Borosilicate glass layers on Mycenaean glass: Surface alterations by glass–borax–gold interactions

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ABSTRACT

A previous report (Möncke et al., PCG 2013) discussed the possible formation of a borosilicate layer on the surface of Late Bronze Age glass samples (Mycenaean period, excavated in Palaia Epidavros, Greece). Now, we investigate potential mechanisms for such suggested borate incorporation into the surface of alkali-lime silicate glasses. Since former studies reported that glass relief fragments have been found alongside gold foils featuring identical reliefs, we tested the hypothesis that borates could have been introduced through gold working via the reaction of a borax-treated gold foil with the silicate glass surface at elevated temperature. A borosilicate layer was successfully generated on replica soda lime silicate glass samples and identified by vibrational spectroscopy. Different approaches were used in order to merge gold sheets to the glass samples. At temperatures above 800 °C, borax could have been used either as a solder in the fusion process between a gold sheet and the glass surface, or borax was transferred from the gold sheet previously in contact with a borax melt, presumably for cleaning gold or during ore extraction. The depth of the borosilicate layer on the thus treated glasses extents from the surface several μm into the bulk glass as probed by micro-Raman and IR spectroscopy, while the dimensions of the spread across the glass surface can be visualized by neutron radiography.

1. Introduction

The Mycenaean palace epoch (14th to 12th ct. BCE) is not only known for its impressive architecture, bronze weaponry and ceramics, but also for a rich variety of turquoise and blue glass products [1–3]. Most of the glass objects found in burial sites and residential areas from the Mycenaean era come in the form of beads. Seals and relief inlays are also known [4–6]. In some cases, relief inlays and relief beads have been excavated together with gold sheets of similar shape to the vitreous SiO2 [16]. Due to the absence of O–H vibrations at ~3600 cm⁻¹, superimposed on the reflectance profile typical of vitreous SiO2 [16]. In addition, an unexpected absorption band was found at ~930 cm⁻¹, at that time, the authors suggested that this band might originate from the presence of a thin borosilicate glass layer.

Because of its low atomic weight, boron is a challenging element for quantitative by common analytical techniques. Most corrosion and restoration studies of antique glasses are based on techniques such as X-ray fluorescence or SEM-EDX [18,21]. However, these techniques are not sensitive to boron contamination, especially when only present in very low levels or in thin superficial layers. Other techniques are often destructive (e.g., mass spectrometric analyses), or require
uncontaminated or coated surfaces (e.g., XPS) [22]. Thus, it is not surprising that many earlier studies missed this aspect especially when boron is present in a thin layer. A possible source for boron, or the process by which boron would have been introduced into the glass surface, was only addressed briefly in the earlier publication by Möncke et al. [16]. In a continuation of this earlier study, we now search for possible sources for boron and/or applications that might have caused the transfer of boron into the archaeological glasses. It is known, for example, that the mineral borax, Na₃B₅O₆·10H₂O, can be used either as a solder [23] or as a simple means to extract gold from ores [24]. The property of borax to bind undesired, often dark coloured oxides at a metal’s surface and, therefore, its application in the purification of gold is mentioned in modern handbooks of gold smelting [23], although we are not aware if this knowledge would have existed during the Late Bronze Age. It is also known that borates will adhere to the oxidised surface of gold samples [25], and as speculated by Schliemann in 1878 ([26], p., 266) this aspect was already known in Mycenaean times. When gold, extracted from ore with the use of borax, was later brought into contact with hot glass, a transfer of borate could have occurred. In both cases, we assume that hot glass might have been cast into a mould, in which a gold sheet was already laid-out. Due to the deterioration of the glass surface by moisture, acidity and interactions with the surrounding [17,18,27–29], we assume that the decorative gold layer delaminated in most cases over time [5,8].

In all experiments, we focus on the reactivity of boron oxides with a silicate glass surface at elevated temperatures. Thereby, we assume that a better understanding of the reaction mechanism of boron with a silicate glass network will help us to explain the origin of the unexpected infrared signature of Mycenaean glass surfaces. In the absence of suitable experimental (non-destructive) tools for direct chemical analysis, we continue with the hypothesis that the observed IR spectroscopic features are caused by the presence of a significant amount of boron on the surface of the glass. We use mostly vibrational spectroscopy to follow structural variations on the surface of the glass samples subjected to different treatments. Depth profiling by infrared reflection spectroscopy was also conducted on broken edges and obliquely polished samples to probe the extent of the borate-silicate interaction layer. In an attempt to visualise the dimensions of the borosilicate layer and to explore the general applicability of neutron radiography to archaeological glass samples that need to be studied non-destructively, radiography experiments were conducted. We note that, while this study can give experimental proof for potential borate transfer reactions to the glass, final proof on the actual mechanisms that caused the layer formation in Mycenaean samples cannot be given.

2. Materials and methods

2.1. Model glass

Previously published quantitative analysis of Mycenaean glass samples revealed an average composition of approximately 64 to 70% SiO₂, 5 to 7% CaO, 15 to 19% Na₂O, 1–3% Al₂O₃ and 2–4% MgO (in wt %); the remaining constituents include among others K₂O, Fe₂O₃, MnO, Sh₃O₆, Cl and S [11,16,30]. Based on this composition, a model soda lime glass (SLS) with similar expected properties and the molar composition 65SiO₂·15CaO·17Na₂O·3Al₂O₃ (in wt%: 64 SiO₂·14 CaO·17 Na₂O·5 Al₂O₃) was prepared.¹ Quartz powder, calcium carbonate, sodium carbonate and aluminium hydroxide (all 99.5%) were used as raw materials. The batch was melted at 1400 °C in an electrically heated muffle furnace using Pt crucible. The melt was cast on a brass block, annealed at 550 °C and cooled to room temperature at 2 K/min⁻¹. In order to compare the borate layer with the silicate glass, a sodium diborate glass (SDB, Na₂B₂O₅) was prepared from borax, Na₂B₂O₅·10H₂O. The substance was heated to 1200 °C, the melt was cast on a brass block, annealed at ca. 800 °C and cooled to room temperature at 2 K/min⁻¹.

2.2. Glass-to-gold fusion experiments at 600 °C - 1000 °C (process A)

The model soda lime glass (SLS) was cut into slices of 1 mm thickness and polished to replicate an as-cast surface. Samples were covered with fine borax powder and fused to a thin gold plate through exposure for 30 min to different temperatures, selected between 600 °C and 1100 °C. For comparison, blind samples without borax layer and/or gold covering were similarly prepared.

2.3. Fusion experiments by direct casting (process B)

A model soda lime glass (SLS) batch was melted at 1400 °C as described above. For direct fusion, a gold plate was coated with borax powder and heated to 800 °C. The glass melt was directly cast on the borax-covered gold plate. A blind sample was prepared by casting the glass melt on a non-borax treated gold sheet. The same procedure was also conducted without preheating the borax-coated gold plate. The general process is schematically visualized in Fig. 1.

2.4. Boron transfer from impure gold sheets (process C)

Gold sheets were coated with borax and annealed at 800 °C to simulate a cleaning process during gold purification [23]. The remaining (visual) layer was subsequently removed either by dissolution in hot water or by removing the residual borax mechanically. The so-prepared gold sheets as well as a non-treated blind sample were fused to a model glass at 800 °C.

2.5. Vibrational spectroscopy

After treatment, all glass samples were analysed by infrared reflectance micro-spectroscopy (Perkin Elmer Spotlight 200i) at 50× magnification with a reflectance angle of 0° within a range of 4000 cm⁻¹ to 500 cm⁻¹, using a gold mirror as reference. The average of 200 spectral accumulations, at 2 cm⁻¹ resolution, from an area covering 200µm × 200µm were used for IR analysis. A selection of samples was polished obliquely and analysed in order to get an idea of the depth profile for the boron distribution from the glass surface into the sample. Here, an area of 5 µm × 400 µm was analysed. The such obtained reflectance data was used without any further treatment.

Kramers-Kronig transformation was not applied to the reflectance spectra due to the limited accessible wavenumber range, which lacked the far infrared region and, as a consequence, did not allow extrapolation to zero frequency. Reflectance spectra show shifts of band maxima compared to absorption spectra and emphasise the intensity of low energy bands compared to the intensity of high energy bands. Nevertheless, the direct comparison of reflectance spectra that have been measured under the same conditions gives important information on structural variations. Also, the spectral similarity is close enough to absorption spectra to allow assigning the measured bands to vibrations of various structural entities.

Some samples were additionally investigated by Raman micro-spectroscopy (Renishaw InVia Raman micro-spectrometer) at 50× magnification, using a laser wavelength of 514 nm for excitation. Spectra were taken within the range of 100 cm⁻¹ to 1650 cm⁻¹ and 2 cm⁻¹ resolution. For each spectrum, 10 accumulations each with 10 s exposure time were applied.

2.6. Neutron radiography

One sample of process B (Fig. 1) was selected for neutron
radiography in order to visualise boron containing glassy material. The measurement was conducted at the Paul Scherrer Institute at Villigen, Switzerland, using the thermal neutron imaging instrument NEUTRA. A scintillator size of 50 μm combined with an Andor Neo camera (resolution: 1950 × 1950 pixel) and a 50 s exposure time was used for neutron detection. The sample was rotated at 360° with a step size of ca. 0.5°. For further information, see ref. [31].

3. Results and discussion

3.1. Fusion experiments with borax as flux

It was found that heat treatment of polished glass plates with gold (process A) leads to adhesion between glass and gold surfaces. Adhesion increases with increasing treatment temperature. Below 700 °C, the gold is only weakly attached to the glass surface. We observed no difference between the adhesion of blank samples and those covered with borax. However, surface crystallisation appears in heat-treated samples without a borate layer, whereas crystallisation was inhibited for borax-coated samples. The glass becomes softer with increasing temperature. Therefore, only samples treated at temperatures below 800 °C remained in their original shape. Samples treated at higher temperatures afford a mould during the heat treatment to prohibit unwanted deformation. Casting a model glass on a gold sheet (process B) leads to strong adhesion between the glass and the gold foil. As in process A, coating the gold with borax powder did not create a significant difference in adhesion. However, borax appeared to spread further on the surface than expected; Fig. 2 shows the neutron radiography of a model glass cast on a coated gold sheet. As seen from the image, the boron-containing layer spreads far over the sample, forming a thin film at the sides.

The modification of gold sheets before fusion (process C) did not affect the adhesion behaviour significantly. This observation is similarly to process A. When treating the gold with borax at temperatures $T > 800$ °C, green coloured drops of borate glass formed at the surface of the gold sheet. The glass could be removed either mechanically by bending the gold sheet or within 2 h by dissolution in hot water.

3.2. Vibrational studies

3.2.1. Processes A and B

Fig. 3 displays the FT-IR reflectance spectra of the two model glasses (SDB, a and SLS, b) of this study. Additionally, the spectrum of borax powder was added to the figure for comparison (light grey filled spectrum). The Na$_2$B$_4$O$_7$ glass (Fig. 3a) exhibits a B–O–B bending vibration at 705 cm$^{-1}$ (very close to the 710 cm$^{-1}$ band of crystalline borax), and a broad reflectance band from about 775 to 1150 cm$^{-1}$ which forms the envelope of several sharp bands that are also evident in the spectrum of borax. The 775–1150 cm$^{-1}$ envelope of SDB, with apparent reflectance maxima at 870 and 980 cm$^{-1}$, is due to the asymmetric B–O stretching vibration in tetrahedral borate units [BØ$_4$]$^-$, where Ø indicates a bridging oxygen atom [32,33]. In

![Fig. 1. Scheme of the casting process B (left), and the corresponding sample that was investigated by neutron radiography (right).](image1)

![Fig. 2. Normalized neutron radiography of a glass cast on a borax-coated gold sheet (process B). The dark area shows high neutron-atom interactions, which is assumed to be due to neutron capture by boron atoms. See supplementary information for a 180° rotation video.](image2)

![Fig. 3. FT-IR reflectance spectra of the two model glasses SDB (a) and SLS (b), together with the fingerprint spectrum of crystalline borax powder. * marks artefacts from CO$_2$ in the air (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.).](image3)
addition, different overlapping bands in the range of 1150–1500 cm\(^{-1}\) for both the SDB glass and borax indicate the presence of stretching vibrations of B–O bonds in trigonal borate groups [32,33]. The SLS model glass spectrum (Fig. 3b) is typical for a soda lime silicate glass. The Si–O–Si bending mode is visible at about 745 cm\(^{-1}\), the Si–O stretching profile from about 800 to 1250 cm\(^{-1}\) shows its maximum reflectance at 1045 cm\(^{-1}\) and designates a dominating presence of Q\(^3\) units, while smaller contributions from Q\(^2\)to Q\(^4\) units add intensity to the lower- and higher-frequency sides of the Q\(^3\) envelope, respectively [16,34]. In comparison to the above two endpoint glasses, SLS and SDB, the spectra of heat-treated model glasses SLS (process A) appear to consist of features present in the spectra of both glasses. After treatment at temperatures below 700 °C the coated model glass (Fig. 4c) shows reduced intensity of the high frequency envelope with apparent maxima at about 1325 and 1375 cm\(^{-1}\), indicating the formation of an amorphous borate phase with trigonal borate groups in contrast to the starting borax crystalline phase (grey spectrum in Fig. 3). A similar spectrum was obtained for glass cast on borax-coated gold sheet (Fig. 4e), which shows an increased content of borate triangles (band at 1342 cm\(^{-1}\)) relative to the spectrum in Fig. 4c. Treatment at temperatures above 700 °C (Fig. 4d) reduces further the relative intensity of the infrared profile corresponding to trigonal borate units (1200–1500 cm\(^{-1}\), suggesting a structural reconstruction process. This is supported by additional changes in the 800–1200 cm\(^{-1}\) envelope including the reduction in intensity of the borate shoulder at ca. 870 cm\(^{-1}\), relative to that of the < 700 °C treatment, and the appearance of a new band maximum at 1020 cm\(^{-1}\) which is in between those of the B–O stretching in SDB (980 cm\(^{-1}\)) and Si–O stretching in SLS (1045 cm\(^{-1}\)). In addition, the band for bending vibrations is measured at 727 cm\(^{-1}\) between those of SDB (705 cm\(^{-1}\)) and SLS (745 cm\(^{-1}\)).

To explore possible interactions between the borate and silicate components, Fig. 5 compares weighted averages xSDB + (1 – x) SLS of the spectra of sodium-diborate glass (SDB, x = 1) and the model soda lime glass (SLS, x = 0) with the measured spectrum of the borax-coated model glass heat-treated at 800 °C (black, solid). In comparison to the progressive evolution of the weighted average spectra from x = 1 to x = 0, the experimental coated model glass spectrum shows a shift of its low-frequency band to ca. 695 cm\(^{-1}\), indicating the formation of Si–O–B bonds [16,33]. Additionally, the high-frequency band of the trigonal borate groups shows two shoulders one at the lower (1325 cm\(^{-1}\)) and the other at the higher-frequency (1325 cm\(^{-1}\)) side of the initial 1342 cm\(^{-1}\) band of SDB glass. This effect suggests a reduction in symmetry of the trigonal borate units [35], which may be the result of trigonal B bonding to Si centres through Si–O–B bridges. Finally, the main reflection maximum of the experimental spectrum in Fig. 5 appears at 975 cm\(^{-1}\), whereas no weighted average spectrum xSDB + (1–x) SLS shows maximum at 975 cm\(^{-1}\). These findings show clearly that heat treatment of the model soda lime silicate glass coated with borax leads to interactions between the silicate and borate structural units.

We have performed depth profiling of the infrared reflectance spectra to probe the extent of the interactions between the borate/ borosilicate layer and the interior of the silicate glass. Samples treated at 800 °C according to process A were polished obliquely and subsequently investigated by infrared reflectance spectroscopy. The evolution of the infrared spectra is presented in Fig. 6 in comparison to the spectrum of the pristine SLS model glass. According to the previous discussion, the first spectrum in Fig. 6 (0 μm spectrum) suggests interactions between the borate and silicate components as manifested by spectral changes at both low- and high-frequency regions compared to the SLS spectrum. The influence of the borate layer is visible very clearly up to ca. 2 μm into the glass bulk. This is especially shown by the gradual disappearance of the high-frequency band of trigonal borate units at about 1370 cm\(^{-1}\), and the shift of the main band from about 1000 cm\(^{-1}\) (overlapping of the [BO\(_4\)]\(^−\) and Q\(^3\) bands) to 1045 cm\(^{-1}\) (main Q\(^2\) band in SLS). The influence of borate addition on the silicate network decreases when measuring deeper into the bulk. In terms of the 1370 cm\(^{-1}\) band, Fig. 6 indicates that no borate triangles are formed at 6 μm or deeper. However, consideration of reflection at about ca. 1000 cm\(^{-1}\) shows that there should be some borate-silicate interactions even at 9 μm. The spectrum measured at 12 μm shows that the activity at 1000 cm\(^{-1}\) is identical to that of the SLS glass. However, a new shoulder appears at ca. 950 cm\(^{-1}\) in the 12 μm spectrum, with relative intensity exceeding that of the similar shoulder for SLS glass. This

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2 Strictly speaking, each asymmetric Q\(^n\) vibration mode consists of three vibrational components with slightly different energies. Here, we will only consider the average band position of the three components without the application of a band deconvolution. Further information can be found in the literature [e. g. 18].
enhanced 950 cm$^{-1}$ shoulder could be related to the 930 cm$^{-1}$ band which was found superimposed on the reflectance profile of vitreous SiO$_2$ measured on Mycenaean glass surfaces [16]. Therefore, this part of this work shows that (i) borate triangles are forming in an amorphous phase with thickness smaller than ca. 6 μm, (ii) borate tetrahedral units [BØ4]$^-$ are forming deeper into the silicate glass, in layers that reach at least 9 μm into the bulk. These results are consistent with earlier findings in ref. [16], where the thickness of the borosilicate layer was estimated to be a few μm.

Raman spectra of heat-treated glass samples (process A) are shown in Fig. 7. The spectrum of the model glass SLS is dominated by bands at 1090 cm$^{-1}$ (symmetric stretching of Q$^2$ units) and 595 cm$^{-1}$ (symmetric stretching-bending of Si–O–Si bridges), while weaker bands are observed at 950 cm$^{-1}$ (symmetric stretching of Q$^2$ units) and 780 cm$^{-1}$ (bending of Si–O–Si bridges) [36]. Heat treatments of the SLS glass – cast on borax-coated gold sheet – at temperatures above 700 °C lead to a broad envelop between 1300 and 1520 cm$^{-1}$, which indicates the formation of trigonal borate units [33,37,38]. In addition, a shift of the bending mode to 755 cm$^{-1}$ is observed and is consistent with the formation of Si–O–B bridges [16]. In contrast to strong signatures of borate species in the infrared reflectance spectra (Fig. 4), no additional Raman evidence is available for the borate species. This is most likely due to the larger depth probed by Raman spectroscopy, leading to spectra dominated by the response of the silicate species. On the other hand, infrared reflectance spectroscopy is a surface-sensitive technique and, thus, its response comes from the top few μm thick layer where the borate species are found in the studied glass samples. Above 900 °C, crystallisation of sodium silicates and sodium borates dominate the spectra, leading to the formation of multiple sharp peaks [39].

3.2.2. Process C

Fusing the model SLS glass to a gold sheet that had been treated with borax at 800 °C or more, leads to similar vibration bands as seen above. As an example, Fig. 8a shows the normalized FT-IR reflectance spectrum of a glass surface that has been fused to a borax-coated and heat-treated gold plate. The presence of the 1315 and 1375 cm$^{-1}$ range. To the contrary, activity was detected at 930 cm$^{-1}$ and was attributed to [BØ4]$^-$ units coordinated partially to less mobile, divalent Co$^{2+}$ ions [16].

3.3. Neutron radiography of cast glass (process B)

Fig. 2 shows the normalized neutron radiograph of a glass block
after casting a model glass melt on a gold sheet covered with borax powder. The untreated SLS glass is almost transparent to the neutron beam. Boron with a high neutron absorption and hydrogen with its high scattering cross section are both expected to show transmission losses for neutron radiation. For the laboratory samples, no significant amount of water is expected, and this assumption was confirmed by optical transmission and reflectance IR spectroscopy in the near IR region.

During the casting process, borax was only present as a thin powder layer on top of the gold sheet. Nevertheless, a thin dark layer (Fig. 2), that – in the absence of water – is indicative for a boron-containing material, is visible in the radiograph of Fig. 2 even at the sides of the glass sample (except for the missing sides, where edges broke off from the sample at the front and backside of the image’s center). The thin layer can be more easily seen in the video of the rotating sample (see Supplementary information, Video S1). Thus, we were able to demonstrate that a layer of boron-containing glass form easily and that this layer can spread over a large surface area, without the necessity of a complete and homogeneous covering of the glass sample with borax powder. Extended borosilicate glass layers could therefore form even if only small parts of the sample were in contact with borate powder.

For the analysis of archaeological glass samples, neutron radiography appears to be a promising method to detect boron even in small quantities. Unfortunately, the sensitivity of neutron imaging to boron atoms, which absorb neutrons, is similar to that of hydrogen atoms that scatter the neutron beam. Although we could exclude the presence of large hydrogen quantities in our laboratory test sample, this is not the case in the corrosion layers of ancient glass samples. However, we do expect a difference in neutron interaction within the surface of archaeological glass samples due to different process intensities. Moreover, the combination of infrared spectroscopy, confirming water bands in the near IR region, with neutron radiography could support such investigations. In the context of this paper, we focused on the remaking of borosilicate layers. We could confirm that neutron radiography is a useful, non-destructive method for the analysis of small glass fragments, and we plan to follow up with neutron tomography of archaeological glass samples in the future.

3.4. Availability of borates in the Aegean region

Recalling the earlier findings regarding the formation of a borosilicate layer on LBA glass samples [16], and without clear evidence that the use of borax was most likely introduced by goldsmiths rather than glassmakers, we want to evaluate the availability of borates in the Aegean region. At the present time, large deposits for borates are located in Turkey [40]. Many of these deposits are rather close to the surface and are well conceivable for ancient mining activities. For gold the most important sources for the Late Bronze Age are well known today: Next to mining activities on today’s Turkish to Greek main land, e.g. in Anatolia, Chalkidiki or Macedonia [41], trade and warfare with the eastern realms supplied the Mycenaean palace culture with gold. Due to the overlap of gold deposits [42] with borate deposits [43] a connection of gold and borate mining is plausible. However, only excavation of such ancient mines will lead to further information.

4. Conclusions

Our continued research regarding new evidence for possible boron incorporation into the surface layer of Mycenaean glass fragments lead us to investigate three possible effects borate usage may have had in the manufacturing process. This starts from the initial speculation that boron-containing glass surfaces formed during gold working when glass artefacts were covered in gold. We argued that compounds from the borax family were used as interlayers to improve the adhesion between gold and the glass’ surface. In addition, borax additions inhibit surface crystallisation during heat treatments between 700 °C but below 1000 °C, leading to more appealing surfaces in secondary hot-forming processes. Finally, borax (or other borates) could have been used as a cleansing agent for gold surfaces. In that case we expect borate residues on the gold surface to react with the glass during fusion.

Replicating all scenarios, we confirmed by vibrational spectroscopy a diffusion of borates into the silicate glass surface at temperatures above 700 °C. While introduction of borates from fusion with contaminated gold plates will lead to rather small amounts of borates at the glass surface, we could show a diffusion of borates into roughly the first 9 μm of a glass surface when working at temperatures of about 800 °C. This demonstrated that the formation of a borosilicate layer can indeed occur as a result of such treatment. By using purified gold or such that has been extracted by the borax method, the introduction of borates into the glass surface would even be a necessary consequence of fusing gold sheets to glass.

Using neutron radiography we obtained further evidence for the boron incorporation into the studied model glasses upon casting on a gold sheet covered with borax. Although a differentiation between boron and hydrogen is not readily possible in this method, we assume that surface layers of corroded glass samples will show differences between a borate layer and a corroded glass layer. Especially in combination with IR reflectance spectroscopy, which can unambiguously identify water bands, neutron radiography could provide further evidence for the formation of such a boron-containing glass layer.

The intentional use of borax as an additional glass-to-gold soldering agent would present a significant step in the understanding of metal-glass interaction. Similarly, the washing of noble metals in borax melts, which is well-known in contemporary technology, would be a notable feat, reflecting the high level of Mycenaean-age processing of materials.

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