Goat path discovery unlocks secrets of medieval zinc

Medieval zinc furnaces, recently excavated in India, represent one of the most sophisticated pyrotechnical processes in use before the Industrial Revolution – a process which is still basic to all high temperature distillation operations today. Paul Craddock, of the British Museum Research Laboratory and a member of IAMS Scientific Committee, describes the expedition in which he took part last December.

Although zinc minerals were used in Roman times in combination with copper to form the alloy brass, the individual metal was not isolated until much later. In the West it has been produced for little more than 200 years, and only became common in the 19th century.

Of all the base metals zinc is the most difficult to process. Most metals smelted in a simple furnace collect as a molten mass in the bottom, but in such conditions, zinc, with its low boiling point of 907°C, would have been produced as a vapour and lost up the flue.

Clearly a very different process was necessary to smelt zinc ore, retain the vapour and condense it. Such a system was patented in 1738 by an Englishman, William Champion, who designed a furnace fitted with an external condenser. He built a plant at Warmsley, near Bristol, where the calcinated ore was reduced in retorts which were set in a furnace with their necks protruding downwards into a cooler chamber where the zinc vapour condensed and was collected in water-filled vessels.

Champion’s plant eventually contained 30 such furnaces and by 1760 was producing metallic zinc at the rate of about 200 tons a year.

Sources unknown

Whether Champion acquired his technology directly or indirectly from the East is not known for whilst he must be given the credit for introducing the first commercial zinc-making process in the West, zinc had been arriving in Europe for a good many years in ships of the Dutch and British East India companies. Historians have never been quite certain where the metal came from, but most likely sources were India and China. How it was produced remained a mystery.

In more recent years metallurgists and mining engineers visiting the modern mines at Zawar, in the Indian state of Rajasthan, reported seeing huge dumps of spent retorts and other debris of zinc production around the old mines. It was not until last December, however, that they were scientifically investigated in a joint project of the University of Baroda, the British Museum and Hindustan Zinc Limited, the company which now owns and works the site.

Already the intact furnaces uncovered by our excavation, still containing their full charge of retorts, and our analysis of the refractories, have given us a detailed picture of the process – a process that predates 18th and 19th century “discoveries” in Europe by several centuries.

We expected to get most of our information from the study of stratified fragments within the heaps, but on only the third day of the excavation one of the Baroda team spotted the corner of a refractory plate sticking out from a heap by the side of a goat path.

Mounting excitement

If our conjectured reconstruction of the furnace was correct then this brick was in its right alignment. With mounting excitement we cleared a small area above and around it to reveal first, the edges of furnace walls, and then the tops of retorts still in situ.

continued overleaf
This was something we had not dared to hope for, and over the next few days as more and more was revealed it became apparent that the whole process was much more sophisticated than we imagined. We had discovered a continuous bank of seven furnaces, four of which we cleared, each originally containing 36 retorts.

Thus a total of 252 retorts could be fired simultaneously. This bank of furnaces is provisionally dated by radiocarbon to the 16th century, and another and even larger set, which we also excavated, is dated to the 18th century.

In principle, the process was quite simple. The ore at Zawar is sphalerite, zinc sulphide. This was roasted in air to convert it to oxide ready for reduction—a process which was not “invented” in Europe until the 18th century. The roasted ore, together with charcoal and other ingredients, was then placed inside an open clay retort. The medieval Indian alchemists listed various exotic ingredients which cannot now be verified. Salt was also specified and this was found in the Zawar retorts which we examined. Once again, the addition of salt was common European practice centuries later to increase the overall efficiency of the process.

**Clay condensers**

Clay condensers were then luted in place and sealed retorts were put in the furnace to rest on a perforated clay plate with their necks protruding through large holes into a cooler chamber below the main furnace. The fire was built above and around the retorts and was maintained at a sufficiently high temperature to cause considerable softening of the clay retorts. Scientific examination of sections of the vitrified retort walls by scanning electron microscope in the British Museum Continued in col. 2 opposite.

Four of the seven furnaces cleared by the expedition. Each originally contained 36 retorts. Picture P.T. Craddock
Facts about zinc

The Romans' method to produce brass was to heat a mixture of zinc and copper oxides, or zinc oxide and metallic copper, with carbon in a closed crucible. The zinc vapour produced during the reduction dissolved immediately in the metallic copper. A similar method was used in Europe until the beginning of the 18th century.

Earliest use of the word "zinc" was by Paracelsus (c. 1490-1541) who described it as a bastard form of copper.

Georgius Agricola (1544-1555) refers to zinc in De Re Metallica by the name "contrexy" but in other works he uses "zincum" for ore found in Silesia.

The term "spelter", employed for commercial grades of zinc until about the middle of this century, came into use in 1600. It was derived from the Dutch word "spant", which meant both zinc and pewter, a fact that emphasizes continuing confusion over the true nature of the metal.

An ancient idol, containing 87% zinc, discovered in prehistoric ruins in Transylvania (modern day Romania), is the oldest known zinc object. Zinc-filled silver bracelets dating to 500 BC have been recovered on the island of Rhodes.

Zinc ores are commonly found in association with those of other metals, notably lead, though frequently with copper or gold and silver. Mining operations are typical of those used generally underground. Owing to the nature of the ore veins, most zinc mines are underground rather than open pit.

The lead-zinc mine at Trepca in Yugoslavia, one of the biggest in Europe, was discovered in 1926 by investigating the remains of mines developed first by the Romans and later by Saxons. In one ancient adit, explorers came across the figure of a Roman centurion carved into the rock. The late Sir Alfred Chester Beatty, who initiated the exploration, ordered the carving to remain undisturbed and always referred to it as "my Roman consulting engineer."

World zinc production is currently running at between 4 million and 5 million tons a year. Major producing countries include Canada, USA, Russia, Belgium, France, Germany and Australia.

Biggest single application for zinc is for the protection of steel against atmospheric corrosion. It also forms the basis for one of the best alloys used in die-casting and, of course, in brass.

Zinc can be readily rolled into sheet and in this form is used in building construction, giving long service at reasonable cost.

A major boost to the zinc industry in the United States recently has been a move to copper-plated rolled zinc for the one cent coin. Composition was switched in 1982 from 95% copper and 5% zinc to 97.6% zinc and 2.4% copper, including the plating. As a result, US consumption of rolled zinc almost doubled, and last year the Mint used more than 50,000 tons.

In common with other base metals, zinc has been in over-supply during the world industrial recession, but demand has increased substantially over the past few months.

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Tourist Gold Mine

Visitors to the Welsh village of Pumpaint in Dyfed will soon be able to inspect the remains of a Roman gold mine at Dolaucothi in the nearby hills.

The mine was worked sporadically since Roman times but finally closed down in 1936. Recently, some of its workings have been exposed by members of the mineral exploration department of University College, Cardiff, and the site – the only place in Britain where evidence has been found of Roman gold mining – is to be opened as a tourist attraction.

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Huelva Report

Copies of Studies in Ancient Mining and Metallurgy in S-W Spain, a report of the exploration by IAMS and the Universities of Seville and Madrid in the Huelva Province in 1974-78, are now available to Newsletter subscribers at the reduced price of £10.

A few copies are still available of the IAMS monograph Chalcolithic Copper Smelting, by Beno Rothenberg, R. F. Tylecote and P. J. Boydell, at £2, half the original price.

Orders should be sent to IAMS secretarial offices, Institute of Archaeology, 31-34 Gordon Square, London WC1H OPY.
IAMS smelting experiments featured in TV serial on metals history

Scenes shot in the Timna valley, Israel, in 1981 when a desert copper smelter was built and operated in a process identical to that followed by metallurgists who worked there more than 3,000 years earlier, are included in a new international television series on the history of metals.

Entitled Out of the Fiery Furnace, the film was written and produced by Robert Raymond at the invitation of Sir Roderick Carnegie, chairman of CRA Limited, Melbourne, Australia. The seven 50-minute episodes are narrated by Michael Charlton, one of the best known television commentators in Britain through his long association with Panorama.

Nothing is more inextricably involved in the daily life of 20th century man than metals. The modern world - even civilization itself - is built on a framework of copper, steel, aluminium and a score of other metals and alloys. Throughout history the control of metal resources has been a determinant of success or failure among nations and empires. And yet man has known and used metals for only a fraction of the time that he has inhabited the earth - for perhaps 10,000 out of the 2 or 3 million years that he has been using stone, wood, clay, bone and other natural materials.

Coloured stones

Stone Age man had come surprisingly far without metals. He had begun to cultivate crops, domesticate animals, build dwellings and was on the verge of establishing the first urban societies. Then - somewhere, somehow - he discovered that many of the coloured stones he had been using for decoration could be made to yield new materials of almost infinite utility.

The growth of metal technology is one of the most significant developments in the history of mankind. It involves all people, everywhere. There have been many television series on science, the arts, exploration, wildlife, economics and many other aspects of the evolution of modern society. But never before has there been an attempt to tell the story of metals in one encompassing sweep.

The new series is based on exhaustive scientific and archaeological research, and took four years to complete. It was filmed on locations in all parts of the world, on sites where significant developments in the history of metals took place.

Timna's copperbelt

One such location was the Timna valley, site of what was probably the world's first great copperbelt, operated by Egyptian mining expeditions sent forth by the Pharaohs in the 13th and 12th centuries BC, and where too there is evidence of primitive copper smelting 3,000 years earlier in the Chalcolithic Period.

Here exploration by IAMS and its predecessor, the Arubah Expedition, had been going on since the 1950s: modern scientific study of ancient mining and metallurgy has developed largely from the important discoveries made over a quarter of a century by these teams in the field and by backroom boys in the laboratories.

In 1981, as part of IAMS's continuing post-excavation analysis of the Timna finds, experiments, using local ores and materials were carried out on the spot to demonstrate how copper was smelted towards the end of the Bronze Age.

Robert Raymond and his crew were present to film the trials which were performed by John Merkel, a young American, as the culmination of his doctoral research with IAMS and the Institute of Archaeology in London.
The cameras whirled as the furnace was fired and volunteers worked foot-controlled bellows to raise the temperature to the required level. They toiled all morning and into late afternoon under a blazing sun. In the end more than 1 kg of copper metal was recovered from "the fiery furnace" - and the name of a great international television serial was born.

**Extensive research**

In a foreword to the inevitable "book of the film" which is to be published shortly, Robert Raymond confesses that he was at first reluctant to accept Sir Roderick Carnegie's invitation to make the film: "Metals play such a pervasive role in modern life, and in fact have had such an influence on the growth of civilization, that it seemed almost presumptuous to consider even outlining the history of metallurgy in a television series... life was too short, I felt, to grasp even the fundamentals of such a vast subject in any reasonable time."

But Sir Roderick had already made some important dispositions. Before Raymond came on the scene he had commissioned Willard C. Lacy to travel the world and collect material on metallurgical history, and to identify relevant physical evidence and archaeological sites. Lacy, a genial but rigorous-minded American, had been a practising mining geologist before becoming Professor of Geological and Mining Engineering at the University of Arizona, and later Professor of Geology at James Cook University in Queensland, Australia.

"Bill Lacy's massive volume of research material, succinctly and lucidly expressed, made both the television series and the book possible," acknowledges Raymond. All his material has now been lodged with the Mitchell Library in Sydney and the Latrobe Library in Melbourne for the benefit of future researchers.

CRA also provided Raymond with a panel of distinguished academic and scientific consultants, some of whom, when they read the draft scripts, were obviously concerned at the limitations which the television medium imposes on the telling of history, and in particular the denial of discussion, comparison and qualification. By its very nature, the television

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**Where to see the film**

*Out of the Fiery Furnace* is being screened by the Australian Broadcasting Corporation starting on May 27.

The series has been sold to West German Television and for showing in the Middle East. Screenings in Germany will begin in November. The film has also been bought by the Irish television authority but no dates for its showing have yet been announced.

When this issue of the Newsletter went to press arrangements had still to be finalized for screening the film in Britain and in North America.

The book was expected to be published, first in Australia, at the end of May.

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...treatment tends to suggest that events follow one another in a chronological line of development; that things happened first here, and then there; that someone must have been the "first" to make discoveries, whereupon others followed.

In fact, although the origins of metallurgy are still obscure, it is known to have developed not by a linear progression of individual achievements, but rather through scattered bursts of innovation and discovery against a broad, slow advance in technology. Whether or not an idea took root in any particular area seems to have depended upon whether society was ready to receive it and economically capable of developing it.
Mystery of Timna’s iron solved by lead isotope ‘fingerprinting’

Scientists have long sought a means to discover where precisely the ancients obtained the ores from which they extracted their metal. Noël Gale and Zofia Stos-Gale describe a “fingerprinting” process by lead isotopes in which they are concerned at the Department of Geology at Oxford University, and which has been applied to solve the mystery of the occurrence of iron at Timna long before its common use.

A solution of the problem of identifying the source of ancient ores is of great importance to archaeology since it would establish beyond doubt trade routes in the metals, cultural contacts and the technology and economy of ancient societies.

Attempts to solve the problem by chemical analysis of metal artefacts and ores have founded, and metallurgical investigations have fared little better. As Professor Coles of Cambridge recently observed: “Chemical analysis of the metal products of the European Bronze Age is perhaps the most monumental disaster of all the contemporary studies... it has provided a few answers in restricted areas of enquiry and created mass confusion in others.”

The failure of this approach is partly due to the heterogeneous distribution in most ore deposits of the minor elements on which reliance has been placed. Moreover, as Professor Tylecote’s experiments have shown, the process of extractive smelting disrupts the pattern of major, minor and trace elements existing in the original ore.

Fortunately, an alternative scientific approach to this problem exists: lead isotope analysis.

Most elements are composed of atoms of different mass. These are called isotopes. For instance, lead consists of the four isotopes Pb-204, Pb-206, Pb-207, and Pb-208, having masses respectively of 204, 206, 207 and 208 atomic mass units. The chemical processes of smelting, refining and even corrosion leave the original proportions of the lead isotope composition unchanged from that in the original metal ore.

... marks the spot

The isotopic composition of the lead in an ancient metal object therefore provides a “fingerprint” which allows the ore source to be pinpointed. All that is necessary is to compare the isotopic composition of the lead in the artefact with those of ores from various ancient mines. Not only can this be applied to lead objects, but also to those made of silver and to copper-based alloys which always contain traces of lead derived from lead impurities in the ores.

Modern mass spectrometers make it possible to measure the lead isotope composition using as little as one millionth of a gram of lead metal.

It remains to be added that the isotopic composition of the lead in various ores differs because some of the atoms of lead 206, lead 207, and lead 208 are formed respectively by the radioactive decay of uranium 238, uranium 235 and thorium 232. Different ores will have a different lead isotope composition depending on the geological age and on the relative amounts of uranium and thorium initially present in the ore-forming fluids.

Work at Oxford

This method of provenancing metals has been applied at Oxford with great success to locating the sources of lead and silver in the Bronze Age Aegean and to ancient sources of copper. In fact the isotopic composition of lead in a particular ore deposit is not quite constant, but analyses of a number of samples from a deposit define a fingerprinting “field” of composition.

Early Bronze Age culture of the Cycladic Islands of the Aegean made use of lead and silver. Lead isotope analyses made at Oxford of four lead boat models dating to about 2900 BC, which were found in a grave on Naxos, fall in the isotopic field characteristic of the lead-silver ores from the mines on the island of Siphnos (see IAMS Newsletter No.4, p.3).

This was first proof that the Siphnos mines were
Iron minerals were usually present in the charge of an ancient copper smelting furnace, either as an unwanted part of the copper ore, or added in order to flux siliceous copper ores like those at Timna. Though most of the iron passes as oxides or silicates into the slag, experimental work both by Rostoker and Tylecote has shown that under certain conditions some may be reduced to the metallic state and incorporated into the copper metal.

Dr Craddock's analyses of copper artefacts found in the Timna temple show that many contain up to 2 per cent, and some up to 7 per cent, of iron. Professor Tylecote has demonstrated by experiment that this iron may be removed from the copper by a refining process in which the impure copper is kept molten for some hours in a crucible. Under the right reducing conditions the iron separates out as an upper layer which is sufficiently malleable to be forged into simple objects such as rings.

**Temple jewellery was iron from Timna**

Many of the votive gifts found in Timna were actually brought from Egypt by the mining expeditions; and those that bore hieroglyphic inscriptions and cartouches have enabled the temple to be accurately dated. As a few iron objects are known from Egypt in the Late Bronze Age, it was reasonable to suppose that the iron jewellery found among the treasures could have been of Egyptian origin.

Comparative lead isotope analyses of copper and iron ores from Timna, together with the iron jewellery, show that this was not the case. The iron objects have the same lead isotope fingerprint which characterises the Timna ores and the few Timnian copper artefacts so far analysed. The iron jewellery here is clearly a local product. The other strands of evidence summarised above suggest strongly that the metal from which the jewellery was made was a rare and accidental byproduct of copper smelting and refining at Timna.

Lead isotope analysis has thus helped to clear up one of the major mysteries of Timna's ancient mining: it may well prove to be the tool to solve other important puzzles surrounding the emergence of iron as one of man's most useful materials.

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**Lead isotope ratios for metal ores from Timna compared with the isotopic compositions of copper objects and iron jewellery found in the Egyptian mining temple nearby**
Important discoveries about the use of metals were undoubtedly made independently in many parts of the world, but only in some did they lead to higher levels of technology.

It is much to the credit of the producer and writer that both the film and the book tend to emphasize those concepts which favour independent discovery and innovation rather than the established ideas of diffusion. Diffusionists hold that the most significant advances began in or near the “cradle of civilization” in the Near East, spreading thence across the ancient world. But in recent years a spate of archaeological and anthropological discoveries has mounted a serious challenge to the traditional view.

Whichever school of thought one may support, Out of the Fiery Furnace is bound to stimulate a greater awareness of the vital part that metals has played in the development of civilization and culture throughout the world. It should also provide first-class entertainment to millions of viewers.

**Arsenical copper studies**

The use of arsenical copper in ancient times before copper and tin were alloyed to produce bronze is being studied in a joint project by IAMS and Lehigh University, Bethlehem, Pennsylvania.

Arsenical copper is known from the early history of metals—in Spain as long ago as the 4th/3rd millennium BC. Natural alloys probably accounted for the first known occurrences: man did not learn to alloy copper and arsenic until much later, and it is likely that he discovered deposits when sources of oxidised copper became more difficult to find.

At Lehigh the researches are being directed by Professor Mike Notis, head of the Department of Metallurgy, who is not only studying the properties and behaviour of the material but also seeks to discover why it virtually disappeared from use with the coming of bronze.

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IAMS as an international organization whose function is to promote, supervise and coordinate research into the origins and development of mining and metallurgy from earliest times.

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