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Transdisciplinary collaboration for the multiscale description of corrosion structures in metallic heritage

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Introduction

The nature of heritage metals (archeological and historical), their history, and the environments to which they have been exposed are all parameters that influence their long-term conservation. The professionals who study them (archeologists, collection managers, museum curators, conservators, conservation-scientists, corrosion experts, and archeometallurgists) perceive them differently depending on their competences and level of expertise. Some limit themselves to the visual (macroscopic) description of the objects studied and their surface appearance, while others examine the materials they are made of at the microscopic level in order to specify their fabrication techniques, study the corrosion mechanisms developed, and predict their future evolution.

Everyone has their own approach methodology, vocabulary, and examination methods to describe what they see. The information obtained is not always shared between the communities concerned and leads, in fact, to an often partial perception of the objects, based on the conservation conditions of the materials.

The online and freely accessible MiCorr application (www.micorr.org), initiated in 2016 and optimized since then in the framework of several projects including Metal-PAT of the Interreg France−Switzerland program (2014−20) [1], aims to promote the sharing of information between communities involved in the conservation of heritage metals and more specifically the diagnosis of their preservation state [2,3]. This sharing is based on object sheets collecting all the information available on these objects and transmitted by those responsible for them—archeologists, collection managers, or

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museum curators (context of discovery and use, dating, inventory number, metal family, etc.), the conservators who have documented them (non- or microinvasive observation of the corrosion structures developed in order, for example, to specify the limit of the original surface of the object), the conservation scientists/corrosion experts who have assisted the latter in the analysis of these structures, on the basis of samples studied in cross-section, and the archeometallurgists who study the metal microstructure to determine its elaboration methods and provenance. Thus, the MiCorr database is enriched by the contributions from all four parties, which improves the relevance of the application.

MiCorr is not just a database. It is also a diagnostic support platform, consisting of three search engines enabling the user to consult the available entries and compare the data observed on an object under study with those contained in the database.

The consultation is possible through keywords ("By keywords" engine). The user can also identify the family of the metal observed from its physical characteristics and surface appearance ("By visual inspection" engine). They also can construct representative stratigraphies of the corrosion forms they have observed on their objects using the "By stratigraphy representation" engine and compare them with those of the database entries [3,4]. Only the last two engines are detailed in the next sections.

Multiscale description of heritage metals

Conservation professionals observe heritage metals at different scales and deduce different but complementary information. In the following, we specify, according to the interested user the needs for documentation and the solutions provided via the MiCorr application.

Global perception of objects and identification of metal families

Collection managers and museum curators see objects as a whole, taking into account the accompanying metadata. This allows them to appreciate the surface appearance of the objects without having a clear perception of the nature of the metal and its preservation state. From this, they deduce the most optimal conservation conditions. Due to the complexity of the chemical processes leading to surface alteration in various environments, errors of assessment are possible. MiCorr "By visual inspection" search engine consolidates this preliminary appreciation.

In engineering, it is common to identify metals by their magnetism, color, and other physical properties (weight, hardness, brightness, etc.) [5]. In conservation, the metal is first characterized by its preservation state: fully or partially visible, or nonvisible. Indeed, the development of the observed alteration (corrosion, dust deposit, etc.) will induce or not urgent and costly intervention strategies.

For the fully visible metal, we are interested first in any surface treatment or/and coating that may distort the appreciation of the underlying metal, then in its physical characteristics: magnetism, weight, presence of a hallmark (purity grade), hardness, and brightness. While magnetism and the presence of a hallmark call for a yes/no answer, for the other characteristics, the answer is more nuanced because it is based on the experience and expertise of each professional.

Partially visible and nonvisible metals can be artificially corroded (patina of outdoor bronzes) or naturally (tarnishing, localized, or generalized corrosion) [6]. Once the forms of corrosion are characterized, the querying on the residual metal is carried out as before.

The MiCorr "By visual inspection" search engine consists of a decision chain built on a series of questions/answers aiming first at characterizing the forms of corrosion developed and then the residual metal, discarding with each question a certain number of metal families. The family(ies) of metals proposed at the end of the query remain(s) hypotheses that the user must validate by consulting the descriptive sheets of each family provided and detailing all the properties of the metals concerned (including those integrated in the decision chain).

The tool is intuitive: the questions are straightforward and the user answers by clicking on the images reflecting their choice (Fig. 2.3.1a). The user follows in real time the progress of their query. Two levels of information are available: the list of probable metal families (Fig. 2.3.1b) and the details of the questions and answers given in addition to the excluded metal families (under "Previous Questions" on Fig. 2.3.1b).

Curators and collection managers trained in the use of this search engine are not all metal experts. They therefore appreciate the information provided and validated by conservation professionals, which enable them to better preserve the artifacts in their care.

Description of the observed corrosion forms

Conservators have sufficient expertise to easily identify heritage metals based on their surface appearance and physical characteristics. They are also able to recognize classical forms of corrosion: generalized corrosion on ferrous, cuprous and lead-based metals, localized corrosion on copper ("bronze disease") and aluminum alloys (pitting with white efflorescence), etc. [6]. The description of the observed corrosion structures using the MiCorr "By stratigraphy representation" search engine allows them to distinguish between corrosion layers above the limit of the original surface (OS) and corrosion layers below it and to propose a cleaning of the surface in order to get closer to the OS [7]. It also allows them to verify the activity of certain localized corrosions by detecting characteristic corrosion products initiating them, such as nantokite at the bottom of the pits of copper-based alloys [8].

Establishment of stratigraphies of corrosion structures under binocular microscope

Conservators rarely have the possibility to take samples from the objects they study. Their diagnosis is therefore based on the simple macroscopic description of the corrosion forms observed visually and under binocular microscope. While visual examination allows an overall assessment of the corrosion forms present, such as those developed on bracelet n°67 from the Bussy/Pré-de-Fond site in Fribourg, Switzerland

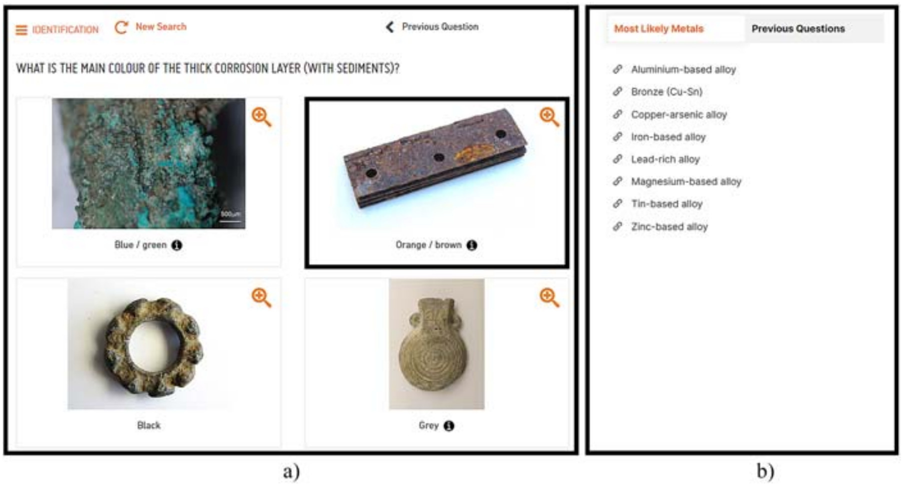


Figure 2.3.1 MiCorr "By visual inspection" search engine interface: (a) Visualization of a question and its answers, with the selected answer highlighted in the black frame and (b) tracking of query's progress with the list of probable metal families.

(Fig. 2.3.2a), a binocular microscope is used to investigate the corrosion structures in place. The strata (sediments, corrosion products, etc.) are progressively and locally cleared with a scalpel. The approach is both visual and tactile as shown by the characteristics that can be described through this mode of observation (color, cohesion, compactness, hardness of the strata [assessed by the possible mark left by a hardened steel tip and equivalent Mohs hardness], etc.). On the basis of these observations, the conservator creates, according to Bertholon's descriptive method [7], a schematic representation gathering all the observations on the different strata constituting the corrosion forms. The user first identifies the strata and encodes them according to their nature: S—sediment, M—metal, CP—corrosion product, etc (Fig. 2.3.2b). The most representative corrosion structure(s) is (are) selected (red frame, Fig. 2.3.2b) and then represented digitally via MiCorr by filling in as best as possible and for each stratum's fields of characteristics (morphology, texture, microstructure, composition, interface) (Fig. 2.3.2c) the available subcharacteristics: shape, continuity, relative thickness, cracks, and nature of the interface with the upper stratum.

Comparison with the stratigraphies of MiCorr database

This digital stratigraphy is then compared to those of the objects in MiCorr database. This operation is made possible by an algorithm that compares stratum by stratum their subcharacteristics to which an importance value is assigned (Fig. 2.3.3, left). The results are given in the form of a matching percentage (%), decreasing from the studied metal (100%) to the proposed metals of the database (Fig. 2.3.3, right). We note, for the example of Fig. 2.3.2, that among the first five proposals (out of 85), we find two other bracelets (n°23 and 38) of the same corpus (red arrows).

Advanced research

The previous search is said by default since it is based on the expertise and professional experience of the conservators. The user can, if desired, artificially modify the values of each subcharacteristic in order to weight its importance. These subcharacteristics are selected according to the specific expertise of the user or determined more precisely by studying the % match calculation. In the previous example, by increasing the values of the relative thickness of the strata and the structure of the cracks, subcharacteristics specific to the binocular observation mode, bracelets n°23 and 38 give better matching scores (Fig. 2.3.4). The user has the possibility to develop further this search, for example by comparing the strata with each other according to their location in relation to the residual metal (match by class function).

Once the best scores have been obtained, the user can click on the proposed stratigraphies in order to study them more closely. Other information (metal family, alloy, object designation) is also available on the corresponding object sheet, which can be accessed by clicking on the information in the table of Fig. 2.3.4.

Establishment of stratigraphies of corrosion structures in cross-section

Conservation scientists and corrosion experts are studying the forms of corrosion in cross-sections. The samples taken are representative of the observed alteration problem. The observation of a corrosion structure in cross-section gives access to information that is not available when observing the same structure under a binocular microscope (precise thickness of the strata, compactness of the strata and percentage (%) of porosity, profiles of the strata, presence or absence of a CM—corroded metal stratum, microstructure of the residual metal). The analytical examination also

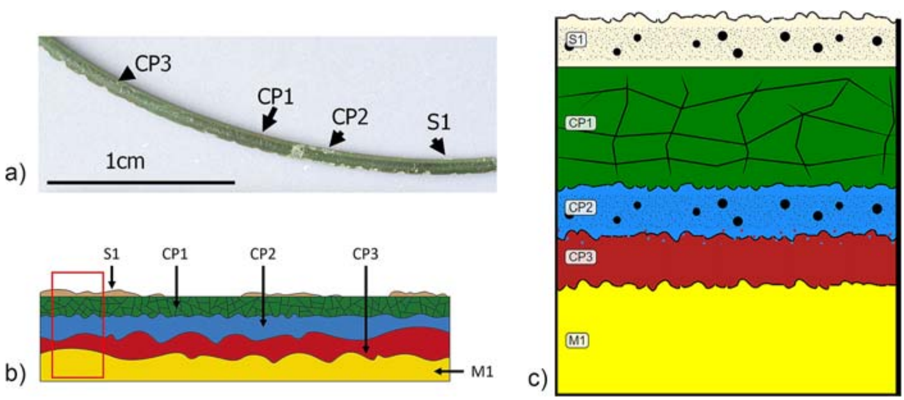


Figure 2.3.2 Bracelet (n°67) from the Bussy/Pré-de-Fond site in Fribourg, Switzerland: Microscopic view with strata codes (a), schematic representation of the corrosion forms with indication of the most representative corrosion structure (red frame) (b) and construction of the digital stratigraphy deduced (c).

Figure 2.3.3 Result of the (by default) MiCorr database query for the binocular stratigraphy in Fig. 2.3.2c. On the left, the subcharacteristics values filled in (in blue, detail). On the right, the matching proposals of the database: the stratigraphy under binocular mode, the matching %, and the information on the corresponding metal, alloy and object (detail).

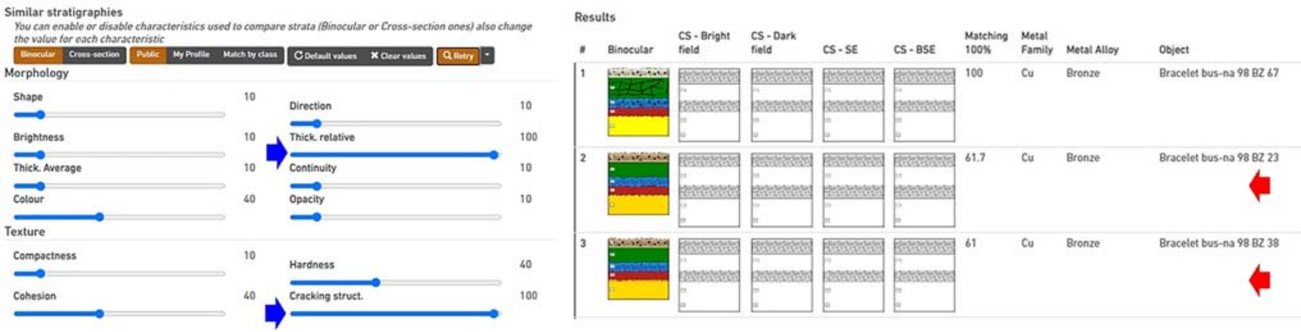


Figure 2.3.4 Result of the (advanced research) MiCorr database query for the binocular stratigraphy in Fig. 2.3.2c. The highlighted subcharacteristics are indicated with a blue arrow.

specifies the composition of the main constituents of the strata. A more accurate diagnosis can therefore be expected.

The MiCorr digital stratigraphies in cross-section reflect the corrosion structures observed under a metallographic microscope in bright—BF and dark field—DF (the latter approaching the observation under binocular microscope) and a scanning electron microscope (secondary -SE and backscattered electrons—BE). The information listed above can be introduced as subcharacteristics of each stratum. Fig. 2.3.5 illustrates this for bracelet n°67 from the Bussy/Pré-de-Fond site in Fribourg, which we were able to sample: the metal microstructure is detailed in Fig. 2.3.5a, while Fig. 2.3.5b and c describe the corrosion structure accompanied by elemental mapping. All these data are compiled in the MiCorr stratigraphy shown in Fig. 2.3.5d.

Once the fields for the four observation modes (BF, DF, SE, BE) have been filled in, the same algorithm as above is used to determine whether the MiCorr database presents similar stratigraphies (Fig. 2.3.6). It can be seen, using the by default mode, that for the same bracelet (Fig. 2.3.2a) the three other bracelets in the corpus (n°38, 23 and 11) come out on top and the matching % are significantly higher (except for bracelet n°11) than those of Fig. 2.3.3. This shows that when working on cross-section a better matching is expected due to the amount and precision of information provided.

Metadata

Proposals of stratigraphies and associated objects following the querying of the MiCorr database might be discarded by taking into account keywords from the metadata related to the object considered. The "By keywords" search engine allows these keywords to be inserted directly or via filters (country of origin, metal family, corrosion forms, object environment) selected to facilitate the querying.

Applications

A corpus of drafts and finished products with different corrosion forms

The bronze bracelet n°67 from the Bussy/Pré-de-Fond site in Fribourg, Switzerland, owned by the Service Archéologique de l'Etat de Fribourg and used to illustrate the previous sections is part of a series of bracelets dated to the first Iron Age (550−450 BCE, Halstatt Culture—Ha D2-3). Two are thought to be drafts (n°38 and 23, Fig. 2.3.7a), while two others are obviously at a more advanced stage of work (n°11 and 67, Fig. 2.3.7a). Fig. 2.3.2 details the corrosion forms observed on bracelet n°67. By comparing its digital stratigraphy under binocular microscope with those of the database, the MiCorr application proposes (Fig. 2.3.3) as close stratigraphies, by default, the stratigraphies of drafts n°38 and 23 (Fig. 2.3.7b), and as a eighth proposal, the stratigraphy of bracelet n°11 which is significantly different (in particular with regard to the thicknesses of CP1 and CP2, Fig. 2.3.7b). The digital

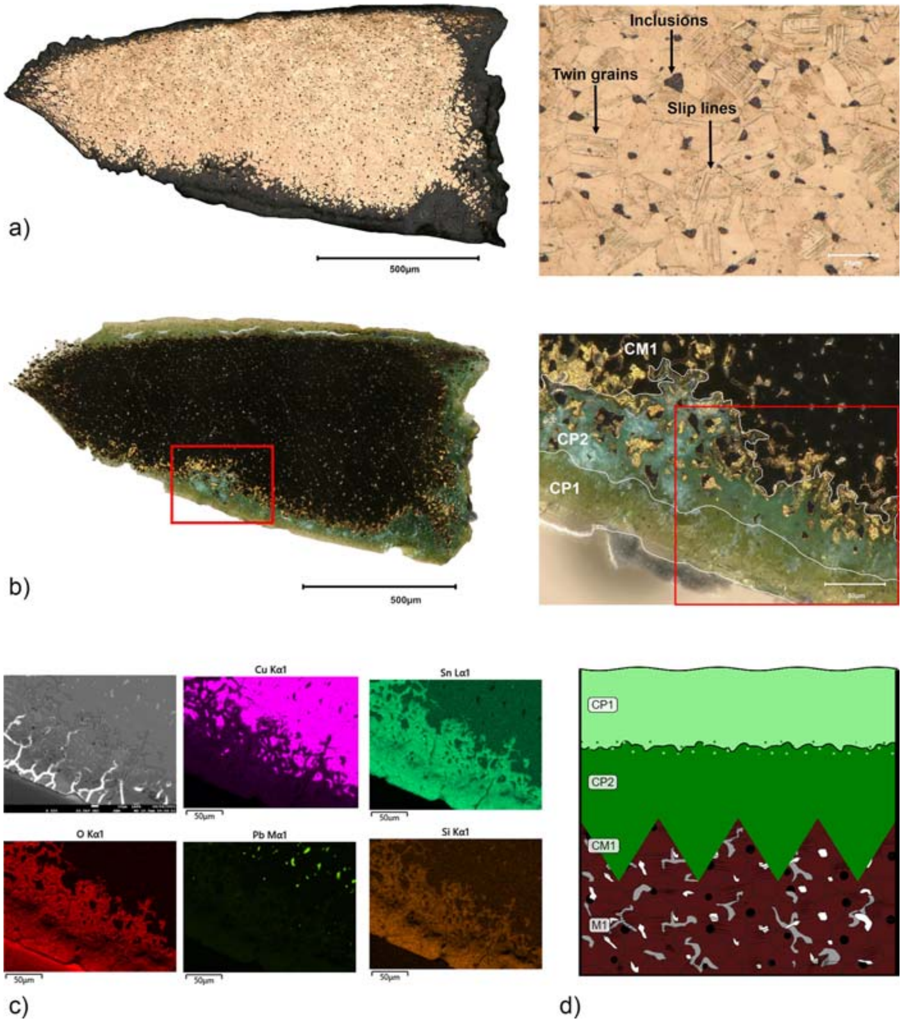


Figure 2.3.5 Cross-section of the sample taken from the bracelet (n°67) from the Bussy/Pré-de-Fond site in Fribourg. Optical micrograph of the sample in bright field after chemical etching of the surface and detail of the observed metallic microstructure (a); optical micrograph of the sample in dark field and detail of the observed corrosion structure with reference to the identified strata (b); SEM picture and EDX elemental mapping of the corrosion structure, showing the decuprification of the corrosion structure with associated Sn enrichment and incorporation of external elements (Si, O) (c) and digital stratigraphy deduced (d).

stratigraphies of the corrosion structures observed in cross-section are more informative (Fig. 2.3.7c). The dendritic microstructure of bracelets n°38 and 23 indicates that they are drafts, while the grain microstructures with twinning and sliding lines of bracelets n°11 and 67 show that they have been worked. The corrosion structure of

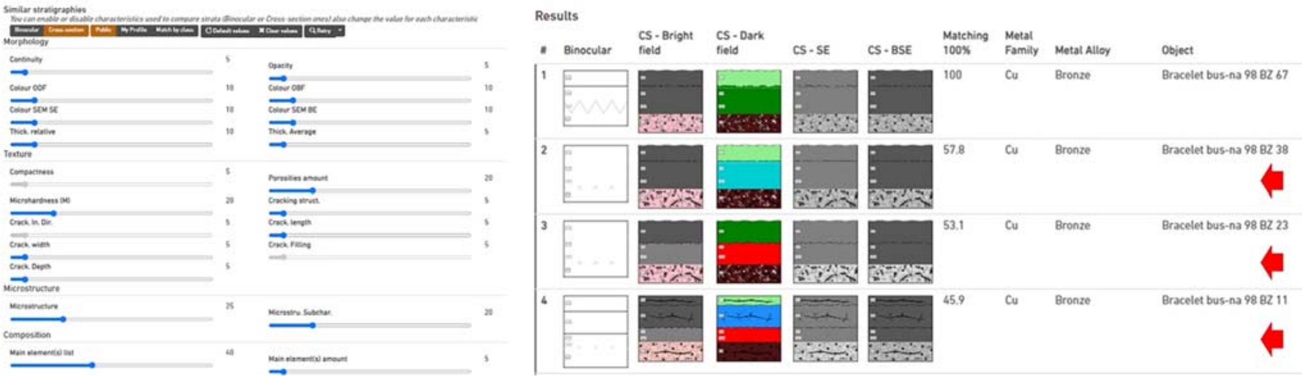


Figure 2.3.6 Result of the query (by default) of the MiCorr database for the stratigraphy in cross-section of the object in Fig. 2.3.2a.

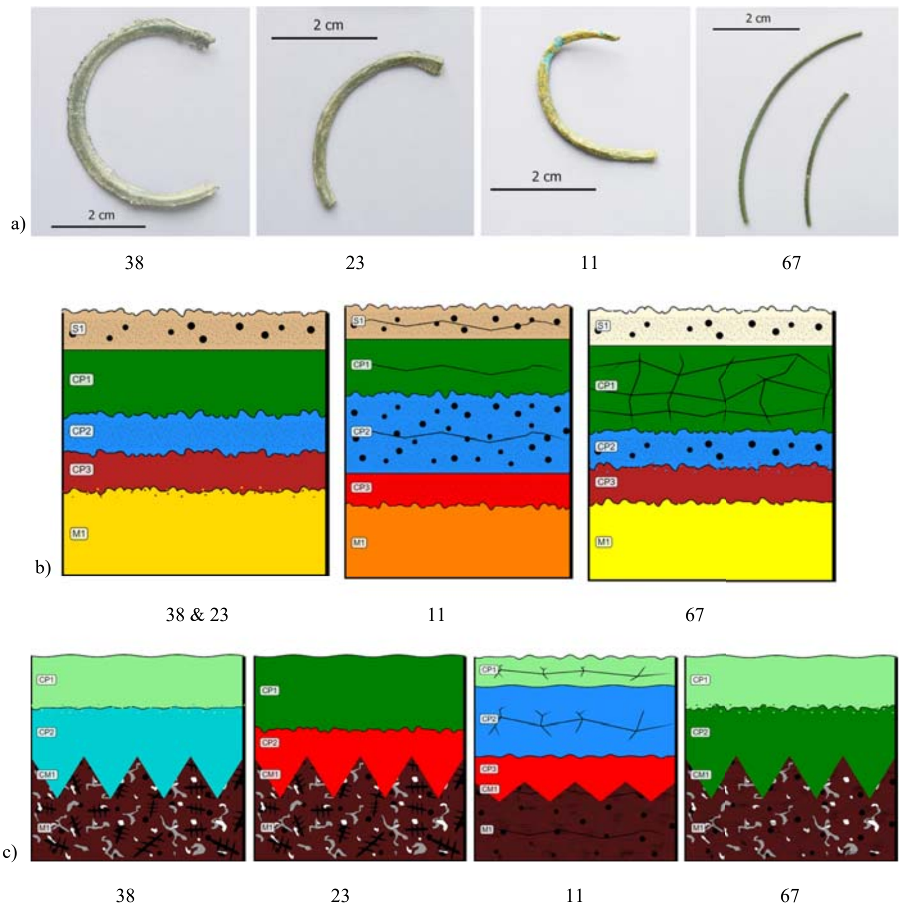


Figure 2.3.7 Bracelets n° 38, 23, 11, and 67 from the Bussy/Pré-de-Fond site in Fribourg, Switzerland: Macroscopic views (a), digital stratigraphies under binocular microscope (b) and in cross-sections (c).

bracelet n°67, however, is closer to that of bracelet n°38, while the corrosion structures of bracelets n°11 and 23 are quite similar.

Lake patina

The MiCorr database contains a number of entries for Late Bronze Age (−1400 to −800 BCE) bronze objects found at the archeological site of Hauterive-Champréveyres, on the shores of Neuchâtel lake, Switzerland. Archeologists and researchers have noticed that these objects had distinct appearances, some with a smooth, dense and yellowish-brown "lake patina" (Fig. 2.3.8a) and others with a thick, greenish-blue "terrestrial patina" (Fig. 2.3.9a). These forms of corrosion were recorded

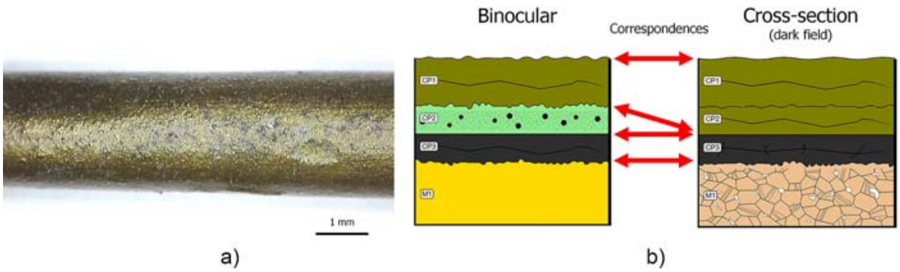


Figure 2.3.8 Surface appearance of a bronze pin (n° 18152, detail) from the archeological site of Hauterive-Champréveyres with "lake patina" (a) and associated digital stratigraphies under binocular and cross-section modes of observation (b).

in MiCorr via digital stratigraphies obtained by observing the objects under binocular microscopy and studying samples in cross-section under metallographic and scanning electron microscopy (Figs. 2.3.8b and 2.3.9b). Correspondences between the two MiCorr stratigraphies could be established.

The analyses carried out confirm those obtained by Schweizer [8] who showed that the lake patina is mainly composed of chalcopyrite ($CuFeS_2$, CP1 under binocular microscope and CP1/CP2 in cross-section, Fig. 2.3.8b). This layer is formed on top of a tin oxide layer (CP3). CP2 under binocular microscope corresponds to secondary corrosion products, formed after exposure to the atmosphere. The stratigraphies under binocular microscope and in cross-section are however rather similar. The terrestrial patina is more complex, and the stratigraphies under binocular microscope (8 strata) and in cross-section (9 strata) are different. Under the sediment (S1), we find, in cross-section, a turquoise blue stratum (CP1) made up of copper sulfate and corresponding to the green-blue strata CP2-4 in binocular mode. Then, still in cross-section, a succession of black and gray layers (CP2-4), a priori copper sulfides with a little iron (perhaps the origin of chalcopyrite), is observed and corresponds to the CP5 under binocular microscope. Finally, a succession of green-brown layers (CP5-CP7 in cross-section) based on tin oxide appears near the residual metal and corresponds to the red CP6 under binocular mode. The formation of the turquoise blue

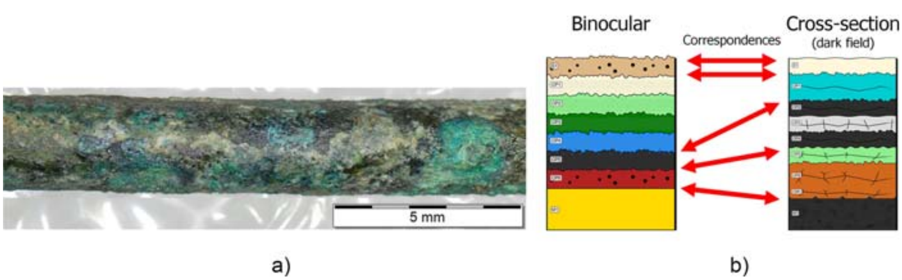


Figure 2.3.9 Surface appearance of a bronze pin (n° 3031, detail) from the archeological site of Hauterive-Champréveyres with "terrestrial patina" (a) and associated digital stratigraphies under binocular and cross-section modes of observation (b).

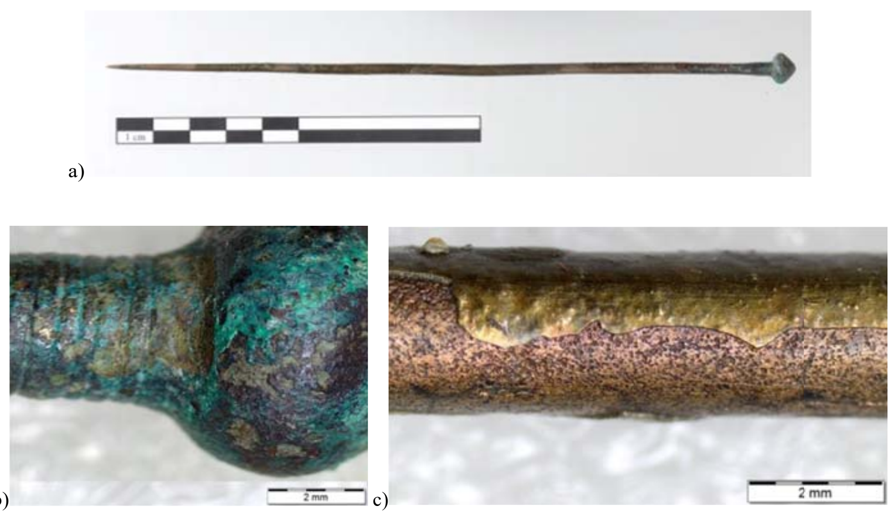
stratum is thought to be due to the lowering of the water level of the lake, favoring aerobic conditions and the transition from sulfides to sulfates and even carbonates [9].

Several other objects from the same site have since been examined, including pin n°3071 (Fig. 2.3.10a). As it was not possible to take samples from these, we were only able to observe them under binocular microscope in order to define the forms of corrosion that had developed (Fig. 2.3.10b and c). Their overall schematic representation shows the corrosion structures present, which were represented digitally via MiCorr (Fig. 2.3.11).

If the corrosion form of the body of pin n°3071 is undoubtedly assimilable to a lake patina as indicated by the first five propositions of the MiCorr ranking, among which pin n°18152 gives the best matching (fifth proposal, Fig. 2.3.12a) compared to the terrestrial patina of pin n°3031 (60th proposal), that of the head would be between a lake (10th proposal for pin n°18152) and terrestrial (25th proposal for pin n°3031) patina (Fig. 2.3.12b), even if optically one would lean toward a terrestrial patina (bluish-green aspect, aerobic environment similar to that of the discovery of pin 3031).

Discussion

As described above, each professional involved in the examination of heritage metals documents them according to their level of expertise. The MiCorr application promotes the sharing of knowledge between the communities concerned via the object sheets: collection managers provide the metadata and the identification of the metal family, conservators a preliminary diagnosis based on the corrosion structures observed under binocular microscope, and conservation scientists/corrosion experts/



a) b) c)

Figure 2.3.10 Pin (n°3071) from the archeological site of Hauterive-Champréveyres (a) and details of the head (b) and the body (c).

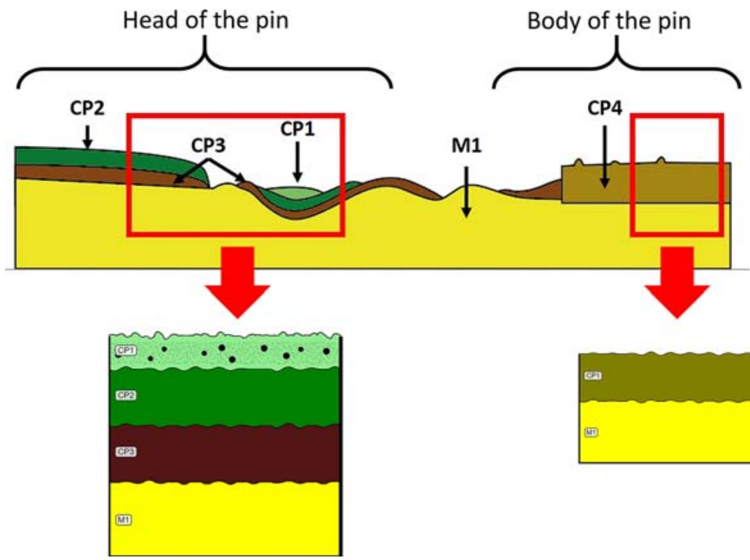


Figure 2.3.11 Schematic representation of the corrosion forms on pin n°3071 of the Hauterive-Champréveyres archeological site and digital stratigraphies deduced from the corrosion structures selected. Note the presence of two CP1 strata imposed by the construction of the digital stratigraphies.

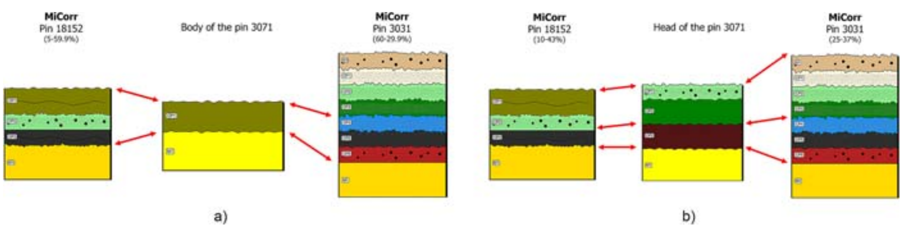


Figure 2.3.12 Comparison of the stratigraphies under binocular microscope of the body (a) and head (b) of pin n°3071 with those of pins n°18152 (lake patina) and n°3031 (terrestrial patina) in the MiCorr database. Rankings and % matches are shown in brackets.

archeometallurgists a complementary diagnosis based on the same corrosion structures observed in cross-section.

MiCorr should also have an impact on everyone's work. For example, thanks to the "By visual inspection" search engine, collection managers should be able to specify the nature of the metal objects in their care, thus facilitating their daily activities of inventory, condition assessment, and preventive conservation strategy.

Conservators, in charge of interventions on these same objects, should be able to establish more relevant diagnoses on them. It should be remembered that in the vast majority of cases, their approach to the objects is noninvasive and their diagnosis is based on the observation of corrosion forms developed under binocular microscope. The comparison of the digital stratigraphies obtained with those of the objects in the

MiCorr database should enable them to identify objects and/or series of objects with similar corrosion structures, as seen with the bracelets from the Bussy/Pré-de-Fond site in Fribourg, Switzerland (Fig. 2.3.4). If they limit themselves to these observations, the diagnosis remains partial. To further support it, digital stratigraphies in cross-section are more relevant (Fig. 2.3.6). Hence the importance of establishing correspondences between the observation of corrosion structures under binocular microscope and in cross-section, which, as we have seen, can be very different (Figs. 2.3.7b and c or 2.3.9b). These correspondences allow a shortcut to be made between the digital stratigraphy obtained following the observation under binocular microscope of an object under study and the digital stratigraphy in cross-section of an object in the database which, via the comparison algorithm, gave the best match.

This approach is illustrated in Fig. 2.3.13 for the two zones of interest of pin n°3071 of the Hauterive-Champréveyres archeological site. At the end of the questioning, the user has, beyond the type of corrosion (lake and terrestrial patina), precious information deduced from cross-section examination on similar objects such as the distribution of the strata, their thickness, the presence or absence of cracks, their composition and the microstructure of the residual metal, allowing them to refine their diagnosis without sampling. This information, although not quantitative, will help to understand how these objects can damage further or be cleaned/stabilized.

Conservation scientists, corrosion experts and archeometallurgists should benefit, via MiCorr, from information on metal objects similar to those they observe, their microstructure and fabrication techniques, as well as a global description of the corrosion forms that they can generate. Working often on samples, mostly representative of

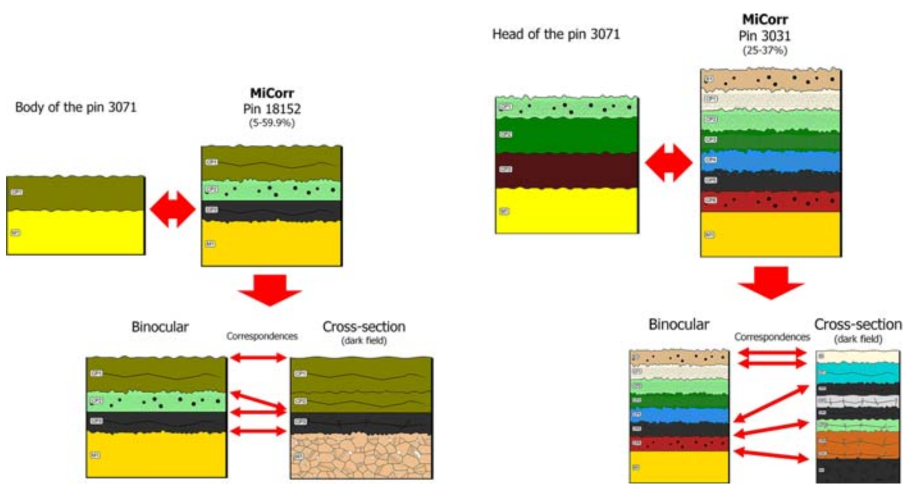


Figure 2.3.13 Facilitated diagnosis of an object studied (pin n°3071 from the Hauterive-Champréveyres archeological site) by comparing its digital stratigraphies under binocular microscope with those closest to the MiCorr database and on the basis of correspondences between the digital stratigraphies in binocular and in cross-section modes of these same objects.

the alterations observed on the objects, this information would allow them to extend their conclusions on the corrosion mechanisms in place to the whole associated object.

Conclusion

The diagnosis of heritage metals should be a shared task between the professional communities involved, taking advantage of the expertise of each. The MiCorr application offers this possibility of a transdisciplinary approach where everyone contributes according to their expertise but for the benefit of the others. The collected data constitute the application's database.

MiCorr should also be seen as a support for scientific research. Thus, the application makes it possible to identify types of corrosion forms for each metal in a specific environment. In addition, researchers can use MiCorr as a platform for sharing "additional data" that can be cited in publications.

The MetalPAT project has shown that beyond the construction of the application itself, the contributions of conservation professionals are essential to its development and its relevance. This requires time and commitment. It is therefore hoped that the whole profession involved in the documentation of heritage metals will adopt MiCorr as soon as possible so that the application continues to serve the needs and interests of the professionals involved.

Acknowledgments

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Bridging the Gap: Corrosion Science for Heritage Contexts explores the decision-making processes for preserving heritage metals and examines the collaborative, interdisciplinary relationships that underpin them.

Through themed chapters, this book is designed to develop and strengthen research collaborations, creating a synergy that benefits science and practice for the preservation of heritage metals. It builds an overview of the challenges faced in metal conservation across a broad range of heritage contexts, from indoor museum displays to fixed outdoor structures and moving objects. Researchers and practitioners provide critical insights into corrosion problems within heritage, current corrosion mitigation procedures, and the evidence supporting best practice guidance.

This book will be a valuable reference resource for corrosion and corrosion protection scientists, heritage preservation scientists, and conservation practitioners and students studying the preservation of cultural objects.

Key Features

- Provides a detailed understanding of the recent advancements in the field and the benefits of a multidisciplinary approach to addressing future challenges
- Provides a contextual understanding of the corrosion of selected heritage metals in different environments
- Discussion of novel characterization techniques as applied to heritage science
- Extensive case studies from experts who deal with the conservation of metal artifacts

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