



Contamination Knowledge Report:

OR-CKP-04-1-S, 0

OR-CKP-05-1-S, 0

Particle investigation

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Summary

This report summarizes the properties of particles collected on mounts OR-CKP-04-1-S, 0 and OR-CKP-05-1-S, 0. Deployment schedules and activities for these contamination knowledge plates are summarized at the end of this report.

OR-CKP-04-1-S, 0: Scanning electron microscopy (SEM) examination identified ~ 40 particles/particle groups* ranging in size from 1.5-32 μm . Most particles are metal/metal oxides and carbonaceous. One Pb-bearing brass particle was identified. One siliceous mineral particle contained K. Three fiber-like particles were observed; one was C-rich and two were Al-rich.

OR-CKP-05-1-S, 0: This mount contained over 200 particles/particle groups* ranging in size from 0.1-35 μm , mostly occurring on the wafer edges. The most common particle type was metal/metal-oxides. One large, morphologically complex particle containing Bi was identified (possibly an Al-Bi alloy). Notably, 16 Pb-bearing particles with an average area of 1 μm^2 each were identified on two corners of the mount. These particles may be Si-carbide with absorbed Pb (II) ions or possibly a Pb-carbonate. Even with the presence of numerous Pb-bearing particles, the permissible Pb mass limit (0.79 ng/cm^2) was not exceeded on this mount ($\sim 0.054 \text{ ng}/\text{cm}^2$).

Recommendation

The presence of Pb-bearing materials is a potentially serious concern and efforts should be taken to identify the source of Pb-particles and mitigate its use if possible. In the case of CKP-04, Pb-bearing brass is most likely from the ATLO facility. In the case of CKP-05 the Pb-bearing particles are only located at the edge of the wafer and are most likely introduced by handling. An additional wafer from this batch of four plates should be examined to confirm this.

Procedures

* Not including Si-only particles derived from the mount.

The mounts were introduced into a SEM for particle characterization. Image mosaic was obtained for each Si wafer mounts (OR-CKP-04-1-S, 0 and OR-CKP-05-1-S, 0) taken at 150X using LAGE, each frame acquired for 30 s. Each mount was then examined through SEM analyses over a one-day period (~ 10-12 hours). All particles $\geq 0.05 \mu\text{m}$ in size were examined. All EDX spectra were obtained for 60 s at 15 keV with one exception (Spt 12 was acquired for 200 s., mount OR-CKP-05-1-S, 0; Spt = spectra are EDX spectra obtained in spot mode).

Observations

The particle population on OR-CKP-04-1-S, 0 is somewhat chemically diverse and consists primarily of particles $< 20 \mu\text{m}$ in size. The SEM image mosaic, shown in Figure 1, shows particle locations on this mount. The majority of particles are located near the edges. The most common particle types are metal/metal-oxides and those containing C. One Pb-bearing particle was identified on the lower right corner of the mount. This particle, $\sim 7 \times 16 \mu\text{m}$, also contains Zn and Cu and so is consistent with brass. Although Zn is not a diagnostic element, three other particles with unknown origins also contained Zn. Three Fe-, Cr-particles contained Ni; these are likely particulate residue from tweezers. One siliceous mineral particle contained K. Three fiber-like particles were observed; one was C-rich and two were Al-rich. The largest fiber on the surface was lost prior to analysis likely due to charging by the electron beam during imaging. However, two particles shed from the fiber remained on the substrate; they indicated the fiber was C-rich. Figure 2 shows a spectrum of the Pb-containing particle and a representative spectrum of a Ni-containing Fe-,Cr-particle. Table 1 summarizes the size, location, shape and major element abundances of these particles.

The particle population on OR-CKP-05-1-S 0 is chemically diverse and consists mainly ($>85\%$) of particles $< 15 \mu\text{m}$ in size. An SEM image mosaic of this mount displaying the particle locations is shown in Figure 3. The vast majority of particles are located along the mount edges. The most common particle type was metal/metal-oxides. One large and morphologically complex particle containing Bi was identified; this is possibly an Al-Cu-Bi alloy. One Ni-containing particle, $\sim 0.3 \times 0.7 \mu\text{m}$, also contained C, O, and Cl. Notably, Pb-bearing particles were identified on the upper left and lower right corners of the mount; they also contained C minor O. We speculate these particles may be composed of Si-carbide with absorbed Pb (II) ions or may be a Pb-carbonate. In the former case, it is not possible however to distinguish Si present in the particles from that contributed by the background using SEM. In the latter case, they may be composed of PbCO_3 although in some analyses the O abundance appears to be low. One unusual Pb particle contained Cu in addition to O and C. *Pb-bearing particles have unusual compositions and based on our assessment, are of unknown origins.* Figure 4 shows a representative spectrum of one of the Pb-containing particles and a spectrum of the Ni-containing particle. Table 2 summarizes the size, location, shape, and major element abundances of these particles. Even with the presence of numerous Pb-bearing particles, the maximum permissible Pb mass limit (0.79 ng/cm^2) was not exceeded on this mount ($\sim 0.054 \text{ ng/cm}^2$). Note the center of this mount is nearly particle free.

Discussion

Key diagnostic elements: The Contamination Knowledge effort is monitoring the abundances of the following diagnostic elements in collected particles: C, K, Ni, Sn, Nd, and Pb. Below is a summary for each of these elements:

OR-CKP-04-1-S, 0

- C: Few particles were C-rich. Most were rich in C or C and O; none had typical elemental signatures of biological contaminants.
- K: K was observed in 1 particle; likely Si-rich mineral.
- Ni: Minor Ni was observed in three Fe-, Cr-particles, two of which were < 5 μm and one of which was 3 x 17 μm in size.
- Sn: Not observed
- Nd: Not observed
- Pb: Minor Pb was observed in one 7 x 16 μm particle near lower mount edge.

OR-CKP-05-1-S, 0

- C: Few particles were C-rich. None had typical elemental signatures of biological contaminants such as skin flakes or perspiration.
- K: Not observed.
- Ni: Minor Ni was observed in one $\sim 0.3 \times 0.7 \mu\text{m}$ particle which also contained C, O, and Cl.
- Sn: Not observed
- Nd: Not observed
- Pb: Minor Pb was observed in 5 regions, all of which were located near edges. The largest of the Pb-containing particles was $\sim 4 \mu\text{m}$. Pb-bearing particles have unusual compositions and unknown origins.

Pb-bearing particles:

OR-CKP-05-1-S, 0

Based upon the size of these particles and assuming that they contain ~ 75 wt.% Pb (e.g. PbCO_3 ; cerussite), we estimate a mass loading of 0.054 ng/cm³, which does not exceed the desired mass limit in TAGSAM.

- Metal
- C-bearing
- Si-bearing
- Al-bearing
- Other

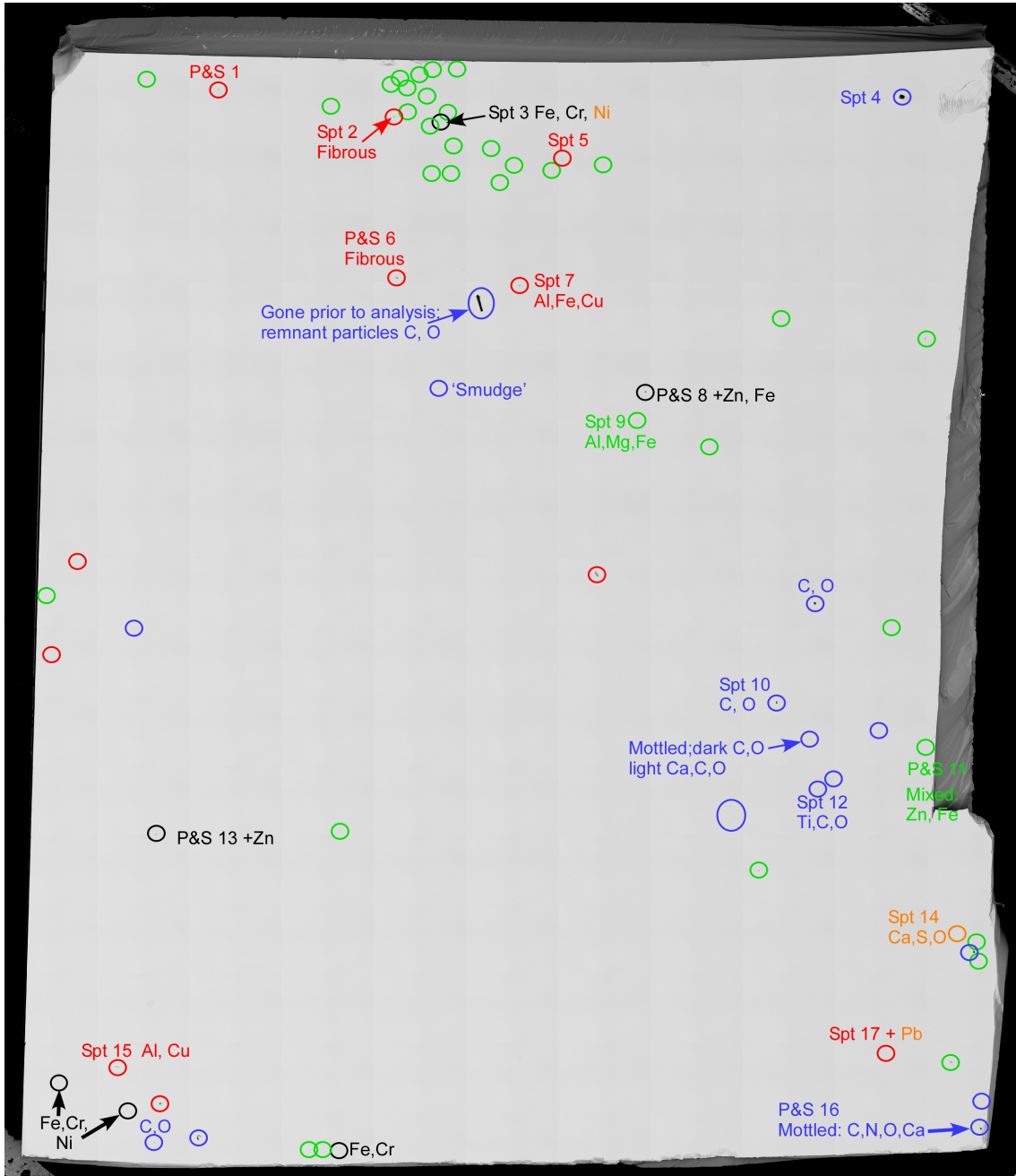
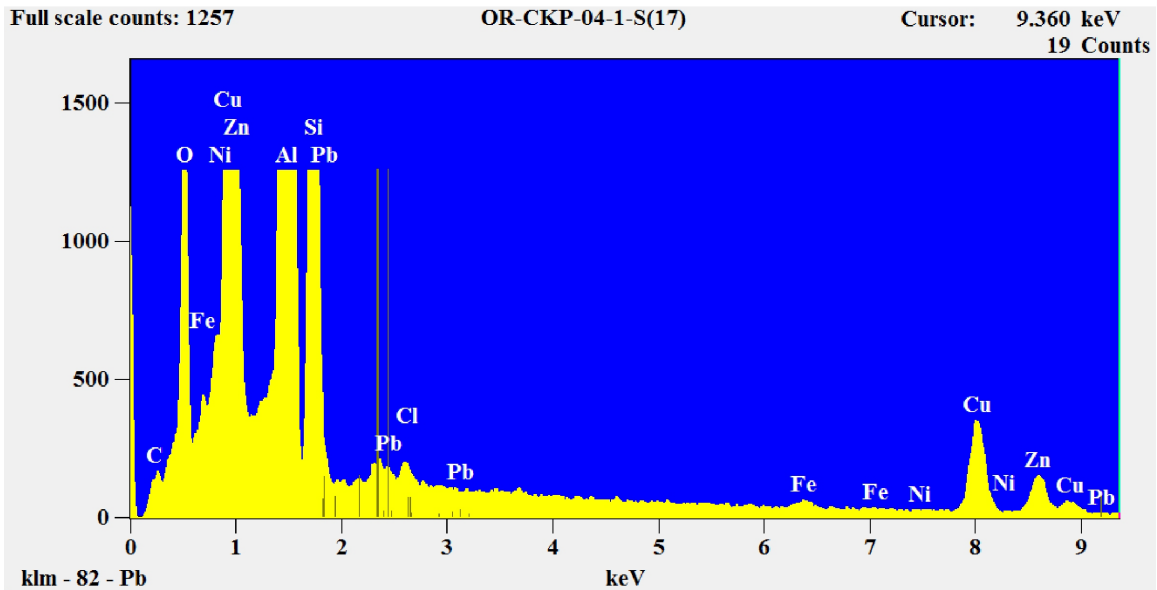


Figure 1: OR-CKP-04-1-S ,0 SEM montage, particle map. Numbers in parentheses correspond to EDX spectral file numbers.

Pb-containing Particle



Ni-containing Particle

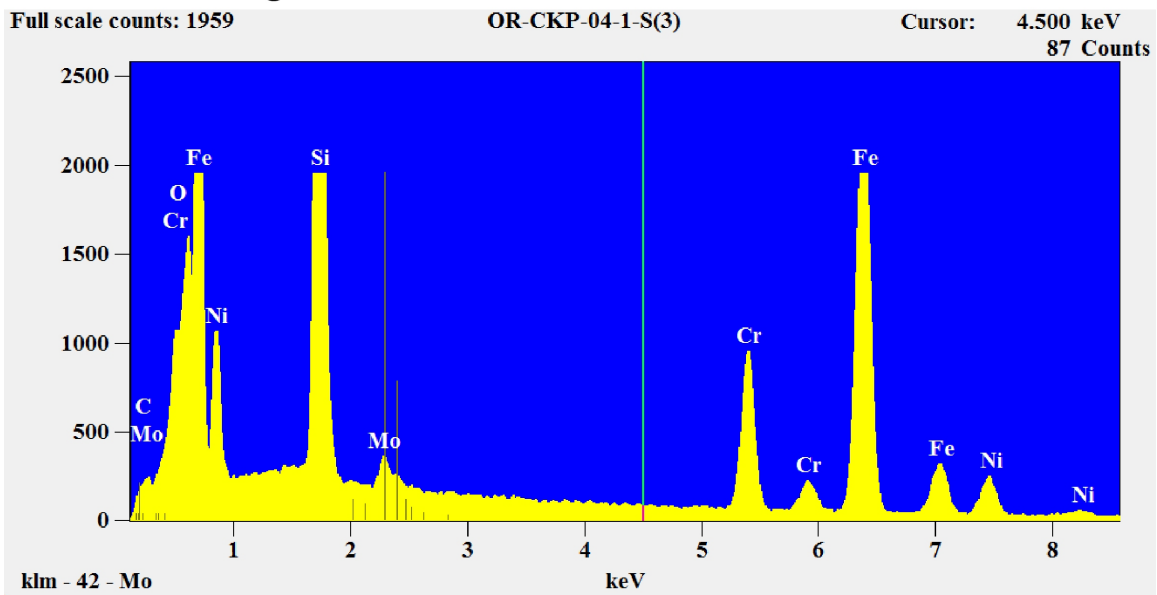


Figure 2: EDX spectra of two particles from OR-CKP-04-1-S,0. Top: Al-rich particle with minor O, Cu, Zn, Cl and Pb. Bottom: Representative spectrum of a Fe-,Cr-containing particle with minor Ni and Mo.

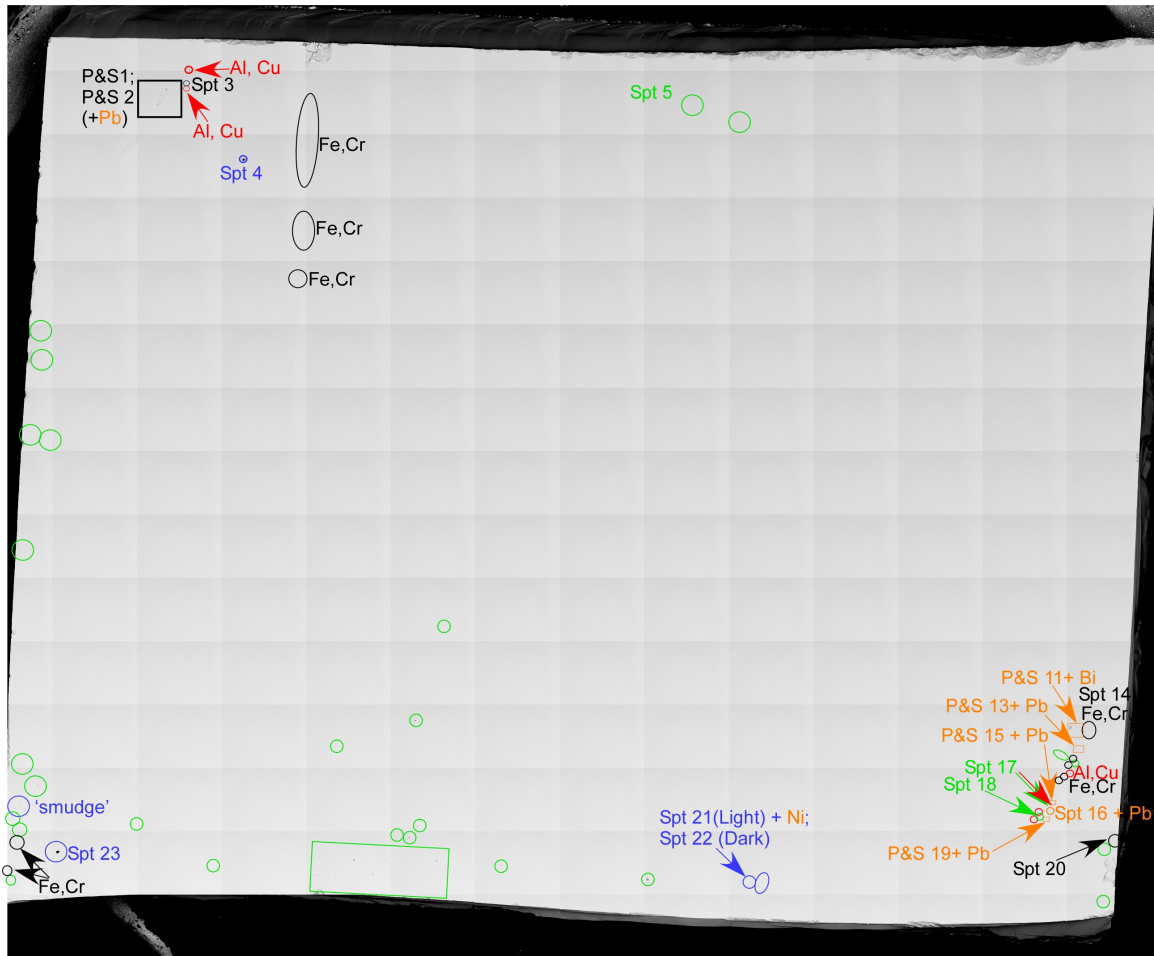
Table 1: Summary of particles identified on OR-CKP-04-1-S ,0. Key diagnostic elements are highlighted in red. Zn is highlighted in blue.

Approximate Size (µm)	#Particles	Montage	Shape	Chemistry
12x15	1		Irregular	P&S (1) 1. bkg 2. Al metal (trace O)
~ 30 (if extended)	1		Fibrous, curled upon self	Spt 2 Al, Si, O, Mg
2x1.5	1		Irregular	Spt 3 Si, Fe, Cr, Ni, Mo
40x42	1		Irregular	Spt 4 Si, C, O
6x4	1		Irregular	Spt 5 Al, Mg, C, O, Fe, Si
18x4	1		Fiber	P&S 6 Al, Mg, C, O (Si) maybe Cu
7x11	1		Irregular	Spt 7 Si, Al, Fe, O, Cu, Mg, C
3x2 - part of very large fiber?	1		Part of huge fiber—lost prior to analysis. Likely beam sensitive. Only tiny piece left	C, O (no spt)
8x4	1		Irregular	Si only for spiral fractured grain – No image or spt
13x11	1		Irregular; mottled light and dark regions	P&S 8 1. Zn, Si, Al, O, C, Cl, S, Ca, P, K, Fe, Ti, Cu 2. Si, Fe, Al, Zn, O, Cl, C, Ca, S 3. Al, Si, C, Zn, O, Cl, Mg, Ca, S
6.5x3.5	1		Irregular	Spt 9 Si, Al, Mg, K, Fe, O, P
4x4	1		Irregular	C-rich smudge (no O; no spt)
4x4	1		Irregular	Al, O (no spt)
5x3	1		Irregular	Al, O, C (no spt)

2.5x2.5	1		Irregular	C only (no spt)
14x10	1		Irregular	C, O sample folded on self— beam damage (no spt)
7x21	1		Irregular	Spt 10 C, O
9x4	1		Mottled, light and dark regions	Dark-C, O Bright-Ca, C, O (no spt)
4x5	1		smudge	C, O (no spt)
Whole region: ~ 18x9	1 (possibly several parts)		Irregular	P&S 11 1. Si, O, Zn, Al, Cl, Mg, Fe, K, Ca, S (no C) (medium dark) 2. Si, O, Fe, Zn, Cl, Al, Mg, S, Ca, C (bright) 3. Ca, O, Ca, Zn, Al, Mg, S, Cl
2x3	1		Irregular	C (no spt)
1x2	1		Irregular	Spt 12 Ti, C, O, Al
2x5, 4x5	2		smudges	C only (no spt)
9x25	1		Sharp, irregular	P&S13 Al, Zn, O, Mg, Cl
19x21	1		Irregular	C, O (no spt)
10x7	1		Irregular	Si, Mg, O, Fe (no spt)
5x3	1		Irregular	Spt 14 Si, Ca, S, O, Fe
40x7	1		Irregular	Spt 15 Al, Cu, O
14x17	1		Crumpled, irregular	Al, hint O
6x63 (bent on itself)	1		fiber	C, O (no spt)

4x6	1		Smudge?	C, O (no spt)
3x17	multiple		Smear of particles	Fe, Cr, Ni (no spt)
5x4, 1x1.5	2		Irregular	Fe, Cr, Ni (no spt)
1.5x1.5; 1.8x2	2		Irregular	Fe, Cr (no spt)
14x17	1		Mottled, light and dark regions	P&S 16 1. dark- Si, C, Ca, O, S, N, Cl 2. light-Si, Ca, C, O, S, N, Mg, Al
6x5	1		C-shaped smudge	C, O (no spt)
7x16	1		Irregular	Spt 17 Al, Cu, Zn, O, S, Cl, Pb, Fe
3x4	1		Irregular	Al, trace O (no spt)
55x15	1		Irregular	Al, trace O (no spt)

- Metal
- C-bearing
- Si-bearing
- Al-bearing
- Other



Spt 1, Spt 6 (Background only)

Out of field of view on left side:

- Spt 7 (C, Cl)
- Spt 8 (C,S,Fe)
- Spt 9 (C)

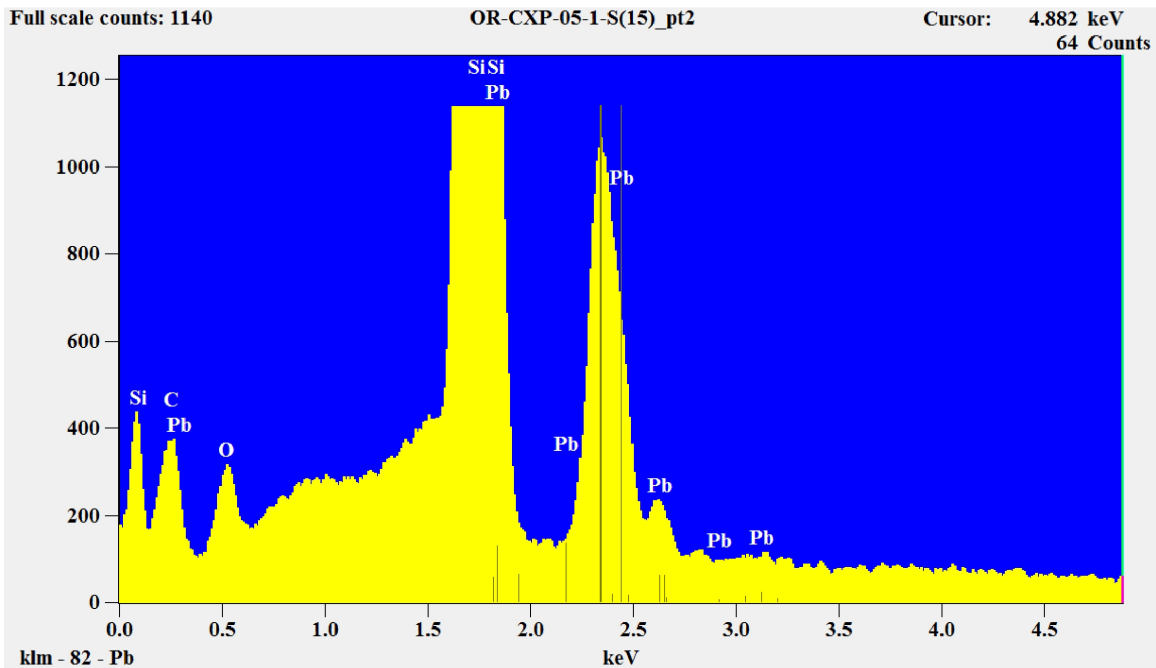
Repeat analysis-same region as P&S 11

Spt 10 (Al, Cu, Bi, O) for 60 s.

Spt 12 (Al, Cu, Fe, K, O, Bi) for 200 s.

Figure 3: OR-CKP-05-1-S ,0 SEM montage, particle map. Numbers in parentheses correspond to EDX spectral file numbers.

Pb-containing Particle



Ni-containing Particle

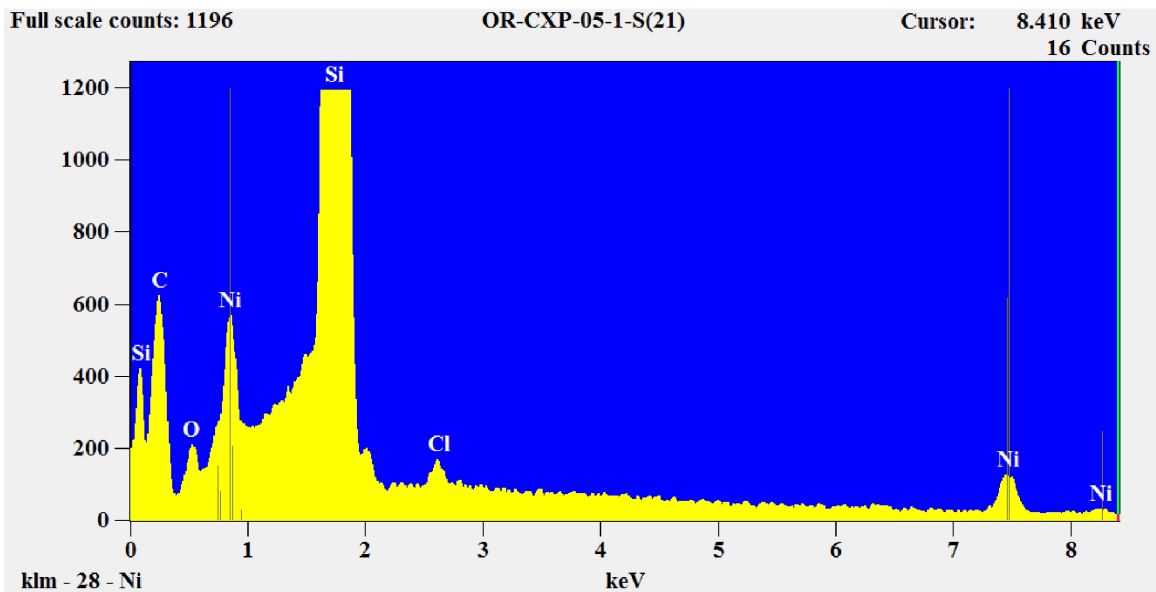


Figure 4: EDX spectra of two particles from OR-CKP-05-1-S ,0. Top: A representative spectrum of a Pb-, C-rich particle with minor O. Bottom: Ni-containing particle with C and minor O and Cl.

Table 2: Summary of particles identified on OR-CKP-05-1-S ,0. Key diagnostic elements are highlighted in red. *Spt* refers to EDX spectra obtained in spot mode.

Approximate Size (μm)	#Particles	Montage	Shape	Chemistry
0.3 to 18 um	multiple		Irregular, near edge	See P&S OR-CXP-05-1-S(1) 1. Si (bkg) 2. Al, Si, O, Cu 3. Al, O, Cu, Si 4. Fe, Cr, Si, Al 5. 5. Al, Si, Cu, O 6. Al, Cu, Si, O
As above	multiple (close up of P&S (1))		Irregular, near edge	See P&S OR-CXP-05-1-S(2) 1. Si, Pb , C, O (2 um) 2. Fe, Cr, Si, Al 3. Fe, Cr, Si, Al 4. Fe, Cr, Si, Al 5. Fe,Cr,Si, Al, O 6. Si, Pb , C, O, Al (1.6 um) 7. Si, Al, Cu, O
5x11	1		Irregular, near edge	Spt 3 Fe, Cr, Si, Al, O
4x8; 7x7	2		Irregular, near edge	Si, Al, Cu (no spt)
11x15	1		Irregular, near edge	Si, Al, Cu, O (no spt)
< 0.5 to 9x12	multiple		Irregular	Fe, Cr, Si
26x22	1		Rounded	Spt 4 C, O, Si, Cl, Ti
4x2	1		Irregular	Spt 5 Si, O, Al, Ti
~0.1 to 2	multiple		Irregular	Fe, Cr (no spt)
6x7	1		Irregular; smudge	Si, hint C (no spt)
10x11	1		Irregular	Spt 7 Si, C, Cl, minor O
Largest 1x2, down to ~ 0.2	multiple		Irregular	Fe,Cr
Range: ~0.5x0.5 to 4x1	multiple		Irregular	Fe, Cr (no spt) Spt 8

~2x2	1		Irregular	Spt 9 Si, C, minor S, Ti, Ca
Bright particles Fe, Cr Range: 1x1 to 6x7 Darker region with Bi ~ 25x35	multiple		Irregular	See P&S 11 1. Al, Cu, O, Bi, Si dark region same as Spt 10 (60 s.) & Spt 12 (200 s.) 2. Fe, Cr, Al, Si bright region 3. Fe, Cr, Si 4. Si, C, O
Range: ~1 up to 2x5	multiple		Irregular	See P&S 13 1. Pb, C, O, Si 2. Pb, C, O, Si 3. Pb, C, O, Si 4. Pb, C, O, Si 5. Pb, C, O, Si 6. Pb, C, O, Si 7. Pb, C, O, Si 8. Si, O, Al, Mg, Fe
Range ~0.2 to 512	multiple		Irregular	Spt 14 Fe, Cr, C, O
Range: 0.2x0.2 to 2x2	multiple		Irregular	See P&S 15 (spt for larger particles only) 1. Pb, C, O 2. Pb, C, O 3. Pb, C, O
1x2	1		Irregular	Spt 16 Cu, Pb, O, C
2x6	1		Irregular	Spt 17 Si, O, C
6x3	1		Irregular	Spt 18 Si, Mg, O, C, Fe
Range: 0.3x0.6 to 1x2	multiple		Irregular	P&S 19 1. Si, Pb, O, C 2. Si, Pb, C, O 3. Si, Pb, C, O 4. Si, Pb, C, O
6x6	1		Irregular	Spt 20 Fe, Cr, Ni, S Si
0.3x0.7	1		Irregular	Spt 21 Si, Ni, C, O, Cl
2x4	1		Smudge/halo	Spt 22 Si, C, O, Cl, S (Dark halo surrounding grain in Spt 21)

25x24	1		Irregular	Spt 23 Si, C, O, S, Cl
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Calculations:

Estimate 16 1x1x1 μm PbCO_3 grains

PbCO_3 density $\sim 6.6 \text{ g}\cdot\text{cm}^{-3}$

M.W $\text{PbCO}_3 = \begin{matrix} 267 & 207 & \text{Pb} \\ & 12 & \text{C} \\ & 3 \times 16 & (48) & \text{O} \end{matrix}$

Fraction of mass of PbCO_3 due to Pb = $207/267 \sim 0.775$

Σ volume PbCO_3 grains = $16 \mu\text{m}^3$

Since $10,000 \mu\text{m} = 1 \text{ cm}$ then $1 \mu\text{m}^3 = 10^{-12} \text{ cm}^3$

Σ volume PbCO_3 grains = $1.6 \cdot 10^{-11} \text{ cm}^{-3}$

Σ mass PbCO_3 grains = $(1.6 \cdot 10^{-11} \text{ cm}^{-3}) \cdot 6.6 \text{ g}\cdot\text{cm}^{-3}$

= $1.056 \cdot 10^{-10} \text{ g}$

Assume 0.775 of this is due to Pb then Σ mass Pb = $(1.056 \cdot 10^{-10} \text{ g}) \cdot 0.775$

= $8.184 \cdot 10^{-11} \text{ g}$

= 0.08184 ng

If the mount surface area is estimated to be $1 \text{ cm} \cdot 1.5 \text{ cm} = 1.5 \text{ cm}^2$

Then mass of Pb/ $\text{cm}^2 = 0.08184 \text{ ng} / 1.5 \text{ cm}^2 = 0.054 \text{ ng} \cdot \text{cm}^2$

Composition comparison of unknown Pb-bearing particle (mount OR-CKP-05-1-S, 0)
with known PbCO_3 standard (<http://webmineral.com/data/Cerussite.shtml>)

Unknown Pb-bearing particle was calculated using Fit method 'Filter without Standards'
and Correction Method 'ZAF' - Si was removed prior to data reduction.

Guestimate for spt OR-CKP-05-1-S (15) pt2

STD (Cerussite, PbCO_3)

Element Element Wt. %

Element Element Wt. %

C K --- 10.69

C K --- 4.49

O K --- 13.77

O K --- 17.96

Pb M --- 75.54

Pb M --- 77.54

Total -----
100.00

Total -----
100.00

ATLO Events

July		Exposed: 7/14/2015-8/17/2015, Location: SSB High Bay																											
Event	Wed	Thu	Fri	Sat	Sun	Mon	Tue	Wed	Thu	Fri	Sat	Sun	Mon	Tue	Wed	Thu	Fri	Sat	Sun	Mon	Tue	Wed	Thu	Fri	Sat	Sun	Mon	Tue	
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28		
TAGSAM Deployment																													
Spacecraft Lift to Rotation Fixture																													
OVIRS Install																													
TAGCAMS Install																													

Color Key

Room 202	SSB High Bay	RAL	SSL	MTF	PHSF
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