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FAIENCE: AN INVESTIGATION OF THE MICROSTRUCTURES ASSOCIATED WITH THE DIFFERENT METHODS OF GLAZING

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INTRODUCTION

In the present context, faience refers to a ground-quartz body with alkaline glaze which first occurs as early as the fourth millennium B.C. and has continued to be used in the Near East for the production of small objects up to the present time.

Recent work by Vandiver (1982, 1983) and Tite *et al.* (1983) has confirmed that the three principal methods used to glaze faience in antiquity were (1) direct application of the glazing mixture to the surface of the ground-quartz body; (2) efflorescence glazing method in which the glazing components were mixed with the moistened ground-quartz body, being carried to the surface during subsequent drying, and (3) cementation glazing method in which the ground-quartz body was fired whilst buried in the glazing mixture.

Vandiver (1983) has studied the external morphology of faience objects with a binocular microscope and, to a more limited extent, their internal microstructure in fracture section with a scanning electron microscope (SEM). On the basis of her observations on both laboratory-produced replicas and ancient objects, she has suggested criteria by which the three different methods of glazing can be distinguished. Tite *et al.* (1983) undertook a preliminary study of the internal microstructure, in polished section with the SEM, of faience produced in the laboratory by the efflorescence and cementation glazing methods. They again suggested tentative criteria for distinguishing between these two methods of glazing.

In the current work, the laboratory replications were extended to include direct application of a glazing mixture, both in the raw state and in the form of prefired and ground frit. The faience thus produced was again examined in polished section with the SEM and the distinguishing characteristics of the microstructures associated with the three principal methods of glazing were defined.

EXPERIMENTAL PROCEDURES

Laboratory reproduction

Ground flint was used to make the quartz bodies in all the laboratory reproductions. This raw material was chosen because its particle-size range (less than 50 μm diameter) closely matches that of the ground quartz in a significant number of ancient faience bodies. Furthermore, even when coarser-grained quartz (100–200 μm diameter) predominates in the ancient faience bodies, significant amounts of fine-grained quartz (less than 50 μm diameter) are still present. The quartz sand with a particle-size range of 100–200 μm diameter, which was used in the previous laboratory reproductions (Tite *et al.* 1983), was therefore no longer considered appropriate.

For the direct application glazing method a mixture, based on the analysis of a shabti glaze (Tite *et al.* 1983) (i.e. 72.5% SiO_2, 5.0% CaO, 19.0% Na_2O and 3.5% CuO) was made up from ground flint, calcium carbonate, anhydrous sodium carbonate and ground malachite. This mixture was then applied, either in its raw state or after prefiring to 800 °C to form a frit and then grinding, to the surfaces of the ground flint bodies which had been shaped into roughly spherical beads. For the efflorescence glazing method, a mixture consisting of 80% by weight of ground flint and 20% by weight of the same glazing mixture but without the addition of silica was prepared. Sufficient water was added to the mixture to produce a paste which was then shaped into beads. These beads were left to dry at a room temperature of about 18 °C during which efflorescence produced a surface layer of glazing mixture. With both methods, the beads were fired at either 850 °C or 980 °C for 1 hour in air in a muffle furnace.

For the cementation glazing method, a sample of glazing mixture, obtained by Mr O. Watson from the workshop at Qom in Iran reported on by Wulff *et al.* (1968), was used. This mixture consisted of plant ash, hydrated lime, powdered quartz, copper oxide and charcoal. An unfired bead of ground flint was buried in the mixture in a porcelain crucible and fired at 980 °C for 1 hour in air in a muffle furnace. At the termination of firing, the glazing mixture was readily crumbled away to leave the glazed bead.

Examination and analysis

Samples were taken from the beads produced in the laboratory in order to provide sections through the glazes into the cores. After resin impregnation and polishing, these sections were examined in the SEM in which the phases present could be distinguished on the basis of their atomic number contrast, that is, the quartz appears dark as compared with the higher atomic number glass phase (figures 2–9).

The glazes were analysed quantitatively using a Link Systems energy dispersive spectrometer attached to the SEM. The results indicated that the compositions of the glazes produced by direct application of prefired and ground frit and by efflorescence (i.e. 70–75% SiO_2, 18–20% Na_2O, 3–5% CuO, 2–5% CaO) are comparable to those for unweathered glazes on ancient faience objects from Egypt (Tite *et al.* 1983). The composition of the glaze produced by cementation (i.e. 60–65% SiO_2, 16–20% Na_2O, 6–8% K_2O, 3–5% CuO, 3–7% CaO) is also similar except for its higher K_2O concentration which probably reflects the different plant-ash used in present-day Iranian workshops as compared to those used in Egypt in antiquity.

RESULTS AND DISCUSSION

The SEM examination showed that, typically, the faience consists of a quartz core containing varying amounts of interstitial glass (figure 1, Layer III). The core is encased in an interaction layer which is made up of quartz embedded in a continuous matrix of glass (Layer II). Overlying the interaction layer, there is in some cases a surface layer of quartz-free glaze (Layer I). The microstructure of the faience can therefore be defined in terms of: (1) thickness of the surface glaze layer; (2) the thickness of the glaze-core interaction layer; (3) the nature of the boundary between the surface layers and the quartz core, and (4) the extent of any interstitial glass phase within the quartz core. Data on these parameters for faience produced in the laboratory by the different methods of glazing are given in table 1. These data are based on the examination of a minimum of two beads produced by each method at each firing temperature, the actual number of beads produced also being listed in table 1.

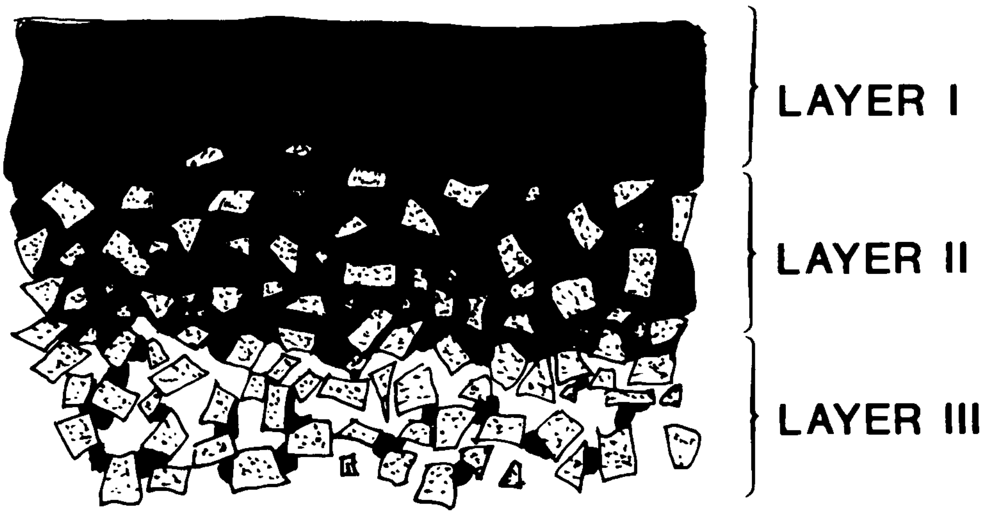

Figure 1 *Diagramatic representation of the microstructure of faience showing surface glaze (Layer I), glaze-core interaction layer (Layer II) and quartz core (Layer III).*

The faience produced by the direct application of the glazing mixture in the form of prefired and ground frit is characterised by thick surface glaze and glaze-core interaction layers, with the former being significantly thicker, and by quartz cores containing no interstitial glass (figures 2 and 3). The boundary between the glaze-core interaction layer and the quartz core is clearly defined. This microstructure is consistent with the use of frit in which the alkali (Na_2O) and colourant (CuO) are effectively immobilised. As a result, reaction occurs only at the interface between the applied frit and the quartz core and there is no transport of alkali or colourant beyond the glaze-core interaction layer.

The faience produced by the direct application of raw glazing mixture is more variable in its microstructure. Although a surface glaze layer is consistently absent, the glaze-core interaction layer varies from thick and essentially continuous (figure 4) to thin and fragmented (figure 5). In both cases, however, the interaction layer tends to merge into the quartz core and the boundary between them is poorly defined. Near to the boundary, the core contains sufficient interstitial glass to produce some bonding between the apices of adjacent quartz grains whereas, away from the boundary, no interstitial glass was observed. These microstructures are consistent with the use of a raw glaze in which the alkali and colourant are potentially mobile and can therefore be transported into the core. The variability in microstructure thus reflects differences in the amount of glaze applied to the surface, as well as differences in the relative moisture content of the glazing mixture and the quartz core. The relative moisture content, in turn, determines the extent to which glaze components are transported into the core.

The faience produced by the efflorescence glazing method is again somewhat variable in its microstructure. The microstructure is, however, consistently characterised, first, by thick surface glaze and glaze-core interaction layers, with the former tending to be of somewhat irregular thickness (figure 6). Second, the glaze-core interaction layers merge into and are not readily distinguished from the quartz cores which themselves contain extensive interstitial glass, sufficient to bond together the faces of adjacent quartz grains and even surround the smaller grains (figure 7). The microstructures are consistent with mixing the glaze components with

Table 1 *Microstructure data*

Reproduction technique	*No. of samples*	*Firing temperature (°C)*	*Surface glaze (Layer I) (thickness – μm)*	*Glaze-core interaction (Layer II) (thickness – μm)*	*Boundary*[1] *Layer II to Layer III*	*Core interstitial glass*[2] *(Layer III)*
Direct application – frit	2	850	300–600	100–200	Defined	None
Direct application – frit	2	980	200–800	50–200	Defined	None
Direct application – raw mixture	3	850	–	200–400	Diffuse	Apex near surface
Direct application – raw mixture	2	980	–	200–700	Diffuse	Apex near surface
Efflorescence	3	850	100–300	200–400	Diffuse	Face
Efflorescence	3	980	50–400	200–300	Diffuse	Face
Cementation	2	980	20–50	50–200	Defined	Apex/face near surface

[1] Defined – boundary between interaction layer and core is clearly defined. Diffuse – boundary diffuse such that interaction layer merges into and is not readily distinguished from core.

[2] Apex – glass produces bonding between apices of adjacent quartz grains. Face – glass produces bonding between faces of adjacent quartz grains.

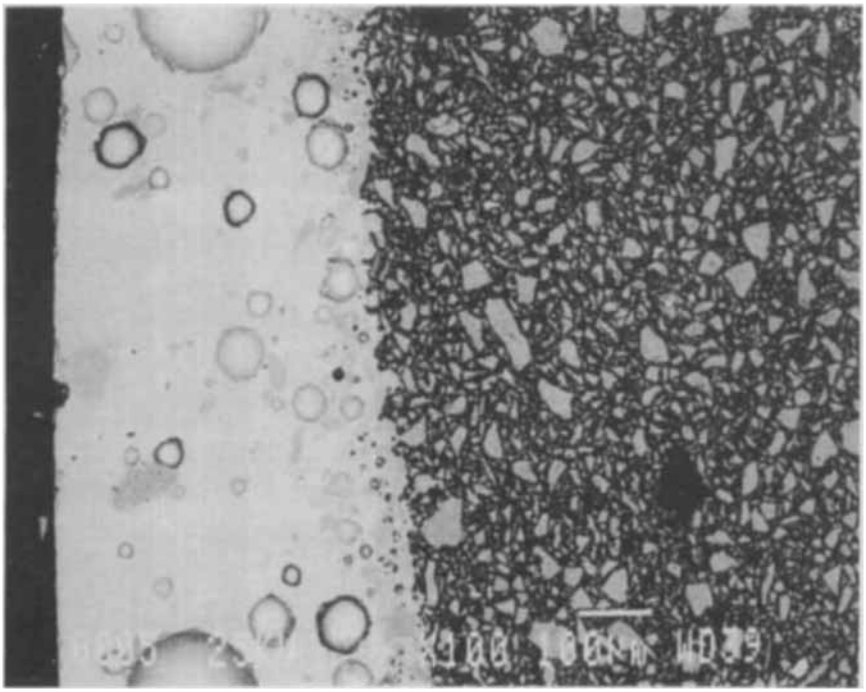

Figure 2 *SEM photomicrograph of section through faience produced by direct application of fritted glaze mixture (firing temperature 850° C), showing surface glaze, glaze-core interaction layer and quartz core containing no interstitial glass.*

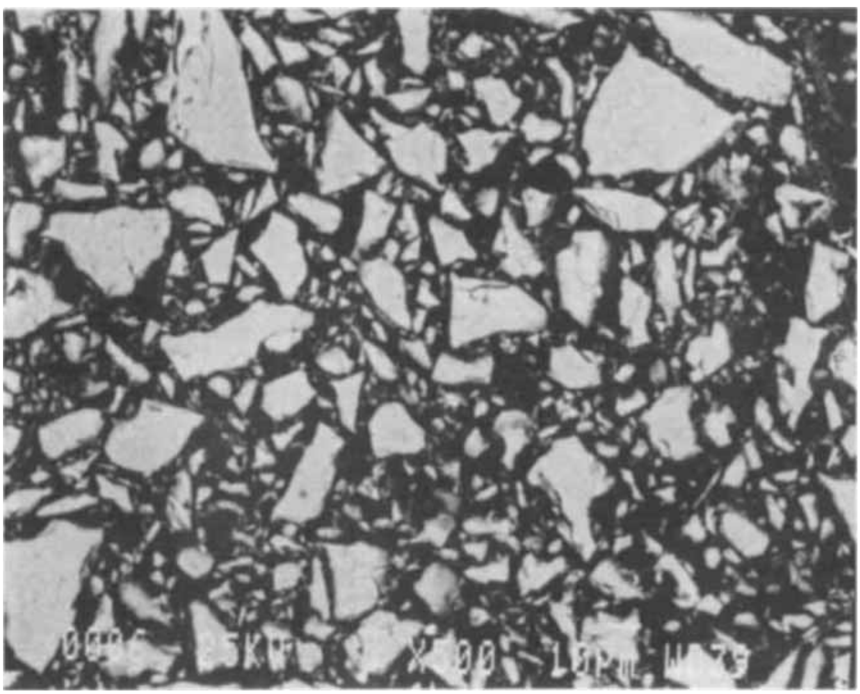

Figure 3 *SEM photomicrograph of section through faience produced by direct application of fritted glaze mixture (firing temperature 850° C), showing quartz core with no interstitial glass.*

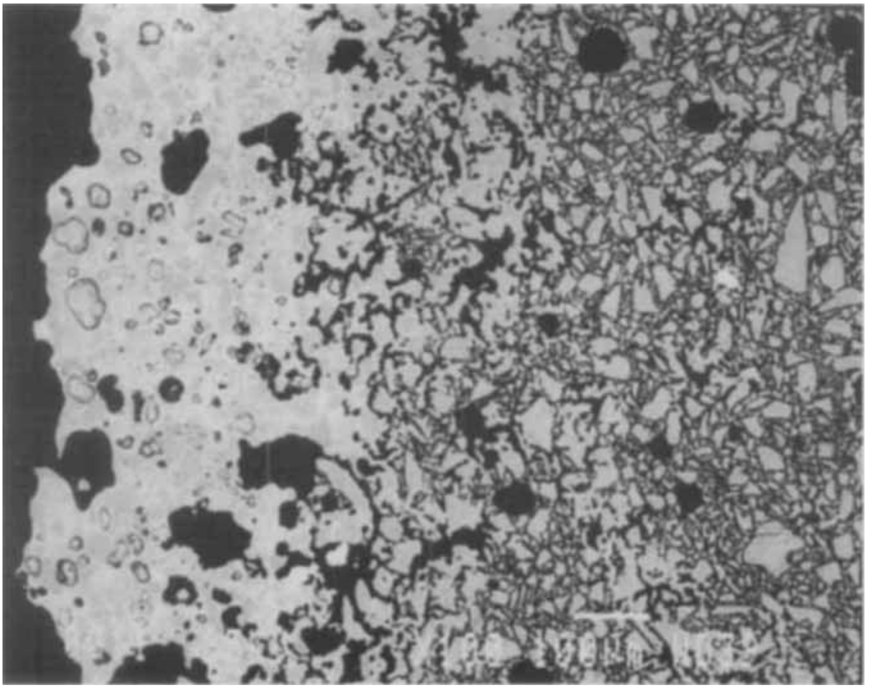

Figure 4 *SEM photomicrograph of section through faience produced by direct application of raw glaze mixture (firing temperature 850 °C), showing thick glaze-core interaction layer which merges into the quartz core containing some interstitial glass near to the interface.*

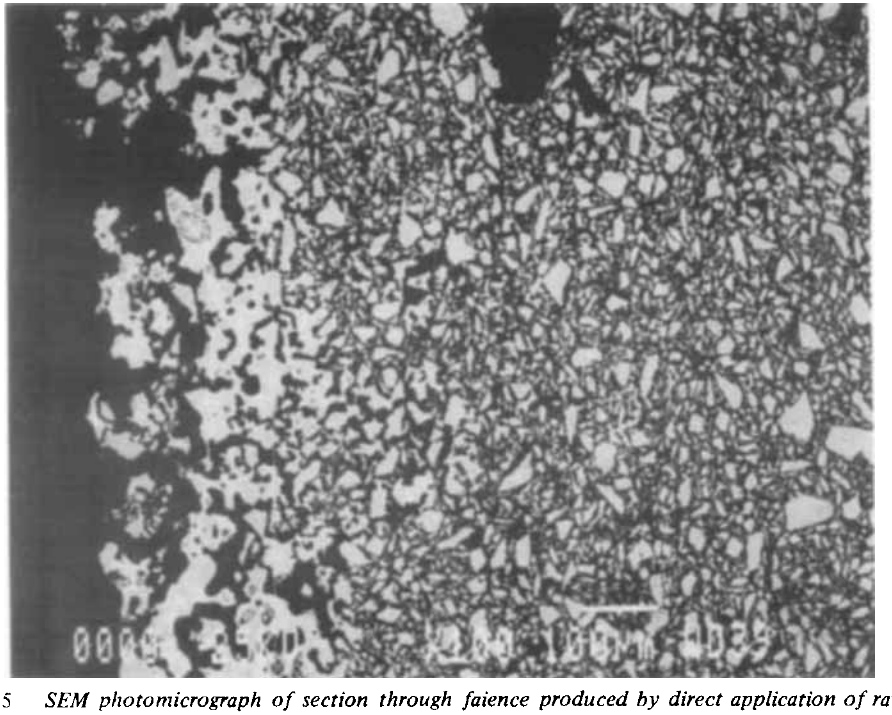

Figure 5 *SEM photomicrograph of section through faience produced by direct application of raw glaze mixture (firing temperature 850°C), showing fragmented glaze-core interaction layer which merges into the quartz core containing some interstitial glass near to the interface. No interstitial glass is visible in the core away from the interface (right hand side of photomicrograph).*

the ground-quartz core material in that a significant amount of the glazing mixture inevitably remains within the core after drying and forms the interstitial glass. The variability in microstructure reflects both the water content and rate of drying of the faience which, in turn, determine the extent to which the glazing components are transported to the surface.

The faience produced by the cementation glazing method is characterised by thin irregular surface glaze layers, somewhat thicker glaze-core interaction layers and clearly defined boundaries between the interaction layer and the quartz core (figure 8). Near to the boundary, the core contains sufficient interstitial glass to produce some bonding between the apices and faces of adjacent quartz grains (figure 9) whereas away from the boundary no interstitial glass was observed. This microstructure reflects some initial transport of the glazing components into the quartz core, possibly in the vapour phase. However, once the glazing mixture has interacted with the quartz at the surface of the bead to form glaze and glaze-core interaction layers, the core is effectively protected from further intrusion by the glazing components.

For the faience produced by the direct application of glaze or by the efflorescence glazing method, increasing the firing temperature from 850 °C to 980 °C did not significantly change the observed microstructure. At the higher firing temperature, there were fewer isolated grains of undissolved quartz remaining in the glaze layer and some of the quartz in the glaze-core interaction layer had recrystallised as cristobalite. In contrast, for the faience produced by the cementation glazing method, the higher firing temperature was essential, no glaze being produced at a firing temperature of 850 °C.

With regard to the physical appearance of the beads, the glaze produced by the direct application of the fritted glaze mixture was the most uniform and had the highest gloss. Conversely, the glaze produced by the direct application of the raw glaze mixture was uneven and, as a result of the absence of a definite surface glaze layer (figures 4 and 5), was predominantly matt. The quartz cores of the beads produced by the direct application of the fritted glaze mixture were white and extremely friable. Those produced by either the cementation glazing method or the direct application of the raw glaze mixture were still white but somewhat less friable. In contrast, the cores of the beads produced by the efflorescence glazing method were, as a result of the presence of extensive interstitial glass (figure 7), pale blue and hard.

CONCLUSIONS

The results presented above confirm that the microstructure of faience, as observed in cross-section in the SEM, should be of considerable help in distinguishing between the different methods used in antiquity for glazing faience. To summarise, thick surface glaze and glaze-core interaction layers, a clearly defined boundary between the interaction layer and the quartz core, and the absence of interstitial glass in the core suggest direct application of fritted glaze mixture. Thin surface glaze and interaction layers, but, again, a clearly defined boundary and the absence of interstitial glass except near to the boundary suggest cementation glazing. Thick surface glaze and interaction layers but, instead, a diffuse boundary and extensive interstitial glass suggest efflorescence glazing. Finally, the absence of a surface glaze layer, a diffuse boundary and no interstitial glass except near to the boundary suggest direct application of raw glaze mixture.

In applying these criteria to ancient faience, the possibility that, prior to glazing by direct application or cementation, alkali was added to the quartz core must be borne in mind. The presence of alkali in the core, whether added in the raw state, as a frit or even as a glass, would

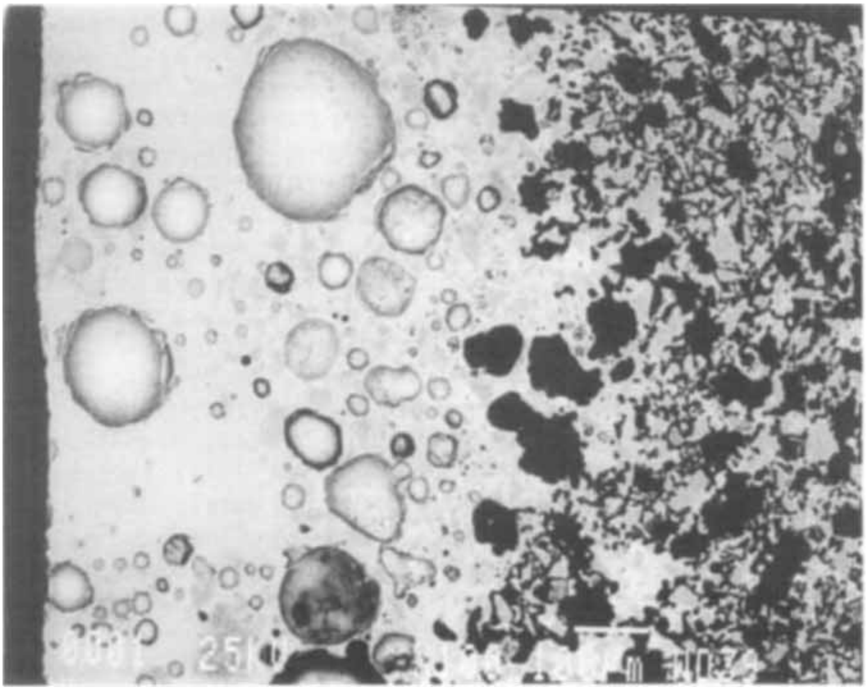

Figure 6 *SEM photomicrograph of section through faience produced by efflorescence glazing method (firing temperature 850 °C) showing surface glaze and glaze-core interaction layer which merges into quartz core containing extensive interstitial glass.*

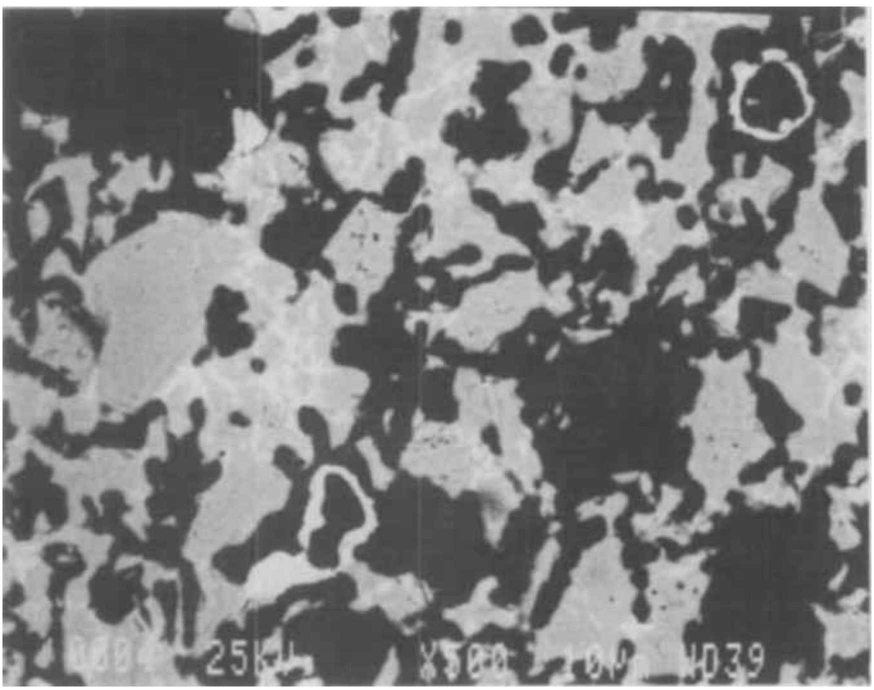

Figure 7 *SEM photomicrograph of section through faience produced by efflorescence glazing method (firing temperature 850 °C) showing quartz core with extensive interstitial glass which bonds together the faces of adjacent quartz grains and surrounds the smaller grains.*

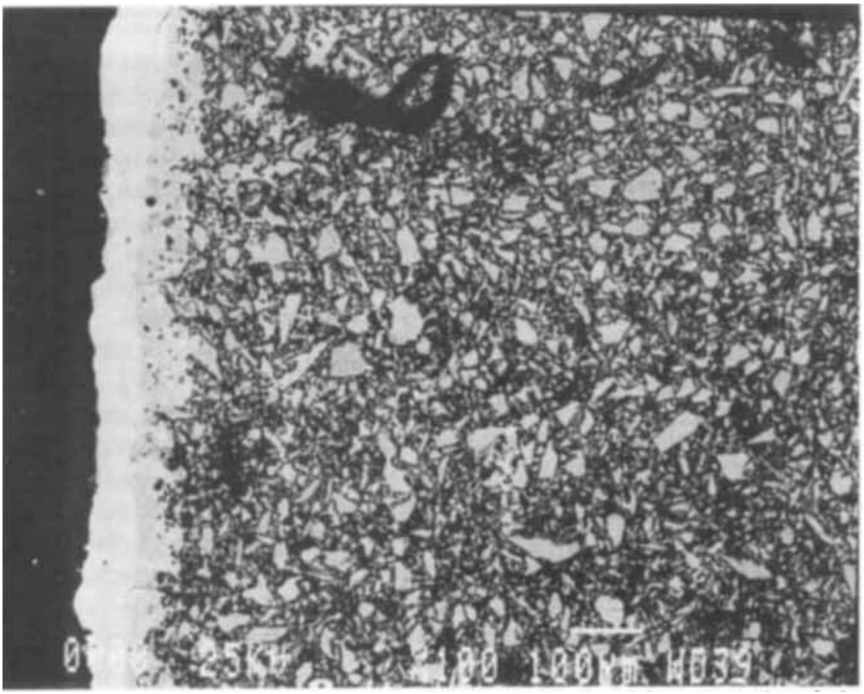

Figure 8 *SEM photomicrograph of section through faience produced by cementation glazing method (firing temperature 980 °C), showing surface glaze, glaze-core interaction layer and quartz core containing some interstitial glass near to the interface. No interstitial glass is visible in the core away from the interface (right hand side of photomicrograph).*

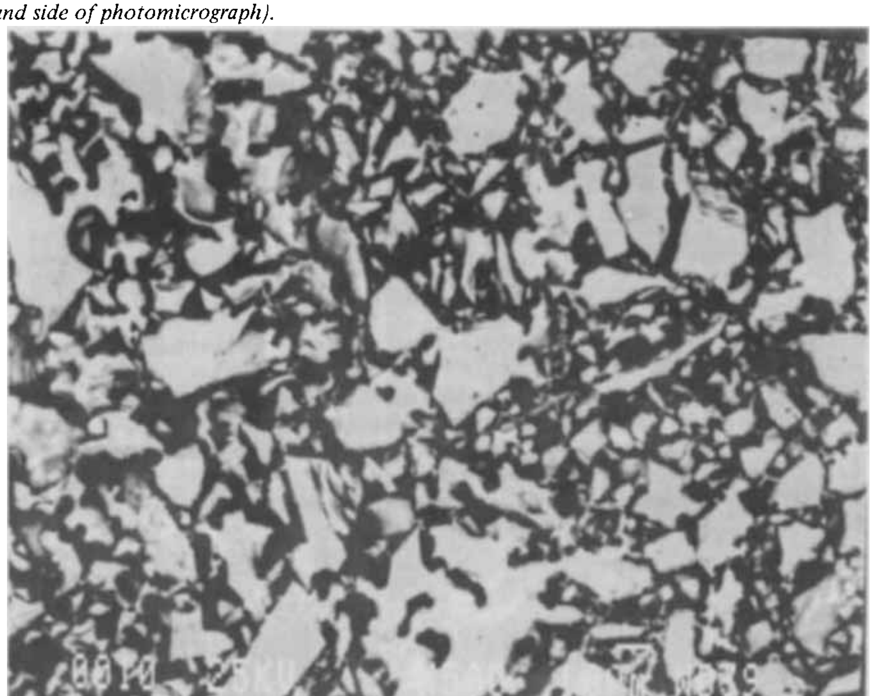

Figure 9 *SEM photomicrograph of section through faience produced by cementation glazing method (firing temperature 980 °C), showing quartz core with sufficient interstitial glass to produce some bonding between the apices and faces of adjacent quartz grains.*

result in the formation of interstitial glass and thus confound any attempt to identify the method of glazing on the basis of microstructure. Further, if a thin object is glazed on both sides, then the interstitial glass which, after glazing by direct application of the raw mixture or by cementation, is present close to the boundary between the glaze-core interaction layer and the quartz core could extend throughout the core. Again, any attempt to identify the method of glazing on the basis of microstructure would be confounded. In addition, when examining ancient faience, the situation is further confused by the fact that the surface glaze could have been subjected to both physical wear and chemical weathering. Some of the surface glaze layer may, therefore, have been lost and alkali may have been leached from the remainder, thus making the study and interpretation of the microstructure more difficult.

In spite of these difficulties, the criteria established above confirm the methods of glazing previously suggested for four faience objects from Egypt (Tite *et al.* 1983). That is, the two rings (BMRL No. 16319 and 16321) were glazed by efflorescence, the shabti (16323) by cementation and the shabti (16322) by direct application of fritted glaze mixture.

Finally, it should be emphasised that, although the current paper is concerned entirely with the examination of faience microstructures, the study of the external morphology of the faience (i.e. distribution of glaze, drying and firing marks etc.), as described by Vandiver (1982, 1983), is of comparable importance. In the first place, to take a section through the glaze for microstructural examination would cause an unacceptable amount of damage to a high proportion of ancient faience objects. Second, even when microstructural examination is possible, the results obtained should always be interpreted in conjunction with those obtained from the study of the external morphology. However, this latter approach depends much more on the individual's experience of, and familiarity with, the material. Also, the criteria are often subjective and, since faience objects do not always show, for example, drying and firing marks, the argument is often based on absence of any evidence rather than on direct evidence for the method of production used.

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