Radiogenic isotopes
in the provenance determination of raw materials
A case of lead and glass recycling at Sagalassos (SW Turkey)

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The concept of recycling as a part of the process of artefact production is well known. It is shown here that the isotopic composition of architectural lead at ancient Sagalassos (SW Turkey) reflects a thorough mixing of resources by progressive lead recycling throughout the history of the building of the city. The lead processed at Sagalassos was derived from at least two distinct sources, but the exact provenance of the raw material cannot be clearly determined. Secondary production (reworking) of glass from imperial to early Byzantine times has also been demonstrated at the ancient city, in that the Sr isotope composition of the glass and the mixing lines indicate the production of "recycled" glass from imported chunks. A Levantine origin is suggested for the blue chunks and an Egyptian origin for the HIMT chunks.

Keywords: glass, isotopes, lead, recycling, Sagalassos, strontium

Introduction

Ancient Sagalassos (Pisidia, SW Turkey; Fig. 1) has been the focus of an interdisciplinary archaeological research project co-ordinated by the Katholieke Universiteit Leuven since 1985 (Waelkens et al. 1999). Although Sagalassos may not have been much more than a provincial *primus inter pares* or a regional pole of attraction in ancient times, this fundamental reconstruction of the economy and ecology of the town within its territory has elevated our understanding of it and its inhabitants beyond the traditional practice of classical archaeology in Asia Minor (Waelkens et al. 1999).

Sagalassos appears in the historical record in 333 BC, when the town and the region of Pisidia were conquered by Alexander the Great. Pisidia witnessed a sequence of Hellenistic kings, from Antigonos Monopthalmos (321–301 BC), to Lysimachos of Thrace (301–281 BC), the Seleucids of Syria (281–189 BC) and the Attalids of Pergamon (189–133 BC). After the latter bequeathed their kingdom to Rome, the region of Pisidia was joined to the Republican province of Asia and later Cilicia. In 48/7 BC Sagalassos once more reverted to Asia, until the northern part of Pisidia was donated to the Galatian Amyntas, a client-king of Rome, by M. Antonius in 39 BC. Upon Amyntas' elimination in 25 BC, Rome decided to incorporate the region into its empire once and for all. The *Pax Romana* introduced by the soldiers of Augustus was to last for centuries, and it was only around AD 400 that Sagalassos felt a need to fortify its civic centre once more, most probably against raids of Isaurian and Gothic bands. Although the town seems to have
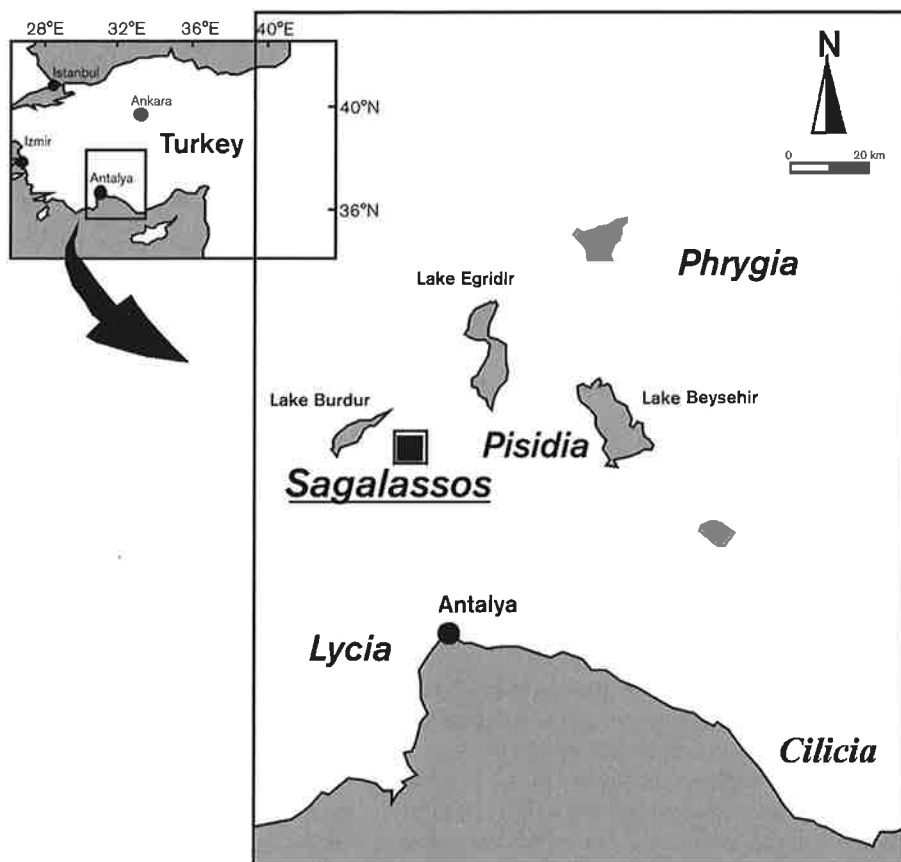
Figure 1. Location of Sagalassos in SW Turkey and ancient Pisidia.

remained relatively prosperous under these conditions, the glory of old was shattered by an early sixth century AD earthquake, after which, largely as a result of a plague in AD 541–42, the settlement may have been reduced more or less to the status of a village. The site was largely abandoned after the middle of the seventh century AD, when a dramatic series of epidemics, Arab raids and another major earthquake took their toll (Waelkens 2001). One of the major research topics at Sagalassos has focussed on the unravelling of the economic network of the town on the local, regional and supra-regional levels.

Apart from exploiting the obvious resources such as agriculture and forestry, the extensive territory of Sagalassos offered a rich variety of mineral resources (Degryse et al. 2003a). By virtue of the numerous occurrences of iron slag and bloom in the excavations, the local processing of several types of metal, including iron and lead, has been demonstrated (Degryse et al. 2003a, 2003b, 2007). Glass chunks, fuel ash slag and kiln fragments related to glass processing have also suggested local secondary production (reworking) of glass at Sagalassos from imperial to early Byzantine times (Degryse et al. 2005, 2006).

Radiogenic isotopes

Lead isotopes have been extensively used in archaeometry to trace the provenance of raw materials used for metal production (e.g. Gale & Stos-Gale 1981; Yener et al. 1991). To be significant, this approach preferably requires homogeneous isotopic signatures for both the studied artefacts and the possible lead sources. Lead ore deposits contain Pb that has been withdrawn in trace amounts from its U and/or Th-bearing source rocks, accumulated over the lifetime of the ore-forming fluid systems and subsequently incorporated into U and Th-free ore minerals, where its isotopic composition is no longer modified by radiogenic growth. Ore deposits have been formed in response to distinct geological processes during the Earth's history that have caused the circulation of ore-forming fluids. Therefore, different lead ore deposits display distinct Pb isotope compositions depending on their geological age and the geochemical character of the Pb source rocks with respect to their U/Pb and Th/Pb ratios (Faure 1986). Conversely, the original isotopic composition of the artefact lead should be identical to that of the ores from which it was produced (Brill & Wampler 1965). As

Table 1. Pb isotopic composition of architectural lead from Sagalassos (precision better than 0.2%).

Sample	$^{206}Pb/^{204}Pb$	$^{207}Pb/^{204}Pb$	$^{208}Pb/^{204}Pb$	$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$
Heroon, 25 BC–AD 1					
94 H 198	18.347	15.684	38.568	0.85487	2.1020
94 H 151	18.640	15.672	38.807	0.84081	2.0820
94 H 203	18.729	15.678	38.857	0.83709	2.0749
94 H 77	18.794	15.698	38.970	0.83528	2.0736
94 H 175	18.333	15.666	38.509	0.85453	2.1003
94 H 255	18.427	15.666	38.593	0.85016	2.0945
94 H 166	18.640	15.684	38.838	0.84142	2.0833
Agora Gate, AD 100					
95 AG 145/1	18.568	15.671	38.671	0.84400	2.0824
95 AG 145/2	18.701	15.673	38.745	0.83807	2.0719
95 AG 145/3	18.820	15.704	38.964	0.83444	2.0703
95 AG 162	18.595	15.681	38.712	0.84334	2.0817
95 AG 84	18.338	15.673	38.530	0.85465	2.1010
Roman Baths, AD 125–165					
96 RB 146	18.344	15.679	38.546	0.85474	2.1013
96 RB 153/1	18.866	15.691	38.873	0.83172	2.0605
96 RB 153/2	18.535	15.669	38.721	0.84538	2.0891
97 RB 2/34/1	18.555	15.656	38.618	0.84375	2.0813
97 RB 2/34/2	18.542	15.658	38.664	0.84446	2.0825
Upper Agora, AD 160–180 to late 4th century					
95 UA 21	18.751	15.697	39.033	0.83712	2.0817
95 UA 46	18.645	15.700	38.897	0.84208	2.0862
95 UA 150	18.580	15.662	38.681	0.84299	2.0819
95 UA 179	18.565	15.674	38.702	0.84431	2.0847
95 UA 150	18.678	15.684	38.854	0.83973	2.0802
95 UAN 124	18.308	15.677	38.538	0.85632	2.1050
95 UAN 150	18.494	15.678	38.649	0.84777	2.0898

some ore lead isotope signatures may coincide, only negative conclusions can be made on the raw materials used for a certain artefact.

The $^{87}Sr/^{86}Sr$ ratio in the Earth's crust varies with the age and type of rock, as radiogenic ^{87}Sr is formed over time by the beta decay of ^{87}Rb While the other naturally occurring isotopes of strontium, ^{84}Sr, ^{86}Sr and ^{88}Sr, are non-radiogenic and show constant $^{84}Sr/^{86}Sr$ and $^{88}Sr/^{86}Sr$ ratios. The $^{87}Sr/^{86}Sr$ ratio has therefore been widely employed to determine the ages of various rock types (Faure 1986). Over the past 25 years, geologists have used $^{87}Sr/^{86}Sr$ ratios for rock and sediment provenance studies and strontium isotope chronostratigraphies. Due to their low mass differences, the isotopes of strontium are not fractionated by natural exchange reactions or kinetic effects, and thus the strontium isotopic composition of an artefact will have been inherited from its raw materials. This gives us an opportunity to use strontium isotopes as tracers for the origin of artefacts (e.g. Wedepohl & Baumann 2000; Freestone et al. 2003; Degryse et al. 2006, 2007; Degryse & Schneider 2008).

On the origin of the lead at Sagalassos

In the first case-study the nature and origin of architectural lead from the ancient city of Sagalassos, SW Turkey, were ascertained by means of its isotopic composition. The architectural and structural fittings at Sagalassos, including clamps, dowels, revetment pins and nails, consisted mainly of iron, while a small number, mainly revetment pins and some nails, were fashioned from a copper alloy. A large amount of lead was used for sealing the dowels and clamps in the ashlar masonry.

Methods

Sampling

A total of 24 samples of architectural lead from dated contexts at Sagalassos were analysed for their isotopic composition. Details of the sample chronology are given in Table 1. The samples designated "UA" were taken from the nymphaeum on the upper agora, the original building phase of which has been dated to
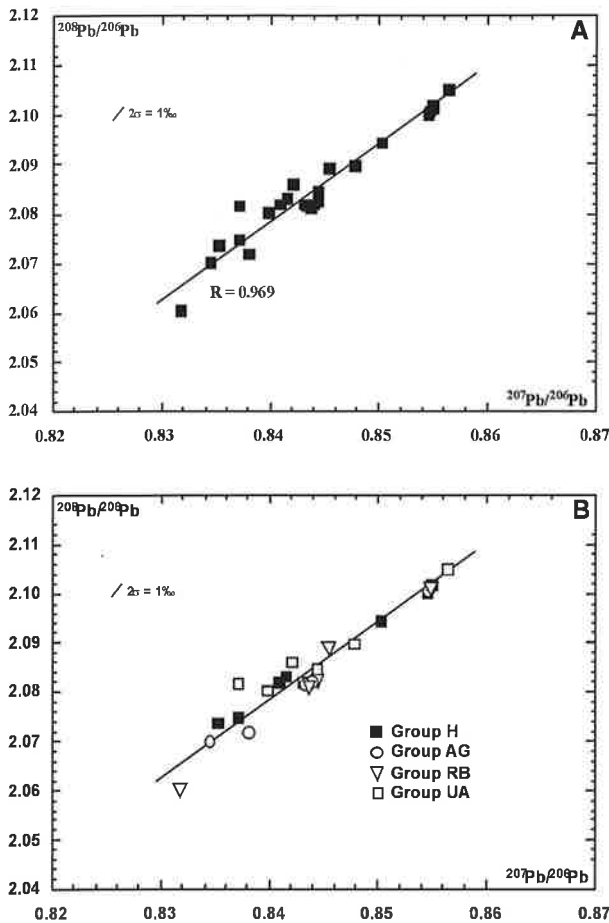
Figure 2. $^{208}Pb/^{206}Pb$ versus $^{207}Pb/^{206}Pb$ biplot showing (a) the entire set of architectural lead samples from Sagalassos, and (b) the chronological groups of samples.
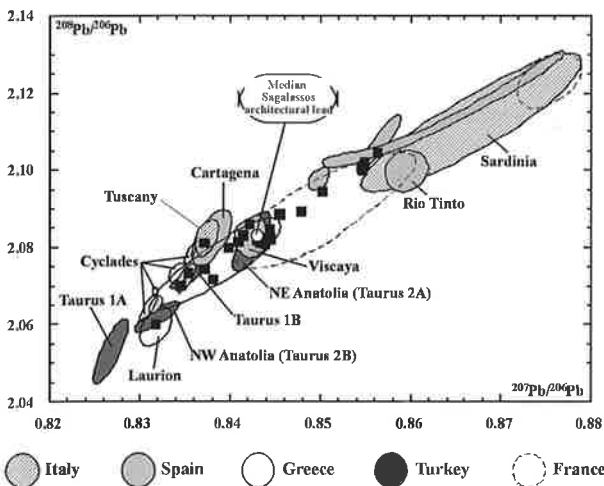

Figure 3. Lead isotope signatures of ore deposits and mining districts in the Roman world of the eastern Mediterranean (ellipsoids) compared with the data for architectural lead from Sagalassos (filled squares). Data for the mining districts and ore types in Turkey are from Yener et al. (1991) and the other data from a compilation by Gebel and Schmidt (2000).

160 to 180 AD, although substantial modifications were made to it in the middle and late fourth century or early sixth century AD. The samples marked "H" were taken from the heroon, which was built during the middle Augustan period, has been dated at 25 BC to AD 1 and was never modified, and the samples labelled "RB" were taken from the Roman baths, the lead being derived from the original ashlar building material, dated to AD 125 to 165.

Pb isotope analysis

For Pb isotope analysis, single lead grains of 10 to 500 micrograms were extracted from the samples by hand, cleaned in deionised water, weighed, and dissolved in 8N HBr. After evaporation to dryness, the residues were dissolved in nitric acid, dried again and redissolved in 1 ml of deionised water.

Sample aliquots of 200 nanograms Pb were loaded onto single Re filaments using silica gel-H_3PO_4 bedding. All the isotope analyses were performed on a six-collector Finnigan MAT 261 thermal ion mass spectrometer (TIMS) running in static multicollection mode. Pb was corrected for mass fractionation using a discrimination factor of 0.123±0.029 (2σ) %/[amu], based on replicate measurements of the NBS SRM 981 common lead (n=34) standard. Errors and error correlations were calculated after Ludwig (1980). The 2σ uncertainties for all the corrected Pb/Pb ratios are better than 0.2%.

Results

The lead isotope data obtained for the samples are listed in Table 1. All the samples plot slightly above the reference growth curves for average terrestrial crustal lead given by Stacey & Kramers (1975) and Cumming & Richards (1975) in both the $^{207}Pb/^{204}Pb$ vs. $^{206}Pb/^{204}Pb$ and $^{208}Pb/^{204}Pb$ vs. $^{206}Pb/^{204}Pb$ diagrams. The lead found in the architectural objects is therefore of crustal origin, i.e. it was produced from metal ores formed by processes within the Earth's crust with no contribution from lead derived from the mantle.

The architectural lead displays considerable variation in its isotopic composition, with

$^{206}Pb/^{204}Pb$=18.308–18.866,

$^{207}Pb/^{204}Pb$=15.655–15.704, and

$^{208}Pb/^{204}Pb$=38.497–39.032,

revealing a systematic linear trend for the entire data set in both Pb-Pb diagrams (Fig. 2). This trend is best illustrated in the $^{208}Pb/^{206}Pb$ vs. $^{207}Pb/^{206}Pb$ biplot, where the data show a close positive correlation along

a straight line (R=0.969, Fig. 2a). Compared with the Pb isotope ratios involving ^{204}Pb, the ^{208}Pb/^{206}Pb and ^{207}Pb/^{206}Pb ratios are less susceptible to mass bias and low signal response, i.e., they can be determined and corrected with better precision.

In Fig. 2b the samples are split into chronological groups according to the date obtained from the contextual analysis (cf. Table 1). These plots show a random distribution of the lead isotope ratios, with all parts of the linear trends, occurring within all the chronological groups. Coeval sets of subsamples of architectural lead are therefore not characterised by distinct isotopic compositions.

Discussion

Significance of the linear trend in lead isotope ratios
The linearity observed in the isotope ratios for architectural lead from Sagalassos awaits further explanation. Linear trends, or even straight lines, in isotope biplots involving a common denominator (e.g. ^{206}Pb in Fig. 2; see Faure 1986) have been observed for various types of geological samples and have generally been interpreted to represent either *isochrons* representing the geological age of a set of co-genetic samples, or binary mixing lines reflecting mixtures of two end components in different proportions.

Linear lead arrays observed for a number of mostly subeconomic ore deposits have also been explained as resulting from small-scale, isotopically heterogeneous ore-forming fluid systems that were variously contaminated by lead derived from distinct lithologies with variable U/Pb and Th/Pb ratios (e.g. Gulson 1986). However, since the spread observed for the isotope signatures of the architectural lead from Sagalassos greatly exceeds the internal isotopic variation of virtually all possible ancient lead mining districts (cf. Fig. 3), it seems highly improbable that the Sagalassos lead was produced from only one ore deposit or district. The chronologically random distribution of the architectural lead along a straight linear trend, especially in the ^{208}Pb/^{206}Pb vs. ^{207}Pb/^{206}Pb space, rather suggests differential mixtures of lead involving at least two end components (see Faure 1986).

Systematic binary mixing would require two isotopically strictly constant, unradiogenic (^{208}Pb/^{206}Pb>2.11) and radiogenic (^{208}Pb/^{206}Pb<2.06) sources of ore lead used over more than four centuries of building history at Sagalassos. As this seems questionable, the linearity of the architectural lead could also plausibly result from mixing of lead derived from numerous, now unidentifiable point sources comprising several radiogenic and unradiogenic types of ore. These must have supplied lead of variable isotopic composition which was subsequently thoroughly mixed and redistributed by continuous in-situ reworking and recycling throughout the history of the building of the city. This type of interpretation has been used by Haack et al. (2004) to provide a plausible explanation for the straight Pb-Pb lines observed for modern environmental samples that had accumulated and redistributed atmospheric leads over long periods of time (20–30y).

Long-term recycling of architectural lead is a comparable process, and would be economically favourable, as no local lead resources were available in the territory of Sagalassos itself and the transport of bulk lead over long distances would have been expensive. It is unlikely that several consignments of lead imported from separate source areas were deliberately mixed to produce architectural lead for one and the same building. Rather, there were various building phases predating our sampled contexts in which lead from one source was used in one particular setting and lead from another source in another, possibly non-coeval context. As history progressed and buildings were altered, destroyed and/or rebuilt, architectural lead was recycled. In this way, a mixing of lead from several distinct sources in one context or building was progressively achieved.

On the origin of the glass at Sagalassos

it has been demonstrated by main element chemical analysis that different raw materials were used in early Byzantine times for the production of the locally found blue, green and HIMT types of glass at Sagalassos, and the finding of chunks of glass has been taken to indicate the use of imported raw glass for the local secondary manufacture of these colours of glass (Degryse et al. 2005, 2006). Conversely, the heterogeneous lead isotopic composition of the local green and HIMT vessel glass was a strong indication of a mixing of several sands or the use of heterogeneous silica raw materials for its manufacture, or could possibly be the result of recycling (Degryse et al. 2005). The end members of this lead isotopic composition were formed by the local blue glass vessels and chunks on the one hand and the local HIMT chunks on the other. Sr isotopes provided analytical evidence for the recycling of glass subsequent to the use of imported raw glass (Degryse et al. 2006).

Table 2. Pb and Sr isotopic data for early Byzantine (AD 450–675) glass from Sagalassos (precision for Pb better than 0.2%).

Sample	Colour	Type	$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$	$^{206}Pb/^{204}Pb$	$^{207}Pb/^{204}Pb$	$^{208}Pb/^{204}Pb$	$^{87}Sr/^{86}Sr$	Sr	1000/Sr	Sr for 100% carbonate
582	blue	vessel	0.843	2.084	18.602	15.685	38.758	0.70881	526	1.90	0.33
583	blue	vessel	0.844	2.084	18.586	15.682	38.738	0.70881	515	1.94	0.31
590	blue	vessel	0.842	2.083	18.618	15.685	38.772	0.70881	509	1.96	0.39
VL8A	blue	chunk	0.845	2.078	18.574	15.693	38.587	0.70886	517	1.93	0.35
727	green	vessel	0.841	2.078	18.616	15.663	38.682	0.70861	483	2.07	0.39
729	green	vessel	0.840	2.075	18.658	15.665	38.719	0.70858	519	1.93	0.41
586	HIMT	vessel	0.831	2.054	18.859	15.669	38.731	0.70849	469	2.13	0.43
720	HIMT	vessel	0.837	2.070	18.730	15.675	38.776	0.70862	493	2.03	0.42
SA00JP25	HIMT	chunk	0.833	2.066	18.782	15.650	38.797	0.70813	398	2.51	0.34
SA00JP25B	HIMT	chunk	0.834	2.067	18.767	15.650	38.796	0.70782	351	2.85	0.41
VL4	HIMT	chunk	0.842	2.077	18.673	15.719	38.775	0.70848	487	2.05	0.46

Methods

Sampling

A series of 11 samples representing the common colour varieties of free-blown vessel glass to be found at Sagalassos were selected for Pb and Sr isotope analysis. The colour varieties were distinguished macroscopically, and the chronology was determined by stratigraphical association, given that all the samples originated from early Byzantine excavation contexts (AD 450–650/75).

Pb-Sr isotope analysis

All the glass sample powders were completely dissolved and split into two aliquots for separate determination of $^{87}Sr/^{86}Sr$ and Pb isotope ratios and Sr-Pb concentrations by isotopic dilution using mixed ^{206}Pb-^{84}Sr tracers. Strontium and lead were chemically separated and purified with 3N HNO_3 using EICHROM Sr resin on 50 μl Teflon columns, following the methods of Horwitz et al. (1991a, 1991b). For mass spectrometry, Sr was loaded onto single W filaments with a $TaCl_5$-HF-H_3PO_4 solution (Birck 1986), and Pb loaded onto single Re filaments using silica gel-H_3PO_4 bedding. All the Sr and Pb isotopic measurements were performed on a six-collector Finnigan MAT 261 solid-source mass spectrometer running in static multicollection mode. Sr isotopic ratios were normalised to $^{88}Sr/^{86}Sr$=0.1194. Repeated static measurements of the NBS 987 standard over the duration of this study yielded an average $^{87}Sr/^{86}Sr$ ratio of 0.71025±4 (2σ mean, n=17). The Pb isotope ratios were corrected for mass fractionation using a mean discrimination factor of 0.123±0.029 (2σ) %/[amu], based on replicate measurements of the NBS SRM 981 common lead (n=96) standard. Errors and error correlations were calculated according to Ludwig (1980). The 2σ uncertainties for all the corrected Pb/Pb ratios were better than 0.2%. Total procedure blanks did not exceed 50 pg for Sr and 70 pg for Pb, and were found to be negligible with respect to the sample weights.

Results

The analytical results for the blue, green and HIMT glass (vessels and chunks) at Sagalassos are given in Table 2. The elemental (Degryse et al. 2006) and isotopic composition of the blue vessel and chunk glass differs between samples 582 and 583, and the isotopic composition of 0.70881 to 0.70886 obtained for the blue glass is similar to isotopic analysis reported for soda-silica-lime glasses from Germany and the Levant, as given by Wedepohl & Baumann (2000) and Freestone et al. (2003). The Sr content is high, around 500 ppm for 7.3 to 9.4% of CaO. The isotopic composition of the HIMT glass chunks from Sagalassos is clearly different from that of the blue (and green) glass. The Sr isotope compositions of the two HIMT chunks are 0.70813 and 0.70782 respectively. The Sr content of the HIMT chunks is also lower than in the blue glass, around 350 to 400 ppm, but this is for a CaO content of only 5.3 to 6.6%. The chemistry of the green and

HIMT vessel glass at Sagalassos corresponds to compositions between the blue vessel glass and the HIMT chunks on all parameters.

Discussion

It has been argued by Wedepohl & Baumann (2000) that glasses that have not been deliberately coloured have a Pb isotope composition characteristic of the glass-making sand. A homogeneous composition would therefore indicate the use of a single sand as a raw material, while a heterogeneous composition would point to the use of different sands. The heterogeneous lead isotope composition of the early Byzantine green and HIMT vessel glass at Sagalassos suggests either the use of heterogeneous sands in their production, or the mixing of different sands in the manufacture of each. The end members of the lead isotopic composition of the green and HIMT species of vessel glass are identical to those of the local blue vessel and chunk glass in the one case and to that of the HIMT glass chunks in the other. Also, the major element composition of the green and HIMT vessel glass species lies on a mixing line between certain types of the blue glass and HIMT glass chunks.

The use of Sr isotopes for interpreting ancient types of glass depends primarily on the assumption that the bulk of the strontium is in many cases incorporated into the lime-bearing constituents of the glass (Wedepohl & Baumann, 2000). $^{87}Sr/^{86}Sr$ values of around 0.709 have been explained by Wedepohl & Baumann (2000) as suggesting the use of recent aragonitic sea shells as a source of lime (and hence of Sr), which will then have undergone some diagenesis, and by Freestone et al. (2003) as an Sr-signature largely caused by the use of recent sea shells with some influence from older limestone. The high Sr content of around 500 ppm for the 7.3 to 9.4% of CaO in the glass may also indicate the use of recent sea shells as a source of lime. It has been shown by Wedepohl & Baumann (2000) and Freestone et al. (2003) that recent aragonitic shells contain about 3000 to 4000 ppm of Sr (for 100% $CaCO_3$), while limestone only contains around 450 ppm on average. The Sr isotope composition of HIMT chunks has been explained by Freestone et al. (2005) as originating from raw materials with ancient limestone as the main lime source.

It has been demonstrated from Sr mixing lines (Fig. 4; Degryse et al. 2006) that the Sr in the green and HIMT vessel glass is a mixture derived from the aforementioned end members. This proves that local
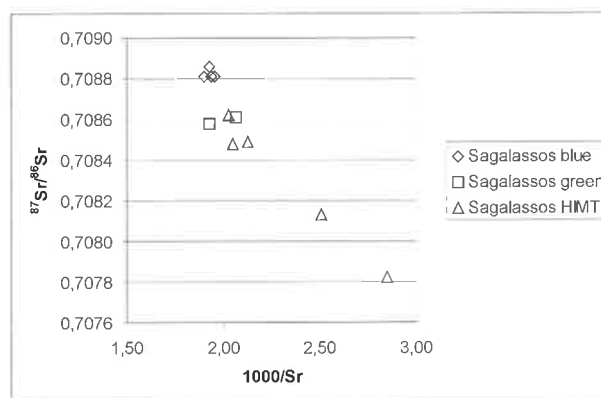

Figure 4. Strontium mixing diagram for the early Byzantine glass from Sagalassos.

recycling and secondary production of glass took place at Sagalassos. It cannot be shown, however, whether the "recycled" glass was produced from a mixture of only chunks, only glass cullet or both cullet and chunks.

It may be suggested on the grounds of the major element composition, the Sr and Pb isotope signatures and the high absolute Sr content of the local blue glass that this type had several production locations (at least two), probably in the Levant, using recent marine shell as the main source of lime. For the HIMT glass chunks, both the Pb and Sr isotope compositions correspond very well to those reported by Freestone et al. (2005), who argued that HIMT glass is produced using a mixture of two proximal sands, possibly beach sand and fluviatile material as a source of silica. The lime in these sands mainly came from ancient limestone, though a recent shell component will have been present as well. It has been suggested earlier that the use of an Egyptian natron flux would not have a great influence on the Sr balance of the resulting glass, but a minimal input of non-carbonates with a low Sr isotope composition may have had an impact on the Sr balance of a certain batch of glass (Degryse et al. 2006).

Conclusions

The concept of recycling as a part of the process of producing objects has for a long time been taken for granted. As a workable definition of the concept, we use that given by Darnay & Franklin (1972): "Recycling is an activity whereby a secondary material is introduced as a raw material into an industrial process in which it is transformed into a new product in such a manner that its original identity is lost. Secondary

materials are those that (1) have fulfilled their useful function and cannot be used further in their present form or composition and (2) materials that occur as waste from the manufacturing or conversion of products."

The nature and origin of the architectural lead and structural fittings at ancient Sagalassos (SW Turkey) has been studied here by means of lead isotopes. No distinct chronological groups can be distinguished within the architectural lead samples in terms of their Pb isotope composition, but rather the data are randomly distributed along a straight linear trend, reflecting thorough mixing by progressive lead recycling throughout the history of the building of the city. It must therefore be concluded that the lead processed at Sagalassos for all buildings and contexts from 25 BC until the late 4th century AD was derived from at least two distinct sources. The exact provenance(s) cannot be clearly determined, however.

The secondary production (reworking) of glass from imperial to early Byzantine times at the ancient city of Sagalassos (SW Turkey) has been demonstrated here by pointing to the existence of glass chunks, fuel ash slag and kiln fragments related to glass processing. The heterogeneous lead isotope composition of the green and HIMT types of vessel glass at Sagalassos, with the end members coinciding with the isotopic composition of the local blue glass vessels and chunks in the one case and with that of the HIMT glass chunks in the other, could indicate the production of "recycled" glass, although heterogeneous raw materials could have been used. The use of Sr mixing lines nevertheless confirms local recycling. It is clear that the Sr in the green and HIMT vessels is a mixture of that in the aforementioned end members, but it cannot be proved whether the green "recycled" glass was produced from a mixture of chunks alone, or from a mixture of cullet and chunks. Suggestions can be made regarding the possible origin of the raw materials: a Levantine origin for the blue glass and an Egyptian origin for the HIMT glass.

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