

tions of iron, along with the low totals, are characteristic of limonite. The ranges for Ni and Cu are most significant.

The concentration range for nickel is much lower than that indicated from the earlier analyses by Desch (1929). While the new analytical results would not completely rule out the possibility of meteoric origin (Photos, 1989), the lower concentrations below 0.2% Ni do certainly decrease the strength of the argument. Although both sets of analyses were done on corrosion products, the earlier set by Desch did include a larger sample; most likely the whole bead. To resolve this matter, a new metallographic section across one of the remaining beads would be required. This could confirm or reject the hypothesis that the bead was originally metallic. Micro-analysis may also discover any remaining metallic iron within the corrosion products.

It is important to establish the origin of this Pre-Dynastic iron metal and this is where the observed copper range in the corrosion products is important. It is uncertain whether any copper or copper alloy artefacts were also in the same grave with the beads. If the copper is, indeed, a component of the original iron metal, then the implication would be that the iron may be a by-product from copper smelting. Again, a section would be necessary to establish the distribution of copper within the iron corrosion products. Investigations of copper in corroded iron artefacts from Timna by Gale et al. (1990),

simply report the presence of copper based upon X-ray fluorescence analysis (XRF). Chronologically, these 'iron beads' from Pre-Dynastic Egypt would be comparable to the Late Chalcolithic or Early Phase of metallurgical activities in Rothenberg's (1990) table for the Sinai-Arabah. At this time, copper smelting was conducted using iron ore flux, which could be the actual source of the smelted iron.

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References

- Buchwald, V. F. 1975. *Handbook of Iron Meteorites*, Vol. 2, Berkeley. University of California Press.
- Desch, C. H. 1929. *Reports on the metallurgical examination of specimens for the Sumerian Committee of the British Association*. Reports of the British Association for the Advancement of Science.
- Gale, N. H., Bachmann, H.-G., Rothenberg, B., Stos-Gale, Z. and Tylecote, R. F. 1990. The Adventitious production of iron in the smelting of copper. In B. Rothenberg, ed. *The Ancient Metallurgy of Copper*. London. IAMS. 182-91.
- Gowland, H. and Bannister, C. O. 1927. *Ancient Egyptian Metallurgy*. London. Chas. Griffin.
- Petrie, W. M. F. 1914/5. *Ancient Egypt Journal*, 12-23.
- Photos, E. 1989. The question of meteoritic versus smelted nickel-rich iron: archaeological evidence and experimental results. *World Archaeology*, 20, 3: 403-21.
- Rothenberg, B. 1990. *The Ancient Metallurgy of Copper*. London. IAMS.
- Wainright, G. A. 1932. Iron in Egypt. *Journal of Egyptian Archaeology* XVIII: 3-15.

Analytical Investigation of Crucible Steel Production at Merv, Turkmenistan

According to the early Islamic texts, three methods are described for indirect production of steel (*fuladh*) as discussed by Allan (1979) and al-Hassan and Hill (1986). The most common, traditional method is solid state carburization of wrought iron. There are many variations on this method. It is also known as 'case hardening' or in other instances 'cementation'. This is a diffusion process in which wrought iron is packed in crucibles or a hearth with charcoal, then heated to promote diffusion of carbon into the iron to produce steel. Alternatively, another indirect method uses wrought iron and cast iron. Although there has been some uncertainty on the translation of the word *daus* in Islamic texts, the cast iron interpretation is generally accepted (Allan, 1979). In this process, wrought iron and cast iron may be heated together in a crucible to produce steel by 'fusion'. This is also called a 'visco-liquid diffusion process' (Needham, 1958) and may operate below the melting point of true cast steel (Smith, 1960). A third indirect method to produce steel is partial decarburization of cast iron or a high carbon steel bloom. Again, there are variations of this method, but generally it is considered very difficult to control (see Rostoker and Bronson, 1990). Outside the Islamic textual evidence, inadvertent direct production of steel during bloomery iron smelting represents another possibility, but it is not considered here in the context of an indirect or multi-stage process to routine production of steel. A detailed account of the many variations is presented by Rostoker and Bronson (1990). It is against these three main methods for indirect steel production, this preliminary report concerns the

archaeometallurgical evidence and its interpretations for early Islamic times at Merv, Turkmenistan.

The archaeometallurgical investigations at the Islamic site of Merv represent only one aspect of an international collaborative project under the direction of Dr Georgina Herrmann and Dr K. Kurbansakhatov. The organizations involved in the International Merv Project are the Institute of Archaeology, University College London, YuTAKE (the South Turkmenistan Multi-Disciplinary Archaeological Expedition), Turkmen Academy of Sciences, Ashgabat, and the Institute for the History of Material Culture, St Petersburg.

During the 1993 season at Merv, two areas with surface concentrations of crucible fragments, green 'glass' fragments and slagged furnace fragments have been located in the survey in area MGK 7.F.II. The scatter of pottery around and within the archaeometallurgical remains in MGK 7.F.II at Merv is predominantly dated as Early Islamic, perhaps 8th or 9th century AD, by the archaeological team. A small-scale excavation was conducted in the 1994 season (Fig. 1) by Dr K. Kurbansakhatov, D. Connolly, St. J. Simpson, Ann Feuerbach and other members of the International Merv Project.

Fragments of crucibles, furnace wall and tuyeres, as well as the glassy slag, were collected from the metallurgical dumps by Dr J. Merkel for technical investigation in the Wolfson Archaeological Science Laboratory at the Institute of Archaeology, UCL. The analytical work is undertaken, in part, as supervised M.Sc. research in archaeometallurgy by Ann Feuerbach. The metallurgical



Fig. 1. Excavations of the remains of crucible steel production at the Islamic city of Merv.

process has now been identified as crucible steel production, based foremost upon the presence of abundant carbon steel droplets in the glassy green slag adhering to the inner surface of crucible fragments collected from the two areas. The steel is identified using etched metallographic sections and microhardness measurements (Hv 140–320). Against metallographic standards, the carbon concentrations of the steel droplets seem to range from <0.1–0.8%. The structures are characteristically variable proportions of pearlite and ferrite. Microanalysis also detected silicon and sulphur in the iron droplets. The droplets range in diameter up to approximately 0.3mm, but most droplets are too small for microhardness and adequate etching for microstructure identification. Quantitative microanalysis of the steel droplets for phosphorous and other possible alloying elements will follow. The Merv oasis is alluvial and without local iron ore deposits, so iron smelting locally is very unlikely. Steel production at Merv was probably based upon recycled wrought iron scrap.

Qualitative compositional analysis (SEM/EDS) of the associated green 'slag' adhering to the interior surface of a crucible fragment (Fig. 2) identified silicon, aluminium, calcium, iron, manganese, magnesium and potassium. The 'slag' does not have a crystalline structure; it is a glass with a variable composition. Concentrations for calcium, potassium and aluminium from the SEM/EDS, however, are quite different from those typical for glass. Fragments of the glassy green slag are observed to melt under reducing conditions in an electric furnace at a temperature of about 1250°C. This is a temperature below the melting point of true cast steel (Smith, 1960). Several glassy slag fragments exhibit viscous flow patterns which will be investigated. Some crucible fragments indicate the upper level of the molten crucible contents as a 'fin' of glassy green slag (Fig. 2). It is this slag which contains abundant steel droplets. Above this 'fin' of slag, the

adhering pattern of corroded iron is interpreted as 'splashes' onto the upper crucible wall. Of course, these forms will be investigated in detail for relict structures in the corrosion products. Below the 'fin' appears a honeycomb pattern in the glassy slag on the inner surface which appears similar to that on the inner surface of other crucible base fragments. Similarities in descriptions of a characteristic honeycomb pattern in the slag (see Percy, 1864) as well as striations in some glassy green slag fragments suggest incomplete fusion (see Smith, 1960).

Crucible fragments are variable in thickness and condition, but appear to represent a single type. Wall



Fig. 2. Detail of crucible wall fragment showing differences above and below the 'fin' of glassy green slag.

thickness ranges from 0.5–2.5cm. Fragmented circular segments with a reconstructed outer diameter of 6–8cm and a thickness of about 0.5cm are interpreted as crucible lids. These fragments have a central perforation of about 1cm in diameter and the outer edges exhibit evidence of a clay seal to a crucible wall. The use of a luted, perforated lid is a variation in accounts of traditional steel making in crucibles during the 19th century (see Rostoker and Bronson, 1990). The thinner fragments and lids are interpreted as having been fired at high temperatures under reducing conditions. The thickest fragments have a light-coloured core and dark, reduction fired surfaces. Investigations of the refractory properties of the crucible fragments and furnace wall fragments will be undertaken in order to characterize the materials relative to their performance at high temperatures and to estimate the duration of firing. These would be important considerations for steel production.

Fragments of furnace wall consist of the local mud brick, but lined with crucible fragments on the interior. The inner surface is covered with a black, adhering layer of slag arising from high furnace temperature and fuel ash which has not yet been investigated. The furnace wall fragments were about 5–10cm in length and 5–10cm in height. Several are interpreted as representing corner fragments. There are examples of crucible base fragments slagged onto a lower ceramic support (Fig. 3) which was placed on the furnace bottom. During the excavations in the summer of 1994, the remains of two furnaces were discovered. The internal diameters of the circular furnaces are about 70cm, so it is estimated that a maximum of some 40 crucibles could be packed into each furnace. Part of one furnace wall was lifted from the site for technical study, currently in progress. Slag remains from melting brass were also present in the archaeological layers with the furnaces. The context for the furnaces is interpreted as a metallurgical workshop involved with several different metallurgical processes.

While these archaeometallurgical samples are attributed to crucible steel production, there remain many questions and lines of evidence to investigate and many samples still to section and analyze. Distinction between the three possible indirect methods (cementation, fusion or decarburization) for crucible steel production at Merv would depend upon further discovery of minute fragments of raw materials in the glassy green slag or adhering to the crucible walls. Raw materials might include wrought iron with elongated slag inclusions, carbon impressions or components (such as rice husks in the later Indian examples presented by Lowe et al. 1991) or perhaps even cast iron coatings on wrought iron droplets. Final products such as ingots or fragments may

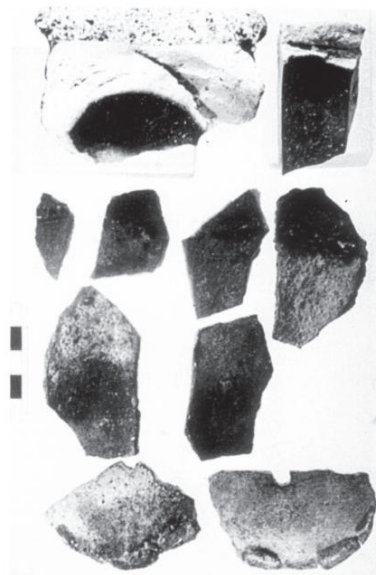


Fig. 3. Interpretation of the crucible fragment positions for steel production. The fragments are arranged from the base to the lid.

not necessarily be conclusive enough to distinguish between fusion or cementation. For the interpretation of these archaeometallurgical specimens from Merv, the most salient observations relate to the abundant steel prills of variable carbon content in a relatively small amount of glassy green slag with a melting temperature of approximately 1250°C adhering to the inner wall of crucible fragments. Further excavations of the archaeometallurgical remains are anticipated.

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References

- Allan, J. W. 1979. *Persian Metal Technology*. London. Ithica Press.
- Al-Hassan, D. Y. and Hill, D. R. 1986. *Islamic Technology: An Illustrated Guide*. Cambridge. Cambridge University Press. 251–60.
- Bronson, B. 1986. The making and selling of Wootz, A crucible steel of India. *Archaeomaterials* 1: 13–51.
- Lowe, T. L., Merk, N. and Thomas, G. 1991. An historical mullite fibre-reinforced ceramic composite: Characterization of the 'Wootz' crucible refractory. In P. A. Vandiver, et al. eds. *Materials Issues in Art and Archaeology II*. Materials Research Society, Vol. 185: 627–32.
- Needham, J. 1958. *The development of iron and steel in China*. London. Newcome Society.
- Percy, J. 1864. *Metallurgy, Vol. 2, Iron; steel*. London. John Murray.
- Rostoker, W. and Bronson, B. 1990. *Pre-Industrial Iron, Its Technology and Ethnology*. *Archaeomaterials Monograph No. 1*. Philadelphia.
- Smith, C. S. 1960. *A History of Metallography*. Chicago. University of Chicago Press.