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Over the rainbow? Micro-CT scanning to non-destructively study Roman and early medieval glass bead manufacture

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ABSTRACT

Desktop micro-CT scanner has become a standard piece of equipment for many materials science laboratories and is increasingly popular in the field of archaeology for the study of archaeological soils and small archaeological artefacts. The technique does have limitations which are reviewed. Then, the usefulness of desktop Micro-CT scanners for the study of archaeological artefacts is demonstrated in a non-destructive study of manufacturing methods of Roman and Early Medieval monochrome and polychrome glass beads. Differences in glass colours show up in these scans as differences in attenuation. The presence and distribution of bubbles and various inclusions (metal, opacifier) are also well visible. Shaft shapes and patterns of bubbles inside the glass make it possible in most cases to distinguish between drawn, wound and constructed (millefiori) beads and to study shaping methods. Shafts shapes also reveal shapes and dimensions of the mandrels used in bad manufacture. Decoration and construction methods are be visualized and studied in virtual cross-sections and semi-3D rendered images of selected glass colours. Visible degradation processes - including fissuring, leaching (especially of opaque yellow glasses) and dissolution processes - are highlighted. The micro-CT scans demonstrate how quality of raw glass, base design and workmanship ion bead manufacture have improved with time. Moreover, it shows how these scans may serve as basis to discuss the organization of workshops where simple or more complex objects were made from non-metallic raw materials, and the availability and distribution of different qualities of raw materials for these workshops.

1. Introduction

1.1. scanning in archaeology

Chemical, physical and microscopic analyses and recording are core and major parts of archaeological science studies. Since many techniques are destructive, choices have to be made between damaging scientific analyses on the one hand and preserving archaeological remains as source for future study and as cultural heritage on the other hand. Studying micro-spatial and micro-structural properties of artefacts often requires especially damaging approaches like making and analysing cross-cuts, petrographic slides, or thin sections. Even then, these techniques are limited by the fact that they provide a 2-D cross-section of what are essentially 3-D spatial properties.

X-ray Computed Tomography (CT) techniques in general have the dual advantage that they are non-destructive, and at the same time provide possibilities for recording, studying and analysing the 3-D spatial characteristics of the materials or objects studied. "Normal" or medical CT scans have been sporadically applied in archaeological contexts, mainly for determining the content of blocks of soil from burial sites (Jansen et al., 2006) and cremation urns (Anderson and Fell, 1995; Harvig et al., 2012) without the risk of damaging vulnerable artefacts during excavation or for inspecting ancient mummies, in order to derive biographical information, detect signs of pathology or allow facial reconstruction (Lynnerup, 2010). The same techniques have also been used to study material properties and manufacture of artefacts – in particular ceramics (Applbaum and Applbaum, 2005) – but for this, usually higher resolutions are required. This higher resolution can be achieved using X-ray micro-CT scans.

In the last decade, the use of X-ray micro-CT has made it possible to study the internal structure of archaeological objects non-destructively, and at high resolution (microns). This non-destructive technique

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(Cantatore and Müller, 2011 and Cnudde and Boone, 2013) can be used to inspect the internal composition and 3-D structure of objects of a wide range of materials, including glass. This makes this technique extremely useful for studying the manufacturing techniques and material properties for non-metallic objects. X-ray attenuation is function of object thickness, material density and mass attenuation coefficient. The mass attenuation coefficient depends on the X-ray energy and the cube of the atomic number of the chemical elements present in the material. Thick objects made of metals with high atomic number like lead obstruct X-ray.Micro-CT is increasingly popular in the field of archaeology for the study of archaeological soils (Ngan-Tillard and Huisman, 2017) and small archaeological artefacts (Bertrand et al., 2012; Tuniz and Zanini, 2014; Barron et al., 2017). With repeated (time lapse) 3D-X-ray imaging, microstructural transformations caused by environmental changes imposed to archaeological artefacts can also be tracked (McBride and Mercer, 2012, Ngan-Tillard et al., 2015, 2016). Both degradation and conservation processes can be investigated (Bertrand et al., 2012).

X-ray micro-CT can be conducted at one of the few synchrotron facilities available worldwide, but also using a desktop micro-CT scanner. The latter has become a standard piece of equipment for many materials science laboratories and is readily accessible to archaeologists. Compared to lab-based micro-CT, synchrotron based micro-CT is faster and provides results with similar or often higher degree of detail. It is performed using a high quality parallel monochromatic beam which eliminates the beam hardening (white corona effect) that is inherent to micro-CT scans obtained with polychromatic beams.

Lab-based micro-CT scanners deliver scans which have a sufficient resolution for the study of small objects: between 5 and 10 micrometres for a 10 mm diameter object, i.e. about one to two thousandth(s) of the width of the object. With the development of local tomography, a higher resolution can be reached for larger objects in a given region of interest (Maire and Withers, 2014).

Sufficient X-ray attenuation contrast between the materials present in an artefact is a sine qua none condition for the conventional micro-CT technique used in this article. With the less common phase contrast X-ray micro-CT (Bertrand et al., 2012), even subtle contrasts in density can be resolved.

1.2. Limitations of CT scanning and post-processing

Several artefacts are inherent to X-ray tomography. Some can be mitigated during scanning by careful choice of appropriate scan parameters and by hardware filters. Others can be corrected for during reconstruction and post-processing of the scanned object (Cheng et al., 2017 and Cnudde and Boone, 2013). Remaining X-ray artefacts have to be understood in order not to mistake them for real features (see further below).

Reconstruction consists in combining radiographs recorded during the scan in order to model the object as a 3D matrix of voxels. Each voxel is assigned a grey value that corresponds to the local X-ray attenuation of the object. As reconstruction algorithms are expected to improve rapidly by integrating a-priori information on the object in the reconstruction or by fusing micro-CT data with other kinds of tomographic data (neutron tomography data, for example), both raw and reconstructed data should be archived to allow better reconstruction in the future. The size of the data sets (1–100's of GB per set) does require large storage capacity.

After reconstruction, the inner structure of the object can be visualized. The data can be further processed to improve image quality and segmented to extract selected features. These features can be subjected to a morphometric and spatial distribution analysis, in order to evaluate, for example, manufacture skills or extent of damage. The volume that they occupy can be represented to better understand their 3D depositional context and their surface can be generated and meshed, for producing 3D printed replicas, that can be manipulated for a closer relation with the object and a better understanding of its use. Abel et al. (2011), for example 3D-printed virtual flints produced from X-ray micro-CT scans for detailed analysis of knapping styles. As the surface of a volume generated from micro-CT scans is oversampled, mesh simplification and smoothening are often required to allow 3D printing. This adds to the uncertainty of the final product.

Image processing and image quantification allow to derive valuable information from micro-CT scans. Even when using high performance computers equipped with fast graphic cards, they can cost 10 to 100 times the data acquisition time (Maire and Withers, 2014) which is about 60 min for small beads! Checking the results of each step of the image processing such as feature segmentation and separation over a whole object is particularly time consuming.

In brief, the models resulting from micro-CT scans have a high spatial and density resolution but are also tainted with uncertainty related to each phase of the scanning, from data acquisition to surface generation, as well as to the scanned object itself and the environmental conditions of the scan (Cheng et al., 2017). Quantification of uncertainty in micro-CT scans deserves more attention.

One must also be aware that, despite being classified as a non-destructive method, X-ray micro-tomography has been reported to alter visibly the colour of some materials, including antique glasses. Colour changes in glasses occur mainly around the impurities that glasses often contain. Some are reversible by exposure to UV light (Bertrand et al., 2012; Bertini et al., 2014). The photon beam sent by bright X-ray sources can also cause non-visible radiation-induced damage at the atomic level. By ionizing the material, it can affect results of future investigations (Bertrand et al., 2012). As the X-ray beam interacts with electrons around the nucleus of atoms, it is more likely to impact analyses based on the number of electrons trapped within the atom such as thermo-luminescence and optically stimulated luminescence-dating (in contrast to e.g. ^{14}C-dating, which relies on the number of neutrons contained in the nucleus of Carbon atoms). Fast electrons generated by X-ray radiation can produce secondary ionization that can degrade molecular bounds in matter. Organic compounds are thought to be among the heritage artefacts the most vulnerable to structural alteration by X-ray radiation (Bertrand et al., 2012). Ongoing research aims at monitoring radiation induced damage and determining tolerable doses of radiation for a wide range of materials. The task is complicated, in particular for composite archaeological artefacts (Bertrand et al., 2012). In any case, all information allowing the calculation of the dose of X-ray to which an artefact has been subjected should be archived so that possible bias on future analyses can be evaluated (Bertrand et al., 2012; Zappala et al., 2013).

1.3. Application in archaeological glass

Archaeological glass artefacts are arguably among the most complex of the non-metallic archaeological artefacts: To a basic, compositionally more or less homogeneous glass, variability is introduced by colourants and decolourizers, giving different properties to differently coloured glass. Opacifiers basically cause the formation of specific minerals in the glass, causing additional small-scale variability. The plasticity of the material at working temperatures allows manufacture of objects in which different types of glass are combined in detailed, complex and intricate 3-D designs. Finally, some types of glass are particularly sensitive to dissolution and fragmentation processes. Observing and understanding the resulting potentially extremely complex structures in (often relatively small) glass objects is not easy, especially if damage to the objects has to be limited.

All these aspects would theoretically be observable in micro-CT scans: As X-ray attenuation is strongly influenced by the concentration and atomic number of (especially the heavy) chemical elements glasses of different chemical composition – often related to colour - can be distinguished. This makes it possible to analyse the way that different glass colours are combined in polychrome objects. Moreover, impurities