# Metal Component Thermal Blackening Process Failure Solved by SIMS

## Production Process Problem

Following a thermal blackening process designed to create the correct thermal emissivity properties, some components within a batch were found still to be silver. They could be separated out from the 'black' components with a magnet.

Process and quality engineers wanted to know the root cause of the failure to blacken, and whether there had been any cross contamination from the silver tubes to the black components. This would mean that the 'filtered' batch could not be used.

## SIMS Investigation of the Root Cause

Blackening and thermal emissivity are governed by controlled growth of the surface oxide so a surface analysis technique was used to investigate this problem.

Secondary Ion Mass Spectrometry (SIMS) is a powerful technique which analyses the surface of materials, by sputtering the outermost surface with gallium ions and separating the sputtered molecules according to their mass to charge ratio in a quadrupole mass spectrometer.

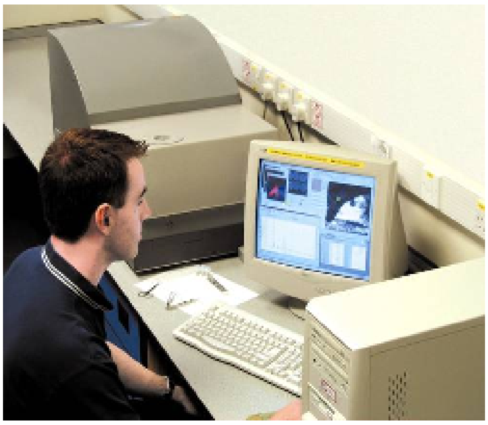

*Fig.1. The Millbrook MiniSIMS instrument.*

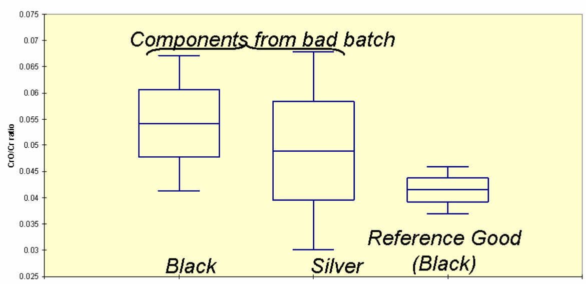


*Fig.2 Box plot showing Cr/CrOH for reference 'good' black components plus silver and black components from the suspect 'bad' batch. The ratios come from SIMS peak heights in the spectra and relate to oxide thickness. A box plot shows the mean value, distribution and range.*

SIMS is able to provide relative quantification of elements and molecules in the surface. The very high throughput of samples possible in the laboratory's Millbrook MiniSIMS instrument means that good sampling statistics can be achieved. Thus, data is representative of the variation within a batch. By comparing components a root cause and a prudent plan of action was quickly reached.

The SIMS analyses in fig.3. were able to show large differences between the normal 'good' black and the 'bad' silver components, which included silicon and hydrocarbon based contaminants.

The silver components were made from a different alloy than normal production. This could be seen in a range of spectral features and peak ratios such as a larger spread in their Cu/Cr and SiOH/Cr ratios. They also had excessive levels of hydrocarbons.

The fact that the CrO/Cr ratios were different for 'good' and 'bad' black components, proved that cross-contamination of the 'bad' black components had occurred. Processing with the incorrect silver components altered oxide growth rate of the rest of the components in the batch.

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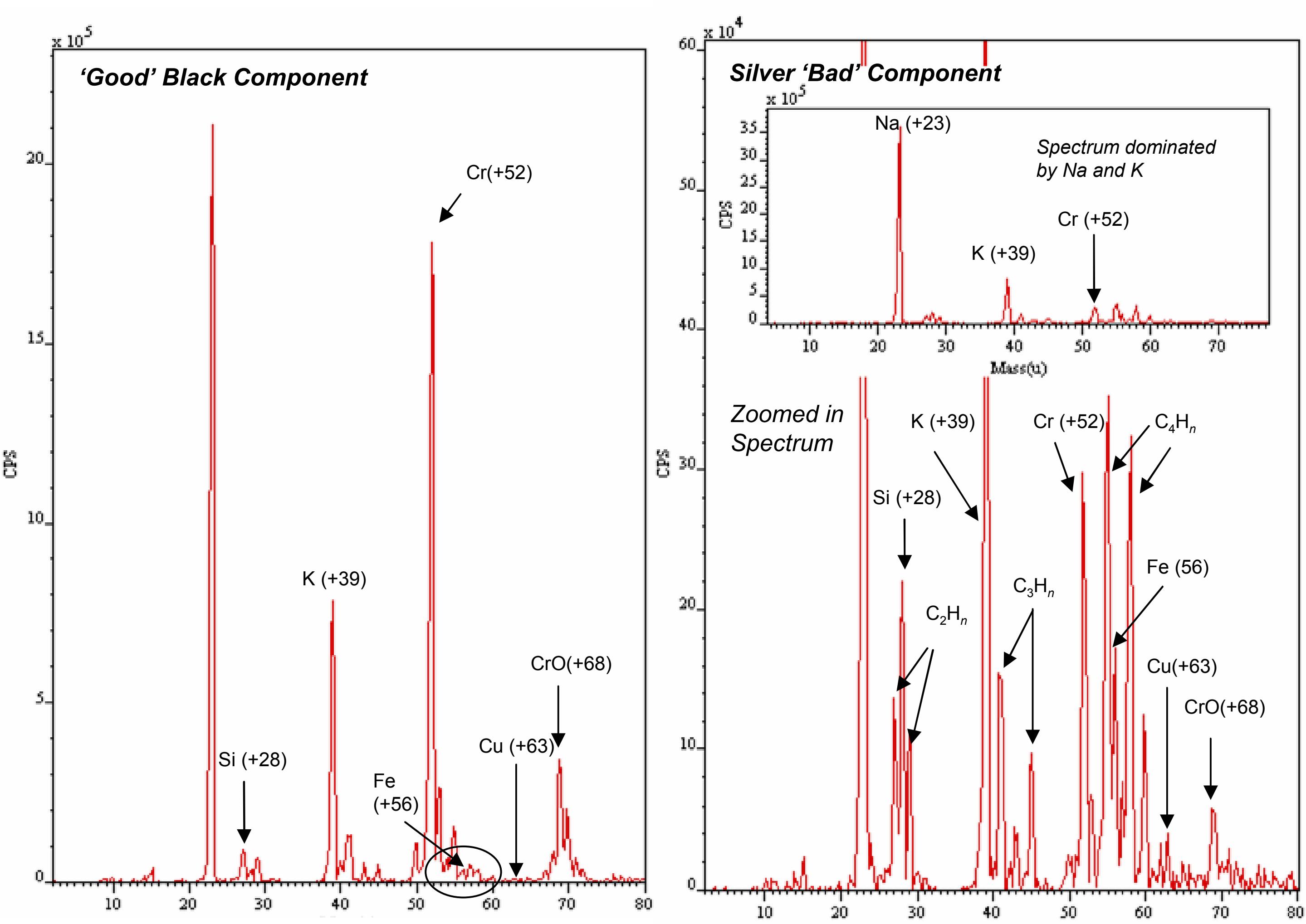


*Fig 3. Example comparison of SIMS spectra for the 2 extreme component samples. The silver tube appears to be organic and silicon contaminated.*

### Process Problem Solution

On the basis of the information from SIMS it was advised that the suspect component batch could not be used for production purposes – even with the silver components removed, because of the risk.

This scrapping decision prevented both yield loss in downstream processes and risking customer returns with all the associated bad publicity. Through further discussion the root cause was determined to be at the supplier. They had mistakenly delivered material intermixed with small volumes of a different metal alloy. A supplier audit process prevented this reoccurring through the resultant improvements in their production traceability.

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