

Mössbauer and XRD studies of Roman amphorae buried in the sea for two millennia

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Abstract During the years 2004–2007 many Roman amphora sherds were found in the sea near Cortiçais, off the southern coast of the Peniche peninsula on the Atlantic coast of Portugal. The amphorae are of the Haltern 70 type and stem from a shipwreck that has been dated to the time of the emperor Augustus, between about 15 BC and 15 AD. They were produced in the Roman Province of Baetica in the south of Spain and used to transport wine and other staple foods by sea to other Roman settlements. We have studied several fragments of these amphorae by ⁵⁷Fe Mössbauer spectroscopy and X-ray diffraction in order to look for changes in the ceramic material caused by two millennia of exposure to sea water. For comparison Mössbauer data on a Haltern 70 type amphora handle excavated on land at the site of Castro do Vieito in the north of Portugal were used. The fragments on which we report here are a body sherd and two handles. The sherds show a visible layer structure. The different layers were studied separately. The Mössbauer spectra of the buff surface layers indicate that up to about 60 % of the iron is present as very fine goethite particles, which are superparamagnetic at RT but exhibit magnetically split spectra at 4.2 K. Their blocking temperature is around or even below 50 K. The goethite is too fine grained to be detected by

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X-ray diffraction. Re-firing experiments confirm the presence of goethite, which is found to convert to hematite between 300 and 600 °C. The results show that the iron in the silicate matrix of the ceramic material converts to goethite under the prolonged influence of the sea water, mainly in the outermost several millimetres and apparently depending on the nature of the ceramic material.

Keywords Mössbauer spectroscopy · Haltern 70 Amphorae · Marine archaeology · Shipwreck · Cortiçais · XRD

1 Introduction

During the years 2004–2007 many Roman amphora sherds were found in the sea at the site of Cortiçais on the southern shore of the Peniche peninsula, on the Atlantic coast of Portugal. Geomorphology combined with historical and geographical data indicate that the Peniche peninsula of today was an island in ancient times [1]. The site of Cortiçais is located on a rocky coast facing south, exposed to occasional high energy marine action from SW, but protected from the predominant N and NW winds and the related swell. Lying among rocky shoals forbidding any kind of navigation, the sea bottom at the location is at a depth of 4–7 m, depending on the tide level. The underwater site was formally identified and geo-referenced in late September 2004 under the direction of Jean-Yves Blot, in collaboration with Luís Jorge, the original finder. On that occasion, archaeologist and ceramics expert A. Dias Diogo examined the lot of ceramic fragments and identified the amphorae as being all of the Haltern 70 type. The amphorae are assumed to have been produced in the Roman Province of Baetica in the south of Spain and used to transport wine and other staple foods by sea to other Roman settlements. The first recovery campaign took place in May 2005. In June 2006 another campaign was organized [2, 3]. The isochrony of the different types of ceramic fragments led to the conclusion that the finds stem from a single shipwreck. The fine ware found in addition to the amphora fragments supports a date around the transition of the era (15 BC to 15AD). The underwater campaigns were supplemented by two campaigns for cataloguing and inventory of the finds performed in November 2005 and 2006. Unfortunately after 2006 no more work could be performed at the site. The fragments of Haltern 70 amphorae represent 98.8 % of the recovered ceramic assemblage [3]. Exposure to major marine events from SW and related cyclical sedimentary and erosive processes during the past two millennia led to characteristic features including primary fracture observed on mostly un-abraded fragments, secondary fracture related with severely abraded fragments, abrasion and rupture.

The amphora finds from the Cortiçais shipwreck provide material for a study of the modifications which ceramic material undergoes during prolonged exposure to sea water. As a reference, land-buried Haltern 70 amphorae from the site of Castro do Vieito are available [4, 5]. Castro do Vieito is situated on the banks of the estuary of the River Lima, on the northern coast of Portugal. About two-thirds of the total area of this archaeological site was affected by the construction of the A28 motorway in 2005, which led to a large scale archaeological intervention between June 2004 and July 2005 directed by A. J. M. Silva [4–6]. The study of these archaeological remains showed that the settlement had a very short period of occupation, corresponding to the very earliest stages of the integration of north-western Iberia into the *orbis romanum*, i.e., from the reign of Augustus until the middle or third quarter of the 1st century AD. Castro do Vieito's setting at a strategic location on the river estuary, close to one of the region's largest mineral veins, made this settlement an

Table 1 Haltern 70 Amphora fragments studied in the present work

Sample no.	Type	Origin	Remarks
CV.A.4125	Handle	Castro do Vieito	Land buried, well preserved
2469	Handle	Cortiçais shipwreck	Well preserved; uniform
823	Handle	Cortiçais shipwreck	Well preserved; pink rim
3066	Body sherd	Cortiçais shipwreck	Strongly abraded

The sample numbers are those given the objects in the archaeological record [3–5]

important node in the military supply network. Regular supplies were needed for the military personnel who controlled prospecting for gold upstream on the River Lima. Even if a significant quantity of food was acquired locally, “comfort food” was imported from other places that might be very distant but that had been part of the empire for a long time. These familiar foodstuffs ensured that the soldiers kept their *humanitas* within an environment of “barbarians”, because the food regime was one of the factors that distinguished civilized men [7]. Amphorae used for food transport are therefore the most common exogenous products found, and the majority belongs to the Baetican Haltern 70 type. It is, in fact, the largest assemblage of amphorae of this type found to date in the whole of the Roman empire. This particular type had an important function in the military supply system, which largely depended on river access in order to reduce the cost of transporting goods between the coast and the inland area where the greater part of the permanent soldiers were billeted [4, 8].

First Mössbauer and X-ray diffraction studies of amphorae found at Castro do Vieito and at the kiln sites in southern Spain have been published [9–11]. The present paper will give a first report on first results obtained on the amphora finds from the Cortiçais shipwreck.

There is only a limited number of Mössbauer studies of ceramics found in the sea. A Mössbauer study of amphorae from the shipwreck of the Madrague de Giens off the coast of southern France [12] showed that under conditions of free access of sea water Mg is deposited in the ceramics as hydrotalcite while K is dissolved. In ceramics covered by marine sediments, the formation of pyrite and sulfates was observed. A Mössbauer study of pottery buried under lagoon conditions [13] reports pyrite and jarosite as alteration products. A laboratory study of the effects of sea water on ceramic material [14] has demonstrated a tendency for the deposition of Mg and the dissolution of Ca present as calcium carbonate.

The ceramics from the Cortiçais shipwreck were found in a part of the sea where the sea water has free access and not much influence of decomposing organic matter is to be expected. The focus of the present paper is on the transformations which the iron in the ceramic material undergoes under such circumstances. We shall demonstrate that the main effect of the sea water is the formation of very fine grained goethite, which is superparamagnetic at ambient temperature and nearly impossible to discover by X-ray diffraction because of its small grain size, but whose presence can be easily verified by Mössbauer spectroscopy at liquid He temperature.

2 Samples and experiments

We present data on three fragments of Haltern 70 amphorae from the Cortiçais shipwreck and, for comparison, on an amphora handle buried and recovered on land at the Castro do Vieito archaeological site (Table 1 and Figs. 1 and 2).



Fig. 1 View of the amphora handle CV.A.4125 from Castro do Vieito. The surface facing upwards resulted from cutting off a piece to expose the interior for sample taking. The handle is on the right, the surface facing left is the inside of the neck of the amphora

The amphora handle CV.A.4125, which bears a workshop mark [4, 5] was cut with a diamond saw in such a fashion that a surface reaching down to the core was laid open. From this cutting face, samples at different distances from the surface were removed with a diamond grinder for Mössbauer measurements at RT and 4.2 K.

All ceramic material from Cortiçais was submitted to a desalination process at the Peniche Museum in order to remove salts, especially sodium chloride. If artifacts from maritime sites are not carefully desalinated, salt crystals may cause the original material to break apart. The desalination lasted for three years, between November 2007 and October 2010, with regular exchange of water. The amount of salts in the desalination baths was monitored by conductivity measurements.

The strongly abraded body sherds from Cortiçais typically have a thin brown, sometimes spotty surface, a buff layer on both surfaces that is 1.5 to 3 mm thick and a grey core. The typical thickness of these sherds is 7 to 10 mm, which is only about half the normal thickness of the walls of Haltern 70 amphorae. Presumably, the amphorae broke in the course of the shipwreck and were abraded by the wave motion during burial in the sea. This is confirmed by the rounded fracture rims. The buff surface goes all around the sherds, indicating that the buff color is due to alterations caused by exposure to the sea water. To study the individual layers separately, first the brown surface was recovered with as little of the underlying ceramic material as possible, removing about 0.3 mm. Sherd 3066 was then subdivided into 7 layers by removing layer by layer from both sides with a diamond grinder. The curvature of the sherds indicates that the outside of the amphora is facing upwards in Fig. 2. Mössbauer spectra of the individual layers were recorded at room temperature (RT) and 4.2 K. Samples of the buff layer near the surface and the grey core of 3066 were also subjected to annealing in air at temperatures between 300 and 900 °C increasing in steps of 100 °C.

The amphora handles from Cortiçais also exhibited brown spots on the surface that were first removed separately. Sections of both handles were then subdivided into layers, beginning on the outside and removing layers about 1 mm thick to a depth of about 10 mm, which is practically the center of the handles. The pink layer of handle 823 (Fig. 2) was subdivided into an outer and an inner half. Handle 2469 does not show such a pink surface layer, but a slightly yellow discoloration reaching 2 to 3 mm into the ceramic material.



Fig. 2 View and fracture edge of the strongly abraded body sherd 3066 (*left*), and sections through the amphora handles 823 and 2469 (*right*)

Mössbauer spectra were recorded in transmission geometry with a $^{57}\text{Co}:\text{Rh}$ source of about 25 mCi and a spectrometer operating with a sinusoidal velocity waveform. The absorbers usually had a thickness of 200 mg/cm^2 . Only those of the brown surface spots were made thinner (mostly 50 mg/cm^2) because of the high iron content. Measurements at 4.2 K were performed in a liquid He bath cryostat with the source kept at the same temperature as the absorber. Isomer shifts are given with respect to the source having the same temperature as the absorber.

X-ray diffraction patterns were taken of several of the different layer samples to obtain information on the mineral content that cannot be gained by Mössbauer spectroscopy. The XRD patterns were recorded with $\text{Co K}\alpha$ radiation source using a Philips PW1070 diffractometer equipped with a graphite monochromator from 5 to $80^\circ 2\theta$ in steps of $0.02^\circ 2\theta$ with a counting time of 5 s for each step.

3 Results and discussion

3.1 Mössbauer spectroscopy

Figure 3 shows RT and 4.2 K Mössbauer spectra of amphora handle CV.A.4125 from Castro do Vieito, which will serve as reference representing unaltered ceramic material of such artefacts. The spectra of the different layers are quite similar, although the color changes very slightly from buff at the surface to brownish below the surface, then reddish and eventually grayish. The RT spectra all consist of a dominant ferric quadrupole doublet with a quadrupole splitting of $QS \approx 0.9\text{ mm/s}$ and an isomer shift of $IS \approx 0.26\text{ mm/s}$. A magnetic sextet of about 30 % of the spectral area is a mixture of hematite and maghemite, whose

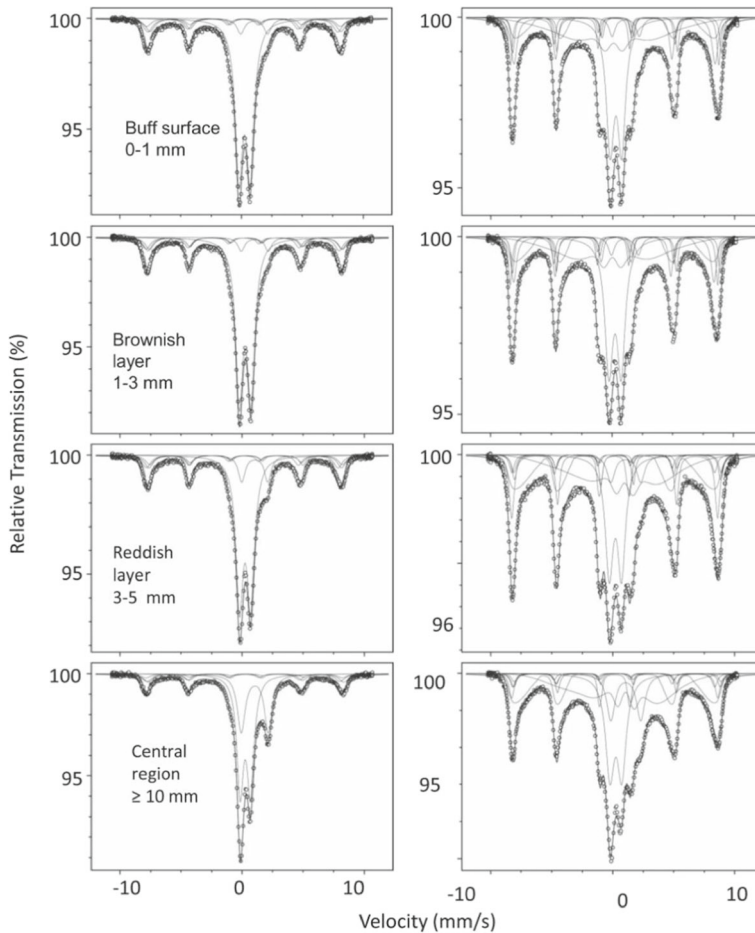


Fig. 3 RT (left side) and 4.2 K (right side) Mössbauer spectra of different layers of the amphora handle CV.A.1425 from Castro do Vieito

relative amounts cannot be determined reliably. A ferrous quadrupole doublet with $QS \approx 2.2$ mm/s and $IS \approx 1.05$ mm/s is present in all spectra. Its area increases from 6 % on the surface to 28 % in the center. This increase can be explained by the model proposed previously [9] assuming that the amphorae were fired under reducing conditions and then re-oxidized at the end of the firing cycle when the kiln atmosphere became oxidizing after the fuel had been consumed. Since the amphorae were already cooling while the oxygen penetrated into the ceramic material, the temperature will have become too low too soon for a complete re-oxidation of the inner parts of the objects.

In the 4.2 K spectra much of the the ferric doublet splits magnetically into two components. One of these is a broad pattern with a mean hyperfine field of about 48 T that can be fitted with an asymmetric Gaussian distribution of hyperfine fields. The second magnetic component can be fitted by an even broader distribution of hyperfine fields around a mean value of only about 25 T. It is unclear whether this component is, indeed, due to a static

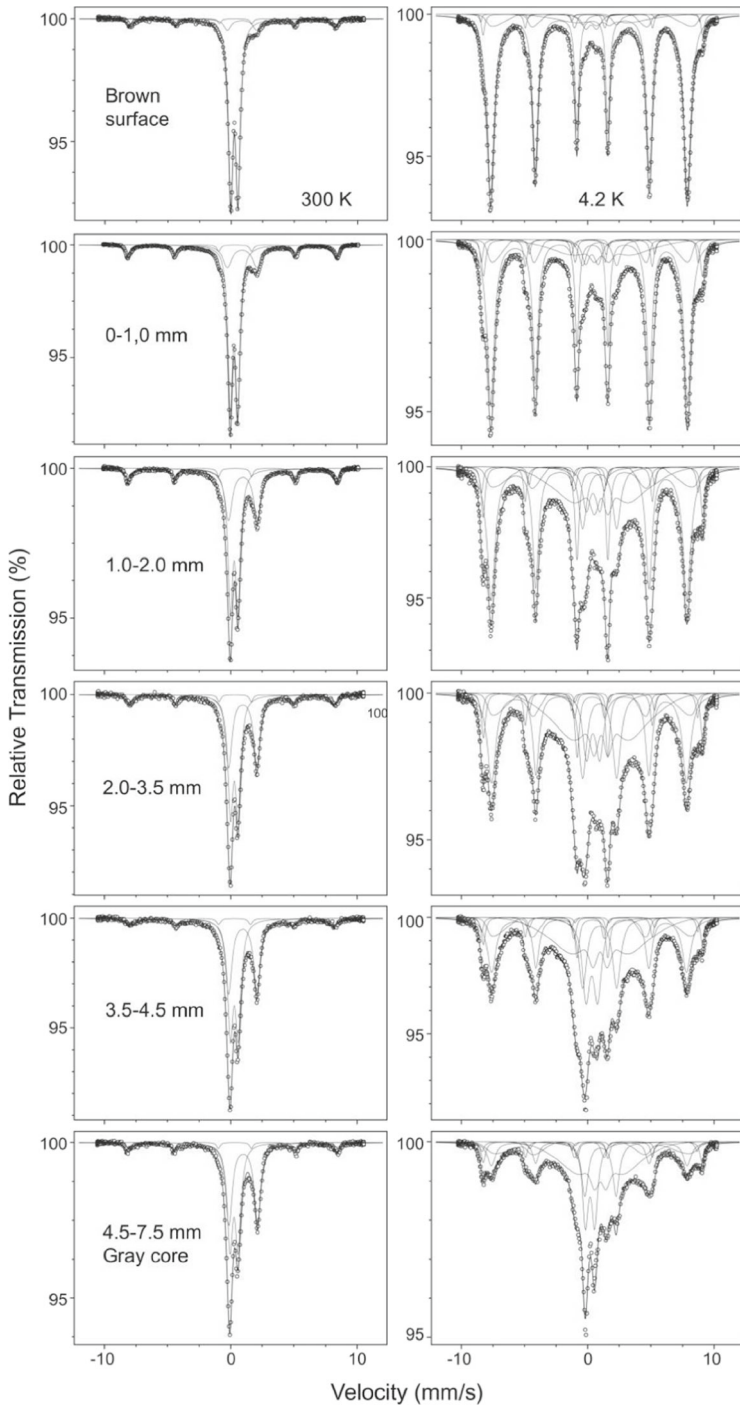


Fig. 4 RT (left side) and 4.2 K (right side) Mössbauer spectra of different layers of the strongly abraded amphora body sherd 3066 from the Cortiçaís shipwreck

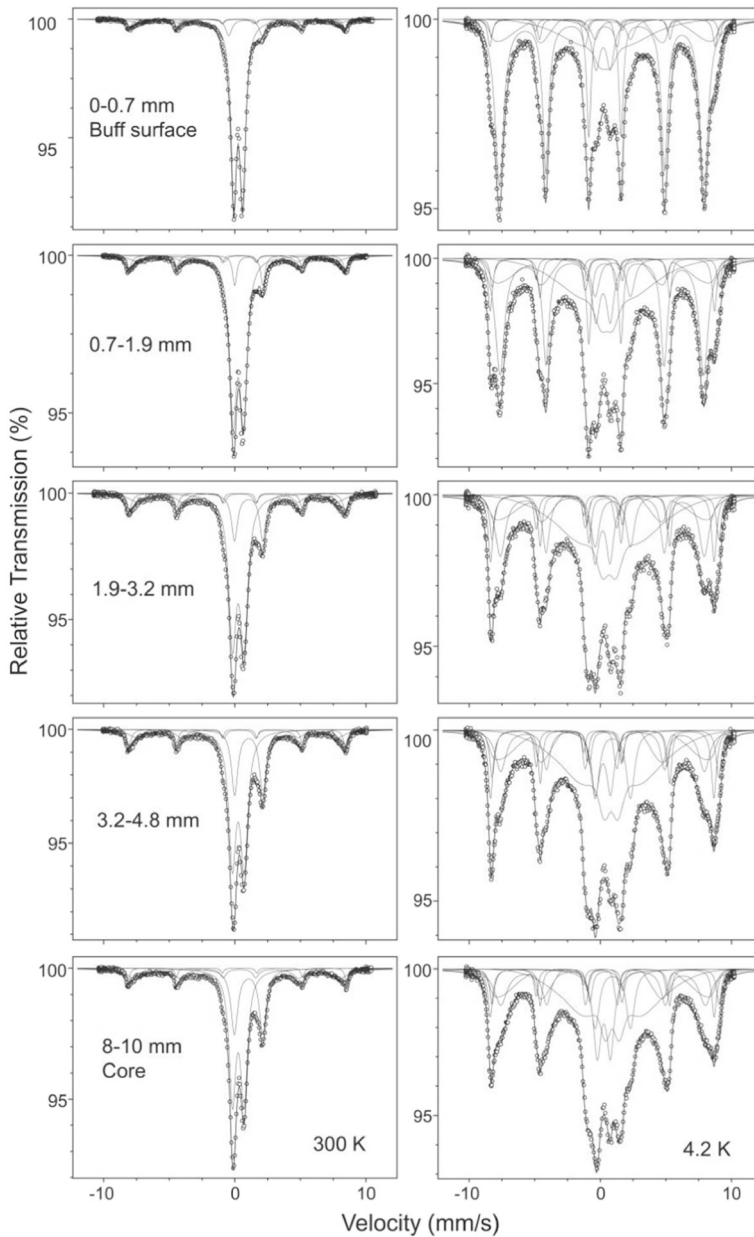


Fig. 5 RT (left side) and 4.2 K (right side) Mössbauer spectra of different layers of the amphora handle 2469 from the Cortiçais shipwreck

distribution of hyperfine fields or to superparamagnetic relaxations with intermediate relaxation times. Both kinds of broad magnetic components have been observed before in the 4.2 K Mössbauer spectra of archaeological ceramics [9, 15, 16]. They have been attributed to small clusters of oxidic iron precipitating in the amorphous silicate matrix that forms

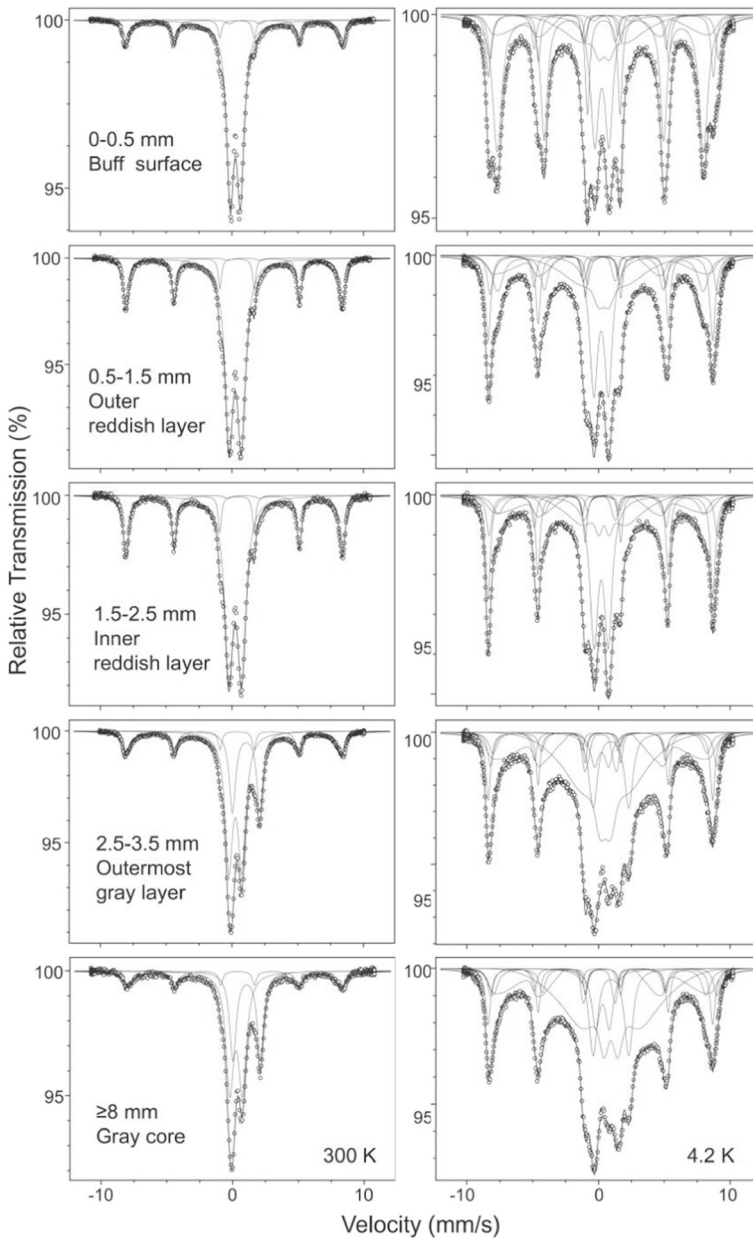


Fig. 6 RT (*left side*) and 4.2 K (*right side*) Mössbauer spectra of different layers of the amphora handle 823 from the Cortiçaís shipwreck

when clay minerals are heated to about 800 °C or higher. The broad magnetic components make up between 40 and 50 % of the spectral area of the 4.2 K spectra. Their magnetic splitting disappears at rather low temperatures. In the case of pottery from the south of Germany it was found to disappear already between 30 and 40 K [16]. Ferric iron that still

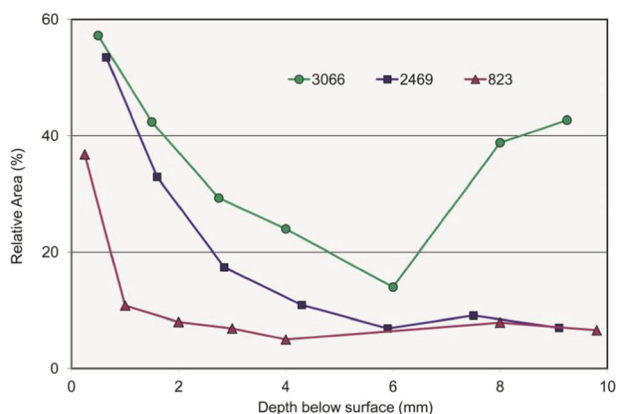


Fig. 7 Dependence of the intensity of the goethite component on the distance from the surface. In the case of sherd 3066 the surface is taken as the surface of the convex side. The surface of the concave side is 10 mm from it at the thickest position, hence the rise of the goethite content for distances above 6 mm from the convex surface

yields a quadrupole doublet at 4.2 K is either so dilute that it does not order magnetically even at 4.2 K, or it forms clusters that are so small that they are still superparamagnetic at that temperature.

The spectra of the different layers of the amphorae from Cortiçais are shown in Figs. 4, 5 and 6. Their spectra are very different from those of the land-buried amphora handle CV.A.1425, particularly those taken at 4.2 K. The RT spectra of the strongly abraded body sherd 3066 (Fig. 4) exhibit mainly a ferric doublet, but with a smaller splitting than for CV.A.1425 ($QS \approx 0.6$ mm/s; $IS \approx 0.26$ mm/s), which changes only slightly with depth and indicates that the ferric component is different from that in CV.A.1425. The ferrous component ($QS \approx 2.25$ mm/s; $IS \approx 1.00$ mm/s) is very similar to that of CV.A.1425. Its intensity increases from 13 % in the buff surface layer to 45 % in the grey core.

The 4.2 K spectra of the outer layers show a dominant component that can be fitted by a Lorentzian sextet with rather broad lines, a hyperfine field near 48 T, a quadrupole shift $eQV_{zz}/2 \approx -0.25$ mm/s and an isomer shift of $IS = 0.26$ mm/s. These are typical parameters for goethite at 4.2 K. When one goes deeper into the sherd, one still finds goethite in the Mössbauer spectra (Fig. 4), but with decreasing intensity. The goethite area of the individual spectra is shown in Fig. 7 as a function of the depth inside the sherd. It decreases from 57 % in the buff surface layer to 14 % in the gray core. When one goes farther away from the outer (convex) surface of the sherd, one comes closer to the inner (concave) surface and the goethite content increases again. The spectra of the gray core are largely like those of the land buried handle CV.A.1425.

The spectra of the brown layer on the surface of 3066 and also of the brown spots on the amphora handles are remarkable since this material contains 3–4 times more iron than the ceramic layers below the surface. The goethite component is particularly strong for the thin brown surface layer of 3066 (Fig. 4, top), where it amounts to 75 % of the spectral area. The brown colour of the surface is thus caused by goethite. The clay matrix components in this spectrum are probably due to ceramic material removed together with the surface material. The goethite in the amphorae does not split magnetically at RT, which shows that it must be present as very small particles.

The spectra of the handles 2469 and 823 (Figs. 5 and 6) are similar to those of the body sherd 3066. The proportion of divalent iron as observed at RT again increases from the surface to the core, whereas that of goethite observed at 4.2 K decreases. The depth dependence of the goethite content (Fig. 7) is, however, different for the two handles. In 2469 goethite is present down to a depth of about 3 mm, but its intensity decreases much more rapidly in 823, where already at a depth of about 1 mm the goethite content has diminished to less than 10 %, where it remains down to the core of the handle. In fact, such low goethite contents may be an artifact of the fitting procedure, which cannot distinguish well between a small amount of goethite and the broad magnetic component of the ceramic material always underlying it.

The spectra of 823 are, however, remarkable for another reason: This handle exhibits a pink rim with a thickness of about 2 mm (Fig. 2) going all around the circumference. The RT Mössbauer spectra (Fig. 6) show that this pink region has a higher proportion of hematite (29 % in the outer and 36 % in the inner half) than either the thin buff surface layer (23 %) or the grayish layer right below the pink one (19 %). This is attributed to the cooling of the amphora in the kiln, not to the influence of the sea burial. Apparently the oxidation front penetrated only to a depth of about 2 mm and with a sharp frontier to the underlying material that was hardly oxidized. Down to the inner pink layer, the Fe^{2+} content is less than 5 %, and immediately below the inner pink layer it jumps to 30 %, where it remains down to the core of the handle.

3.2 Annealing experiments

To further test the nature of the goethite in the amphorae, specimens from the surface layer of 3066 were subjected to annealing in air at temperatures rising in steps of 100 °C from 300 to 800 °C. It turned out that already after annealing at 300 °C the goethite lines in the 4.2 K spectra began to broaden and shift towards higher hyperfine fields, indicating an incipient transition to hematite, which was found to be practically complete on annealing at 600 °C. Remarkably, this hematite was found to be superparamagnetic at RT, but showed the typical magnetic hyperfine splitting at 4.2 K, a feature that is understandable since it was formed from very small goethite particles.

3.3 Temperature dependence

A detailed study of the temperature dependence of the Mössbauer spectra of samples from the surface of 3066 is still ongoing, but first results show that even at 80 K the spectra still exhibit mainly a quadrupole doublet. To obtain the magnetic hyperfine splitting of most of the material, temperatures of about 40 K or lower are needed. This shows that the goethite particles must be very small, probably having sizes below 7 nm.

3.4 X-ray diffraction

X-ray diffraction patterns of the core and the surface material of 3066 are shown in Fig. 8. The diffractograms are nearly identical, except for a slightly higher background in the pattern of the surface layer between 20 and 50 °2 θ . The most interesting feature is that one does not observe the presence of goethite in the diffractogram of the surface layer of 3066. This can be understood if the goethite particles are so small that their diffraction peaks are too broad to be distinguished from the background in the diffraction patterns. Figure 8 illustrates this with a diffraction pattern of a synthetic goethite with a mean particle size between

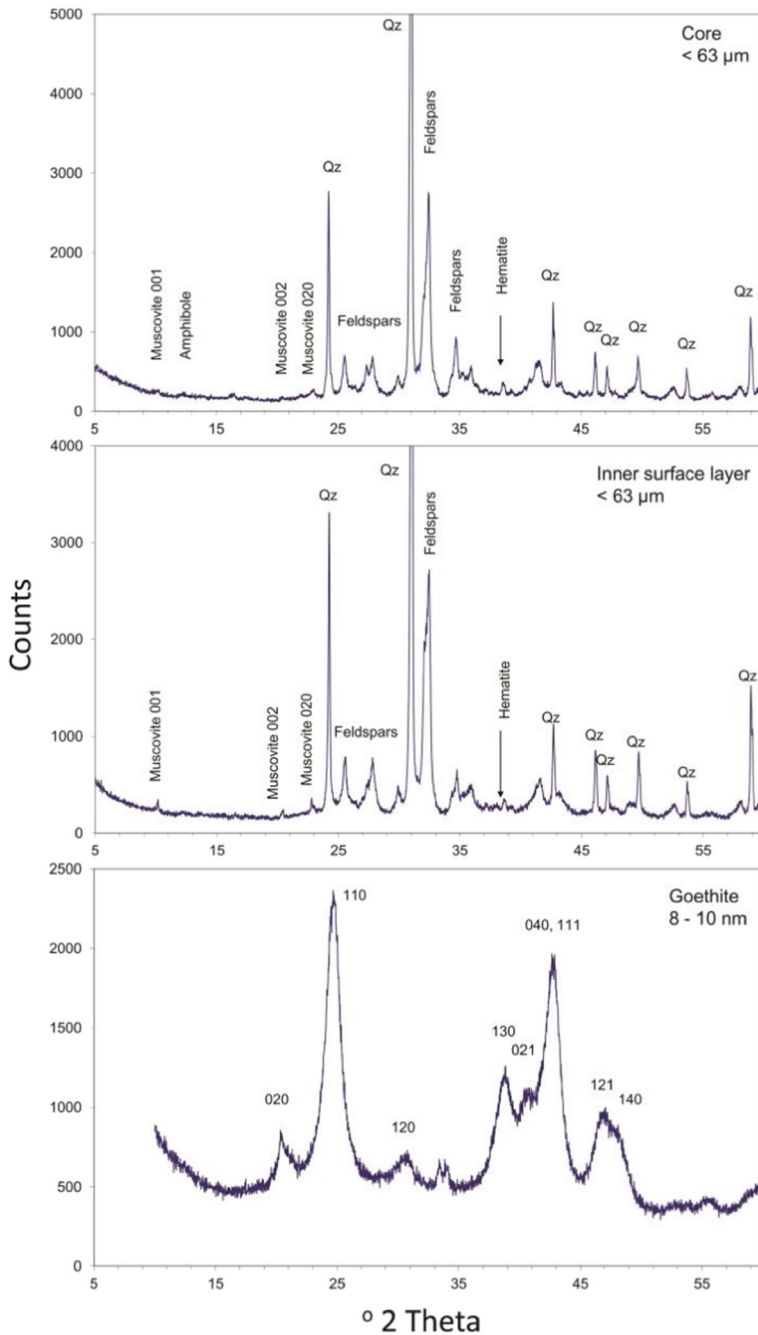


Fig. 8 X-ray diffraction patterns recorded with Co K α radiation for the core and the inner surface layer of the amphora sherd 3066 from Cortiçais. The diffractogram of a synthetic goethite with a particle size of 8–10 nm is shown to demonstrate where the reflections of the goethite in the surface layer of the amphora sherd are to be expected

8 and 10 nm according to from the widths of the diffraction peaks. This goethite shows a complete magnetic hyperfine splitting around 80 K, indicating that the goethite particles in the amphorae are even smaller, since they split completely only below 50 K. Their X-ray diffraction peaks will thus be far too broad to be detected reliably by X-ray diffraction, but they can be detected by Mössbauer spectroscopy.

4 Conclusions

The sea-buried amphora sherds have undergone a conversion of the iron in their amorphous silicate matrix into goethite. Oxidic nanosized iron-rich clusters seem to have converted to similarly small goethite particles with sizes around 7 nm. The conversion to goethite begins at the surface and proceeds into the ceramic material, reaching a depth of several millimeters within two millennia, but apparently also depending of properties of the ceramic paste that still deserve more detailed studies. The strongly abraded body sherd 3066 is remarkable since the converted layer on it is even thicker than that on the handles despite of the abrasion of the material. We presently have no explanation for this.

The brown sometimes spotty surface layer observed on many of the amphora fragments from Cortiçais contains an unusually high proportion of goethite. These goethite rich layers seem to have formed by dissolution of the silicate matrix at the surface of the sherds in the sea water, with the iron remaining on the surface as goethite. We consider this as the more probable explanation compared with the deposition of goethite from iron originally dissolved in the sea water.

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