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Abstract

This report describes analytical results from a small suite of specimens drawn from the material recovered during excavation at Brownslade and analysed as comparative material during post-excavation analysis of residues from South Hook Pembrokeshire. It is not a full post-excavation study of the Brownslade assemblage.

The analysed material included two samples of slag, both interpreted as smelting slag. One of these, BS1, was a tapped slag flow and the sole stratified slag find from the site; the other, BS2 was a slag with a lobate, steep contact, probably formed against a stone and just possibly indicative of being from a non-slag tapping furnace. Two samples of iron ore were also analysed. One of these, BS3, was a typical example of the goethitic layered ores that were found abundantly on the site and the second, BS4, was a large block of irregular concretionary ore with a texture suggesting it was a reworked Carboniferous concretion, but for which an origin as iron pan or bog ore cannot be excluded.

Analysis showed that the two slags were very similar and also showed similar chemical characteristics to the goethitic ore. The concretionary ore, in contrast, was much closer in chemical signature to the smelting slags found at South Hook, on the opposite side of Milford Haven.

Contents

Methods

All materials were examined visually with a low powered binocular microscope as part of the evaluation (Young 2006). The evaluation identified this assemblage as being slags and associated residues produced in the processes of iron smelting and smithing. The catalogue of the material is presented again here in Table 1. A small programme of analysis was undertaken in conjunction with the full post-excavation analysis of the archaeometallurgical residues from South Hook, the only other early medieval iron-production site then known in SW Wales. This study does not constitute a full post-excavation analysis of the Brownslade assemblage.

Two samples of slags (one a tapped slag, one a possible non-tapped smelting slag) and two contrasting iron ore samples were selected for detailed analysis (Table 2).

Electron microscopy was undertaken on the LEO S360 analytical electron microscope in the School of Earth, Ocean and Planetary Sciences, Cardiff University. Microanalysis was undertaken using the system's Oxford Instruments INCA ENERGY energy-dispersive x-ray analysis system (EDX). All petrographic images presented in this report are backscattered electron photomicrographs. The polished blocks for investigation on the SEM were prepared in the Earth Science Department, The Open University. Chemical analysis was undertaken using two techniques. The major elements (Si, Al, Fe, Mn, Mg, Ca, Na, K, Ti, and P) were determined by X-Ray Fluorescence using fused beads, on the Open University Earth Science Department's Wavelength-Dispersive X-Ray Fluorescence (WD-XRF) system. Whole-specimen chemical analysis for minor and trace elements was undertaken using samples in solution on the ThermoElemental X-series Inductively-Coupled Plasma Mass Spectrometer (ICP-MS) in the School of Earth, Ocean and Planetary Sciences, Cardiff University.

Whole-specimen bulk chemical analyses are presented in Table 3 and EDS microanalyses in Table 4, wth their locations in Plates 1-5.

Results

Iron slag

BS1: This lobate slag was a small piece, but had all the morphological characteristics of a tapped slag. The upper surface showed well-developed raised flow lobes and the base showed dimples, probably from contact with charcoal.

In the SEM (Plate 1) it shows a moderate fine vesicularity and a very variable density of primary wustite (compare Plate 1a with Plate 1e), mainly in the form of stubby rounded dendrites. The slag is within flow lobes with iron oxides (probably wustite) on the chilled margins (Plate 1c and 1d).

The secondary mineral phase is an iron silicate (probably fayalite) forming delicate, “feather-like” dendrites. This morphology is very unusual and is closer to textures more normally associated with pyroxene or even amphibole, than olivine in slags. The dendrites are set in an aluminous, calcium-rich glass. EDS analysis of this material inevitably samples a mixture of the two materials. The bulk EDS compositions obtained are actually close to the composition of an amphibole and given the problems of mixing of dendrite and glass in the analyses the identification of the dendritic mineral is not possible.

Major element analysis of this slag shows it to have a high iron content (FeO = 70%). Besides alumina and silica other elements are in low concentrations, although calcium and potassium are towards the upper end of typical abundances for smelting slags in the Bristol Channel Orefield. The trace element concentrations are typically low, but vanadium, uranium and chromium are moderately elevated.

The rare earth elements (REE) shows a gently inclined profile when normalised against Upper Crust (Figure 1; normalisation factors after Taylor and McLennan 1981) with progressive relative depletion of the light REE (LREE) compared with the heavy REE (HREE). There is a small negative cerium anomaly.

BS2:
This sample was of broken piece of a vesicular grey slag, formed in the angle of a horizontal and vertical surface. The original base of the piece is rather eroded, but appears to have been slightly dimpled and may have been a contact with sediment. The vertical side shows the surface of a series of rounded lobes, which are coated with a white material (lime?), except for the lowest lobes, which are free of coating and appear to have undercut the vertical wall slightly. On morphological criteria, the block appears to be a smelting slag, with the accumulation of lobed slags against a vertical face either occurring in the tapping channel of a slag tapping furnace, or in the basal pit of a non-slag tapping slagpit furnace.

The microstructure of the sample (Plate 2) shows a rather variable texture. In some areas discontinuities are visible within the slag (e.g. running almost vertically through the field of view in Plate 2c) that are suggestive of the margins of cooled flow lobes. To the left of the discontinuity in Plate 2c the slag texture appears broadly similar to many tapslags, although perhaps rather coarse-grained, whereas to the right the degree of vesicularity is much higher and there are patches of slag devoid of primary wustite. This texture is much closer to that of furnace slags, or indeed to some smithing slags. The coarse grain size is particularly apparent in Plate 2f. Unlike some furnace slags, however, this sample does not appear to develop potassium rich zones (often characterised by the development of leucite-rich rims) around the large vesicles. The potassium enrichment is produced by the capture by the slag of potassium from the fuel ash that has been volatilised during combustion. This suggests that the present sample is not a slag that has developed over a long period in a furnace.
The interstitial areas include large zones of finely dendritic olivine (too fine for EDS analysis) which appear to be in crystallographic continuity with the larger crystals, together with an iron-poor groundmass.

Microanalysis of the olivine shows that the cores are Fa92-94 with 2% Ca and 3%Mn substitution grading out to margins of Fa94-97 4-7%Ca and 3%Mn substitution (Table 5).

The major element analysis of the specimen indicates this slag is more iron-rich than BS1 (FeO = 76%; reflected in the much higher proportion of wustite). Levels of calcium and potassium are proportionately lower (compared to silica) in this sample than in BS1, suggesting a lower relative contribution to the melt from the fuel ash slag. Phosphorus is slightly more abundant than in BS1. Most trace elements are at broadly comparable concentrations to BS1, though uranium is elevated, leading to a much higher U:Th ratio.

The REE profile for BS2 is almost identical to that for BS1 (Figure 1).

Iron Ore

BS3:
This specimen was a small piece (38g) of goethitic ore showing a somewhat laminated structure, with parallel zones of dense ore bounding a more porous zone. On cutting for specimen preparation the material appeared much richer in haematite than had been apparent from the surface.

In the SEM (plates 3 and 4) the ore can be seen to have a very complex internal structure. The primary mineralisation seem to consist of dense zones of oxide (probably rich in haematite) which have either a dense, laminated microstructure (Plate 3b), or comprises mineral with a botryoidal texture (e.g. Plate 3d, f, g).

Between the areas with dense oxide textures there are zones of more mixed material, incorporating some botryoidal lamination (e.g. Plate 3b; Plate 4a), but is more frequently rich in silicate and carbonate grains suggestive of sediment which has infiltrated the voids and become mineralised during secondary processes.

The polished block produced for the SEM work was unfortunately slightly contaminated by metallic indium in the preparation lab. This is documented here (Plate 4b and c) to avoid future confusion.

Major element analysis shows the ore to contain almost 81% Fe_2O_3 with almost 11% LOI (suggesting that the dominant mineral is likely to be hydrated, probably goethite). All other major elements are present in low concentrations.

The trace elements too are present typically in low concentrations, although titanium, lead , zinc, uranium and vanadium are present in levels that are relatively elevated for the Bristol Channel Orefield.

These trace elements may be plotted onto the bivariate diagrams employed by Young (2000, Figure 10.4) to examine provenance of samples from the western part of the Bristol Channel Orefield. On all of those plots for which relevant data are available for BS3 (the current incarnation of the ICP-MS system used for the analysis does not routinely provide date for germanium, arsenic or cadmium, which proved useful elements in the Paviland analysis) the sample plots with the field of analyses of ores from Gower, plus those from Paviland interpreted to have been originally sourced in Gower. This suggests that there is potentially a degree of homogeneity between the trace element chemistry of the ores of Gower and of South Pembrokeshire.

The Upper Crust-normalised REE profile (Figure 1) shows a relative depletion of the LREE, but a relatively flat profile for the HREE. There is a slight negative Ce anomaly. This profile differs from the profiles of the ores from Paviland and eastern Gower (Young 2000, Figure 10.5), which show broadly humped profiles with progressive depletion of the LREE and HREE centred on dysprosium, but does resemble an analysis of the mineralised (haematised) shale at Port Eynon, Gower. Young (2000, Figure 10.6) shows the fields of analyses from the western Forest of Dean and Glamorgan also extending down values of Gd_N/Lu_N, corresponding to profiles without progressive HREE depletion.

BS4:
This large, rounded block of iron rich rock shows a rather contorted, laminated, texture in section. The layering is suggestive of banding produced by concretionary mineral precipitation and in the uncut specimen this showed some indication of being concentric. The iron impregnation is relatively even, although varies slightly giving a variable yellow-brown colour to the cut face of the sample. The texture is unlike typical bog iron ores and is somewhat closer to the texture in oxidised concretions reworked from bedrock, particularly from the Carboniferous coal measures.

In the SEM (Plate 5) there is an abundance of quartz at a silt / very fine sand grade. The quartz is very irregular in its contacts with the surrounding iron oxides, suggesting that some reaction and etching of the quartz has occurred. The rock also contains somewhat rounded voids, typically filled with clay minerals and also angular grains which may be clay filled voids after carbonate crystals (possibly also some relict carbonate grains too). The distribution of quartz is variable, suggesting an original sedimentary bedding foliation. The texture is reminiscent of that of an oxidised carbonate concretion, although the clay mineral aggregates are unusual. On balance, the petrography of the material does not distinguish with any degree of certainty between an interpretation as an oxidised carbonate concretion and as an unusual bog iron ore.

Major element analysis of this sample shows an Fe_2O_3 content of about 69% (but an unexpectedly high discrepancy exists with the 78% indicated by the ICP-MS analysis). The high silica content (almost 13%) corresponds to the high level of detrital silt observed in the polished bock. The phosphate content was around 1%, with MnO also slightly elevated compared with the goethite ore BS3.

The trace elements are present in generally rather low levels, with the REE, uranium, vanadium and thorium all particularly low. The Upper Crust-normalised REE profile (Figures 3, 4) shows a progressive LREE depletion with a superimposed enrichment of the MREE, giving a pronounced hump from samarium to terbium, together with a slight, but marked negative cerium anomaly. This profile is markedly different from those of the, admittedly few, Carboniferous claystone ironstone concretions available for comparison (Figure 4).

Interpretation

The data described above allow a very good correlation to be made between the chemical signature of the goethitic ore sample, BS3, and the two smelting slags. Since there are so few analyses of smelting slags and no analysis of the likely furnace material, then no attempt can be made to calculate mass balance for smelting reaction following the technique of Thomas and Young (1999a, b). None the less the similarity of the REE profile leaves little doubt of the link between the two materials.

The interpretation of the ore sample BS4 is more difficult. There is clearly no link between this ore and the analysed slags. That is not to say the material was not smelted at Brownslade, merely that there is no evidence on the basis of these two analyses. The interpretation of the origin of this material is problematic because although it appears similar to an oxidised nodule from the Carboniferous, the REE profile does not match the few profiles available for comparison (Figure 4). The profile does match those of the smelting slags from South Hook (Figure 3), where the smelting slags show high manganese and other elements which are strongly suggestive that the source was a bog iron ore. The question of whether the concretionary ore specimen was derived from the solid geology, or is a superficial concretion of “bog ore” remains uncertain.

Discussion

This limited programme of analysis has shown that smelting slags found at Brownslade can be linked to the goethitic iron ore fragments. These fragments are of ores which are closely related by both texture and chemical composition to others in the Bristol Channel Orefield.

The nature of the goethitic ore fragments at Brownslade is interesting. They are relatively abundant; the assessment report (Young 2006) recorded approximately 80 fragments of ore from the excavations with a weight roughly equal to that of the total slag. This appears a very high ratio and it may well be that some or all of the ore recorded may have been on the site through natural agency; in other words the same resource that the early smelters were exploiting. The Bristol Channel ores are commonly present as void-filling deposits within the Carboniferous Limestone (which underlies Brownslade) and it is possible that the ore fragments

have been released through dissolution of the host limestone.
This study has not produced a definitive answer on the technology behind the smelting slags. While the smaller fragment has both the appearance and microstructure of a tapped slag, the larger block appears to have cooled more slowly, and possibly with the furnace. The overall structure, however, does not reveal whether this was in a tapping or non-tapping furnace.

The new data adds nothing to the discussion of the possible date for the iron-production given in the assessment report. The combination of slag-tapping with smithing hearth cakes ranging up to a little less than a kilogramme would suggest a date of Roman to earlier medieval.

The large block of ore (BS4) is interesting because of the similarity between its trace elements, particularly the REE, and those recorded from the tapped smelting slags at South Hook. The ore is a completely different type of ore from oxide ores of the Bristol Channel Orefield; this is a sedimentary ore in which iron minerals have precipitated within a pre-existing sediment. The distinction to be made is whether this is a concretion reworked from the Carboniferous bedrock (in which case the original iron mineral would have been iron carbonate, siderite, which has subsequently been oxidised on exhumation) or whether the concretion is of more recent origin with iron minerals precipitated into an unconsolidated sediment (bog iron ore). The microstructures most closely resemble those of a Carboniferous claystone ore, but the REE pattern does not resemble the few analysed examples from South Wales. The sample has a high content of silica, because the mineralisation is hosted by a siltstone, which is not compatible with the South Hook slags, but claystone ironstones may occur in a variety of host sediment types. If the specimen is of a Carboniferous ore, then there are no local primary sources. The piece must either have been moved by human agency, or by glacial action, most likely from the coalfield lying to the north of Milford Haven.

The alternative interpretation of this piece is that it is of relatively recent origin. Bog iron ores typically have a mottled texture and the more homogeneous texture here might be a reflection of the relatively coarse-grained host sediment – in other words this specimen might be closer to deposits usually known as iron-pan than to lithologies strictly referable to the bog iron ores.

Whatever the origins of this specimen, there is currently no evidence for materials of this type having been smelted at Brownslade.

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Illustration Captions

Figure 1: Upper Crust-normalised (Taylor & McLennan 1981) REE profiles for slag samples BS1 and BS2, together with that of the goethitic ore BS3 and a comparative sample MUM3 (a haematised shale from Port Eynon).

Figure 2: Upper Crust-normalised (Taylor & McLennan 1981) REE profiles for ore sample BS4, together with comparative slag samples SHL1-7 from South Hook.

Figure 3: Upper Crust-normalised (Taylor & McLennan 1981) REE profiles ore sample BS4, together with comparative claystone ironstone samples (BP1, Bla1, ExO and U28; all from the eastern part of the South Wales coalfield)

Plate 1: Backscattered electron photomicrographs of slag sample BS1.
a. Area 1. Scale bar 100µm.
b. Area 2. Scale bar 100µm.
c. Area 3. Scale bar 1mm
d. Area 4. Scale bar 100µm
e. Area 5. Scale bar 100µm
f. Area 6. Scale bar 60µm

Plate 2: Backscattered electron photomicrographs of slag sample BS2.
a. Area 1. Scale bar 1mm.
b. Area 2. Scale bar 100µm.
c. Area 3. Scale bar 1mm
d. Area 4. Scale bar 100µm
e. Area 5. Scale bar 1mm
f. Area 6. Scale bar 1mm

Plate 3: Backscattered electron photomicrographs of ore sample BS3.
a. Area 1. Scale bar 2mm.
b. Area 2. Scale bar 2mm.
c. Area 3. Scale bar 2mm
d. Area 4. Scale bar 2mm
e. Area 5. Scale bar 2mm
f. Area 6. Scale bar 1mm
g. Area 7. Scale bar 200µm
h. Area 8. Scale bar 600µm

Plate 4: Electron photomicrographs of ore sample BS3.
a. Area 10 (backscattered electron photomicrograph). Scale bar 1mm.
b. Area 11/12 indium contamination (backscattered electron photomicrograph).Scale bar 100µm.
c. Area 11/12 indium contamination (secondary electron photomicrograph). Scale bar 100µm.

Plate 5: Backscattered electron photomicrographs of ore sample BS4.
a. Area 1. Scale bar 1mm.
b. Area 2. Scale bar 1mm.
c. Area 3. Scale bar 1mm
d. Area 4. Scale bar 100µm.
e. Area 5. Scale bar 60µm.
f. Area 7. Scale bar 800µm
g. Area 8. Scale bar 800µm

context	*weight*	*description*
102	240	small dense SHC shattered into 12 pieces - very altered
105	38	dense grey tapslag-like sheet, but only a small piece, charcoal dimples on base (sample BS1)
114	62	small fragment of dense slag (from SHC?) broken in two
131	178	rounded cobble of iron-enriched rock - needs cutting to determine precise nature
133	152	small glassy SHC with included quartz pebbles. Top smoothly lobed, base dimpled
	138	fragment from small but very dense SHC, weathered pale grey, has lots of tubular vesicles
	10	fine debris
134	216	very dense, probably most of small SHC, with very dense iron rich lower part
	14	corroded iron
136	74	4 pieces of iron ore
	66	grey glassy slag in nub with khaki surface, lots of small charcoal impressions
	260	part of small dense SHC. Slightly prilly base with probable large tool mark, interior vesicular, top covered with fine rusty material
143	62	rather porous iron ore
145	14	small dense ore fragment
146	10	possible vitrified lining
	12	corroded iron - shrapnel
	12	small piece of tapslag-like material
	14	3 pieces of iron ore
147	198	13 pieces of iron ore
	6	iron - shrapnel
	100	natural coarse limestone
	12	vesicular grey indeterminate slag
	72	most of small SHC, dense
148	10	corroded iron - shrapnel
	10	iron ore

context	*weight*	*description*
150	14	corroded iron - shrapnel
	60	angular lump of slag with a very large charcoal impression
	30	4 pieces of vesicular slag
151	62	low density piece - probably stone, but needs fresh surface
153	138	5 pieces of goethite ore
	4	stone
	10	charcoal-rich slag
	78	fragment from small very dense SHC, thick crust, but small puddle
154	6	limestone pebble
	186	8 pieces of goethite ore
	64	dense angular slag piece - probably from margin of SHC
	42	smooth topped but lobate, with dimpled base - fragment of a very thin SHC?
	20	small nub of dense vesicular slag
155	16	iron rod
	1	lining slag
	8	corroded iron lump
	472	23 pieces of goethite ore
	52	natural stone
	66	small block of vesicular dense grey slag - probably an SHC fragment
	<	charcoal piece
	140	small complete SHC, with good charcoal impressions on top and dimples underneath. Slag rather pale
156	76	small tap slag like piece, mainly single flow with rough base, c 15 thick, lobed top
	38	small nub of charcoal-rich lining dominated slag
	232	10 pieces of goethite ore (sample BS3 from these)
	502	accumulation of lobate slag on floor and against object. Possibly from basal pit of slagpit furnace or from tapping channel (sample BS2)
	30	3 pieces of vitrified lining?
	2	low density finely vesicular slag fragment
176	40	3 pieces of iron ore

context	*weight*	*description*
213	22	probable corroded iron lump
	10	poorly mineralised boxstone piece
	252	iron ore
243	2	low grade iron ore fragment in red clay
248	1065	block of concretionary ore (sample BS4)
	728	part of medium sized conventional SHC, <80%, charcoal rich. In 3 bits
	50	fine slag debris
	282	indeterminate charcoal-rich rusty slag block
	100	2 ore fragments
	218	charcoal-rich slag piece - possibly an SHC burr
256	262	dense rounded nub of slag, possibly part of an SHC, grey slag abundant vesicles near surface
	36	tapslag fragment in two pieces
	26	probable tapslag-like piece with charcoal dimpled base in two pieces
	22	corroded iron - shrapnel
	66	5 fragments of vesicular slag - some possibly SHC material?
	18	2 pieces of concretionary material - both probably from growth around corroding iron
266	16	small smithing slag nub
	12	piece of slagged lining
	12	probable iron ore

Table 1: Summary catalogue by context.

sample	context	description
BS1	105	Small apparent tapslag flow. Stratified
BS2	156	flow slags against stone? side
BS3	156	layered goethite ore
BS4	248	large weathered concretionary ore

Table 2: Samples selected for detailed analysis.

sample	SiO_2	Al_2O_3	Fe_2O_3	FeO*	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	LOI	LOI*	Total	Ba ppm	Cr ppm	S wt%
BS1	15.84	3.57	78.16	70.33	1.42	0.83	3.88	0.46	2.12	0.20	0.54	-6.88	0.95	100.14	150	159	0.05
BS2	10.12	2.40	84.14	75.71	0.93	0.73	1.58	0.33	0.79	0.13	0.88	-5.74	2.69	96.27	152	69	0.06
BS3	2.89	1.09	80.72	72.63	0.74	0.27	0.50	0.13	0.08	0.03	0.44	10.79	18.88	97.67	65	111	0.01
BS4	12.87	0.80	68.80	61.91	1.90	0.38	0.75	0.17	0.23	0.05	0.99	12.12	19.01	99.06	74	36	0.02

Table 3a. Major elements determined by XRF, expressed as wt% oxides except where stated. Columns in grey (*) are alternative calculations with iron expressed as FeII instead of FeIII. LOI = loss on ignition

sample	TiO_2	Fe_2O_3	MnO	P_2O_5
BS1	0.18	71.79	1.36	0.49
BS2	0.14	81.50	1.01	0.92
BS3	0.04	79.35	0.77	0.44
BS4	0.10	77.57	2.35	1.14

Table 3b. Major elements determined by ICP-MS expressed as wt% oxides

sample	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Mo	Sn	Cs	Ba
BS1	10.7	172.1	116.8	4.0	6.8	3.7	91.0	5.3	19.9	75.8	134.4	65.0	3.20	3.17	20.63	1.47	138.1
BS2	9.5	76.5	43.7	2.7	4.3	12.9	67.1	4.0	10.3	100.4	104.1	65.4	3.10	2.69	2.38	0.77	165.2
BS3	2.1	141.9	84.9	6.8	50.4	6.4	228.9	1.8	3.4	35.0	21.1	22.9	1.03	8.30	2.23	0.64	49.0
BS4	1.3	18.9	31.6	42.8	437.8	9.0	949.6	6.2	11.0	104.2	25.4	28.6	0.50	1.11	1.16	1.19	87.0

Table 3c. Trace elements determined by ICP-MS in ppm

sample	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Hf	Ta	Pb	Th	U
BS1	31.63	31.30	9.26	36.67	8.10	1.84	8.61	1.66	11.62	2.55	7.79	1.14	7.08	1.15	1.41	0.17	1.57	3.22	8.30
BS2	28.59	21.76	7.18	29.60	6.02	1.30	6.45	1.20	8.62	1.94	5.86	0.85	5.22	0.86	1.52	0.16	2.30	2.72	11.47
BS3	5.73	8.63	1.42	5.62	1.23	0.33	1.60	0.24	1.86	0.41	1.26	0.18	1.08	0.20	0.43	0.04	598.68	1.35	8.57
BS4	1.90	1.75	0.46	2.40	0.86	1.08	4.39	0.38	0.86	0.20	0.62	0.11	0.74	0.13	0.77	0.04	24.80	0.12	0.64

Table 3d. Trace elements determined by ICP-MS in ppm (continued)

Sample	*Area*	*#*	*notes*	*N*	*Na*	*Mg*	*Al*	*Si*	*P*	*S*	*Cl*	*K*	*Ca*	*Mn*	*Fe*	*In*	*O*	*Total*
BS1	2	1	feathery olivine		0.63	0.77	3.91	15.84	0.40			1.95	2.70	0.85	14.39		58.55	100.00
BS1	2	2	feathery olivine		0.70	0.58	4.01	15.85	0.36	0.15		1.98	3.43	0.70	13.58		58.67	101.23
BS1	2	3	feathery olivine		0.83	0.28	4.68	16.21	0.50	0.21		2.54	5.30	0.44	10.00		59.02	101.17
BS1	2	4	feathery olivine		0.82	0.30	4.19	15.77	0.43	0.10		2.00	5.09	0.59	12.06		58.65	98.93
BS1	2	5	wustite				0.41							0.56	48.75		50.16	98.20
BS1	2	6	wustite			0.49	0.31							0.57	48.39		50.13	97.08
BS1	2	7	bulk area		0.69	0.56	3.59	13.05	0.38			1.77	2.94	0.65	19.29		57.09	102.84
BS1	3	1	bulk area		0.72	0.57	3.20	10.37	0.27			1.47	2.50	0.62	24.66		55.64	98.83
BS1	3	2	bulk area		0.62	0.71	3.08	9.55	0.24			1.32	2.31	0.64	26.28		55.24	119.11
BS1	4	1	feathery olivine		0.82	0.51	4.20	16.17	0.50	0.09		2.27	4.42	0.67	11.51		58.83	100.51
BS1	4	2	feathery olivine		0.75	0.58	4.21	16.29	0.44	0.12		2.19	4.89	0.64	10.99		58.91	102.19
BS1	4	3	feathery olivine		0.69	0.71	4.16	16.11	0.46	0.10		2.30	3.59	0.79	12.31		58.79	98.90
BS1	4	4	feathery olivine		0.79	0.56	4.23	16.35	0.51	0.17		2.44	4.67	0.62	10.69		58.98	99.32
BS1	4	5	wustite			0.57								0.65	48.78		50.00	99.02
BS1	4	6	wustite			0.57	0.33							0.67	48.35		50.08	100.95
BS1	4	7	wustite			0.57	0.37							0.69	48.28		50.09	100.72
BS1	4	8	wustite			0.52	0.62	1.03				0.13	0.32	0.68	46.05		50.64	102.26
BS1	4	9	wustite			0.52	0.42							0.69	48.26		50.10	101.47
BS1	4	10	wustite			0.44	0.42	0.34						0.70	47.84		50.27	100.72
BS1	4	11	wustite			0.56	0.33	0.29						0.65	47.78		50.28	100.29
BS1	4	12	wustite			0.46	0.34	0.31						0.75	47.90		50.24	100.76
BS1	4	13	wustite			0.50	0.29	0.24						0.65	47.96		50.25	100.49
BS1	4	14	wustite			0.46	0.34							0.63	48.31		50.14	100.33
BS1	4	15	wustite			0.58	0.36						0.12	0.72	47.97		50.14	99.76
BS1	6	1	tiny equant olivine		0.33	1.69	1.10	14.53	0.15			0.46	3.48	1.15	19.66		57.45	106.20
BS1	6	2	tiny equant olivine		0.44	1.58	2.67	14.58	0.29			1.42	3.84	0.97	16.50		57.71	107.17
BS1	6	3	tiny equant olivine		0.31	1.77	1.45	14.54				0.78	3.49	1.09	19.21		57.36	103.87
BS1	6	4	dark interstitial		1.56		8.55	15.48	0.81	0.28		4.82	2.97	0.19	6.17		59.17	99.82
BS1	6	5	dark interstitial		1.53	0.19	8.24	15.34	0.81	0.26		4.85	3.12	0.20	6.45		59.00	100.03
BS1	6	6	dark interstitial		1.55	0.24	7.80	14.96	0.79	0.18		4.62	3.24	0.30	7.67		58.65	96.47
BS1	6	7	wustite				0.43							0.55	48.91		50.11	98.79
BS1	6	8	wustite				1.61	2.61				0.48	0.46		43.24		51.59	138.62

Sample	*Area*	*#*	*notes*	*N*	*Na*	*Mg*	*Al*	*Si*	*P*	*S*	*Cl*	*K*	*Ca*	*Mn*	*Fe*	*In*	*O*	*Total*
BS2	2	1	olivine core			1.63		14.24	0.12				0.52	0.90	25.36		57.21	104.39
BS2	2	2	olivine outer			1.01		14.11					0.78	0.94	26.11		57.05	103.57
BS2	2	3	olivine margin			0.77	0.19	14.07					1.10	0.88	25.89		57.08	104.13
BS2	2	4	olivine core			1.71		14.18					0.53	0.91	25.58		57.09	102.19
BS2	2	5	olivine margin			1.96	0.29	13.98	0.22				0.47	0.89	24.96		57.23	101.65
BS2	2	6	olivine margin			0.91	0.29	13.88	0.19			0.21	1.43	0.85	25.13		57.10	100.50
BS2	2	7	interstitial glass/olivine		1.96		10.69	12.56	2.11	0.22		3.16	4.08	0.12	5.47		59.52	103.06
BS2	2	8	interstitial glass/olivine		2.14		10.29	13.44	2.04	0.22		3.59	4.27		4.39		59.61	105.43
BS2	2	9	wustite				0.65	0.23						0.46	48.38		50.28	100.54
BS2	2	10	wustite				0.47	0.22						0.38	48.54		50.28	101.81
BS2	4	1	olivine core			2.07		14.23					0.59	0.87	25.12		57.12	101.76
BS2	4	2	olivine margin			1.88		14.10	0.13				0.77	0.92	25.05		57.15	100.63
BS2	4	3	olivine core			2.23		14.10					0.45	0.90	25.27		57.05	100.90
BS2	4	4	olivine margin			1.78	0.26	14.13	0.22				0.90	0.94	24.47		57.30	100.34
BS2	4	5	olivine margin			1.36	0.84	13.82	0.55			0.34	1.81	0.89	22.94		57.45	99.12
BS2	4	6	late mixed olivine		1.10	0.53	3.80	14.45	0.63			1.51	2.96	0.63	16.41		57.99	104.07
BS2	4	7	late mixed olivine		1.13	0.31	3.51	14.08	0.71	0.14		1.42	2.97	0.62	17.16		57.96	103.48
BS2	4	8	dark interstitial		0.71		8.76	15.47	1.73	0.65		3.10	3.56		5.12		60.92	78.58
BS2	5	1	bulk area			0.72	2.65	6.19	0.30			0.51	0.64	0.41	34.75		53.85	96.73
BS2	6	1	bulk area		0.74	0.56	4.00	9.51	0.58			0.96	1.36	0.47	26.06		55.76	87.05
BS2	6	2	w-poor sub area		1.05	0.63	4.41	13.03	0.85			1.62	2.14	0.51	18.18		57.58	89.03
BS2	6	3	w-rich sub area		0.52	0.50	3.46	7.30	0.47			0.69	0.95	0.48	31.05		54.57	97.48
BS3	7	1	inner spheroid				0.43	1.28	0.19				0.27	0.22	46.72		50.89	74.43
BS3	7	2	inner spheroid				0.59	1.24	0.24				0.34	0.21	46.45		50.94	76.65
BS3	7	3	inner spheroid				0.63	1.16	0.29				0.18	0.41	46.37		50.96	73.06
BS3	7	4	inner spheroid				0.47	1.23	0.21				0.26	0.37	46.56		50.89	71.64
BS3	7	5	outer fibrous spheroid				0.70	1.56					0.22	0.24	46.33		50.95	72.95
BS3	7	6	outer fibrous spheroid				0.41	1.76	0.22					0.22	46.25		51.15	72.38
BS3	7	7	inner layered botryoidal coat				0.76	1.96					0.14	0.20	45.77		51.17	73.92
BS3	7	8	outer layered botryoidal coat				1.75	1.62	0.21				0.18		44.82		51.41	74.62
BS3	7	9	dense particle				1.05	1.49	0.21					0.38	45.71		51.16	68.82

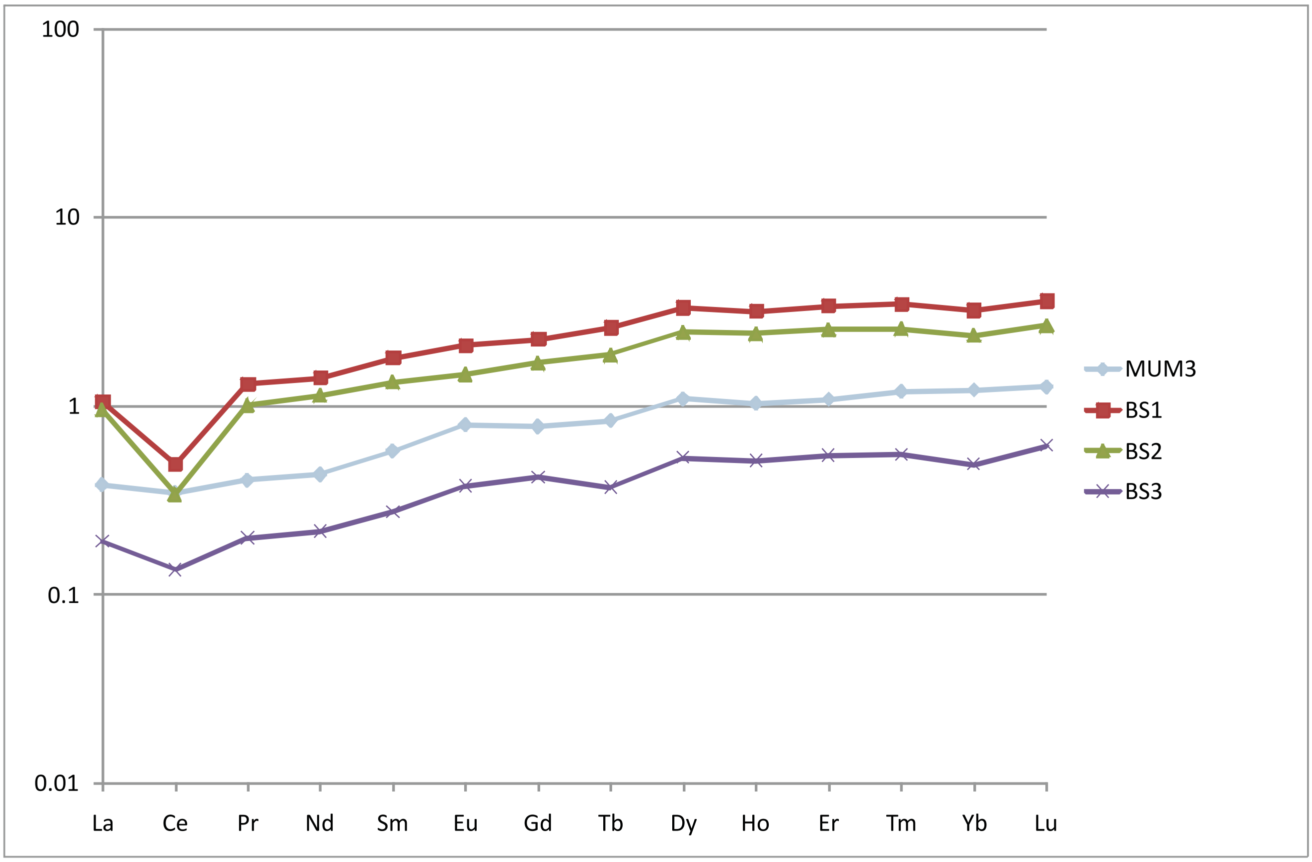
Sample	*Area*	*#*	*notes*	*N*	*Na*	*Mg*	*Al*	*Si*	*P*	*S*	*Cl*	*K*	*Ca*	*Mn*	*Fe*	*In*	*O*	*Total*
BS3	7	10	point in open texture				1.54	2.08	0.26				0.21	0.38	43.91		51.62	74.68
BS3	7	11	bulk area				3.89	1.50	0.20		0.21		0.29	0.24	41.91		51.77	70.18
BS3	8	1	delicate layering			0.49	0.68	1.41					0.26	0.21	46.07		50.88	66.88
BS3	8	2	indium			0.76	5.23	1.05		0.31		1.83			2.09	29.13	59.18	73.66
BS3	8	3	indium			3.93	4.50	1.85		0.57		1.71			1.68	26.86	58.90	80.59
BS3	8	4	moderately bright				0.65	1.33	0.29				0.25	0.31	46.13		51.04	82.59
BS3	8	5	finely porous layer				6.46	2.12	0.18				0.21	0.22	37.98		52.81	74.15
BS3	10	1	error				6.29	9.14					0.95		23.80	2.95	56.88	24.57
BS3	10	2	bulk area of crust/layered			0.47	6.09	2.50	0.30		0.20	0.14	0.32	0.41	36.71		52.86	62.90
BS3	11	1	indium contamination	7.99								1.75			1.12	26.81	62.18	134.72
BS3	11	2	indium contamination									2.91			1.05	37.28	58.42	116.81
BS3	11	3	indium contamination	7.86								1.70			0.56	27.42	62.20	137.99
BS3	11	4	indium contamination					0.63				2.55			2.10	35.96	58.57	103.74
BS3	11	5	indium contamination			0.42	11.83	0.59				1.51			0.60	25.67	59.21	123.83
BS3	11	6	indium contamination				6.25	0.57	0.40			1.94	1.15		3.58	27.47	58.64	73.62
BS3	11	7	indium contamination				3.96	1.25				1.02	0.68		20.78	16.76	55.55	72.03
BS3	11	8	indium contamination				0.96	1.39					0.24	0.32	45.87	0.23	50.99	73.74
BS3	11	9	indium contamination				3.66	1.24	0.25			0.95	0.39		24.02	14.35	55.13	71.32
BS4	7	1	bulk area, silty			0.63	9.13	5.66	0.53		0.65	0.14	0.51	0.44	27.15		55.15	57.75
BS4	8	1	bulk area, denser oxide			0.30	5.57	7.26	0.40		0.27	0.11	0.41	0.68	29.83		55.16	75.04

Table 4: Archive of EDS microanalytical data. Values are expressed as normalised atomic %, with analytical total in wt%. Oxygen calculated by stoichiometry.

				Fayalite/(Fa+Fo)	%Ca subst.	%Mn subst.
BS2	SOI 2	Spot 1	olivine core	0.94	2%	3%
BS2	SOI 2	Spot 2	olivine outer	0.96	3%	3%
BS2	SOI 2	Spot 3	olivine margin	0.97	4%	3%
BS2	SOI 2	Spot 4	olivine core	0.94	2%	3%
BS2	SOI 2	Spot 5	olivine margin	0.93	2%	3%
BS2	SOI 2	Spot 6	olivine margin	0.96	5%	3%
BS2	SOI 4	Spot 1	olivine core	0.92	2%	3%
BS2	SOI 4	Spot 2	olivine margin	0.93	3%	3%
BS2	SOI 4	Spot 3	olivine core	0.92	2%	3%
BS2	SOI 4	Spot 4	olivine margin	0.93	3%	3%
BS2	SOI 4	Spot 5	olivine margin	0.94	7%	3%

Table 5: EDS microanalyses of olivine in sample BS2

Figure 1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Tm
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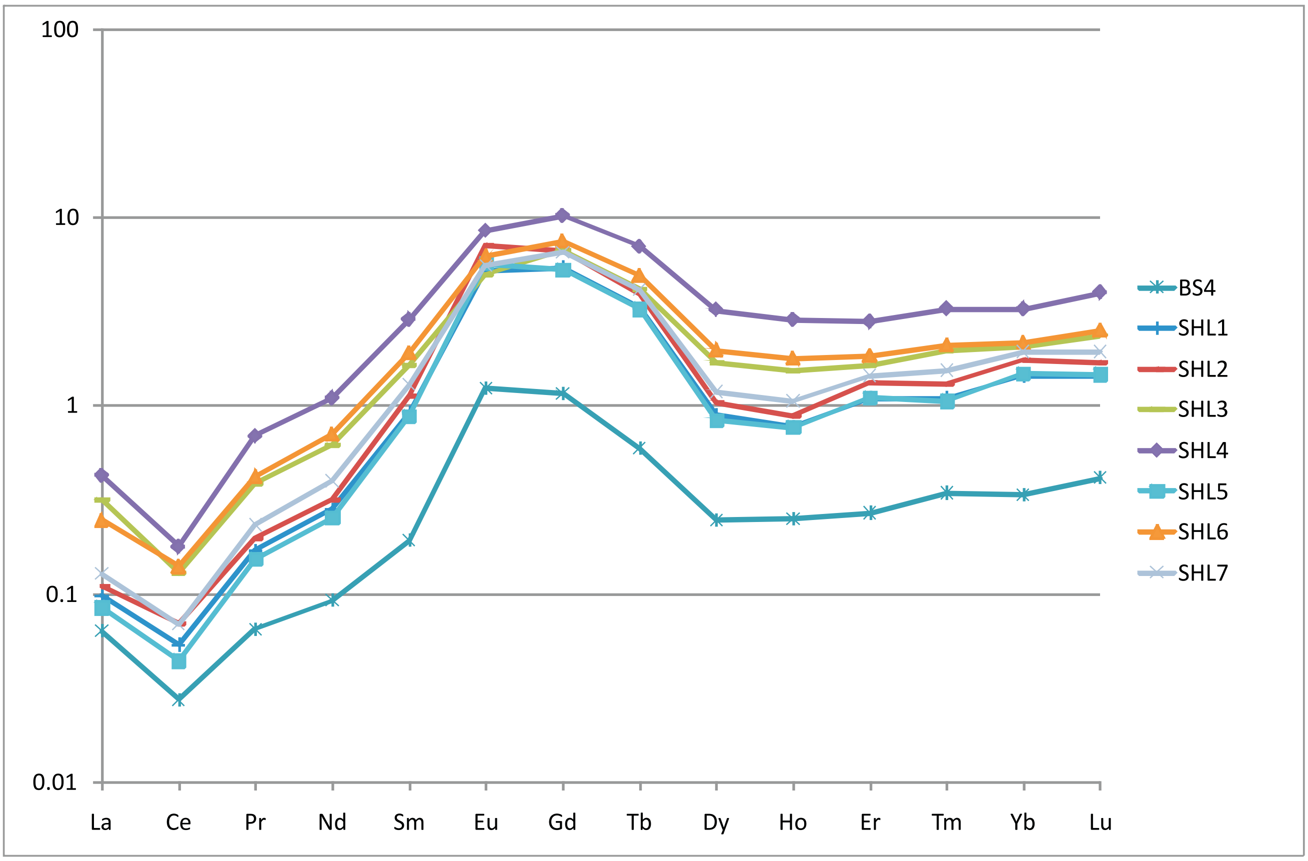
Lu
MUM3
BS1
BS2
BS3

Figure 2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Lu
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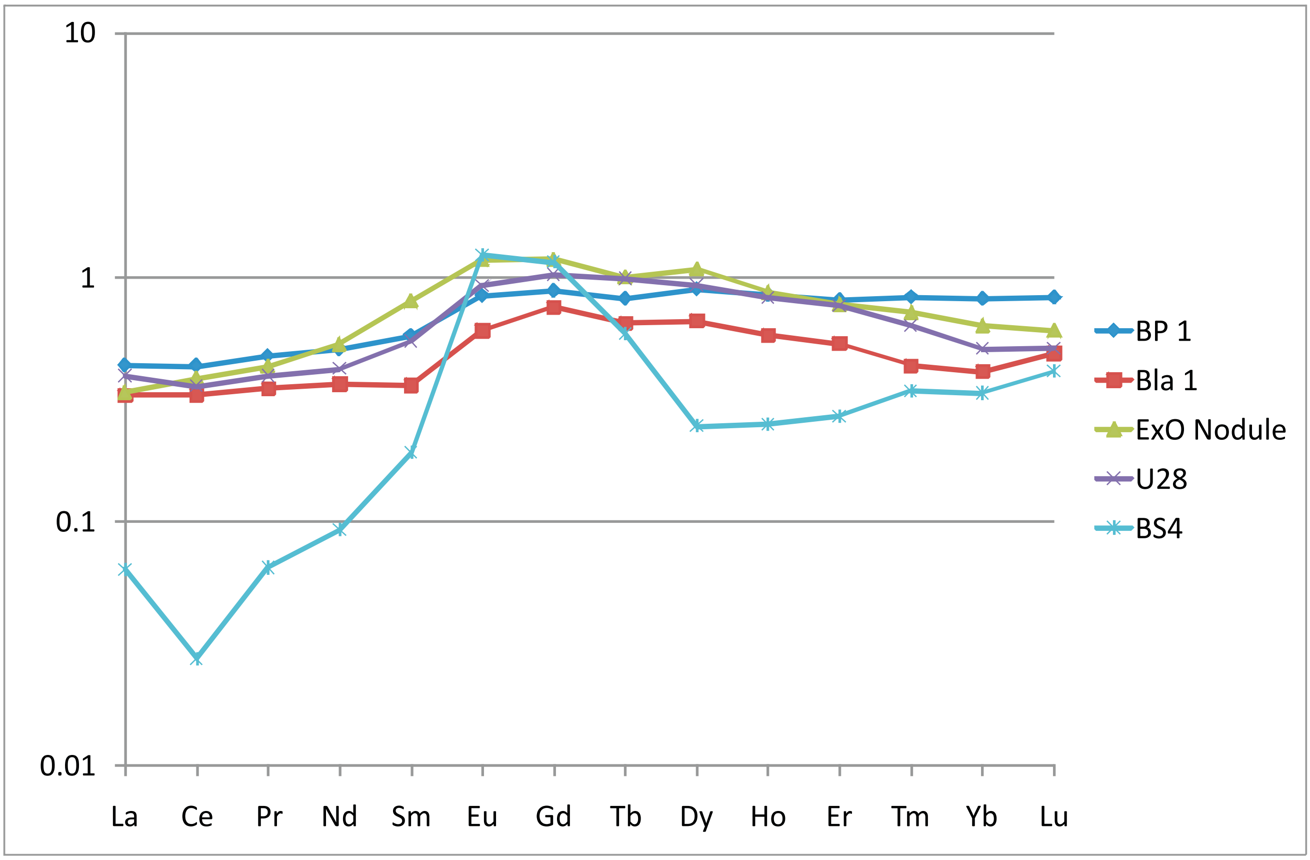
SHL2
SHL3
SHL4
SHL5
SHL6
SHL7

Figure 3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Lu
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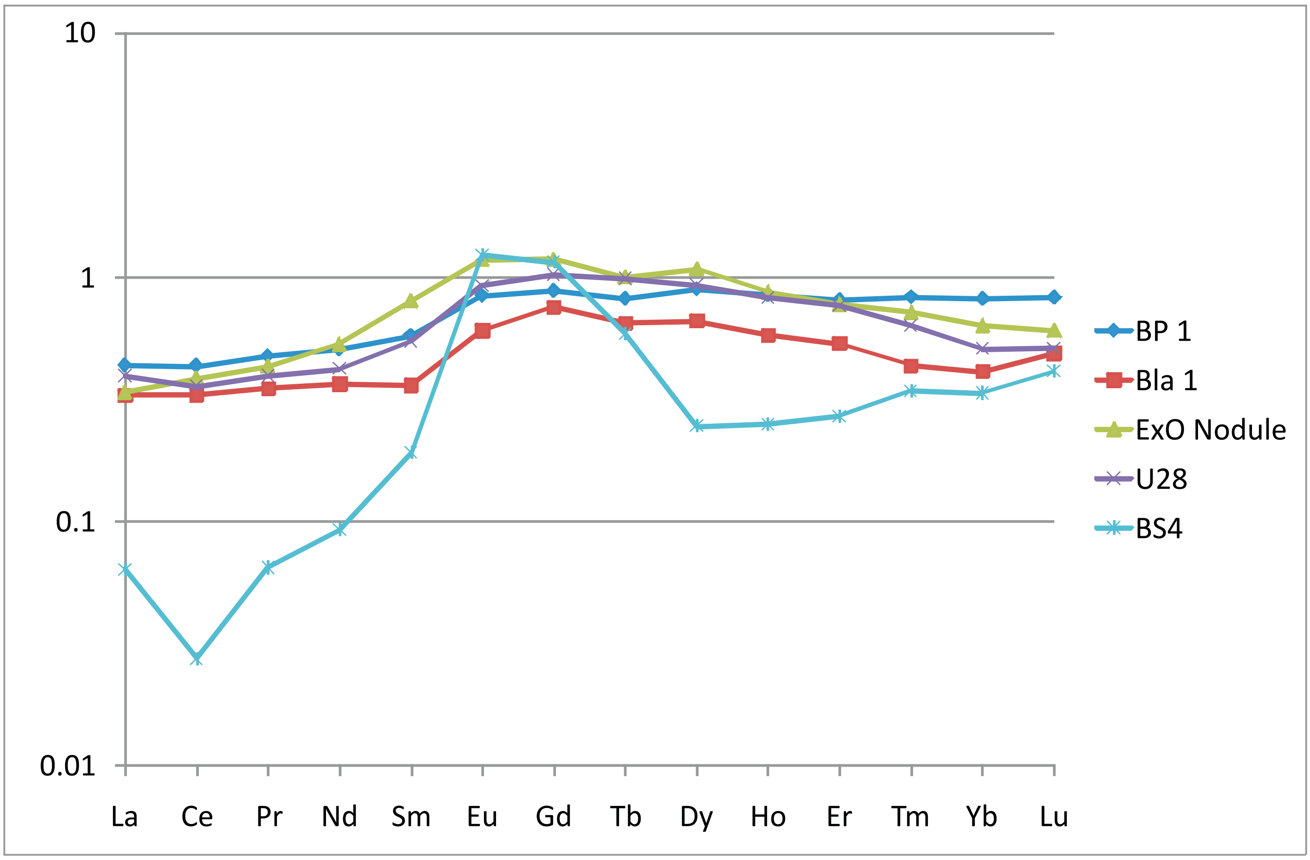
Bla 1
ExO Nodule
U28
BS4

Figure 3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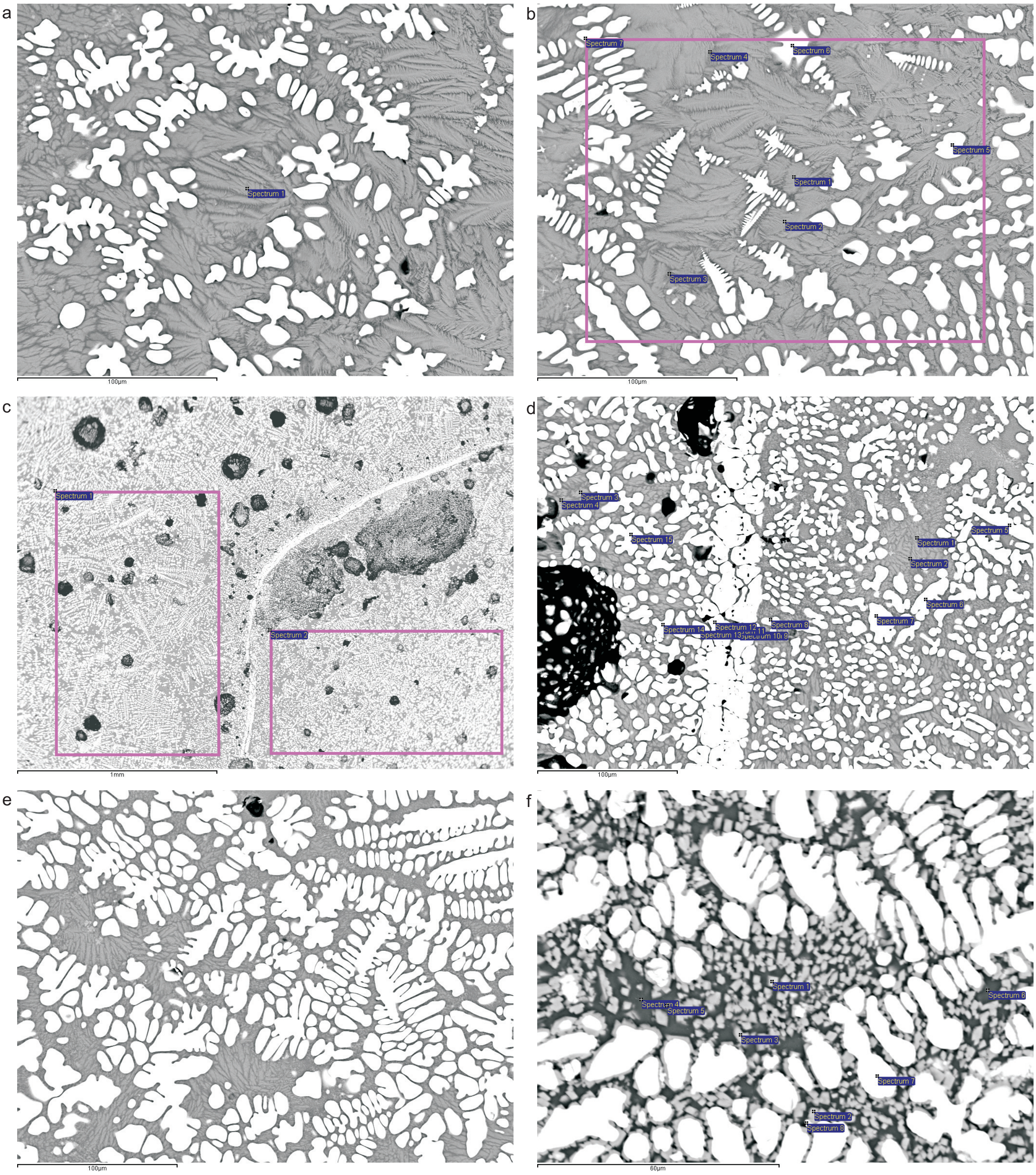
Lu
BP 1
Bla 1
ExO Nodule
U28
BS4

Plate 1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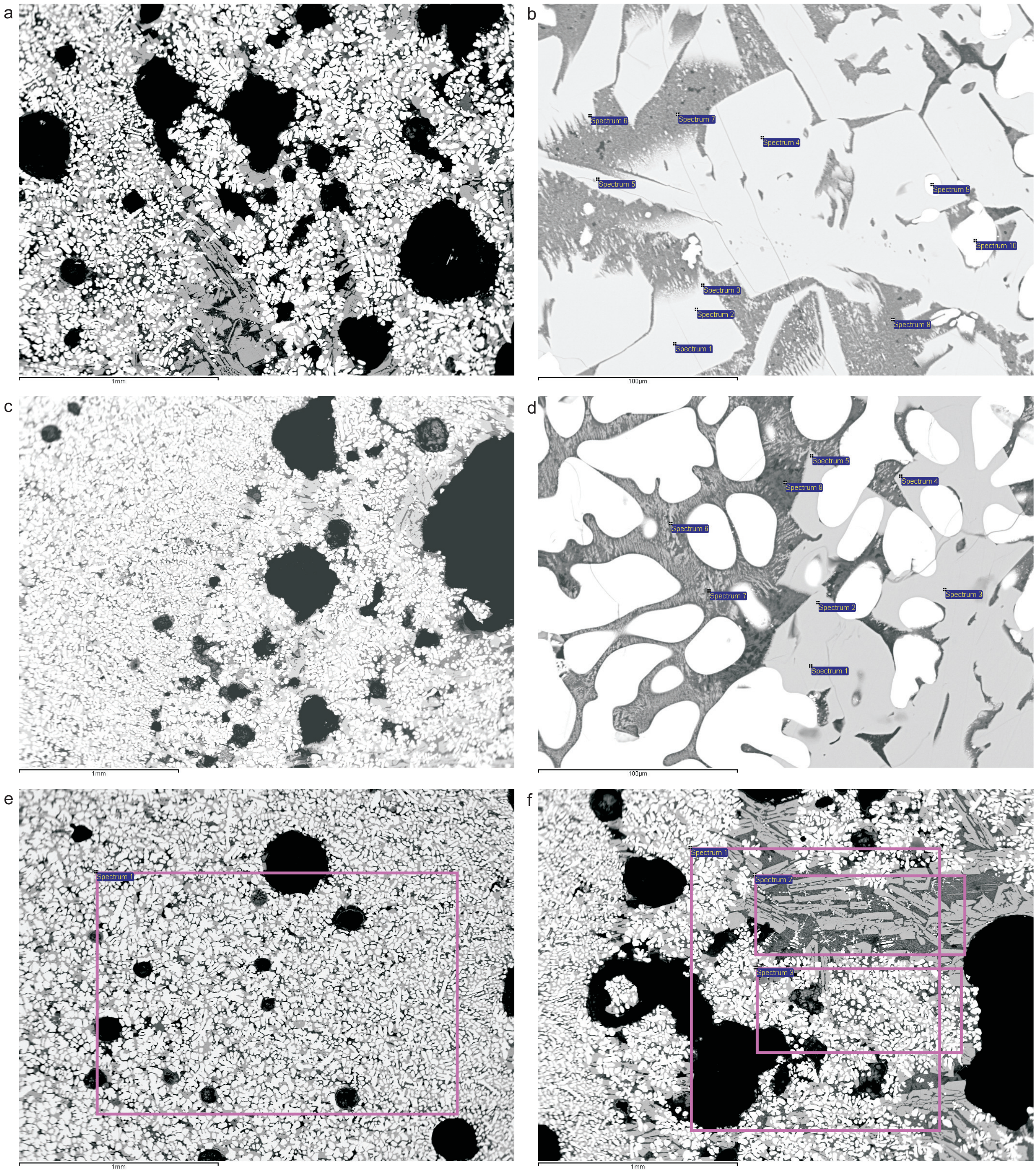
100µm
b
Spectrum 7
Spectrum 4
Spectrum 6
Spectrum 5
Spectrum 1
Spectrum 2
Spectrum 3
100µm
c
Spectrum 1
Spectrum 2
1mm
d
Spectrum 3
Spectrum 4
Spectrum 15
Spectrum 1
Spectrum 5
Spectrum 2
Spectrum 6
Spectrum 7
Spectrum 14
Spectrum 12
Spectrum 13
Spectrum 11
Spectrum 10
Spectrum 9
Spectrum 8
100µm
e
100µm
f
Spectrum 1
Spectrum 6
Spectrum 4
Spectrum 5
Spectrum 3
Spectrum 7
Spectrum 2
Spectrum 8
60µm

Plate 2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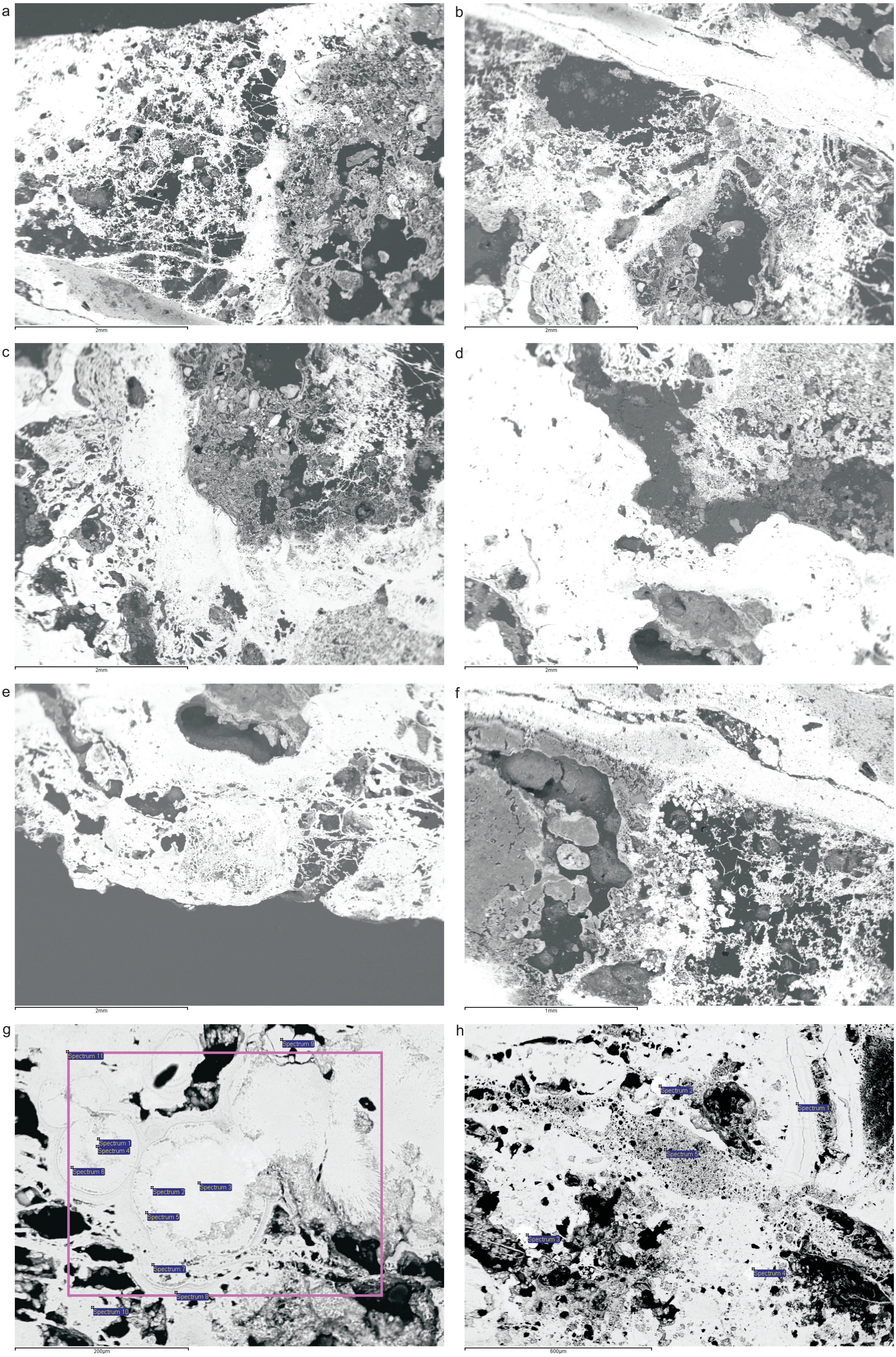
1mm
b
Spectrum 6
Spectrum 7
Spectrum 4
Spectrum 5
Spectrum 9
Spectrum 10
Spectrum 3
Spectrum 2
Spectrum 8
Spectrum 1
100µm
c
1mm
d
Spectrum 5
Spectrum 8
Spectrum 4
Spectrum 6
Spectrum 7
Spectrum 2
Spectrum 3
Spectrum 1
100µm
e
Spectrum 1
1mm
f
Spectrum 1
Spectrum 2
Spectrum 3
1mm

Plate 3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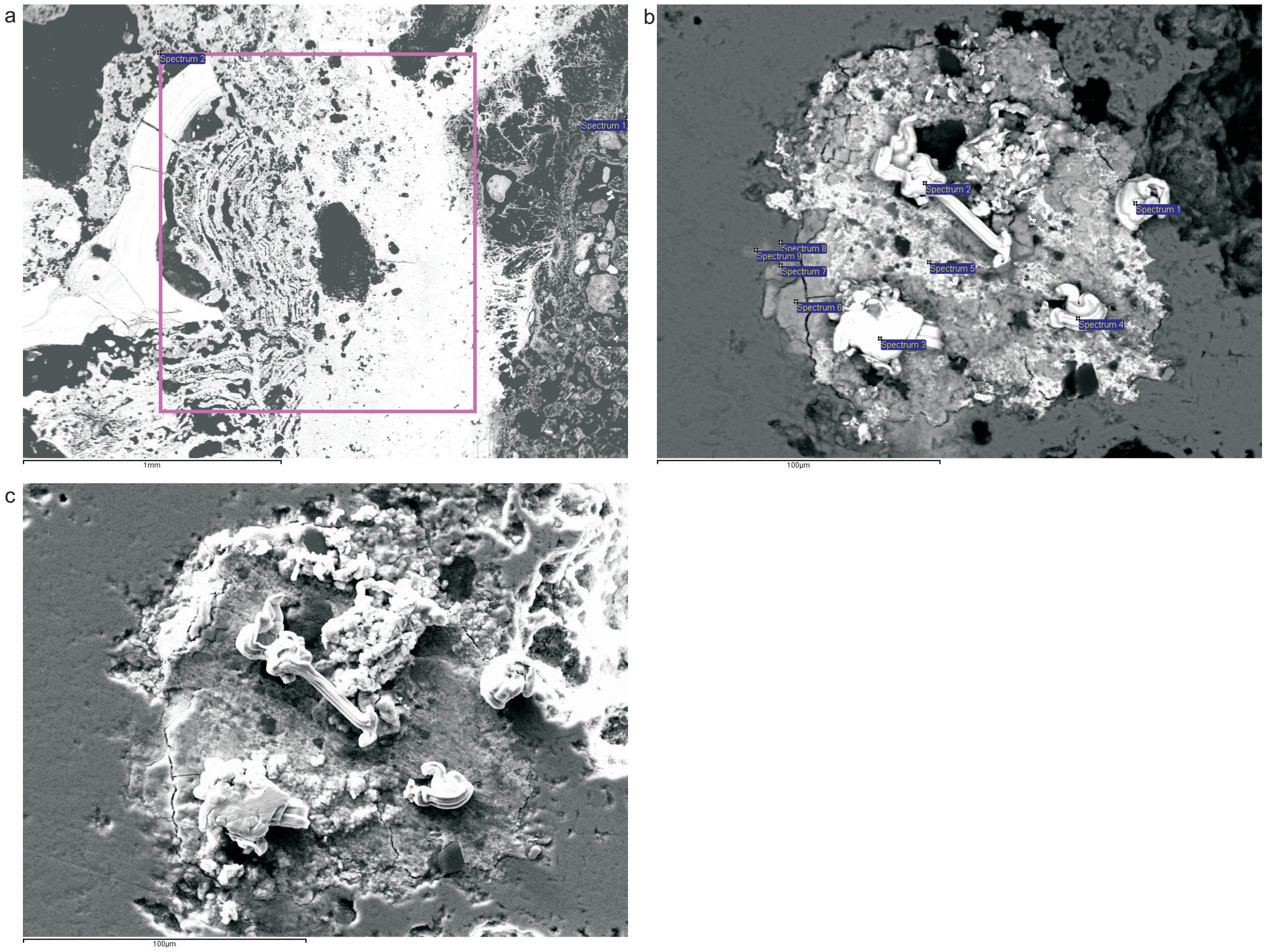
2mm
b
2mm
c
2mm
d
2mm
e
2mm
f
1mm
g
Spectrum 11
Spectrum 9
Spectrum 1
Spectrum 4
Spectrum 6
Spectrum 2
Spectrum 3
Spectrum 5
Spectrum 7
Spectrum 8
Spectrum 10
200µm
h
Spectrum 2
Spectrum 1
Spectrum 5
Spectrum 3
Spectrum 4
600µm

Plate 4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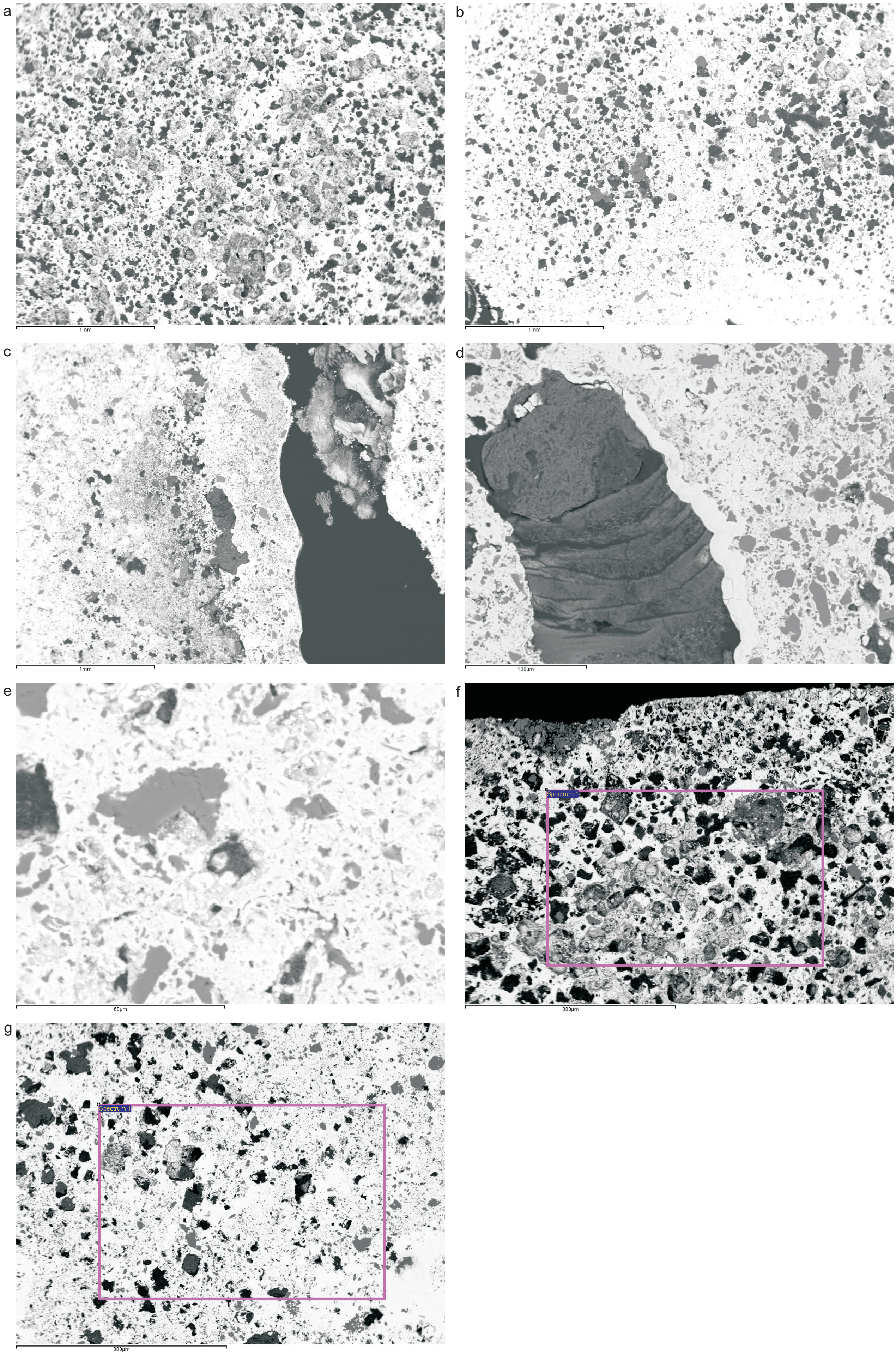
Spectrum 2
Spectrum 1
1mm
b
Spectrum 2
Spectrum 1
Spectrum 8
Spectrum 9
Spectrum 7
Spectrum 5
Spectrum 6
Spectrum 4
Spectrum 3
100µm
c
100µm

Plate 5


a
1mm
b
1mm
c
1mm
d
100µm
e
60µm
f
Spectrum 1
800µm
g
Spectrum 1
800µm

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