



Contamination Knowledge Report: OR-GCKP-03-1,0 Particle Investigation

Kathie Thomas-Keprta (Analysis)
Scott Messenger (SampleWG lead)
Harold C. Connolly Jr. (MSS)
July 1, 2015

Summary

This report summarizes properties of particles collected on a Si-wafer from the third deployed GAKMAPS (**OR-GCKP-03 or 'CK3'**). This unit was deployed during *Avionics box assembly and SRC functional post-vibe*.

Scanning electron microscopy examination of the Si wafer identified 43 particles/particle groups ranging in size from 2-100 μm . The most common particle types were metal/metal oxides (27 particles), followed by biogenic/C-rich (10 particles), and minerals, salts, or other (6 particles). Notably, two Pb-bearing brass particles were identified. In addition, one very large and complex carbonaceous particle was identified. The presence of two large Pb-bearing brass particles is troubling because the desired Pb mass limit (0.79 ng/cm^2) may not be satisfied by the 100A/2 visibly clean standard and was exceeded on this mount (1.5 ng/cm^2)

Recommendation

The presence of Pb-bearing materials in TAGSAM is a potentially serious concern. Since the science-driven abundance limit of 0.79 ng/cm^2 cannot be guaranteed by the established cleaning standard, efforts should be taken to identify the source of brass and mitigate its use if possible.

CK3 Deployment

CK3 was deployed May 11 – June 10, 2015 during avionics box assembly and SRC functional post-vibe. Anomaly reported: *'About halfway through deployment, it was noticed some of the foils looked bent – could have been a sign someone bumped it or even set something on it. Moved plates to SSL on June 8th just before SARA lift and removed June 10th when the chamber closed for SARA TVAC - SRC was opened during this time.'*

Procedures

One randomly selected Si-wafer mount each was removed from CK1 and CK2 and placed in a pre-cleaned Al can with a glass lid. The mount was then introduced into a scanning electron microscope for particle studies. An image mosaic was obtained for the entire Si wafer mount (OR-GCKP-03-S, 0) taken at 150X using LABE, each frame 30 s. The mount

was then examined over a one day period by SEM. All particles >2 μm in size were examined. EDX spectra were obtained for 100 s at 15 keV.

Observations

The particle population on CK3 is chemically diverse and consists mainly (>75%) of particles >10 μm in size. An SEM image mosaic of CK3 showing particle locations is shown in Fig 1. A total of 43 particles/particle groups were examined, ranging in size from 2-100 μm . Based upon EDX analysis, the most common particle types were metal/metal oxides (27 particles), followed by biogenic/C-rich (10 particles), and minerals, salts, or other (6 particles). Table 1 summarizes the size, location, shape, and major element abundances of these particles.

Several particles have compositions that stand out on this sample mount. Two Pb-bearing brass particles were identified and their spectra are shown together in Figure 2. The largest particle on this mount (106x72 μm) is carbonaceous and is encrusted with various salts and minerals. An SEM image of this grain is inset in Figure 1. K-bearing particles are usually associated with biogenic origin. However on this mount several K-bearing carbonaceous grains are found that have unusual compositions. An example EDX spectrum of one such grain is shown in Figure 3.

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. So long as the cleaning standard of 100A/2 is met for the TAGSAM surface, the abundance limits of C, K, and Ni will be satisfied. On the other hand, this cleaning standard may not be sufficient to protect certain types of scientific studies if highly unusual abundances of Sn, Nd, and Pb are found. Below is a summary for each of these elements:

- C: On the order of 25 % of collected particles were C-rich. A few of these had typical elemental signatures of biological contaminants such as skin flakes or perspiration. Other C-bearing particles included a probable calcite and inorganic particles.
- K: K was observed in 6 particles and at least 3 of these are of probable biogenic origin (such as skin flakes). Several K-bearing particles have unusual compositions and unknown origins.
- Ni: Trace Ni was observed in one 3x5 μm Fe,Cr-rich particle. Given its location near the edge of the wafer, it may have been introduced by handling with stainless steel tweezers.
- Sn: Not observed
- Nd: Not observed
- Pb: Minor Pb (1-few wt. %) was observed in two brass particles. These were large particles (10 – 20 μm) in the interior of the mount.

Pb-bearing particles: Based upon their EDX spectra, two particles have Cu, Zn, and Pb abundances that are consistent with brass. Pb is a common trace (~2%) metal used to improve the workability of brass. Pb-bearing brass is used in screws, nuts, gears, and keys. Based upon the

size of these particles and assuming that they contain 2 wt% Pb, we estimate a mass loading of 1.5 ng/cm³, approximately twice the desired mass limit in TAGSAM.

- █ Metal (likely)
- █ C-bearing
- █ Si-bearing
- █ Al-bearing

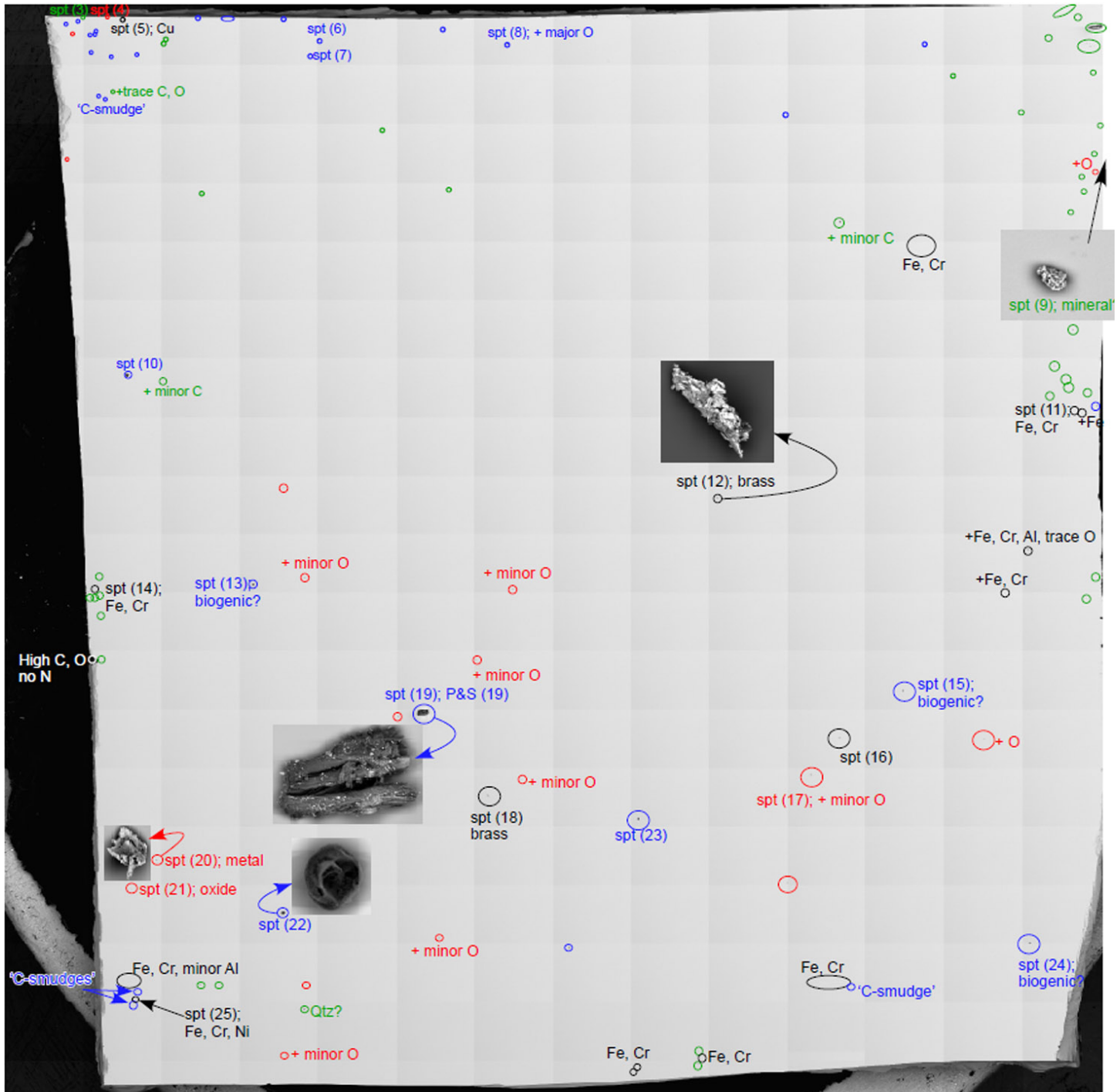


Figure 1: OR-GCKP-03 or 'CK3' SEM montage, particle map. Numbers in parentheses correspond to EDX spectral file numbers.

Table 1: Summary of particles identified on OR-GCKP-03. Particle entries are color shaded as metallic/metal oxide (blue), organic/C-rich (yellow), of potentially warranting

Approximate Size (µm)	#Particles	Montage	Shape	Chemistry
6x6			Irregular	Si, Al (no spt)
4x6	Grouping on edge		Irregular	Si, Na, O, C (Mount 1 62515(3))
9x6	On edge	Al-oxide	Irregular	Si, Al, minor O, Cu, P, S (Mount 1 62515(4))

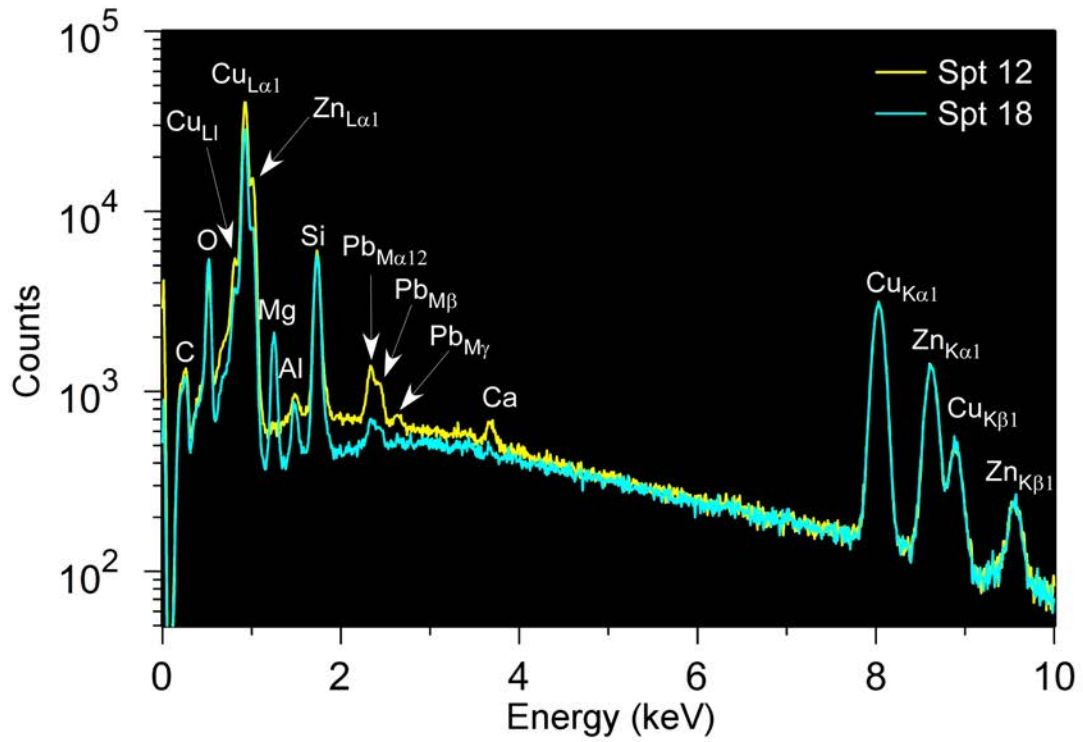
further study (pink), other (no color)

3x2		Stainless	Irregular	Fe, Cr, Al, O, C (Mount 1 62515(11))
28x7		Brass	Irregular	Zn, Cu, Pb , O, C, Al, Ca (Mount 1 62515(12)) see pic:
7x19			Irregular	Al, minor O (no spt)
17x8			Irregular	Al, minor O (no spt)
15x17		Biogenic	Irregular	C, N, O, Na, Cl, S, K (Mount 1 62515(13))
6x2		Stainless	Irregular	Fe, Cr, very minor O (Mount 1 62515(14))
10x18			Irregular	Al, O (no spt)
5x5; 7x9; 17x4; 20x2; 10x2; 12x2	Group; stringers		Irregular	Fe, Cr (no spt) also see image (25)
32x21		Biogenic	Irregular	C, N, O, Na, S, Cl, K (Mount 1 62515(10))

- Acicular** needle-shaped
- Angular** sharp-edged or having roughly polyhedral shape
- Crystalline** freely developed in a fluid medium of geometric shape
- Dendritic** having a branched crystalline shape
- Fibrous** regularly or irregularly thread-like
- Flaky** plate-like
- Granular** having approximately an equi-dimensional irregular shape
- Irregular** lacking any symmetry
- Modular** having rounded, irregular shape

4x7			Irregular	Fe, Cr (no spt)
46x7			Irregular	Al, O (no spt)
14x20		Biogenic	Irregular	C, N, O, S, Ca, Na, K (Mount 1 62515(15))
9x7			Irregular	Al, O (no spt)
9x10			Irregular	Fe, C, O, Ca, S, Na, Al, Mn (Mount 1 62515(16))
14x6		Al metal, oxidation on surface	Irregular	Al, minor O (Mount 1 62515(17))
16x21		Brass	Irregular	Cu, Zn, C, Mg, Al, Pb , O (Mount 1 62515(18)) See image (18)
7x10			Irregular	Si, Al no O (no spt)
106x72		Largest particle on surface. C-rich with encrusted salts/minerals. Lamellae-like structure.	Irregular	C, N, O, Na, Cl, K , S, Mg, Al (Mount 1 62515(19)) (see images (19& 19A)) also P&S (19)(1) points 1-7 on this sample
13x12		Al metal	Irregular	Al, Fe, hint O (Mount 1 62515(20)) (see image (20))
3x4		Al-oxide	Irregular	Al, O, C, P, S (Mount 1 62515(21))
22x24			Modular	C, O, K , Ca, trace Cl, Mg (Mount 1 62515(22)) See image (22)
17x11			Irregular	Al, hint O (no spt)
20x20			Irregular	C, O, K , Ca
25x22			Irregular	C, Cl, hint O, Na, S (Mount 1 62515(23))
10x11			Irregular	Al (no spt)
20x10		Biogenic	Irregular	C, N, O, S, Cl, K , Ca (Mount 1 62515(24))

4x5			Irregular	Fe, Cr (no spt)
8x10			Irregular	Al, hint O (no spt)
12x10		Mineral?	Irregular	Si, O (Qtz?) (no spt)
7x10			Irregular	Al (no spt)
4x5; 2x3; 2x3; 2x3; 2x3; 1x2 (several)	grouping			Fe, Cr, hint Al no spt
~ 10 in longest direction for each				C (smudges) approximately 5 associated with stringers ; see image (25)
3x5		Stainless	Irregular	Fe, Cr, Ni, minor O (Mount 1 62515(25)); see image (25)
4x4; 3x6			Irregular	Fe, Cr (no spt)
10x4 largest	grouping		Irregular	Fe, Cr Stringers (no spt)



Unless otherwise stated,
peaks without designation are 'K' emission lines

Figure 2: Energy dispersive X-ray spectra of two brass particles.

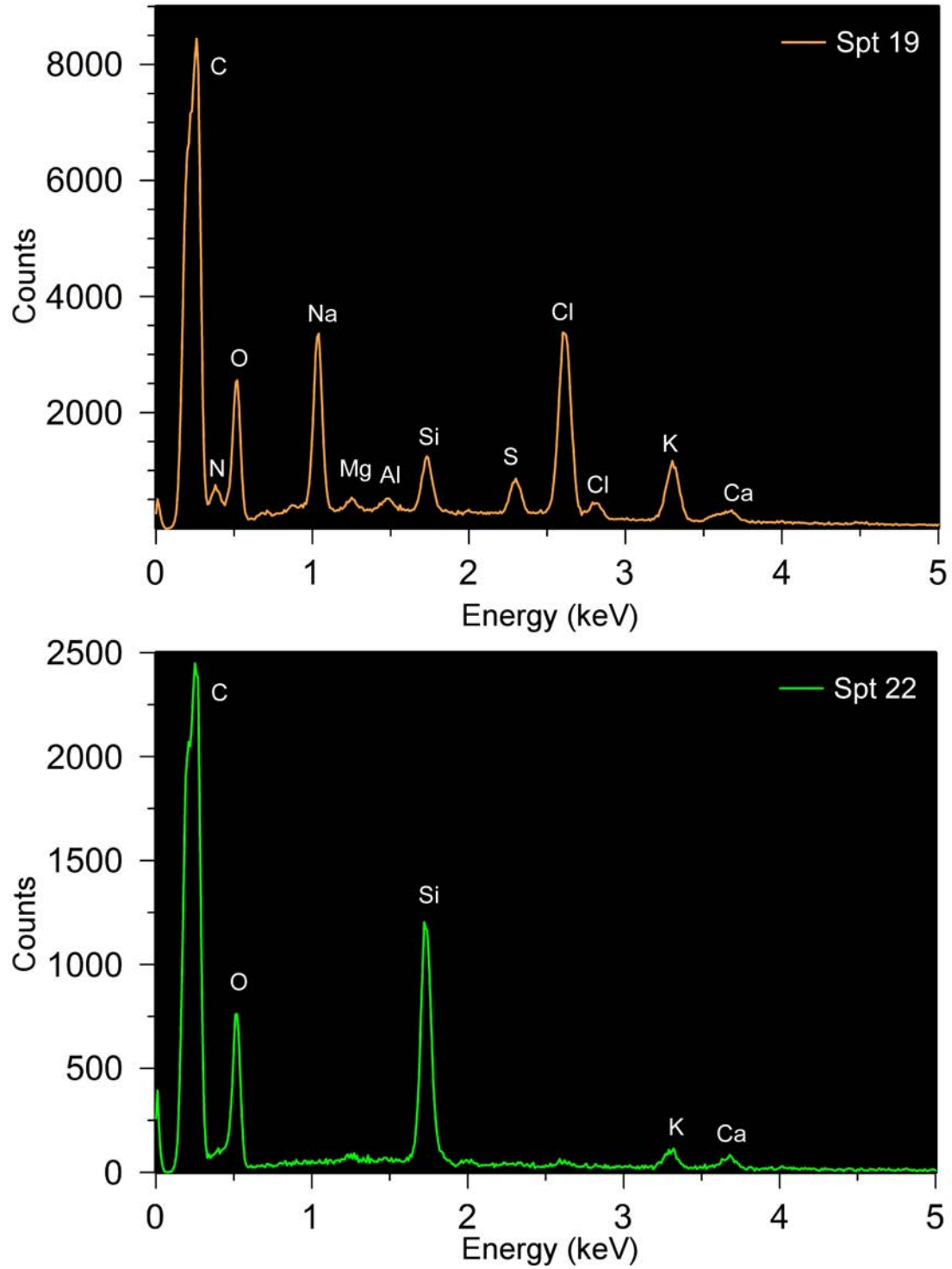


Figure 3: EDX spectra of a large, complex carbonaceous grain (top) and an unusual K-bearing grain of unknown origin (bottom).