



Contamination Knowledge Report:

Diffuse Black Kapton

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Summary

Diffuse black Kapton (DBK) is a candidate material for wrapping the TAGSAM arm to reduce light reflection and improve imaging of the TAGSAM head. Samples of DBK were examined to assess the potential science impact of particle shedding from the DBK during TAGSAM arm movements.

Particles are easily shed from diffuse black Kapton (DBK) during handling through either abrasion or mechanical flexure. Shed particles range from sub-mm to sub-micron with a complex morphology composed of nanometer-to-micron sized aggregates of nitrogen and oxygen-rich carbonaceous matter and mineral grains composed of silica (SiO_2) and rarely alumina (Al_2O_3). These particles share chemical and physical properties associated with primitive meteoritic materials. Many of the principal mass peaks identified by μ -L²MS from DBK are associated with *O* (e.g., carbonyl $C=O$) and *N* (e.g., nitrile $C\equiv N$) containing organics. The distribution of organic peaks overlay the typical range of masses found in the solvent extractable organics from carbonaceous chondrites.

Recommendation

Given the propensity of DBK to shed particles containing abundant and compositionally diverse organic matter of meteoritic interest, we strongly recommend avoiding use of this material in any location likely to allow for sample contamination.

Procedures

Sample Preparation

We received a ~ 15 cm² piece of DBK film and a particle fallout silicon wafer for analysis. It was not necessary to examine the silicon wafer because plenty of particles were shed from the DBK film during shipping for our analysis. For optical microscopy and μ -L²MS, samples of DBK were prepared by cutting small squares of the DBK film ($\sim 3 \times 3$ mm), using either a stainless steel surgical scalpel or SS scissors, and attaching to a SS sample mount using a small piece of double-sided 3M™ Scotch tape. Separate samples of the DBK film were used for analysis of both the top and bottom surfaces. The

'top' side designating the darker corrugated matte surface and the 'bottom' side designating the lighter coloured smooth, partially reflective surface.

For FESEM and EDX analysis similarly sized patches of the DBK film were attached to a ½-inch diameter *Al* sample stub using a Pelco™ double-sided conductive *C* adhesive tab. Since the DBK film is itself electrically conductive no sample coating was necessary. In addition to both top and bottom surfaces a thin cross-section of the DBK film was prepared using a SS surgical scalpel as well a selection of small black particulates that were found adhering to the interior surface of the polyethylene pouch in which the DBK film was shipped.

Optical & Fluorescence Microscopy: Optical and fluorescence imaging were performed using an Olympus BX60 microscope using a 50× (1.4 N.A.) objective. Extended depth-of-field images and surface topography were determination from image stacks collected over a range of z-focus planes. For fluorescence images a narrow pass 490 nm excitation filter was used in combination with a 520 nm long pass emission filter.

Microprobe Two-Step Laser Mass Spectrometry (μ -L²MS): Organic mass spectrometry was performed using a two-step laser desorption laser ionization time-of-flight mass spectrometer. Laser desorption was achieved using a spatial-filtered pulsed CO₂ laser (λ 10.6 μ m) equipped with a plasma shutter to minimize sample surface damage. For ionization a pulsed 118 nm vacuum ultraviolet (VUV) laser source was used through generation of the 9th harmonic of a Nd:YAG (λ 1.064 μ m) laser.

Field Emission Scanning Electron Microscopy (FESEM) & Energy Dispersive X-ray Spectroscopy (EDX): Scanning electron microscopy imaging was performed using a field-emission JEOL 7600F microscopy operating a 15 keV, with concurrent acquisition using low-angle backscatter (LBE) and secondary electron (SEI) detectors. Elemental composition of selected regions of interest were determined by energy dispersive X-ray spectroscopy using a *Si(Li)* drift detector.

Observations

Optical & Fluorescence Microscopy: Optical microscopy showed the DBK film to be dimorphic. The bottom side appears smooth and contiguous while top side has a chaotic dimpled surface topography composed of pseudo-hemispherical mounds ranging in size from sub-micron to over 100 μ m with the entire surface criss-crossed by numerous cracks. The presence of occasional shallow circular depressions in the top surface was attributed to delamination of surface mounds either through abrasion or flexing of the DBK film. This was supported by observation of fine black particulate matter adhering to the interior surface of the polyethylene pouch in which the sample shipped. Under UV-blue illumination both top and bottom surfaces show strong and distinctive native fluorescence signatures. The bottom surface was dominated by a diffuse orange fluorescence consistent with that observed from other Kapton™ polyimide films. In

contrast the top side show a strong green heterogeneous fluorescence associated with lighter colored micron and sub-micron sized angular particulates (Fig 1.).

μ -L²MS: On introduction into the μ -L²MS vacuum chamber relatively little outgassing was observed from the DBK sample (Fig. 2). The outgassing spectrum was composed primarily of a mixture saturated and unsaturated hydrocarbons. For example, peaks at 44, 58, 72 & 86 m/z are assigned to the C₃ – C₆ alkanes (C_nH_{2N+2}; n = 3, 4 ... 6) propane, butane, pentane and hexane while peaks at 78, 178 are assigned to the aromatics benzene (C₆H₆) and phenanthrene (C₁₄H₁₀). At higher masses (> 200 m/z) there was a broad distribution of waxy/paraffinic like hydrocarbons. In under an hour under high vacuum the degassing reduced sufficiently to allow two-step analysis.

Spectra were acquired from both the top (Fig. 3) and bottom (Fig. 4) surfaces respectively by summation of 100 individual point spectra (~7 μ m diameter) acquired at random positions on the sample surface so as to provide an overall characterization of all desorbable species. On the bottom surface the shot-to-shot variability of spectra was small. This was not, however, true of the top surface which showed considerable shot-to-shot variability in terms both of relative abundance and composition. Some but not all of this apparent heterogeneity may be a result of optically dark nature of the top surface combined with its topographic variability which made it difficult to accurately maintain the sample surface at the CO₂ laser beam focus. Comparison of the top and bottom surface spectra (Fig. 5) indicates that, while sharing many of the same peaks they are compositionally different. Because of the particulate contamination concern associated with the top surface this was the primary focus of spectral analysis. It is immediately evident many of the principal mass peaks are associated with O (e.g., carbonyl C=O) and N (e.g., nitrile C≡N) containing organics. For example the peak sequence at 59, 117 & 175 m/z represents radical fragment species formed by successive loss of an enolate ion (CH₃CO₂⁺), while the peak sequence at 198, 224, 250 m/z represent the successive loss of a nitrile ion (CN⁺). Overall the distribution of organic peaks would overlay the typical range of masses found in the solvent extractable organics from a carbonaceous chondrite.

FESEM & EDX: The cross sectional structure of the DBK film appears to be composed of a Kapton backbone laminated with organic-rich coatings on both the bottom and top surfaces (Fig. 6). The bottom coating is composed of a smooth amorphous organic material in which is embedded nanometer sized particulates. In contrast the upper surface is structurally inhomogeneous composed of rounded and angular particulates ranging in size from sub-micron up to several tens of microns enveloped in a very fine nanometer-scale fractal, wispy carbonaceous coating. At lower magnification the presence of numerous cracks and fissures are apparent in the surface suggestive of coating fracture from flexing or abrasion. Figure 7 shows several particles that were shed from the top surface of the DBK film. Compositional analysis by EDX indicates the DBK to be composed of one or more organic polymers containing O, N and possibly S functional groups. Interspersed with in this are nanometer-to-micron sized mineral grains composed primarily of silica (SiO₂) and rarely alumina (Al₂O₃) along with micron-sized carbon grains.

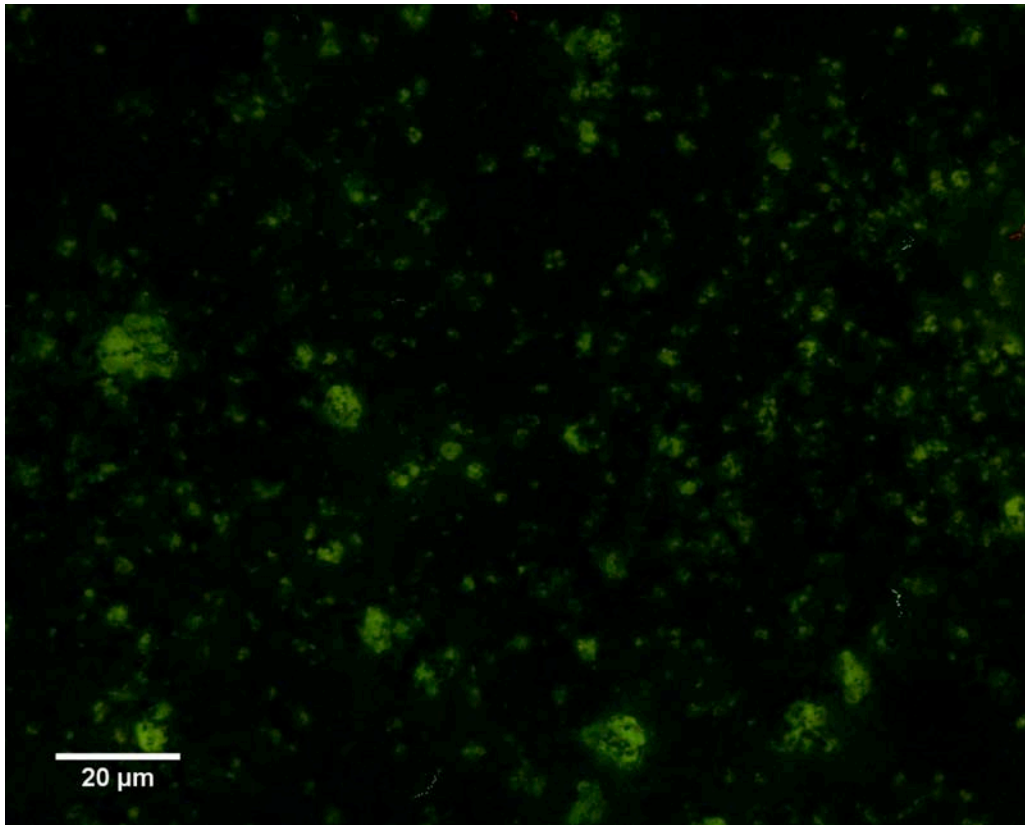
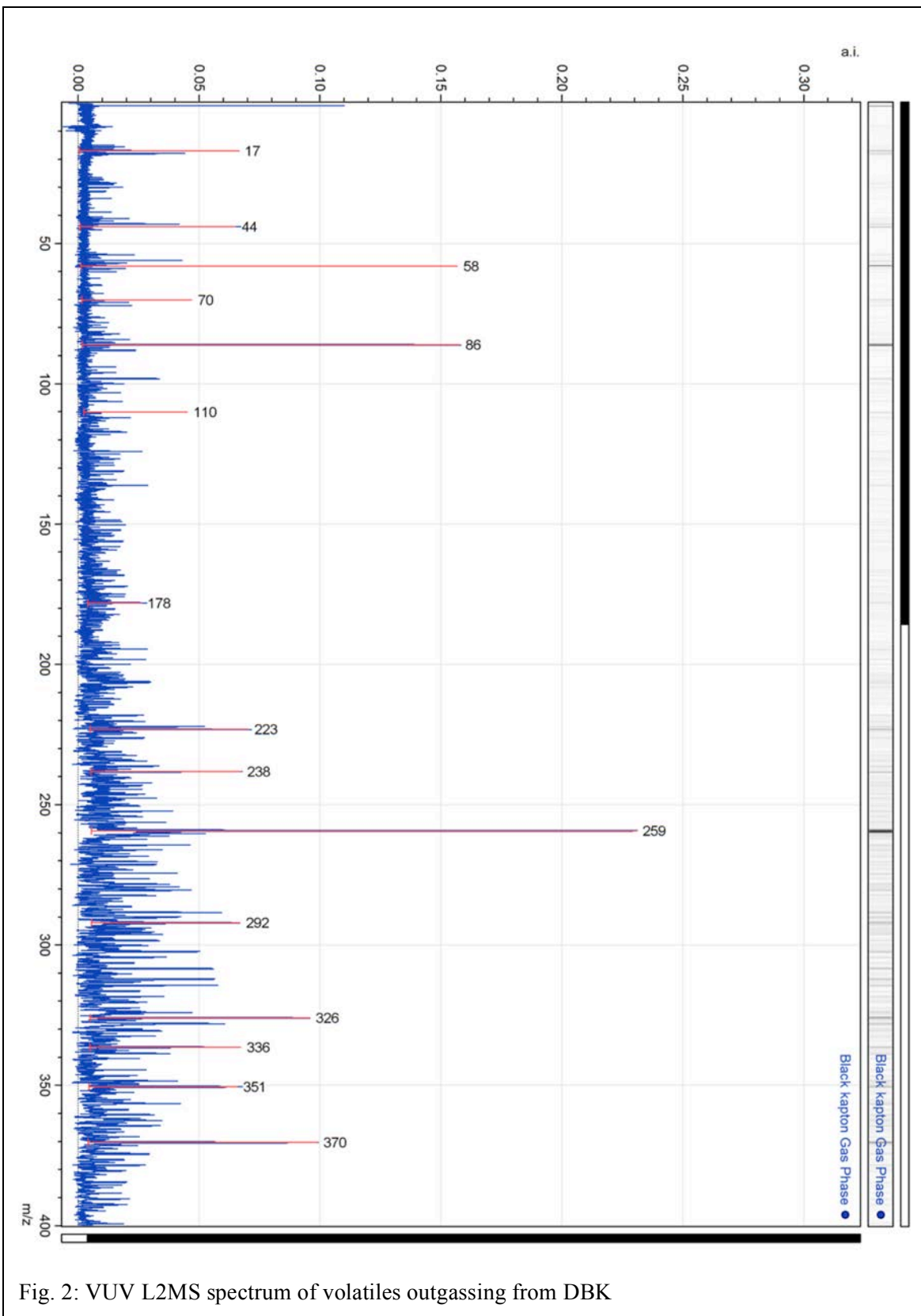


Fig. 1: Native UV fluorescence image obtained from the top surface of the diffuse black Kapton.



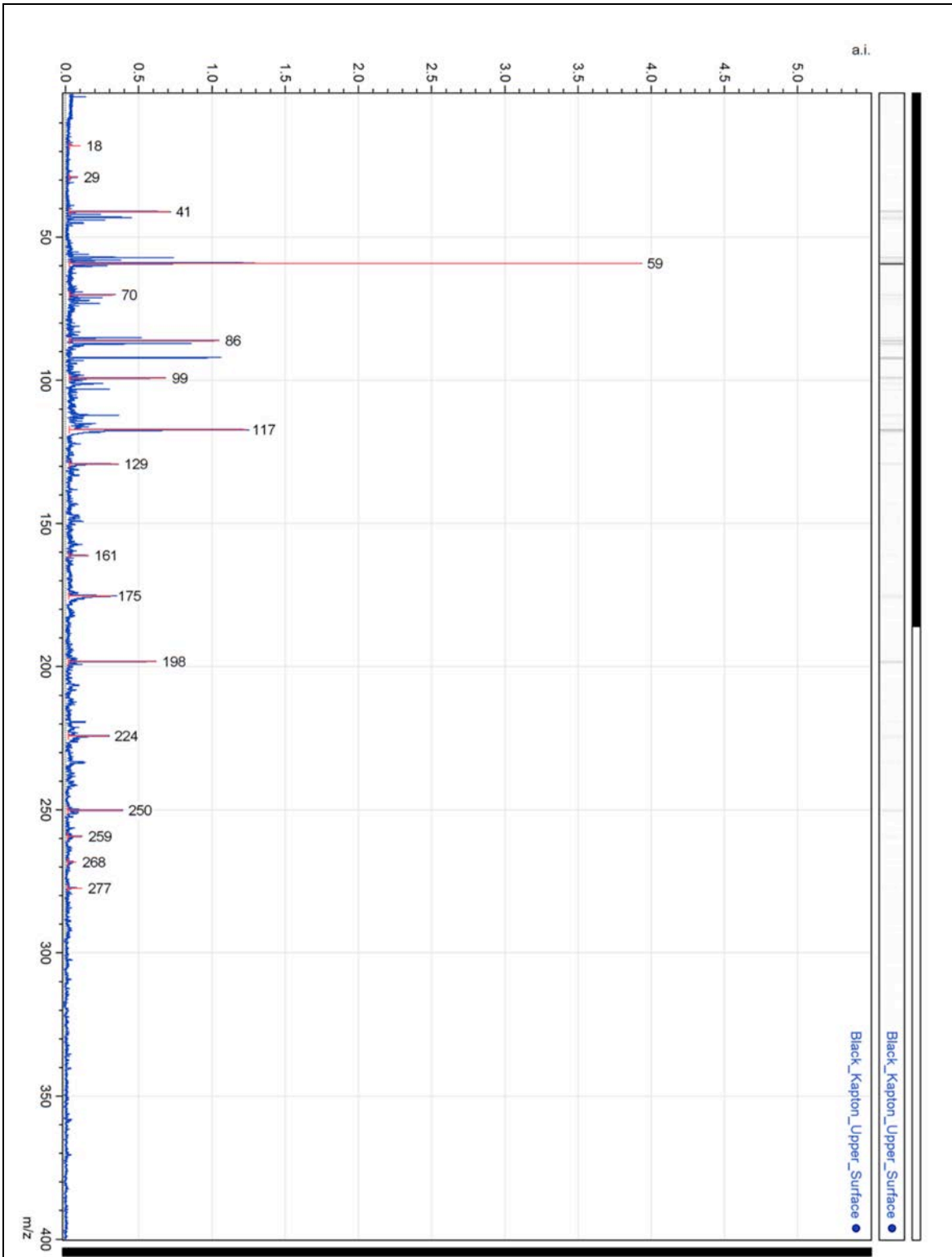


Fig. 3: VUV L2MS spectrum of the DBK top surface

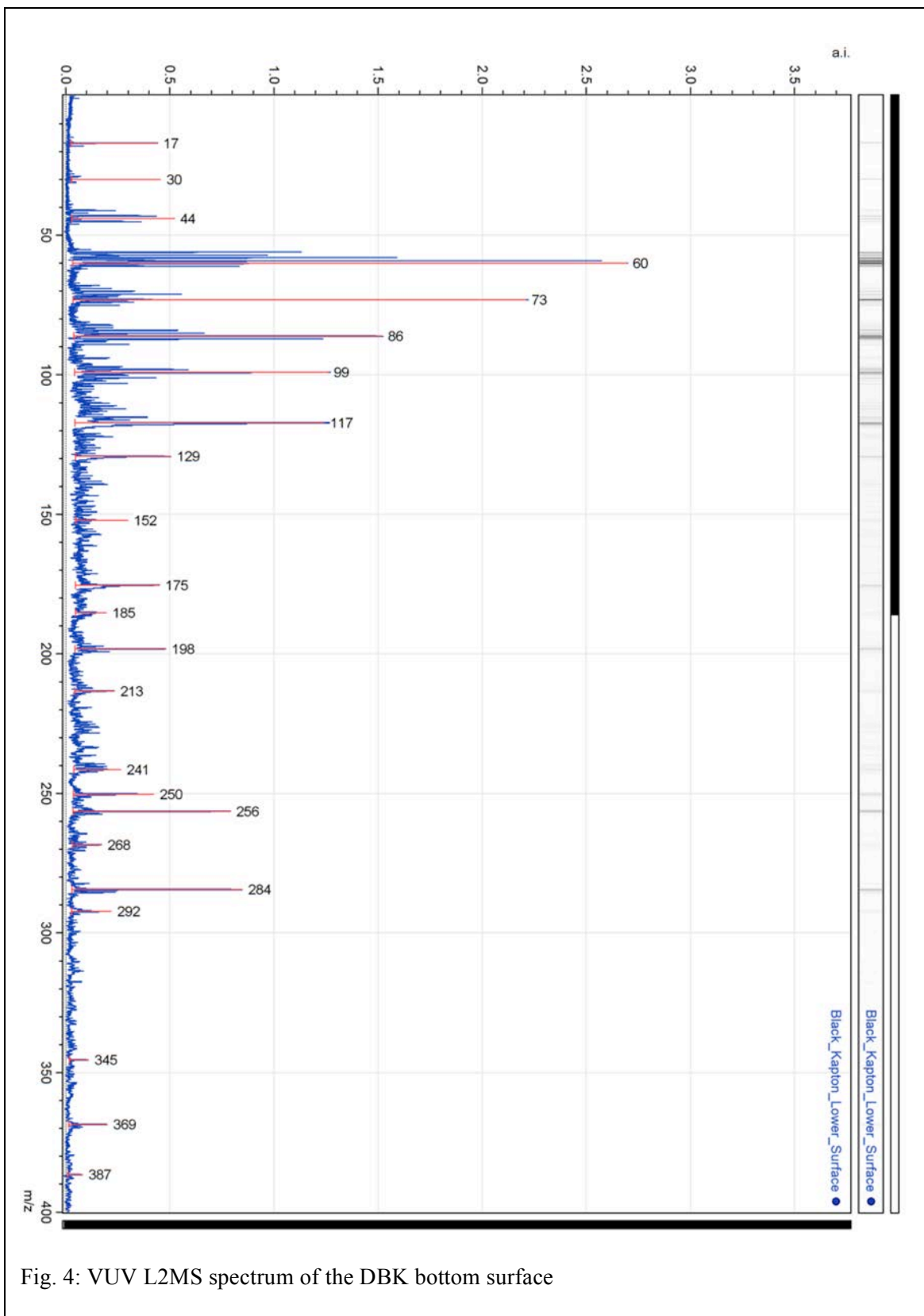


Fig. 4: VUV L2MS spectrum of the DBK bottom surface

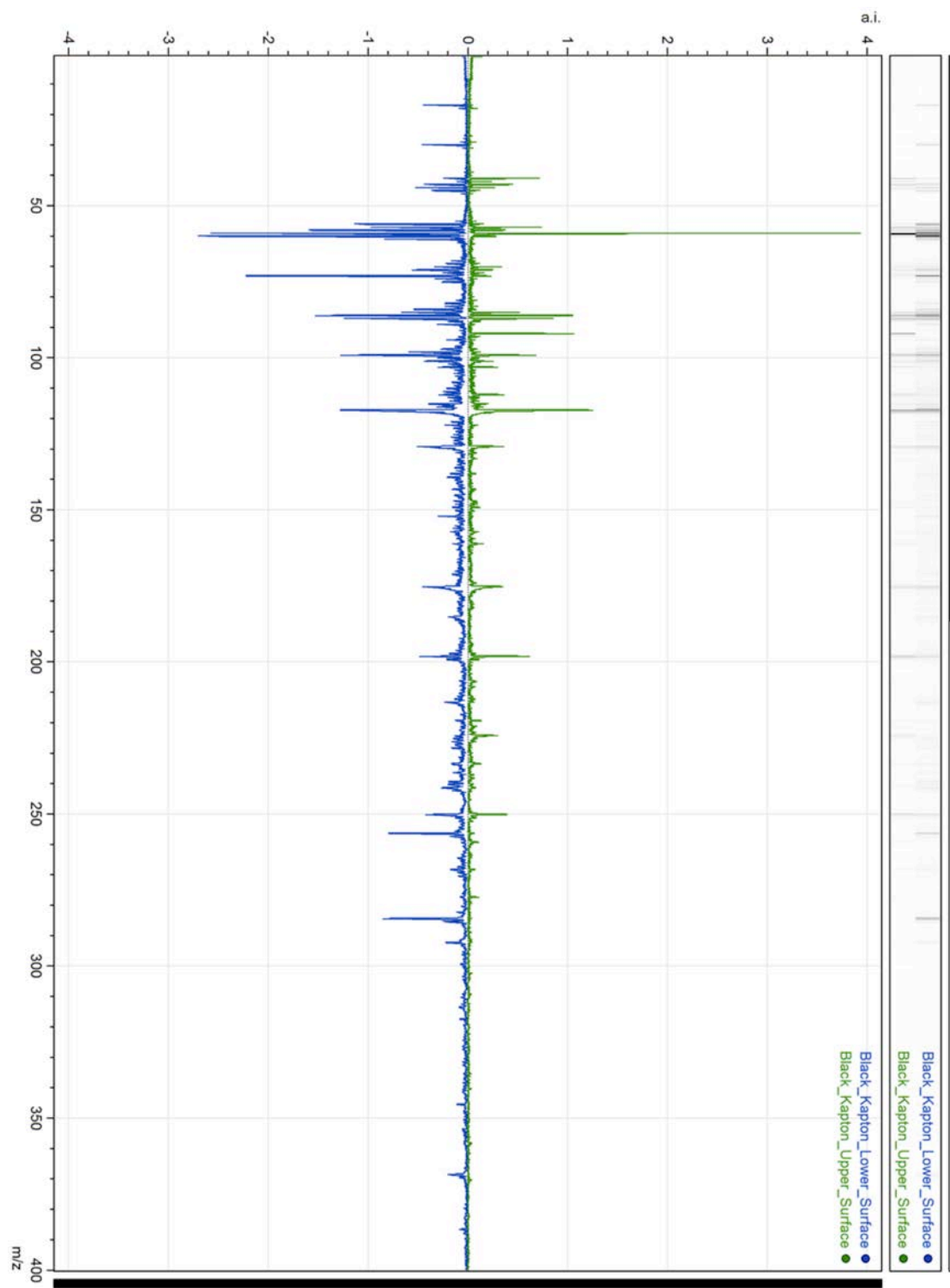


Fig. 5: Comparison of VUV L2MS spectra of the DBK top and bottom surfaces

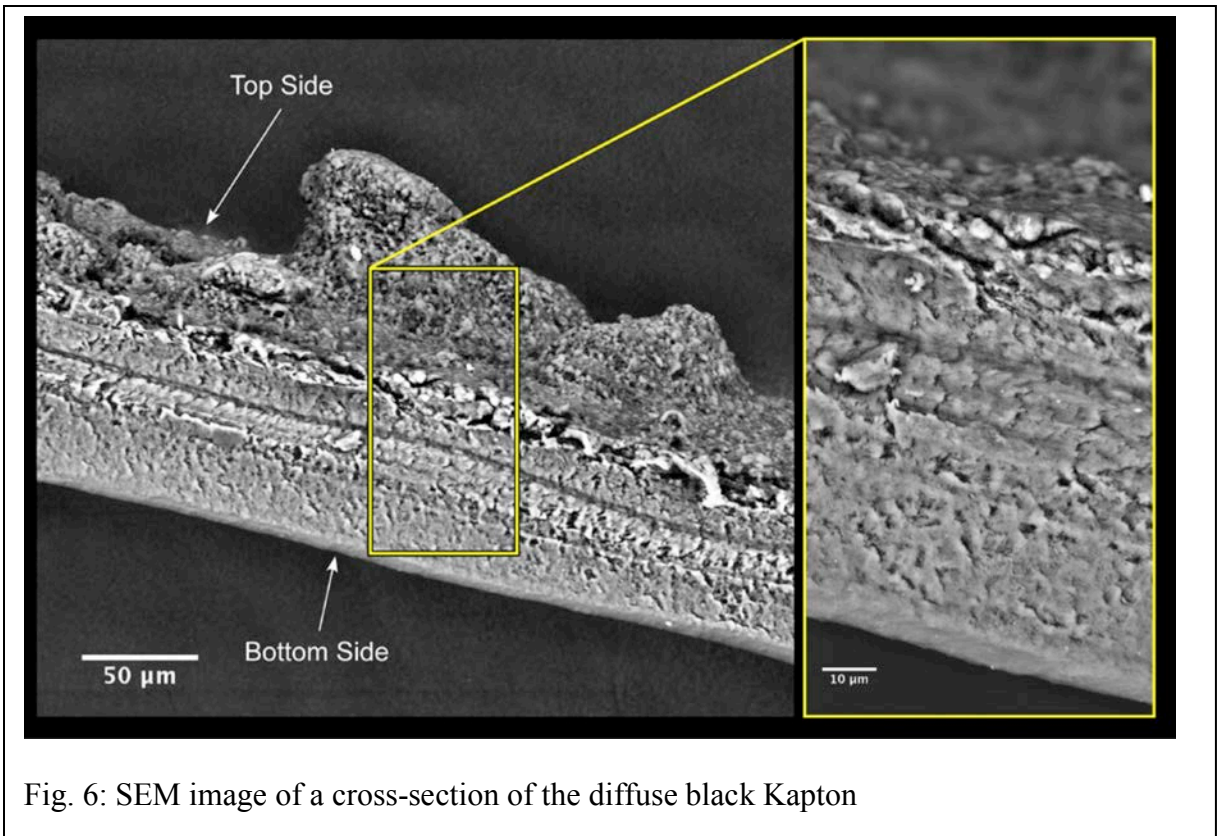


Fig. 6: SEM image of a cross-section of the diffuse black Kapton

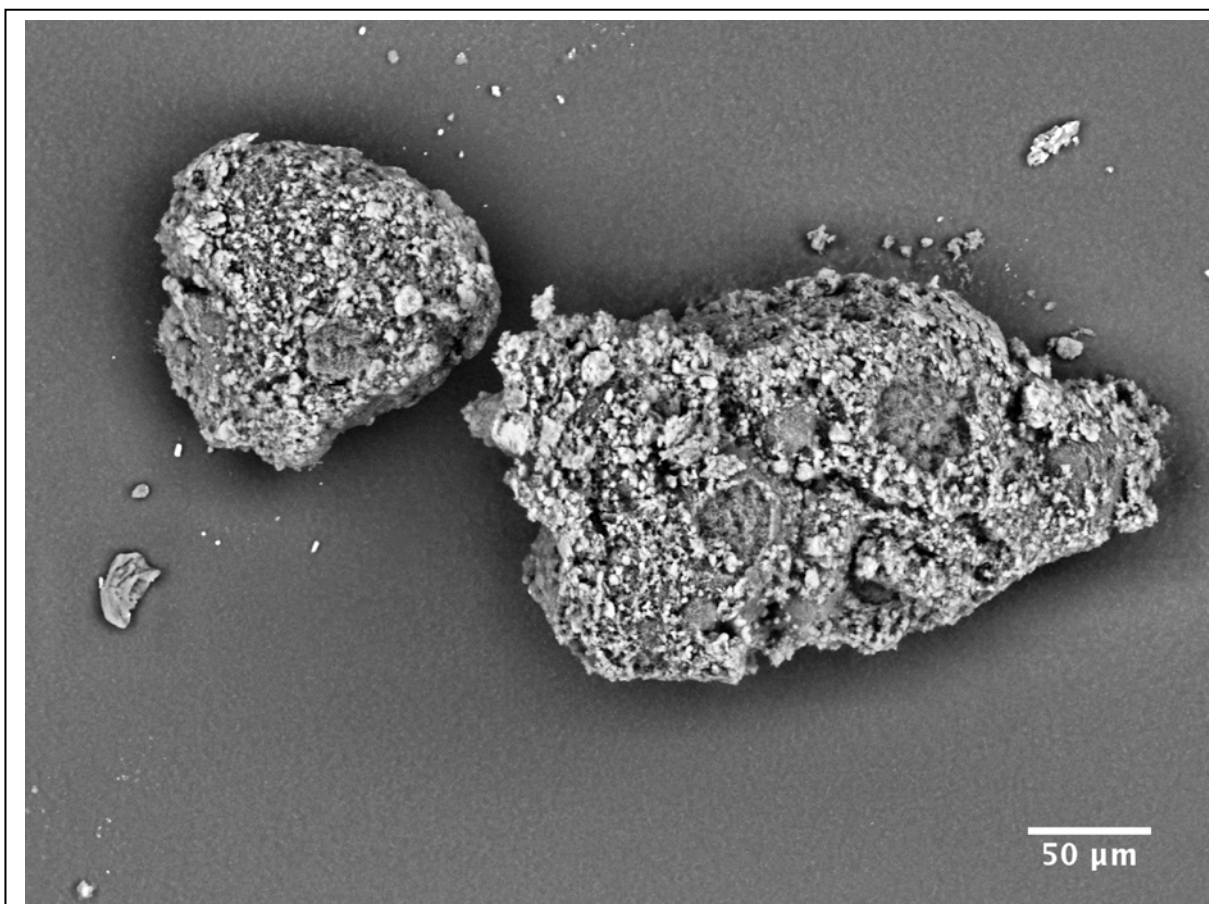


Fig. 7: SEM image of particles shed from the diffuse black Kapton