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Ultrahigh-carbon "wootz" from crucible carburization of molten iron: Hypereutectoid steel from "Tamil Nadu Process" at Mel-siruvalur

Sharada Srinivasan

School of Humanities, National Institute of Advanced Studies, Bengaluru, India

ABSTRACT

As European and Mediterranean accounts indicate, India has been famed for the production of steel, apparently made by crucible processes. Late medieval traveler's accounts record the making of "wootz" steel in several places in southern India. This material was used for the fabled Damascus swords, which were later found to be of ultrahigh-carbon steel. Whereas studies on Asian crucible steel making from India, Central Asia and Sri Lanka have discussed various processes ranging from co-fusion of cast iron and wrought iron to solid-state carburization of wrought iron, it has been difficult to find clear evidence relating to an end product of ultrahigh-carbon steel. In this light, the archeometallurgical evidence from Mel-siruvalur in Tamil Nadu, presented in this paper, is significant in that it shows unmistakable remnants in crucibles of ultrahigh-carbon, hyper-eutectoid steel, with a likely production mechanism of molten carburization of wrought iron to steel. The favorable comparison with ultrahigh-carbon steel finds dated to early historic or megalithic times in Tamil Nadu and southern India also suggest that this method of crucible steel manufacture, which may be described as the "Tamil Nadu process", might have been earlier or more archaic than the co-fusion process.

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Introduction

There has been considerable recent interest in the ancient production of high-carbon iron alloys and steel with new evidence from different parts of Asia. For its part, India has been famed for a traditional crucible steel in literary and historical accounts right from Greek and Roman times. This industry flourished at any rate by the late medieval period in southern India, as testified by numerous European descriptions of "wootz" crucible steel. "Wootz" corrupted from "ukku", the south Indian word for steel, may derive from the Tamil word "uruku", implying melting in a container [1–3]. In Europe, interest in south Indian "wootz" steel intensified in the 17th–18th centuries, especially since it was linked to the manufacture of the fabled Oriental "Damascus" swords, reputed to cut gauze kerchiefs, first encountered by the Europeans in the Near East during the Crusades, but also known from the Indian subcontinent. "Wootz" steel was also the subject of much scientific scrutiny in the 19th century and the early 20th century, and in its own way spurred developments in modern metallurgy with no less than Michael Faraday, inventor of electricity, being involved in numerous experiments to understand its alloying constituents [4, 5]. It was eventually found that "wootz" was a high-carbon steel with over 1% C, a novelty in Europe where high-carbon steels were not previously known. Figure 1 is a photograph of a wootz specimen from Science Museum, London, showing what appear to be radial casting fins at the top of the ingot from solidification from the molten state.

As for the antiquity of Indian crucible steel processes, three sword blades with 1.2–1.7% carbon were reported in the 1950s from Taxila in the northwestern part of the Indian subcontinent [6]. The Greek physician Ctesias of the late 5th–early 4th century BCE is said to have mentioned the wonderful swords of Indian steel presented to the King of Persia [7]. There is also an account by the Egyptian alchemist Zosimus of the 2rd c. CE to the effect that Indian iron was melted in crucibles to make swords [8]. The Roman account of the Periplus of the Erythrean Sea mentions imported Indian iron, steel and cotton cloth in the district of Ariaca [9].

There are several lucid accounts from different parts of southern India by European travelers of steelmaking practices by crucible processes in the 18th–19th centuries, such as of Voysey [10] from the Nizam's dominions corresponding to the modern state of Telangana, and of Buchanan-Hamilton [11], who traveled in the provinces of Mysore after the fall of Tipu Sultan.

However, the accounts of various travelers and scholars suggest that there is still much to be understood in terms of the mechanisms and the exact nature of the final product formed from south Indian wootz crucible processes, whether it was necessarily a high-carbon steel or a general steel of a homogenous composition, which might include even white cast iron. There is a bewildering array of late medieval descriptions by travelers, throwing up the possibilities of a range of ferrous crucible processes being used, from carburization of low-carbon wrought iron, to de-carburization of cast iron, and co-fusion of high-carbon cast iron with low-carbon iron.

CONTACT Sharada Srinivasan sharada@nias.iisc.ernet.in National Institute of Advanced Studies, Bengaluru 560012, India.
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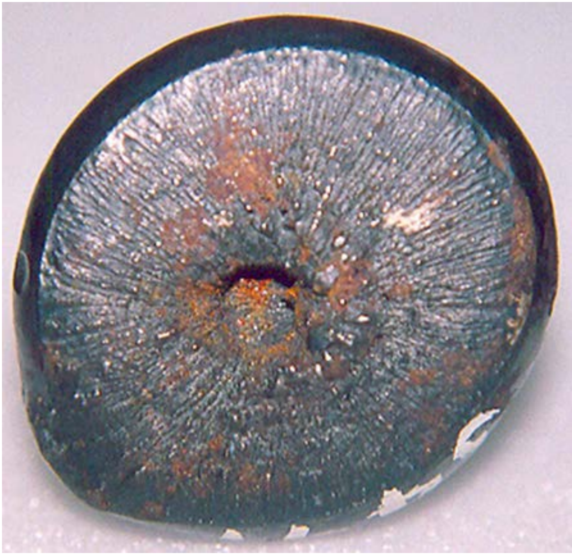

Figure 1. Specimen of wootz ingot from Science Museum, London.

Moreover, the accounts differ as to whether the charge or metal was at all completely molten during carburization or merely viscous, or whether the process involved was solid-state cementation. There is still a dearth in terms of the archeometallurgical record toward more fully understanding the processes and their antiquity.

Materials and Methods

Nineteenth century observers of the manufacture of wootz steel in India, such as Percy, have commented on the carburization of iron to steel in crucibles. By this process a batch of closed crucibles with low carbon iron charge was stacked in a large furnace and fired in a strongly reducing atmosphere in a long 14–24-hour cycle, at high temperatures of not less than 1200°C [12]. In the author's opinion, three different types of crucible processes seem to have been described by nineteenth century travelers, varying from region to region, i.e., the Deccani or Hyderabad process, the Mysore process and what may be described as the Tamil Nadu process. Wood made significant observations on crucible steel processes in the Salem and Arcot districts of Tamil Nadu [13]. The Mysore process can be especially discerned from the accounts of Francis Buchanan. In the Tamil Nadu process and the Mysore process, the charge consisted of wrought iron or bloomery iron (i.e., low-carbon iron with no more than 0.1% C) produced separately, which was then stacked in closed crucibles and carburized in a large furnace [14]. But while the Mysore process charged the wrought iron with carbonaceous matter, Wood's observations on crucible processes in Salem and Arcot districts in Tamil Nadu suggest that only iron was charged and the crucible containing the ingot was not fast cooled in water as in the Mysore process. Gatihosahalli is one of the well-known sites from the Mysore region from which wootz-making debris have been identified and well studied by Anantharamu el al. [15] and also by the author [16]. This site in Chitradurga district fell into Tipu Sultan's dominions, from where crucible steel making was described and illustrated by Francis Buchanan. "Gati" can also be taken to mean "hard" or congealed in Tamil/Kannada, and might refer to slag or ore processing.

The Deccani process from the Hyderabad/Golconda region was renowned for the best-quality wootz. The best-known site is Konasamudram. By the late 1600s, shipments of tens of thousands of wootz ingots were made from the Coromandel coast to Persia. In fact, Tavernier, who in 1679 wrote of the export of steel from Golconda to West Asia and Persia to make the fabled Damascus blades, went so far as to mention that steel from Golconda was the only sort that could be damascened by Persian artists by etching with vitriol [17]. One of the processes followed here was not of carburization of a wrought iron bloom, but of the fusion of two separate pieces of cast iron (i.e., 2–4% C) and a low-carbon iron bloom, thus producing an alloy of intermediate composition. This may be also inferred from studies on Deccani crucibles. Lowe, who extensively surveyed sites in the area of Nizamabad in the 1980s, reported finds of white cast iron remnants in some Deccani crucibles from Konasamudram, which she considered "failed products of crucible steel production" [18]. Cast iron prills with 4% of iron were found in crucibles from Telangana from the author's collaborative technical investigations on material collected from sites, identified by S. Jaikishan and then surveyed in the collaborative UKIERI project with G. Juleff, S. Ranganathan and B. Gilmour [19].

The investigation presented here is on crucibles from a mound in Mel-siruvalur village, South Arcot district in Tamil Nadu. The site of Mel-siruvalur is about 5 km from the hillock of Mamandur, where outcroppings and old workings for copper are found. Mamandur is also about 40 km from the temple town of Tiruvannamalai. Megalithic cairns circles were observed by Sasisekaran [20] at Mel-siruvalur, near the main road, while herostones with 6th century Pallava inscriptions were also identified by him in the vicinity. In 1992, the author noted debris from crucibles (Fig. 2) scattered all the way up to a close-by canal, in which she noted stacks of fragments of elongated and tapering terracotta tubes (Fig. 3) of shapes recalling the legs of megalithic sarcophagi. Sherds with rim fragments of 3 cm, belonging to large storage jars of some 60 cm in diameter with no slip and tempered with rice hulls, were also found. The late C.S. Patil (pers. comm.) of the Mysore Archaeological Survey pointed out their resemblance to

Figure 2. Crucibles from Mel-siruvalur showing fin, bottom and crucible lid.

Figure 3. Terracotta tubes stacked in the canal in Mel-Siruvalur.

Figure 4. Evidence of ore crushing on boulder, Mel-siruvalur.

Figure 5. Tuyere and debris from bloomery iron smelting area, Mel-siruvalur.

Figure 6. Sectioned crucible lid from Mel-siruvalur, also showing rusty splashes from the molten charge.

megalithic storage jars of red ware without slip. Evidence of ore crushing, looking considerably worn, was also seen on low hillocks near the canal (Fig. 4). While the main mound consisted of abundant crucible debris, just behind one of the villager's houses, close to the mound, was a trench from where fragments of tuyeres were retrieved, along with debris from bloomery iron smelting (Fig. 5). Although yet undated, the crucible debris from the mound were clearly from preindustrial production, looking rather worn when compared to those from Gatihosahalli. Microstructural investigations and scanning electron microscopy (SEM) and electron probe microanalysis (EPMA) analyses were undertaken on the fragments of the crucibles from Mel-siruvalur, as reported further, elaborating previous preliminary findings [1].

Results and Discussion

Cross-sections of a fragment that would have formed the lid of a crucible (Fig. 6), and of a crucible fragment from the glassy fin region were studied. Microscopic metallic prills (i.e., solidified metal droplets) were found in cross-sections from different parts of crucible assemblages, such as the remnants of the rusty charge within the crucible (Fig. 7), the interior glassy slag linings of the crucible (Fig. 8) in the fin areas, representing the upper meniscus of the molten ingot, and the glazed exterior portion of the crucible lid (Fig. 9). What is remarkable is that the etched prills from various parts had all fairly uniformly a

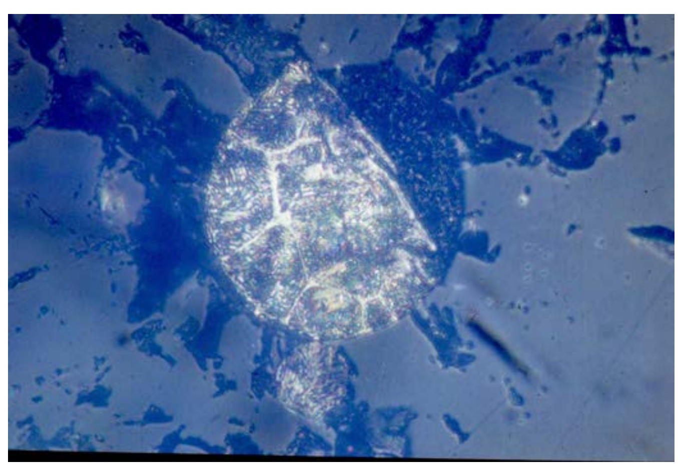

Figure 7. Prill in the glassy matrix in cross-section of crucible fragment from Mel-siruvalur. 462X.

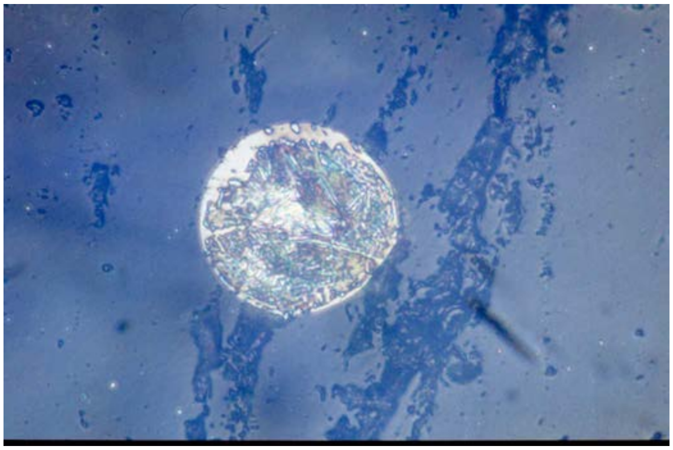

Figure 8. Prill in glassy fin lining inside the crucible from Mel-siruvalur. 462X.

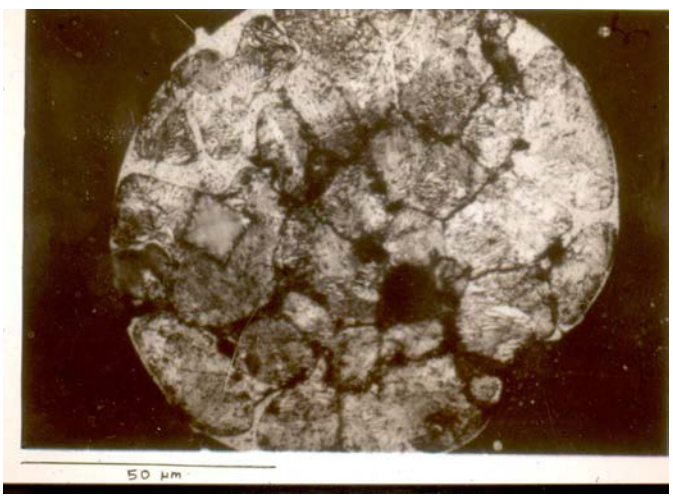

Figure 9. Prill of 80μm diameter in lid fragment from Mel-siruvalur, hardness 400 VPN.

typical microstructure of ultrahigh-carbon hypereutectoid steel (i.e., with a carbon content of about 1.25%). This is typically indicated by the equiaxed honeycomb-shaped prior austenite grains, containing fine lamellar pearlite eutectoid (darkly etched), surrounded by the whitish network of pro-eutectoid cementite or Fe_3C (which does not etch easily) that formed along the grain boundaries. Under present-day laboratory conditions, this structure would usually form from the solidification of a largely melted high-carbon steel, with the pro-eutectoid cementite-carbide precipitating on the austenite grains at the upper critical temperature of not more than 900°C, and with the austenite transforming to pearlite eutectoid during cooling below the lower critical temperature of around 700°C. A prill in the lid of the crucible had a high hardness of 400 VPN, which is about the hardness of normalized steel.

The rusted globules of metal that formed along the sides of the crucible walls are indicative of splashing of the charge. As such, the splashing of some of the charge that may have molten at the top surface need not be taken as a decisive indicator that the entire charge had been molten, rather than being generally viscous or partially liquid. However, the markedly globular nature of all the metallic prills from the various parts of the crucible cross-sections seems to suggest that the charge had indeed become molten. The slag fins, formed from a small amount of molten slag that separated from the molten charge, are also found to have a convex curvature, suggesting again that the charge or ingot was molten with a convex meniscus.

In the case of crucible steel finds from Islamic Merv in Turkamenistan, evidence for co-fusion could be construed from the finds of metallic remnants of composition ranging from cast iron to wrought iron in crucibles [21, 22]. However, in the case of the Mel-siruvalur crucibles, the absence of metallic remnants of cast iron suggests that the process was, rather, the carburization of wrought iron to obtain ultrahigh-carbon steel.

The evidence for the carburization of a wrought iron charge is also supported by the fact that the crucible fabrics are very black, and very carbonaceous with remains of rice hulls as well as intact pieces of charcoal. This suggested that carbon-rich matter was packed into the crucible fabric for the purposes of carburization of low-carbon iron, i.e., wrought iron. The fabric of the crucible was porous, and also consisted of a glassy network with distinctive coked rice hull relics dispersed in the matrix (Fig. 10), the interiors of which were either voids, had charred carbonaceous remains or fused glassy remains. The porous nature due to the voids left from the rice hull relics also had a function to serve, since it would have helped the escape of gases from within the more or less closed crucibles, which would otherwise have burst at high temperatures from the expansion of gases. The carbon-rich crucible strongly suggests that the process followed was of carburization of wrought iron charge. Wrought iron melts at around 1500°C, with the melting point being depressed by the highly reducing conditions to about 1400°C.

The inclusion of rice hulls in the refractory material was noted by Lowe in the manufacture of Deccani wootz crucibles [23]. She postulated that these were added for their high silica and carbon contents, making the crucible a particularly effective reinforced composite refractory material, both to withstand very high temperatures over a very long firing cycle and to maintain a highly reducing environment.

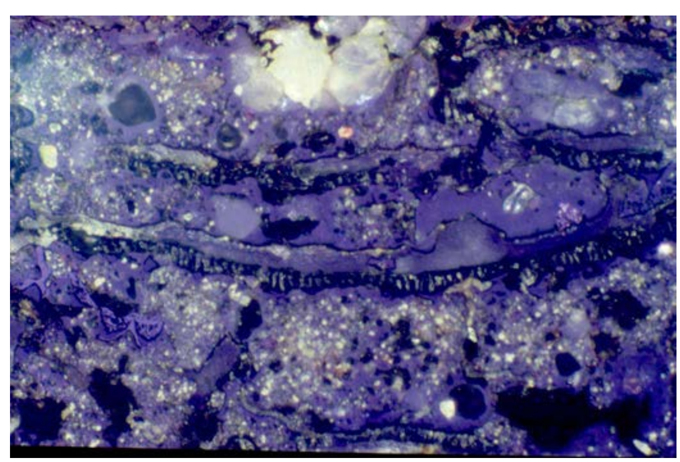

Figure 10. Coked rice hull relics in the crucible fabric from Mel-siruvalur. 462X.

Table 1. EPMA/SEM analyses of cross-sections of crucibles from Mel-siruvalur.

Constituent	Fe	Al	Si	Ca	K	P	S	Ti	Cu	O	Total
Prill	99.94	0.03	1.82	0.08	0.0	0.14	0.02	0.0	0.0	–	102.03
Crucible cross-section (86 mm)	4.95	11.89	34.96	28.06	2.92	1.37	8.36	7.31	0.17		99.99
Area within rice hull (spot)	2.94	3.73	10.05	0.46	0.48	0.0	0.01	0.25	0.06	–	17.98
Area around rice hull 1 (spot)	0.09	0.0	38.9	0.0	0.0	0.01	0.0	0.0	0.0	–	39.00
Area around rice hull 2 (spot)	0.13	0.0	38.74	0.0	0.0	0.1	0.0	0.0	0.0	–	38.97
Glassy matrix within crucible	5.8	7.4	22.99	11.23	3.36	0.27	0.02	0.77	0.03	–	51.17
Slag fin	8.47	6.53	14.47	5.64	1.6	0.41	2.52	13.0	0	–	52.64
Glazed exterior	2.78	5.9	15.16	3.82	2.35	0.06	0.02	0.59	0.0		30.68
White inclusions in glazed exterior	0.17	0.0	46.6	0.0	0.03	–	0.01	–	–	–	53.17

SEM with EDAX analysis by HITACHI S-570 with link AN-1000 with ZAF correction with an instrumental accuracy of around 8% at 25%. Lighter elements, such as O, C, N etc., are not determined.

In the glassy matrix at large the presence of both silicon of up to 23% and aluminium of 6–7% is seen. Analysis of a white inclusion in the exterior of the crucible confirmed that it was quartz or SiO_2 as seen by the composition of 47% Si. Several such white inclusions can be seen in the exterior layer of the crucible, suggesting that the outer layer was packed with crushed quartz fragments to make it more refractory. It may be significant that SEM-EDS analysis (undertaken at Institute of Archaeology, London) of the fused networks just around or circumscribed by the rice hull relics showed them to have a fixed composition of 38–39% silicon, with no other major constituent (Table 1). The levels of lighter elements of carbon, oxygen and nitrogen were not measured. The generally significant level of silicon in the cross-section of the crucible of about 34% is indicative of its good refractory properties. Wootz refractories from Konasamudram were found by Lowe et al. to be reinforced by the formation of fibers of the alumino-silicate mullite, a strengthening material in the field of high-performance ceramics [24]. Formation of mullite and chrystobalite was noted by Rao et al. [25] in crucibles from Gatihosahalli. The practice of inserting rice hulls to reinforce the fabric of pottery is also seen in pottery sherds collected from Mel-siruvalur, as mentioned earlier.

Conclusions

It is significant that investigations on broken crucible fragments from a wootz crucible steel site production site at Mel-siruvalur, Tamil Nadu, in south India, strongly suggest that a high-carbon hypereutectoid steel was indeed produced by the molten carburization of wrought iron in crucibles. The temperatures hence needed for this molten carburization process, which could have been lowered under highly reducing conditions, but nevertheless not much below ca. 1400°C, may have been amongst the highest achieved in preindustrial processes. The silicon-rich and carbon-rich matrix also suggests that the crucible was highly refractory to be able to withstand high temperatures. The addition of quartz fragments, rice hulls and pieces of charcoal to the crucible fabric contributed to such a silicon- and carbon-rich matrix. The finds of debris from a bloomery iron furnace in the vicinity supports the idea that wrought iron was being carburized at the site.

The microstructures of the prills from the crucibles from Mel-siruvalur are consistent with the structures and composition of superplastic ultrahigh-carbon steels (around 1.25–1.5% C). Ultrahigh-carbon steels, exhibiting superplasticity, were patented by Wadsworth and Sherby and were shown to correspond to "true" Damascus steel or Bulat blades [26, 27]. It may be noted that the "true" Damascus blades had watered silk patterns, known to emanate from the typical etched crystalline structure of hypereutectoid high-carbon steel, with the alternation of light pro-eutectoid cementite with darkly etched pearlite. These are, hence, different from the "welded" Damascus blades, made by welding together high-carbon and low-carbon iron. Figure 11 shows such a pattern in an Indian example of a watered steel blade in a sword from the National Museum, New Delhi. Therefore, the significance of the investigations on these crucibles is that they tend to confirm the literary references, suggesting that an ultrahigh-carbon steel was produced by crucible processes in south India of the kind from which the "true" Damascus blades could have been made. Indeed, Tavernier's comment mentioned earlier, that steel traded from the Coromandel coast of southern India was the only sort that could be damascened by Persian artists to make Damascus blades by etching in vitriol, might be significant in implying some regional prowess in making such true ultrahigh-carbon steels.

Another archeologically significant aspect is the striking match between the microstructure of metallic remnants in prills from Mel-siruvalur with an especially similar hypereutectoid steel structure in a nail excavated from the early historic site of Pattinam in Kerala, investigated by the author [28], and similarities also to a chisel from the megalithic site of Kodumanal, 3rd century BCE, studied by Sasisekaran [29], having the honeycomb structure of prior austenite surrounded by pro-eutectoid cementite networks. Early experimentation in southern India with higher carbon steels is also suggested by investigations by the author on a cross-section of a tiny ring from the megalithic site of Kadebakele in Karnataka, carbon dated to about 800–700 BCE, excavated

Figure 11. Pattern in Indian watered steel blade in National Museum, New Delhi.

by a joint team of the University of Chicago and University of Michigan, and found to be of a high carbon through pearlitic steel with at least 0.8% carbon, suggestive of crucible carburization [30].

The structures from Mel-siruvalur suggest that the ingot was slow cooled, as seen from the formation of fine lamellar pearlite within the equiaxed prior-austenite grains, as there is no evidence of the formation of bainite or martensite from fast cooling or quenching. This may be inferred as being closer to the "Tamil Nadu process", as previously described in the accounts of Wood, which suggests that the charge consisted of iron (indicative of a process of carburization of iron), and is distinct from the Mysore process, by which fast cooling or quenching was reportedly observed. The closer match in terms of the remnant pearlitic structure within a honeycomb structure, noted in finds from early historic and megalithic southern Indian contexts, mentioned earlier in the sites of Pattinam and Kodumanal, suggests that these were also made by something approaching the so-called "Tamil Nadu process". This may thus have been a more archaic method of crucible steel production than some of the other processes that have been noted. Further archeological excavation followed by further archeometallurgical investigations at Mel-siruvalur and archeometallurgical surveys in the Arcot and Salem districts for identifying production sites would help throw more light on the production mechanisms and historical trajectory of the processes.

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