

Preface

The research has been focused on the optimization of imaging techniques for the study and characterization of metal artefacts. The potential of two imaging techniques, which can be almost considered complementary in terms of achievable results, was explored: Absorption Contrast Neutron Imaging, a complex technique that provides information regarding the bulk composition of the object under examination, and Reflectance Transformation Imaging (RTI), which is instead a rather simple technique that provides information of the artefact surface.

Most of the work was dedicated to the calibration of a Neutron Imaging station dedicated to the study of Cultural Heritage at the LENA Applied Nuclear Energy Laboratory in Pavia (Italy). The main aim was to provide a set of reference alloys with known chemical composition and microstructure similar to ancient metal artefacts to be able to correlate the information obtained from the image processing, in particular the mass attenuation coefficient, with the composition of the alloy. In fact, having an instrument that allows alloy discrimination would be an important upgrade in the characterization of metals by using neutrons. As will be fully explained in the next chapters, the use of neutrons presents several advantages. In particular, neutrons can penetrate heavy metals (i.e. lead) and can discriminate elements not otherwise discriminable with other techniques. Neutron Imaging is a non-invasive and non-destructive technique which represents a valuable complement and alternative to conventional X-Ray imaging.

The obtained results of the calibration turned out to be quite promising, especially in the discrimination of alloys made up of percentages of different elements (i.e. brass, tin bronze, leaded tin bronze). Additionally, an attempt was made to study the corrosion patina formed on the metallic surface. For this purpose, some metallic references were selected to be artificially aged to obtain corrosion product layers on the surface. The discrimination of the composition of the corrosion products layers

is also a challenge in the safeguarding of metallic objects. In particular, in the case of Cu-based artefacts, corrosion patinas have to be preserved for historical and aesthetic reasons. Implementing the Neutron Imaging resolutions is therefore a fundamental issue.

In advance of the application of Neutron Imaging to historical metal artefacts, it will be necessary to take into consideration the strict radioprotection rules which provide mandatory quarantine (six months) for samples potentially activated by the neutron beam. Until now, this is one of the biggest problems evidenced in anticipation of using this technique with real metal artefacts. Despite this, the calibration was carried out to implement the Neutron Imaging facility.

Eventually, for the study of the morphology, an RTI system was developed in parallel to provide information about the surface. Since Neutron Imaging was unable to provide the necessary information regarding the superficial corrosion layer of the sample, the development of a system that allows the morphological and compositional analysis of the corrosion products became necessary. In this way, it was developed a portable and low-cost system for the characterization of small-sized metal artefacts, to simultaneously obtain morphological and compositional information. This was done by pairing two different types of lighting: visible and ultraviolet. In this way, the acquisition of RTI images in the visible and UV can provide a first characterization of a sample in a totally non-invasive way.

The VIS-UVL RTI system was tested for the analysis of some bronze coins, therefore carrying out an initial chemical characterization using Raman spectroscopy and then comparing the results with the appearance of the surface (more or less fluorescent areas). Even in this case, the results were promising, as will be better explained in the following chapters.

Finally, these two techniques, so different from each other in terms of instrumentation, cost and above all achievable results, appear to be almost complementary. They have proven to be very useful in the characterization of metal artefacts, providing a generally non-invasive and non-destructive tool for the study and conservation of metals. The role of the measurement expert is therefore fundamental in attempting to improve the current limitations so that they can effectively be two independent techniques that can be used in an independent way.

Some case studies were also followed to develop measurement protocols devoted to the study of iron metallic artefacts using Raman spectroscopy. In the first case

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study, a protocol was developed and tested on archaeological iron nails to study the corrosion product layers and provide information about the conservation state. The second case study involved the evaluation of long-term effectiveness of different protective treatments applied to archaeological iron nails.