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Eternal Fragile: Preliminary Analytical Approach to Glass Characterization at Early Islamic Glass Workshop in the City of Istakhr

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Abstract:

Glass finds from the archaeological excavation of the city of Istakhr in the Fars province was subjected for the first time to archaeometrical investigations. Based on their archaeological context, these pieces were dated back to early Islamic period, the last period of growth and prosperity of the city, before its final decline in the tenth century AD. The interdisciplinary investigations on glass pieces from Istakhr confirm an essential scientific approach due to the chemical composition of the most important antique glass workshops in the Iranian plateau based on their material characteristics and cultural evidence. Glass fragments have been analytically studied by WXRF, optical microscopy and ESEM-EDS in order to evaluate their chemical composition, micro-structure and for textural characterization. the clustering of the objects was of interest, due to discriminate between the glass finds in this region. Based on the chemical analysis, the investigated objects are therefore categorized as high “Potash-Calc”.

Keywords:

Ancient Glass, Structural Properties, Chemical Composition, Early Islamic Period, Istakhr.

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1. Introduction

Glass is a silicates containing other oxides, notably CaO, Na_2O, K_2O, and sometimes Al_2O_3. This chemical composition influence the glass properties, as well as giving some clues about the manufacturing of this material. Literary mentioned, a typical classification of glass in the antiquity can be divided in two groups; sola-lime silica glasses and potash-lime silica glasses (Degryse et al., 2014; Rosenow et al., 2018). soda–lime glass consists of approximately 70 wt% SiO_2, which were balanced by adding mainly Na_2O (soda) and CaO (lime) within the batch. The information of the glass industry and the usage of plant ash in the Near Eastremains principally poor (Phelps, 2018). The description of a framework about glass manufacturing processes, followed by the divers types and model of manufacturing and purification organisation for the plant ash industry, is still lacking (Brill, 1999;Emami et al., 2020; Freestone, 1987; Freestone, 2006; Henderson et al., 2004).

Glass is an archaeologically magnificent object which highlights the tendency of ancient technicians to strive for perfection. Technically, glass is a highly complex material, since it is a kind of substance which performs to be built – thermodynamically – in a metastable situation. The specific origin and birthplace of glass and the origin of its manufacture is unclear. It is thought that the first glass production was probably documented in the Mitannian or Hurrian region of Mesopotamia, perhaps as an extension of glaze productions (ca. 5000 BC) (Cummings, 2002). The manufacture of glass objects highlights the awareness, curiosity, and high levels of technological perfection in a characteristic triangle. Its development likely stemmed from many years of technological learning from the manufacture of ceramics, stone working (e. g. obsidians), and metal extraction. As a matter of fact, the first evidence of artificial (or accidental) glassy materials is metallurgical slag and other amorphous remains such as high tempered kiln walls (Gliozzo et al., 2013). Whereas clay-based materials such as ceramics and bricks have been routinely subjected to multi-analytical and elemental analysis to determine the structural and chemical characteristics as well as provenance determinations, archaeological glass has received less attention (Freestone, 2006). Administratively, this is mostly based upon the rarity of glassy samples or uniqueness of some glass finds which are not allowed to be analysed via destructive methods. Important databases of major, minor and trace element analyses of glasses are reported (Brill, 1999; Brill and Rising, 1999), but beyond extensive scientific attachments, substantial compositional clustering has been difficult to define (Freestone, 1991). This problem can be discussed by structural properties of glass as well as its chemical influences to the surrounding environment (Dutrizac, 1983; Emami and Pakgohar, 2017). The environment reflects the mobility processes of the elements (leaching) and assumes different signals during burials (Emami et al., 2016; Heimann, 2017). In a considerably different aspect, glass is a material which highlights meta-stability. This means that the glass recrystallization that occurs during melting will be executed by a transitional temperature state through super cooling of a liquid at high temperature (Shackelford and Doremus, 2008). The metallic Ions within the texture can alternatively achieve this in many different ways (Cholakova et al., 2016; Okada et al., 2018; Quartieri et al., 2002). The investigation on archaeological glasses are therefore interesting, since studying such objects will offer both the know-how about the manufacturing process as well as possible tracking of the burial conditions based on the glass texture as amorphous material over time (Henderson et al., 2018; Li et al., 2014).

2. Area of Study

The Fars province, as the origin of two great Iranian empires (Achaemenid and Sassanid), has always held great significant in archaeological studies (Benech et al., 2012; Boucharlat, 2002). The city of Istakhr (29° 58’ 51’’ N, 52° 54’ 34’’ E) is situated in the Fars province, about 60 km north of Shiraz and approximately 5 km north of Persepolis, in the narrow valley of the Pulvār River, on the northern edge of the plain of Marvdasht (Figure 1). The town was enclosed by walls with round towers, today clearly discernible on the high earthen mound surrounding the archaeological area. Only very limited areas of the town were excavated in the years following the 1st World War. In fact, the earliest archaeological researches were carried out by the Chicago Oriental Institute in the 1930s. Ernst Herzfeld first, from 1932, and Erich F. Schmidt later, from 1935, executed some on-site tests, but the area was only partly excavated and unfortunately the results of their research were never fully published. The investigations only partially brought to light at least one building with a square hypostyle plan identified by Herzfeld, and in turn by Schmidt, as a mosque (Fontana 2018:11,12). In fact, archaeological investigations at Istakhr have been extremely limited, and many of the proposed interpretations of its origins and earliest phases are based on rather speculative textual evidence alone. One hypothesis suggests that a settlement existed at the site of Istakhr as early as Achaemenian or even pre-Achaemenian times (Callieri 2018:31). Based on written and archaeological sources, the city then flourished during the post Achaemenid and Sasanian periods. Despite all the conflicts and wars that took place during the early Islamic period between the inhabitants of the city of Istakhr and the Arab invaders (Casari 2018 :77-85), it seems that this city also had a period of growth and prosperity, especially in the eighth and ninth centuries AD. Some of the city's distinct products, including molded pottery (Asadi 2018:413- 437) and glass studied in this article, date back to this period of prosperity.

An Iranian-Italian cooperation agreement was signed in November 2011 to enable the start of the Istakhr Project, the aim of which was the historical-archaeological study of the early Islamic phase of the town. The field work undertaken in two seasons in 2012 aimed to provide a better understanding of the complexity of the archaeological area of the town and its surroundings, as well as to carry out an in-depth investigation of a sector within the city walls. The work included archaeological and topographic surveys and the creation of an archaeological map of the site. Moreover, in the chosen urban sector – after digital terrain modelling and a targeted geo-physical investigation - a test trench was excavated and a preliminary study was made of the finds (Fontana2018:13-14). The glass fragments, which are studied here are found in these field work seasons. In fact Based on this reconstructed layout of the first Islamic city, a trench was opened during the second campaign of the Joint Iranian-Italian Mission at Istakhr in the autumn of 2012 to investigate the area west of the western wall of the ‘mosque - identified by Whitcomb as the qiblī wall (Jaja2018:304). In the end of the excavation, an paved stone street – extended with a south- north direction- was found parallel with the mosque wall.

The samples were gathered from the first compacted and well-smoothed floor level found immediately to the west of the mentioned street, in a context unlikely to be earlier than the 11th century AD. The main aim of the research was to chemically analyse the glass objects from the mentioned area to characterize them and to verify the presence of a definite local glass composition.

Huge amounts of pottery sherds as well as glass fragments have been found in this layer, suggesting a 11th century dating (Noruz Zadeh Chegini et al., 2013). Similarly, one fragment of 10th – 11th century graffiti has also been found. Finally, numerous unglazed wares featuring inclusions (including vegetal chaff) on their surfaces (Priestman, 2005; Whitcomb, 1985) have also been found there. This kind of fabrication suggests that they might be related to the unglazed painted wares from later phases of excavation. This phase, (phase 4) has been attributed to a range of dates from the 11th century to the 14th century (Chegini et al., 2013). Overall, the recovered glass objects reveal critical information about the technological know-how necessary for production as well as the exploitation of thematerial sources and their chemical composition. A multi-method chemical analysis on some glass fragments from Istakhr has been conducted. Thus this study contributes as a key insight into glass characterization, chemistry and use in the early Islamic era.

3. Materials & Methods

Based on the unique character of the samples, and the aversion to destructive analyses for such rare objects, the sampling process was performed only on seven fragments (Figure 2). The location of sample extraction differed, as samples were taken from the body as well as the neck, bottom or mound of the objects. All samples were collected from the excavation in 2015. Macroscopically, they were demonstrably blue, white, green, violet, and olive green, as a result of the accumulation and leaching of certain elements, followed by an iridescence effect on the surface (Emami et al., 2016). Heat treatment and manufacturing temperatures of these objects were crucial, due to the technological circumstances in this region. The laminar structure of weathering crusts on soda-lime-silica and on potash-lime-silica glasses is associated with variations in selective leaching due to seasonal variation of temperature and availability of water as well electrolyte solution. The samples show typical macroscopically structures which will be described as a group of finds as follows.

Table 1. Macroscopical observations of the objects.

Sample	Description	Color	Type of Damages
SU129	Part of Body	Dark Blue	Pitting, Getting Layered & Sugary
SU141	Piece of Edge	Metal Grey	Iridescence, layered , Alkali ion concentration, shelly appearance
SU146	Piece of a container's neck	Green/Blueish	Alkali ion concentration, Micro bubbles
SU156	Part of body	Green	Micro bubbles, Discoloration & Getting layered
SU160	Handle	Green	Iridescence, Layered, Pitting
SU184	Piece of bottom	Green	Discoloration, Leaching, Milky, Pitting, Alkali ions Concentration, Glass Seek
SU190	2 part of a container	Green	Getting layered, Milky & Alkali ion concentration

The SU 129 group originates from the body of a jar and features a dark blue colour. The surface character of this group is described as sugary, associated with pitting corrosion and layering character.

The SU 141 group consists of some pieces from the edge. They are metallic dark grey coloured and show already iridescence effects as a result of alkali ion concentration and getting layered (Nazarov et al., 2013). SU 146 contains mostly pieces of a container's neck. They show green blueish colours and iridescence effects. On the surface, little bubbles are visible due to the selective corrosion procedure (Nazarov et al., 2013). The SU 156 group is part of the container body. It exhibits a green colour and bubbles associated with discoloration effects of the surface. The external surface character is layered. The SU 160 group contains mostly samples from the handle. These have a green colour with an iridescence effect. Surface character is again layered with tiny pitting corrosion structure. The SU 184 groups are mostly samples from the bottom of an object. They also feature a green colour. The surface character is described by means of discoloration, encrustation of huge contamination layers, and alkali ions concentration followed by iridescence effects. Finally, the SU 190 group contains pieces of the container's body. Like others, these are green coloured. The surface character is considered to be layered with a milky structure and sharp iridescence effects.

The samples have also been investigated by means of wavelength XRF (WXRF) with a Bruker instrument PW 2400 WXRF in Razi Metallurgical Foundation in Tehran to provide insight into the bulk chemical compositions of the glasses. To avoid any contamination of the results only the core of the glass samples and non-corroded parts of the glasses have been analysed.

For a higher resolution microstructural analysis, ESEM was used. For the investigation of the glass and the glassy character of these materials, samples were glued with silver glue on a sample holder and investigated with a FEI FEG Quanta 240 ESEM in low vacuum mode at approximately 100 Pa at the University Siegen, department of building materials chemistry. In this regard the inner structures of glasses as well as related damaging conditions on the surface were investigated.

4. Results and Discussion

Bulk chemical composition of the glasses by wavelength X-ray fluorescence

Chemical compositions of the glass fragments are provided in table 2. From the chemical point of view, the glass fragments from Istakhr are classified as high sodium-calcium-siliceous glasses (high natron calc silica glasses) (Fig. 3). The chemical composition of the glasses was quantified as major elements (>5wt%), minor elements (1-0.1 wt.%) and trace elements (<0.01 wt.%). The correlations between the major elements in chemical clusters of ancient glasses are critical for the discrimination between the samples from the eastern Mediterranean region and Europe (Brill and Rising, 1999; Henderson, 1985; Rehren, 2000). In this case study, SiO_2 is generally included in sand and gravel. However, alkali oxides (e.g. Na_2O and K_2O) within the glass used as flux are possibly derived from wood ashes (Angelini et al., 2004). CaO and MgO can be obtained from calcite or dolomitic rock forming stone. This mineral shows high solubility in water and therefore leaches toward to the surface of glasses and results in white encrustation (Shortland and Tite, 2000).

The glass fragments from Istakhr showed to have an essentially differing amount of K_2O from the Iron-Age and Roman Period glasses in Europe (Henderson, 1988). The fragments proved to have a specific alkali composition with a nearly constant amount of K_2O (2.37-5.39) and inconsistency in quantity of Na_2O (10.9-45.3 wt.%). This can be crucial for the assessment of alkali elements in glass production. The MgO content is consistency approximately 1-2 wt.%, and the mean CaO/MgO weight ratio is about 1:4.77. The amount of FeO is roughly related to the amount of Al_2O_3 at a mean weight ratio about Al_2O_3/FeO=1.43, which indicates a possible common source for the glasses (Angelini et al., 2004; Brill, 1963). The main common source of material can also be interpreted by means of the remarkably similar correlation between FeO and MgO which is calculated to MgO/FeO=1.46 as weight ratio. This is indeed very similar to the Al_2O_3/FeO ratio (1.43). In summary, the Fe, Mg, Ca, and Al all possibly share a common heritage. P_2O_5 has had only minor influence on the chemical composition due to its value being considered as a minor oxide with little variations among these fragments. The amount of P_2O_5 in ancient glasses indicates that it was probably accessible in a vegetable potash source of alkali (Henderson, 1988). In the glass fragments from Istakhr, P_2O_5 has a positive correlation with K_2O. This is also reported for most of the plant ashes and particularly strong for soda-rich plants (Barkoudah and Henderson, 2003).

Table 2. Bulk chemical composition of the glasses with WXRF. Results are given in wt.%.

Sample	SU146	SU156	SU160	SU184	SU129	SU141	SU190
Na_2O	34.86	45.3	20.48	10.9	21.96	19.54	18.21
K_2O	2.96	2.37	5.29	3.24	2.76	2.57	3.04
CaO	5.97	5.51	9.37	4.67	7.44	5.78	5.52
Fe_2O_3	0.98	1.57	2.02	0.67	1.06	1.89	0.65
SiO_2	48.9	37.17	58.58	76.21	58.86	62.27	64.58
MgO	1.1	0.92	1.06	1.14	2.37	1.81	2.02
Al_2O_3	2.27	1.72	1.49	0.95	1.86	1.39	1.27
TiO_2	0.06	0.07	0.04	0.04	0.07	0.06	0.04
Co_3O_4	0.01	<0.01	<0.01	<0.01	0.19	0.65	0.24
ZnO	<0.01	<0.01	0.01	0.01	<0.01	0.02	<0.01
BaO	0.05	0.04	0.04	0.07	0.14	0.15	0.17
Cl	0.38	0.65	0.63	0.76	0.51	0.92	0.53
Cr_2O_3	<0.01	0.01	<0.01	<0.01	0.21	0.38	0.39
SrO	0.04	0.06	0.06	0.14	0.07	0.04	0.09
NiO	<0.01	0.01	<0.01	<0.01	0.01	0.02	0.01
SO_3	0.25	0.36	0.41	0.28	0.43	0.24	0.28
P_2O_5	0.01	0.02	0.04	0.01	0.03	0.12	0.12
CuO	0.03	<0.01	<0.01	<0.01	<0.01	0.2	0.01
MnO_2	2.13	4.22	0.48	0.92	2.12	1.95	2.83
La&Lu	<1	<1	<1	<1	<1	<1	<1

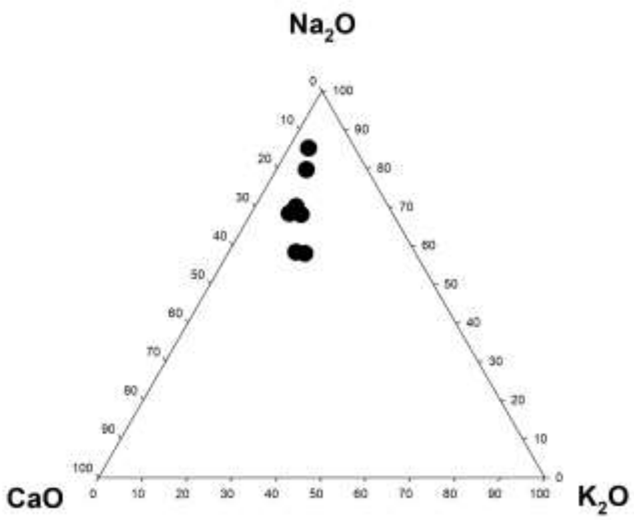

Figure 3. Ternary diagram for clustering the chemical composition of glass fragments as a function of three major oxides CaO-Na_2O-K_2O.

4. Micro- structural analysis by ESEM

Microstructures of the glasses have been studied by means of SEM without sputtering. Multi-layered structures of silicon oxides and glass networks were formed (Figure 4a) as well as the bubbles.

The temperature required to form a glass melt varies with the composition of the batch. Typical melting temperatures are in the range of 1300–1600°C, which is rarely reported during antiquity. At this stage of the process, the melt may contain many gas bubbles (Rehren, 2000), mainly consisting of CO_2 and SO_2 which are formed through dissociation of carbonates and sulphates (Amadori et al., 2018). These are mentioned as reaction products between the glass melt and the applied refractories. Indeed, bubbles were also important historically. In antiquity, blowing glass (perhaps with a pipe) made it possible to not only produce simple round spheroidal shapes but also to create other thin layered structures around the bubble (Figure 4b). The interspersed porosities caused by means of bubbles in millimetre or micrometre size have been formed through two main processes. Firstly, they are products of the high temperature reaction of raw materials (boiling). Later, a second group of bubbles can appear and are formed during the cooling process within viscose state.

The high amounts of Na_2O and lower amounts of MgO (network modifier) in the glass charge causes its viscosity to remain elevated even at high temperatures (Freestone et al., 2000; Herb and Willburger, 2016). Therefore, the structures appear by spherulitic growths of semi-crystals (Figure 4c). The layers formed over each other are caused by exfoliation of glass which might be the result of a burial environment over time (Freestone and Middleton, 1987). The bubbles formed within the structure of the glasses are a result of high boiling temperatures and then rapid cooling of the liquid, causing them to become trapped in the matrix (Eramo, 2005). In this regard, figure 4d shows clearly non-uniform glass structures on the surface and smooth uniform structures within the core. The intensity of the borders may be evidence of contamination of heavy metal ions through leaching. In essential parts, the structure of glass has less density due to the immigration of ions and the iridescence effect. Regarding the structures that are formed within the glassy matrix, the estimation of refractory temperature would be of interest. High temperature reactions cause the developed boundaries toward their centre (Figure 4e). It has been supposed that each two-dimensional connection resulted in angles of approximately 120° (Wakai and Aldinger, 2003). However, if the total number of boundaries around a grain is less than six, each boundary must concave inward (Figure 5). This process can be observed through shrinkage and eventually disappearance of the boundaries during sintering. Small grains will thus have less than six boundaries and will grow within the matrix more rapid and periodically.

5. Conclusion

As stated at the beginning of this article, despite the fact that the city of Istakhr suffered from many conflicts and wars during the early Islamic period, it experienced a period of economic growth in the eighth and ninth centuries AD. During this period, it seems that some of the city's products, including its moulded pottery, flourished. The glass sherds introduced in this article also have high quality and special construction method. If these pieces of glass are part of the products made in the city of the Istakhr itself, it can be considered as a tradition of Istakhr glassmaking, especially due to the use of soda in the manufacturing process.

The glass fragments analysed here showed many varieties regarding their external shape as well as their application and colour. Due to the environmental characters of the location of excavation, they reflect essentially corroded factors, such as iridescence, decolouring, encrustation, pitting corrosion on the surface, cracking and exfoliation.

Seven glasses have been analysed to characterize their chemistry and micro-structure. Based upon many other studies, it is typically expected that the composition of ancient glasses reflects the entire batch used in their production. The glasses here are characterized as soda-lime-silica glasses. The high amount of Na_2O (10.9-45.3 wt.%) as soda was seemingly used as flux for decreasingthe temperature of glass workability. CaO showed to have a particularly constant amount. This can be achieved by the same recipes which have been applied in these workshops. The manufacturing process of glasses in Istakhr can be interpreted as a total batch melting. This may be a result of testing recipes and raw material control. The viscosity of the batch in this case study is directly induced by high amount of soda even at high temperatures. Finally, the existence of gas bubbles found particularly within theglass sample should provide an interpretationfor the highest temperature which the melt has reached.

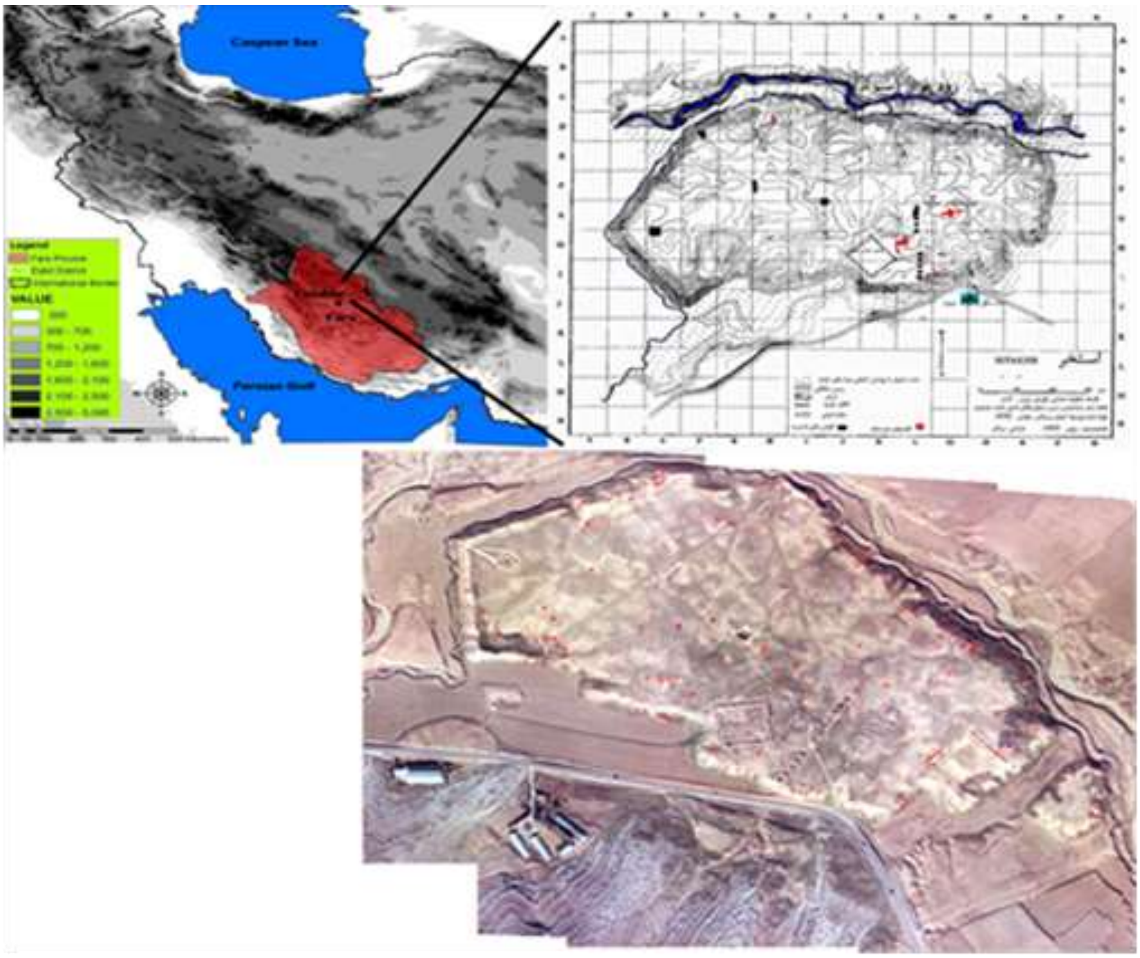

Figure 1. The location of the city of Istakhr on the Marvdasht plain.

Figure 2. Investigated glass groups from Istakhr excavation in 2012.

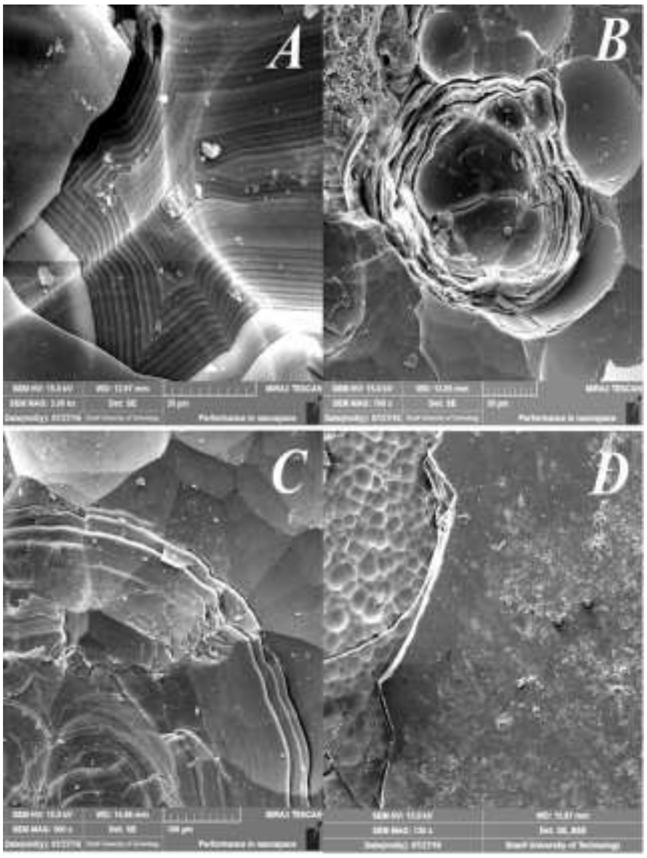

Figure 4.

a) Multi-layer structure of viscous glass texture. b) Multi-layered structures formed from the bubbles within the structure. c) Spheroid growth of crystals within the texture. d) Shelly layers of the corroded surface and the smooth uniform structure of glass beneath.

b)

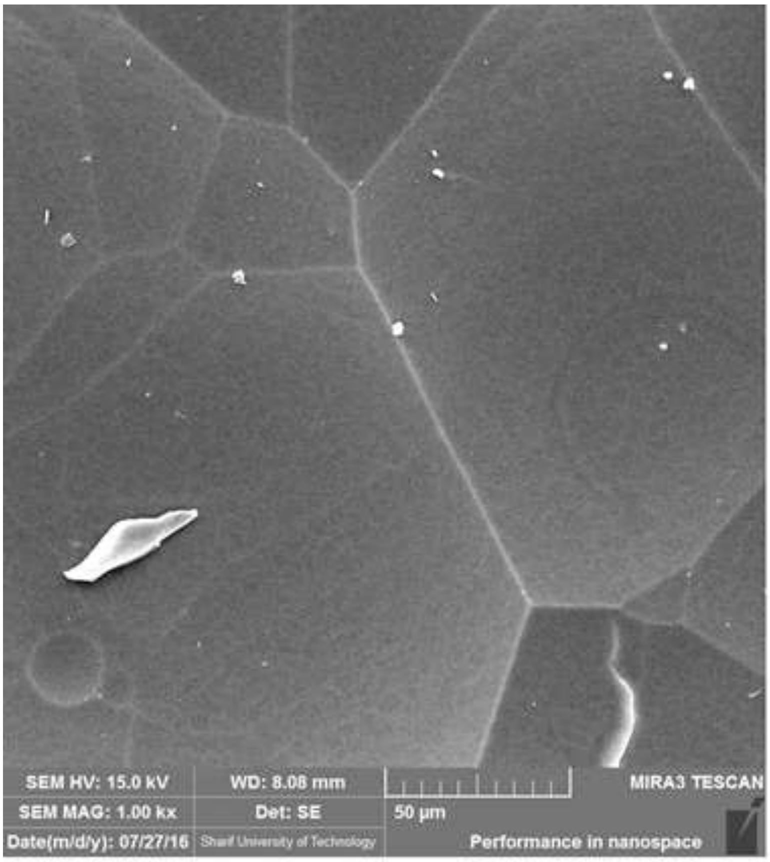

Figure 5. Evaluation of boundaries within the glassy matrix which its assessment with less than six boundary lines.

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