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Characterization of corrosion products on archaeological iron coins

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Abstract

Purpose – The purpose of this paper is to investigate the microstructures, slag inclusions, morphology and composition of ancient Chinese iron coins exhumed from Emei Mountain and Baoji after 818-966 years of being imbedded underground.

Design/methodology/approach – Metallography, scanning electron microscopy, energy dispersive spectrometry, X-ray photoelectron spectrometry and Fourier transform infrared spectroscopy were employed.

Findings – The results showed that archaeological coins exhibited characteristics of a typical hypoeutectic white cast iron, with slag inclusions of FeS strips and phosphate. Porous or hexagonal platey corrosion products were discovered on the archaeological iron coin, which were mainly identified as Fe_2O_3 , FeOOH, $Fe_x(OH)_{1-x}CO_3$ and $Fe_x(OH)_{1-x}SO_4$. The possible corrosion mechanisms for the iron coins were discussed based on the corrosion products.

Originality/value – This study revealed the characterization of corrosion products on archaeological iron coins and may provide guidance for the preservation of archaeological iron.

Keywords Archaeology, Iron, Rust, Corrosion products, China

Paper type Research paper

1. Introduction

The Song Dynasty (from 960AD to 1279AD) was the longest dynasty in Chinese history when iron coin was used as common currency. A great number of Song iron coins were exhumed recently. In 1988, about 20,000 kg of Song iron coins were excavated from Baoji, a city in the Northwest China. About 14 years later, 16,320 kg iron coins were recovered from Emei Mountain and Mianyang city. All of the hoarded coins were seriously corroded. They were partly mineralized and difficult to separate from the adhering soil, due to the attack of the soil environment. Most of the iron artifacts in China were made of cast iron, which could be classified into grey cast iron, mottled cast iron, white cast iron and decarbonized cast iron (Wang *et al.*, 2007a, b).

The corrosion of iron artifacts involved several factors such as their chemical composition, the metallographic structure, slag inclusions, as well as design configuration. However, the environment also has important effects on the preservation of

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Anti-Corrosion Methods and Materials

ironware. So far, little work has been done on the evolution of corrosion products on ancient iron coins, and in China the preservation of large numbers of ancient coins was imperative.

In this paper, metallographic techniques, scanning electron microscopy (SEM), energy dispersive spectrometry (EDS), X-ray photoelectron spectrometry (XPS) and Fourier transform infrared spectroscopy (FTIR) were used to analyze the morphology and composition of the corrosion products. The work aimed to clarify the corrosion mechanism of archaeological iron coins and provide guidance for coin preservation.

2. Methodology

2.1 Samples

The samples of Chinese ancient iron coins are listed in Table I.

2.2 Observation of metallographic structure and corrosion morphology

Cross-sections of samples E1 and B1 were polished with emery paper up to 1,000 grade followed by etching in nitric-alcohol solution for 10 s. The morphology was observed using a Quanta-600F SEM manufactured by America FEI.

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Table I Details of Chinese ancient iron	coins
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Sample number	Excavation year	Site	Production year	Characteristics	Fracture face
E1, E2	2002	Emei Mountain	About 1174-1189AD	Rusted; adhering together; characters on individual coin can be identified; metal core remained	White
B1	1988	Baoji	About 1041-1125AD	Seriously corroded, adhering together, unidentifiable characters, and metal core remained	White

2.3 Identification of corrosion products

Slag inclusions were analyzed using EDS manufactured by Link (Oxford Instrument Analytical Limited).

XPS was performed using a MCROLAB MKIIX (VG, England) employing a hemispherical analyzer to evaluate the composition of the corrosion products. The analyzer was operated in the constant analyzer energy mode at pass energy of 50 eV. The XPS spectra were recorded with Al Ka radiation (300 W, 15 keV) of an Al/Mg twin anode. Argon ion beam etching was performed at 3 keV incident energy for 5 min. The angle between the ion gun and sample surface was 45°. In the analyzer chamber, the base pressure was better than 0.5×10^{-6} Pa.

FTIR spectroscopic measurements were conducted using a spectrometer (FTIR, JASCO 470). The corrosion products and KBr were mixed with a sample/KBr weight ratio of 1/100 and were compressed to pellets. The region of test spectrum was $400-4,000 \text{ cm}^{-1}$.

3. Results

3.1 Microstructure of ancient iron coin

The microstructures of ancient iron coin samples E1 and B1 are shown in Figure 1. They both comprised austenite, cementite and ledeburite. The grey part with white spots is ledeburite, and the white part is the corroded austenite, branches of which are clearly observed in the image E1. The dark grey area that surrounds the austenite grains is cementite. The iron coins exhibit characteristics of typical hypoeutectic white cast iron. During the casting process of the iron coins, the cooling step is rapid due to small thickness, so the carbon in the iron does not have sufficient time to form graphite, but changes instead into Fe_3C . This is the reason that iron coins have a white cast iron structure. In addition, white cast iron is the hardest and the most wearable one in the cast irons, which reduces the effects of abrasion when the coins are in circulation.

3.2 Slag inclusions

There are various kinds of metallurgy defects, such as pores or slag inclusions in the matrix of iron artifacts because of the Volume 58 · Number 1 · 2011 · 39-45

Figure 1 Microstructures of ancient coin samples E1 and B1



ancient foundry technique. The typical morphology of slag inclusions in iron coins E1 and B1 is presented in Figure 2. The EDS results showed that the white strips in the cavity on the surface of iron coin E1 consisted of FeS, and the black agglomerant in B1 consists of phosphate.

The dissolution products of sulfide slag inclusions like S^{2-} and HS^{-} are considered to be activators of the local corrosion attack due to their high moisture adsorption and activation behavior towards iron. They are capable of reducing the amount of Fe²⁺ in the solution, and lowering open circuit potential. Clearly, S^{2-} and HS^{-} will first accelerate corrosion around the slag inclusions. Because the phase boundary energy between the sulfide slag and the iron matrix is higher than other places, the location around sulfide slag inclusions is prone to corrosion (Chen *et al.*, 2000).

It is well recognized that phosphor is able to improve the anti-corrosion performance of iron and steels. Naito Hirondts has suggested that elements such as P, W and Mo in steels will form XO_4^{-} (where X refers to P, W, Mo), which combines with metal ions and deposits in the region nearby anode, thus preventing the anodic reactions. Balasubramaniam (2000) investigated the corrosion resistance of an ancient Indian object – the Delhi iron pillar, which is approximately 1,600 years old. The Indian research showed that the minor element P had an important effect on the corrosion resistance of the pillar to the atmosphere. Initially, the corrosion rate of iron is high due to the presence of slag particles. This results in enhancement of surface P content. In the presence of P, the

Figure 2 Slag inclusions in iron coin E1 and B1



formation of a protective amorphous compact layer of δ -FeOOH next to the metal surface is catalyzed and this confers the initial corrosion resistance. In addition, the formation of iron hydrogen phosphate hydrate, or the crystalline phosphate, lowered the rate of corrosion (Ramesh and Balasubramaniam, 1998; Balasubramaniam, 2000).

3.3 Observations relating to corrosion morphology

Figure 3 shows an SEM image of iron coins E1 and B1. The section of E1 is composed of an iron matrix, product layer (PL) PL1 and PL2. It can be observed that PL1 comprises corrosion products and a few of SiO_2 grains from soil, and the composition is shown in Table II.

Neff *et al.* (2005) and Chitty *et al.* (2005) studied 40 iron archaeological artifacts that had been buried in soil for several centuries. Samples taken with the adhering soil and crosssections of the ferrous archaeological artifacts were analyzed with the description as follows from the metal to the soil:

- The metallic substrate (M) contains various amounts of minor elements and also slag inclusions.
- Dense product layer (DPL), a layer of corrosion products made of iron oxides, oxy-hydroxides, chlorides and/or carbonates. These phases are generally well crystallized and more compact, compared to the external zones of the corrosion system. The DPL may contain slag inclusions coming from the metallic substrate.
- Transformed medium (TM), a transition zone between the DPL and the soil, containing both corrosion products and quartz grain from the soil. The surface between the DPL and the TM corresponds to the ancient original



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surface of the object whose position probably has moved during the corrosion processes.

The soil (S), where no corrosion products can be found.

The above description presents the characteristics of the cross section of artifacts from metal to the soil in most cases. However, the cross-section of archaeological iron coins is different from these common characteristics. It seems that PL1 in Figure 3 E1 is similar to the TM in Neff's classification, but the outer layer PL2 is not soil but FeOOH, which is confirmed by the EDS analysis on area A (Table II). The characteristics of iron coin E1 were attributed to its heap-stored buried state in the soil. They probably were bonded together either by corrosion product, or by soil. There was insufficient space between the coins to allow stack storage, and thus there would be sections where the corrosion product and soil were interspersed between adjacent iron coins.

The cross-section of iron coin B1 was made up of an iron matrix, a transformed layer (TL), and a corrosion PL. The TL is a transition zone between the iron matrix and the corrosion product, and it had the same structure as the iron matrix. Nearby, at the interface between the TL and the iron, there are crevices along the interface with some perpendicular cracks in the TL zone. Along the interface, a little corrosion product was distributed as shown in Figure 3. It can be observed that the corrosion PL was loose and porous and was similar to the skeleton of sponge. Therefore, water or aggressive ions in the electrolyte, such as Cl^- and SO_4^{2-} from soil, could be absorbed and may accumulate and could attack the adjacent iron core. Formation of corrosion products is associated with a volume expansion, which causes the cracks and exfoliation of the corrosion layers or even whole coins.

Figure 3 Cross-section on the iron coins E1 and B1





Notes: PL: product layer; TL: transformed layer

Table II EDS analysis on iron coin E1

Element (atomic %)	А	В	С
с	5.96	_	8.74
0	68.91	68.70	63.85
Si	1.25	26.66	5.94
Fe	23.88	4.63	21.47

3.4 Morphology and composition of corrosion product An important characteristic of the ancient storage was that a large mound of iron coins was heaped together. Therefore, the gaps between piled coins varied in shape and volume, and accordingly the contents of air and ground water varied in the gaps, leading to a complicated morphology and composition of corrosion products intermingled with the iron coins. After separating the coins mechanically, it was found that there were several corrosion products with different colors at different regions. Some was an orange powder; some was brown or dark green crystalloid to the naked eye. 3.4.1 Morphology and composition of corrosion product on iron coin E2

The morphology of the orange powder on iron coin E2 is shown in Figure 4. It grew in plate shape and showed a uniform honeycomb structure. The FTIR in Figure 5 shows that the orange rust was composed of mainly γ -FeOOH and a little amorphous layer of δ -FeOOH. The brown rust on E2

Figure 4 Morphology of the corrosion products on iron coin E2





Figure 5 FTIR analyses on the corrosion product on an iron coin



exhibited hexagonal plates that were stacked together. The layers were very compact and they exhibited plate-shaped particles with a very large average thickness of about 10 μ m, as is shown in Figure 4 (E2-b).

The major elements in the corrosion product of E2-b (i.e. Fe, O, C and S) were analyzed by XPS. Figure 6 shows the fits of the Fe 2p, O1s, C1s and S2p responses; the calculated peaks of the signals of the different components were corrected. Binding energies of the compounds from published literature were used for calibration wherever possible. The standard binding energies are listed in Table III.

An examination of the Fe1p peaks revealed a $Fe2p_{3/2}$ peak at a binding energy of 711.24 eV, corresponding to Fe^{3+} . binding energies of O1s can be separated into 529.63, 531.18

Figure 6 Fe 2p, O1s, C1s and S2p signals on rust E1-b

and 532.96 eV, associated with the iron oxide, hydroxide, carbonate or sulfate, respectively. The scan of the C1s binding energies revealed three peaks: the one at 284.80 eV corresponded to the carbon adsorbed on all the specimens, and the other two at 286.30 and 288.53 eV both corresponded to iron carbonate. The S1s peak is located at 168.57 eV, taken as the standard binding energy for iron sulfate. In summary, it was confirmed the corrosion products of E2-b consisted of Fe₂O₃, FeOOH, Fe_x(OH)_{1-x}CO₃ and Fe_x(OH)_{1-x}SO₄ regardless of the adsorbed carbon on the specimen induced by contamination. The Cl2p binding energy is located at 199.00 eV, which means that possibly

there were some chlorides in the corrosion products.



Tabl	e	III	Binding	energies	for	all	standard	signals	s from	references	
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	Fe 2p _{3/2}	01s	C1s	S2p	Cl2p
Fe(II)	707.15-711.00				
Fe(III)	710.20-711.05				
С			284.60		
CO ₃ ²⁻		532.2-532.8	288.0-290.3		
SO ₄ ²⁻		532.1		168.70-169.00	
0 ²⁻		529.2-532.2			
OH^-		531.2			
H ₂ O		533.1			
cl [−]					199.00

Sources: Descostes et al. (2000), Keller and Strehblow (2004), Wang et al. (1998), Lin et al. (2005), and Fuente et al. (2006)

3.4.2 Morphology and composition of corrosion product on iron coin B1

The morphology of the brown rust on iron coin B1 is shown in Figure 7. The SEM image revealed a lot of broken hexagonal platelets covered with many particles. According to the analysis shown in Figure 8, the brown rust was α -FeOOH, which is supported by the earlier studies by Wang (2007a, b): the grain-shaped corrosion product on archaeological iron was identified as α -FeOOH.

4. Discussion

According to the investigation above, it was concluded that the corrosion products on ancient iron coins of Emei Mountain and Baoji were made up of Fe₂O₃, α -FeOOH, γ -FeOOH, Fe_x(OH)_{1-x}CO₃ and Fe_x(OH)_{1-x}SO₄. The formation of these various kinds of corrosion products resulted from attack on the iron coins in the deeply buried soil over several centuries. In the beginning, the iron coins dissolved by reaction with the underground soil water:

Figure 7 Morphology of the corrosion products on iron coin B1



Figure 8 FTIR analysis on the corrosion product on iron coin



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$$Fe \rightarrow Fe^{2+} + 2e$$
 (1)

then Fe^{2+} hydrolyzed to ferrous hydroxide $\text{Fe}(\text{OH})_2$ on the surface of iron coins:

$$Fe^{2+} + 2H_2O \rightarrow Fe(OH)_2 + 2H^+$$
⁽²⁾

in presence of various anions such as Cl^- , SO_4^{2-} or $HCO_3^$ which are the main corrosive sources to metal in the soil. The resulting ferrous hydroxide $Fe(OH)_2$ usually oxidizes into ferric oxyhydroxides via an intermediate Fe^{II} - Fe^{III} hydroxylsalt designated as green rust (GR), e.g. GR(Cl -) (Refait, 1998), $GR(SO_4^{2-})$ or $GR(CO_3^2 -)$ (Abdelmoula, 1996). The $GR(Cl^-)$ has honeycomb structure, while $GR(SO_4^{2-})$ is piled hexagonal platelets, and $GR(CO_3^{2-})$ are compact layers with platy particles.

The reactions (Refait, 1994) are as follows:

$$GR(Cl^{-}): 4Fe(OH)_{2} + Cl^{-} + 2H_{2}O$$
$$\rightarrow Fe_{3}^{II}Fe^{III}(OH)_{8}Cl \cdot 2H_{2}O + e \qquad (3)$$

$$GR(SO_4^{2-}): 6Fe(OH)^2 + SO_4^{2-} + 2H_2O$$

$$\rightarrow Fe_4^{II}Fe_2^{III}(OH)_{12}SO_4 \cdot 2H_2O + 2e \qquad (4)$$

$$\begin{aligned} GR(CO_3^{2-}) &: 6Fe(OH)_2 + HCO_3^- + 2H_2O \\ &\to Fe_4^{II}Fe_2^{III}(OH)_{12}CO_3 \cdot 2H_2O + H^+ + 2e \end{aligned} \tag{5}$$

when SO_4^{2-} or CO_3^{2-} are present, $GR(Cl^-)$, owing to its moderate oxidation number of Fe (+2.25), can be oxidized into $GR(SO_4^{2-})$ or $GR(CO_3^{2-})$ compounds, which are more stable than is $GR(Cl^-)$ due to the higher oxidation number of Fe (+2.33) (Refait, 1997).

As an intermediate transition compound, the GR gradually became darker, yellow or brown, due to the state of Fe in the rust changing from Fe^{II} to Fe^{III} over 900 years while buried deeply underground. Consequently, $Fe_x(OH)_{1-x}CO_3$ and/or $Fe_x(OH)_{1-x}SO_4$ containing Fe^{III} determined as the hexagonal platelets by XPS, is the correct result from the transformation of $GR(CO_3^{2-})$ and/or $GR(SO_4^{2-})$.

Usually, GR may transform into an orange rust y-FeOOH after contact with the open atmosphere. The γ -FeOOH will further oxidize to brown α -FeOOH. Relatively, the latter should always be a long-term process at room temperature (Huang et al., 1996). Because of insufficient oxygen supply to deeply buried iron coins, the GRs have sustained very slow oxidization during more than 900 years. Although the slow effect increasingly changed the composition of GR including $GR(Cl^{-})$, $GR(SO_4^{2-})$ and $GR(CO_3^{2-})$ into the new species like γ -FeOOH and/or α -FeOOH, the particular structure of hexagonal platelets for GR itself still remained. This is the probable reason for the morphology and composition of corrosion products on the iron coin B1. Furthermore, XPS disclosed that there was Fe₂O₃ in the rust on iron coins such as E2, which possibly resulted from the decomposition of oxyhydroxides when the coins were heated to dry them after being excavated. According to previous investigations, the phase transformation of corrosion products on archaeological iron coin can be summarized by the sequence shown in Figure 9.

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Figure 9 Phase evolution of corrosion products on archaeological iron coin



5. Conclusions

Ancient Chinese iron coins exhibit characteristics of typical hypoeutectic white cast iron. Slag inclusions such as sulfide and phosphide are found entrapped in the iron coin matrix. The location around sulfide slag inclusions is easily lost, while phosphide is able to improve the corrosion resistance of the iron coins. The cross-section of these specific archaeological iron coins was different to common characteristics mentioned by previous investigators because of the storage method of these coins.

Corrosion products on the surface of the iron coins presented complex morphologies. They exhibited a honeycomb structure and hexagonal platelets with the composition Fe_2O_3 , FeOOH, $Fe_x(OH)_{1-x}CO_3$ or $Fe_x(OH)_{1-x}SO_4$. These rusts have evolved over several centuries as a result of attack by the exposure environment.

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