


Object Number	K546	Description	Helmet-crest channel, cast in silver and gilded, with animal ornament and animal-head terminal. Catalogue no. 589.
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	Sample Description and location.
	Sample K546-1 collected from dark material indicated in images below

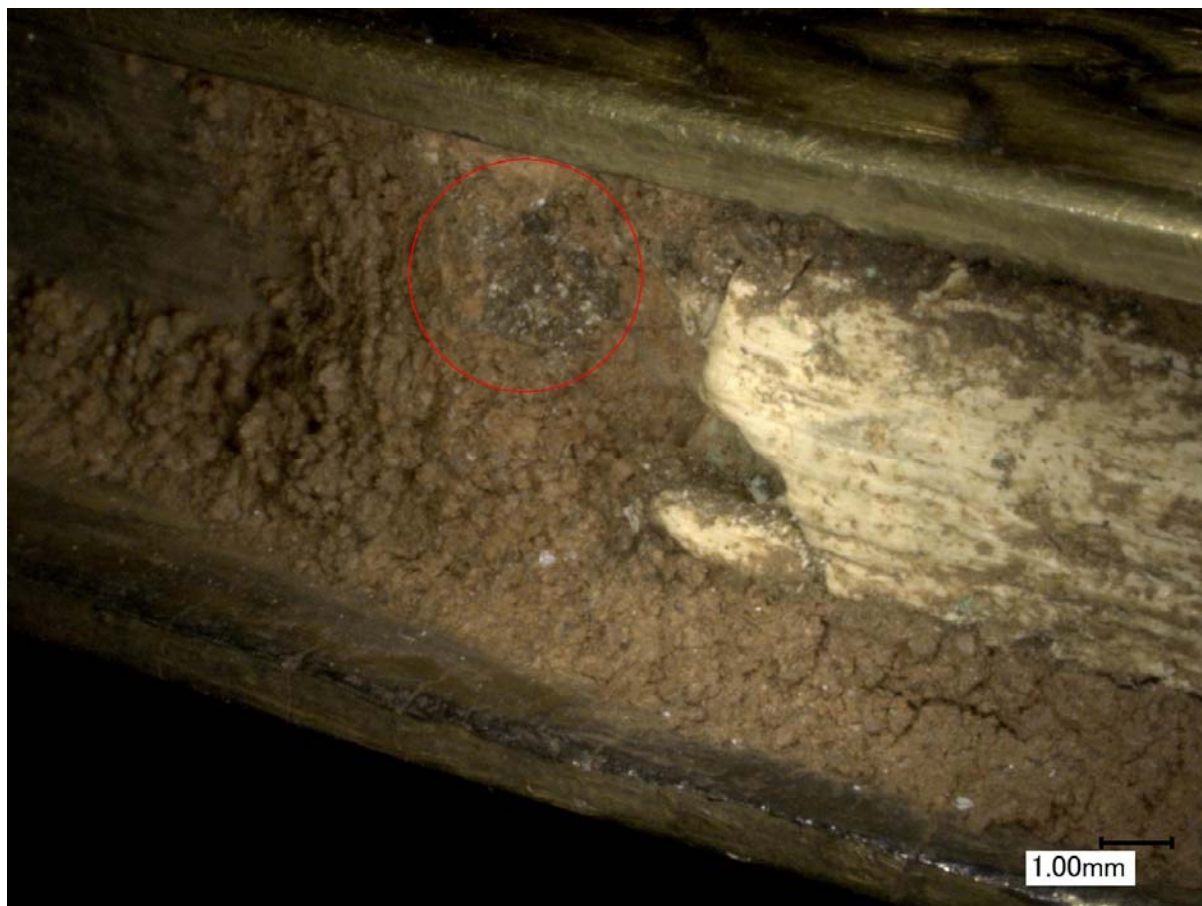


Figure 1. Sample K546-1 collected from dark material circled in image above.



Figure 2. Detail of sample K546-1 collection area (circled).

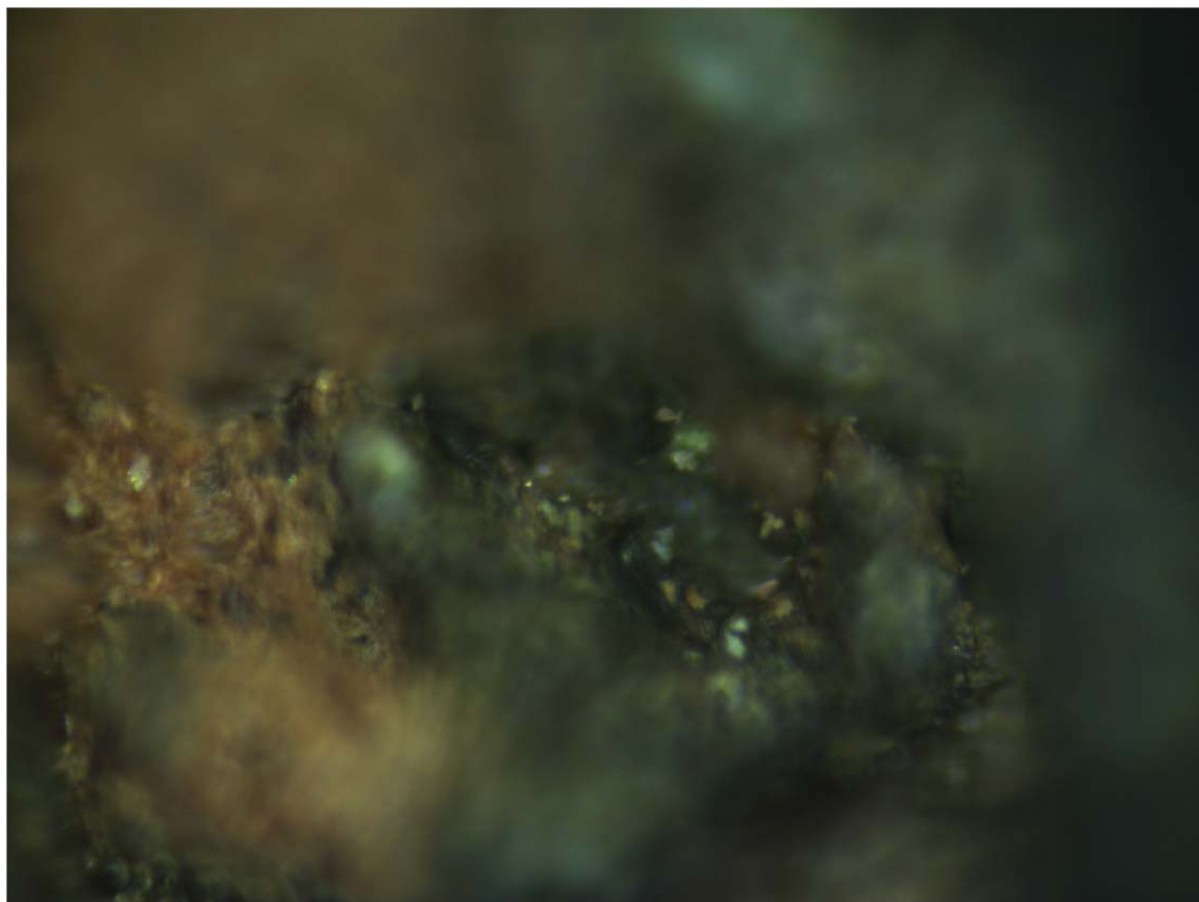


Figure 3. Detail showing sub-sample K546-1-1 FTIR analysis area.

FTIR Analysis

Comments: Spectrum K546-1-1 (top, pink) appears to be a mixture of a keratin based animal glue such as hoof glue and trace beeswax. The FTIR spectra for keratinous materials exhibit characteristic bands for amide I (C=O stretching) between 1700 and 1600 cm^{-1} , and amide II (CN stretching and NH bending) between 1560 and 1500 cm^{-1} , and a broad band centred at around 3300 cm^{-1} related to N-H stretching. A doublet between 3000 and 2800 cm^{-1} relating to C-H stretching of methylene groups are characteristic of keratinous proteinaceous materials. (Welsch et al. 2012, Kennedy et al. 2013, Mansilla et al. 2011, Derrick et al. 1999). In oxidised keratinous materials such as the oxidised horse hair shown in Fig.4, an intense peak at about 1030 cm^{-1} is attributable to S=O bonding in cysteic acid. (Welsch et al. 2012, Kennedy et al 2013, Mansilla et al. 2011). This peak should be interpreted with some caution in this sample however as silicate based material such as is found in earth minerals also exhibit a sharp band at approximately 1000 cm^{-1} which relates to Si-O bonding. The peak at 1030 cm^{-1} may derive from cysteic acid in oxidised keratinous tissue, silicate based earth minerals deliberately added to the paste or from the burial environment, or a combination of both.

The FTIR spectrum for beeswax is characterized by dominant absorption bands around 2950 cm^{-1} and 2850 cm^{-1} that relate to C-H stretching of the methylene (CH_2) groups, a band at 1740 cm^{-1} that relates to C=O bonding characteristic of the ester groups, a band around 1460 cm^{-1} relates to C-H bending, and a doublet between 720 and 730 cm^{-1} relating to non planar skeletal deformation vibrations of long chain hydrocarbons (Derrick *et al* 1999, Birshtein and Tul'chinskii 1977). The bands in the region 1350 - 1180 cm^{-1} may be assigned to a phenomenon known as a 'band progression' present in fatty acids and fatty acid esters which result in a series of evenly spaced bands in this region. These are due to wagging and twisting vibrations of successive carboxyl coupled methylene groups (Baeten et al. 2010) shown in more detail in Fig. 5.

Representative Spectrum

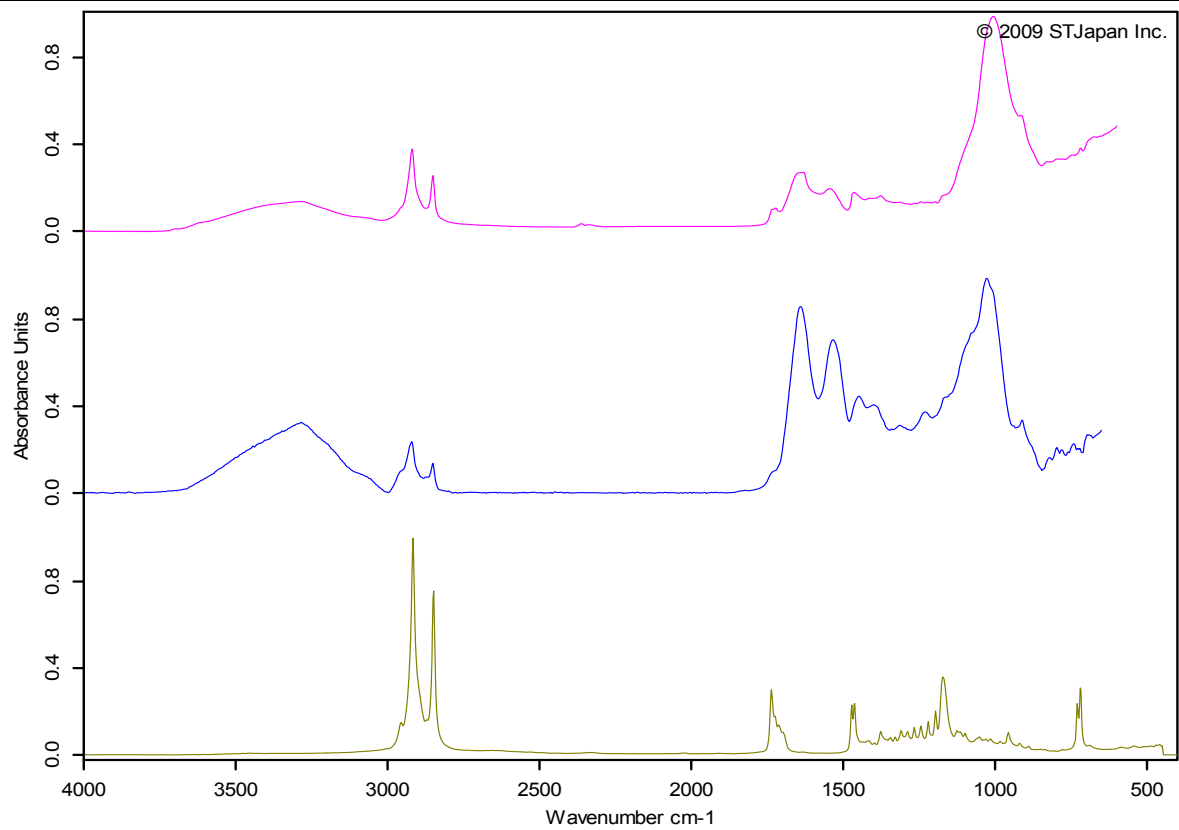


Figure 4: Top (red) K545-1-1. Middle (blue) Oxidised horse hair reference sample, ST Japan 2009. Bottom (green) Beeswax reference spectrum, ST Japan 2009.

Representative Spectrum

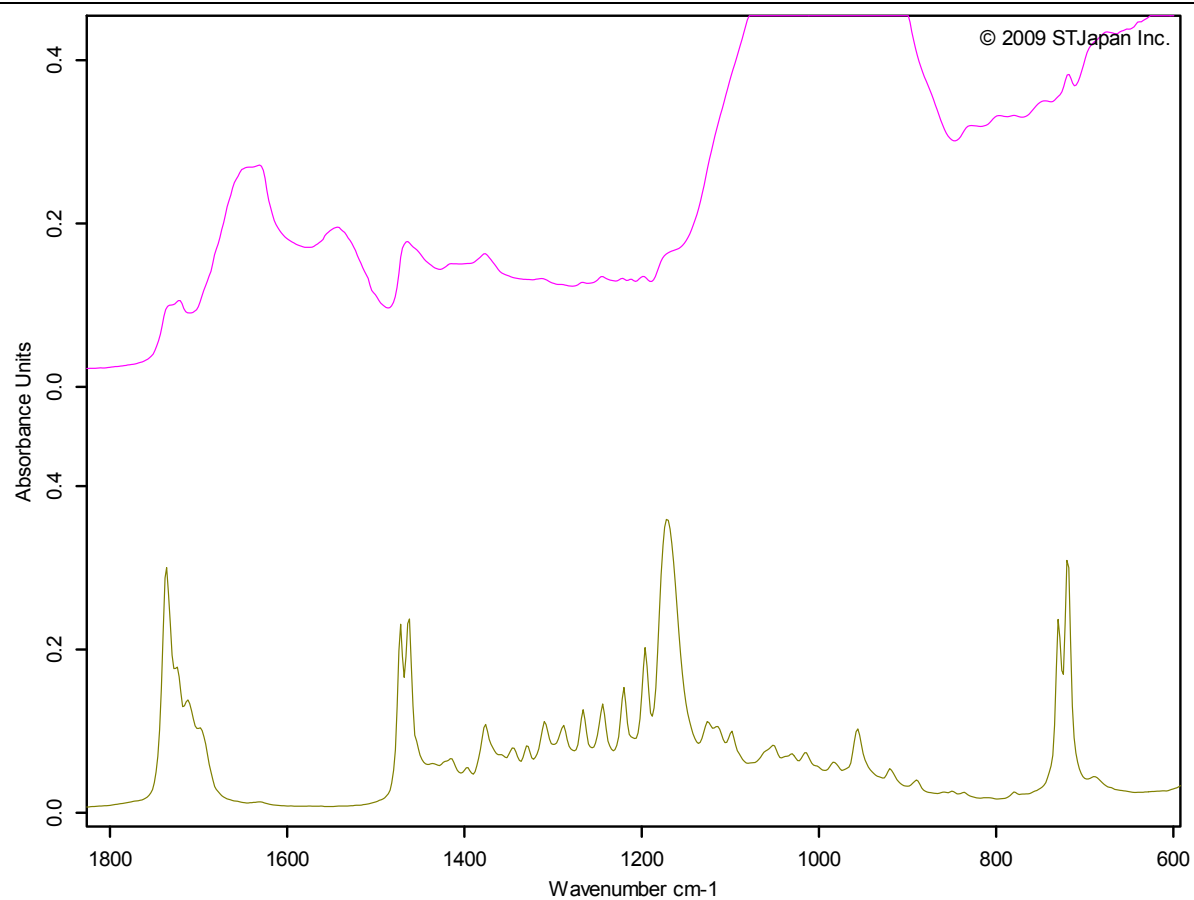


Figure 5: Detail of 600-1800 cm⁻¹ region for FTIR spectra top (pink) K546-1-1, and bottom (green) Beeswax reference spectrum, ST Japan 2009, showing subtle confirmative peaks for beeswax.

SEM Analysis

SEM analysis was carried out on K546-2 to examine the microstructure of the material and to elementally characterize the particulate distributed across the sample surface. The white specks visible in the BSE image at figure 9 map closely for silver, and while chlorine is not present in the mapping, in other samples, silver specks like this frequently relate to silver chlorides. The silver is unlikely to be silver dust. The sample has an overall coating of particulate formed from aluminium, silicon, and oxygen which likely relates to alumina silicates commonly found in earth minerals. These likely derive from the burial environment.

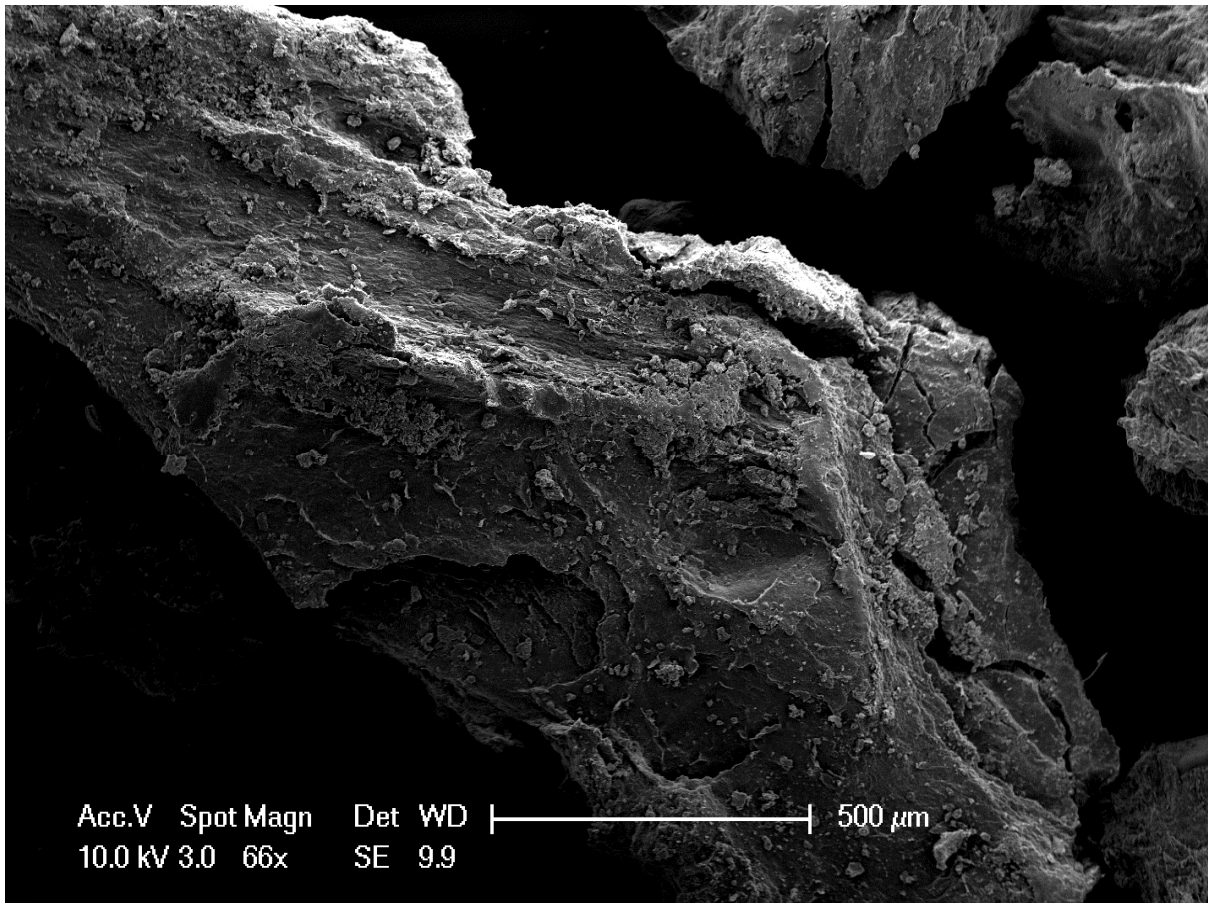


Figure 6. Secondary electron image of K546-2 showing texture of black material collected from helmet crest channel. Material appears to have a texture consistent with beeswax, and has a surface distribution of particulate overall.

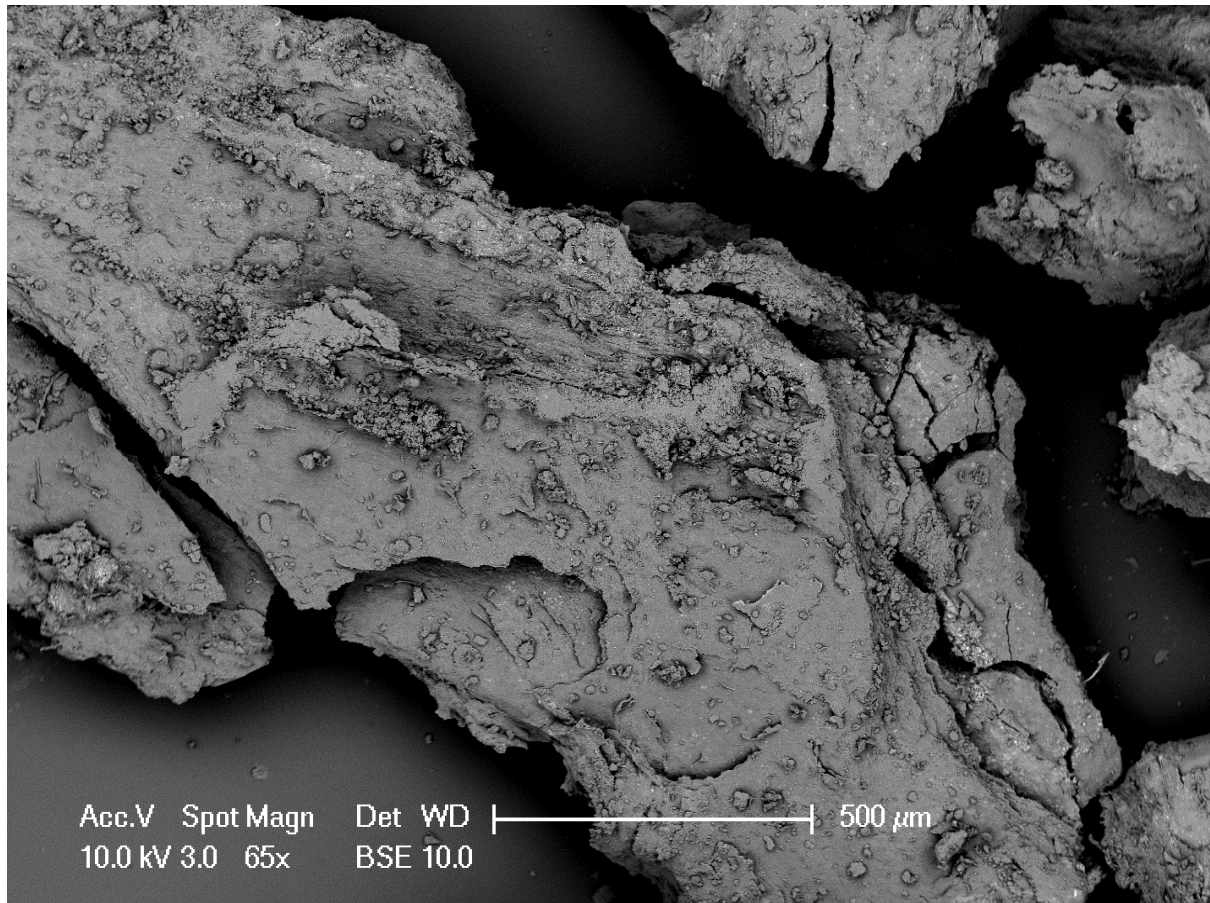


Figure 7. Back scatter electron image of K546-2. The material appears elementally homogeneous.

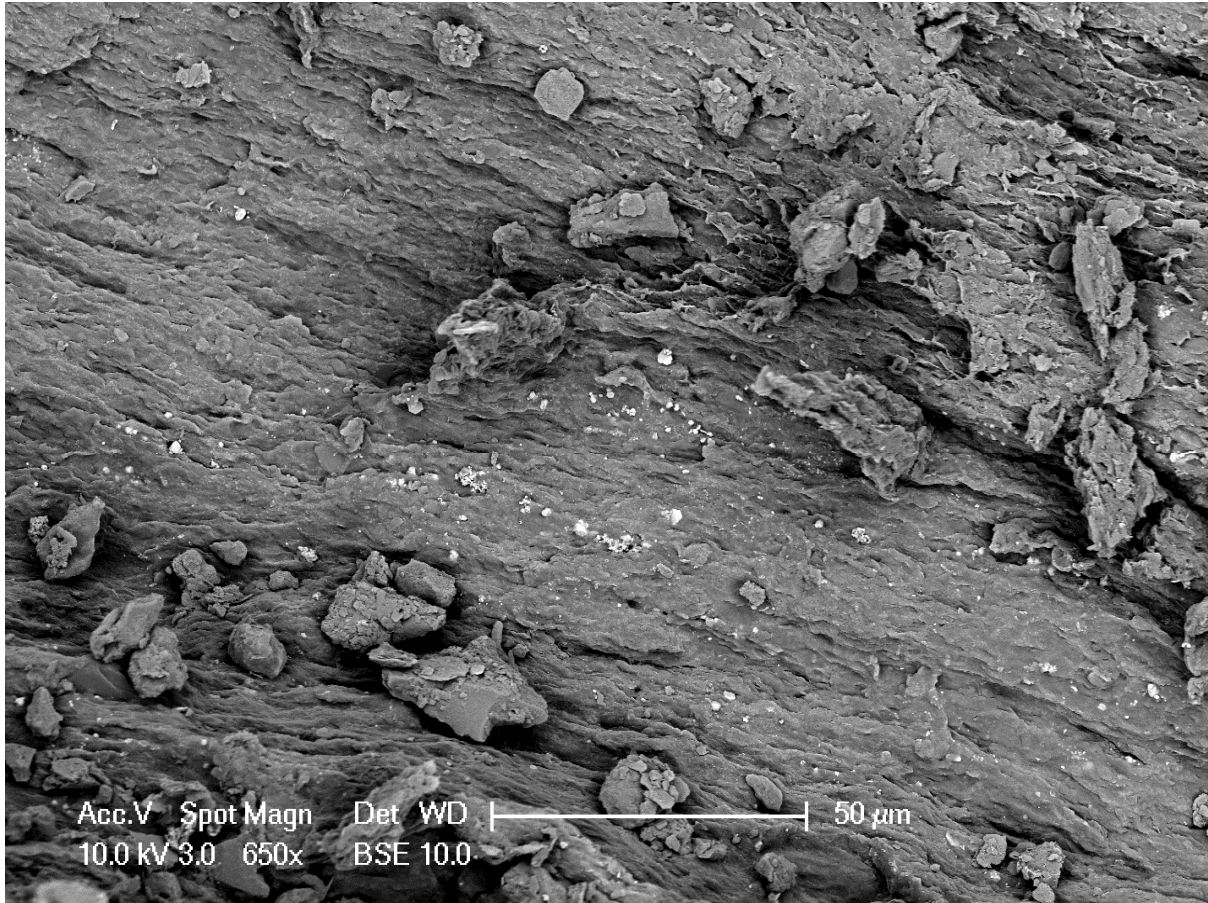


Figure 8. Back scatter electron image showing distribution of elementally different white specks across surface of K546-2.

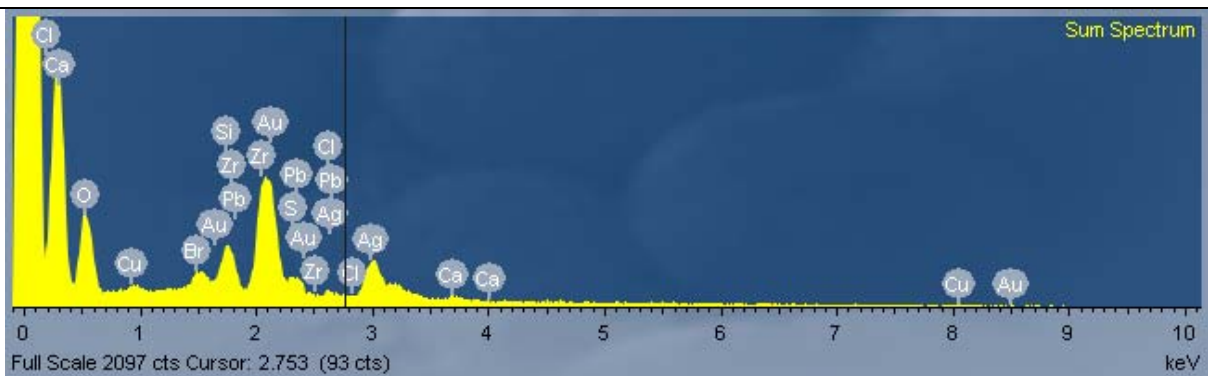
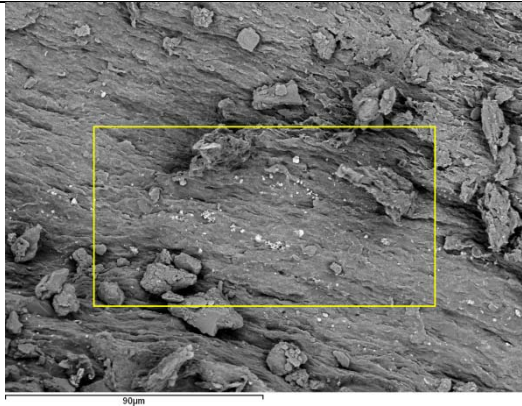
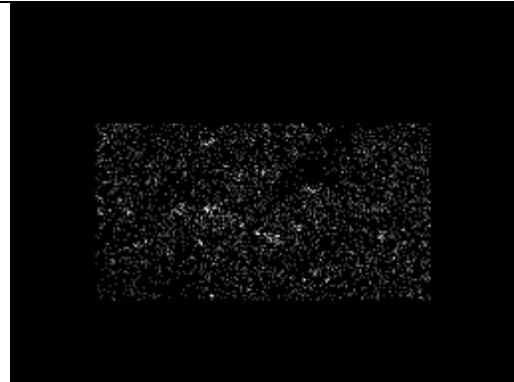


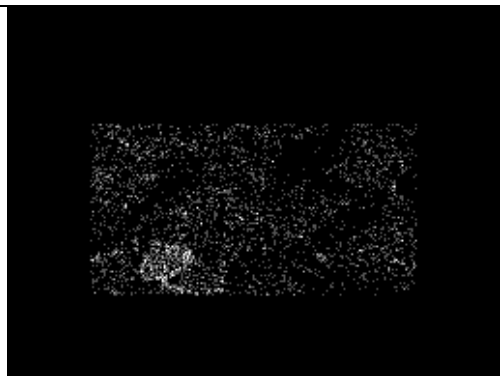
Figure 9. EDS sum spectrum for K546-2.



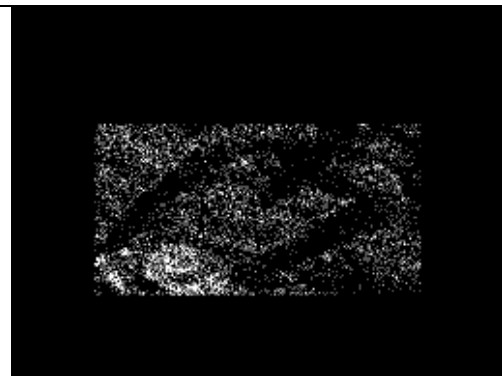
K546-2 SOI1 Electron Image



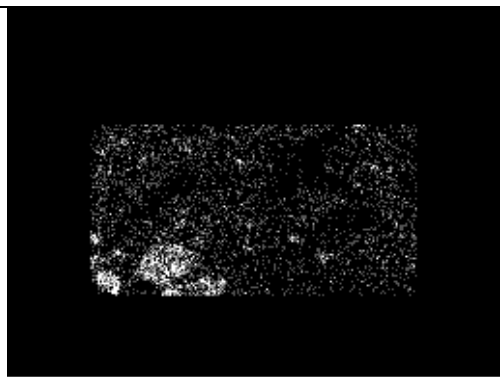
Ag La1



Al Ka1



O Ka1



Si Ka1