Microsc. Microanal. 19, 1248–1254, 2013
doi:10.1017/S1431927613001608

Microscopy AND Microanalysis

Multifocus Optical Microscopy Applied to the Study of Archaeological Metals

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Abstract: Studies on cultural metal artifacts can benefit greatly from microscopy techniques. The examination of microstructural features can provide relevant information about ancient manufacturing techniques, as well as about corrosion/degradation processes. In the present work, advantages of the use of multifocus imaging techniques in optical microscopy for the study of archaeological metals are presented. An archaeometallurgical study of a large collection of bronzes demonstrates the possibility of a microstructural study with no need for sample removal, which is a great advantage in the study of cultural objects. In addition, the study of mounted samples illustrates the advantages of the multifocus technique in the examination of particular corrosion features, with the possibility of three-dimensional reconstructions.

Key words: multifocus imaging, optical microscopy, microstructure, 3D reconstructions, cultural heritage, archaeometallurgy

Introduction

Optical microscopy has been used in the study of archaeological metallic artifacts (e.g., Davy, 1826). Despite the present availability of more sophisticated microscopy techniques, such as scanning electron microscopy (SEM), which can also be used for the study of archaeological metallic artifacts (e.g., Ingo et al., 2004; van Hoek et al., 2011), optical microscopy is still a very relevant technique as it delivers important information regarding the microstructure of ancient materials, manufacturing methods, and degradation processes in a relatively simple, inexpensive, and fast manner (e.g., Panseri & Leoni, 1957; Kienlin et al., 2006).

However, optical microscopy studies on metallic artifacts traditionally involve the removal of a sample (MIT, 2003), which can be a drawback in studies on cultural materials. Because of its uniqueness and historical/artistic value, or even because of the small size of some artifacts, severe restrictions on sample removal are frequently imposed by heritage holders (e.g., museums). As a result, studies involving optical microscopy techniques can be easily dismissed despite the very relevant information they provide.

In the last few decades, in part because of the fast improvements in hardware and software, a variety of imaging and microscopy solutions have become available. Among these, the multifocus (stacking) imaging technique, applied to commercially available optical microscopes, is capable of producing images at high magnifications with a considerably extended depth of field. This technique relies on the acquisition of multiple images along the optical axis (z-axis) until the required depth of field is covered (successive images must have a close focus so that focused portions of an image overlap focused portions of the next image), with the subsequent combination of in-focus portions of each image to produce a single final image with greatly increased depth of field. By this image-processing method it is possible to overcome the limited depth of field of objectives. Also, the multifocus technique can allow three-dimensional (3D) reconstructions if the positions along the z-axis are recorded. This feature can be used for the reconstruction of textured models of the objects or surfaces observed. Some detailed explanations of this technique and comparisons with other microscopy examination techniques such as confocal microscopy and SEM have been previously published and can be found in Niederöst et al. (2003), Zamofing and Hügli (2004), Cisneros (2010), and Thiéry and Green (2012), and in the references therein. Generally, when compared with these other techniques, multifocus optical microscopy is a simple, efficient, relatively inexpensive, and rapid approach. In addition, it can provide significant complementary information, making it a very valuable technique.

The multifocus technique, when associated with microscopy techniques, has been used for interesting applications in diverse research areas. Some recent applications in the geomaterials area include 3D reconstructions of inclusions in thin petrographic sections (Thiéry & Green, 2012) and imaging of internal and external features in single translucent/transparent crystal grains (Thiéry, 2013). Among paleontological studies it has been used for 3D reconstructions of fossil inclusions (Haug et al., 2009). In the materials science areas it has found distinct applications in areas such as 3D map surface measurements in wood and metal samples (Zamofing & Hügli, 2004).

Despite its potential, e.g., for 3D reconstructions, to date, its applications in the archaeological or cultural mate-

Received January 20, 2013; accepted April 19, 2013
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rials fields are scarce (Pavlidis et al., 2007). Among these fields, examples of application include enhancing depth of field in images of bone tools (Backwell & d'Errico, 2008) and shell beads (d'Errico et al., 2008).

Given its characteristics, multifocus optical microscopy can be of considerable value in the study of archaeological metals, to overcome sample removal drawbacks and offer new imaging possibilities. When sample removal is not allowed, a small area in the surface of an artifact can be manually prepared for microstructural observations. As the resulting area is usually nonplanar, the use of multifocus techniques can be of considerable interest for the production of sharp and clear images of the artifact microstructure. Also, because of the possibility of generating 3D reconstructions from the multifocus image sequences, the examined surfaces that may exhibit relevant topographic information can be illustrated with added 3D value.

In the present work, multifocus optical microscopy has been applied for the study of archaeological metals, showing the advantages of this technique in the examination of nonsampled artifacts and for the study of particular topographic features in conventional sampled sections of metals. In particular, a study of a large collection of archaeological bronzes with different typologies, sizes, and microstructures will show the adequacy of this technique in the evaluation of ancient manufacturing techniques, without sample removal. In addition, the results of the examination of some conventional mounted samples of copper and bronze artifacts are presented to show the adequacy of this technique for detailed visualization of particular corrosion features with topographical information.

Materials and Methods

Archaeological Metals

Copper-based artifacts, such as bronzes, regularly present thick superficial corrosion layers that have developed over long periods of time. When sampling is performed, the sample generally includes the superficial corrosion layers and the unaltered metal that lies below, allowing the examination of the corrosion profile and possible gradients in grain size and shape along the cross-section. Sample preparation normally involves mounting in resin, grinding, and polishing in order to obtain a planar surface for examination under a conventional optical microscope wherein 2D photographic records can be obtained (MIT, 2003).

For the present study of artifacts without sample removal, a specific procedure involving a small surface preparation in selected areas of the artifacts was developed (Fig. 1). The surface preparation included removal of the superficial corrosion layers in a small area (<25 mm^2) of the artifacts with a grinding tool accessory attached to a small hand-held electric drill, followed by a metallographic preparation of the metal, which consisted of manual polishing with decreasing size of diamond suspensions in a cotton swab (until a 1-μm diamond size). The resulting surface was nonplanar, frequently with a slightly concave shape. The procedure

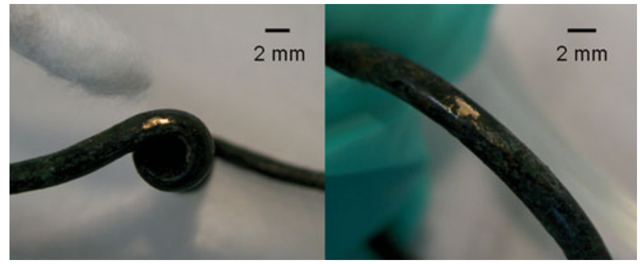

Figure 1. Photographs showing small surface preparation artifacts for microstructural examination with multifocus optical microscopy. The artifact on the left is a fibula and the artifact on the right is a ring, both attributed to the late Bronze Age period. The prepared area in both artifacts is $\sim 2 \times 1$ mm^2 in size.

allowed the observation of the microstructure at selected near-superficial areas of the artifacts, involving a minimally invasive procedure that left no structural damage to the artifact.

After the study, the small prepared areas could be sealed by a local conservation treatment.

Multifocus Optical Microscopy

The microstructural observations were performed using a motorized inverted metallurgical optical microscope Leica DMI5000M (Leica Microsystems, Wetzlar, Germany), equipped with a Leica DFC290 digital camera (Leica Microsystems). Because of the inverted configuration, the microscope allowed the positioning of relatively large-sized objects (Fig. 2).

The microscope and camera were connected to a desktop computer installed with the Leica Application Suite (LAS V2.6) (Leica Microsystems) and multifocus module software (http://www.leica-microsystems.com). This allowed the automatic acquisition of digital images at different focus positions (Z-stacks), followed by an automatic combination of the areas in focus of the various images, in order to produce a single, sharp, final composite image with significantly extended depth of field.

Five objective lenses were used, of magnifications 5×, 10×, 20×, 50×, and 100×, all semi-apochromatic (Plan-Fluotar), adequate for brightfield (BF)/incident light darkfield, and working with air as the medium. In Table 1 the numerical aperture values as well as the distance between each z-step performed in the automatic multifocus mode (default values) for each objective lens are presented.

Observations were made with BF illumination and under polarized light (POL), with the surfaces in unetched and etched conditions. The etching of the surfaces was made using an aqueous ferric chloride solution (6% (w/v) ferric chloride in a 6% (v/v) hydrochloric acid solution) impregnated in a cotton swab. The combination of these modes of observation was relevant for the study and for identification of different metallic phases present in the alloy, as well as for the study of inclusions and for corrosion evaluation. In particular, the observations under POL were important for the differentiation of a variety of corrosion

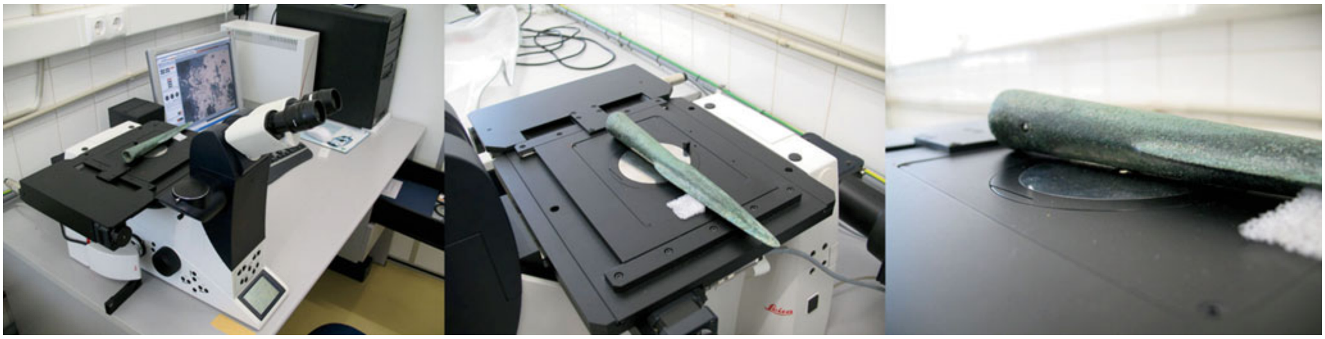

Figure 2. Examination of a prepared surface in a bronze spearhead by inverted reflected microscopy assisted by multifocus imaging. The prepared surface that is being examined is located in the socket of the spearhead. In the right image the light that emerges from the objective lens can be observed; the prepared surface area of the socket to be examined is positioned over the lens.

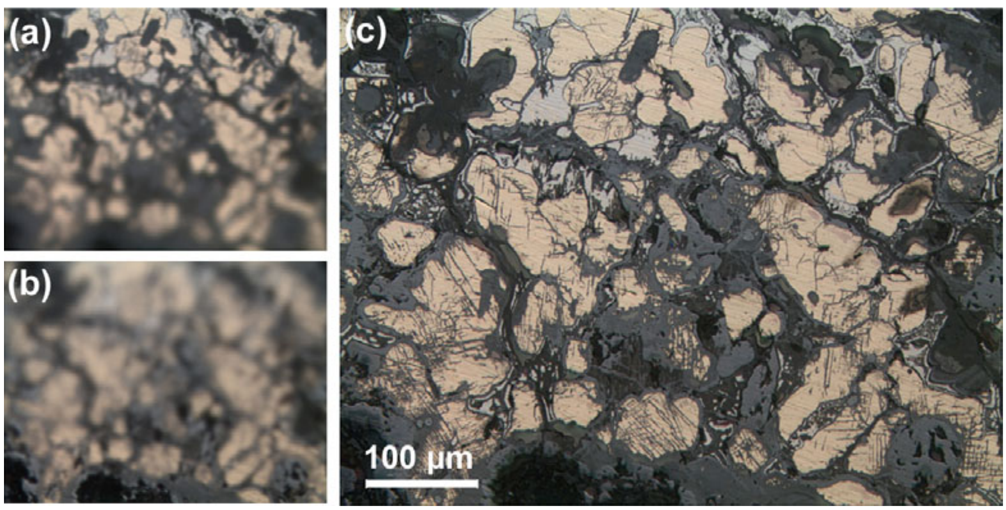

Figure 3. Examination of a nonplanar surface of an archaeological metal with inter- and transgranular corrosion, involving the acquisition of various images at different Z-stack positions (brightfield, unetched, 20× objective). **a,b**: Extreme positions of focus. **c**: Resulting multifocus image made with a combination of 44 stack images (distance between each z-step is 1.87 μm).

products that present particular colors under this light, and observations under etched conditions were important for the study of the manufacturing techniques, because of the enhancement of microstructural features such as grain boundaries, annealing twins, and slip bands in the grains.

Results and Discussion

Examination of Nonplanar Surfaces

The examination by multifocus optical microscopy of nonplanar surfaces, such as those resulting from manual surface preparations in the artifacts, involved the acquisition of various images at different Z-stack positions. In Figure 3 an example is shown illustrating the examination of a prepared corroded surface area of a bronze artifact. In Figures 3a and 3b, two extreme planes of focus are shown, which illustrate the constraints related to limited depth of field in a conventional optical microscopy examination, without applying the multifocus imaging technique. In contrast, Figure 3c, which is the multifocus image resulting from a stack of 44 successive steps, is totally in-focus, illustrating the advantages offered by this technique. In the multifocus image created, the coarse alpha-phase Cu-rich grains can be clearly identified, with intergranular corrosion along grain boundaries, as well as transgranular corrosion, which developed inside each grain in the form of parallel and thin gray lines.

The time needed for the acquisition and combination of the 44 images by the LAS software was <1 min, which proves to be perfectly adequate for microstructural studies

Table 1. Characteristics Related to the Set of Microscope Objectives Used in the Present Work.

Objective	5×	10×	20×	50×	100×
NA	0.15	0.30	0.50	0.80	0.90
Distance between each z-step (μm)[a]	23.73	5.72	1.87	0.51	0.29

[a]Automatic optimized step size. NA, numerical aperture.

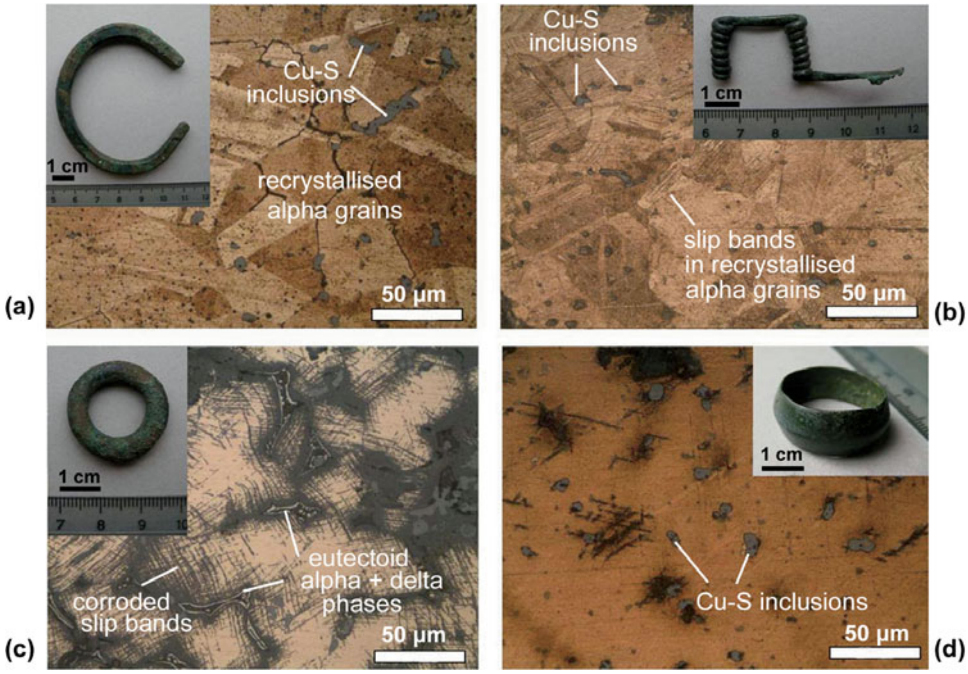

Figure 4. Resulting multifocus images of nonplanar small prepared surfaces in various artifacts showing different microstructural features. **a**: Bracelet with recrystallized (twinned) alpha grains [brightfield (BF), etched, 50× objective]. **b**: Fibula with recrystallized (twinned) alpha grains with some slip bands (BF, etched, 50× objective). **c**: Ring with alpha dendrites and slip bands enhanced by corrosion, with the presence of (eutectoid) delta phase in interdendritic regions (BF, unetched, 50× objective). **d**: Finger ring with recrystallized large-size alpha grains and some slip bands (BF, etched, 50× objective). Cu–S inclusions are frequently present and visible in the microstructures, as annotated.

of one or more artifacts involving several multifocus image acquisitions.

Study of a Large Collection of Bronzes Without Sample Removal

The multifocus imaging technique was used for the study of the microstructure of a collection of protohistoric bronzes that had previously been analyzed by energy-dispersive micro-X-ray fluorescence spectrometry and SEM with energy-dispersive X-ray spectrometry (SEM-EDS) in the small prepared surfaces for the determination of alloy composition and inclusions (Figueiredo et al., 2011, 2013). The collection is composed of 30 rings of various sizes and shapes, as well as six bracelets and one fibula.

The microstructure of each artifact can be directly related to the thermomechanical processes employed in its manufacture. Different manufacturing techniques can be found among artifacts of different typologies and cultural contexts, even when exhibiting similar alloy compositions (as bronzes with ~13 wt% Sn; Figueiredo et al., 2013).

The multifocus technique allowed a very clear picture of the microstructures of the various artifacts. These could vary from cored alpha-Cu dendrites (as-cast) to recrystallized alpha grains (indicating cycles of thermomechanical processing), with or without slip bands (indicating final plastic deformation). In Figure 4 some multifocus images representing different microstructural features among different artifacts are shown.

The presence of the eutectoid alpha + delta phases was observed in some artifacts (e.g., Fig. 4c), and it was also possible to observe some dark gray inclusions (BF observations) among the primary alpha grains (clearly visible in Figs. 4a, 4b, 4d), which had previously been identified in SEM-EDS analysis as Cu–S inclusions. Other inclusions, but

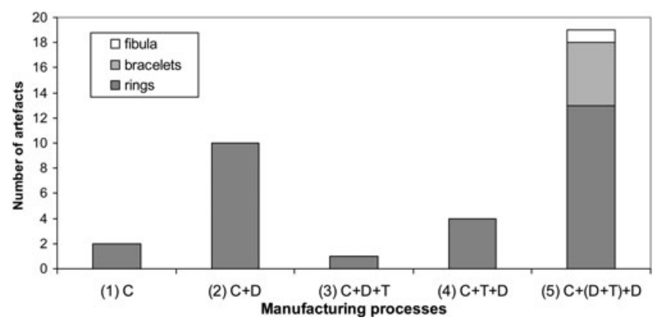

Figure 5. Different manufacturing processes that have been recognized under the multifocus optical microscopy study of a large collection of bronzes of different typologies. C, cast; D, plastic deformation; T, thermal treatment (annealing).

of much smaller size, such as Pb rich or Sn–O, which were also identified in the previous SEM-EDS analysis, and within the Cu–S inclusions, were more difficult to observe because of their diminutive size (<5 μm; Figueiredo et al., 2011). Nevertheless, careful examination of the larger Cu–S inclusions at high magnifications revealed some occasional dark spots inside them (in both BF and POL observations), some with regular/globular shapes and others with more irregular/sharp-edged shapes, which might refer to Pb-rich and Sn–O inclusions, respectively (some can be visualized in Fig. 4d, despite their relatively small size in the pictures).

In general, the examination by multifocus optical microscopy allowed a clear identification of the various techniques used in the manufacture of the artifacts (Fig. 5), with no need for sampling. Results showed that most artifacts suffered multiple cycles of thermomechanical processing, which ended with a final plastic deformation (manufacturing process no. 5 in Fig. 5). This final deformation could, in some cases, be due to a final surface finishing, such as polishing

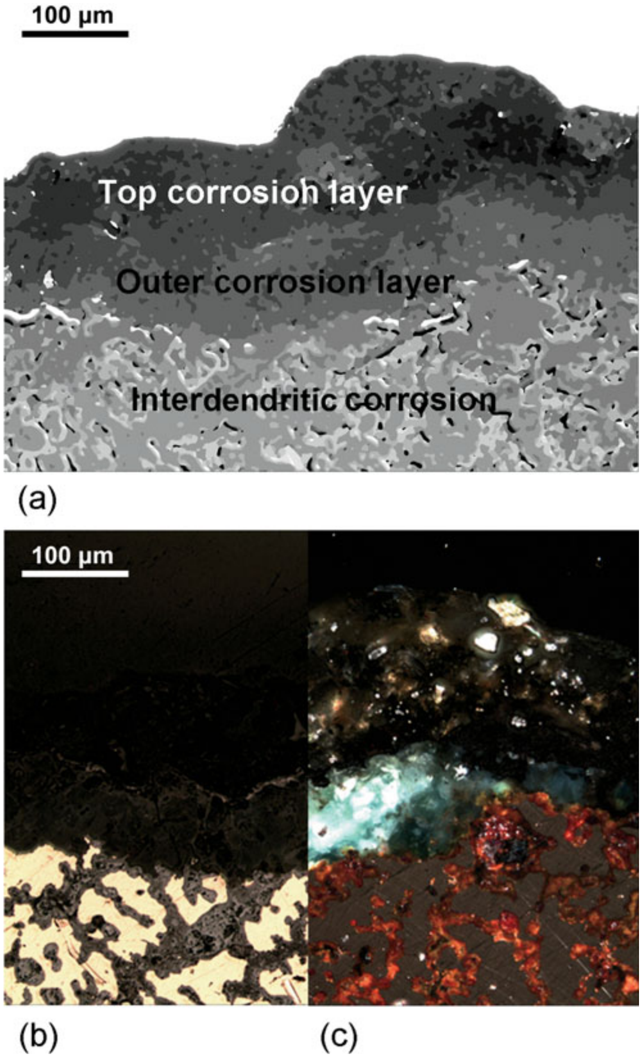

Figure 6. Mounted and polished cross-section sample of a leaded bronze artifact with well-developed corrosion. **a**: Multifocus "depth map" image (generated in the LAS software) showing the top, outer, intergranular corrosion layers and metal at different focal planes (given by different gradients of gray). **b**: Multifocus left-sided image of the examined area under brightfield illumination (sample unetched) and (**c**) multifocus right-sided image of the examined area under polarized light illumination mode. Multifocus images resulting from a stack of 11 pictures (20× objective).

(Wang & Ottaway, 2004). The presence of different finishing degrees among similar types of objects, such as rings, can be related to different uses that these artifacts had; e.g., those most carefully finished could be worn in the more exposed areas, or even with the production of artifacts in different workshops/cultural contexts.

Study of Particular Corrosion Features and 3D Reconstructions

Some samples representative of artifact cross-sections, mounted in resin and prepared by traditional metallographic methods, were also studied under the multifocus optical microscope to evaluate this examination technique for the study of some corrosion particularities.

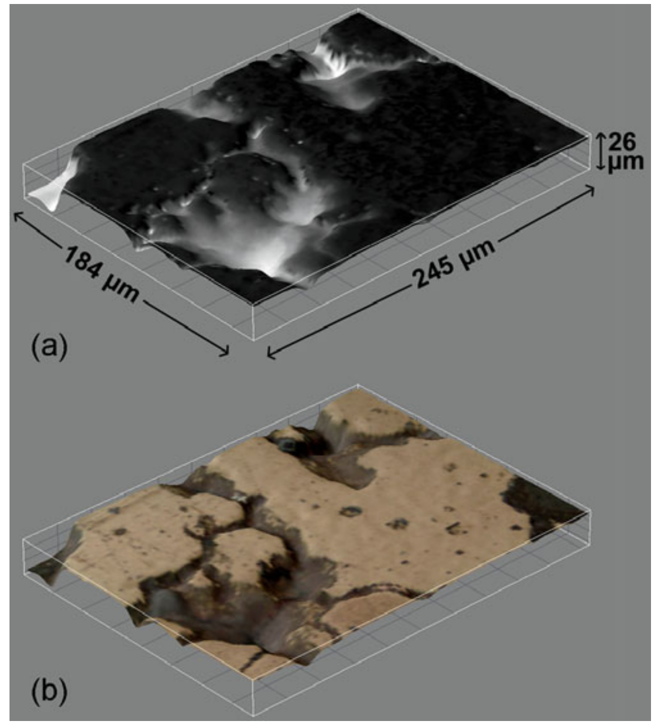

Figure 7. Detail of a microstructure of a mounted and polished sample of a bronze artifact with intergranular corrosion. **a**: Three-dimensional (3D) surface plot (generated using ImageJ) created from the multifocus "depth map" image (generated in the LAS software). **b**: Same 3D surface plot as previous but with the multifocus brightfield image loaded as "texture." Multifocus image resulting from a stack of 50 pictures (50× objective, field of view 245 μm, and maximum depth of 25.5 μm).

Ancient artifacts suffer from long-term corrosion processes, which can originate particular and defined structures, such as thick superficial corrosion layers and deep intergranular corrosion (Robbiola et al., 1998; Figueiredo et al., 2010). The study of corrosion by optical microscopy is of interest in cultural material studies as distinctions can be made between different corrosion products on the basis of their color or crystal morphology. Specific corrosion phenomena can be related to environmental burial conditions (Robbiola & Portier, 2006), or even to the current state of preservation of an artifact or collection, and can thus determine specific conservation and restoration procedures (Scott, 1990).

During grinding and polishing procedures of conventional mounted samples, materials with different degrees of hardness, such as metal and different corrosion layers, can suffer distinct abrasion. This can result in slightly different focal planes for different materials when observed under an optical microscope. In multifocus optical microscopy and with adequate software a "depth map" (a 2D grayscale picture) can be created that distinguishes the regions with different focal planes. In Figure 6a such a map generated by the LAS software program is illustrated, which shows how this feature can assist in the interpretation of some archaeological samples in which a clear mapping of different

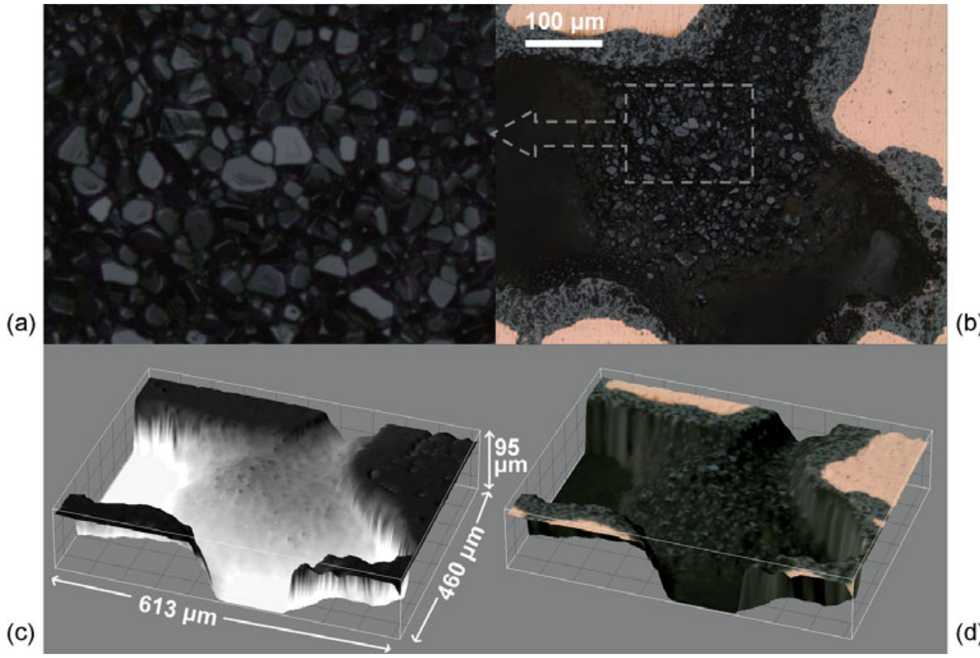

Figure 8. Detail of a microstructure of a mounted and polished sample of a copper artifact with intergranular corrosion. **a**: Detailed multifocus image of a concentration of small-sized crystals growing in intergranular spaces. **b**: Multifocus image in which the crystals were observed. **c**: Three-dimensional (3D) surface plot (generated using ImageJ) created from the multifocus "depth map" image (generated in the LAS software). **d**: Same 3D surface plot as previous but with the multifocus brightfield image loaded as "texture." Multifocus image resulting from a stack of 58 pictures (20× objective, field of view 613 μm, and maximum depth of 95 μm).

corrosion layers is important and other modes of observation (Figs. 6b, 6c) may not be sufficient to provide the relevant information on the distribution of metals and the different corrosion types.

The "depth map" can also be used to generate 3D surface reconstructions with adequate software. For the present study, the freely available program ImageJ (Rasband, 1997–2012) was used to produce 3D representations of specific morphological details of corrosion in an easy and fast manner. For that, the "depth map" image created by the LAS software program was used to produce the topographic information in the "interactive 3D surface plot" function from the "3D Viewer" plugin (Schmid, 2007). Previously, if needed, a noise reduction filter could be applied to suppress isolated pixels. Thereafter, the multifocus image could be loaded as "texture."

In Figure 7, a polished area of a sampled bronze artifact with intergranular corrosion is shown. Figure 7a shows the generated 3D surface plot with the "depth map" loaded as texture, and it can be observed that the lighter areas correspond to the far focus planes and the dark areas to the near focus planes. In Figure 7b, the same surface plot has the multifocus BF image loaded as "texture," and here it is clearly visible that the far focus areas correspond to intergranular corrosion. This image clearly illustrates the loss of material related to this type of corrosion, which is very common among archaeological artifacts.

In Figure 8, a polished area of a sampled copper artifact with intergranular corrosion is shown. Here, the ability of the multifocus technique to produce images of complex 3D objects is shown, with the clear representation of a concentration of small crystals (each one <20 μm) that have grown among the grain boundaries of the metal as a result of severe intergranular corrosion phenomena.

Conclusions

In the present study advantages of the multifocus imaging technique applied to optical microscopy for the study of cultural metals are presented. This technique allows the study of a complete archaeological bronze collection without the need of sampling, providing information about the range of ancient manufacturing techniques employed in the fabrication of different types of artifacts, as well as about the presence and distribution of specific inclusions in the metallic matrix. It also has tremendous advantages in the imaging of conventional mounted samples, such as for detailed observation of corrosion features with topographical information, showing that in some cases multifocus optical microscopy may even provide more information than obtainable from a standard optical microscopy examination. Further advantages may also be found in the application of this technique to the observation of translucent materials, such as some corrosion or metallurgical slag products, where 3D structures can develop.

Finally, it can be concluded that the method is relatively simple to implement and provides relevant information for both archaeometallurgical and corrosion studies in a relatively fast manner. It is suitable for the study of large collections of artifacts, artifacts of complex shapes, artifacts with aesthetical significance, which are not allowed to be sampled, as well as for conventional mounted samples, thus being of considerable value in cultural heritage studies.

Acknowledgments

This research work was financed by the Portuguese Science Foundation (FCT-MCTES) through the Early Metal project (PTDC/HIS-ARQ/110442/2008) and the grant SFRH/BPD/73245/2010 to EF. The financial support of CENIMAT/I3N through the Strategic Project-LA25-2011-2012 (PEst-C/CTM/LA0025/2011) is also acknowledged.

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