Identification of the Type and Source of Particulate Contamination in a Ceramic Component Using SEM / EDX

Production Process Problem

A ceramic preform is used not only to hold together several metal components in a holder for thermionic cathodes but also to provide an electrically insulating barrier.

The ceramic is white, and in-line optical inspection is used to look for the presence of contamination on the preform.

This inspection rejected holders for the presence of dark specks on the surface of the glass that could lead to later insulation or poisoning problems for the cathode at the customer.

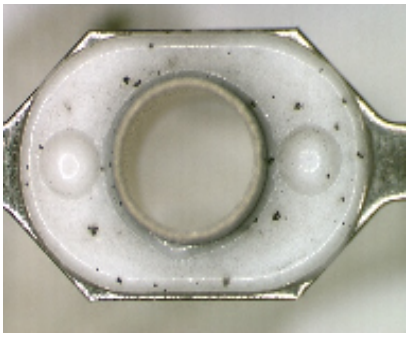

Fig. 1. Optical image of dark specks of contamination in sintered glass ceramic

The preform is produced from a glass powder, which is pressed into shape and sintered.

The metal components are then assembled together with the preform and thermally processed to first melt the glass around the metal and then re-crystallise it to form a rigid ceramic.

SEM and EDX Investigations

The scanning electron microscope (SEM) was used to examine the contamination at a higher magnification to determine whether the contamination was lying on the surface of the ceramic (surface contamination), embedded in the surface (contamination from the pressing operation) or within the surface (contamination in the glass powder).

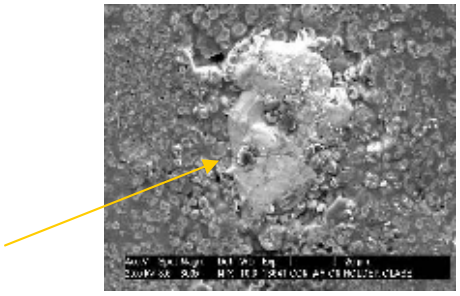

Fig. 2. Contamination on the holder ceramic

The contamination appeared to be within the surface, therefore some samples were fracture sectioned to look for the presence of contamination inside the ceramic.

Backscatter electron imaging was used to look for particles of a different chemical composition to that of the surrounding ceramic.

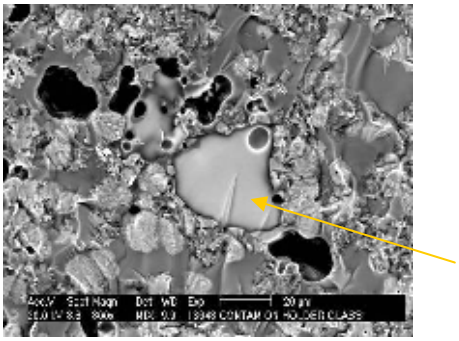

Fig. 3. Contamination inside the holder ceramic

By taking samples of sintered preforms from earlier in the process, it was possible to confirm that the contamination was present before the final assembly and thermal processing, implying that it was most likely to be present in the original glass powder, either as a result of contamination in the preform pressing process or from contamination in the incoming glass powder.

The particle of contamination was analysed by EDX and compared to the composition of the holder ceramic.

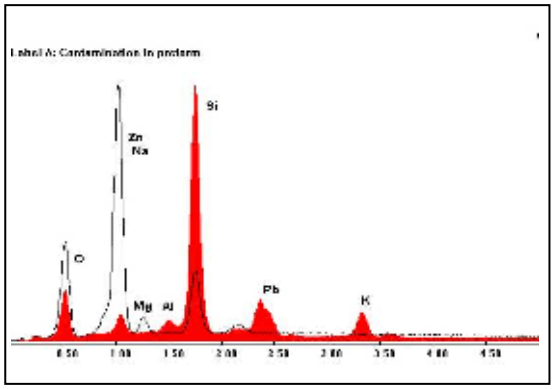

Fig. 4. EDX spectra of particle (red) and the normal ceramic (black outline)

The particle contained a mixture of silicon, lead, potassium, aluminium, oxygen, sodium and zinc, whereas the normal holder ceramic contained mainly zinc, silicon and oxygen.

The spectrum from the particle was quantified to give a composition (in weight %) of :

SiO_2	PbO	K_2O	Na_2O	Al_2O_3	ZnO
64.0	20.8	4.9	4.6	3.4	2.3

No materials of this composition were used on the site that manufactured the holders, but when the supplier of the glass powder was contacted, they recognised it as being very similar to another type of glass that they manufactured.

SiO_2	PbO	K_2O	Na_2O	Al_2O_3	ZnO
64.4	20.0	7.0	6.7	1.3	-

The composition of the glass particle had been slightly altered by being sintered inside the holder glass with its very different composition. However, all the main elements were present in characteristically similar concentrations.

An investigation by the glass powder supplier revealed that the machine used to prepare the holder glass powder had been used immediately before to prepare glass powder of the type found in the contaminated holders.

Although the machine had been cleaned before preparing the holder glass, the cleaning process had not been good enough to remove all traces of the previous glass powder.

The foreign glass powder would normally have been clear when melted, but the SEM analysis also revealed that where the glass had been exposed to the dry hydrogen atmosphere of the stove used to manufacture the holder, the lead oxide in the particle had been reduced back to form a film of metallic lead on the surface, and hence appeared black when seen optically.

The presence of a film of metallic lead on the surface of the ceramic could lead to insulation problems for the cathode, therefore batches of holders manufactured with the contaminated glass powder were identified with further SEM analysis and removed from production.

Process Problem Solution

The severe consequences that this type of glass contamination could have on the product was such that the supplier of the glass powder agreed to dedicate one machine to the preparation of glass powder, thus removing the risk of any further cross-contamination.