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THE MAKING OF BLACK
GLASS IN LATE ROMAN
DOCLEA, MONTENEGRO

IZRADA CRNOG STAKLA U
KASNO-RIMSKOJ DOKLEJI,
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Introduction

Doclea, one of the most important Roman and Late Roman archaeological sites in Montenegro, is situated close to the modern capital of the country, Podgorica (Fig. 1). In the centre of the ancient town, in the immediate vicinity of the *forum* and just across the small *thermae*, the capitol temple of *Doclea* was discovered in 2009. The fieldwork in this area continued in 2010 and 2011. By the early/ mid 4th c. AD the temple had lost its significance and had been abandoned. The space was then transformed into a workshop area and continued its life in a different form. This paper presents preliminary results of the analytical work done on glass fragments related to local secondary glass working which took place in the centre of the Late Roman town.

The above mentioned temple complex or building IX (Fig. 2) is located in a dominant position of the town, on the east side of the *forum*, on the intersection of the *decumanus* and *cardo* streets. The entire building is circa 50 m (east-west) x 40 m (north-south) large. In the middle of the north part of the building, a *cella* is raised with a preserved size of 7 x 9.5 m. In front of the *cella* a large *atrium*, paved with high quality stone slabs is surrounded to the west, east and south by a number of different size rooms. On the western and southern sides of the rooms there is a porch, while

Uvod

Dokleja, jedan od najvažnijih rimskih i kasno rimskih arheoloških nalazišta u Crnoj Gori, nalazi se blizu glavnog grada Podgorice (sl. 1). U središtu antičkog grada u neposrednoj blizini gradskog foruma, a preko puta malih termi 2009. godine otkriven je kapitolni hram Dokleje. Terenska istaživanja u ovoj oblasti nastavljena su 2010. i 2011. godine. Početkom odnosno sredinom IV vijeka n.e. hram gubi na značaju i biva napušten. Prostor se transformiše u radioničarski kvart i život nastavlja u znatno izmjenjenom obliku. Ovaj rad predstavlja preliminarne rezultate analitičkog rada izvršenog na fragmentima stakla koji se dovode u vezu sa sekundarnim staklarskim aktivnostima koje su se odvijale u središtu rimskog grada.

Gore pomenuti hramovski kompleks odnosno objekat IX (sl. 2), nalazi se na dominantnoj poziciji



Fig. 1. Location of Doclea site in Western Balkan region
Sl. 1. Lokacija arheološkog nalazišta Dokleja, na Zapadnom Balkanu

on the eastern side the existence of the porch is still not confirmed. Based on stylistic characteristics and the dimensions of the *cella*, it is assumed that the *Doclea* temple probably had a four-columned *prostilos* (Baković 2011: 15-17).

The temple is dated to the period after the 1st c. to the middle of the 4th c. AD (Baković 2011: 18-21), when it was finally abandoned. Subsequently, during the second half of the 4th and throughout the 5th century, certain repairs took place in the south-eastern part of the complex. During that period one of the rooms of the building was transformed into a small scale glass workshop (Fig. 2). In the room 3/IX (with dimensions of 8 x 6.90 m) were discovered the remains of a pot furnace, a possible annealing oven (a construction with *hypocaust*), and glass blowing waste (moils, collars, test pieces) and fragments of raw glass chunks (Živanović 2011: 41-48, sl.7.1, Fig.8). The archaeological material showed that the workshop was active for a limited period during the second

antičkog grada, na mjestu presjeka glavnih ulica *cardo* i *decumanus*, a sa istočne strane foruma. Cijela građevina ima dimenzije oko 50 m (istok-zapad) x 40 m (sjever-jug). Na sredini sjevernog dijela objekta izdignuta je cela hrama sačuvanih dimenzija 7 x 9,5 m. Ispred cele se nalazi prostrani atrijum popločan kvalitetno obrađenim kamenim pločama kojeg sa zapada, istoka i juga okružuje slijed prostorija različitih dimenzija. Na zapadnoj i južnoj strani ispred ovih prostorija nalazi se trem, dok sa istočne strane on još uvijek nije potvrđen. Na osnovu stilskih karakteristika i dimenzija cele pretpostavlja se da hram Dokleje najvjerojatnije pripada tetrastilnom prostilosu (Baković 2011: 15-17).

Hram je datovan u vremenu nakon I vijeka pa do sredine IV (Baković 2011: 18-21), kada je konačno napušten. Naknadno, tokom druge polovine IV i V v. n.e., na jugo-istočnom dijelu kompleksa su uslijedile prepravke i tom prilikom jedna od prostorija objekta je transformisana u staklenu radionicu malog kapaciteta proizvodnje



Fig. 2. Plan of building IX of Doclea site with the find spots of the analysed fragments in the north-eastern part of the complex, and location of the glass workshop in room 3/IX
Sl. 2. Plan objekta IX, Dokleja, sa mjestima nalaza analiziranih fragmenata u sjevero-istočnom dijelu kompleksa, i lokacija staklarske radionice u prostoriji 3/IX

half of the 4th or the 5th c. AD, and possibly the early 6th c. AD.

The glass finds – selection, description and context

Doclea glass and its characteristics have been recorded in archaeological studies from the end of the 19th century onwards, but only recently found evidence started the debate on local glass production. In particular, significant research conducted in 2009, 2010, and 2011 provided a large amount of glass debris, including small glass chunks, moils, droplets, test pieces, fragments of glass-melting crucibles, etc.

Collaboration between Museums and Galleries of Podgorica and the UCL Institute of Archaeology led in 2011 to an initial selection of nearly 40 samples of glass fragments and glass working waste for analysis in the Wolfson Archaeological Science Laboratories at the UCL Institute of Archaeology. Here we present preliminary observations and results obtained from a small sub-set of 5 samples. The main criteria for choosing these particular samples is the adhering ceramic, i.e. a strong evidence that these fragments are related to the glass melting process, as well as the colour of the glass – dark-green/black in four of the five samples. Furthermore, the selected pieces are linked to each other also in their distribution in the north-eastern part of building IX, as became apparent by mapping the find spots (Fig. 2).

Therefore, the finds discussed here do not originate directly from the glass workshop. They have been found during excavations in 2010 and 2011 in the north-eastern corner of the *atrium* (rooms 5/IX and 8/IX). The cultural layer they originate from is composed of dark brown earth, fragments of roof tiles and crushed stone, and is characterized by an abundance of archaeological material. In that area the mentioned layer is formed unequally, and from the middle of the room 5/IX it rises slowly to the north and gets most massive in the room 8/IX. It is dated by small finds and coins to the 4th and 5th c. AD, even though among the numismatic material a 3rd century coin is also found (Baković 2011: 20). The formation of this layer is most likely due to the

(sl. 2). Naime u prostoriji 3/IX, dimenzija 8 x 6,90 m, otkriveni su ostaci ložišta staklarske peći, pretpostavljene peći za hlađenje gotovih proizvoda (konstrukcija s hipokaustom) kao i otpad od proizvodnje stakla duvanjem (kaplje, djelovi stakla koji se bacaju sa lule, komadi za testiranje) i ulomci komada sirovog stakla (Živanović 2011: 41-48, sl.7.1, sl.8). Arheološki materijal je pokazao da je radionica bila aktivna tokom druge polovine IV i V vijeka i krajnje nesigurno početkom VI v. n.e.

Nalazi stakla – selekcija, opis i kontekst

O staklu Dokleje i njenim osobenostima pisalo se u arheološkoj nauci počev od kraja XIX vijeka, ali tek nedavno pronađenim dokazima, započete su rasprave o lokalnoj produkciji istog. Naročito značajna istraživanja, sprovedena 2009., 2010. i 2011. godine, dala su veliki broj fragmenata staklenog materijala među kojim i ulomke staklenog otpada tačnije ostataka od staklarskih aktivnosti (komadi stakla, rastopljeni stakleni otpaci, kaplje - probni uzorci, ulomci keramičkih posuda za topljenje stakla itd.).

Saradnja JU Muzeji i galerije Podgorice i UCL Instituta za arheologiju, 2011. godine, dovela su do prvobitnog odabira od skoro 40 uzoraka fragmenata stakla i staklenog otpada koji su dati na analizu u Wolfson arheološkim naučnim laboratorijama na UCL Institutu za arheologiju. U ovom radu, ćemo predstaviti preliminarna zapažanja i rezultate jednog manjeg podskupa od 5 uzoraka. Glavni kriterijum za odabir upravo ovih primjeraka jeste prilijepljena keramika, to jest čvrst dokaz da su ovi ulomci u vezi sa procesom topljenja stakla, kao i boja stakla – tamno zelena/crna u četiri od pet uzoraka. Nadalje, odabrani komadi su povezani jedni s drugima i na osnovu njihove distribucije u sjevero-istočnom dijelu objekta IX, što je postalo očigledno mapiranjem mjesta pronađenih nalaza (sl. 2).

Dakle, nalazi o kojima se ovdje raspravlja ne potiču direktno iz radionice stakla. Oni su pronađeni tokom iskopavanja iz 2010 i 2011. godine u sjevero-istočnom uglu atrijuma i sjeverno od njega (prostorije 5/IX i 8/IX). Kulturni sloj iz kojeg potiču nalazi se sastojao od tamno mrke zemlje,

accumulation of waste in Late Antiquity, when the temple was abandoned. The temple abandonment is shown in the spreading of the layer directly on the sidewalk with a channel in the *atrium* of the building IX, and also proved by a grave discovered in room 8/IX, dated to the 4th – 5th c. AD and dug into this layer (Baković 2011: 19-20, Fig. 1). It is possible that this area was used as a junkyard of extensive living and glass workshop production, which is reinforced by the analysed samples. Based on the stated facts, the finds can be more generally dated to the period between the 4th and 6th c. AD.

Using the numbering of the samples from the initial selection, a brief description of the five pieces analysed so far is given below.

Sample 36 (Fig. 3/1) is a wall fragment of a ceramic vessel used as a crucible for glass melting/modifying. The vessel is made of refined clay with

ulomaka krovne opeke, usitnjenog kamena i karakteriše ga obilje arheološkog materijala. Na tom prostoru spomenuti sloj je formiran neravnomjerno i on se od sredine prostorije 5/IX polako uzdiže sjeverno i dobija najveću masivnost u prostoriji 8/IX. Datovan je analogijom nalaza i novcem u IV i V vijeku iako je među numizmatičkim materijalom pronađen i novac III v. (Baković 2011: 20). Nastanak sloja se najvjerojatnije treba objasniti gomilanjem otpada u kasnoj antici kada je hram napušten. Da je hram tada bio napušten pokazuje prostiranje sloja direktno na pločnik sa kanalom u atrijumu objekta IX a isto tako i otkriveni grob 1/2010 u prostoriji 8/IX, oprijedjeljen u IV vijek a ukopan u opisani sloj (Baković 2011: 19-20, T1/fig.1). Nije isključeno da je ovaj prostor poslužio kao smetlište opsežnog života i rada staklarske radionice čemu u prilog idu i analizirani uzorci koji se na osnovu iznesenih

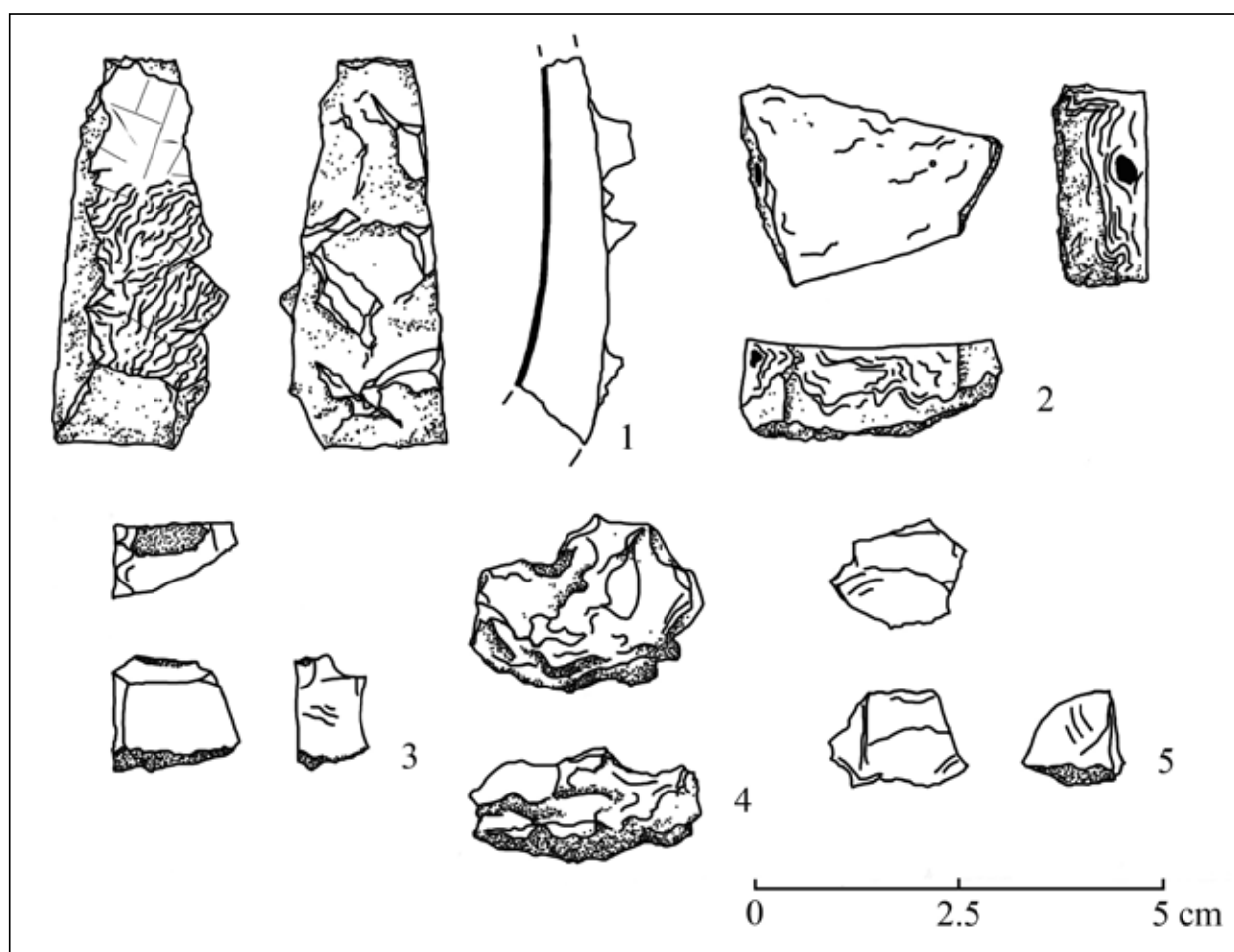


Fig. 3. Glass melting/ colouring related finds from building IX of Doclea site: 1) a crucible fragment, sample 36; 2) a piece of black appearing glass, sample 17; 3) a chunk of dark green glass, sample 19a; 4) a chunk of black appearing glass, sample 19b; 5) a chunk of aqua glass, sample 28

Sl. 3. Nalazi koji su povezani sa topljenjem/ bojenjem stakla pronađeni iz objekta IX, Dokleja: 1) fragment posude za topljenje stakla, uzorak 36; 2) komad "crnog" stakla, uzorak 17; 3) komad tamno zelenog stakla, uzorak 19a; 4) ulomak "crnog" stakla, uzorak 19b; 5) ulomak "akva" stakla, uzorak 28

dark red to dark grey colour. Its internal surface is covered by a thin layer of glass – smooth and transparent on the upper part of the fragment, and more heterogeneous and appearing black and opaque on the lower part. The external surface of the fragment is not preserved, but irregular pieces of broken dark-green glass adhering to it suggest contact with the glass melt also from the outside.

Sample 17 (Fig. 3/2) is a broken piece of black appearing glass which cooled on a ceramic surface. Its upper part is flat and the glass shows a certain degree of inhomogeneity, including small quartz particles. Visually this glass is very similar to the remains preserved on the internal lower part of the crucible fragment. Sampling a small and thinner fragment from that piece helped to recognise its true dark-green colour. Underneath the glass a tiny layer of dark grey ceramic is partly preserved, probably part of clay of a furnace, since its irregular appearance does not allow interpretation as a vessel wall/ crucible.

Sample 19a (Fig. 3/3) is a small chunk of dark-green transparent glass with an adhering layer of light-yellowish refined clay on one side, and a small area with traces of dark grey clay on the other side. Sample 19b (Fig. 3/4) is a chunk of black appearing glass cooled between irregular and fragile layers of light brown clay with small quartz particles and traces of vitrification and bloating. Similarly to samples 17 and 19a it is difficult to relate this piece to crucible glass melting/ modifying, since the ceramic resembles more furnace clay lining than a vessel wall.

Sample 28 (Fig. 3/5) is also a small chunk of glass with an adhering layer of fine white clay; the piece differs from the other four samples by its clean aqua colour and transparency of the glass.

The finds were selected as a small set for initial analysis to address certain questions. Obviously all five samples are waste of glass melting, most probably local secondary re-melting of pre-existing chunks and/or cullet. However, the dark-green/black colour of most of them is unusual, and could come from that pre-existing material, but also could be a result of colouring that took place locally. The analyses we present here are aimed to determine the additives used to colour the glass,

činjenica datuju u vrijeme između IV i VI vijeka.

Uz korišćenje numerisanih uzoraka iz prvobitne selekcije, slijedi kratak opis pet analiziranih primjeraka.

Uzorak 36 (sl. 3/1) je ulomak trbuha keramičke posude koja je služila za topljenje stakla. Posuda je izrađena od prečišćene gline tamno crvene do tamno sive boje pečenja. Njena unutrašnja površina je prekrivena tankim slojem stakla - gladak i providan na gornjem dijelu fragmenta, i više mutan, crnkast i neproziran na donjem dijelu. Spoljna površina fragmenta nije sačuvana, ali nepravilni komadi razbijenog tamno-zelenog stakla koji su prilijepljeni za nju sugeriraju kontakt topljenja stakla i sa spoljašnje strane posude.

Uzorak 17 (sl. 3/2) je slomljeni komad stakla naizgled crne boje hlađen na keramičkoj površini. Njegov gornji dio je ravan i staklo pokazuje određeni stepen nehomogenosti, uključujući male čestice kvarca. Vizualno, ovo staklo je veoma slično ostacima sačuvanim na unutrašnjem donjem dijelu fragmenta posude za topljenje stakla. Uzimanje uzoraka manjeg i tanjeg fragmenta tog komada pomoglo je prepoznavanju njegove prave tamno-zelene boje. Ispod stakla, mali sloj tamno sive keramike je djelimično očuvan, vjerovatno dio gline sa peći, jer njegov nepravilan izgled ne dopušta tumačenje kao keramičkog ulomka posude za topljenje stakla.

Uzorak 19a (sl. 3/3) je mali komad tamno-zelenog prozirnog stakla sa prilijepljenim slojem prečišćene svijetlo-žučkaste gline s jedne strane, i male površine s tragovima tamno sive gline na drugoj strani. Uzorak 19b (sl. 3/4) je komad naizgled crnog stakla ohlađenog između nepravilnih i poroznih slojeva svijetlo smeđe gline sa malim kvarcnim česticama i tragovima vitrifikacije i ispupčenja. Slično uzorcima 17 i 19a teško je povezati ovaj komad sa posudom za topljenje/ oblikovanje stakla, budući da keramika više nalikuje glini sa peći koja je njome obložena, nego na zid posude.

Uzorak 28 (sl. 3/5) je takođe mali komad stakla sa prilijepljenim slojem fine bijele gline; komad se razlikuje od ostala četiri uzorka po svojoj čistoj vodenoj boji i providnošću stakla.

Primjeri nalaza selektovani su kao mali skup za

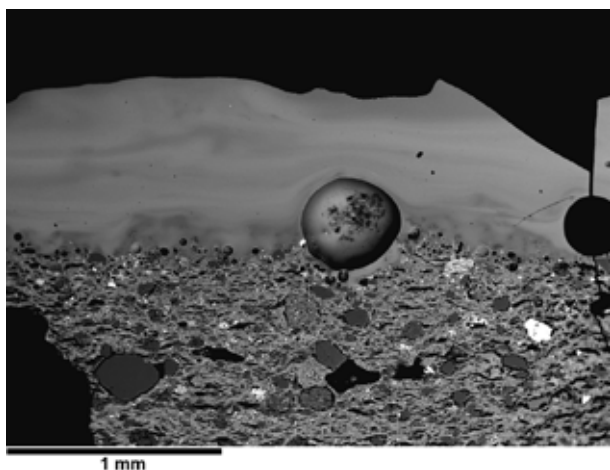


Fig. 4. Back-scattered electron image of the inhomogeneity of the glass layer (schlieren) inside the crucible (sample 36)
Sl. 4. BSE slika nehomogenosti sloja stakla (schlieren) unutar posude za topljenje (uzorak 36)

and to shed light on the character of the process that has been used by the *Doclea* craftsmen (i.e. glass melting only, or glass colouring). Then, the composition of the glass can explain whether and how the finds are related to each other and what the possible origin of the glass is. At this stage of our research analysing the ceramics in the samples is not an objective, but certainly will become part of future work.

Methods

Small fragments of samples 36, 17, 19a, 19b and 28 were mounted in epoxy resin and prepared as polished blocks. The samples were studied by optical microscopy (OM) and scanning electron microscopy (SEM), and their chemical composition analysed by energy dispersive spectrometry (EDS) using the JEOL 8600 instrument at the Wolfson Archaeological Science Laboratories. Two to five area analyses and/or spot analyses were done on each sample/ particular phase in order to understand the bulk glass composition and to identify different structures found in the glass. The results were then averaged to obtain a composition representative of the sample. These average values are reported in Table 1 and Table 2; for oxides found to be present below 0.5 – 0.2 wt% it is believed that reliable quantification is not possible due to the detection limits of the EDS technique.

početne analize kako bi se dobili odgovori na određena pitanja. Očito je da su svih pet uzoraka otpad od topljenja stakla, najvjerovatnije od lokalnog sekundarnog i ponovnog topljenja već postojećih sirovih komada i/ ili staklenih otpadaka. Međutim, tamno-zelena/ crna boja dominantna za većinu fragmenata je neobična, a možda voditi porijeklo iz tog već postojećeg materijala, ali takođe može biti i rezultat bojenja koje se desilo na lokalnom nivou. Analize koje ćemo predstaviti ovdje imaju za cilj određivanje primjesa korištenih za bojenje stakla, a da stave naglasak na karakter procesa koji je korišten od strane zanatlija Dokleje (tj. samo topljenje ili bojenje stakla). Zatim, sastav stakla može objasniti da li je i kako su uzorci međusobno povezani i koje je potencijalno porijeklo stakla. U ovoj fazi našeg istraživanja analiza keramike u uzorcima nije cilj, ali zasigurno će postati dio budućeg rada.

Metode

Mali fragmenti uzoraka 36, 17, 19a i 19b i 28 su postavljeni u epoksidne smole i pripremljeni kao polirani blokovi. Uzorci su izučavani optičkom mikroskopijom (OM) i pomoću skenirajuće elektronske mikroskopije (SEM), a njihov hemijski sastav je analiziran energetska disperzivnom spektrometrijom (EDS) pomoću JEOL 8600 instrumenta u Wolfson arheološkim naučnim laboratorijama. Tri do pet područnih analiza i/ ili analize mjesta je urađeno na svakom uzorku/ u posebnoj fazi kako bi se shvatio opseg sastava stakla i identifikovale različite strukture pronađene u staklu. Rezultati su potom uprosječeni kako bi se dobio sastav svakog uzorka. Ove prosječne vrijednosti su predstavljene na tabeli 1 i tabeli 2 ; za okside za koje se utvrdilo da su prisutni ispod 0,5 - 0,2 wt% se vjeruje da pouzdana kvantifikacija nije moguća zbog granica detekcija EDS tehnike.

Rezultati

Kompoziciono, svi uzorci pripadaju soda-kreč-silikatnoj porodici stakla koja se proizvodi sa

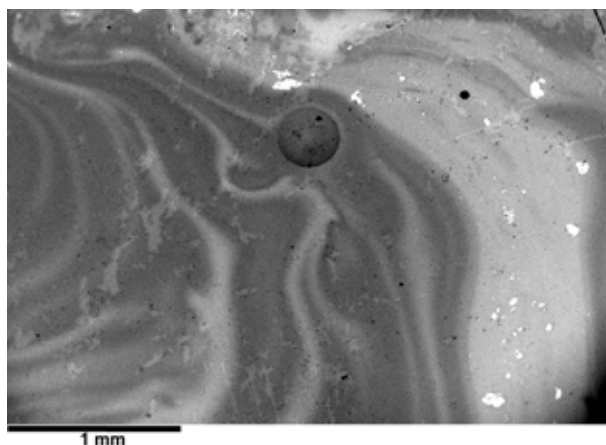


Fig. 5. Back-scattered electron image of the inhomogeneity of the glass (*schlieren*) in sample 17; lighter areas have elevated iron oxide values and contain small iron oxide particles nearly dissolved in the glass melt

Sl. 5. BSE slika nehomogenosti stakla (*schlieren*) u uzorku 17; svjetlije površine imaju vrijednosti oplemenjenog oksida gvožđa i sadrže male čestice gvožđe oksida skoro rastopljene u staklenu masu

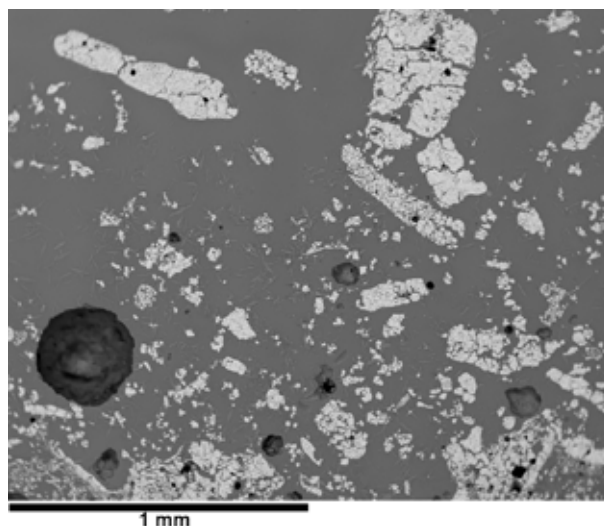


Fig. 6. Back-scattered electron image of iron oxide particles in the glass in sample 17

Sl. 6. BSE slika čestica gvožđe oksida u staklu uzorka 17

Results

Compositionally all the samples belong to the soda-lime-silica glass family produced with mineral natron, as it is expected for the Roman/ Early Byzantine period. The dark-green/ black colour of most of the finds is due to the high amount of iron oxide, most likely intentionally added to the glass (from about 3.5 wt% up to 10% and even higher in certain areas). Sample 28 contains about 0.5 wt% FeO, which is a value due to the natural content of iron oxide in the sand used to produce the glass. OM and SEM observations confirmed the conclusion from the initial visual inspection that the glass in samples 17, 19b and 36 (on the inner lower part of the crucible wall) is not homogeneous.

The crucible fragment (sample 36) represents homogeneous glass with iron oxide levels at about 4 wt% in the broken layer on the outside and on the inner upper part of the vessel wall. However, on the inner lower part the glass is heterogeneous, with well visible *schlieren* in it (Fig. 4) showing incomplete mixing of the melt. In the areas with higher concentration of iron oxide the levels are about 6 wt% FeO and more. Elevated values of alumina and magnesia are also observed in certain measurements, probably because of the contact with the ceramic,

mineralnim natronom, kao što se i očekuje za rimsko/ ranovizantijsko doba. Tamno-zelena/ crna boja u većini nalaza je nastala uslijed velike količine oksida gvožđa, najvjerovatnije namjerno dodatom staklu (sa oko 3.5 wt% do 10%, a još više u određenim područjima). Uzorak 28 sadrži oko 0,5 FeO wt%, što je vrijednost prirodnog sadržaja oksida gvožđa u pijesku koji se koristi pri proizvodnji stakla. OM i SEM istaživanja su potvrdila zaključak koji je donesen na osnovu vizualne observacije da staklo u uzorcima 17, 19b i 36 (na unutrašnjem donjem dijelu ulomka posude za topljenje stakla) nije homogeno.

Fragment posude za topljenje stakla (uzorak 36) prikazuje homogeno staklo sa nivoima gvožđe oksida na oko 4%wt u slomljenom sloju na spoljašnjem i na unutrašnjem gornjem dijelu zida posude. Međutim, na unutrašnjem donjem dijelu staklo je heterogeno, sa dobro vidljivim *schlieren* u njoj (sl. 4) i pokazuje nepotpuno miješanje rastopljenog stakla. U područjima sa visokom koncentracijom gvožđe oksida stepeni su oko 6 wt% FeO i više. Povišene vrijednosti glinice i magnezijum oksida su također primijećene u pojedinim mjerenjima, vjerovatno zbog kontakta s keramikom, ali nisu povezane sa područjima visoke koncentracije gvožđe oksida.

Nepotpuno miješanje je jak pokazatelj da je primjesa za bojenje stakla (gvožđe oksid) dodata

but not linked to the areas of high iron oxide.

The incomplete mixing is a strong indication that the glass colorant (iron oxide) was added to the glass in this crucible, and was not already present in the glass when it was put into the crucible for (re-) melting. The incomplete mixing can be explained in two ways; either as an attempt of the glassworker to avoid stirring the glass close to the bottom of the crucible since the touch of his tool to the wall of the vessel can cause a crack or breakage. An alternative probable explanation is that the process of melting was not entirely completed, but possibly interrupted by the breakage of the vessel. That would explain the external thick glass layer and its broken outer surface, which can result from a leakage of hot glass and its eventual cooling between the crucible fragment and furnace wall.

The fragment of glass cooled on a ceramic surface (sample 17) shows the highest degree of inhomogeneity compared to the rest of the samples, as well as many inclusions in the glass. The *schlieren* in the glass (Fig. 5) are stronger than those in sample 36. Areas with low iron oxide content have values of about 6 – 8 wt% FeO, while in the areas with high content this additive is present with about 18 wt% FeO in some measurements. The best evidence that the fragment is a waste of glass colouring and not just glass re-melting are numerous inclusions in the melt. Iron oxide particles with specific elongated/rounded rectangular cross section are concentrated in the lower part of the fragment, close to its ceramic base, forming a kind of residue layer (Fig. 6). Some of these inclusions are well shaped but others are partly melted in the glass (Fig. 7), and very small pieces of them are visible in the high iron oxide *schlieren* (Fig. 5) suggesting that they are the source of iron oxide intentionally added to the glass. The composition of the particles (Table 2) proves that these are nearly pure iron oxide that can contain just minor parts of one percent as traces of other metal oxides (MnO, MgO, Al₂O₃, TiO₂). Such pure material cannot be easily explained as a natural iron oxide. Moreover, the artificial man-made character of the additive is confirmed by a small metal sphere irregularly shaped and with a gas bubble inside (Fig. 8). It consists of pure metallic iron (Table 2) with traces of slag inclusions in it. Both iron oxide particles and the metallic iron sphere resemble very

staklu u posudu za topljenje stakla, i da nije bila ranije prisutna u staklu kada je stavljeno u posudu za (ponovno) topljenje. Nepotpuno miješanje može se objasniti na dva načina; bilo kao pokušaj da staklar izbjegne miješanje stakla blizu dna posude jer bi dodir njegove alatke o zid posude uzrokovao pukotine ili lom. Alternativno moguće objašnjenje je da se proces topljenja nije u potpunosti završio, već je vjerovatno prekinut uslijed loma posude. To bi objasnilo spoljni debeli sloj stakla i njegovu izlomljenu spoljašnju površinu, koji je mogao nastati curenjem vrućeg stakla i njegovog eventualnog hlađenja između fragmenta posude i zida peći.

Ulomak stakla ohlađen na keramičkoj površini (uzorak 17) pokazuje najviši stepen nehomogenosti u odnosu na ostale uzorake, kao i mnoge inkluzije u staklu. *schlieren* u staklu (sl. 5) je jači od onih u uzorku 36. Područja sa niskim sadržajem gvožđe oksida imaju vrijednosti od oko 6 - 8 wt% FeO, dok je u područjima s visokim sadržajem ovaj aditiv prisutan sa oko 18 wt% FeO u nekim mjerenjima. Najbolji dokaz da je ulomak otpad od bojenja stakla, a ne samo ponovno topljeno staklo jesu brojne inkluzije u samoj topljenoj masi. Čestice gvožđe oksida s određenim izduženim/ zaobljenim pravougaonim poprečnim presjekom koncentrisani su u donjem dijelu ulomka, u blizini svoje keramičke podloge, stvarajući neku vrstu sloja taloga (sl. 6). Neke od ovih inkluzija su dobro oblikovane, ali druge su dijelom istopljene u staklo (sl. 7), i vrlo mali komadi od njih vidljivi su u visoko gvožđe oksidu *Schlieren* (sl. 5) sugerirajući da su oni izvor gvožđe oksida, namjerno dodatog staklu. Sastav čestica (tabela 2) dokazuje da su skoro pa čist oksid gvožđa koji može sadržati samo manje dijelove od jedan posto u tragovima drugih metalnih oksida (MnO, MgO, Al₂O₃, TiO₂). Takav čisti materijal ne može se jednostavno objasniti kao prirodan oksid gvožđa. Štaviše, vještački (ljudski) karakter primjesa potvrđuju male metalne sfere nepravilnih oblika i sa gasnim mjehuom unutar (sl. 8). Sastoje se od čistog metalnog gvožđa (tabela 2) s tragovima inkluzije zgure u njemu. Obje, i čestice gvožđe oksida, i metalna gvoždena sfera veoma nalikuju na ljupe metala - tipičnom otpadu koji nastaje kao proizvod kovanja gvožđa. Njihovi oblici su u skladu s dvije poznate vrste ove kovačke mikro šljake - pahuljice i sfere. Kao rezultat hlađenja

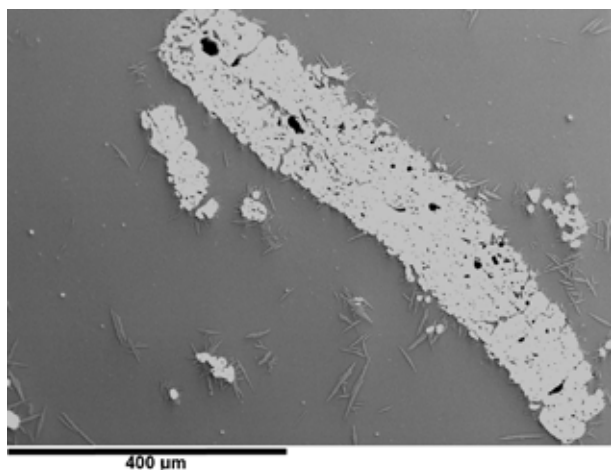


Fig. 7. Back-scattered electron image of an iron oxide flake in the glass in sample 17; it is partly melted in the glass, and pyroxene crystals are formed on it
Sl. 7. BSE slika ljuspi gvožđe oksida u staklu uzorka 17; djelimično istopljenih u staklu, i piroksenih kristala koji su se formirali oko njih

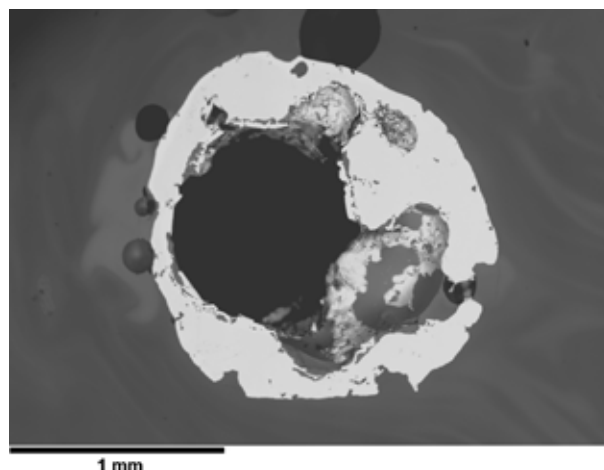


Fig. 8. Back-scattered electron image of the metallic iron sphere in sample 17; its cross-section allows seeing the irregular shape and gas bubble inside
Sl. 8. BSE slika metalne gvoždene sfere u uzorku 17; njegov presjek dopušta da vidimo nepravilan oblik i gasni mjehur unutar

much hammer scale – a typical waste product of iron smithing. Their shapes are consistent with two well-known varieties of this smithing micro slag – flakes and spheres. As a result of cooling of this high iron glass melt numerous crystals are formed in it, mainly growing on and near the iron oxide flakes (Fig. 9). The shape and composition of the crystals (Table 2) identify them as members of the pyroxene group, most probably augite crystals.

The heterogeneous and complex structure of sample 17 confirms that the fragment is a waste of glass colouring that has been carried out locally, as it is evident also from the crucible find. The overall glass composition and levels of added iron oxide in sample 17 are comparable to those observed in the crucible fragment (sample 36), and also in sample 19a – a small chunk of dark green glass with adhered ceramic on two sides (Table 1). All the oxides in the glass of these three samples, except the iron oxide, have values which proportionally do not differ much from the recipes of most Roman and Late Roman glasses, i.e. no specific oxides are positively correlated with the elevated iron oxide content. This is an indication that the source of the iron oxide used as a colourant of the melt is relatively pure – one more argument in support of the conclusion that artificial iron oxide, i.e. hammer scale is the actual raw material. The close chemical similarities between the three fragments (samples

ovog topljenog stakla sa visokom koncentracijom gvožđa su brojni kristali koji su formirani u njemu, a koji se uglavnom grupišu na ili u blizini pahulja gvožđe oksida (sl. 9). Oblik i sastav kristala (tabela 2) identifikuje ih kao članove piroksene grupe, najvjerojatnije augit kristala.

Heterogena i složena struktura uzorka 17 potvrđuje da je fragment otpad od staklarskog bojenja koje je sprovedeno na lokalnom nivou, što je očigledno i zbog pomenute posude za topljenje stakla. Ukupni staklarski sastav i nivoi dodatog oksida gvožđa u uzorku 17 su uporedivi sa onim iz posude za topljenje stakla (uzorak 36), te takođe i u uzorku 19a - mali komad tamno zelenog stakla sa prilijepljenom keramikom na dvije strane (tabela 1). Svi oksidi u staklu ova tri uzoraka, osim oksida gvožđa, imaju vrijednosti koje se proporcionalno ne razlikuju puno od većine receptata rimskog i kasnog rimskog stakla, tj. nijedan posebni oksid nije pozitivno povezan s povišenim sadržajem gvožđe oksida. To je pokazatelj da je izvor oksida gvožđa korišćen za bojenje rastopljenog stakla relativno čist - još jedan argument koji ide u prilog zaključku da je vještački gvožđe oksid, tj. ljuspe metala od kovanja zapravo sirov materijal. Bliske hemijske sličnosti između ova tri fragmenata (uzorci 36, 17 i 19a) mogu sugerisati da se odnose na jedan i isti proizvodni proces ili događaj koji je vjerovatno bio nepotpun/ prekinut kao što se može vidjeti

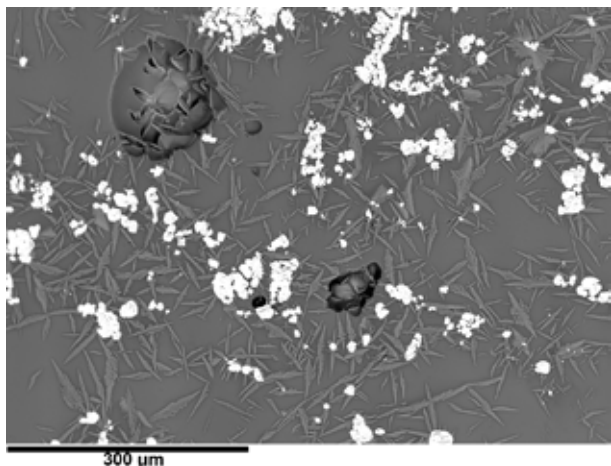


Fig. 9. Back-scattered electron image of the pyroxene crystals growing on and near the iron oxide particles in sample 17
Sl. 9. BSE slika piroksenih kristala koji su grupisani na i blizu čestica oksida gvožđa u uzorku 17

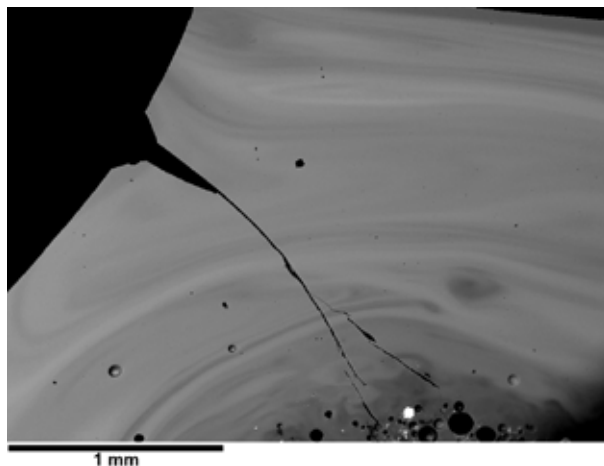


Fig. 10. Back-scattered electron image of the inhomogeneity of the glass (schlieren) and ceramic layers in sample 28
Sl. 10. BSE slika nehomogenosti stakla (schlieren) i keramičkih slojeva u uzorku 28

36, 17 and 19a) may suggest that they are related to one and the same production process or event which probably has been incomplete/ interrupted as it can be seen from the leakage and cooling of the melt, partly left inhomogeneous. However, the fact that these finds come from a secondary waste deposit but not from their original context does not allow us to see that as something more than a hypothesis.

The fragment of black glass cooled between burnt clay layers (sample 19b) resembles the above mentioned group. The melt has also same *schlieren* (Fig. 10) as marks of incomplete mixing, i.e. indicating again a process of colouration, demonstrating local production of black glass. As before, iron oxide is the reason for this colour, with values close to 10 wt% FeO. But in contrast to the glass modified with hammer scale particles, in sample 19b higher contents of alumina, titania and also manganese oxide are correlated with the elevated concentration of iron oxide (Table 1), indicating the possible use of a different source of the raw material used, probably geological magnetite.

The last find – a small chunk of transparent aqua glass (sample 28) – again has a base glass composition very close to the composition of the four black glass samples (Table 1). As mentioned above, the iron oxide content in sample 28 of about 0.5 wt% FeO is typical for its natural levels in the glass-making sand; therefore glass melting in this

iz curenja i hlađenja rastopljene staklene mase, koja je dijelom ostavljena nesjedinjena. Međutim, činjenica da ovi nalazi potiču iz sekundarnog sloja otpada, a ne iz njihovog izvornog konteksta, ne dozvoljava nam da o tome govorimo van okvira hipoteze.

Fragment crnog stakla ohlađenog između izgorjenih slojeva gline (uzorak 19b) podsjeća na gore pomenutu grupu. Stopljena masa ima isti *schlieren* (sl. 10) kao oznaku nepotpunog miješanja, tj. ponovno ukazuje na proces bojenja, demonstrirajući lokalnu proizvodnju crnog stakla. Kao i u predhodnim slučajevima, gvožđe-oksidi je razlog ove boje, s vrijednostima blizu 10 wt% FeO. No, za razliku od stakla modificiranog sa česticama metala od procesa kovanja u uzorku 19b veći sadržaji glinice, titanijum dioksida i takođe magnezijum oksida, su povezani sa povišenom koncentracijom gvožđe oksida (tabela 1), što upućuje na moguću upotrebu drugog izvora sirovog materijala, vjerovatno geološkog magnetita.

Posljednji nalaz - mali komad prozirnog "akva" stakla (uzorak 28) - opet ima osnovni sastav stakla, koji je vrlo blizak sastavu četiri crna staklena uzoraka (tabela 1). Kao što je već spomenuto, sadržaj gvožđe oksida u uzorku 28 od oko 0,5 wt% FeO je tipičan za svoje prirodne nivoe u pijesku; te stoga topljenje stakla u ovom slučaju nije imalo za cilj da proizvede crno staklo s prekomjernom količinom FeO. Nasuprot tome, staklo u uzorku 28 sadrži oko 1 wt% mangano oksida koji je puno

case has not been aimed to produce black glass with an excessive FeO amount. In contrast, the glass in sample 28 contains around 1 wt% manganese oxide which is much higher than natural MnO levels in the glass-making sand. It can be concluded that manganese oxide, a widely used raw material in the Roman and Late Roman glass industry, has been added deliberately to the batch to give a clearer and more colourless appearance of the glass. At this preliminary stage of research on *Doclea* local glass working we do not have enough arguments to say whether this decolouration was done by *Doclea* craftsmen, or whether they simply re-melted glass that already had elevated manganese oxide content. Nevertheless, the observation of similarities in the base glass composition in all five sample analysed (Table 3) can imply a constant use of the same raw glass source in the local workshop, at least as a preliminary hypothesis.

Interpretation

The data available so far enable us to suggest some initial interpretation of the activities of the glass workshop of *Doclea*. The small finds related to dark-green/ black glass originate from a mixed deposit opposite to the workshop proper; this deposit may well represent collective waste or refuse. The nature of this material closely relates it to the glass workshop.

Three of the DG/B (dark-green/black) samples appear to be closely related to each other. They all share a close association with ceramic; sample 36 is a likely crucible fragment with a thin glass film on one side and a thick broken layer of DG/B glass on the other. Samples 17 and 19a are mostly DG/B glass but with adhering ceramic, suggesting they cooled in a crucible, furnace, or crack in the furnace wall. Also chemically they are very similar. Their base glass, re-calculated without the excessive iron oxide, is very similar (Table 3, FeO re-calculated to 0.5 wt %) for all three, and shares many analogies with the widely used Roman blue-green glass type. The added iron oxide content, however, is very variable between and within these samples; it ranges from c. 3.5 – 4 to more than 8 wt % FeO, and reaches in some parts even 18 wt %.

viši od prirodnih MnO nivoa u pijesku. Može se zaključiti da je mangano oksid, naširoko upotrebljiv sirovi materijal u rimskoj i kasno rimskoj industriji stakla, dodat namjerno masi kako bi dao jasniji i bezbojniji izgled stakla. U ovoj preliminarnoj fazi istraživanja lokalne staklarske djelatnosti Dokleje, nemamo dovoljno argumenata da kazemo da li je ova dekolorizacija urađena od strane Doklejskog zanatlije, ili da su jednostavno ponovo istopili (reciklirali) staklo koje je odveć imalo povišen sadržaj mangano oksida. Ipak, primjećene sličnosti osnovnog sastava stakla u svih pet analiziranih uzoraka (tabela 3) mogu nagovjestiti stalnu upotrebu istog izvora sirovog stakla u lokalnoj radionici, barem kao preliminarnu pretpostavku.

Interpretacija

Dosada dobijeni podaci omogućuju nam da predložimo neka početna tumačenja aktivnosti staklarske radionice Dokleje. Manji nalazi u vezi sa tamno-zelenim/crnim staklom potiču iz mješovitog depozita nasuprot odgovarajuće radionice; ovaj depozit može dobro predstaviti kolektivni otpad ili šut. Priroda ovog materijala se usko odnosi na staklarsku radionicu.

Tri od TZ/C (tamno - zelena/crna) uzoraka čine se da su blisko povezani međusobno. Svi oni dijele usku povezanost sa keramikom; uzorak 36 je vjerovatno ulomak posude za topljenje s tankim staklenim slojem na jednoj strani i debelim izlomljenim slojem TZ/C stakla na drugoj. Uzorci 17 i 19a su većinom od TZ/C stakla ali sa prilijepljenom keramikom, što ukazuje da su se hladili u posudi za topljenje, peći ili u pukotini na zidu peći. Takođe, hemijski su vrlo slični. Njihov osnovni sastav stakla, ponovno izračunat bez pretjerane količine gvožđe oksida, je gotovo isti (tabela 3, FeO ponovno izračunat na 0,5 wt%) za sva tri primjerka, i dijele mnoge sličnosti sa naširoko upotrebnim rimskim plavo-zelenim tipom stakla. Dodatni sadržaj gvožđe oksida, međutim, vrlo je promjenjiv između i unutar ovih uzoraka, varira od oko 3.5 – 4 do više od 8 wt% FeO, i doseže u nekim dijelovima čak 18 wt%.

Dva opažanja su od posebnog značaja za interpretaciju ovih nalaza. Prvo, TZ/C staklo nije

Two observations are of particular significance for the interpretation of these finds. Firstly, the DG/B glass is not very homogeneous, but has numerous *schlieren* ranging from 5 wt% FeO to about 18 wt% FeO. This indicates that the DG/B glass here is not simply re-melted black glass from cullet, but that the actual coloration took place in this particular workshop. Secondly, this is further indicated by the observation of numerous hammer scale particles in one of the fragments (sample 17). We assume that these are the actual source of the added iron oxide, and not chance flakes fallen off the glassworker's tools. Such chance contamination is well known from worked glass, particularly on moils and from the internal side of bangles or the hole of pinched beads. However, these chance hammer scale particles only ever stick to the hot glass and never seriously dissolve in the molten glass. Also, they are typically isolated particles but not enough to increase the amount of FeO content of the glass melt to the levels seen here. Furthermore, the sphere of metallic iron is only consistent with proper hammer scale from smithing, probably formed during welding (Starley 1995), but not with the superficial oxidation of the glassworker's tools.

The observation of newly formed augite crystals in the immediate vicinity of the hammer scale particles is also interesting. Hammer scale consist primarily of magnetite (Fe₃O₄) (Young 2011), often with a thin outer layer of haematite (Fe₂O₃). Thus, the iron in hammer scale is predominantly oxidised, with more than 2/3 of it being present as Fe³⁺. In contrast, most of the iron in the augite surrounding the hammer scale particles is present in the 2+ valency, suggesting that the melt itself was strongly reduced. Divalent (reduced) iron is a much stronger chromophore than the oxidised trivalent iron (Weyl 1959; Pollard & Heron 1996). It would therefore have made sense for the glass worker of *Doclea* to maintain strongly reducing conditions in his glass melt when aiming to produce black glass; not least to counter-act the oxidising effect of adding magnetite/ hammer scale to the melt as a source of iron oxide. This is also consistent with the dark red to almost black colour of most of the ceramic attached to these finds, indicating strongly reducing furnace conditions.

baš homogeno, ali ima brojne *schlieren* u rasponu od 5 wt% FeO oko 18 wt% FeO. Ovo pokazuje da TZ/C staklo ovdje nije samo ponovo rastopljeno crno staklo sakupljeno od polomljenih ulomaka za reciklažu, već da je pravo bojenje sprovedeno u ovoj konkretnoj radionici. Drugo, to dodatno upućuje na razmatranje brojnih čestica - ljusti metala od kovanja u jednom od ulomaka (uzorak 17). Pretpostavljamo da su oni pravi izvori dodatog gvožđe oksida, a ne moguće ljuste otpale sa alata staklarske radionice. Mogućnost kontaminacije je dobro poznata kod konačnih staklenih proizvoda, posebno na obodima i vratovima koji se otkidaju sa staklarske lule, i sa unutrašnje strane narukvica ili kod rupica za nizanje perli. Međutim, čestice - ljuste metala samo se ponekad lijepe na vrelo staklo i nikada se u potpunosti ne rastope u izlivenu staklenu masu. Takođe, one su obično izdvojene u čestice, ali ne dovoljno da povećaju iznos sadržaja FeO u rastopljenom staklu do nivoa koji se mogu vidjeti ovdje. Nadalje, sfere metalnog gvožđa su dosljedne samo sa pravilnim ljustama metala od kovanja, vjerovatno formirane tokom kovanja-zavarivanja (Starley 1995), a ne površnom oksidacijom sa alata staklara.

Izučavanje novonastalih augit kristala u neposrednoj blizini čestica metala od kovanja takođe je zanimljivo. Ljuste metala od kovanja sastoje se prvenstveno od magnetita (Fe₃O₄) (Young, 2011), često sa tankim spoljašnjim slojem hematita (Fe₂O₃). Dakle, gvožđe u ljustama metala pretežno oksidira, sa više od 2/3 od njega je prisutno kao Fe³⁺. Nasuprot tome, najveći dio gvožđa u augit mineralu koji okružuje čestice metala od kovanja je prisutan u 2+ valentnosti, sugerirajući da je rastopljena masa sama od sebe snažno redukovana. Divalentno (redukovano) gvožđe je puno jači hromofor od trovalentnog oksidiranog gvožđa (Weyl 1959; Pollard & Heron 1996). Stoga bi imalo smisla da je staklar Dokleje održavao snažne redukcijske uslove rastopljenom staklu u nastojanjima da proizvede staklo crne boje; ne i manje važno da bi suprotno djelovao na oksidirajući učinak dodavanja magnetita/ ljusti metala rastopljenoj staklenoj masi kao izvor gvožđe oksida. Ovo je takođe u skladu sa tamno crvenom do gotovo crnom bojom većine keramike koja je prilijepljena na ovim nalazima, što upućuje na snažne redukcijske uslove unutar peći.

Sample 19b differs slightly from the other fragments analysed here. It is based on the same, or a very similar, base glass, and also shows strong *schlieren*, suggesting again local production/coloration. However, in addition to increased iron oxide, it also has elevated levels of alumina and titania correlated to the increased iron oxide content. While the increased alumina content could possibly be due to some absorption of ceramic material or soil swept up together with the hammer scale, the same cannot be said for titania. Instead, it appears more possible that the titania (and the alumina) was part of the original magnetite. This would then suggest that the magnetite was not hammer scale, formed from the hot oxidation of iron metal, but instead the mineral magnetite, which quite regularly contains certain amounts of TiO_2 in its composition. Similarly, Al_2O_3 is a relatively common component of natural magnetite. Therefore these two oxides indeed suggest the use of natural magnetite sand being used as the colorant in this case.

Conclusion

This preliminary investigation of a set of five glass working samples has given strong evidence for the local production of black glass, by intentionally adding significant amounts of iron oxide to a typical Roman base glass. The process was probably done under strongly reducing conditions, using partly hammer scale as the source of pure iron oxide, and partly geological magnetite, possibly as black sand gathered from local deposits (from a river or beach nearby). The source of the base glass used is unclear, and may have been cullet, collected from the ruins of the former temple complex. It is possible that this dark green glass, appearing black when shaped in thicker objects, was intended to be used for the production of bangles or other types of small jewellery. The presence of a versatile workshop in the centre of the town contributes to our understanding of the Late Roman and Early Byzantine regional economy, and is testament to the ingenuity and technological traditions of the local glass workers.

Uzorak 19b se u manjoj mjeri razlikuje od ostalih fragmenata koji su analizirani. On je baziran na istoj ili veoma sličnoj osnovnoj strukturi stakla, i takođe pokazuje snažni *schlieren*, što ponovo ukazuje na lokalnu proizvodnju/ bojenje stakla. Međutim, pored povećanog oksida gvožđa, takođe ima i povećane nivoe glinice i titanijum dioksida koji su povezani sa povišenim sadržajem gvožđe oksida. Dok se povećani sadržaj glinice može opravdati apsorpcijom keramičkog materijala ili zemlje sakupljene zajedno sa ljuspama metala od kovanja, isto se ne može reći za titanijum. Nasuprot, čini se prije moguće da je titanijum dioksid (i glinica) bio dio izvornog magnetita. Ovo bi dalje sugerisalo da magnetit nije od ljuspi metala, koje su nastale vrelom oksidacijom gvožđa, već mineralni magnetit, koji skoro redovno sadrži izvjesne količine TiO_2 , u svom sastavu. Slično, Al_2O_3 je relativno uobičajen sastojak prirodnog magnetita. Stoga, ova dva oksida zaista ukazuju na korišćenje prirodnog pješanog magnetita koji je korišten za bojenje u ovom slučaju.

Zaključak

Ovo preliminarno istraživanje seta od pet uzoraka stakla je dalo jake dokaze o lokalnoj proizvodnji crnog stakla, time što su namjerno dodavane značajne količine gvožđe oksida na tipično rimsku osnovnu strukturu stakla. Proces je najvjerovatnije odrađen pod jako redukcionim uslovima, uz korišćenje ljuspi metala od kovanja čekićem kao izvora čistog gvožđe oksida, a dijelom i geološkog magnetita, vjerovatno crnog pijeska sakupljenog sa obližnjih ležišta (iz rijeke ili plaže). Porijeklo osnovne strukture stakla je nejasno, i možda potiče od ulomaka polomljenih staklenih predmeta, sakupljenih za reciklažu, sa ruševina nekadašnjeg hramovskog kompleksa. Moguće je da je ovo tamno zeleno staklo, koje izgleda crno kada se oblikuje u vidu debljih predmeta, bilo namijenjeno za proizvodnju narukvica ili drugih vrsta sitnog nakita. Prisustvo raznovrsne radionice u centru antičkog grada doprinosi i našem shvatanju kasno rimske i rano vizantijske regionalne ekonomije, i predstavlja dokaz o dovitljivosti i tehnološkim tradicijama lokalnih staklara.

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Zahvalnost

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wt%	SiO ₂	Na ₂ O	Al ₂ O ₃	CaO	K ₂ O	MgO	FeO	MnO	TiO ₂	P ₂ O ₅	Cl	SO ₃	CuO	CoO	PbO	Sb ₂ O ₃	Total
Corning B measured	60.46	16.97	3.92	9.05	1.06	0.98	0.3	0.24	0.27	0.56	0.25	0.5	2.76	0.10	0.57	0.56	98.55
Corning B recommended	61.55	17.0	4.36	8.56	1.0	1.03	0.3	0.25	0.09	0.82	0.2	0.54	2.66	0.046	0.61	0.46	99.52
wt% normalised quantitative results					SiO ₂	Na ₂ O	Al ₂ O ₃	CaO	K ₂ O	MgO	FeO	MnO	TiO ₂	P ₂ O ₅	Cl	SO ₃	
sample 36 – glass on the external surface of the crucible (bulk composition)					64.7	18.4	2.3	6.6	0.6	1.0	4.2	0.7	0.1	0.01/ bdl*	1.1	0.5	
sample 36 – glass on the internal surface of the crucible/ upper part (bulk composition, mean of 3 measurements)					65.1	17.3	2.9	6.5	0.9	1.1	4.3	0.5	0.2	0.1	0.9	0.4	
sample 36 – glass on the internal surface of the crucible/ lower part, I (mean of 3 measurements)					65.5	17.8	3.3	6.6	0.6	1.1	3.4	0.5	0.2	0.05/ bdl	0.6	0.3	
Sample 36 – glass on the internal surface of the crucible/ lower part, II (mean of 2 measurements)					62.8	17.5	3.6	5.4	0.8	1.0	6.9	0.5	0.2	bdl	0.8	0.5	
sample 17 – areas with lower FeO concentration, I (mean of 5 measurements)					63.3	17.8	2.2	7.1	0.6	1.0	5.9	0.7	0.1	0.04/ bdl	1.0	0.4	
sample 17 – areas with lower FeO concentration, II (mean of 3 measurements)					61.5	16.9	2.2	7.4	0.6	0.9	8.3	0.6	0.1	0.1	0.9	0.4	
sample 17 – areas with higher FeO concentration (mean of 2 measurements)					54.5	14.5	3.1	6.6	0.6	0.9	18.0	0.4	0.1	0.1	0.8	0.2	
sample 19a – bulk glass composition (mean of 4 measurements)					65.7	17.6	2.2	7.1	0.7	1.0	3.7	0.6	0.05/ bdl	bdl	1.0	0.3	
sample 19b – areas with higher FeO concentration (mean of 2 measurements)					60.9	15.5	4.6	5.4	0.7	1.0	10.0	0.5	0.3	0.1	0.7	0.2	
sample 19b – areas with lower FeO concentration (mean of 4 measurements)					64.2	14.7	3.5	6.7	0.7	1.1	7.2	0.7	0.2	0.1	0.7	0.2	
sample 28 – bulk glass composition (mean of 4 measurements)					69.3	15.4	2.4	8.4	0.8	0.7	0.5	1.1	0.1	0.09/ bdl	1.1	0.2	

*bdl - below detection limits

Table 1. Chemical composition of the glass samples analysed and reference measurements of Corning B glass standard (SEM-EDS data)
 Tabela 1. Hemijski sastav uzoraka analiziranog stakla i referentne mjere po Korning B standardu za staklo (SEM-EDS podaci)

wt%	Fe	O			
metal sphere I	99.5	0.5			
metal sphere II	99.7	0.3			
wt% normalised quantitative results	FeO	MnO	MgO	Al ₂ O ₃	TiO ₂
iron oxide flake I (mean of 4 measurements)	98.5	0.5	0.5	0.3	0.2
iron oxide flake II (mean of 3 measurements)	99.5	0.2	0.2	0.1	traces
iron oxide flake III (mean of 4 measurements)	99.2	0.3	0.3	0.2	traces
wt% normalised quantitative results	SiO ₂	FeO	CaO	Al ₂ O ₃	MgO
pyroxene crystals I (mean of 4 measurements)	49.3	28.0	17.0	2.8	2.9
pyroxene crystals II (mean of 5 measurements)	55.6	26.5	12.5	3.2	2.2

Table 2. Chemical composition of the inclusions in the glass, sample 17 (SEM-EDS data)
Tabela 2. Hemijski sastav inkluzije u staklu uzoraka 17 (SEM-EDS podaci)

wt%	SiO ₂	Na ₂ O	Al ₂ O ₃	CaO	K ₂ O	MgO	FeO	MnO	TiO ₂	P ₂ O ₅	Cl	SO ₃	total
sample 36	67.3	18.4	2.9	6.8	0.7	1.1	0.5	0.6	0.2	bdl*	0.9	0.4	99.8
sample 17	67.2	18.7	2.2	7.6	0.6	1.0	0.5	0.6	0.1	bdl	1.0	0.4	100.1
sample 19a	67.9	18.2	2.3	7.3	0.7	1.0	0.5	0.6	bdl	bdl	1.0	0.3	99.8
sample 19b	69.4	16.8	2.5	6.7	0.7	1.2	0.5	0.6	0.1	0.1	0.7	0.2	99.5
sample 28	69.3	15.4	2.4	8.4	0.8	0.7	0.5	1.1	0.1	bdl	1.1	0.2	100

*bdl - below detection limits

Table 3. Re-calculated base glass of the sample analysed with iron oxide content reduced to the typically observed natural level
Tabela 3. Ponovo izračunata osnovna struktura stakla uzoraka analiziranih sa smanjenim oksidom gvožđa, na po pravilu izučavanom prirodnom nivou

