



Parting Layers, Ash Trays, and Ramesside Glassmaking:
An Experimental Study

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Summary

A series of glassmaking and glass colouring replication experiments was undertaken in order to test some of the current hypotheses concerning Late Bronze Age glass production at Qantir-Piramesses. These were based on the model of glassmaking developed in this volume, and aimed in particular to test the behaviour of the parting layer and the local ceramic under the proposed chemical and thermal conditions. Modern ash trays made out of Egyptian Nile silt clay were used as proxies for LBA reaction vessels and crucibles, and both raw glass and coloured glass ingots were produced in them. This experimental study, based on detailed observation and technical studies of archaeological samples from Qantir-Piramesses, not only provides material readily comparable to the archaeological finds, but brings to the forefront practical issues concerning the nature of the parting layer, its application, the melting procedures, the re-use of crucibles, and indirect evidence of primary production, such as the impact of sodium chloride, a major component of plant ashes, on the ceramic. Although this string of experiments does not fully replicate LBA glassmaking technology, much information was obtained and further areas of ambiguity identified.

1. Introduction

Previous work has identified two main types of ceramic vessels related to glassmaking in Qantir-Piramesses, namely Reaction Vessels which were used to produce semi-finished or raw glass from its raw materials, and Cylindrical Crucibles which were used to produce coloured ingots.¹ Both vessel types were made from local Nile silt, Vienna system I.E.01. Much of the reconstruction in this volume of the Ramesside glassmaking technology is based on the investigation of fragments of these vessels, identified by their characteristic inner calcareous coating, dubbed Parting Layer. This layer had first been identified some fifty years ago on glass crucibles from Amarna, thought to serve as a means to separate the glass charge from the ferruginous ceramic.² More recently, a series of discolorations and changes to the ceramic underneath the parting layer have been identified and tentatively interpreted as evidence for glassmaking from raw materials, as opposed to re-melting existing glass.³ The differentiation between glassmaking and glass melting is not only of fundamental significance for the question of native Egyptian glassmaking in view of overwhelming textual and iconographic evidence suggesting large-scale import of glass ingots into Egypt but not out of it, but also for a better understanding of the practicalities of Ramesside glassmaking in Qantir-Piramesses. The archaeological evidence points towards a sequential process by which raw glass was made in ovoid domestic vessels, followed by the re-melting and coloration of the raw glass in crucibles to produce ingots suitable for export and further processing. Both vessel types were made from the same ceramic and prepared in the same way by applying a parting layer on their inside; the main difference between the two vessel types appears to be in the firing temperature to which they were subjected. However, there is no apparent reason why the first step should have required the use of a different vessel type. Indeed, many crucible fragments from Qantir-Piramesses seem to have remains of semi-finished glass, and it is not clear why raw glass should not have been made in crucibles instead of – or in addition to – reaction vessels. Part of the purpose of this study is to investigate the reactions between the glass charge, the parting layer and the ceramic of the vessels under controlled conditions, and to identify diagnostic traces, preserved in the technical ceramic, to distinguish between glassmaking and glass melting vessels. It is hoped that these criteria then can be applied to the archaeological material, not only in Qantir-Piramesses but also in other sites where such glassmaking vessels are preserved.

1 REHREN/PUSCH 2005.

2 TURNER 1954.

3 SCHOER/REHREN this volume; REHREN/PUSCH 2007.



To recreate the ancient technology of glass production, a firmly grounded understanding of the limiting and exploitable factors surrounding production must be obtained, such as raw materials and material properties. Much work has been done already to identify these factors. A major limiting factor in LBA glassmaking is the quality of the technical ceramic. The crucibles and reaction vessels were produced from clay which does not withstand temperatures in excess of 1150 °C and readily reacts with the components of the glass batch. The raw ingredients of the glass from Qantir-Piramesse have been established to be crushed quartz and alkali rich plant ash.⁴ In particular the latter material is likely very variable in composition, and consists not only of compounds conducive for glass forming, but also contains varying amounts of non-reactive salts, most notably sodium chloride. The reactive salts, on the other hand, would strongly absorb material from the ceramic, discolouring the glass. Despite these limiting factors, the glass quality from Qantir has been shown to be equal to other Egyptian glasses, and not significantly affected by contamination from either the technical ceramic or the non-reactive components of the batch.⁵ The parting layer, whose understanding much of this paper is devoted to, has been identified as a very beneficial element in the design of the glassmaking vessels, whose exploitation helped in over-coming many of the limitations identified above. It is crucial in preventing contamination of the glass charge during melting, and to facilitate release of the coloured glass ingots from the crucibles at the end of the process. It is a highly calcareous layer applied to the inside of the ceramic vessels prior to their use in glassmaking. Its main function is to act as a parting agent between the ceramic of the glassmaking vessels and the liquid glass, based on its limited reactivity with silicate melts.

Although no suitable furnace structures have yet been archaeologically identified, heavily vitrified spots around the base of the crucibles suggest use of a small furnace with localised heat aimed at the wall of the crucibles near their base, and the funnel attachment means the furnace was probably accessible from the top to add more glass to the crucible during melting.⁶ The operating temperatures can be extracted from our knowledge of the melting properties of glasses,⁷ but also by the levels of vitrification in the industrial ceramics used,⁸ and are thought to be in the range of 900 to 1100 °C.

All in all, much can be interpreted from the archaeological finds and their visual and chemical analysis, but to test the reconstructed process in the physical world is important in validating it. Reconstructing this technology in its entirety would be a formidable task, therefore only certain aspects of

production were simulated for this experimental study. To reliably control aspects of temperature, duration and furnace atmosphere, a programmable electric kiln was used rather than a probably wood-fired furnace of unknown design. Nine ceramic vessels, otherwise used in Egypt as ash trays, were available for glass making experiments. These modern ash trays were chosen because they are made from local Nile silt clay, presumed identical to the clay used to make the archaeological glassmaking vessels. This is the pivotal aspect of the study because the permeability and chemistry of the crucible ceramic plays an important role in promoting comparability, but also in the understanding of the movements and reactions of compounds among the various zones developing from the interaction of the charge, parting layer and ceramic. The overall shape of the ash trays was not dissimilar to the crucible finds from Qantir, although their useful volume was much less. Their inner diameter near the rim varied from 6 to 7 cm, their inner height was around 3.5 to 4.5 cm, and the wall thickness ranged from 1 to 2 cm. The typical finished glass ingot made in these vessels weighted from 150 to 320 g, as compared to an estimated 2 to 4 kg for the Qantir ingots.⁹

The specific objectives of this study were to determine the composition of the parting layer and to replicate it; to investigate different methods of application of the parting layer; to test the behaviour and efficiency of the parting layer during glassmaking and glass melting; to document and interpret the effect of common salt (NaCl) as part of the glass charge on the ceramic underneath the parting layer; to document the effect of temperature on the ceramic and of temperature and length of firing on the glass charge; and to illustrate the volume differences between raw material batches, raw glass, crushed raw glass and finished glass.

4 SCHOER/REHREN this volume.

5 SCHOER/REHREN this volume; BRILL 1999, 27-37.

6 REHREN/PUSCH 2005, 1757.

7 REHREN 2000a, 1227.

8 TURNER 1954.

9 PUSCH/REHREN this volume, chapter 3.2.3 (Tiegelvolumina).

1.1 Parting layer replication

The archaeological crucibles were lined with a calcium-rich coating of variable composition which has been referred to in the past as parting layer between the ceramic and the glass,¹⁰ and seen as a non-reactive separating material and/or a buffer from which the glass melt could draw additional calcium oxide to augment glass forming.¹¹ The apparent variability of the composition of the parting layer could be due to several possibilities: a mis-identification of what layer is, in fact, the parting layer; the parting layer being at different stages of decay or reaction with the surrounding materials; or variation in the actual production recipes. To recreate the parting layer for our experiments, some preliminary analyses were undertaken on crucible fragments with particularly well preserved parting layer material, to better understand how this layer was produced. Since the parting layer is predominantly comprised of calcium carbonate, at the temperatures above 800 °C required to make glass, any such layer would likely decompose into powdery quick lime (CaO) which would readily react with its surrounding materials, and which also lacks mechanical strength. Therefore, theoretically such a layer would be problematic seeing how it should not likely survive in the archaeological record, but luckily archaeological examples of the highly calcareous parting layer exist.

To determine the original composition of the parting layer as best as possible we selected Sample GT 281 because of its exceptionally thick calcium-rich layer (Fig. 1). This sample is a glass-making crucible fragment that is heavily vitrified with corroded glass laying underneath the thick friable white coating. The powdery quality of the parting layer contrasts with the dense, highly vitrified ceramic. The superficial boundary and lack of interaction between the two materials imply that the coating was applied only after the ceramic was subjected to high heat. The analysis shows that the white layer is an extremely fine grained lime plaster with few haphazard inclusions of ceramic, quartz, corroded glass and a small fragment of material similar in composition to Egyptian blue. The survival of this material in such thickness on a crucible fragment is unusual, and is thought to represent an unfired or unused parting layer. One possible explanation is that this crucible sherd was used in the mixing or application of the wet plaster mixture. The thick parting layer on this sherd shows a high standard of integrity, and it is on



Fig. 1: GT 281 with an exceptionally thick coat of parting layer material near the rim.

the analysis of this plaster that the experimental parting layer is based. However, it cannot be ruled out that the GT 281 parting layer was used in glass melting in the past, and or may have a composition different from the usual parting layer when fresh. The composition of this layer was determined by bulk EDS analysis of five areas of 2 mm x 1.5 mm each. The average composition is about 72 wt% CaO, just under 20 wt% SiO₂, and about 10 wt% combined minor oxides, mostly magnesia, alumina, soda, iron oxide and titania. With this, it is quite comparable to the thin parting layers of used vessels and crucibles analysed by SCHOER/REHREN in this volume, chapter 3.3, but is overall significantly richer in calcium oxide. We believe that this discrepancy is at least in part due to the different use history of this particular sample compared to the more silica-rich ones, and this hypothesis of a de-calcification of the parting layer during use is among those to be tested by the experimental work reported here.

1.2 Materials, Compositions and Procedures

The primary questions this study was undertaken to explore are how the parting layer works in practice, how the glass and Nile silt crucibles react at various temperatures and durations, and how the addition of NaCl affects the system. Due to limited availability of Nile silt vessels for this study, in all, four different types of ceramic vessels were used to produce and to re-melt glass, though the Nile silt vessels were the main focus of the study.

¹⁰ SCHOER/REHREN this volume.

¹¹ REHREN 1997.



The vessels used

Previous work had identified that both the archaeological reaction vessels and the crucibles were made from a sandy Nile silt local to Qantir-Piramesses, equivalent to the Vienna System I.E.01.¹² It is this fabric which is showing distinct and diagnostic use traces during the glassmaking process, mostly as different zones of discolouration and vitrification of the ceramic underneath the parting layer. Our experiments aimed to replicate these traces as closely as possible under controlled conditions, in the hope to better understand the process parameters applied in the LBA glassmaking process. For this purpose, we had a total of nine open vessels available, made locally near Qantir in a fabric which strongly resembles Vienna system I.E.01. To determine whether the ash trays were sufficiently similar in composition, comparative SEM-EDS analyses were done on several off cuts from the modern vessels, and on two archaeological vessel fragments. Table 1 shows that they are indeed very similar in composition, although some differences exist between the two groups in their magnesia, sulphate, potash and lime contents.

	AT4	AT8	AT1	AT Av.	RG 080	RG 080	QCR2	Average
Na ₂ O	2.2	3.8	3.6	3.2	4.4	4.8	2.4	3.9
MgO	2.8	2.8	2.4	2.7	2.0	1.5	2.3	1.9
Al ₂ O ₃	14.5	15.0	15.2	14.9	15.3	14.2	13.0	14.2
SiO ₂	64.6	61.4	58.2	61.4	58.3	59.1	64.4	60.6
P ₂ O ₅	0.7	0.5	nd	0.4	0.8	1.1	nt	0.6
SO ₃	nd	0.4	nd	0.1	1.5	1.6	nt	1.0
K ₂ O	1.5	1.7	1.0	1.4	4.4	3.8	3.2	3.8
CaO	5.6	5.2	5.6	5.5	3.3	3.6	3.4	3.4
TiO ₂	1.4	1.7	2.2	1.8	1.4	1.2	1.4	1.4
FeO	6.5	7.5	10.7	8.2	7.8	8.5	9.8	8.7

Table 1: Semi-quantitative SEM-EDS area analyses of modern ash trays (AT) and archaeological ceramic vessels from Qantir. The data is normalised to 100 wt% to account for variable porosity and to enable better comparison.

The other vessels used for our experiments include small white mullite crucibles purpose-made for glassmaking experiments; a commercially available stoneware flower pot; and a hand-made cylindrical crucible produced specifically for these experiments using commercially available potters' clay. Unless otherwise stated, all vessels were fired with the same parting layer applied. Both the hand-made cylindrical crucible and the flower pot were used to produce additional quantities of raw glass for the re-melting experiments using ash trays, while the hand-made cylindrical crucible was made to replicate the characteristic shape of glass crucibles from Qantir-Piramesses while at the same time fitting into the existing electric kiln.

The glass charge

It was decided to use a typical LBA base-glass composition of 67% SiO₂, 20% Na₂O, 8% CaO, and 5% MgO prepared from finely crushed quartz and carbonates of the other three components. This charge has a theoretical melting temperature of about 950 °C. One factor we wanted to control was the presence of chloride, and some batches contained an additional 20 grams of common salt (NaCl) for every 100 grams of anticipated glass produced. To eliminate measurement error caused by humidity, the powdered ingredients were heated in a furnace at 100 °C for more than an hour prior to weighing. Over the course of the experiments, five batches of raw material were produced, all to the same recipe. Glass batch 1 was measured to produce 100 grams of glass while glass batches 2 through 5 were measured to produce 500 grams of glass. Glass batches 1, 2, and 5 contained 20 grams of NaCl per 100 grams of glass, and batches 3 and 4 contained none. Throughout the subsequent text, we will refer to glass batches and reaction vessel firing when talking about making glass from raw materials, and raw glass re-melting and crucible firing when pre-fired semi-finished

glass was further processed. However, in our experiments the same vessels were used as reaction vessels and crucibles, i.e. contrary to the archaeological evidence we did not use vessels of different shapes for the different processes. For the colouration of the final ingots we used commercially available copper oxide and manganese oxide, respectively.

The parting layer

Our parting layer recipe was based on the analysis of GT 281. Slaked lime, silica and finely crushed ash tray ceramic were mixed together in weight proportions of

¹² ASTON 1998.



75%, 8%, and 17% respectively. It is recognized that a plaster of this composition would have problems with shrinkage because of the low proportion of aggregates. The aggregates seem to be an unintentional contamination possibly from both the clay/silt in the water used to mix the plaster and industrial debris. There is the alternative that an additional organic aggregate, such as finely ground charcoal, was used that would not be identified on the SEM/EDS because carbon was not included in the analyses. SCHOER/REHREN in this volume report a grey variety of the parting layer, found predominantly at the low-temperature reaction vessels, and tentatively suggest that the grey colour is due to the presence of charcoal dust. However, the assumed unreacted parting layer analysed here is white, not grey, and microscopic investigation did not show any trace of charcoal particles. If the parting layer had been fired with the crucible then it may have been possible that there was a calcareous aggregate component that decomposed upon firing, making it indistinguishable from the matrix. For the sake of simplicity, the possibility of the use of an organic or calcareous aggregate was not further considered in these experiments.

The practicalities of applying a parting layer onto the ceramic have not previously been explored. When a slaked lime-rich plaster is put in contact with a dry porous ceramic, it instantly loses a considerable quantity of water, leaving behind a thick plaster coating. Brushing is time consuming, while pouring liquid slurry into the crucible and draining the excess seemed to promote cracking in the layer during drying.

Two ways have worked well experimentally in building up a thick parting layer. A combination of brushing and pouring, gradually building up the thickness of the parting layer with thin coats work well, but was also time consuming. The second method is to make a slurry with excessive amounts of water to produce a thick, but fluid coating and then sprinkling on dry powder to soak up the excess water. Both these methods achieve the end goal of a one millimetre thick or greater parting layer with a minimal problem with cracking. How best to apply the parting layer was discovered through trial and error over the course of the experiments, and not all the experiments yielded excellent results. The main flaws were caused by cracking of the plaster and it not being thick or durable enough to withstand the glass melts.

Charging and firing procedure

The crucibles and ash trays were individually coated with the parting layer as the experiments progressed. After coating and drying, the vessels were then filled with either glass batches or crushed raw glass. Since

the ash trays are small in volume and vary in size and shape, it was chosen that they should be filled so that the maximum surface area of the ceramic was exposed to the melting glass so that we could obtain the most information. For this reason, a standard amount of raw batch or glass was not used, and crucibles were filled to their capacity. In preparation for the melting experiments, the ash trays with a previously applied calcareous slip were sanded to reveal the red ceramic underneath. This was done to be better able to distinguish the ceramic surface from the parting layer. Ash Tray 6 was to be used without a parting layer and was left un-sanded while Ash Tray 3 had no such slip and was saved for the last experiment.

The exact firing procedure varied from melt to melt because this is one of the independent variables to be tested by these experiments, but several aspects of the firing procedure were standardized. The same electric furnace equipped with a thermocouple was used for all experiments. The vessels were inserted into the furnace at room temperature, and the temperature in the furnace increased at three degrees per minute until the maximum temperature for the particular experiment was reached. Then that temperature was either held for a measured length of time or allowed to cool immediately. The cooling, due to the well insulated furnace walls, was in the range of one degree per minute until the furnace was slightly cracked open to increase the cooling rate. The furnace was opened at around 400 °C and cooled within a few hours. Annealing the glass was not a priority in this set of experiments, although two ash trays of glass were allowed to reach room temperature with the furnace closed. In most cases, the crucible with glass was removed from the furnace still warm. To follow a proposed two stage system of glass production, raw glass was produced from batches of raw materials, crushed to homogenize and then re-melted, with or without added colorant, to form a glass ingot. The raw glass produced in the ash trays, for the most part, was not re-melted, instead additional raw glass fused in the large stoneware flower pot and the hand-made cylindrical vessel were used for re-melting.

In most cases, the used ash trays with their glass charge in place were then cut into half to view the interior, and to observe any changes to the ceramic in profile. Cutting was done using a water-lubricated diamond-impregnated circular saw. In two cases the fired vessels were filled with cold-setting resin prior to cutting to stabilise the assemblage of ceramic vessel, residual parting layer, and glass, but the majority of the vessels were cut without this precautionary measure.



2. Experiments

The first four ash trays (AT1, AT2, AT6 and AT8) were used to make a semi-finished or raw glass, and five were used to make coloured glass ingots of various qualities from raw glass. After two test firings of the batch used throughout these experiments, 925 °C for the duration of 8 hours yielded a glass that was not fully reacted. For ease of discussion, the ash trays fired to this regime from the raw ingredients will be referred to as 'reaction vessels'. The five other ash trays were used to re-melt raw glass at temperatures of 1000 °C and above, and will be called 'crucibles' to better differentiate the two steps in the production sequence.

2.1 Ash trays as reaction vessels

Ash Trays 1 and 2 were identically prepared and fired together in the furnace. The parting layer was applied to a thickness of 1-2 mm and then filled with salt-containing glass batch. After cooling, AT1 was sliced in half, but while cutting the raw glass ingot became dislodged from the friable parting layer. The water lubricating the saw washed away the remains of the parting layer, revealing that the ceramic on the interior surface of the reaction vessel showed a buff discolouring (Fig. 2). Little parting layer adhered to the reaction vessel, though in few places glass stuck to the ceramic. The separation of the glass melt from the ceramic was nearly complete (Fig. 3). To preserve the structure of AT2, the interior of the reaction vessel was filled with resin prior to cutting (Fig. 4). The profiles of the two vessels show that reactions occurred to the glass, parting layer and ceramic. Note the buff discoloured zone where the ceramic was in contact with the parting layer and the pinkish discoloration in the interior of the walls of the vessels (Fig. 5). These two features are frequently seen in archaeological samples from Qantir, and their successful replication in the first two firings was encouraging.

As a control, AT6 was used without a parting layer. It was filled with salt-containing glass batch and fired at the same temperature as before. The glass in this case was securely fused to the ceramic. However, the semi-finished glass formed bubbles near the ceramic making it easy to break the glass out if wanted, but despite this the reaction vessel remains to have a coating of glass and there is much glass firmly adhering to the ceramic which is difficult to manually separate. The same pinkish discoloration as seen in the previous examples occurred in the walls of the reaction vessel, but there is no evidence of a buff discoloured zone into the ceramic body.

AT8 finally was prepared with a parting layer to a thickness of 1-2 mm, although this time a salt-free

glass batch was used (Fig. 6). The profile after firing seen in Figure 6 is starkly different from the previous examples. Immediately behind the parting layer, where the buff discoloured layer of ceramic should be, there is a zone of more vitrified ceramic. No pinkish discoloration of the ceramic occurs (Fig. 7). Visually and chemically the reaction zone of this vessel is dissimilar to the first two, although all three vessels had the same parting layer applied to them.

A more detailed discussion of the interpretation of these observations will be pursued later in the paper. However, it is reasonable already now to attribute the characteristic discoloration zones to the joint presence of sodium chloride salt in the glass batch, and a calcareous parting layer. The highly reactive sodium carbonate present in all four melts as the main soda source apparently does not on its own lead to the formation of either the buff or the pinkish zones in the underlying ceramic. Both samples with a parting layer and salt in the batch developed a buff discoloration zone in the ceramic near the parting layer, while all three melts with salt present in the batch developed the pinkish discoloration of the ceramic. The sample with a parting layer but no salt in the batch did not show the buff discoloration. In summary, these first four melts showed that the parting layer is both effective and necessary to prevent contamination of the glass and facilitate removal of the raw glass. Furthermore, it appears that the characteristic double discoloration, buff and pinkish, of the ceramic only develop in contact with the parting layer and in the presence of common salt in the glass batch.

2.2 Larger reaction vessels

Because the finished fully melted glass is much denser than the raw ingredients, for the next stage of experimentation, large quantities of raw materials had to be fused for re-melting to allow complete filling of the crucibles. A single 2-liter stoneware flower pot was successfully used three times to produce raw glass. This vessel was lined with parting layer and the hole in the bottom plugged with parting layer and crushed ceramic. In every case the raw glass ingot was removed intact without damaging the reaction vessel. Different quantities of raw ingredients were fused each firing, but using the same batch formulation and with a standardized firing procedure of 925 °C for 8 hours. Interestingly, the quality/density of the glass appears to vary depending on the mass of the charge. In the last firing using this vessel, it was filled with 675 g of batch material to produce 500 g of raw glass, and in the centre there was an un-reacted core, whereas the first firing to make 300 g of raw glass produced a



Fig. 2: AT1 with residual parting layer and buff and pink discoloration zones in the ceramic.



Fig. 3: The semi-finished glass cake from AT1, with buff-yellow parting layer material adhering.



Fig. 4: AT2 with residual parting layer and buff and pink discoloration zones in the ceramic, consolidated with resin.

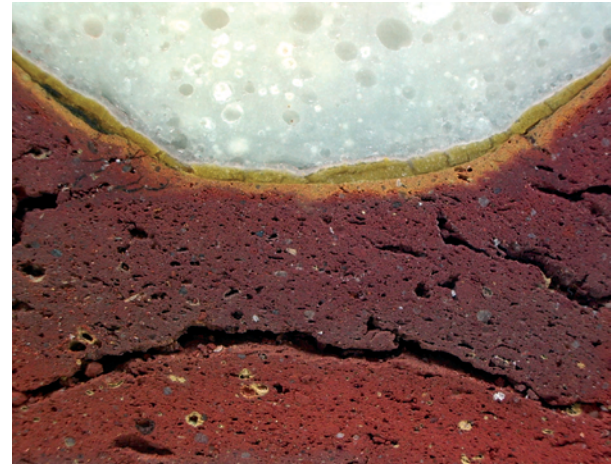


Fig. 5: Close-up of the reaction zone at the bottom of AT2, showing a green to buff zone between the glass and the ceramic, and the pink discoloration of the ceramic..

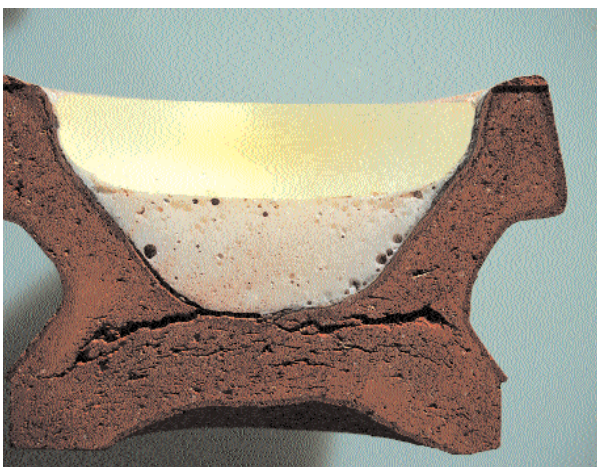


Fig. 6: AT8 consolidated with resin. No discoloration of the ceramic occurred despite the same firings conditions being used as for AT1 and 2. AT8 did not contain salt in its batch.



Fig. 7: Close-up of the reaction zone at the bottom of AT8, showing only a faint buff zone between the glass and the ceramic, and almost no discoloration.



much more dense and evenly reacted raw glass. Though it is possible that there was a variability in charge homogenization, clearly, volume and vessel shape are other important factors to be considered because of the heat gradient from the externally-heated vessels into the centre of the charge; however, these were outside the remit of this study.

Eventually, a hand-made cylindrical vessel was used to produce about 400 g of dense raw glass from a batch containing 20 g salt per 100 g of ready glass. This vessel was specifically made to have its shape similar to the crucibles found archaeologically at Qantir. After firing, the ingot of raw glass was removed by flipping the vessel and tapping until the ingot of raw glass simply fell out. Almost no glass adhered to the vessel and the vessel was undamaged in the procedure. The vessel was then broken open for a better view of the interior. In this case, the ceramic of this vessel reacted differently to the Nile silt. One possible reason for this is that it is not a calcareous fabric. The effect of salt penetrating into the ceramic is easily visible in a clean cut revealing two tide marks: one at the level of the original fill, one at the level of the fused raw glass. The undercut throwing marks on the interior possessed no problems because the parting layer was thick enough to even them. This vessel shows that reaction vessels can be re-used repeatedly, provided a sufficient parting layer is present, due to the ease of glass separation and the low level of vitrification and cracking of the ceramic occurring at these temperatures (Fig. 8).

2.3 Ash trays for re-melting

The other five ash trays (AT3, AT4, AT5, AT7 and AT9) were used in the re-melting of raw glass to form coloured glass ingots. The raw glass was crushed with a mallet generally to a grain size smaller than 5 mm. Contrary to what had been suggested earlier, the glass was not washed at this stage because components such as NaCl, an important variable in the experiments, would be lost, and here, we wanted to test whether enough salt would be carried through the two-step process to produce similar discolorations as in the initial firings.¹³ Another main objective at this stage was to trace the amount of discoloration in the ceramic when a salt-free charge is being melted, in order to see whether the archaeologically observed discoloration pattern are indeed characteristic for glassmaking, or also occur in re-melting glass. Finally, we wanted to test how much, if any, of the parting layer is being absorbed during the re-melting at higher temperatures, testing this aspect of the partial melting model put forward earlier.¹⁴

Each of these ash tray firings was unique, exploring a different array of variables and conditions; there were not enough Nile silt ash trays available to do repeat experiments or single variable variation. The complexity of producing a perfectly separated, fully fused and well coloured glass is seen in the experimental replications. Thus, through trial and error and with varying degrees of success, in the experiments a range of temperatures, durations, and parting layer thicknesses were used to produce glass, with and without salt in the glass charge.

AT7, re-melting salt-containing glass

Ash Tray 7 was the first of the crucible firings. The crucible was prepared like AT1 and 2 with a parting layer of 1-2 mm. The crucible was filled to the rim with finely crushed salt-containing raw glass with an addition of 2% by weight of Cu_2O . The charge was heated at 1050 °C for 8 hours and resulted in a perfectly fused, deep blue transparent glass (Fig. 9). The glass overflowed the rim of the crucible and dripped down one side (Fig. 10). It was of very good quality, but the parting layer did not withstand the firing, resulting in a fusion of glass and ceramic. The first millimetre of the glass at the contact to the ceramic shows more of a greenish tinge, indicating a contamination from the ceramic. The differential shrinkage caused a crack to develop between the glass and ceramic allowing the glass to be removed, but a layer of red ceramic stuck to the glass (Fig. 11). The adhering ceramic is difficult to remove and this cannot be ideal because it should be removed before re-melting to prevent further contamination. Although the initial batch used to make the raw glass for this melt contained 20 wt% salt, no buff discoloured zone or pink discoloration of the crucible ceramic is apparent after the re-melting.

Two observations appear particularly relevant here; first, the higher temperature of the re-melting compared to the initial melting went together with an almost complete absorption of the parting layer, and the level of salt present in the raw glass was not enough to produce the characteristic discoloration zone in the underlying ceramic.

AT4, salt in parting layer, shorter firing

To see how the system would react with a shorter duration/higher temperature, Ash Tray 4 was used. It is very likely now that NaCl is responsible for the discolorations that occur in the crucible fragments from Qantir. A further objective of this experiment was to explore whether different methods of introducing salt into the crucible, other than as part of the flux (assumed plant ash in the LBA), would

¹³ REHREN/PUSCH 2005, 1757.

¹⁴ REHREN 2000 a, b.



Fig. 8: A hand-made cylindrical vessel after firing a salt-containing batch to produce semi-finished glass. The vessel was deliberately broken to reveal the discoloration of the ceramic.

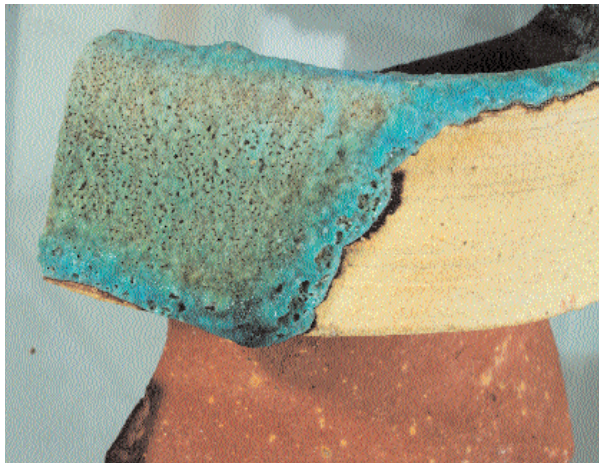


Fig. 10: Glass overflowing the rim of AT7.

result in the same discoloration. Thus, the parting layer plaster was mixed with a saline solution of 3.5 g NaCl per 100 g of water to imitate slaking the quicklime with sea water. A funnel was added to the rim of the crucible to prevent overflow and to allow much more crushed glass into the crucible. After drying, crushed raw glass made from a salt free batch was charged with the addition of MnO and Cu₂O to produce a blackish-purple glass. This crucible was then fired to 1100 °C for 2 hours and was allowed to slowly cool in the furnace. The resulting glass was fully fused, transparent (where thin) and, despite the slow cooling, was not properly annealed causing it to fracture during cutting (Fig. 12).

For the most part, the parting layer did not withstand this firing either, though here it is not as clear cut as the previous firing. The chloride, in some areas, discoloured the ceramic but did not penetrate far into the interior. Immediately behind the buff discoloured ceramic is a region of more heavily

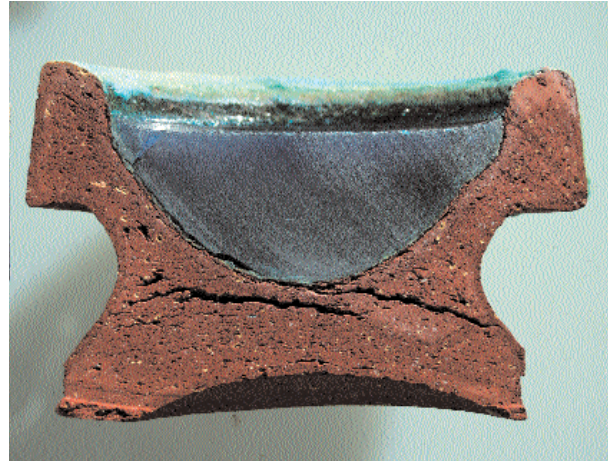


Fig. 9: AT7. Note the complete absorption of the parting layer, and the absence of any discoloration of the ceramic.

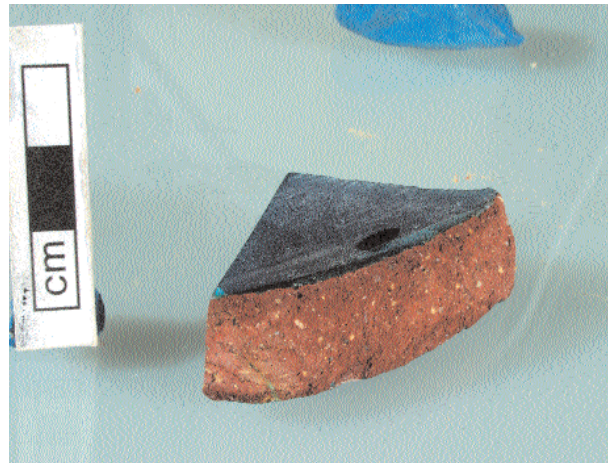


Fig. 11: Part of the ingot removed from AT7 with ceramic material firmly attached to the glass.

vitrified ceramic. Where the parting layer was, is now a devitrified opaque layer bonded to the vitrified glass. The devitrified layer is quite brittle and glass was fairly easily removed from the ceramic despite the almost complete absorption of the parting layer. It preferentially broke along the devitrified layer, some sticking to the glass some sticking to the crucible (Fig. 13). The subsequent glass is suitable for crushing and re-melting, as no ceramic material stuck to the glass.

AT5, very short firing time, no salt

AT5 was used to test an even further shortening of the firing duration. The parting layer was applied as normal, without added salt. Crushed salt-free raw glass along with a few fragments of dark purple glass from the previous melt were placed in the crucible and melted at 1100 °C with no soaking time. The glass was allowed to cool after the furnace reached the peak temperature. This time the parting layer



Fig. 12: AT4 with some surviving parting layer visible.



Fig. 13: The glass separated badly from AT4, with some glass still attached to the ceramic.

held up better. The glass was removed from the crucible with minimal effort, though much of the parting layer was gone, and where there were cracks in the parting layer, glass leaked through and stuck to the crucible (Fig. 14). The glass, however, is not homogeneous due to incomplete crushing and mixing. The cullet used in this melt was of up to 1 cm in size. Areas high in the purple glass became vitreous while there are some areas that are not fully reacted from the raw glass. On the bottom surface of the glass, a thin rough white parting layer residue is present (Fig. 15). Areas where this is absent correspond to the glass being stuck to the surface of the crucible. There is no discoloration in the crucible, yet there is a deep red zone of more vitrified ceramic bordering the parting layer.

AT9, low temperature, short firing, and added salt

This experiment was designed to test the lower limits of temperature and duration of firing. AT 9 was filled with finely crushed raw glass with the addition of 1 wt% Cu_2O and 20 g of NaCl for every 100 g of raw glass. The charge was fired at 1000 °C for 4 hours and fused to form a frothy, opaque blue glass. Specks of white, un-reacted material dot the glass. Despite the low temperature and short firing time, the parting layer failed in many areas causing glass to adhere to the ceramic. For the first time in the re-melting experiments, this firing resulted in similar discoloration zones developing in the crucible as AT1 and 2, with a buff zone next to the parting layer and internal pinkish/purple discoloration of the ceramic. This observation underlines the causal relationship between a significant salt content of the charge, and the development of the buff and pink discoloration zones in the ceramic (Fig. 16). The temperature and duration of the firing were not sufficient to produce a well fused glass ingot, suggesting that glass of this composition needs temperatures in excess of 1000 °C to form fully reacted ingots.

AT3, extra-thick parting layer

The final glass melt was to test whether an excessively thick parting layer would, in fact, halt the procession of the molten glass into direct contact with the ceramic. Over the course of a week, a parting layer 4-7 mm thick was applied to AT3. Finely crushed salt containing raw glass with the addition of 1 wt% Cu_2O was held at 1025 °C for 10 hours. In this case, the parting layer withstood, losing 1-2 mm of its original thickness (Fig. 17). The glass was easily removed from the powdery parting layer without any damage to the crucible (Fig. 18). The crucible actually showed little change as a result to the melting. The residual salt from the raw glass caused only a superficial bleaching in some areas, but mostly had no effect. A region of denser ceramic is adjacent to the parting layer, which is similar to AT8, the reaction vessel used to produce salt-free raw glass. Although the parting layer was powdery and flaky after removal from the furnace, after a week's time the crust has become cement and would most likely survive archaeologically (Fig. 19).

So far, this may seem like an ideal replication success, but the glass does not appear to be comparable. It was fully fused and fully separated with a rough white crust where in contact with the parting layer, but the glass itself was almost totally devitrified and opaque. Further analysis is necessary on this subject matter to know more precisely what the cause of this devitrification is, but it is thought to be a result of a super-saturation of calcium in the melt and slow cooling rates. Such a devitrification is also seen to begin forming in the crucible AT4, which was allowed to cool very slowly after a two-hour soaking time, and the blue glass from the mullite test crucible.



Fig. 14: AT5 with some glass still attached to the ceramic.

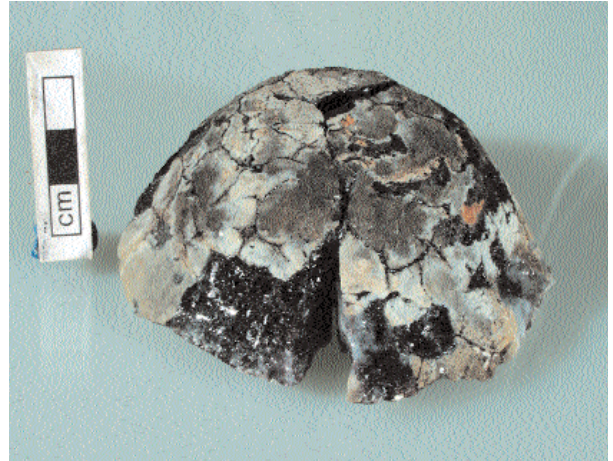


Fig. 15: The glass ingot produced in AT5, with residual parting layer material attached to the glass.

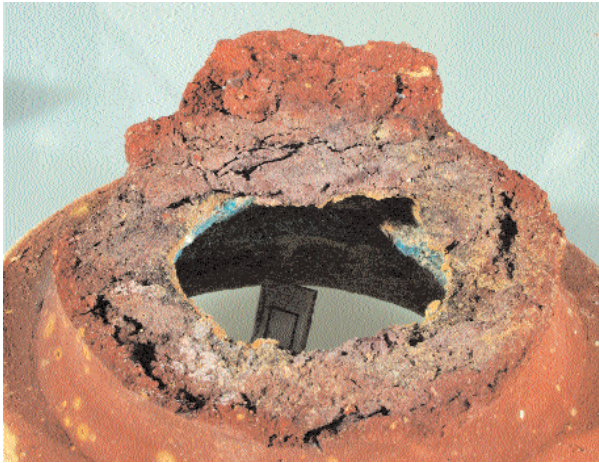


Fig. 16: AT9 broken open at the bottom to expose the discoloration caused by the salt content of the charge.



Fig. 17: AT3, fired with extra-thick parting layer, some of which is well preserved.



Fig. 18: The ingot from AT3, with residual parting layer material attached.

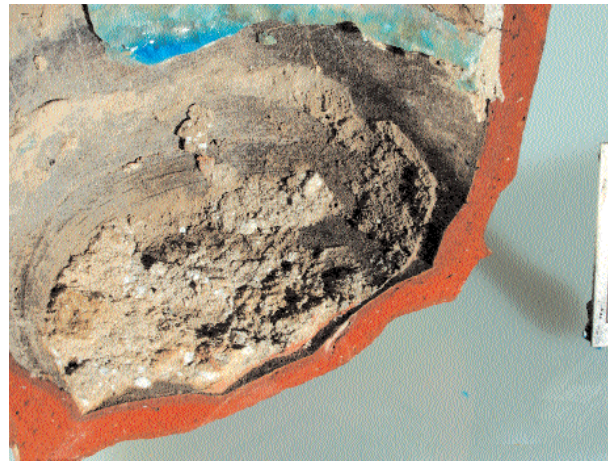


Fig. 19: Residual parting layer material at the bottom of AT3 after removing the ingot. Note the glass patches visible at the top of the image.



Fig. 20: Modern Nile silt ceramic, as received (left) and re-fired at 900 °C (right).



Fig. 21: Modern Nile silt ceramic, re-fired at 950 °C (right) and 1000 °C (right).



Fig. 22: Modern Nile silt ceramic, re-fired at 1050 °C (right) and 1100 °C (right).



Fig. 23: Modern Nile silt ceramic, re-fired at 1150 °C (right) and 1200 °C (right).

2.4 Ceramic colour thermometry

A series of test firings of Nile silt ceramic was done to illustrate and ideally quantify the effects of different firing temperatures on the colour of the ceramic.¹⁵ Freshly cut slices taken from a single piece of modern Nile silt pottery were each fired for six hours. The kiln used was the same as the one used in our glassmaking experiments, operating at ambient atmosphere, that is under oxidising conditions. The test firings were set at temperature intervals of 50 °C, starting at 900 °C and going up to 1200 °C. An original, that is not re-fired, slice was kept for reference and comparison. The slightly darker carbon core of the original ceramic visible in Fig. 20 disappears during re-firing; however, no other colour changes are apparent up to about 1050 °C (Fig. 20, 21), when the ceramic turns a slightly darker red (Fig. 22). This

colour change, however, is only recognizable in direct comparison to the reference fragment and is within the normal colour variability to be expected among different Nile silt vessels. More significant colour changes occur only at higher temperatures, from a strongly developed darkening of the fabric at 1100 °C (Fig. 22), turning almost black at 1150 °C, to a complete breakdown of the ceramic structure and the formation of a fused black mass at 1200 °C (Fig. 23). When broken, this revealed significant bloating of the fused material, equivalent to the archaeologically observed features of collapsed vessels.

¹⁵ We are grateful for M. OWNBY for conducting this set of experiments.



3. Discussion

It is apparent from this experimental study that the calcium-rich parting layer is not an inert material that blocks all interaction between the vessels' charge, ceramic and itself. Rather, the parting layer reacts with and is absorbed by both the ceramic and glass under specific conditions. In particular, it permits the interaction and transfer of certain elements and compounds such as soda, chlorine and calcium. The presence of common salt in the glass batch is a major factor in the final appearance of the ceramic, whereas temperature/duration of firing are the most important factors concerning glass quality and appearance. In all cases, these factors produce a distinctive layered structure of different zones which may help in the interpretation of the archaeological remains.

3.1 Layered zones

To augment visual analysis of the layers, three of the experimental samples were set into resin and polished for SEM/EDS analysis. Sections from reaction vessels AT2 and AT8, and from crucible AT4 were thought to provide the most fruitful comparison to the archaeological samples because of their visual similarities. Series of area analyses at measured intervals attempt to characterize the chemical compositions of the different reaction zones. These analyses are preliminary, as the work is still ongoing at the time of writing this, and only qualitative summary information of these is given as a general impression of what is happening. The variability in the compositions of these zones has not been assessed systematically yet and there may be a risk of some over-interpretation of these figures.

The buff discoloured ceramic and the parting layer

The buff zone has been provisionally identified in the past as being residual parting layer material.¹⁶ More recently it was thought to be a layer in the ceramic that was depleted in iron oxide, the latter carried away as iron chloride as sodium chloride penetrated through the parting layer and ceramic and then re-deposited further inside the ceramic, producing an iron-enriched zone.¹⁷ SEM/EDS analysis shows that this layer is characterized by high silica and lime with alumina and iron oxide, and is distinctly ceramic in nature rather than parting layer material. The ash tray experiments served to investigate the relationship between the presence of common salt in the charge, and the development of the buff zone in the ceramic underneath the parting layer.



Fig. 24: Inside of AT1 after the firing and removal of the glass cake. Note the discoloration of the ceramic, and the patch of glass adhering to the ceramic (centre right).

The reaction vessels AT1, AT2, and AT9 were greatly impacted by the presence of chloride in the charge (Fig. 24). Optical microscopy reveals six main layers and zones: glass, parting layer/interaction zone, buff discoloured ceramic, deep red/melted ceramic, pinkish ceramic, and unchanged ceramic. The most notable compositional trend across this sequence is that of lime. It is apparent from the analyses of vessels with added salt in the charge that lime is more mobile in the presence of chloride, moving easily into the ceramic and glass. EDS analysis from AT1 and 2 shows that the first millimetre of ceramic adjacent to the parting layer absorbs 2 to 4 times the amount of lime present further in the interior.

Crucible AT4 had salt added in the parting layer which in some areas produced a similar visual effect. This zone, however, is rather thin and the glass, having absorbed the parting layer, is in most parts in direct contact with the ceramic. The preservation of the buff discoloured zone is better on the bottom of the crucible. It is unknown how thick the buff zone would be if the glass charge had not been in direct contact with the ceramic. Behind the buff zone is a zone of more heavily vitrified ceramic. Here, the ceramic is fluxed with Na_2O and, with its lower lime levels, began to melt. The composition of the fluxed ceramic is similar to that of the bottle-green or interface glass,¹⁸ and it can be concluded that in AT4 we see the early stages of interface glass formation.

As a direct contrast to the buff zones of the previous examples, AT8 shows no sign of the formation of a calcium-rich layer in the ceramic. This reaction vessel had a normal parting layer applied to it, but no salt in the glass batch or in the parting layer. As a result, more lime stayed in the parting layer, and the movement of lime into the glass/ceramic played

¹⁶ SCHOER/REHREN in this volume, chapter 3.3 (Parting layers).

¹⁷ REHREN/PUSCH 2007.

¹⁸ SCHOER/REHREN in this volume, chapter 3.2 (Interface glass).



little or no role. What this does show is that lime appears to be less mobile when there is no salt present, with no substantial increases in either the glass or the ceramic in contact with the parting layer, while a greater amount of lime is preserved in the parting layer.

The contrast between salt-rich and salt-free charges in terms of colour changes to the ceramic is compounded by the formation of calcium-iron silicates in the ceramic. This is encouraged by the movement of lime and salt into the ceramic, making the colour change from red to buff. At high temperatures, NaCl is known to accelerate the decomposition of calcium carbonate into lime, to encourage the migration of lime in calcareous clays, and to encourage the formation of silicate phases that incorporate iron oxide at the expense of free iron oxide, the main source of the red colour in ferruginous ceramics. These properties have been exploited in the past to produce buff ceramic surfaces for decoration,¹⁹ although in the present case, the altered zone appears to be a by-product rather than exploited for intentional reasons.

The thickness of the buff zone seems to be a function of three variables: the shape of the vessel and the location of sampling, the quantity of salt in the system, and the durability of the parting layer. NaCl melts at around 800 °C but is not incorporated into the silicate melt system; it creates a separate and much less viscous melt that is not contained by the parting layer. Therefore, by gravity and capillary action, as the liquid salt drains through the porous parting layer and into the ceramic, there is a greater effect near the base of the vessel than on the vertical walls. This has been observed both in the ash tray experiments and on archaeological samples. AT4 with its relatively small amount of salt present developed only a thin zone of buff discoloration, further indicating that the quantity of salt present controls the degree of discoloration. Thus, the stronger discoloration nearer the bottom indicates that more salt reaches the bottom of the vessel than the sides, suggesting that the active agent was present as a fluid rather than a vapour.

Since the glass slowly absorbs the parting layer, if this is not thick enough, the glass will eventually come in contact with the buff ceramic and begin to absorb it. It is difficult to determine the original thickness of a buff zone if it is in contact with the glass because it is unknown how far the melt proceeded into the ceramic, though there may be some indirect evidence through compositional analysis of the glass. From behind the buff zone, soda leaking through causes the formation of the so-called interface glass. Therefore glass is eroding away at the buff zone from either side, making reliable thickness determination difficult.



Fig. 25: Cross section through the cylindrical vessel (see Fig. 9) with 'tide-marks', visible in the cut.

Deep red zone and the formation of bottle-green interface glass

In all of the experiments, none exhibited a comparable thickness and quality of the interface glass of many of the archaeological samples. Due to the present sampling procedures, it is unknown how representative these samples are, but clearly some samples show a green glass layer up to 1 mm thick between the parting layer/buff ceramic and the crucible wall.

It is not immediately clear whether the Na₂O flux in the interface glass comes from soda ash in the charge or if it is released from sodium chloride reacting with the ceramic material, but it is apparent from both the archaeological specimens²⁰ and the experimental data presented here that some sodium compound is passing through the parting layer and the buff ceramic zone to react with the ceramic underneath.

At low temperatures the soda appears less aggressive and less mobile causing only a slight increase in the vitrification of the ceramic behind the buff zone. This more vitrified zone seen in the experimental samples is of a deep red colour. In our experiments, AT4 was subjected to the most extreme heat, at 1100 °C for two hours. It has the most

¹⁹ MOLERA et al. 1998, 189. HUDSON 1997, 136.

²⁰ SCHOER/REHREN in this volume, chapter 3.3 (Parting layer).

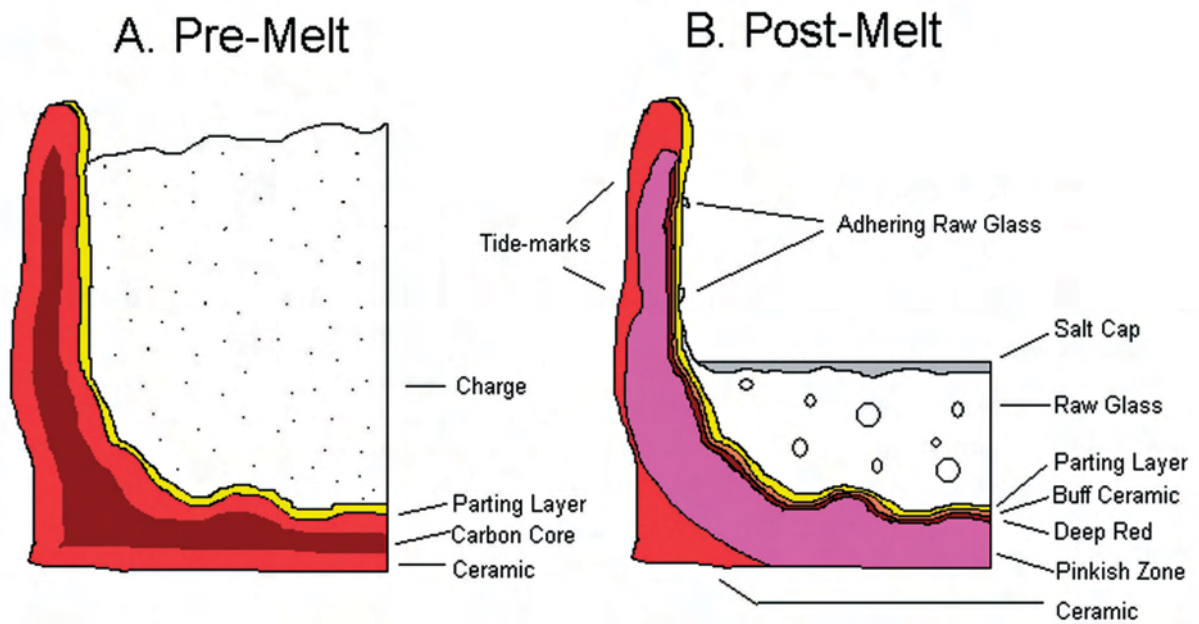


Fig. 26: Schematic of the colour changes in the ceramic caused by the firing of a salt-rich charge, and the volume change from batch (left) to raw glass (right).

pronounced vitrification in the deep red zone, but still does not come close to the thick layer of interface glass seen archaeologically. This may be due to one or more of several factors; AT4 had salt only in the parting layer to simulate lime slaking with sea water, but not in the glass batch. Furthermore, the quantities being melted in the ash trays are only about a tenth of the amount melted in the crucibles at Qantir-Piramesses, and the greater volume of the charge would exude larger amounts of soda into the ceramic. Another possibility is that the interface glass builds up after successive use. It is seen from the experiments that when the parting layer is successful, the ingot is removed from the vessel without damaging either. The quality of the ceramic after one glass melt is still excellent and does not show the levels of vitrification found archaeologically. None of the glass melting vessels used in the experiments appears to have come close to their structural limits; to the contrary they seem able to withstand several melts. Further experiments would be necessary to more fully explore the relative significance of these factors, amount of salt in the charge, glass volume, and repeated use of crucibles.

Pinkish zone

The thick pinkish zone seen in the archaeological vessels and the ash trays is less understood because no work has been done yet to characterize chemically or

mineralogically what is happening. The reaction vessels AT1 and 2 and crucible AT9 all contained an added 20 g of salt per 100 g of glass and showed a considerable effect, but AT4 which had salt only in the parting layer shows no signs of a deep penetration of salt into the body, and the others, which had no or only residual salt in their charge, showed no effect. Like the buff discoloured zone, its formation appears dependent on the presence of large amounts of chloride in the charge. The sodium chloride possibly causes some phase changes that alter the macroscopic properties of the ceramic.²¹ Little understood as it is, its existence may give a considerable amount of information concerning the quantification of the charge. Since it is fairly certain that the melt systems used in Qantir contained chloride due to the frequent occurrence of both discoloured zones archaeologically, conclusions based on visual analysis would seem valid.

The formation of the pinkish zone seems to occur in two stages: one marking the initial level of the batch in the vessel as the salt is leaving the powdered charge and the second the melted level where excess salt separates to form a liquid cap on top of the glass melt which possibly flows into the ceramic. Even in the dense mullite crucible, tide-marks of discoloration can be seen on the exterior of the vessel which mark the initial fill level and the melted glass level. It is perhaps easiest to see in the profile of the large earthenware vessel despite the dissimilarity of colour to the archaeological samples (Fig. 25 and 26). A clean-cut profile of the vessel shows both tide-marks clearly, but it is quite difficult to see from a rough break.

²¹ BAYLEY 1991 has reported a similar pink discoloration for vessels used to separate gold from silver using common salt, and attributed it to a mobilisation of iron oxide and its re-deposition as fine grained haematite.



If the glassmaking vessels from Qantir were subjected to profiling in this way, perhaps it would give insight into the volume of the initial/final batch. Since there are usually two tide-marks in the experimental reaction vessels, two tide-marks in the archaeological vessels would show that the vessel was placed in the furnace with the charge and fused without the need of accessibility for repeated charging. If the vessels were filled successively during the melting process, as a funnel top might suggest, then there should not be a two-tide profile, but something more complex. If the funnel top, on the other hand, was simply used to provide more room for fill, then the vessel may have only one developed tide-mark near the top of the vessel. There is need still for speculation here because this has not been studied in any detail, but research into this area shows great potential.

The significance of salt

How chloride entered the melt system is a question of paramount importance in the argument for glassmaking in Egypt. It is shown that the presence of common salt provides little technical advantage in the glass-making process, and therefore it seems unlikely to be a deliberate addition. However, cursory analysis of the crucible and reaction vessel fragments from Qantir-Piramesses gives the impression that colour changes in the ceramic which are consistent with the presence of significant quantities of salt in the charge are widespread, and that the chloride comes from either the glass charge itself or from the parting layer. Both cases will be explored.

Chloride from the glass charge would likely come with the flux. BRILL has listed chemical analysis on many types of plant ashes and natron suitable for glassmaking.²² The compositions vary considerably with some high in soda and others in potassium. Chloride is another very variable component, which generally ranges between 2 to 20 wt% in plant ashes. The natron samples analyzed also show large quantities of chloride, so the presence of salt-induced discolorations cannot be used to argue what kind of flux was used. As mentioned earlier, much of the chloride leaves the glass melt at 800 °C when it melts, but only large quantities leave a noticeable effect on the ceramic. If the chloride is coming from the flux then the first time the raw ingredients are heated above 800 °C, it would start to drain out from the charge well before the silicate system starts to melt. It is unclear at present how the sodium carbonate component of the batch behaves at this temperature, and whether some of it joins the sodium chloride melt. However, the experimental results presented here strongly indicate that the discoloration effect is limited to those charges which have a substantial salt

component, either in the raw batch or added to the crushed glass. Thus, sodium carbonate seems not to be a required component for the discoloration to form, while common salt is. The low viscosity and low melting temperature of common salt and the porosity of the parting layer combine to allow the latter to act almost as blotting paper, soaking up the liquid sodium chloride and channelling it away from the charge and into the underlying porous ceramic. Assuming that in practice no salt would be added to the re-melting of semi-finished glass (as we did in the crucible experiment AT9), a two staged system could therefore be differentiated: the raw glass melting with the presence of chloride-induced discolorations, and the re-melting of the semi-finished glass without. The argument that there could be enough residual salt in the semi-finished glass to still have an impact on the re-melting crucible is frustrated by the fact that in the experiments this residual salt component had little if any effect. Observing the degree of discoloration present in the archaeological fragments thus potentially offers a detailed insight into the nature of the charge processed in these vessels.

3.2 Temperature effects

The colour of the ceramic is not only affected by the chemically reactions with the charge, but also by the firing temperature. This relationship, of course, is not new but routinely used to estimate firing temperatures of ancient pottery. REHREN/PUSCH used it to identify systematic differences in firing temperature between reaction vessels and crucibles, using a rather qualitative system of 'cold', 'warm', 'hot' and 'very hot'. The temperature label 'hot' was used for samples which showed a considerable darkening of the ceramic, and 'very hot' for almost black ceramic up to the stage of bloating and complete fusion as seen in the hot spots. The associated temperatures were tentatively estimated to cover 900 °C to 1050 °C for 'hot' and accordingly higher for 'very hot'.²³ Our experiments here have shown that a significant darkening of the ceramic only occurs at re-firing at more than 1050 °C, and its fusion and collapse below 1200 °C. The limit of stability for a crucible probably is 1150 °C due to gravity and substantial shrinkage causing cracks to develop, as seen in the re-fired test piece.

We matched the test-fired pieces against the fired ash trays, and found a very good agreement in colour shades. However, one has to be cautious in using the

²² BRILL 1999, 480-486.

²³ REHREN/PUSCH 2005.

colours of the test-fired pieces as a thermometer for the archaeological crucibles and reaction vessels. Our analysis of the modern Nile silt ceramics has identified some compositional differences, primarily in their alkali and earth alkali content (see above, Table 1). In particular the soda and potash concentrations in the modern vessels were lower by a few percent, while the magnesia and lime concentrations were only slightly higher. Alkali oxides have a much stronger fluxing effect on ceramics than earth alkali oxides; thus, the net effect of the compositional differences between the modern and archaeological samples will result in a more ready vitrification of the archaeological ceramics. More experimental work is necessary to quantify how much of a temperature shift results from two percent by weight more total alkali oxide content in Nile silt ceramic. Similarly, we are at present not in a position to assess the combined effect of chemical and temperature attack on the colour of the ceramic, nor do we know whether the kiln atmosphere was always kept oxidising. Thus, it is reasonable to assume that the experimentally established 'colour thermometer' is too high in its absolute values, due to the different alkali oxide contents of the different ceramics; but we can not say by how much.

3.3 Conclusions and further work

The work reported here has very clearly demonstrated that the parting layer is not a passive, inert barrier between the charge of the glassmaking vessels and their ceramic; to the contrary, a significant amount of interaction occurs between both the parting layer and the glass charge, and between the parting layer and the underlying ceramic. The former consists of a degree of absorption of parting layer material into the glass melt, necessarily increasing the lime content of the glass and possibly influencing its devitrification behaviour. It is planned to investigate the glass samples produced in these experiments through electron microprobe analysis to quantify the effect this has on the lime content; already now the noted absorption of the parting layer into the glass is strong support for the validity of the partial melting model and temperature dependency of the glass composition put forward earlier.²⁴ It also has major significance for the interpretation of the lime and magnesia content of archaeological glass samples. Not only are both likely to reflect only in part the original plant ash composition, but are affected by the interaction with



Fig. 27: Cross section through a reaction vessel from Qantir-Piramesses, showing a tide mark developed in the pink discoloration zone. We assume that this indicates the level to which the vessel was filled when fired. FZN 00/0617a,0119.

the parting layer and the formation of crystalline phases such as wollastonite and diopside at the contact zone between the two. Also, the proportion of lime coming from the parting layer is likely to have a different strontium content and strontium isotopic ratio than the proportion coming from the plant ash. Interpreting strontium concentration and isotopic ratios in LBA glasses will have to take this mixing effect into account, and it is important to quantify the extent this mixing can have. Our analytical work on these experiments will be published separately in due course.

The implications for the practicalities of glass-making and its identification in the archaeological record brought to light by the experimental work are also relatively complex and could change several aspects of our understanding of Ramesside glass production presented in this volume. These experiments raise so many questions and possibilities that a comprehensive re-examination of the archaeological material is absolutely necessary.

It has been mentioned earlier that the archaeological samples chosen as basis for this research were selected for having particular strongly developed features related to use. Thus, they are not representative of the entire assemblage of reaction vessel and crucible fragments found at Qantir-Piramesses. Based on the evidence from these archaeological fragments available at the time of writing this contribution, not only reaction vessel but crucible fragments also show the dramatic effects of

²⁴ REHREN 2000a, b; SHUGAR/REHREN 2002.



chloride discoloration. So far, crucibles have been seen almost exclusively as related to re-melting and colouring glass, while the glassmaking was thought to be restricted to the reaction vessels. The observation of strong discolorations, and their dependency on a high salt content of the charge, could have far-reaching consequences for our interpretation of the function of these crucibles. If tide-marks or strong buff and pink discolorations are to be found on essentially all archaeological crucible fragments, then it would provide a strong argument against the two-stage, two-vessel system, showing that the crucibles were routinely used in making glass from the raw ingredients as well as making coloured glass ingots. If, however, only some of the crucibles show significant discoloration zones, and others not, then one could consider a way of making glass by which some crucibles were used for glassmaking and others for re-melting and colouring the semi-finished glass. The archaeological presentation given above reports that about equal numbers of crucible fragments have identifiable remains of semi-finished glass and red glass, respectively.²⁵ Thus, future investigation will have to test whether those with semi-finished glass have a stronger discoloration of their ceramic compared to those with remains of red glass. In this case, half of the crucibles would have been used for glassmaking, and the other half for glass colouring.

The possibility that crucibles could have been used several times could complicate matters. Layers produced through several uses could be difficult to interpret, but it would seem that if semi-finished glass was re-melted in the crucibles, even repeated use would probably not provide the chloride necessary to produce the effects documented in many crucible fragments.

The reaction vessels on the other hand generally have thin walls and the effects of chloride may show through the entire thickness of the wall. This would make the discoloration less noticeable as it would provide little contrast within the fragment, and go unnoticed within the overall variability of fabric colours. One thing to look for when analyzing the reaction vessels is the formation of a calcareous film on the exterior of the vessel. Common salt is known to cause the formation of lime on the surface of vessel due to a mobilization of lime during firing.²⁶ It is likely that any free lime present in the ceramic was leached out in this process, theoretically leaving behind a layer of lime on the outer surface. The homogeneity of the reaction vessels is not yet known, and it is nearly impossible at this stage to hypothesize about colour or chemical variability with any degree of certainty, but a more systematic analysis of the fabrics might yield results or at least help support the current hypotheses.

To move away from chloride being present in the charge, an alternative possibility is that salt or salt water was used in the mixing and application of the parting layer. This is a less persuasive argument though because the small quantities of sodium chloride in sea water may not be enough to produce the visual effects seen, even if the parting layer is 5 mm thick. If the chloride comes from the parting layer then there should be no tide-marks on the crucible fragments. The salt coming out of the parting layer would cause bleaching and, if enough, also a continuous pink discoloration along the wall. AT4 had a parting layer prepared with saline solution, but it was of insufficient thickness to prevent melting and should not be used as evidence against this option. Preliminary observations of temperature effects on the colour and stability of the re-fired technical ceramic indicates a maximum operating temperature of probably around 1100 °C, similar to the values established half a century ago by TURNER for the crucibles from Amarna.²⁷ This is sufficiently higher than the working temperature of 900 °C to 1000 °C²⁸ to enable routine firing procedures, including the inevitable over-firing of vessels due to limited temperature control in the kiln.

Overall, the experiments reported here have shown how drastically the presence of common salt, NaCl, affects the use traces on the glassmaking vessels, and how these use traces differ from those which had no or only little salt present in the charge. This alone provides already a very powerful tool for the assessment and interpretation of relevant archaeological material from Qantir-Piramesses and other sites, such as Amarna. Work scheduled for autumn 2006 will attempt to apply this visual tool systematically to the entire assemblage in Qantir (Fig. 27). In parallel, we are planning to complete the analytical investigation of the experimental samples, including the glasses produced and the various zones and layers of the ceramic materials, in order to better understand the mechanism and chemistry behind these discolorations.

²⁵ PUSCH/REHREN in this volume, chapter 2.10.2 (Gläser).

²⁶ HUDSON 1997, 136.

²⁷ TURNER 1954.

²⁸ Estimated based on the glass compositions, SCHOER/REHREN in this volume, Figure 8.



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