L'OR DANS L'ANTIQUITÉ

DE LA MINE À L'OBJET

Sous la direction de Béatrice Cauuet

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Couverture

Photo du haut : *Détail de la maquette de la mine d'or des Fouilloux (Jumilhac, Dordogne, France), exploitée à la Tène finale. Conception B. Cauuet, réalisation P. Maillard de MAD Entreprise (cliché : Studio 77).*

Photo du bas : *Extrémité d'un collier d'or datant du Bronze final, Gleninsheen, Co. Clare, Irlande (cliché National Museum of Ireland).*

Dos de Couverture

Photo du haut : *Bouloun-Djounga (Niger) : mine d'or ouverte dans la latérite (cliché G. Jobkes).*

Photo du bas : *Femme Fulbe (Mali) parée de boucles d'oreilles massives à lobes effilés (cliché B. Armbruster).*

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Or, économie et symbolique dans le monde antique

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Production Techniques of Celtic Gold Coins in Central Europe

Résumé

Les techniques de production de monnaies d'or utilisées à l'époque celtique en Europe Centrale peuvent être décrites comme suit :

- du cuivre ou du bronze associés à des grains ou de la poudre d'or étaient pesés et versés dans des moules en argile. L'or et l'argent provenaient probablement d'objets antiques issus des régions méditerranéennes hellénistiques, essentiellement sous forme de monnaies.
- Les moules étaient couverts de charbons incandescents dans un petit fourneau, large de 25 x 25 cm. Ce fourneau était ventilé par des soufflets.
- Après environ deux à cinq minutes le point de fusion de l'alliage était atteint. Le moule à flans refroidissait en atmosphère réductrice.
- Les flans étaient aplatis, recuits et frappés. Les alliages riches en cuivre qui sont très sensibles à l'oxydation au cours de l'opération de fusion pouvaient être recuits en atmosphère réductrice après la frappe.

Abstract

The technique used in the production of gold coins in Celtic times in Central Europe can now be described as follows :

- Gold grains or dust with added copper or bronze were weighed and filled into clay moulds. The gold and silver sources were probably antique objects from Hellenistic Mediterranean areas, mainly coins.
- The moulds were covered with glowing charcoal in a small furnace with a section of about 25 x 25 cm. Air was blown into the furnace by means of bellows.
- Within about 2 to 5 minutes the melting point of the alloy was reached. The coin mould containing the blanks was left to cool down in reducing atmosphere.
- The blanks were flattened, annealed and minted. Copper rich alloys which are very sensitive to oxydation during the melting process could be reheated in reducing atmosphere after the minting.

The analysis of Celtic gold took a central place within the recent project on "Prehistoric gold in the Czech Republic and Bavaria - Provenance and Technology" [1]. The project was supported by the *Volkswagenstiftung*, which is gratefully acknowledged. This report presents the reconstruction of the metallurgical processes used in Celtic times for the manufacture of precious metal coins. It focuses on three points :

1) archaeological remains of workshops in the *oppidum* of Manching,

2) analysis of the alloys, metallography of the coins and possible sources of raw material,

3) analysis of the technical ceramics, furnace fragments and coins moulds, involved in the manufacture process.

The results of this work permit a detailed reconstruction of gold alloying and minting in Celtic times. Melting and minting experiments were performed to verify the antique manufacturing processes. The analysed coins are from different sites in Central Europe, most of them were found in big hoards in Southern Bavaria. The studied pieces of technical ceramics stem from the excavations in the *oppidum* of Manching.

Archaeological remains of workshops in the oppidum of Manching

The late Iron Age (3rd to 1st c. BC) is characterised by a high industrial standard. The ability to invent new products or techniques largely depended on the transfer of techniques already in use to new areas of production. This development was favoured by the settlement structure of the *oppida*, as the towns of the Celts were named by the Romans [2]. The organisation of the *oppidum* enabled the specialized craftsman to earn his living without doing additional farming. The scale of industrial production in Celtic *oppida* was very large. Major changes can be observed mainly in metal working, but also in other industries such as woodworking or pottery production.

In the late Iron Age, industrial production appears with an unusual abundance of iron tools and products. The industrial structure is mainly based on the central position of the smith. The experienced iron smith produced objects and special tools for his own requirements as well as for other craftsmen. The situation in the *oppidum* facilitated a close cooperation with the other craftsmen. Tools made by the smith could easily be modified, embellished or perfected on the request of the other craftsmen. This close contact between different craftsmen encouraged the fast exchange of experiences and promoted the invention of new techniques.

The Celtic *oppidum* of Manching, near the modern town of Ingolstadt and south of the river Danube in Southern Bavaria, plays a prominent role in the discussion of Celtic town structures. Since 1955, systematic excavations have been conducted in Manching by the German Archaeological Institute. These excavations have brought to light essential parts of a densely populated settlement [3]. Estimates based on the number of excavated animal bones (remains of slaughtering) indicate that the population was between 5,000 and 10,000 people in the heyday of the settlement. The settlement was divided into quarters enclosed by fences or palisades. The analysis of various groups of finds shows that there were mainly two different types of quarters, one of predominantly agricultural and one of predominantly industrial use [4].

Remains of the working of bronze, iron and precious metals, especially of gold, have been found. However, iron working was most important [5]. Intensive iron work is proved in the north and in the south of the central excavation area, for instance by finds of iron slags, hammers, tongs, and anvils. In the south, a high concentration of such items has been found close to the borders of an enclosure built in the late 2nd c. BC. The northern part of this enclosure was a built-up area, while the southern part was not developed, presumably because of the risk of fire arising from the furnaces.

Indications of gold and silver melting are concentrated in two regions of the central excavation area (fig. 1), which are defined mainly by the distribution of coin moulds. The distribution is significant compared to the waste distribution in the central excavation area [6]. The unique find of fur-

1. Lehrberger *et al.* 1997.
2. Collis, 1984.
3. Krämer, Schubert, 1970.
4. Gebhard, 1993, fig.80 et 88.
5. Jacobi, 1974.
6. Gebhard *et al.*, 1991.

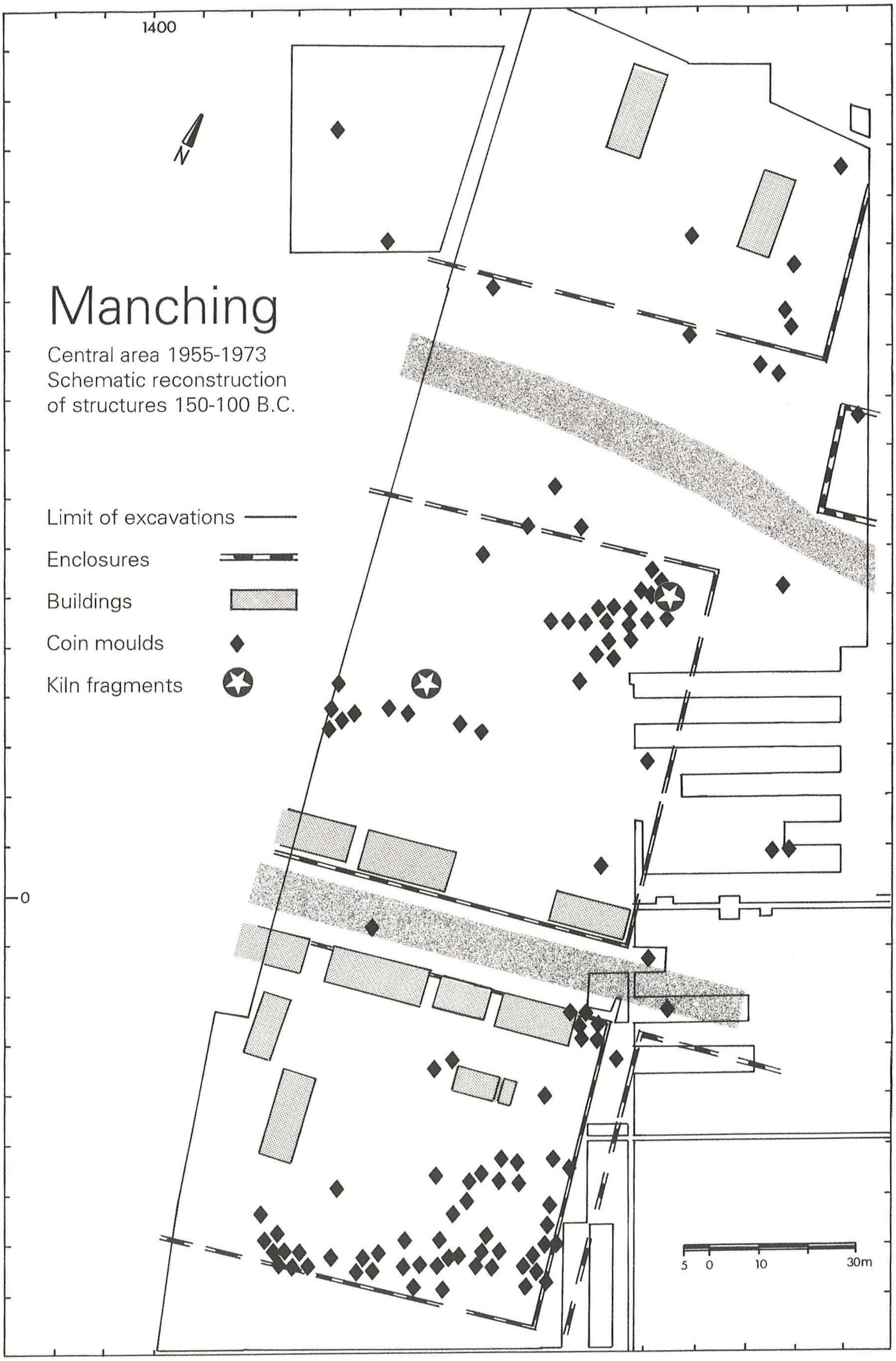
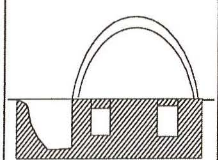
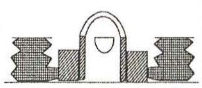
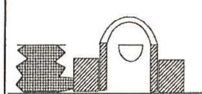
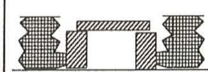
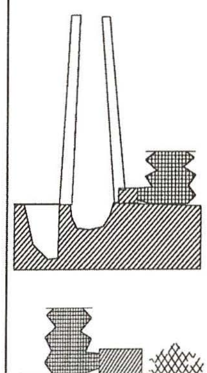


Fig. 1
Schematic plan of the settlement structures and remains of gold workshops in the central excavation area of the Celtic oppidum of Manching.

Fig. 2
Types of kilns and furnaces used in Celtic times.

	Pottery	Bronze	Glass/ Enamel	Gold/ Silver	Iron
temperatures	850-950 °C	800-1000 °C	~1050 °C	1100-1250 °C	1250-1350 °C
process		melting (secondary)	melting (secondary)	alloying	iron ore reduction
kiln atmosphere (cf. ceramic)	reducing oxidizing	reducing	reducing oxidizing?	reducing	reducing
kiln design	2 chambers 	bellow(s) 	bellow(s) 	bellows 	bellows 

nace fragments with gold and silver traces, datable to the 2nd c. BC, proves that the concentration of coin mould fragments coincides with the location of the melting furnaces. A comparison with the distribution of iron slags and nozzles of iron working furnaces shows that iron working was clearly concentrated in the southern enclosure in the same area where the gold was processed. In the middle part of the central excavation area there are two other areas of high concentrations of coin moulds and furnace fragments. These indicate gold working in a second enclosure. Here the buildings are in the southern half of the area and the working places are in the northern half. Remains of ironwork could be found here as well.

A study of the distributions of finds shows that a connection between iron working and precious metal working is likely. This relationship can be explained if one considers the necessity of the transfer of technical skills between the different crafts. The kilns and furnaces used for firing pottery, for iron working and for the melting of precious metals appear of particular interest in this context. We studied therefore excavated furnace structures, of which there are very few, and material from destroyed furnaces, *i.e.* wall fragments and *tuyères*. In this investigation X-ray radiography was used for the detection of metal inclusions and the metal composition of the inclusions found was determined by an energy-dispersive X-ray spectrometer attached to the scanning electron microscope (SEM-EDS). The analysis of the ceramic material by Mössbauer spectroscopy, thin section microscopy and X-ray diffractometry is very useful for reconstructing the temperatures that were reached during the firing process (fig. 2).

The construction of the furnaces is mainly determined by the requirements of controlling the temperature or atmosphere (fig. 2). Kilns for advanced pottery making have to be built mainly according to two aims, namely : a good control of the kiln atmosphere to produce reproducibly oxidized or reduced wares ; and to reach sufficiently high temperatures without the use of bellows, which was necessary because of the long duration of the firing cycle. The Celtic pottery kiln fulfilled both conditions in a perfect way. It was divided into two superimposed chambers. The main chamber holding the

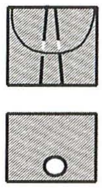
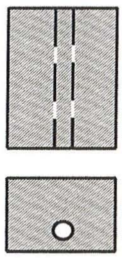
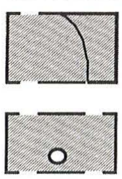
tuyères	scheme	clay	Ø nozzle	work
a) big square type with hemispherical cavity	furnace ⇑ bellow  >10 cm front 16 cm 13 cm	organic temper fired in reducing atmosphere.	2 cm	melting of *bronze* and *(?) glass.*
b) big square typ	furnace ⇑ bellow  18.5 cm front 13-15 cm 17-21 cm	organic temper fired in red. atmosph., vitrified surface, few metal inclusions.	1.3-1.5 cm	melting of *bronze* and *(?) glass.*
c) square type	furnace ⇑ bellow  ~7.5 cm front ~10 cm 22 cm	mineral temper oxidized on bellow side, vitrified surface on furnace side, metal inclusions (Au, Ag).	1.5 cm	melting of *gold* and *silver* .
d) tube type	furnace, hearth ⇑ bellow  >15 cm front Ø 6 cm	mineral temper 2.0 - mainly oxidized, contains iron slag on hearth side.	2.4 cm	working of *iron.*

Fig. 3
Shapes of *tuyères* excavated in the Celtic *oppidum* of Manching.

pottery was built as a dome above a firing chamber which was separated from the main chamber by a grid made of loam or clay. The small air volume of the dome with a diameter of about 1 m prevented heat loss, and the separate firing chamber facilitated the control of the air supply during the firing process. Firing was possible from one or two sides through a short tunnel. This construction provided a good control of the desired kiln atmosphere. The pottery products of the late Iron Age are excellent in material, shape, and the colours achieved by reducing or oxidizing firing.

Most furnaces used for metallurgical processes other than iron smelting were much smaller than the pottery kilns. They were not dug into the earth and usually destroyed completely after becoming useless. Thus fragments of such furnaces have to be used to reconstruct the original shapes and size.

The most common remains of furnaces are *tuyères* made of clay (fig. 3). They were exposed to high temperatures during their use in the furnaces. At points of extremely high temperatures, the clay surface became vitrified. Two types of *tuyères* were used for melting bronze. They consist of massive clay bricks with a tube canal inside. One type has a hemispherical cavity, obviously destined to support the crucible. The clay was mixed with organic temper and was reduced during the melting processes. The front end with the nozzle pointing toward the furnace has a partly vitrified surface. In most *tuyères* found the tube canal is blocked by vitrified clay and the *tuyère* must thus have become useless. Typical *tuyères* for iron working are made in the shape of tubes. The clay is strongly tempered with sand and mainly oxidized. These *tuyères* were probably used on an open hearth. The last type of *tuyères* or wall fragments could be connected with precious metal working. As yet, two sites with fragments of precious metal furnaces have been discovered in Manching (fig. 1).

All fragments were studied by X-ray radiographs, which reveal metal inclusions in the glass matrix of the side directed towards the furnace chamber. There are two wall fragments with nozzles, each of them totally vitrified on one side. The EDX micro-analysis of one of these fragments reveals two separate clusters, one of mainly gold and the other of silver inclusions. Presumably these were caused by a second nozzle on the opposite side which blew molten metal from the coin mould in the centre of the furnace. The second fragment could be used for the reconstruction of the side of the furnace. It can be completed to a brick of about 22 x 10 x 7.5 cm. The shape is very similar to the *tuyères* used in bronze work [7]. Another fragment from the same furnace can be identified as part of the bottom. It has a rectangular shape and is completely covered by spots of metal inclusions, mainly of silver. Different types of metal inclusions sampled from the bricks have been analysed by SEM-EDX. The metal inclusions consist of a variety of gold-silver alloys as well as pure silver and gold. These analyses correspond to the variety of gold-silver alloys found in the coin moulds. The information obtained from the furnace fragments allows a reconstruction of the furnaces used in precious metal working : between two opposed bellows there must have been a rectangular chamber of not more than 22-25 cm in width. This size fits quite well with the size of the coin moulds that were used for the melting of coin blanks. The largest coin moulds measure from 14x14 cm up to 17x17 cm. All coin moulds used in these furnaces have been exposed to strongly reducing conditions during their use (cf. below). Thus there must have been a cover on the top, probably a removable plate that facilitated reloading the furnace with charcoal. Analyses of the coin moulds and furnace fragments show that very high temperatures of up to 1100-1200°C have been reached in the centre of these furnaces.

Fig. 2 summarizes the firing techniques used in late Celtic industry. Craftsmen in the *oppida* were well experienced in handling temperature and furnace atmosphere. This was achieved by special design schemes. Furnaces for extremely high temperatures, necessary for iron and precious metal melting, were sustained with bellows. The highest temperatures had to be reached for the production of iron. Since bronze working is not very popular in Manching it can be assumed that the experiences from iron smelting formed the basis of the gold melting process.

Analysis of the alloys and metallography of the coins

A variety of techniques were used for the analysis of the coins [8]. Optical and scanning electron microscopy revealed surface structures and mineral inclusions in the coin alloy. EDX-analysis allowed to identify the chemical composition of the inclusions. Surface wave-length-dispersive XRF-analysis was used to determine the major elements in the alloy. Density measurements were conducted to control the reliability of the analyses. Minor and trace element analysis was done by optical emission spectroscopy and instrumental neutron activation analysis.

Polished sections of the cut coins were prepared for metallographic interpretation of the internal structures which reflect the deformed and heat treatment history of the coin. Additional micro-hardness measurements helped to reconstruct the processes of minting. EDX-line traces on the polished sections allowed to identify zonation and corrosion of the coin.

When performing surface analysis it is necessary to discuss the possible corrosion and enrichment features of buried precious metal coins. Comparisons between analyses of gold coin surfaces and the measured density of the coins indicated that the coins are enriched in gold at the surface. These observations had earlier been made for placer gold grains. We purchased five different types of gold coins for a detailed program of investigations, including cutting and preparation of polished sections, and micro-chemical analysis of the polished surface. The gold coins studied are listed in Tab. 1 below ; in the text they are referred to as Coins No. 1 to 4 following this list.

7. Jacobi, 1974, Nr. 1805-1808 : Traces of silver have also been found in Nr. 1808.
8. Lehrberger, Raub, 1995.

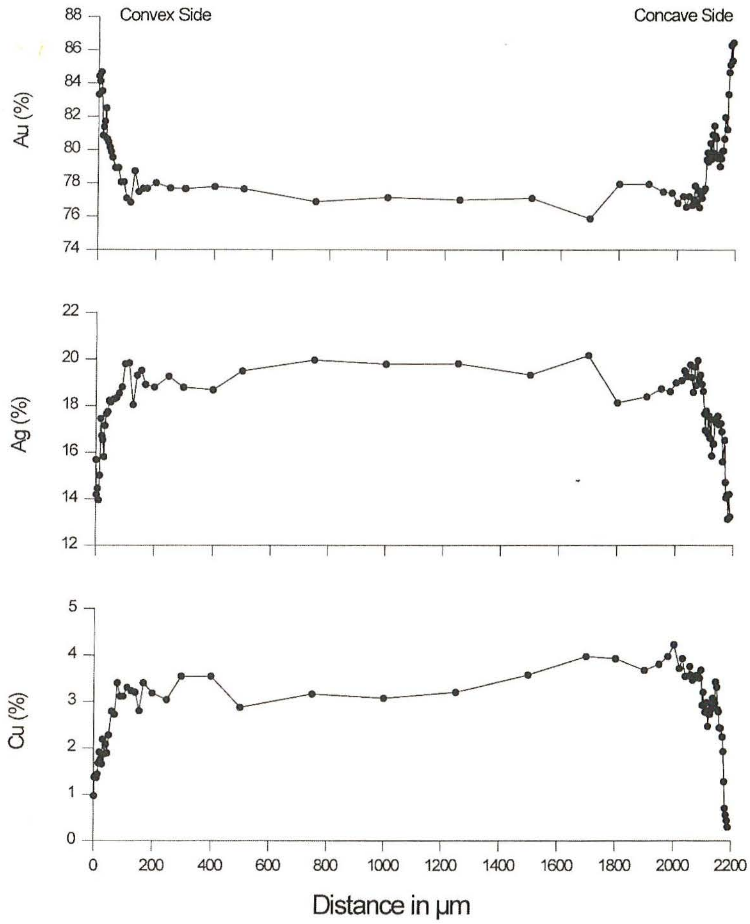


Fig. 4
EDS line traces of gold, silver and copper contents of Coin No. 1.

Table 1. List of investigated Celtic gold coins. (analyses in weight - %)

Coin 1 : 1/1 Stater, Regenbogenschüsselchen, Type VA (Southern German type),

Weight : 8.037 g, Composition (XRF) : 80 % Au, 16 % Ag, 4 % Cu.

Coin 2 : 1/1 Stater, Regenbogenschüsselchen, Type RIIE (Southern German type),

Weight : 7.504 g, Composition (XRF) : 57 % Au, 29 % Ag, 14 % Cu.

Coin 3 : 1/3 Stater, earlier gold minting (Boiian),

Weight : 2.747 g, Composition (XRF) : 96.7 % Au, 2.8 % Ag, 0.5 % Cu.

Coin 4 : 1/1 Stater, Mussel type ; earlier minting (Boiian type),

Weight : 7.230 g, Composition (XRF) : 97.6 % Au, 1.8 % Ag, 0.6 % Cu.

The main results of the analysis summarized here gave information on corrosion phenomena, details of the striking process and of the provenance of the metal.

Corrosion of gold alloys

Natural corrosion processes are well known from placer gold deposits under nearly all climatic conditions all over the world. Nuggets typically show alteration rims some tens of micrometres wide and are doubtless caused by silver leaching processes mainly under oxidizing conditions in the soils or sediments. The alteration rims show homogeneous composition and sudden changes can be observed at the reaction front from the silver depleted rim to the silver bearing core.

The scanning electron microscope allows to investigate the surfaces of objects at a very high resolution and, in combination with an X-ray spectrometer (EDX), the composition of the object can be directly measured at the spot seen on the screen. For quantitative analysis, however, polished surfaces are required for reliable results. Profiles have been measured across sections of the gold coins. The results (fig. 4) clearly show the surface alterations of the gold coins. Thus the data obtained from the surface are not representative for the composition of the alloy.

Therefore the results of the X-ray fluorescence analyses (XRF) have to be controlled by the measurement of the density in comparison to the calculated density using the data from the surface XRF-analysis. Density measurements of the coins, and a comparison with calculated density from surface analyses allow an estimate of the reliability of those analyses. The measurements were conducted with a laboratory balance in normal water with a few drops of a wetting agent. With leaching of Cu and Ag from the surface, the analysis of the Au-enriched surface would lead to a higher calculated density than really existing. The correction of the analytical results using the density measurements was firstly applied by Voûte [9]. Practically, the slices of coins show that the values from the density measurements of the whole coins correspond fairly well with the density calculated from analyses of the cores of the coins. This means that density is a feature of the coin, which is little affected by alteration processes and therefore can be regarded as more characteristic of the coin than surface analyses can ever be. Differences in density of more than 0.5 g/cm^3 indicate, that strong compositional differences between surface and core exist. In such cases it is not possible to perform a non destructive analysis.

Results of the metallography

The polished slices of the coins were etched with a cyanide solution and the structures studied under a metallographic microscope. The fabric of the coins will reflect the metallurgical history through the minting process and show the effects of the different stages : melting, preparation of the blank through forging, annealing and striking.

Coin No. 1 shows a fully recrystallized equiaxed grain structure with prominent annealing twins, typical of solid solution gold alloys. There is no marked gradient in the grain size indicating that the blank had been uniformly worked before heat treatment. Hot striking can probably be ruled out as this would have led to severe fire staining of the metal and, historically, mint practice avoids interfering with the coin after striking. Even if the blank was hot struck, it must have been cold forged and annealed first : it is improbable that temperature, strain rate and energy input would have been sufficient to produce the observed structure from an "as cast" blank. The porous zones observed just below both obverse and reverse surfaces will then be the result of the removal of copper oxide by acid blanching after annealing the cold hammered blank. It is possible that there were two or three cycles of cold working and annealing. A final annealing after striking may have been necessary because of an unsufficient reducing atmosphere during the hot working process. The copper oxides on the surface can finally be completely reduced by annealing the coins in charcoal.

Coin No. 2 shows a coarse and irregular grain structure penetrated by major cracks. The structure is typical for blanks which were molten and cooled slowly. This is supported by the observation of some dendritic segregation, which is still preserved in parts of the structure, with extensive interdendritic porosity generated by exsolution of gases and/or shrinkage during solidification. The habit of using unfired coin moulds to melt the blanks probably added to the evolution of gas. There are no traces of recrystallisation. The cracks are very probably the result of stress corrosion, something to which low carat gold alloys are rather susceptible.

Coin No. 3 shows a rather coarse recrystallised grain structure with annealing twins. The recrystallised grain size is a function of time, temperature and prior cold working ; grain size increases with annealing time and temperature but decreases with prior deformation. In this case the deformation before the last annealing step was the determining factor as annealing times were probably short and the temperature appears to have been insufficient to completely homogenise the structure.

Coin No. 4, the Boiian stater also has a fully recrystallised grain structure with a large grain size and some residual segregation (termed "coring"). In this case, though, the structure is severely deformed in the area illustrated with elongated grains, bent twin boundaries, and numerous deformation bands or slip traces. The cold reduction in this area since the last annealing is of the order of 30-40 %.

9. Voûte,1985 (see section 8, fig.10).

The interpretation of the metallographic data is greatly enhanced by micro-hardness testing ; the etch used on these alloys does not always successfully reveal slip traces, so moderate amounts of cold working or of precipitation and order hardening are not always detected.

Possible sources of raw material

Inclusions of natural alloys of platinum group elements (PGE) have been frequently observed in jewellery and coins from the Mediterranean and of Western Asia. W. Ogden [10] presented a first summary of the known PGE-containing artefacts. Meeks and Tite [11] analysed the PGE-inclusions in gold objects from the Eastern Mediterranean and the Near East with an energy dispersive X-ray fluorescence spectrometer attached to a scanning electron microscope. They found a whole variety of PGE alloys, mainly osmium, iridium and ruthenium. The analysis was in good agreement with the geological samples described by Harris and Cabri [12].

The PGE-inclusions in Celtic gold coins are very small and in most cases they can be identified only under an optical microscope. We use an optical stereomicroscope with a magnification range from 7 to 70 times. The steel-grey inclusions can be detected with some experience by their colour and hardness. The identification is facilitated by using a colour filter, *e.g.* a day-light film filter. The shape of the inclusions is irregular and the size varies from a few microns to approximately 1 mm. At higher magnification one can observe that large grains often consist of minute particles. The inclusions are confined to one side of the coin, the obverse or the reverse, which can be explained by the observation that the PGE-inclusions usually collect at the top of the gold drop during the melting process due to the surface tension of the gold melt. But compared to the mass of the gold object the size and the number of the inclusions are too small to influence the specific gravity of the object noticeably.

The analysis of the PGE-inclusions was performed with an energy-dispersive X-ray fluorescence spectrometer attached to a scanning electron microscope. The results show that the inclusions consist mainly of osmium, iridium and ruthenium forming different minerals according to their specific concentration [13].

The coin hoard of Wallersdorf, Lower Bavaria (1st half 2nd c. BC) with 366 rainbow-cup (*Regenbogenschüsselchen*) staters was partly examined, in order to get information on the quantitative occurrence of PGE-inclusions. We could identify PGE-inclusions in about one third of the coins but the total number of coins with PGE-inclusion could even be higher because the method described above is limited to certain particle sizes exceeding about 10 µm and to particles visible on the surface of the coins. It therefore seems very likely that the majority of the coins was made from gold containing PGE.

The association of gold with PGE-minerals indicates placer gold as the predominant raw material. In primary deposits PGE are usually present in ultrabasic rocks. Since those formations are found neither in the Bohemian Massif nor in the primary sources of the Bavarian rivers, platinum group elements do not occur in the placer gold deposits of this areas [14]. Therefore the gold used for the Celtic coins in Central Europe can only be of foreign origin. Possible placer deposits containing PGE-minerals can be found in Western Europe, the Eastern Mediterranean and the Middle East [15]. The composition of the PGE-grains does not allow to determine the gold sources more exactly, but archaeological evidence and historical reports may help to answer the question how the gold came to southern Germany.

Systematic geological research on the occurrence of PGE in European placer gold deposits is still missing, but so far no considerable PGE-deposits are known for Western Europe. A first investigation of randomly selected early Gaul gold coins, which obviously consist of local gold [16], shows no PGE-inclusions. In contrast, they seem to be common in jewellery and coins from the Mediterranean and Western Asia [17].

In Southern Germany pre-Celtic gold objects never contain PGE-inclusions. Such inclusions are confined to Celtic coins and do not occur until the late 3rd c. BC, when the Celts started to mint their own coinage. The introduction of coinage in Central and Western Europe is a result of the various contacts between the Hellenistic and Celtic world. The first Celtic gold coins were imitations of

10. Ogden, 1977.
11. Meeks, Tite, 1980.
12. Harris, Cabri, 1973.
13. Steffgen *et al.*, 1995.
14. Lehrberger, 1994.
15. Hatzl *et al.*, 1990.
16. Hartmann, Nau, 1976.
17. Williams, Ogden, 1994.

posthumous staters of Philip II of Macedonia (Gaul) and staters of Alexander III (Bohemia, Moravia), which were also minted by his successors. Since the 4th c. BC. Celtic mercenaries have been common in the Mediterranean area. Their number increased during the 3rd c. BC, when each Hellenistic army had its contingent of Celts. Information on the payments of these troops is very scarce but they were probably paid in local coinage. These local coins may have contained PGE-inclusions as, for example observed in Greek staters. The raw material for the Hellenistic coins may stem from the enormous amount of gold Alexander the Great seized after the conquest of the Persian empire. The Persians obviously exploited PGE-bearing gold deposits since PGE-inclusions are frequent in Western Asian jewellery and coins. But this assumption is just one of several possible explanations because Persian gold coinage had been widespread long before Alexander's campaign and such coins may have been molten down not only by him and his successors but also by other sovereigns and states. The famous gold of the river Rhine is also known to contain PGE-minerals, but so far there is few of them.

Analysis of the technical ceramics, furnace fragments and coins moulds involved in the manufacture process

Analytical Methods

The reconstruction of the metallurgical processes used in Celtic times for the manufacture of gold objects must largely be based on the scientific analysis of the gold and of the ceramic material involved in the production process. The Celtic coins were struck with bronze or iron dies from blanks of the proper weight by melting gold particles or pieces, usually mixed with some copper or silver metal. For the melting of such blanks, the Celtic craftsmen used flat clay plates with small circular or square depressions on the upper side (fig. 5).

The use of these coin moulds was widespread throughout the Celtic *oppidum* civilization, even in peripheral areas like southern Poland. Maier and Neth [18] have listed 46 sites in Central and Western Europe where coin moulds have been found ; about half of these sites are *oppida*. The connection of these early examples of industrial ceramics with precious metal metallurgy is fully confirmed by gold and silver inclusions which can often be seen even with the naked eye on the surface of the coin moulds and fragments of hearth lining.

Metallurgical studies of the microstructure of Celtic gold coins have shown [19] that the raw materials for the coin blanks were heated to or above the point of fusion of the respective alloy rather than being merely sintered, for which significantly lower temperatures would have been sufficient. Since many Celtic gold coins contain only a few percent of silver or copper, melting required that a temperature of or even exceeding 1000 °C has to be reached. This, as well as the archaeological evidence, suggests that the hearths were fired with charcoal and operated with bellows to supply the air required for obtaining these high temperatures.

Although several studies of the ceramic material and the metal inclusions of coin moulds have become known [20], no attempt has been made at a systematic scientific analysis to extract all the available

18. Maier, Neth, 1987.
19. Lehrberger, Raub, 1995.

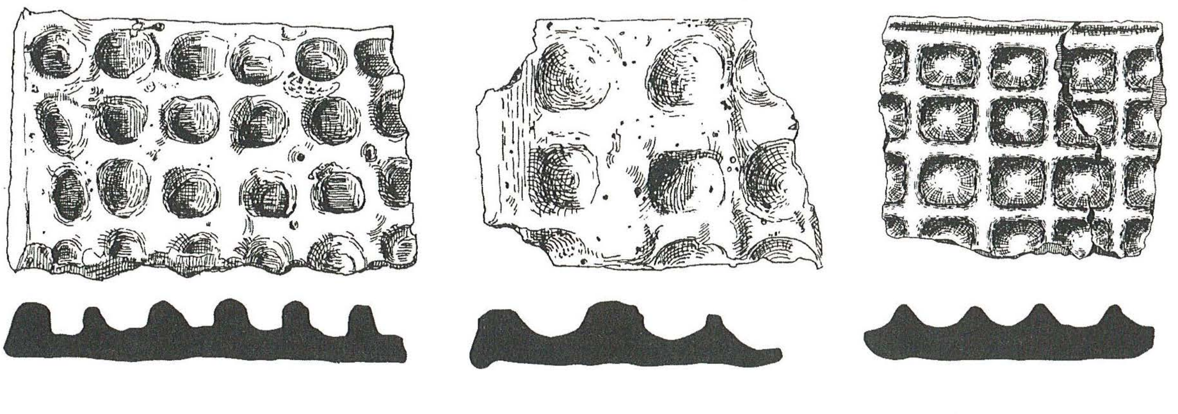


Fig. 5
Coin moulds from the excavations in the Celtic *oppidum* of Manching.
Scale 1:2.

information on the melting techniques used in Celtic times [21]. The results presented here were achieved by a study of both coin moulds and ceramic material from Celtic precious metal melting furnaces excavated in the *oppidum* of Manching.

Neutron activation analysis (NAA) has been used to characterize the clay from which the ceramics were made by their major and trace element contents. Optical thin section microscopy (TSM) gives information on the mineral contents of the ceramic material. Scanning electron microscopy (SEM) provides further information on details of the structure of the ceramic material and reveals how the metal inclusions found in the coin moulds are embedded into the largely vitrified ceramic matrix, while X-ray radiography (XRR) turned out to be a convenient, non-destructive method for studying the distribution of metal inclusions below the surface of the coin moulds. Energy dispersive X-ray fluorescence analysis (EDX) performed in the scanning electron microscope yielded information on the alloy composition of these inclusions. Finally, Mössbauer spectroscopy (MOS) provides information on the chemical and physical state of the iron in the ceramic material, and thus indirectly on the furnace atmosphere and the temperatures to which the coin moulds were exposed during the gold melting process.

Metal Inclusions in Coin Moulds

Sometimes small spherical grains of precious metal are visible on the surface of the coin moulds but, more often, these metal inclusions are hidden in the vitrified clay matrix. In order to make a survey of such inclusions non-destructively, X-ray-radiographs were made of about 150 fragments of coin moulds from Manching with a commercial X-ray tube operated at a voltage of 60 kV. Owing to the strong X-ray absorption of gold and other metals, these radiographs show the metal inclusions as white spots.

A microscopic examination of the radiographs allows us to study the number, size distribution and morphology of the inclusions. A preliminary evaluation shows that most of the inclusions are smaller than 0.3 mm. About 40 % of the coin moulds studied were found to contain a high density of inclusions, mainly in the walls of the pits. The abundance of such metal inclusions suggests that the gold dust melted in the coin moulds was not or, at best, very loosely compacted before the melting process.

In about 10% of the coin moulds no precious metal inclusions were observed, while the remainder had small to moderate precious metal contents. Most of the precious metal inclusions are not on but below the upper surface of the coin moulds. In most cases this surface is vitrified because of the high temperatures sustained during the melting process.

A study of the surface of the coin moulds by scanning electron microscopy (SEM) revealed that the vitrified zone of the ceramic material has typical hot tears. These are best developed where metal inclusions are found at the surface. The metal inclusions form rounded droplets, which are often slightly flattened. Normally only about one half of a droplet protrudes from the ceramic surface while the rest has sunk into the vitrified mass.

Energy dispersive X-ray analyses (EDX) performed in the scanning electron microscope reveal a broad spectrum of different precious metal alloy inclusions in the coin moulds. Au-Ag-Cu alloys with Au contents between 55 and 80 wt.%, Ag contents from 15 to 33 wt.% and Cu contents from 4 to 15 wt.% are predominant. A few of the analysed droplets were found to be pure silver, which indicates that pure silver was deliberately added to the gold alloys. An important result of EDX is the detection of metallic tin and tin-copper bronze inclusions in the coin moulds. In the vicinity of the tin-bearing inclusions one also observes the formation of needle shaped crystals in the vitrified matrix. These crystals have been identified as tin oxide needles formed by the oxidation of tin originally contained in the bronze, while the copper was completely incorporated into the Au-Ag-Cu alloy of the coin blanks. The occurrence of tin oxide and of bronze inclusions in the coin moulds is taken as an indication that bronze rather than pure copper was used as a source of copper in making the desired coinage metal alloys.

Optical Microscopy

In the interpretation of the micrographs, results from a continuing study of pottery from the *oppidum* of Manching [22] have been of assistance. One group of the pottery sherds from Manching is characterized by the presence of large amounts of

20. Jansová, 1974 ; Tournaire *et al.*, 1982 ; Raub, Fingerlin, 1984 ; Maier, Neth, 1987.
21. Tylecote, 1962 ; Castelin, 1960 ; Castelin, 1965.
22. Bott *et al.*, 1994a ; Bott *et al.*, 1994b.

quartz and muscovite. Another is rich in quartz, muscovite, plagioclase, microcline and biotite.

Graphite ware, which is frequently found in Manching, often belongs to this type of ceramics, but additionally contains graphite flakes. A typical feature of all coin moulds examined is the presence of quartz grains with strikingly sharp edges, which suggests that these grains are the result of a deliberate crushing of larger quartz pieces. Presumably this quartz was added on purpose as a temper. Whether this was done to produce particularly temperature resistant ceramics or for a different as yet unknown reason, requires further clarification, since it might give interesting insights into the technical skills of the Celtic craftsmen.

Occasionally, grains of garnet are found in the thin sections. Garnet can be associated with sands found in the Manching area, which may have been used as temper. The presence of garnet is of some interest because the Mössbauer spectra also show the presence of a mineral which, on the basis of its Mössbauer parameters, can be identified as a garnet, presumably almandine with a non-ideal composition.

Mössbauer Spectroscopy

Mössbauer spectroscopy with the 14.4 keV g-rays of ^{57}Fe is a convenient method for studying the chemical and physical state of iron, which usually is present in percent concentrations in clays and ceramics [23]. The sensitivity of Mössbauer spectra for the solid state properties of the absorber material results from the fact that hyperfine interactions between the ^{57}Fe nuclei and their electronic environment split and shift the resonance line in a way that allows conclusions to be drawn, for example, on the oxidation state of iron, on the symmetry of the environment of the iron atoms, and on the magnetic properties of iron compounds. Since the hyperfine patterns of the various iron-bearing mineral phases usually present in ceramics are known, Mössbauer spectroscopy allows one to identify these mineral phases even in complicated mineral mixtures. An advantage of the Mössbauer method, for instance over X-ray diffraction, is that natural iron contents of about one percent are sufficient for obtaining good Mössbauer spectra without interference from the major mineral phases that do not contain iron. Moreover, the iron yields informative Mössbauer spectra even in cases where X-ray diffraction would fail altogether, for instance if iron is present as colloidal oxide particles or in a vitrified silicate matrix.

The Mössbauer spectra of ceramic materials usually are a superposition of the Mössbauer patterns of the various iron-containing phases present. The typical changes in the Mössbauer spectra when clays are fired have been studied in some detail [24]. In unfired clay minerals the iron may be either trivalent or divalent [25]. In technical clays, some iron is usually present in form of oxides, mainly as hematite and more rarely as magnetite, or as oxyhydroxides like goethite or ferrihydrite. When such clays are fired in air, the oxyhydroxides start to transform into hematite at about 400 °C. Such hematite often consists of small particles which show super-paramagnetic behaviour in the room temperature Mössbauer spectra. The clay minerals undergo dehydroxilation between about 300 and 500 °C. This usually goes along with an increase of the quadrupole splitting of the Fe^{3+} in the clays, which may reach values as large as 1.5 mm/s [26]. Above 800 °C the layer structure of the clay minerals breaks down altogether and the iron initially contained in the clay minerals tends to form hematite, except in clays that are rich in calcium, which causes the formation of gehlenite, $Ca_2[(Al,Fe)_2SiO_7]$ [27].

Above about 1000 °C, vitrification occurs and the hematite tends to dissolve in the glassy matrix. Firing in reducing atmospheres, as are often encountered in pottery kilns and are also expected in the furnaces studied in the present work, leads to the formation of divalent iron, mainly in the form of minerals which have rarely been identified. A compound that seems to play an important role in this context [28] is the spinel hercynite, $FeAl_2O_4$. Hercynite is known to form from oxides of iron and aluminium in a reducing CO/CO_2 atmosphere at temperatures near 1000 °C [29]. When ceramics that were first fired in a reducing atmosphere are fired again in an oxidizing environment, the iron in the Fe^{2+} containing minerals transforms into hematite. On the other hand, hematite formed by oxidizing

23. Wagner *et al.*, 1986.
24. Wagner *et al.*, 1988 ; Wagner *et al.*, 1994.
25. Coey, 1980 ; Murad, Wagner, 1991.
26. Murad, Wagner 1989 ; Murad, Wagner, 1991.
27. Maniatis *et al.*, 1981.
28. Bott, 1992.
29. Kunnmann *et al.*, 1963.

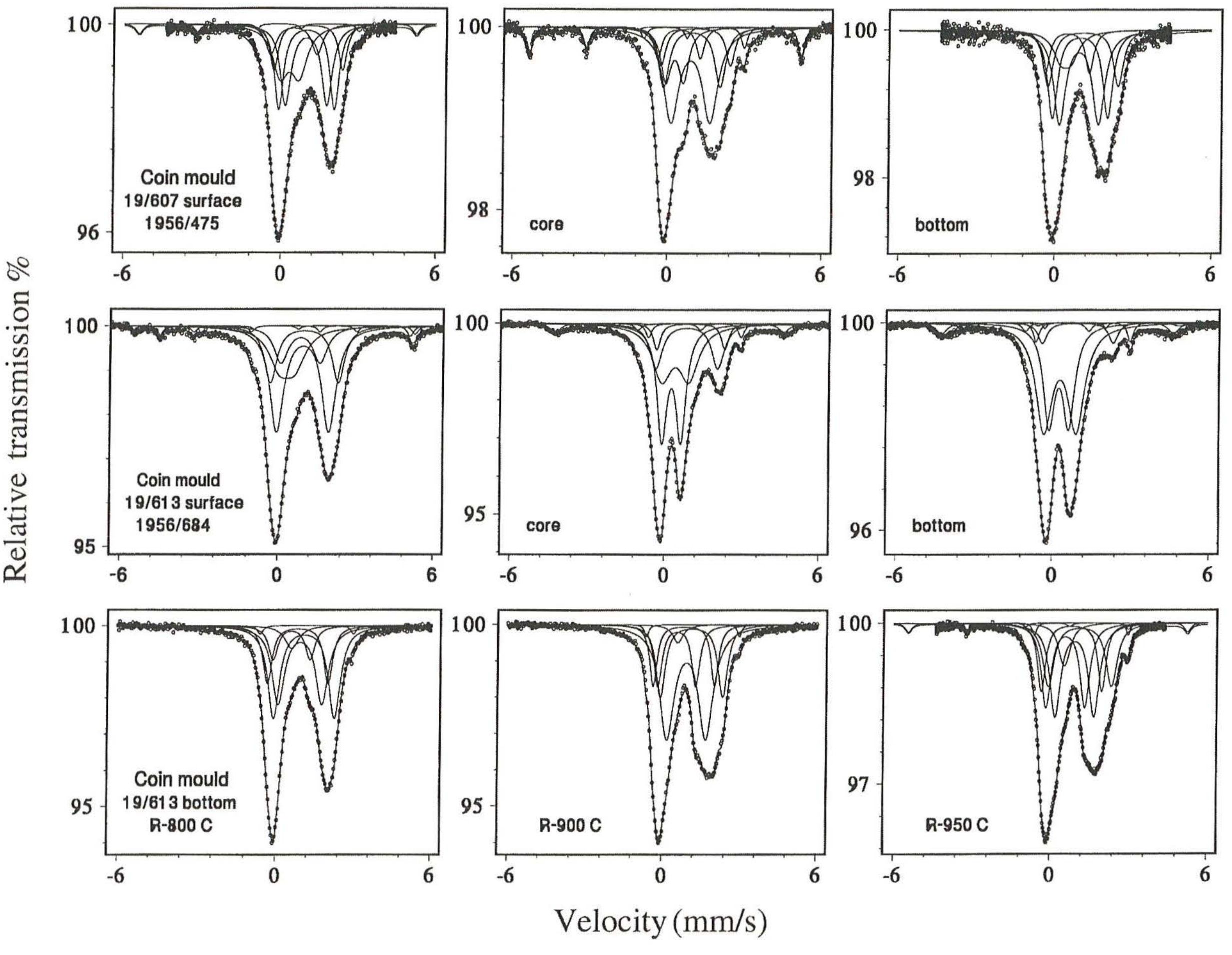


Fig. 6
Room temperature Mössbauer spectra of material from the surface, the core and the bottom part of coin moulds 19/607 (top) and 19/613 (middle). Spectra of material from the bottom of coin mould 19/613 after reduction at 800, 900 and 950°C for 3 h. are shown in the bottom row.

firing can be transformed into Fe^{2+} containing minerals by firing in a reducing atmosphere [30].

In the case of the coin moulds, the microscopic studies have already shown that the upper faces are vitrified, obviously because they had to sustain higher temperatures than the inner and bottom parts. For this reason, samples for Mössbauer spectroscopy were taken separately from the vitrified upper surface, from the core, and from the bottom of each of the studied coin moulds. Mössbauer spectra were measured at room temperature (RT) and in many cases also at 4.2 K with sources of ^{57}Co in Rh, which were kept at the same temperature as the absorber.

The individual layers of ten coin moulds were studied by Mössbauer spectroscopy. The surface layer usually shows a noticeable degree of vitrification, the core is of grey colour, while the bottom is often reddish. Some results are shown in fig. 6.

The Mössbauer spectra of the surface, the core and the bottom of the coin moulds can be interpreted consistently, though there are interesting differences between individual coin moulds. Coin mould 19/607 is somewhat uncharacteristic, as its bottom part is grey. The fractional area of Fe^{2+} in the room temperature Mössbauer spectra of its surface layer (fig. 6, top row) is 72 % and does not vary much between the core and the bottom, indicating that this mould was strongly reduced from top to bottom. An interesting feature is the presence of iron metal in the surface layer and in the core. The metallic iron can easily be identified by its hyperfine field of 33 T. Its fractional area is 12 % for the core. In order to survive 2000 years of burial in the ground without oxidation, the iron particles must have been protected by a tight cover.

For coin mould 19/613, only the Mössbauer spectrum of the surface layer shows 5.6 % of iron metal (fig. 6, middle row). In 19/613 the surface is

30. Bott, 1992 ; Bott *et al.*, 1994a.

completely reduced with a fractional area of 72 % Fe^{2+}-silicates and an additional Fe^{2+}-component which, according to its Mössbauer parameters, may be wuestite. The core and the bottom are less strongly reduced, the Fe^{2+}-species being only 22 and 12 %, respectively. An iron-bearing component with a large quadruple splitting of 3.6 mm/s is observed in material from the core and from the bottom.

This component was identified as almandine by its Mössbauer parameters [31] and by thin section microscopy. Almandine is a garnet stable during firing up to about 950 °C in oxidizing as well as reducing atmosphere. The presence or absence of almandine in different layers of a coin mould can therefore be used as an indicator for the temperature experienced by the mould in antiquity.

Material from the bottom of coin mould 19/613 was used for re-firing experiments at different temperatures and in varying atmospheres. Up to 500 °C, re-firing in air reflects the oxidation of the Fe^{2+}-component, and increases the fractional area of the Fe^{3+}-doublet. The important observation, however, is that the Mössbauer patterns still change on heating above 700 °C, indicating that the temperature experienced by the bottom part of the coin mould in antiquity had not exceeded 700 °C. Almandine disappears between 900 and 950 °C.

Samples from the bottom of coin mould 19/613 were also fired in a reducing environment by heating them with charcoal in closed vessels for 3 h. Spectra taken after reduction at 800, 900 and 950 °C are shown in the bottom row of fig. 6. With 89 % of the iron in the form of Fe^{2+}-components, reduction is almost complete already at 800 °C. Iron metal begins to form at 900 °C and is up to about 12 % after reduction at 950 °C. As during re-firing in air, almandine disappears between 900 and 950 °C.

Field experiments

From the data of the laboratory investigations the following conditions for the melting of coin blanks can be inferred. The coin moulds were used in a strongly reducing atmosphere. The difference in the temperature reached at the top and at the bottom of the coin moulds suggests that they were embedded in glowing charcoal and then air was blown from above with bellows to briefly reach the temperature required for melting the metal pieces or dust into bulk ingots.

To confirm this notion, field experiments were performed in a replica furnace using replica coin moulds [32]. During this experiment, the tempera-

31. Murad, Wagner, 1987.
32. Gebhard *et al.*, 1996.

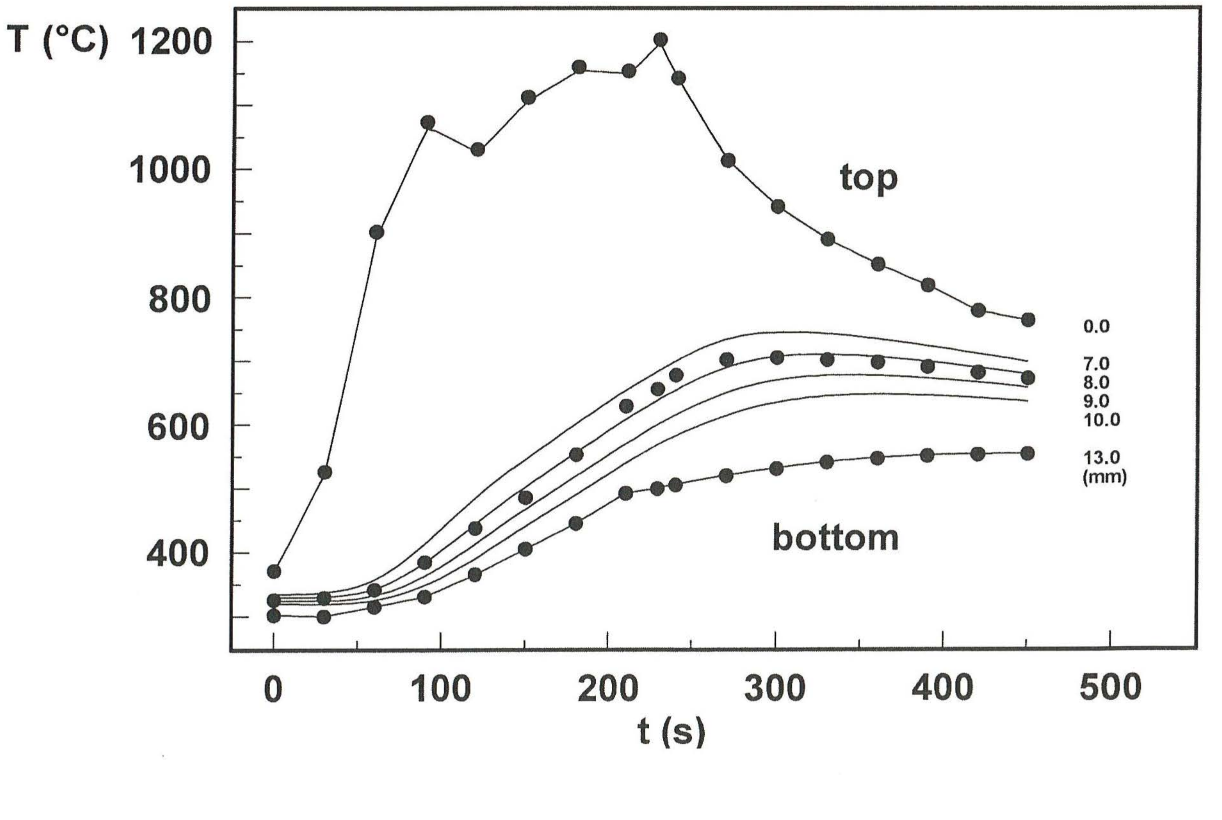


Fig. 7
Temperatures recorded during the field firing of a replica furnace at the top, the core and the bottom of a replica coin mould. There is good agreement between temperatures measured in the core (8 mm from the top), and the temperatures simulated by solving the heat transfer equation.

tures in the upper surface layer, in the core and in the bottom layer of the replica coin mould were measured by thermocouples. The coin moulds were covered with red glowing charcoal and air was blown into the furnace. Pieces of gold and silver could be melted within three to five minutes. The temperatures recorded during the firing process (fig. 7) confirm the conclusions from Mössbauer spectroscopy that only the uppermost part of the coin mould reached temperatures near 1000 °C, while the core and the bottom reached temperatures between 400 and 800 °C only. The observed temperature distribution in the coin moulds can be simulated by solving the one dimensional heat transfer equation using the measured temperatures at the top and at the bottom of the mould as boundary conditions and a value of $l = 0.0034$ W/(cm K) for the thermal conductivity (fig. 7).

From the Mössbauer data for many of the coin moulds one concludes that the hearth must have been allowed to cool under reducing conditions. If it had been opened before the temperature had fallen below about 400 °C, the divalent iron in the ceramic material would again have oxidized to trivalent iron. During this cooling process, the temperature must have dropped so rapidly that the oxidized bottoms and cores of the coin moulds did not become hotter than about 600 °C by heat conduction from the top.

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