Characterization and Treatment Study of a Handcraft Brass Trumpet From Dhamar Museum, Yemen

by Mohamed M. Megahed, Mohamed M. Abdelbar

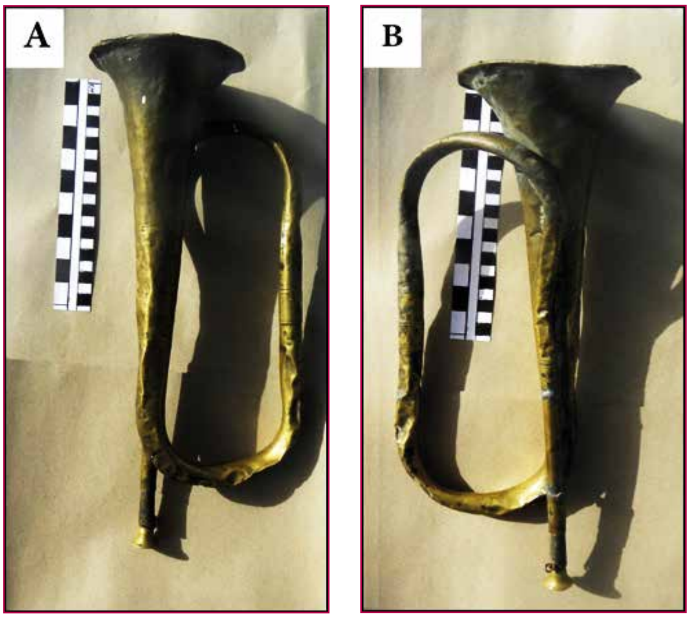

Fig. 1 - Show both sides of the trumpet surface (a, b).

The goal of this paper is to present an analytical and conservation study of a Brass Trumpet from the handcrafts collection of the Dhamar Museum (Yemen). Metallographic examination and scanning electron microscope were performed to identify the microstructure of the alloy.

Brass alloys are mainly composed of copper and zinc, copper is the main component, while zinc is about 10 and 45% (Lanord F. A. 1980; Scott D.A. 1991; Shreir L.L et alii 1994).
Brass alloys in early times were equally binary alloys consisting of 90-70% copper and 10-30% zinc, the colour of the alloy becomes yellower as the proportion of zinc increased (Hodges H.,1964). The colour ranges from a red similar to pure copper through a pleasing yellow to white, above about 45% zinc (Selwyn L., 2004).
In order to make brass, calamine was melted with copper, it is a compound of zinc carbonate and zinc silicate, which was the main ore from which the zinc was, extracted (Van der Heide G. J., 1991). Great caution should be taken when making an alloy of copper and zinc, because the boiling point of zinc (917°C) is below the melting point of copper (1083°C) (Craddock P.T., 1995). For this reason, zinc was not smelted in antiquity, but calamine ore was added to the molten copper in a crucible, the carbon and oxygen being then given off as gaseous carbon dioxide while the other impurities formed, in addition to silica, a slag that could be skimmed off the surface of the molten metal (Hodges H., 1964, p.69).
This process was used into the 19th century until it was replaced by the melting together of zinc and copper metals (Selwyn L., 2004, p.53). It is well known that the addition of can cause a significant change in the color of the alloy and its chemical prosperities as well as its ability to resist corrosion (Scott A. D., 2002).
Brass alloys have been used in making wind instruments since the 16th century, and became an essential part of the orchestra during the 18th century, mainly due to its good properties like malleability, durability, solderablity, the ease of manufacturing of the instruments and its good corrosion resistance. This is also due to the sound produced by a brass instrument could not be compared by that of any other alloy (Baines A., 1993; Leencwadi L., 2011; Deck C., 2016).
The first brass instrument is a trumpet found in a drawing in King Tutankhamen's tomb in Egypt dating to 1500 BCE. In this drawing, two trumpets were found, one was made of bronze (or possibly copper) and the other of silver; both were made by means of flattening the metal with a hammer ((Van der Heide G. J., 1991, p. 126).
The main instruments of the brass family include the trumpet, horn, trombone and tuba. The trumpet is the highest-sounding member of the brass instruments whereas the tuba is the lowest sounding. The trombone is larger than

the trumpet, and has a softer sound (Leencwadi L., 2011).
The processes of flattening and hammering brass were the most important techniques used in the making of trumpets, horns, and trombones until about 1820. By using the appropriate hammers, anvils, and working procedures, the manufacturers were able to form the required bell shape (Van der Heide G. J., 1991, p. 130).
It is well known that brasses are copper zinc alloys which are extensively used in many fields and they combine many of the favorable features of both copper and zinc. Since zinc is the active component of brass, it has a tendency to corrode leaving its surface enriched in copper. The corrosion of a metal is often considered as an inconvenience because it implies a change of the objects in the course of time, this damage caused to a metal by chemical, electrochemical or even biological reactions between metal and the surrounding medium (Hammouch H. et al., 2007).
The exhibition of an old object is accompanied by a change depending on the environmental conditions, which can sometimes accelerated objects degradation [Wadsak M. et al. 2000; Organ R. M., 1963; Tylecate R.T., 1979; Cronyn J. M., 1990;).
The study of the causes of corrosion is essential to develop a control technology and to improve the means of protection. It is anticipated that a thin oxide film (patina) is immediately formed on Brass artifacts surface, when it is exposed to an Oxygen- containing environment. The thickness of this passive oxide film is about a few nanometers and acts as a protective barrier in the corrosive media (Kannan S.et al., 2005). However, the film isn't sufficient during long- term implantation procedures as they are susceptible to corrosion- related problems and lack of biocompatibility to ensure new bone formation at the implant site. A metallic surface, which quite passive due to protective oxide layer may still allow a significant release of ions or atoms into the surrounding tissue under certain conditions. Hence, the development of Brass coatings on a passivated surface tends to ensure bioactivity and resists the metal leach from the surface.
The aim of this work is to represent an analytical and conservation study of brass Trumpet from the handcrafts collection of Dhamar museum in Yemen.
To achieve that Metallographic examination and scanning electron microscope were performed to identify the microstructure of the alloy. Energy dispersive spectroscopy EDS and x-ray fluorescence (XRF) were used to identify the alloy composition used for manufacturing the musical instruments and the solder joints. X-ray diffraction (XRD) was used for the identification of the crystalline corrosion products. Finally exploited the previous info., in a systemic treatment and conservation for the selected object.

MATERIALS AND METHODS: DESCRIPTION AND CONDITION

The investigations and treatment were conducted on a trumpet from the collection of traditional handcrafts in Dhamar museum, Yemen. This trumpet dates back to 1850 A.D, made of brass alloy, it consists of a long oval tube, with two curves, a mouthpiece at one end and a wide bell at the other. The dimensions of the trumpet are 41 cm length, 12 x 11 cm diameter of the front part and 2.2 cm diameter of the back port (Figures 1a, b &2). The trumpet suffered from deterioration aspects such as a thin black layer of corrosion products, calcareous spots, missed parts, micro cracks and wrapped parts (Figure 3 a, b).

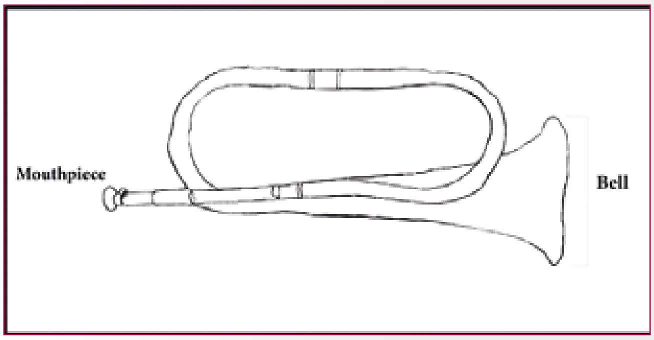

Fig. 2 - Shows the trumpet components.

EXAMINATIONS AND ANALYSIS

Metallographic and Scanning Electron Microscope were used to study the surface morphology and microstructure of trumpet, corrosion products were analyzed by X-Ray Diffraction and X-Ray Florescence was used to identify the chemical composition of the trumpet. The examinations and analyses were performed as the following:

Metallographic Examination (ME)
A ZEISS, AXIO Scope partorus was used to examine two samples from the selected object,
the metallographic examination results are given in (Figures 4. a, b).

Scanning Electron Microscope Examination and Energy Dispersive Spectrometry (SEM&EDS)
Fig.5 a, b show SEM&EDS examination which detects the microstructure, the appearance of deterioration spots and Figs. 6 &7 show the quantitative chemical composition of the trumpet analyzed using an Inspect S50 (FEI).

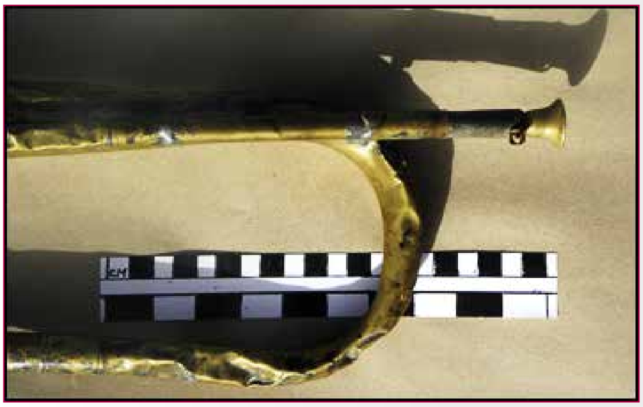

Fig. 3 - Show the deterioration aspects of the trumpet such as a thin black layer of corrosion products, missed parts, micro cracks, wrapped parts and the soldering material (a, b).

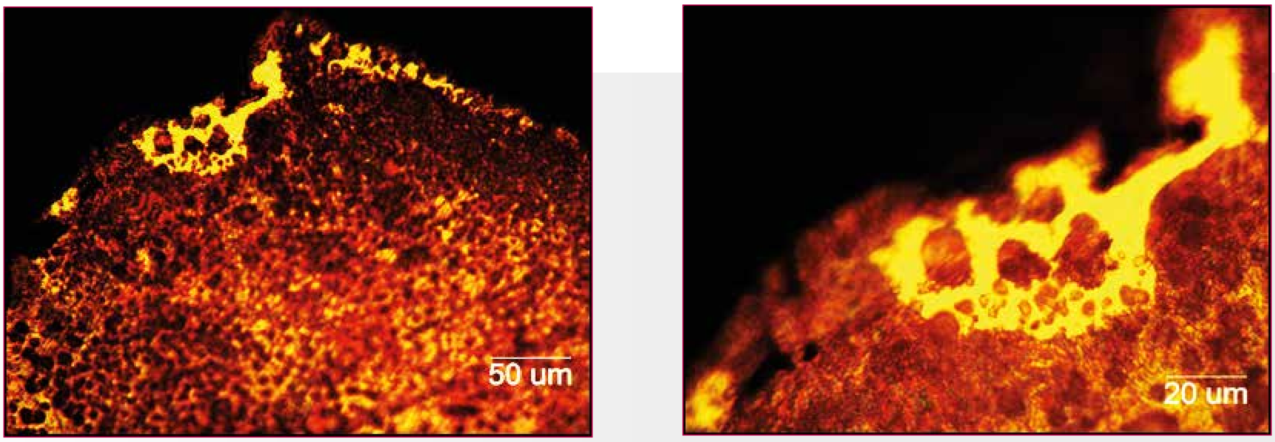

Fig. 4 - Metallographic examinations of the trumpet show: the grains of the alloy filled with pitting corrosion (a). The microstructure of the alloy, selective corrosion and the distortion of the edge (b).

X-ray Fluorescence analysis (XRF)
X-Ray Fluorescence is a non-destructive, powerful and easy to use technique for the elemental analysis of a wide variety of materials; two small samples from the trumpet and the solder joint were analyzed by this technique to determine its composition, by using: NITON/XL8138 (USA), driven with software version 4.2E. The results are shown in Table (1).

X-ray Diffraction analysis (XRD)
Sample from corrosion products of the trumpet was analyzed by using a Philips X-ray Diffractometer with Cu Kα radiation. The aim of this analysis is identification the corrosion compounds in order to decide whether it is authentic, stable and suited to certain kinds of conservation treatment. This information can assist in choosing the best environment of display or storage for the trumpet. The obtained diffraction-scan given in Figure (8) and the identified compounds represented in Table (2).

TREATMENT AND CONSERVATION
Brass tends to oxidize (tarnish) quickly when exposed to air, which is a major reason why most brass is given a clear coating to prevent future tarnishing (Deck C., 2016). The trumpet was subjected to mechanical cleaning with tooth brushes to remove dry dust and dirt. After that, a paste of precipitated chalk and water was used by a soft cloth to remove the residual rouge of corrosion products. The trumpet was washed carefully by distilled water and dried by immersion in acetone, finally it was isolated with 3% Paralaid B-72 dissolved in Ethanol, figure (9. a, b).

RESULTS AND DISCUSSION
Metallographic examination of the trumpet showed: a) the grains of the alloy filled with pitting corrosion, b) Shows the microstructure of the alloy, selective corrosion and the distortion of the edge (Fig.4 a, b). These deterioration aspects due to the manufacture process, the brittleness therefore, might be due to the chemical composition and micro-chemical structure as well as to the ageing process, inducing a drastic change in the metallurgical and micro-chemical structure of the object. An electrical potential between the copper and zinc, in the presence of water, oxygen and other impurities, causes an exchange of electrons resulting in intergranular corrosion, which weakens the brass and makes it especially susceptible to stress damage. Any attempt to reshape brass, such as dent removal or straightening, without prior heat treatment will result in distortion and damage, especially if corrosion-related micro-fissures already exist. Because brass must be heat-treated before working, any specific orientation in the material due to original manufacturing techniques will be obliterated (Barclay B., 1989). In the selective corrosion, corrosion processes cause removal of one component of alloy. A typical example of this form of corrosion is dezincification of brass, when part of the original material, alloy of zinc and copper, turns to spongy copper. Dezincification plays its role in the formation of corrosion cracking of brass. All brasses having Zn content higher than 15 wt. % incline to dezincification, the mechanism of dezincification lies in dissolution of Cu and Zn, and Cu subsequently re-deposits. The dezincification often happens in waters containing chlorides and is a frequent cause of fail-

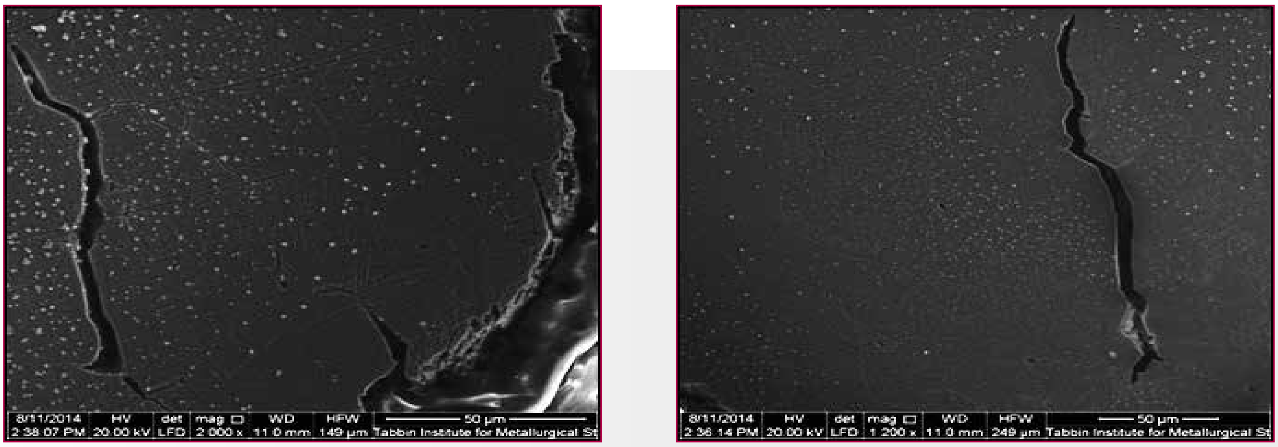

Fig. 5 (a, b) - SEM scan of the alloy shows the white block of zinc dispersed in the alloy and stress corrosion (a). A crossed section shows the stress corrosion caused micro cracks dispersed in the alloy (b).

Samples	Cu	Zn	Sn	Pb	Total
The trumpet	61.14	32.68	5.90	0.28	100%
The solder	0.47	0.53	8.61	90.39	100%

Tab. 1 - Shows XRF analysis results of the trumpet and the solder join.

ing of the brass fittings in water circuits (Novák P., 2007). Localized atmospheric corrosion can also be observed on the surface of brass and copper-zinc alloys due to the reaction of the distinct alloying metals in contact with the environment. In this case, the corrosion is referred to as selective corrosion.

SEM examination showed the white globules of zinc dispersed in the alloy and stress corrosion, which caused micro cracks dispersed in the alloy (Fig.5 a, b). Stress corrosion cracking (SCC) is typical especially for brass, but is less for other copper alloys. The effect appears under tension stress above a certain limit in a polluted (industrial) atmospheric environment, especially when it contains ammonium compounds. SCC affects copper alloys containing 20%or more zinc, but only rarely the other alloys (Knotkova D. & K. Kreislova, 2007). The EDS qualitative analyses proved the trumpet was manufactured from brass alloy (Figs. 6, 7).

XRF analysis was carried out to determine the alloy composition and the solder joints of the trumpet component. The results revealed that the trumpet was manufactured from a yellow brass alloy containing 61.14% Cu, 32.68% Zn, 5.90% Sn and 0.28% Pb (Table no 1).

Yellow Brasses (containing 23 - 41 % of zinc) as the major alloying element and may contain up to 3 % of lead and up to 1.5 % of tin as additional alloying elements. The brasses are generally divided into three categories depending on the phase type: alpha brasses with up to about 35% zinc; alpha + beta brasses with between 35% and 46.6% zinc; and beta brasses with between 46.6% and 50.6% zinc. As zinc content increases the brittle δ phase begins to appear and thus alloys with more than 50% zinc are generally avoided. Beta phase brasses are very much harder than the alpha and can withstand very little cold-working. The beta phase begins to soften at about 470 °C (as the lattice changes from an ordered to a disordered state), and at about 800 °C it becomes much easier to work. Based on the Cu–Zn phase diagram, the solubility of Zn in Cu can be as high as approximately 32.5 wt. % , brass alloys in this range (α-brass) are ductile, easily cold-worked, can be rolled into thin sheets, and have good corrosion resistance in a salt-water atmosphere (Ashkenazi D. et al., 2014).

The alpha brasses, which include most of the ancient specimens, are much better when they are cold-worked and annealed rather than hot-worked because, if hot-worked, impurities tend to segregate at the grain boundaries and make the brass very weak (Scott D.A., 1991, pp. 19-20).

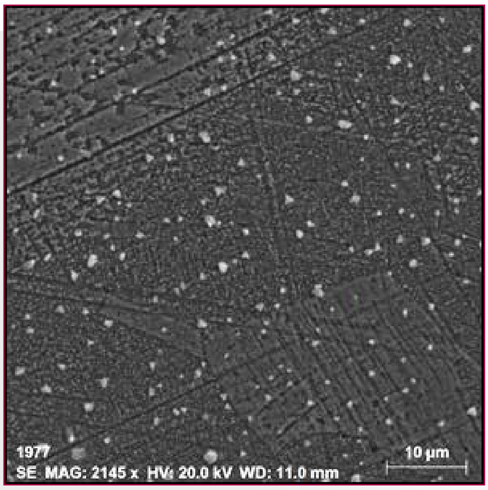

Fig. 6 - Shows SEM image shows the white globules of zinc dispersed in the alloy.

The presence of lead in a small amount about 0.28% as purities may be affects the mechanical properties of the material to a considerable degree. Sixteenth-century brass is a characteristically inhomogeneous material that includes several trace elements. Among these, lead (Pb) is the most significant one, as it affects the mechanical properties of the material to a considerable degree. Different raw materials as well as different production processes were employed in the manufacture of early brass. Modern manufacturers use the direct process, melting the two principal elements of copper and zinc directly into each other, but early craftsmen used the cementation method (Vereecke H.W. et al., 2012).

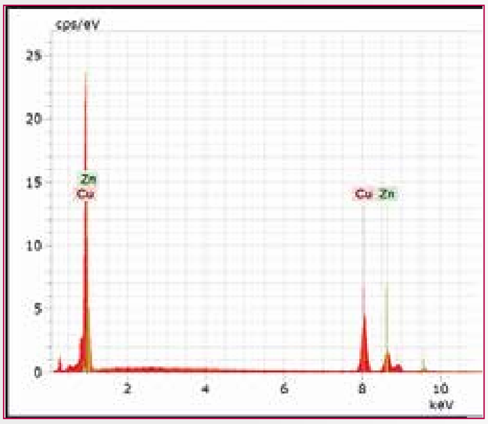

Fig. 7 - Shows the corresponding EDS analysis of the trumpet.

Samples	Compounds		
	Major	Minor	Traces
Trumpet Corrosion products	Covellite CuS (33.57%) Paratacamite $Cu_2(OH)_3Cl$ (32.98%)	Massicate PbO (16.74%)	Cuprite Cu_2O (9.26%) Calcite $CaCO_3$ (7.44%)

Tab. 2 - Shows XRD analysis results of corrosion products of the trumpet.

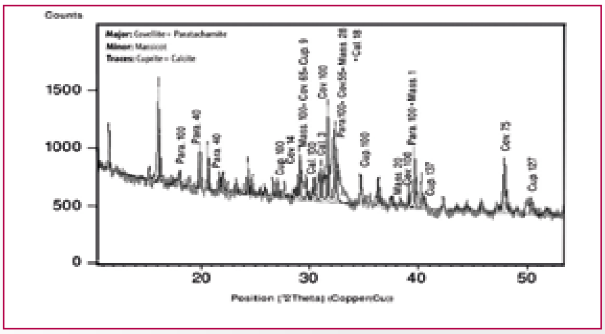

Fig. 8 - Shows XRD scan for the corrosion products of the trumpet.

In general, copper and brass alloys are highly resistant to atmospheric corrosion due to the formation of protective layers of corrosion products, which reduce the rate of attack. Thickness and composition of the corrosion product layer formed are governed by the relative humidity and pollution of the environment. Inside the instruments that are regularly played, a very thin film of water at the surface can be present for quite a long time and damage of the artifacts (brass instruments) might occur on long-term (Elsener B. et al. 2016).
The interior corrosion in brass wind instruments dues to the effects of moisture and saliva inside the instruments, which consequently increases the risk of metal corrosion inside the instruments.
Measurements have shown that after 5 min playing the relative humidity (RH) inside the instruments exceeds 90% and it takes several days to reach ambient RH again (Scott A. D., 2002, p.6).
Thus conditions for atmospheric corrosion, the formation of a very thin film of liquid water at the surface [Craddock P.T., 1995, p. 295; Hodges H., 1964, p.69; Selwyn L. 2004, p.53.) are in principle present for quite a long time when the instruments are regularly played and damage of the artifacts (brass instruments) could be possible on long-term (Elsener B. et al., 2016).
The solder joints of the trumpet are a soft soldering assembled totally with tin-lead solder containing 90.39%% Pb and 8.61% Sn, 0.47% Cu and 0.35% Zn (Table no.1). Soft solders have been used to join pieces of metal together at low temperatures (below 300°C), and produce joints in copper, bronze, brass, and silver objects (Maryon H., 1941; Ashkenazi D. et al., 2014).
It is generally composed of lead and tin mixed in proportions that vary from as little as 30% lead (and 70% tin), to as much as 98% lead (and only 2% tin) ((Maryon H., 1971; Goffer Z., 2007). The flux is zinc chloride, resin, tallow, or some other oily substance was used to prevent film formation and then burn off (Cronyn J. M., 1990, p. 162).
This technique of soldering was used to attach a very small piece of metal to an under layer of the same metal (Ashkenazi D. et al., 2014, p. 51). In the trumpet, all connections between tubes were made by means of a lap joint, in which usually each tube fits into the next tube, moving toward the bell (Van der Heide G. J. , 1991, pp. 122-150). But in the trumpet the soft soldering process may be performed in a later time to fix the disjointed tubes.
As a consequence of the joining of different metals, the behavior and the rate of corrosion are remarkably influenced by the intimate contact between metals with different electrochemical potential. This contact induces the more reactive and less noble metal to become anodic in a couple strongly conductive to corrosion.
X-Ray diffraction analysis of the corrosion products of the trumpet revealed the presence of different minerals including Covellite Cu S, Paratacamite $Cu_2(OH)_3Cl$, Massicate PbO and traces from Cuprite Cu_2O, Calcite $CaCO_3$ (Fig. no. 8&Table no.2).
The formation of sulfates and chlorides (Covellite Cu S, Paratacamite $Cu_2(OH)_3Cl$), resulted from the interaction between surrounding environment and the trumpet. The impact of sulfides on the corrosion of copper alloys has received considerable attention, including published reports documenting localized corrosion of copper alloys by Sulfate-reducing bacteria (SRB), a diverse group of anaerobic bacteria isolated from a variety of sulfur-containing. A porous layer of cuprous sulfide with the general stoichiometry forms in the presence of sulfide ions. Copper ions migrate through the layer, react with more sulfides, and produce a thick, black scale, which can be altered by oxygen from the environment to a complex sulfide-oxide scale. The sulfide scale does not confer much protection against further attack, but the sulfide-oxide scale provides even less. Corrosion products on copper alloys were more adherent and in some cases difficult to scrape from the surface. In all cases, bacteria were closely associated with sulfur-rich deposits. There is one class of conditions under which biofilms appear not to produce sulfide minerals.
The presence of basic copper chloride (paratacamite) is related to the saline nature of the surrounding medium, whereas the trumpet was preserved. Paratacamite always found as a powdery, light green secondary corrosion layer on the patina surface, while the compound of Atacamite occurs as a sugary-looking coating of dark green glistering crystals (Scott D.A., 2002, p.124). Often this dark green crystalline Atacamite is altered to a paler green powdery product of paratacamite (Gettens R.J., 1963; Frondel C. 1950).
The existence of lead in the soldering alloy as globules or independent islands causes a non-homogenous structure. In this case a galvanic corrosion tendency when the alloy is exposed to moist air or soil. As a result of this reaction lead corrosion products such as Massicate (PbO) was deposited on the object surface.
The presence of Cuprite (Cu_2O) as a trace in the corrosion products due to the selective corrosion of the main alloying element, which is re-deposited after dissolution onto the surface of the objects, thus forming a copper enriched layer, Cuprite is the most widely occurring alteration mineral

Fig. 9 (a, b) shows the both sides of the trumpet after treatment and Conservation.

of ancient copper and its alloys. Most of it was concealed beneath overlying green basic salts of copper. It is formed as a result of reaction between the metal and oxygen that present in every environment but in different proportions. Rust layer on ancient brass is constituted by Cu_2O (Cuprite) and Cu O (Tenorite). The metal/ oxide interface is relative irregular and some intergranular penetration can be detected.
The presence of Calcite [$CaCO_3$] crystals as an identified compound on the trumpet surface, which is most probably formed by the reaction of soluble calcium bicarbonate with hydroxide ions produced in the Cathodic reduction of Oxygen (North N. A. & I.D. Macleod, 1987), indicated that the surrounding medium of the trumpet was a calcareous aerobic medium. Such medium usually has high carbon dioxide and may be chemically very aggressive because the carbon dioxide may react with water to form carbonic acid, which may attack metals directly and prevent the formation of a protective film on the metal surface.
Cleaning is one of the most common operations in conservation. The choice of method for cleaning depends on what is required from the object, what is made of, and what condition it is in. It is known that the mechanical cleaning of metals is preferred method for removing disfiguring corrosion. It allows more control and has less effect on the metal alloy. The Mechanical cleaning carried out for removing the external crust corrosion products; the trumpet was subjected to mechanical cleaning with tooth brushes to remove dry dust and dirt. After that, a paste of precipitated chalk and water was used by a soft cloth to remove the residual rouge of corrosion products. The trumpet was washed carefully by distilled water and dried by immersion in acetone, finally it was isolated with 3% Paralaid B-72 dissolved in Ethanol (Fig. no. 9 a, b).

CONCLUSIONS

ME, SEM and XRD results show the occurrence of selective localized or general chlorine corrosion phenomena induced also by the separation of the alloying elements, which creates reactive electrochemical areas.
The chemical composition, the micro-chemical structures and metallurgical feature of the object have been determined and can be used to identify some technological aspects of the ancient manufacturing processes.
The morphology of the surfaces and the elemental compositions of the corrosion products depend strongly on the chemical composition of the alloys.

Abstract

Brass is more malleable and has better acoustic properties than pure copper or zinc; consequently, it is used in a variety of musical instruments, including trumpets, trombones, bells, and cymbals. The overall goal of this work is to represent an analytical and conservation study of brass Trumpet from the handcrafts collection of Dhamar museum in Yemen. Metallographic examination and scanning electron microscope were performed to identify the microstructure of the alloy. Energy dispersive spectroscopy EDS and x-ray fluorescence (XRF) were used to identify the alloy composition used for manufacturing the musical instruments and the solder joints. X-ray diffraction (XRD) was used for the identification of the crystalline corrosion products. Finally, exploited the previous info., in a systemic treatment and conservation for the selected object.

Parole chiave

Brass; Handcrafts; Trumpet; Characterization; ME; SEM&EDS; XRF; XRD; Conservation

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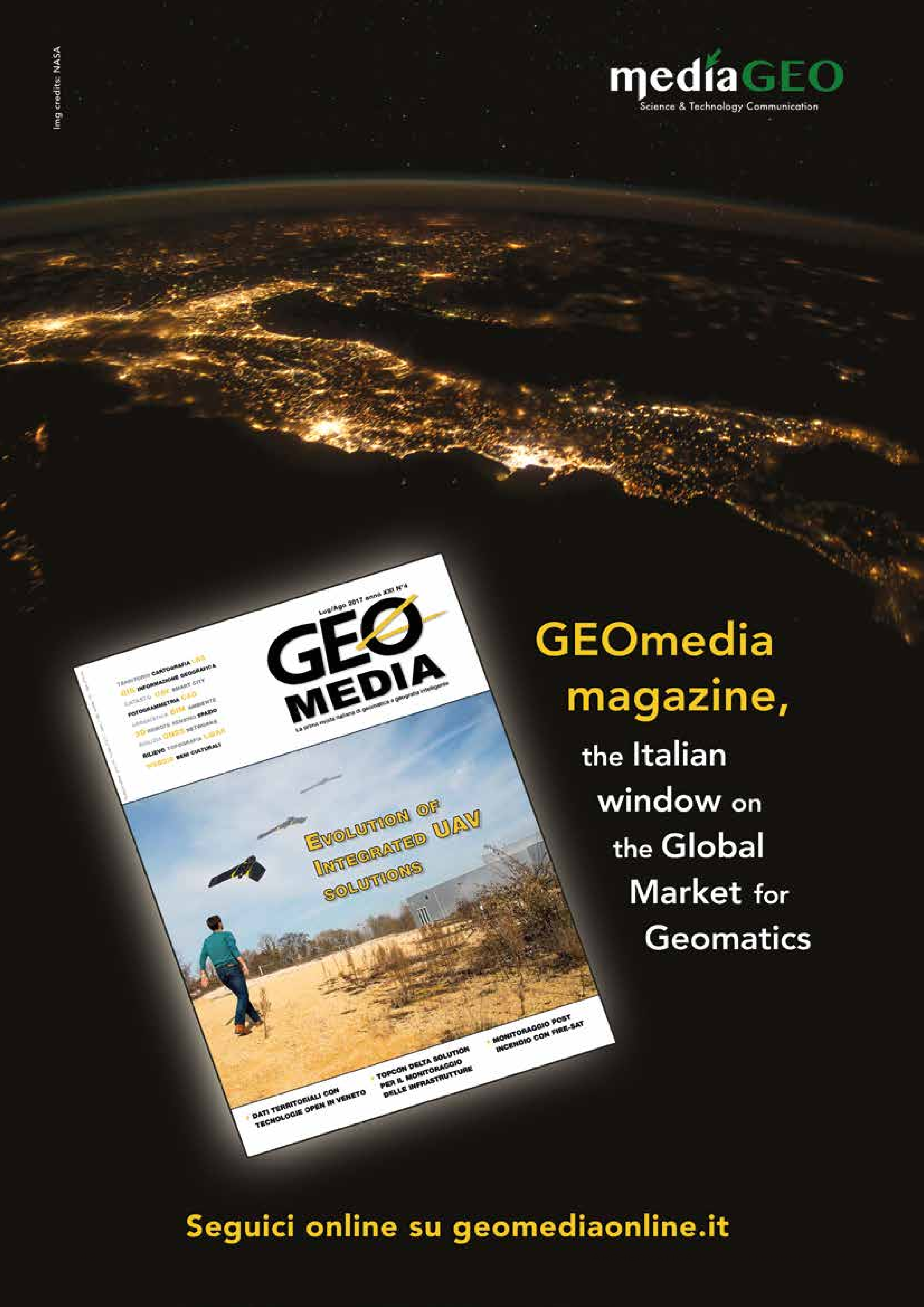

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