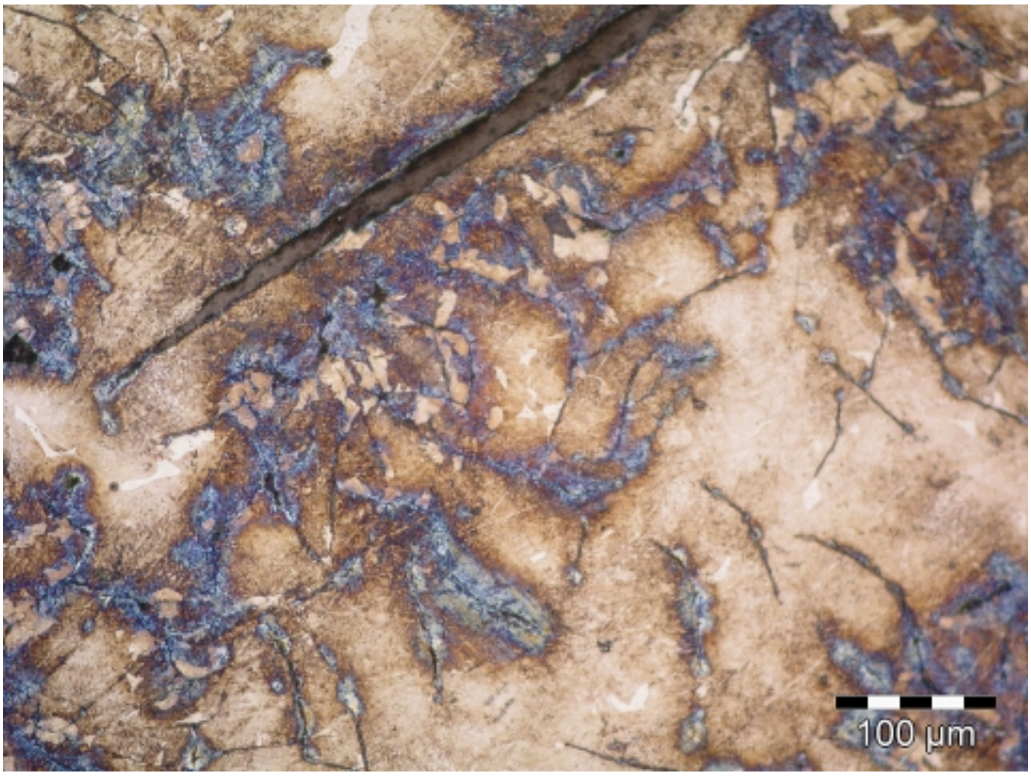
Fig. 9 A piece of slag with iron alloy prills cought in the slag

Close up of a piece of slag of the Georgian technique. The slag contains multiple prills of iron alloys, with a high carbon content. Please note that the corrosion of the prills happened after their discovery.

Credits: Klaas Remmen

21 These droplets measured 0,1 to 5 mm in cross-section, and were situated throughout and on top of the slag layer. Prills from different parts in the glass layer were studied with metallography. The structure of these prills showed to be high carbon iron alloy, with a carbon percentage well into the area of cast iron. The structure of a prill is seen on ill.10: Structure of a iron alloy prill found in the slag (Klaas Remmen) showing grey graphite lamellae, typical for a cast iron structure.

Figure 10 Structure of an iron alloy prill found in the slag



Micro structure of a prill found in the slag layer. The big black line is a piece of graphite and is essentially pure carbon. The smaller black curvy lines are also graphite. The white blocky structures are cementite formations. The matrix is hard to resolve at this magnification.

Credits: Klaas Remmen

Discussion

22 The results of the experiments, following Nonikashvili's technique, showed to be particularly interesting. A remarkable resemblance with the archaeological findings from an ancient crucible steel production site in Merv, Turkmenistan (Feuerbach, 2002) is noticed. An objective comparison is made, in which arguments suggest it is regarded possible that metallurgists in ancient Merv could have used a technique similar to Nonikashvili's. The comparison is made with the data provided by Feuerbach and accessible in her Phd: 'Crucible steel in Central Asia: production, use, and origins' (University College London, 2002).

The prills

23 Excavation evidence shows that the crucibles in Merv were particularly large and had a relatively thin layer of glass on top of the solidified ingot. The produced steel showed a hypereutectoid microstructure. Moreover, the remnants from the workshop showed that the glass slag that was found on top of the ingots, also contained small prills of a iron-carbon alloy with a high percentage of carbon, having a structure range between hypereutectoid iron and cast iron.

24 Given the very few original first-hand descriptions available on the manufacturing of crucible steel, especially in the Central-Asian region, it is uncertain which technique might have been used to obtain the crucible steel in this region. The ancient metallurgists must have used iron with a relatively low percentage of carbon, together

with some source of carbon to be fused in the sealed crucible. According to dr. A. M. Feuerbach it is highly doubtful that, unlike in other known techniques to produce crucible steel, cast iron with a high carbon level would have been used as the carbon source. As a hypothesis for the trapped iron, Feuerbach describes the prills, found in the slag layer were possibly getting trapped in the glass layer during the process after being slung from of the molten metal due “the CO boiling of steel”. This cooking is a well known and described reaction which occurs when steel solidifies and is less able to bind with oxygen. As the oxygen binds with carbon upon cooling it forms CO bubbles which make the molten steel boil (Verhoeven, 2007). Pieces of steel got trapped in the slag layer, as well as to the crucible walls. Many scientists and crucible steel enthusiasts follow this theory.

25 If however, these prills were originating from the liquid crucible steel, they would have to increase their carbon level to get the cast iron structure, after they were slung out of the liquid mass. This means the atmosphere inside the crucible would had to be of a reducing kind, while the slag layer is said to be added to prevent the liquid steel from the oxidising. Moreover, in any other known crucible steel method, the amount of carbon charged into the crucible in whatever form, is calculated to be entirely consumed by the steel. Theoretically there should be little carbon left for the prills to react with when they were shot into this part of the crucible. On the other hand, the archaeological prills are very small, and would not need that much carbon to transform from high carbon steel to cast iron. The prills caught in the slag would not have been able to react with the atmosphere above altogether.

26 The prills that were found in the slag layer of the experiments probably had a different origin. Logically, it is assumed that these prills originate from the upper part of the crucible charge and were not able to travel through the molten glass before the process was ended and completely solidified.

The slag

27 The slag, found on excavations showed to be very similar to slag from nearby iron smelting residues. This suggest that the iron, used to produce the crucible steel was obtained in the nearby smelting area, and contained the slag as a contamination which got separated during the process. Another answer could be found in the deliberately adding of the slag in the crucible to act as a flux, or maybe as a filter in an self-containing process as described above. Slag is regularly tapped from the shaft furnaces in use at that time. The recycling of shaft furnace slag would be beneficial as it is dense and would take up less space in the crucible when added, unlike the use of sand. Furthermore, the melting point of shaft furnace slag is in the right range to act as the described filter, as it was tapped from the furnace when the iron inside was in a semi-molten state, at about 1200-1250°C (Ouden, 1988).

Simplicity

28 All known techniques to make crucible steel need calculations or a empirical knowledge of quantities to get the carbon level of the desired ingot in the right range. It is only a difference of a few weight percentages of carbon which make up the difference between high quality crucible steel and unworkable cast iron (2% carbon or more) (Budinsky and Budinsky, 2005). Dr. Nonikashvili’s self-regulating system is rather simple in use and does not need accurate calculations for the carbon levels to be

effective. Executed in the right way, there is only a small chance of ending up with too much carbon in the structures.

Conclusion

29 The experimental results show a simple technique for making crucible steel which is suitable to obtain the highly prised material. The remains of the metallurgical activities from the experiments match the archaeological finds in Merv very well. This observation gives the impression that a similar self-regulated technique might have been used in ancient Merv.

30 Since the presented research was made as a part of a one-year masterproject, and started of as a pilot-project, there was not enough time nor recourses to make more comparison between the different techniques. For instance we know that also types of crucible charges and firing techniques play a key role process, and have a great effect on the qualities (as well physically as aesthetically) of the material. Future research can surely provide more solid facts on the different theories.

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Notes

1 This pattern is called the 'Damask' and is described more in detail later in this paper

Table des illustrations

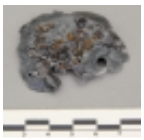
	Titre	Fig. 1 Sabre from the collection of KLM-MRA made from crucible steel
	Légende	The picture shows a Kozakian Shamshir, a typical weapon made of crucible steel from the collection of the KLM-MRA of Brussels.
	**Crédits**	Credits : Klaas Remmen
	URL	http://ceroart.revues.org/docannexe/image/2557/img-1.jpg
	Fichier	image/jpeg, 772k
	Titre	Fig. 2 Damask on the crucible steel
	**Légende**	The wavy pattern on the polished and etched surface of the sabre shown in ill.1. This pattern is made out of groups of cementite (white lines) and the steel matrix (black lines)
	Crédits	Credits : Klaas Remmen
	URL	http://ceroart.revues.org/docannexe/image/2557/img-2.jpg
	Fichier	image/jpeg, 1,4M
	Titre	Fig. 3 Section of crucible and its charge
	**Légende**	The scheme shows the cross section of the crucible charge of the Georgian technique. the black parts represent charcoal, the red parts iron and the green part sand or glass.
	Crédits	Credits : Seerp Visser
	URL	http://ceroart.revues.org/docannexe/image/2557/img-3.jpg
	Fichier	image/jpeg, 36k
	Titre	Fig. 4 Crossection of crucible after cooling
	**Légende**	The picture shows a crucible after firing. The different parts are visible, steel on the bottom, a layer of dark blackish glass and pieces of charcoal on top.
	Crédits	Credits: Klaas Remmen
	URL	http://ceroart.revues.org/docannexe/image/2557/img-4.jpg
	Fichier	image/jpeg, 772k

Titre	Fig. 5 Structure of raw crucible steel
Légende	Structure of raw crucible steel. The white lines are grainboundry cementite and needle like Widmanstätten cementite. The fine fingerprint-like zones are pearlite grains.
Crédits	Credits: Klaas Remmen
URL	http://ceroart.revues.org/docannexe/image/2557/img-5.jpg
Fichier	image/jpeg, 1,6M

Titre	Fig. 6 SEM photograph from ancient sri lankan crucible steel
Légende	Structure of ancient Sri Lankan crucible steel. The microstructure is very similar to that of ill.5, and is consisting out of Widmanstätten cementite in a pearlite matrix. (G. J. M L Wayman, 'Crucible steelmaking in Sri Lanka', Historical Metallurgy 33 (1999), 26-42.)
Crédits	Credits: M. L. Wayman
URL	http://ceroart.revues.org/docannexe/image/2557/img-6.jpg
Fichier	image/jpeg, 192k

Titre	Fig. 7 Banding of cementite in forged crucible steel
Légende	Structure of forged crucible steel after minor reductions. The cementite is broken up into smaller particles and starts to "ball up" to round edged pieces and small spherical structures. These cementite particles are beginning to group into lines.
Crédits	Credits: Klaas Remmen
URL	http://ceroart.revues.org/docannexe/image/2557/img-7.jpg
Fichier	image/jpeg, 1,8M

Titre	Fig. 8 Polished and etched surface of a forged piece of crucible steel
Légende	Polished and etched surface of forged crucible steel. The groups of balled up cementite make out the white spots and lines.
Crédits	Credits: Klaas Remmen
URL	http://ceroart.revues.org/docannexe/image/2557/img-8.jpg
Fichier	image/jpeg, 876k



Titre	Fig. 9 A piece of slag with iron alloy prills cought in the slag
Légende	Close up of a piece of slag of the Georgian technique. The slag contains multiple prills of iron alloys, with a high carbon content. Please note that the corrosion of the prills happened after their discovery.
Crédits	Credits: Klaas Remmen
URL	http://ceroart.revues.org/docannexe/image/2557/img-9.jpg
Fichier	image/jpeg, 1,8M

Titre	Figure 10 Structure of an iron alloy prill found in the slag
Légende	Micro structure of a prill found in the slag layer. The big black line is a piece of graphite and is essentially pure carbon. The smaller black curvy lines are also graphite. The white blocky structures are cementite formations. The matrix is hard to resolve at this magnification.
Crédits	Credits: Klaas Remmen
URL	http://ceroart.revues.org/docannexe/image/2557/img-10.jpg
Fichier	image/jpeg, 1,1M

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Droits d'auteur