Journal of Archaeological Science 38 (2011) 2475–2479

Contents lists available at ScienceDirect

Journal of Archaeological Science

journal homepage: http://www.elsevier.com/locate/jas

Late Pleistocene/Early Holocene seafaring in the Aegean: new obsidian hydration dates with the SIMS-SS method

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ARTICLE INFO

Article history:
Received 11 November 2010
Received in revised form
12 May 2011
Accepted 24 May 2011

Keywords:
Obsidian hydration dating
SIMS-SS
Late Pleistocene
Early Holocene
Seafaring

ABSTRACT

Archaeological evidence regarding the presence of obsidian in levels that antedate the food production stage could have been the result of usage or intrusion of small obsidian artifacts from overlying Neolithic layers. The new obsidian hydration dates presented below employing the novel SIMS-SS method, offers new results of absolute dating concordant with the excavation data. Our contribution sheds new light on the Late Pleistocene/Early Holocene exploitation of obsidian sources on the island of Melos in the Cyclades reporting dates c. 13th millennium - end of 10th millennium B.P.

1. Introduction

Seafaring before the Neolithic (c.7th millennium B.C.) constitutes a controversial issue in Aegean Archaeology and generally in the Mediterranean (Cherry, 1990, 1992; Broodbank, 2006; Mavridis, 2003, 2007; Sampson, 2008; Sampson et al., 2010). However, current evidence from systematic research in different parts of the Aegean started gradually changing this picture and opened up new dynamics for understanding the character of exploitation and the importance of early coastal and island environments, see the example of Crete (Kopaka and Matzanas, 2009; Strasser et al., 2010), the new site of Ouriakos on the island of Lemnos (dated according to preliminary evidence to the end of the Pleistocene and possibly to the beginning of the Holocene c.12 000 B.P., N. Eustratiou, pers. com. 17/9/2010), and the new Middle Palaeolithic (~80 000–35 000 BP) site in Agios Eustratios Island (pers. com. A. Sampson). Our contribution sheds new light on the Late Pleistocene/Early Holocene (ca.12th millennium BP) exploitation of obsidian sources on the island of Melos in the Cyclades.

The main source of information for these early visits on the island of Melos comes from Franchthi cave in the Argolid (Perlès, 1987, 1990). Provenance studies of the material from this site indicated its Melian origin (Renfrew et al., 1965; Renfrew and Aspinall, 1990, 269), but obsidian hydration dating was not applied to the artifacts recovered. In the case of Franchthi cave, obsidian finds consisted of a few pieces (Perlès, 1987, 142–145, Renfrew and Aspinall, 1990), dating to the end of the Upper Palaeolithic (ca.35 000–11 000 B.P), while the use of obsidian continues during the Mesolithic (~9600–6800 B.C), rising to 3% of the lithics in Upper Palaeolithic levels (Runnels, 1995, 720, Perlès, 1999, 314, Broodbank, 2006, 208). Regarding the presence of obsidian at Franchthi, two routes have been considered as possible: a direct one of c. 120 km with islets in between and another one through Attica that included crossings of c. 15–20 km between islands (Sampson, 2002, 2010; Broodbank, 2006, 209). The presence of obsidian in mainland and island sites indicates that these exploitations included successful return journeys.

The new obsidian hydration dates presented below (Table 1), employing the novel SIMS-SS method, offer a new reliable source of absolute dating. Since the archaeological evidence of the presence of obsidian in levels that antedate the food production stage could have been the result of trade or the intrusion of younger age obsidian artifacts from the overlying Neolithic layers (see the discussion regarding Cyclops cave on the island of Youra, Sampson, 2003; Kaczanowska and Kozlowski, 2008, 172; Sampson, 2010a) that exist in all sites discussed here, the novel SIMS-SS method was employed to confirm excavation data (Sampson, 2010a).

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0305-4403/$ – see front matter
doi:10.1016/j.jas.2011.05.019

Table 1
The SIMS-SS dates.

n	Lab. n/site	Sample details	X_s (cm)	C_s (grmol/cc)	C_i (grmol/cc)	e^k	$D_{s,\mathrm{eff}}$ (cm^2/year)	Age SIMS-SS (years BP)
1	Yr–1 Cyclops cave, Youra	Trench Γα, Layer 14, Sq. 11,12, 13,14, AA 412 6-7-95	3.76e-5 ± 3.25e-6	0.001679 ± 2.66e-5	0.0004051 ± 1.10e-5	40	1.2411e-11	10968 ± 640
2	Yr–2 Cyclops cave, Youra	Trench Γ, Layer 4 A/A45, 3-7-1993	7.1699e-5 ± 1.8396e-6	0.0007738 ± 1.47e-5	0.0001589 ± 5.52e-6	220	1.120e-12	11236 ± 776
3	Yr–3 Cyclops cave, Youra	Trench Γα, Layer 7, AA 259	0.00011835 ± 4.04e-6	0.0005165 ± 8.69e-6	5.5296e-5 ± 3.18e-6	40	2.3589e-13	12017 ± 1875
4	Sar–4 Sarakenos cave, Boeotia	Trench A21, Layer T6	0.0001283 ± 3.6e-6	0.0009746 ± 1.53e-5	0.000149 ± 7.30e-6	60	4.73e-13	7921 ± 1206
5	Sar–8 Sarakenos cave, Boeotia	Trench A11 Layer T5	6.01e-5 ± 1.8523e-6	0.0008731 ± 6.98e-6	0.0001347 ± 7.13e-6	220	1.73e-12	7214 ± 327
6	Sar–9 Sarakenos cave, Boeotia	Trench A10, Layer T6	0.0001671 ± 5.73e-6	0.00084119 ± 1.8070e-5	0.0001456 ± 8.849e-6	140	7.71e-13	6985 ± 297
8	Rho–891 Kerame, Ikaria	T,C, Spit 4 Sq. 5 + 6	3.1486e-5 ± 1.87e-6	0.00129197 ± 1.79e-5	0.000106014 ± 6.24e-6	360	1.1365e-12	11085 ± 3282
9	Rho–892 Kerame, Ikaria	Trench D Spit 4	4.90433e-5 ± 2.24e-5	0.0007287 ± 2.24e-5	9.8763e-5 ± 4.44018e-6	80	3.42e-13	10152 ± 1643
10	Schisto–1 Schisto cave, Attica	Trench 1A/B Layer. 4 Spit 11,B1 N1	5.99e-5 ± 5.67e-6	0.00013830 ± 2.052e-5	0.0001538 ± 1.2851e-5	480	2.1526e-12	14539 ± 1280
11	Schisto–2 Schisto cave, Attica	Trench 2, Layer3 Spit 8, B12	3.39e-5 ± 5.31e-5	0.001494 ± 4.71e-5	0.0001714 ± 7.66e-7	680	2.833e-12	9533 ± 1198

2. The SIMS-SS obsidian hydration dating method

Since Friedman and Smith (1960) first report on the Obsidian Hydration Dating, the potential of obsidian as a chronometer in archaeology has been subjected to several drawbacks and detailed studies (Anovitz et al., 1999; Rogers, 2008; Hull, 2001) inspite the defence of this (Ebert et al., 1991; Eerkens et al., 2008).

In fact, the classical approach to obsidian hydration dating (OHD) uses optical microscopy to read hydration rims, makes approximate corrections for temperature history by calculation, and computes age from the square-root-of-time dependence of rim thickness on age. Though in several cases it seems successful (e.g. in the western United States and Australia, where most work has been made) it is not widely used by archaeologists (Liritzis and Laskaris, 2011b).

Amongst several drawbacks is the exponent of time (Stevenson et al., 1989, 1998, 2004; Anovitz et al., 1999; Liritzis, 2006). At any rate the square-root-of-time relationship is introduced also in the SIMS-SS procedure, since in the derivation of equation (1) the Boltzmann transformation introduces a variable, η, which is inversely proportional to $t^{1/2}$ (Liritzis et al., 2004:53, eq. (6); Crank, 1975:105, eq.(7).6).

In 2002 an alternative novel approach towards obsidian hydration dating, named SIMS-SS, has been initiated (Liritzis and Diakostamatiou, 2002; Liritzis et al., 2004; Liritzis, 2006), based on modelling the hydrogen profile acquired by secondary ion mass spectrometry (SIMS), following Fick's diffusion law, together with the assumption of surface saturation (SS) layer with water molecules.

The rationale of the SIMS-SS dating method is based on the modelling of the diffused water profile, especially along the first 1–10 μm. The formation of a saturation layer near the exterior of the obsidian tool surface is a crucial and basic parameter in the diffusion age modelling. In fact, since the cutting of an artifact by prehistoric man, water from ambient humidity enters rapidly and presumably perpendicularly to the obsidian's surface. In the first 1–5 μm the diffusion is faster and saturation occurs, while subsequently a slower diffusion rate continues from this layer towards the interior of the blade (Liritzis et al., 2004; Liritzis, 2006).

The age calculation procedure is separated into two major steps. The first step concerns the calculation of a 3rd order fitting polynomial of the SIMS profile. The second stage regards the determination of the saturation layer, i.e. its depth and concentration. The whole computing processing is embedded in stand-alone software created in Matlab (version 7.0.1) software package with a graphical user interface and executable under Windows XP (Liritzis, 2004; Liritzis and Ganetsos, 2006). The software uses the age equation proposed earlier (eq. (1)) (Liritzis and Diakostamatiou, 2002; Liritzis et al., 2004) in order to calculate the age in years.

$$T = \frac{(C_i - C_s)^2 \left(\dfrac{1.128}{1 - \dfrac{0.177kC_i}{C_s}} \right)^2}{4D_{s,\mathrm{eff}} \left(\left. \dfrac{dC}{dx} \right|_{x=0} \right)^2} \qquad (1)$$

In the above mentioned equation, C_i is the concentration of the intrinsic water, C_s the saturation concentration, k is a factor derived from a correlation of the non-dimensional profile with a family of curves by Crank (1975) and $D_{s,\mathrm{eff}}$ is the effective diffusion coefficient calculated using the depth of saturation layer (X_s), with errors attached the 1sigma standard deviation of respective data processing. In fact $D_{s,\mathrm{eff}}$ is empirically derived from a set of well known ages and equation (2) as the effective value of the diffusion coefficient D_s for $C = C_s$, and k is derived from the family of Crank's curves (Liritzis and Diakostamatiou, 2002). It is:

$$D_{s,\mathrm{eff}} = a^* D_s + b / \left(10^{22^*} D_s \right) \qquad (2)$$

where $D_s = (1/(dC/dx))^* 10^{-11}$ assuming a constant flux and taken as unity. The eq. (2) and assumption of unity is a matter of further investigation.

In Fig. 1 the hydration profile of sample YR-2 (Youra Island, Cyclop's Cave, Greece) is shown along with the two diffusion mechanisms and the main attributes of saturation concentration and depth, as well as the intrinsic concentration.

The SIMS-SS method includes a wide range of suitability criteria and procedures that reduce the propagation of errors and maximize the efficiency of the method (Liritzis and Laskaris, 2009, 2011a, Laskaris, 2010; Liritzis and Laskaris, 2011). These criteria along with the Taylor's error propagation statistics provide overall

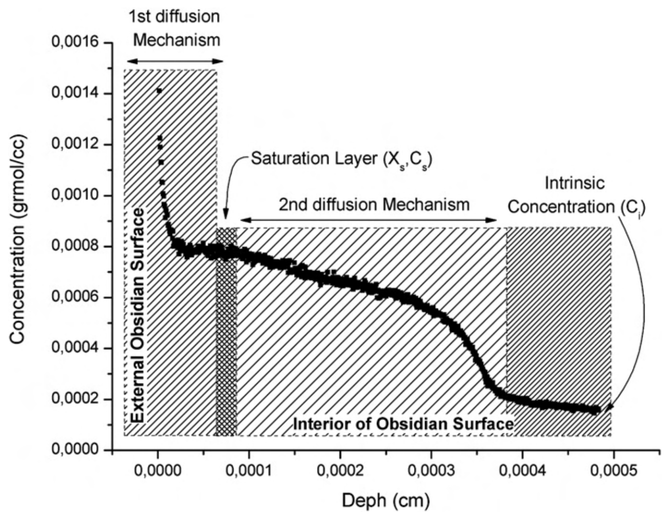

Fig. 1. Hydration profile (H^+) of sample YR-2 and demonstration of the two diffusion mechanisms. 2nd Mechanism is slower than the 1st mechanism and therefore a saturation layer is formed with attributes Xs (depth) and Cs (concentration). Ci is the concentration of intrinsic water.

accuracy. The most important criteria and procedures are the following: According to the theory of SIMS-SS method, one of the crucial points in the dating process is the location of the saturation layer. In fact, the combined factors of the faster diffusion rate of water in the obsidian surface, together with the kinetics of the diffusion mechanism for the water molecules and the specific stereochemical structure of obsidian, as well as the external conditions for the diffusion temperature, relative humidity and pressure, all result in the formation of an approximately constant boundary concentration value in the external surface layers. This is the surface saturated layer (the SS layer) (Liritzis, 2006). Assuming that the second diffusion – from the saturation layer to the interior of the obsidian – follows Fick's 2nd law of diffusion, the attributes of the layer (concentration and depth) are very important as initial/boundary conditions. Liritzis and Laskaris (2009, 2011a) developed an accurate and safe way to determine the saturation layer. This procedure is based on the combination of the first derivative of a 3rd order polynomial that models the SIMS profile along with repeated linear regressions between the data points of the profile.

The saturation attributes X_s and C_s are very important parameters in the dating process. Often the obsidian surface in the micro- and nano- scale is not smooth, a fact that influences the diffusion of water and subsequently the SIMS H^+ profile. Despite the data provided by the PLM (Polarized Light Microscopy) and SEM (Scanning Electron Microscopy) on the structure of the obsidian surface, such as the presence of crystallites in the micrometre scale, the AFM (Atomic Force Microscopy) provided more representative images of roughness and structure. The features of these images consist of cracks and voids, which were sometimes within the first 10 μm of the tool surface, that is within the expected hydration depth of about 10 000 years. Based on these assumptions Liritzis et al. (2008a, 2008b) proposed the use of SEM and AFM for the examination of the surface prior to SIMS H^+ analysis for the selection of a proper spot on it (smooth and free of inclusions) for the SIMS measurement. The hydration profile of the H^+ (as well as other useful cations) data is experimentally measured with SIMS. The SIMS method is based on the bombardment of the sample with a beam of primary ions and the measurement of the backscattered secondary ions emerged from the formed crater; the latter application is considered non-destructive in archaeolometry/archaeological science due to the fact that the area of the crater is only of a few square microns. The measured profile differs from its ideal theoretical (step function) shape and has a sigmoid (S-like) shape. In the SIMS-SS dating method this sigmoid profile is modelled with a 3rd order polynomial. The reason for this is to create a polynomial that describes best the experimental data and use it for the calculation of the effective diffusion coefficient D_S. A significant factor to this fitting is the sufficient modelling of the hydrated region and of the tail, because the hydrated region represents the present condition while the tail represents the initial non-hydrated phase of the artifact. For the efficient calculation of the best fitting polynomial Liritzis and Laskaris (2009, 2011a) proposed the use of a procedure that involves repeated polynomial fittings and leads to a polynomial that describe in the best way the hydrated and the un-hydrated region.

These new procedures and criteria were tested with a comparison of SIMS-SS ages with independent methods for a wide variety of obsidian samples from all over the world (Liritzis et al., 2008a; Liritzis and Laskaris, 2009; Laskaris, 2010). The commensurability between the archaeological expected ages and the SIMS-SS ages is quite high and reinforces the validity and wide applicability of the novel dating approach (http://www.rhodes.aegean.gr/tms/sims-ss).

3. Implications for archaeology: Early exploitation of the obsidian sources on the Island of Melos

The dates of Table 1 represent the only effort made so far for dating the obsidian artifacts, of Late Pleistocene/Early Holocene age (ca.12 000 BP), themselves. Also Table 1 includes three calculated ages for artifacts from Middle Neolithic layers (80 000–35 000 BP) of the Sarakeno Cave (Sampson, 2008b). It is suggested that the exploitation of the obsidian sources on the island of Melos could have been an even earlier phenomenon in relation to what was known so far from Franchthi cave. The rest of the dates are in accordance with the evidence from Franchthi cave, where obsidian is present from the 11th millennium B.P. levels, while its presence continues during the Lower Mesolithic (2nd half of the 10th millennium B.P.), reaching its peak in the Upper Mesolithic (Perlès, 1987, 1995, 186). The SIMS-SS dates (Table 1) from the site of Kerame in Ikaria (Sampson, 2006, 2010; Sampson et al., 2008), Youra cave in Sporades (Sampson, 2008a) and Maroulas on Kythnos (Sampson et al., 2002, 2010) manifest that during the Mesolithic a rather well established system of obsidian exploitation and circulation existed, a phenomenon that has its routes even earlier, as the SIMS-SS dates from sites such as the Schisto cave in Attica (Mavridis and Kormazopoulou, 2009) indicate. Furthermore obsidian artifacts have recently been found in two other Mesolithic sites in Greece, one in the island of Naxos and the other one in the small island of Halki, Dodecanese (Sampson personal comm). As pointed out by Perlès (1995, 186) "the economy of the Late Pleistocene and especially at the end of it presents many characteristics of a Mesolithic economy" (c. 13th millennium - end of 10th millennium B.P.). Exchange systems therefore brought obsidian in the eastern (Ikaria) and the north-west Aegean (Cyclops cave in Sporades), while it even reached coastal inland sites of mainland Greece such as Attica (Schisto cave) though not yet found in mainland sites. Possibly through sites in this latter region obsidian was also brought to the Peloponnese (see Franchthi cave and also finds from cave I in the Kleisoura Gorge, Argolid - Koumouzelis et al., 2003, 117) (see Fig. 2). It is evident that the novel SIMS-SS method has important applications in the relevant work and can aid researchers to address specific questions to their excavation data.

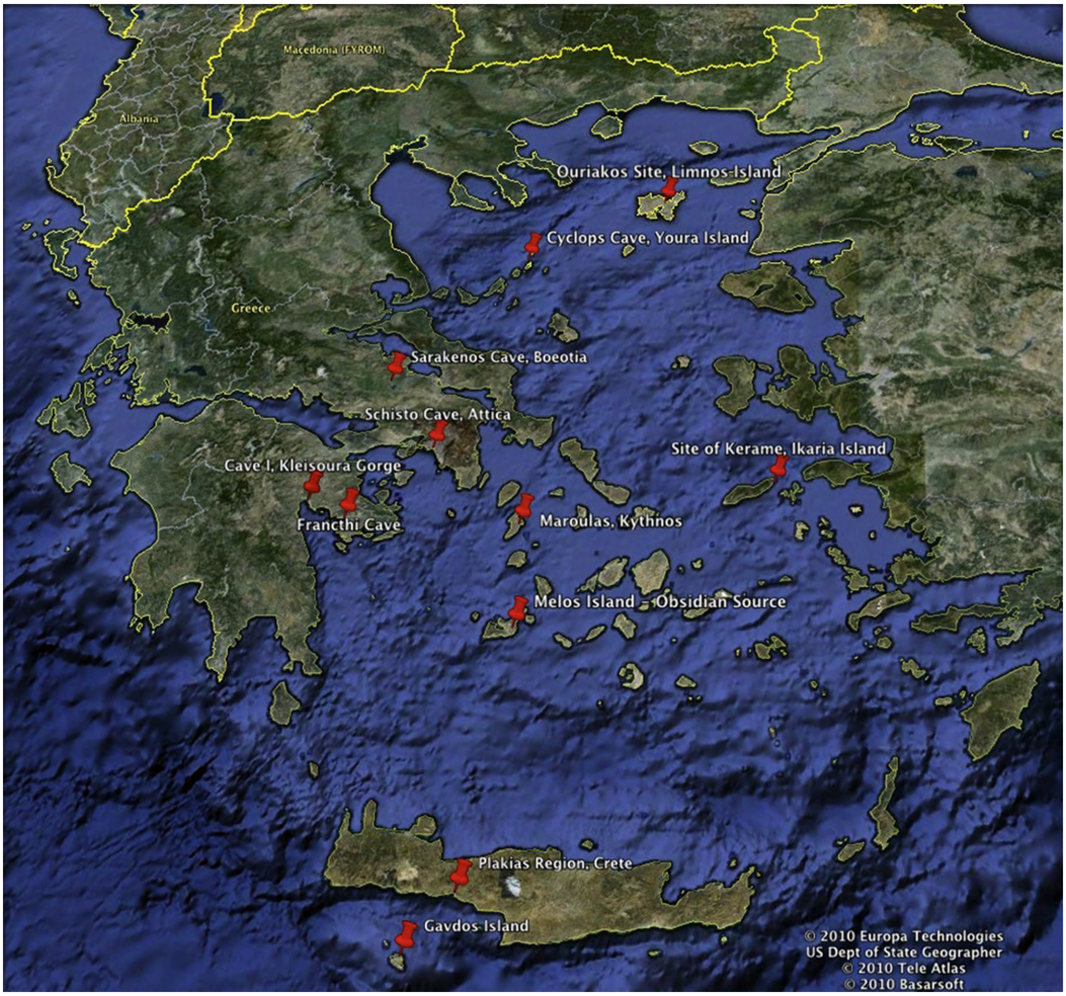

Fig. 2. Sites mentioned in the text.

Acknowledgements

We would like to thank Dr. G. Kotzamani and Dr. N. Zacharias for their useful comments on the draft of the paper. Also N.L. would like to thank Dr. Jerry Hunter and Virginia Tech for their help with Dynamic-SIMS analyses.

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