ANALYSIS AND CONSERVATION OF AN IRON AGE DAGGER FROM TALL ABU AL-KHARAZ, JORDAN VALLEY: A CASE STUDY

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ABSTRACT

The aim of this study is to report from the analyses of a corroded iron dagger from the Iron Age city at Tall Abu al-Kharaz, Jordan Valley, and to present the conservation procedures. Preliminary condition assessment was carried out by visual examination and stereomicroscope. X-ray radiography was used to reveal surface details under the external deposits and corrosion layers. X-ray diffraction (XRD) analysis was used to identify the mineralogical composition of the corrosion products. Polarized light microscopy (PLM) was used to examine a cross-section taken from the dagger to determine its corrosion profile and internal metallographic microstructure. The dagger's radiograph showed that the edges and tip of the dagger were its weakest and most corroded parts. XRD results showed that goethite (α-FeOOH), lepidocrocite (γ-FeOOH) and magnetite (Fe_3O_4) were the main corrosion products. PLM examination showed that the dagger had greatly converted into magnetite. It also showed signs of hammering, carburization and heat treatment during the manufacturing process of the artifact.

The dagger was treated by immersion in an alkaline solution to extract any chloride ions present in the artifact. It was then treated with tannic acid to produce a coherent film of black ferric tannate. Finally, it was coated with Paraloid B72 to consolidate it and protect it from atmospheric humidity and corrosive ions in the environmentally uncontrolled storage area.

1. INTRODUCTION

The analytical investigation of archaeological metal artifacts is essential to identify their corrosion products and conservation needs. It can also provide information about their archaeological history. Archaeological iron artifacts are usually found in an advanced state of corrosion due the reactivity of the metal in various environments. Iron corrodes when exposed to oxygen and humidity, and the corrosion process is accelerated by the presence of dissolved anions, particularly chloride ions. The external corrosion layer usually contains iron oxide hydroxides (goethite α-FeOOH and lepidocrocite γ-FeOOH) mixed with extraneous accretions and soil deposits. The internal corrosion layer is usually composed of iron corrosion products of a lower oxidation state, particularly magnetite (Fe_3O_4) (Selwyn et al., 1999; Selwyn, 2004; Neff et al., 2005; Tamura, 2008; Reguer et al., 2009). Most of the chloride ions are not combined in a stable solid corrosion product. They take part in the metal corrosion cycle, and once the artifact becomes completely mineralized, they diffuse into the burial soil (Turgoose, 1982; Watkinson, 1983). In other words, chloride containing akaganeite (β-FeO$_{0.833}$(OH)$_{1.167}$Cl$_{0.167}$) is a metastable corrosion product, its long term hydrolysis will release chlorides trapped in its tunnel structure and act as an electrolyte for the corrosion of iron. Upon the complete mineralization, the chlorides diffuse to the burial soil and the final corrosion product will be the α-FeOOH goethite (Gilberg and Seeley, 1981; Watkinson and Al-Zahrani, 2008).

Corroded archaeological iron artifacts need to be treated to remove the corrosive agents and increase their stability in the exhibit or storage area. Aqueous alkaline solutions have been used to extract chloride ions from corroded archaeological iron artifacts (North and Pearson, 1978; Gilberg and Seeley, 1982; Turgoose, 1985; Watkinson, 1996; Selwyn and Argyropoulos, 2005; Watkinson and Al-Zahrani, 2008; Wang et al., 2008; and Rimmer et al., 2012). Alkaline sulfite (a mixture of sodium hydroxide and sodium sulfite) and sodium hydroxide are the most common solutions used to extract chloride ions from corroded iron. Both solutions showed little differences in the chloride extraction rate and residual chloride in the artifact, even a solution of low concentration was found to be efficient and did not need frequent changes (Costain, 2000). In fact, the hydroxide ions (OH⁻) from sodium hydroxide are extractors of chloride ion (Cl⁻), they supplant the chloride ions (Cl⁻) as counter ions and allow them to diffuse away from the artifact.

For an artifact with a remaining metal core, it is preferred to use de-oxygenated alkaline solution, which improves the extraction of chloride ions and protect the artifact from corrosion during the treatment (Gilberg and Seeley, 1982; Turgoose, 1985; Watkinson, 1996; Selwyn and Argyropoulos, 2005). De-oxygenation is achieved by either sodium sulfite (Na_2SO_3), nitrogen gas (N_2) or vacuum (Schmutzler and Eggert, 2010). The sulfite ions (SO_3^{2-}) scavenge oxygen by reacting with it and converting to sulfate ions (SO_4^{2-}), while nitrogen gas physically displaces the oxygen (Turgoose, 1985). However, for highly mineralized artifacts, the use of strong reducing alkaline sulfite solution is not recommended. The artifact may become softer, more fragile, and eventually disintegrate. The treatment may also leave sulfate residues in the artifact, and have a negative effect on the mineralized organic matter preserved in the corrosion layer (Keene, 1994). Therefore, lower cost, less environmental damage and easier monitoring of chloride ions suggest using sodium hydroxide alone in the desalination solution (Wang et al., 2008).

This case study aimed to investigate the corrosion and conserve an iron dagger excavated from Tall Abu al-Kharaz in Jordan. Preliminary condition assessment was conducted by visual examination and stereomicroscope. X-ray radiography was used to reveal details and compositional differences under the external surface lay-

ers. X-ray diffraction (XRD) analysis was used to identify the mineralogical composition of the corrosion products, therefore explain the state of preservation of the artifact. Polarized light microscopy (PLM) was used to examine a cross-section taken from the dagger to determine its corrosion profile and internal metallographic microstructure. The artifact was treated by immersion in alkaline solution, followed by tannic acid treatment and finally coating to protect the artifact from atmospheric humidity and corrosive ions in the environmentally uncontrolled storage area.

2. THE CONTEXT

The Swedish excavations at Tall Abu al-Kharaz, a twelve-hectare tell in the central Jordan Valley (Fig.1), continued in 2013 in order to shed further light on the Iron Age occupation of this city, first settled around 3200 BC, which corresponds to the conventional Early Bronze Age IB (Fischer, 2008). The city was also occupied during the Middle and Late Bronze Ages (Fischer 2006). Intensive Iron Age occupation lasted from the 12th century BCE until 732 BCE (local Phases IX-XIV) when the city was conquered by the Neo-Assyrians (Fischer, 2013). At the end of the Iron Age only squatters occupied the city (Phase XV).

The excavation in 2013 concentrated on the area north and east of the well-preserved, 46 m long, two-storey compound in Area 9 which dates from Iron Age I (local Phase IX), that is around 1100 BCE (Fischer and Bürge, 2013). Test trenches to the north of the compound, in Area 11, revealed remains from the Islamic/Abbasid, Late Roman, Iron Age, Late and Middle Bronze Age periods (Fischer and Bürge, in press). Finds from domestic Iron Age IIB contexts, viz. the local Phase XIV (770-732 BCE), include several complete ceramic vessels, an excellently decorated cosmetic palette of limestone and a number of tools and weapons. Amongst the latter is a dagger of iron which preliminarily is ascribed to Phase XIV (770-732 BC).
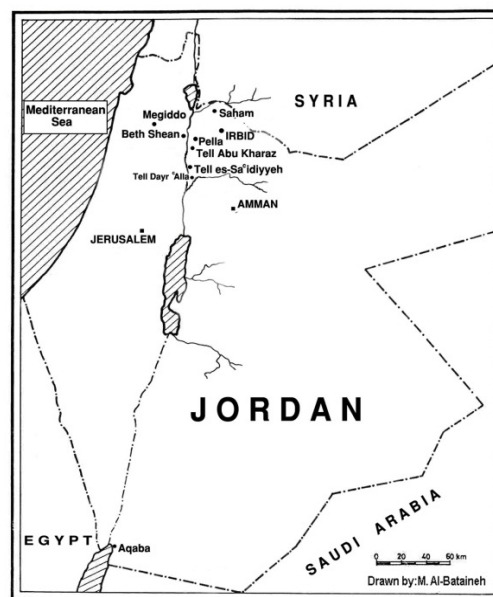

Figure 1 Location map of Tall Abu al-Kharaz

3. MATERIALS AND METHODS

Preliminary condition assessment was carried out by visual examination aided with stereomicroscopy.

X-ray radiography was used as a preliminary non-destructive imaging technique for the aim of inspecting the condition, degree of corrosion and any internal cracks hidden underneath the external deposits and corrosion products of the dagger. Shimadzu MU125M Portable X-Ray instrument was used to take the X-radiograph. The exposure was 80 kv, 3 mA at 100 cm from the source for 10 seconds.

The mineralogical composition of the corrosion products was determined using a Shimadzu LabX, XRD-6000 X-ray diffractometer. A small piece was taken from the broken base of the dagger, ground into powder then analyzed according to the following operating conditions: voltage U= 40 kV, current I= 30 mA, X-ray from anticathode copper (Cu), wavelength CuKα= 1.54178 Å, graphite monochromator, test range 10-95° 2θ, step size of 0.02 in 2θ, and 0.6 s time constant (per step). The assignment of the minerals was based on the database of the Joint Committee Powder Diffraction Standards-International Centre for Diffraction Data (JCPDS-ICDD).

A cross-section was taken from the broken base of the dagger to examine its metallographic microstructure and mode of corrosion using polarized light microscopy (PLM). The dagger was photographically documented before taking the XRD and cross-section samples and after the end of the examination and treatment. The cross-section was embedded in an Araldite 2020 epoxy resin. Following curing for 24 hours, the sample was ground on a series of silicon carbide papers from 150 to 2000 grit, then polished on diamond laps beginning at 9–3 μm and reducing down to a highly polished surface using 1–0.25 μm diamond pastes. The cross-section was then decontaminated in ethanol in an ultrasonic bath and finally dried. The embedded cross-section was etched with 2% nital (2% nitric acid in ethanol). A Zeiss stereomicroscope was used for the preliminary general examination of the dagger and its cross-section. An Askania-Labor polarizing microscope was used for the metallographic examination. Photomicrographs were taken using an AxioCam ERc 5s camera attachment.

An alkaline solution of 0.5M sodium hydroxide (NaOH) was used to extract any chloride ions present in the dagger, therefore increase its stability. The concentration of diffused chloride ions into the alkaline solution was measured using a pHoenix chloride selective electrode connected to a CyberScan pH2500 ion concentration meter. The dagger was immersed in a liter of the 0.5M NaOH solution in a plastic box with an airtight lid, and the chloride ion concentration was measured after one week. The process was repeated for another week with a fresh solution of 0.5M NaOH to insure maximum removal of chloride ions.

After the alkaline treatment, the artifact was washed in several baths of de-ionized water until the pH of the water stopped to rise. It was then dehydrated by immersion in ethanol followed by heating in the oven at 50 ºC for 1 hour. The artifact was then brushed with several coats of a 5% solution of the corrosion inhibitor tannic acid. After the development and drying of ferric tannate layer, the artifact was coated with a 5% solution of Paraloid B72 that was applied by brushing. Finally, a layer of microcrystalline wax applied to decrease the glossiness of the Paraloid B72 coating.

4. RESULTS AND DISCUSSION

4.1 Condition Assessment

Preliminary visual examination of the dagger show that the surface is completely mineralized (Figs. 2 and 3). The base layer is composed of substantial dark brown / black corrosion products. Over this layer are batches of reddish brown corrosion products, fossilized fragments of wood, and soil deposits. These materials are hiding the original surface of the dagger. Some parts of the outer surface have small cracks. The handle of the dagger is missing; the tang had broken from the base of the blade and was not found in the excavation. There are traces of powdery green corrosion at the edge in the middle of the broken base. This type of corrosion is related to copper and indicates that the dagger would have had copper rivets. It would have also had surface decorations; this is indicated by the presence of two main nodules in the central area of the blade, these nodules have missing parts that separated as a result of the corrosion process. In general, the artifact is in a substantially stable corroded state; although it has missing parts and fragile surface; its overall shape is maintained by the corrosion products and surface incrustations.

Figure 2: The dagger's obverse

Figure 3: The dagger's reverse

4.2. X-ray Radiography

X-ray radiography is used to reveal surface details under the external corrosion layers and to indicate the manufacturing techniques and compositional differences which are the result of corrosion products of more than one material in the artifact (Lang, 2005). The X-ray radiography image of the dagger shows that the it has two different zones (Fig. 4); the lighter and cracked zone concentrates at the edges of the dagger and its triangular tip, while the denser zone (brighter in the radiograph) concentrates at the central area of the dagger. It can be deduced from the density variation appearing in the dagger's radiograph that the extensive corrosion concentrates at the edges and tip of the dagger because these areas are thinner and weaker as a result of hammering during the manufacturing process.
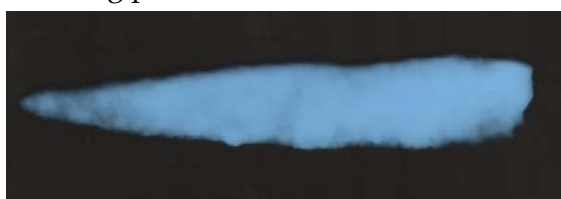

Figure 4: X-ray radiography image of the dagger

The interpretation of X-ray radiographs is limited, especially with the lack of substantial uncorroded metal in the artifact (cf. Watkinson, 1983). Therefore, X-ray radiography was succeeded by chemical and metallographic analyses to examine the identity of the artifact and confirm the composition of different phases.

4.3. XRD Analysis

The XRD characterization of the corrosion products shows the presence of three corrosion products: goethite (α-FeOOH), lepidocrocite (γ-FeOOH) and magnetite (Fe_3O_4) (Fig. 5). No chloride containing product (i.e. akaganeite β-$FeO_{0.833}(OH)_{1.167}Cl_{0.167}$) was detected, which indicates that either the surrounding soil did not contain a significant amount of corrosive chloride ions, or the artifact has completely corroded and the chloride ions have diffused from the artifact to the surrounding soil (Turgoose, 1982; Watkinson, 1983). It seems that the formation of the compact magnetite had a protective effect on the artifact and maintained the overall shape of it (Jegdić et al., 2012). The molar volume of magnetite is 14.9 cm^3, while it is 20.9 cm^3, 26.7 cm^3 and 21.7 cm^3 for α-, β- and γ- FeOOH respectively (Selwyn, 2004). Therefore, the dagger was found in a fairly stable condition due to the stable corrosion products.
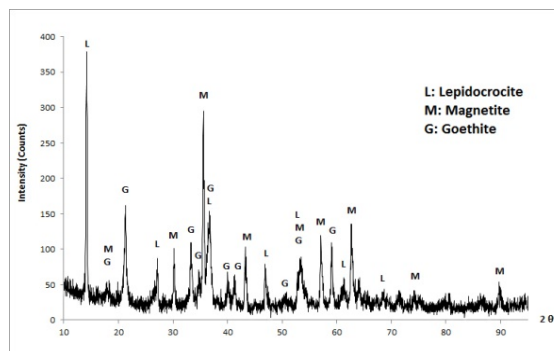

Figure 5: XRD spectrum of the dagger's corrosion products

4.4. Metallographic Examination

A preliminary stereomicroscope examination of the dagger's cross-section shows an external reddish brown corrosion layer (i.e. goethite and lepidocrocite in the XRD analysis), and internal black mass (i.e. magnetite in the XRD analysis) containing long slag stringers (Fig. 6). This internal structure demonstrates how the original ferrite and inclusions have been folded over in the fabrication process.
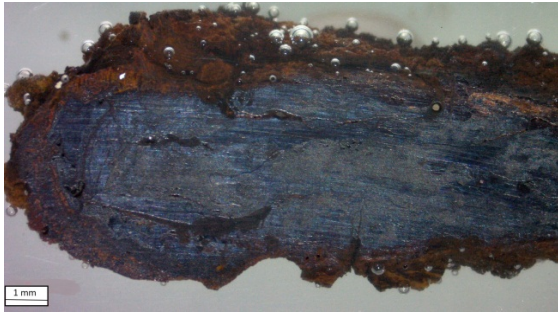
Figure 6: Stereomicroscope image of the dagger's cross section

Figure 7: Polarized light microscopy image showing the corrosion profile of the dagger (starting from left to right in the image)

Figure 7 shows a PLM image of the corrosion profile and internal structure of the dagger. The image shows that the external layer of the dagger contains a reddish brown corrosion product, which is consistent with the detection of goethite and lepidocrocite in the XRD analysis. The areas appearing grey in the external and internal parts of the daggers cross-section can be attributed to the magnetite detected in the XRD analysis. The black inclusion appearing in the internal part is a slag inclusion, while the white lustrous spots are the etched surviving ferrite grains. The image shows clearly that the dagger greatly converted into magnetite. This explains its resistance to etching and its slight magnetism as little metal is remaining in the artifact. It also explains the retained overall shape of the corroded dagger because of the compactness of magnetite (Balos et al., 2009). Comparing the external and internal parts of the cross-section shows that the external part has signs of hammering, carburization and heat treatment during the manufacturing process as appearing from the distortions and graphite particles. This special treatment aimed to increase the hardness and sharpness of the edges of the dagger (Pense, 2000). The effect of this special treatment did not reach the internal part, which appears as a relatively plain area of magnetite that resulted from the corrosion of un-carburized ferrite.
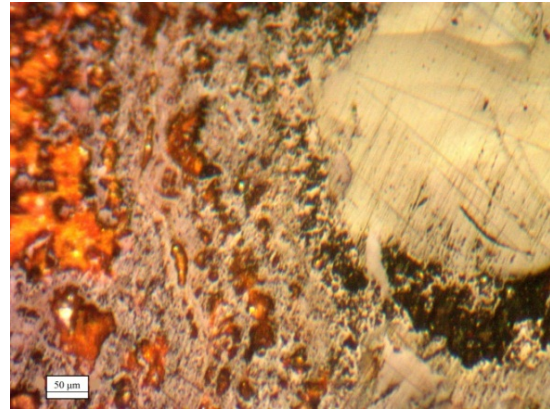
The result of this metallographic examination is consistent with X-ray radiography examination result where the lighter and cracked zone concentrates at the edges of the dagger (goethite and lepidocrocite area) and the denser zone (magnetite area) concentrates in the internal central area of the dagger.

Further metallographic investigation of other parts of the dagger shows surviving pearlite and ferrite colonies in the external carburized layer (Fig. 8). There is also surviving ferrite and pearlite grains in the internal cracks that also contain slag stringers and corrosion products (Fig. 9). This suggests that the original iron bloom used to manufacture the dagger had been forged to remove the entrapped slag, then the compact iron was subjected to a carburization and hot forging process. This included heating in a bed of hot charcoal so that some carbon diffused into the iron, then the artifact was hot forged to give it the final shape. The presence of ferrite and relatively spheroidal pearlite suggests that it had a hypoeutectic structure (<0.8% C) and the hot forging was at a temperature around 700°C (Maxwell-Hyslop et al., 1978).
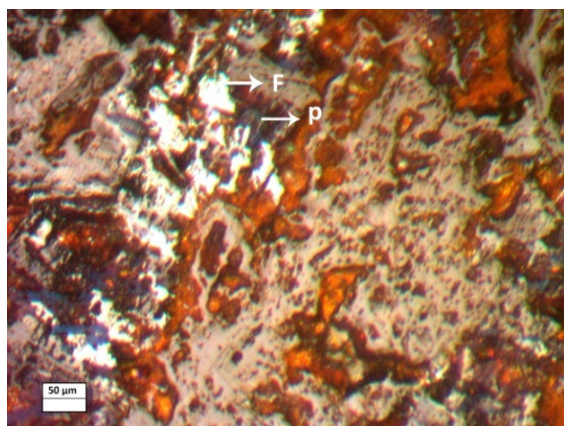
Figure 8: Surviving pearlite (P) and ferrite (F) colonies in the external carburized layer
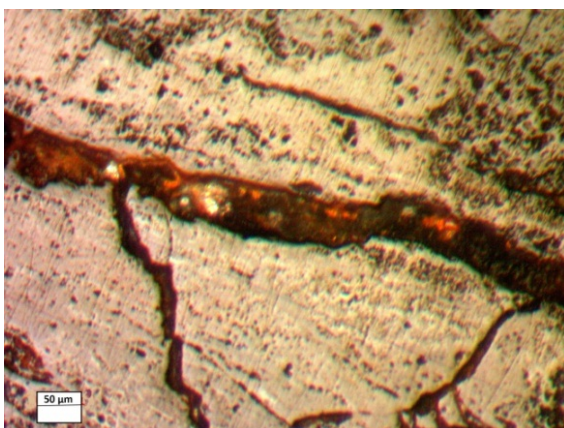

Figure 9: Surviving ferrite and pearlite grains in the internal cracks of the dagger

4.5. Conservation Treatment

The external soil and accretions were cleaned with the aid of the stereomicroscope using mechanical cleaning tools such as scalpels, dental tools and soft bristle brushes.

Although akaganeite was not detected in the corrosion products, there could be a slight amount of chloride ions that need to be removed to stabilize the artifact before its final treatment (Jegdić et al., 2012). Since the dagger is highly mineralized, sodium hydroxide alkaline treatment was selected to remove any chloride ions present in the dagger (Keene, 1994). This treatment will increase the stability of the dagger when stored after the treatment, which means it will not need special environmental conditions required for artifacts infected by the corrosion accelerator chloride ions (Selwyn, 2004). After one week of immersion in a liter of the 0.5M NaOH solution, the chlo-

ride ion reading was 4 ppm. This is a low level of chloride ions and indicates that the corrosion products are stable. Another fresh 0.5M NaOH solution was prepared and the dagger was left in it for a week. The chloride ions reading of the solution was 2 ppm. The treatment was stopped at this stage as the level of chloride ions diffused to the alkaline solution was too low. Heavily mineralized artifacts should only be left in alkaline solutions long enough to remove as much of the chloride ions as possible without damage to the shape of the artifact (Argyropoulos et al., 1997). The treatment did not cause a loss or damage to the mineral preserved organic deposits on the surface of the dagger, which agrees with what is reported in the literature (Wang et al., 2008). No attempt was made to measure the amount of chloride ions left in the dagger after the treatment as this requires digesting the artifact.

The artifact was then brushed several times with a 5% solution of tannic acid. This corrosion inhibitor reacts with the iron to form a blue-black ferric tannate protective film, which in the short term will inhibit the most susceptible areas from reacting with water vapor. It also produces a uniform finish that enhances the appearance of the artifact. Several layers were applied on the surface of the dagger. Brushing and drying was repeated until the artifact was coated with an even, coherent film of black ferric tannate (Logan, 2007).

Finally, to consolidate and protect the artifact from atmospheric humidity and corrosive ions, it was coated by a 5% solution of Paraloid B72 applied by brushing. This coating was chosen because of its availability and proven effectiveness and reversibility. Three layers of Paraloid B72 were applied over the ferric tannate layer. Once the final Paraloid B72 coating layer dried, it was topped with a layer of microcrystalline wax to decrease its glossiness (Scott and Eggert, 2009). Figures 10 and 11 show the dagger after the end of the treatment.

Figure 10: The dagger's obverse after treatment

Figure 11: The dagger's reverse after treatment

5. CONCLUSION

The analytical examination of the Iron Age dagger from Tall Abu al-Kharaz showed that the formed corrosion products were stable, however slightly cracked and obscuring the surface details. Metallographic examination showed that the dagger had greatly converted into magnetite. It also showed signs of hammering, carburization and heat treatment during the manufacturing process of the artifact. The formation of the compact magnetite had a protective effect on the artifact and maintained the overall shape of it. However, the conservation treatment was needed to consolidate and protect the artifact from atmospheric humidity and corrosive ion in the environmentally uncontrolled storage area.

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