A Non-invasive Investigation of Egyptian Faience Using Long Wavelength Optical Coherence Tomography (OCT) at 2 μm

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ABSTRACT

Egyptian faience is a non-clay ceramic semi-transparent material formed of a quartz core and alkali-lime glaze. Previous investigations have identified production techniques by using microstructure images obtained from invasive methods. Optical coherence tomography (OCT) is a non-invasive 3D imaging technique that produces virtual cross-sections of transparent and semi-transparent materials. A previous study by one of the authors demonstrated the feasibility of non-invasive investigation of microstructures of Egyptian faience using 930 nm OCT, but the limited probing depth prevented viewing down to the quartz core of the objects. This paper shows that an in-house developed OCT system using a longer wavelength (2 μm) was able to image the full microstructure from the top glaze layer down to the core, allowing rapid and non-invasive studies of intact objects and demonstrating the potential for surveying large museum collections. OCT virtual cross-section images at 5 wavelengths, 550, 810, 930, 1300 and 1960 nm, were compared and the optimum wavelength for OCT investigation of Egyptian faience microstructure was found to be 2 μm.

INTRODUCTION

Ancient Egyptian faience is a non-clay ceramic material consisting of a fine- or coarse-grained silica (quartz) core, an alkali-lime glaze and, in most cases, an interaction layer connecting the glaze to the core. The glazing methods for faience have been categorised into three main types: efflorescence, application and cementation (Nicholson and Peltenburg 2000). These types are categorised by their microstructure, using scanning electron microscope (SEM) images of polished sections extracted from the fragments (Tite, Freestone, and Bimson 1983). The cementation technique buries a formed quartz body in the glazing salts. During the firing process, the glaze forms on the outside of the object, and therefore, it is unlikely for there to be inter-particle glass in the core (Tite, Freestone, and Bimson 1983). The efflorescence technique mixes the glazing salts into the quartz body which is moulded and then fired; as the glazing salts are mixed within the core, this method results in vitrified material, known as inter-particle glass, within the core after firing. The application technique applies the glaze onto the quartz body as a slurry. Since the glaze slurry is applied to the surface, inter-particle glass is unlikely to be found within the core after firing. The SEM images of the polished sections of laboratory replicas made with the three different techniques clearly show that the glazing techniques and the particle size of the raw material can significantly influence the microstructure (Tite and Bimson 1986). Tite, Freestone, and Bimson (1983) suggested that the objects can be differentiated and categorised by the presence of glass within the core. However, there has been much debate and speculation about the validity of this method. In particular, the glazing methods could have been used in combination with each other (Tite, Freestone, and Bimson 1983; Vandiver 1983; Nicholson 1993; Vandiver 1998). Nevertheless, the non-existence of inter-particle glass in the core can exclude efflorescence as a possible glazing method. While it may not be possible to determine with certainty the glazing method given the potential complications of hybrid techniques, it is still possible to sort objects into groups according to their microstructure without necessarily identifying the exact technique.

The method of employing SEM (Tite, Freestone, and Bimson 1983) requires sampling, which is an issue for museum objects, preventing the study of intact objects. In addition, each polished section only provides a limited view of the microstructure through a single cross-section. Previous investigations (Liang et al. 2012; Liang, Sax, and Saunders 2012b) have demonstrated the feasibility of using OCT, a non-invasive 3D volume imaging technique based on a scanning Michelson interferometer with a broad spectral
band laser source, to image the subsurface microstructure of Egyptian faience without sampling. However, OCT at a central wavelength of 930 nm had limited probing depth that allowed only the glaze and interaction layers to be imaged in most cases. This is because it operates at a spectral range close to the absorption band for Cu$^{2+}$ ions that gives the typical turquoise colour to faience. A long wavelength OCT at 2 μm has since been developed, which has improved the probing depth by moving away from the copper absorption band to a longer wavelength where optical scattering is also reduced (Cheung et al. 2015a; Liang et al. 2015). This paper focuses on using 2 μm OCT to examine the microstructure of ancient Egyptian faience from the British Museum.

Materials and methods

Ancient Egyptian faience objects

The Egyptian faience objects used in this investigation were from a collection of reference objects, dating from approximately 1600 – 300BC, housed in the British Museum’s Department of Scientific Research. Five broken fragments of rings and shabtis (Table 1) were selected and imaged at multiple areas on each object.

OCT systems

OCT systems are based on a Michelson interferometer with a broad spectral band laser source. The probe head is typically situated centimetres from the object. The Michelson interferometer essentially compares the optical path travelled by the photons scattered back from the object with the reference path, to measure the depth of layers. OCT systems typically have superior resolution in the depth direction, determined by the bandwidth of the laser spectrum, to distinguish between thin layers. In comparison, the transverse resolution in the plane of the object surface, determined by the numerical aperture of the objective lens, tends to be of much lower resolution. The field of view of OCT in the depth direction tends to be 1–3 mm, while in the transverse direction it is usually around 5–10 mm.

Five OCT systems at central wavelengths of 550, 810, 930, 1300 and 1960 nm were used in this study. All OCT systems belong to the category of Fourier domain OCT where a fixed reference path is used in the interferometer, and a spectrometer is used to

Table 1. Egyptian faience objects surveyed.

| BMRL No. | Object Type | Date                        |
|---------|-------------|-----------------------------|
| 4698-16320-M | Ring         | New Kingdom (c. 1550–1077 BCE) |
| 4698-16321-P | Ring         | 18th Dynasty (c. 1550–1292 BCE) |
| 4698-16322-R | Shabti       | Late Period (c. 664–332 BCE)  |
| 4698-16323-W | Shabti       | 21st Dynasty (c. 1069–945 BCE) |
| 4698-16324-Y | Shabti       | 21st Dynasty (c. 1069–945 BCE) |
collect the interference signals as a function of wavelength. A Fourier transform of the interference signal produces a depth profile. Scanning of the laser beam over a line segment produces a virtual cross-section image and scanning over an area produces the 3D microstructure of the surface and subsurface volume in the form of a 3D image cube. OCT measures optical distances, or the time it takes for the light to travel through the medium in the depth direction (or direction of the optical axis of the OCT). All the OCT images presented in this paper are in optical distances in the depth direction. Physical thickness of a layer is determined by dividing the optical thickness by the refractive index (RI). OCT is sensitive to changes in RI, since larger changes in RI means higher reflectivity, as governed by Fresnel equations. The brighter areas in an OCT image represent regions of high reflectivity; the dark areas are where there is little change in RI, or high absorption and therefore very few photons scattered back from these regions.

The main OCT used in this study was the system at a central wavelength of 2 μm (1960 nm) developed by the Imaging and Sensing for Archaeology, Art History and Conservation (ISAAC) Lab at Nottingham Trent University (Cheung et al. 2015a; Liang et al. 2015). The resolution of the OCT in distinguishing thin layers in depth is ∼6 μm (in material, assuming its RI is ∼1.5) and the transverse resolution in the plane of the object surface is 17 μm. The speed of capturing a typical image cube of 500 by 500 depth profiles is ∼2 min and several areas of 6 × 7 mm each were scanned per object.

The OCT systems at 550 nm and 810 nm were also developed in-house by the ISAAC Lab using broadband supercontinuum sources from NKT Photonics (Cheung, Spring, and Liang 2015b). They have much higher depth resolutions of 0.8 μm and 1.2 μm (in material) and transverse resolutions of 5 μm and 7 μm respectively. Since CCD detector arrays can be used at these wavelengths, the capture speeds are much faster, at 10 s for a typical image cube of 500 by 500 depth profiles.

The OCT systems at 930 nm and 1300 nm were commercial off-the-shelf systems from Thorlabs (Callisto and Telesto respectively) with depth resolution of 4.5 μm (in material) and transverse resolution of 9 μm and 15 μm respectively.

**Results and discussion**

The 2 μm OCT allowed new aspects of the microstructure of Egyptian faience to be investigated. This is mostly due to the increased probing depth of imaging in the 2 μm regime, which allowed viewing down to the quartz core (Figures 1–3(b)). The increased probing depth shows characteristics of each layer throughout the microstructure of the object. The most important aspect that the 2 μm OCT shows is how very different the microstructures of the objects appear in the virtual cross-section images.

Even at first glance, the OCT images of the Egyptian faience show differences in microstructure. However, the interpretation of an OCT image is key to the understanding of exactly how the microstructures differ from object to object. As mentioned, OCT is sensitive to the change in RI between media, as well as the particle size. Simplistically, in terms of RI, the Egyptian faience has three components: glass (glaze), silica (quartz), air bubbles and pores. At 2 μm, the RI of soda-lime glass is ∼1.50 (Rubin 1985), fused silica quartz is ∼1.44 (Malitson 1965) and air is ∼1. When the RI change is large, for example, when the light reaches the air-to-glaze interface, a bright well-defined line appears. If the difference in RI is small, for example, between quartz and glaze, the OCT will show varying levels of weak scattering, depending on the grain size of the quartz and the orientation of the surface of a grain relative to the optical axis. When there is no change in RI, for example, within the glaze of the Egyptian faience, or within a large grain of quartz or large pore, that area in the OCT image will be dark.

The advantage of 2 μm OCT over 930 nm OCT is that all three layers can be seen using 2 μm OCT, rather than 2 layers in most cases (Figures 1–3(d)). However, this increased probing depth also brings with it a decreased depth resolution from ∼4.5 μm (930 nm OCT) (Liang et al. 2012a) to ∼6 μm (2 μm OCT) (Liang et al. 2015), which means any small features or details close to each other, such as thin gel layers (<6 μm) in the glaze due to weathering may become unresolvable. On the other hand, the difference in depth resolution between the two OCT systems did not make much difference to the visibility of any of the small features in the faience examples given here. Another trade-off is the image contrast, which is lower at 2 μm than 930 nm, since scattering is lower at longer wavelength which is also why it is more transparent at 2 μm.

The 2 μm OCT image of the 18th Dynasty ring (BMRL 4698-16321-P) (Figure 1(b)) shows, at the top of the image, the air-glass interface, and a well-defined transparent glaze layer. There is a gradual transition from the interaction layer into the core suggesting the presence of inter-particle glass in the core, which is also seen in the SEM image (Figure 1(c)). Judging by the interaction layer in the OCT image, it has fine-grained quartz particles compared with larger objects, such as the 21st Dynasty *shabti* (Figure 2).

The images of the 21st Dynasty *shabti* (BMRL 4698-16323-W) (Figure 2) show a very different structure from that seen in the 18th Dynasty ring (Figure 1). At the top of the image, there is a bright air-glass
Figure 2. A 21st Dynasty *shabti* (BMRL 4698-16323-W) from the British Museum Department of Scientific Research Reference Collection. (a) The *shabti* © British Museum Department of Scientific Research. (b) 2 µm OCT virtual cross-section image of the *shabti* with scale bars of 200 µm. (c) SEM image of polished section of the *shabti* (Tite, Freestone, and Bimson 1983) with scale bars of 100 µm. (d) 930 nm OCT image with scale bars of 200 µm.

Figure 3. A Late Period *shabti* (BMRL 4698-16322-R) from the British Museum Department of Scientific Research Reference Collection. (a) The *shabti* © British Museum Department of Scientific Research. (b) 2 µm OCT virtual cross-section image of the *shabti* with scale bars of 200 µm. (c) SEM image of polished section of the *shabti* (Tite, Freestone, and Bimson 1983), with scale bars of 100 µm. (d) 930 nm OCT image with scale bars of 200 µm.
interface followed by glaze of varying thickness and an interaction layer with large quartz particles. The strong scattering in the core is an indication of more quartz-pore interfaces and a lack of inter-particle glass in the core. This object has coarse-grained quartz judging by the structure of the interaction layer. The OCT image is consistent with the SEM image: the irregular glaze thickness, distinct interaction layer and coarse-grained quartz particles with no inter-particle glass in the core.

The OCT image of the late period shabti fragment (BMRL 4698-16322-R) (Figure 3) again shows a very different structure from what was seen for the previous two objects (Figures 1 and 2). The top of the image shows the bright air-glass interface, under which there is a very scattering glaze layer which is different from that seen in the other objects. Similar to the SEM image, there is no interaction layer in the OCT image, but a dark band is seen at the beginning of the core layer. Large air bubbles are seen in the glaze layer (Figure 1(b,d)), with the characteristic curved bright top and bottom interfaces with a dark centre (Liang et al. 2008).

Further images of another 21st Dynasty shabti (BMRL 4698-16324-Y) (Figure 4(a)) and New Kingdom ring (BMRL 4698-16320-M) (Figure 4(b)) fragments show similarities with the objects discussed above. The OCT image of the shabti (Figure 4(a)) shows similarities to that seen in the first 21st Dynasty shabti (Figure 2). The OCT image of the New Kingdom ring (Figure 4(b)) is very similar to that seen in the 18th Dynasty ring (Figure 1).

In summary, OCT images can be used to sort the faience microstructures into 3 groups. The OCT images show very different microstructures between the 18th Dynasty ring (Figure 1), 21st Dynasty shabti (Figure 2) and the Late Period shabti (Figure 3). The images also show similarities between the 21st Dynasty shabtis (Figures 2 and 4(a)) and between the 18th Dynasty and New Kingdom rings (Figures 1 and 4(b)). Even though OCT has a low resolution compared with SEM, the advantages of OCT being a rapid, non-contact and non-invasive technique mean that in the case of Egyptian faience, OCT can be used in larger and more extensive surveys than SEM. OCT allows intact objects to be imaged over a large enough area that is better representative of the whole object, while SEM requires sample removal, and each sample prepared as a cross-section yields only one image.

**Optimum OCT wavelength for examination of Egyptian faience**

When deciding on which wavelength to use, it is important to consider the purpose of the investigation. A prior survey conducted for common historical artists’ pigments in oil and egg tempera binders found the optimum wavelength to maximise penetration depth to be around 2.2 μm over the spectral range 400–4500 nm (Liang et al. 2013). The light scattering coefficient of materials tends to decrease with increasing wavelength over this range, making materials more transparent at longer wavelength. However, the depth resolution of OCT systems tends to decrease with increasing wavelength since it scales as wavelength squared. Therefore, it is a trade-off between whether the investigation requires high resolution with a shallow probing range or deeper probing depth with lower resolution. If both are required for the investigation, a middle ground should be selected where the depth penetration is balanced with the required resolution.

To demonstrate the optimum wavelength for OCT investigations of Egyptian faience, the 21st Dynasty shabti (BMRL-4698-16324-Y) was imaged using four different wavelengths for OCT from 550 nm to 1960 nm. The OCT images show shallower penetration depth but higher resolution at shorter wavelengths (Figure 5(a,b)) compared with the longer wavelengths. The 2 μm OCT shows the best depth of penetration. For the purpose of investigating the production methods and overall microstructure, for example to differentiate between those with and without inter-particle glass in the core, the 2 μm OCT is most suitable. However, if surface detail is important, such as monitoring the gel layer for glass deterioration, a shorter wavelength OCT would be more suitable (Read et al. 2019).
Figure 5. OCT cross-section image of a 21st Dynasty shabti fragment (BMRL-4698-16324-Y) using different wavelength OCT systems. (a) 550 nm. (b) 810 nm. (c) 1300 nm. (d) 1960 nm. Scale bars are 200 µm.

Figure 6. (a) Reflectance spectra of 21st Dynasty shabti (BMRL-4698-16324-Y) between 0.35 µm and 2.5 µm measured with an ASD LabSpec fibre optic reflectance spectrometer, with the spectral range covered by the OCT systems at 550, 810, 930, 1300 nm and 2 µm indicated in coloured bands. (b) Reflectance spectra of 21st Dynasty shabti (BMRL-4698-16324-Y) between 1.85 and 5 µm measured with a Bruker Alpha FTIR spectrometer in external reflection mode.
The relative appearance of the OCT images in Figure 5 can be understood by comparing them with the reflectance spectrum (Figure 6(a)) taken with an ASD LabSpec fibre optics reflectance spectrometer at a 45-degree angle to the surface, in retro-reflection mode. There is more scattering at 550 nm than at 810 nm as can be seen in both the OCT images (Figure 5(a,b)) and the reflectance spectrum (Figure 6(a)). The 810 nm OCT range coincides with the strong Cu²⁺ ion absorption band which results in the limited probing depth in the OCT image. Light scattering is stronger at 1300 nm than at 2 μm as can be seen in the OCT images, while reflectance is higher at 1300 nm in Figure 6(a). The spectral coverage of the 2 μm OCT includes the water absorption line centred around 1.9 μm found in relation to hydration of glass in the process of degradation which varies from object to object (Read et al. 2019).

The 2 μm OCT system appears to give the best depth of penetration in imaging the subsurface microstructure of ancient Egyptian faience. OCT systems at shorter wavelength than 2 μm are hindered by absorption and scattering, however, there is still the question of whether it would be beneficial to employ an even longer wavelength OCT than 2 μm. Recent development of mid-infrared OCT at 4 μm (Zorin et al. 2018; Israelsen et al. 2019) have shown promise in imaging industrial ceramic materials made of alumina and zirconia, achieving deeper depth of penetration compared with 1300 nm OCT and even 2 μm OCT (Zorin et al. 2020). However, currently 4 μm OCT is still one to 3 orders of magnitude slower than the 2 μm OCT used in this study, which makes it impractical for analysing large collections. In addition, FTIR reflection spectra collected by a Bruker Alpha spectrometer in reflection mode shows that there is a strong increase in absorption at 2.7–5 μm (Figure 6(b)). Therefore, considering all aspects of current technology, the characteristics of Egyptian faience and the purpose of the investigation, the optimum OCT for imaging Egyptian faience is at 2 μm.

Conclusion

Longer wavelength OCT at 2 μm provides an increased probing depth down to the core, which allows all layers of Egyptian faience objects to be examined online to examine the quartz particle size, glaze thickness, indications of the presence of inter-particle glass in the core and whether there is an interaction layer. Combined with the non-invasive nature of the technique and the rapid imaging speed, 2 μm OCT was found to be the optimum system for studying the microstructure of intact Egyptian faience objects, allowing multiple areas of acquisition per object and enabling large number of objects to be studied. The similarities and differences of raw material and manufacturing technique will enable investigations into correlations between microstructure, object type and size, period and place of manufacture.

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