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high voltage behaviour of
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The influence of preparation methodology on high voltage behaviour of alumina insulators in vacuum.

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Abstract

The flashover characteristics of an insulator bridged high voltage vacuum gap can play an important role in the overall performance of a high voltage device, for example in the extreme environments of high energy particle accelerators. The detailed preparation of the insulators is, at present, governed by the commercial production methods and by standard bulk cleaning processes, which for a particular application may be far from optimum. The influence of particular cleaning technique have been investigated for commercially available alumina samples, with measurement of surface characteristics by scanning electron microscopy and laser diffraction and analysis of the high voltage performance with the possibility of applied fields up to 200kV/cm. The results of the different measurements are discussed in the overall context of the problems encountered in the full sized high voltage devices, and suggestions are made as to how the performance of alumina insulators could be improved by modification of the production and preparation specification

1 Introduction

Ceramics insulators are widely used for pulsed power and high-voltage (HV) applications. However their performances are limited by the occurrence of surface flashover [1]. For instance HV devices used in CERN's accelerators and exposed to various radiation fields are sensitive to this phenomenon, often adversely affecting accelerator performance [2]. In the case of beam electrostatic extractors [3], solid and hollow rods of sintered alumina are used respectively as insulating supports and HV feedthroughs to power electrodes at up to -300 kV, providing working fields as high as $15 \text{ MV} \times \text{m}^{-1}$. A high reliability level is required for these permanently powered devices.

In addition, machining steps are necessary to achieve the required geometries. It has been recently shown that electrical properties of hard ceramics, such as space charge trapping [4, 5] or the HV holdoff [6, 7], are sensitive to many kind of stresses, e.g. machining, polishing, handling, brazing and cleaning operations. Further, concerning cleaning operations, the prohibition of Freon for safety environmental considerations made it necessary to find new cleaning agents and/or techniques which could provide equivalent results.

Also, it has been recently shown that UV stimulated flashovers on sintered alumina could be dramatically reduced by appropriate physical surface treatments [8]. A low spark rate under UV irradiation would be a useful criterion to choose the suitable global process for insulating materials which are to operate in harsh dynamical environments like in neutron and X-ray sources, or particle accelerators.

These specific problems motivated our choice to study commercial polycrystalline material instead of single-crystals. Thus our programme is aimed at investigating the influence of sample preparation on HV performance of a bridged vacuum

gap under UV bombardment, focusing in the present paper on effects of surface chemical cleaning procedures. In section two, the descriptions of a new ultra high vacuum HV test stand coupled with an optical UV facility are given with the experimental procedures. The third section presents and discusses HV test results obtained with samples cleaned with different chemical agents. In section four conclusions and outlook are given.

2 Experimental set up

2.1 High voltage test facility

The HV apparatus consists of a UHV stainless steel chamber housing an insulator bridged vacuum gap. The spacer has a simple cylindrical shape with 10mm diameter and 8mm thickness, whereas the electrode geometry (see Figure 1) has been designed so that the distribution of the equipotential lines across the insulator are similar to that of HV insulating supports and feedthroughs for CERN's electrostatic separators [9]: the presence of the small shim provides a light shielding effect of the triple junctions.

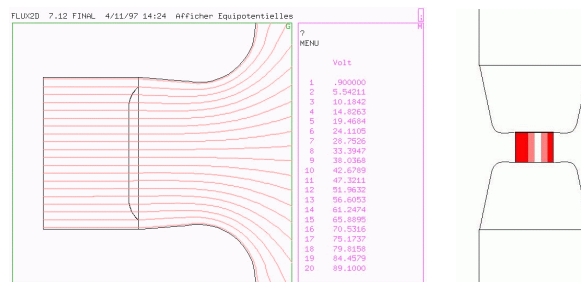


Figure 1 : Equipotential lines and final geometry after Flux2D (ver7.12) calculations.

The anode (upper electrode) is powered by a 220kV DC power supply, however the maximum applied potential across the gap does not exceed 120kV. The occurrence of sparks is detected via two separated systems : the first one based on a capacitive sensor for fast potential drops placed upstream of the HV plug, the second one detecting fast current discharges, placed downstream of the cathode. However, the number of sparks discussed later is given by the first system, with a threshold level for detection of 8kV due to capacitive effects of the HV cables. At present the resolution of our leakage current monitoring is poor ($\sim 1\mu\text{A}$), but its upgrade is planned for the near future.

The vacuum chamber is filled with dry nitrogen before being opened for samples' replacement. Before starting a series of HV tests, a three day baking step is necessary to achieve a working pressure better than 6.10^{-10} Torr. The temperature is slowly increased ($20^\circ\text{C}/\text{hour}$) at up to 300°C . After a 24 hour plateau at high temperature, then the vacuum chamber is let to cool down to room temperature at the same rate. A mass

spectrometer is also mounted for residual gas analysis purposes.

The main feature of the HV test stand is the very fast turn-over of the samples : a set of ten insulators per test series is loaded on an alumina tray. Placing or removing a sample is done under UHV conditions with the operating procedure shown in *Figure 2*. Firstly, while approaching the alumina tray with a linear rack towards the electrodes, the motorised bottom electrode is moved down. Secondly, a mechanical hand equipped with Macor™ jaws catches the sample to be tested. Then the bottom electrode is moved up to an intermediate level so that one can place the sample. Finally, the mechanical parts are retracted and the bridged vacuum gap is formed, with an applied pressure of ~0.5 kg. The reverse procedure is used for removing a sample. The complete operation lasts about 15mn.

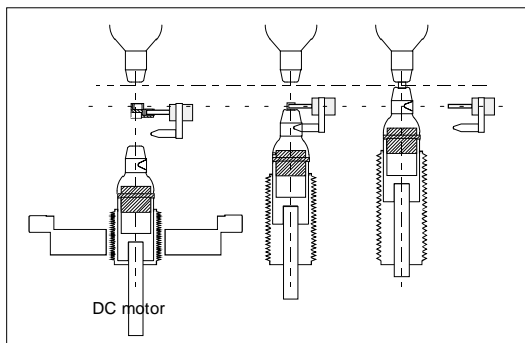


Figure 2 : Operating procedure for placing a new sample

2.2 Optical bench

An external optical facility is added to trigger UV stimulated flashovers (see *Figure 3*). The UV source consists of an Oriel housing for a 200W Hg/Xe lamp whose light is collimated by two diaphragms. Further, a simple optical bench is implemented to investigate some effects of photon energies. The system is based on a set of 1" diameter Excimer filtering mirrors providing a high reflectance level (>99% at 45° angle of incidence), as compared to UV filters whose transmission coefficients do not exceed 20% in the UV range. The selected wave lengths are 253nm, 313nm and 365nm, which correspond respectively to photon energies of 4.9, 4 and 3.4 eV.

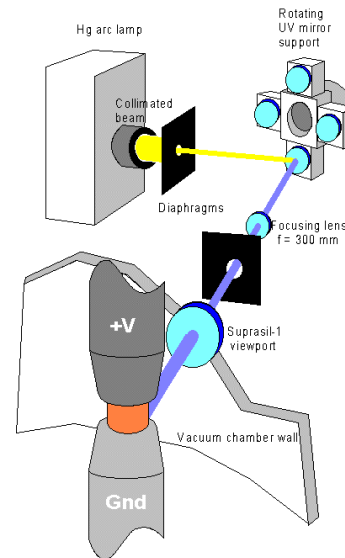


Figure 3 : Optical bench overview.

An extra Al coated mirror is also used to reflect the whole spectrum light. The mirrors are set on a wheel which is activated by a stepping motor. A 300mm focusing lens is also placed on the optical path to increase the radiation intensity on the samples through a fused silica UHV viewport. Half cylindrical area of the spacer is irradiated by the UV spot.

2.3 Sample preparation

Cylindrical insulators made of 97.6% sintered polycrystalline alumina material (Al300) were obtained from Wesgo Ceramics, Erlangen, Germany. The macrofinishing was achieved using a grinding wheel which contains 75-90µm diamond grain size bond in metal with a concentration of 3.3 carat/cm³. As illustrated in *Figure 4*, ground surfaces present an extensive porosity resulting from the insufficient plastic deformation of the material that produces grain removal and cracks. The dimension of the pores are typically 10-20µm diameter and 15-30µm depth, whereas the R_a value is small : 1.8µm.

After being machined, the spacers underwent a cleaning process containing steps in aqueous solutions with and without cleaning agents, mostly using deionized water and ultrasonic tanks. Precise details of the latter step were not communicated by the manufacturer for confidentiality reasons. Before placing the samples in the vacuum chamber, some of them underwent an extra cleaning step performed by the EST/SM group at CERN. Others were used as received from Wesgo. A total of six defined cleaning procedures have been tested so far (see Table 1), two insulators being cleaned by each agent. "Recycled" Axarel, as opposed to "new", means the use of worn and filtered product and refers to CERN's standard practice.

Ensolv	10mn at 50°C +US; 10mn at 50°C +US; 10mn at 50°C +US; dry for 30' at 80°C in air
Biosane	10mn at 60°C +US; 10mn at 60°C +US; 10mn at 60°C +US; dry for 30' at 80°C in air
Freon	20mn at 23°C; 20mn at 23°C + US; dry for 20' at 50°C in air
Axarel 9100 (new)	20mn at 75°C; 20mn at 50°C + US; 20mn at 75°C; dry for 5 hours at 150°C in air
Axarel 9100 (recycled)	20mn at 75°C; 20mn at 50°C + US; 20mn at 75°C; dry for 5 hours at 150°C in air

Dowclene PX 16 S	10mn at 55°C +US; 10mn at 50°C +US; 10mn at 50°C +US dry for 3 hours at 60°C in air
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Table 1: Details of the surface cleaning procedures. "US" stands for ultra sonic bath.

2.4 Testing procedure

A new sample is first conditioned to V_c (120 kV here) prior to any further investigation. During this first step, the potential increase is monitored by a software regulation loop based on the current supplied by the HV generator and vacuum level feedbacks. The other parameters recorded are the potential, the conditioning time, the number of sparks and current discharges. After reaching V_c , the potential level is maintained for a 30 mn plateau. Then, the potential is switched off and then back on to $V_w = 115\text{kV}$ for studying the HV performance. The applied procedure consists of three 20 minute sequences shown in Figure 5. At the end of a complete test, the sample is replaced by a new one as described earlier in §2.1.

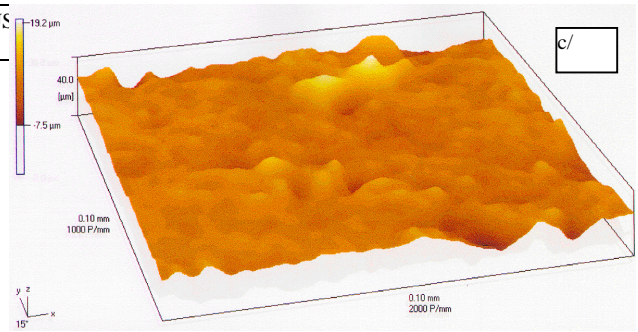
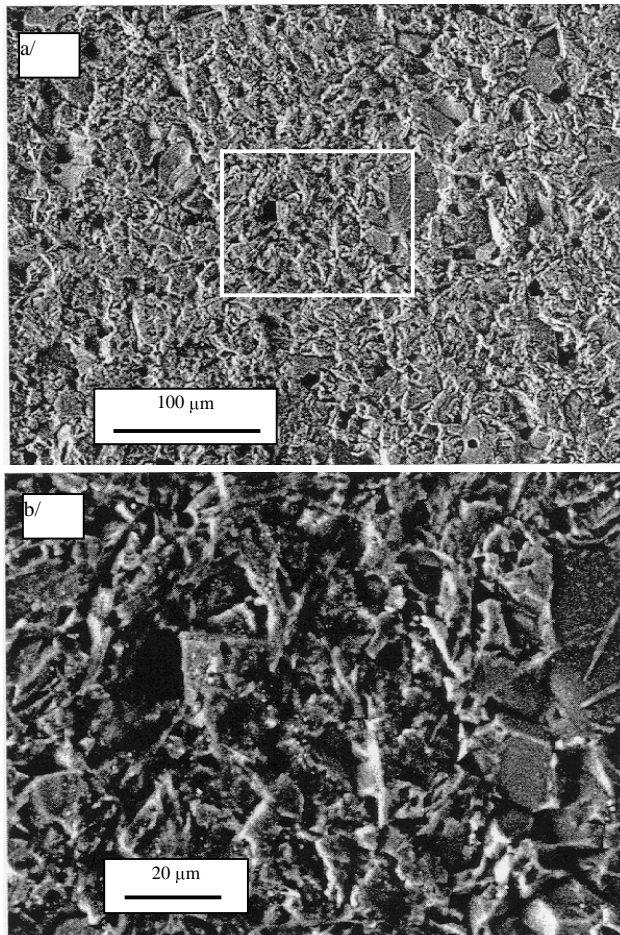


Figure 4 : Al300 alumina surface characterisation : a/ and b/ Scanning electron micrographs, c/ Topography by laser diffraction technique (from EMPA/COMET).

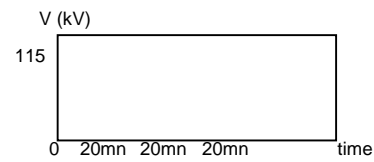


Figure 5 : Testing sequence for all samples, after conditioning.

3 Findings and discussions

3.1 Preliminary results

The commissioning of the HV facility was performed with a batch of ten "as received" insulators. Details of our observations will be published in a separate paper, but one can summarize as follows :

Conditioning step : Many current discharges up to 500-600 μA were detected at $\sim 20\text{ kV}$. The number of such discharges is in the range 60-120 before the occurrence of the first flashover, i.e. producing a potential drop. The first flashover level was remarkably reproducible, with an average value of 40.8 kV and a standard deviation of 1.35 kV. About 32 mn are necessary to reach V_c , with 120 to 140 sparks. The mean spark rate during the 30 mn plateau is 1.2 mn^{-1} with a standard deviation of 0.56 mn^{-1} .

Working sequence : The motivation for working at a single high potential is due to the absence of dramatic increase of UV induced sparks, whatever the photon energy, below a threshold value of V_w : the spark rate stood then between 0 and 0.7 mn^{-1} . At $V_w = 115\text{ kV}$, the steps without UV always still exhibited similar low values. However, the figures significantly increased with UV irradiation to range between 1.8 and 2.6 mn^{-1} for 15mn. With longer exposure time to UV photons, say 3 hours, it was observed that the breakdown rate was decreasing. Other authors had already reported this observation [10]. The main feature of these tests is a very low leakage current of $\sim 2\text{ }\mu\text{A}$ though operating at high field levels i.e. 14.3 and $15\text{ MV}\cdot\text{m}^{-1}$.

Visual inspection of the sample surface after HV test : Systematic yellow/brown discoloration of the cylindrical surface, preferentially near the anode, has been observed. These patterns tend to homogenize on UV irradiated area. Such discolorations were scarcely found on both base areas. Patches of metalisation were localized randomly on both triple junctions.

3.2 Effects of cleaning procedures

In the present study, aluminium coated mirror is only used to light up the sample with UV.

Conditioning step : The first flashover level and the conditioning time are represented in Figure 6. The different cleaning processes produced a light effect on the first flashover level, decreasing the average value to 40.2 kV and increasing the standard deviation to 2 kV. In a recent paper presenting results of flashover strength studies in a pulsed mode, where the samples experienced only few sparks to avoid damages, Sudarshan and Li [7] established that the chemical cleaning did “not have a significant influence on the flashover strength of alumina”. Concerning the conditioning time, it is striking to see how reproducible it is for a given batch. If one compares “recycled” Axarel and PX16S, the conditioning time can be three times longer though the feedback parameters are identical for all samples tested. As shown in Figure 7, the integrated number of flashovers during conditioning and flat top steps exhibits the same trend described above.

Working sequence : As previously mentioned, a low inter electrode current could be measured between two spark events, with and without UV light. Without UV irradiation, Biosane and both Axarel feature a low spark rate, yielding maximum values of 0.3mm^{-1} and 0.05mm^{-1} respectively for the first and the last step, as compared to those of Freon which are 3 to 4 times higher. Under UV spot light, a sample cleaned with PX16S exhibited bulk/surface damage features : the abnormally large number of flashovers, 615 here, and a nearly linear number of events with time - note that the latter phenomenon represents about 10% of the 40 spacers tested so far. Concerning the other cleaning procedures, on one hand, Axarel and Biosane still seem to be less sensitive to UV bombardment, though there is a larger scatter in the spark count. On the other hand Freon, Ensolv and PX16S processes did not yield a good HV behaviour. This may be linked to the polar nature of these three agents whose chemical classification is respectively CFC 113, Aliphatic (meaning benzenic cycle free) hydrogenated hydrocarbon and Modified alcohols, presenting a high electronic affinity with alumina. Axarel and Biosane are classified respectively as Blend aliphatic hydrocarbon and Azeotropic hydrocarbon, that are free of chemical polar functions. A long 300°C baking cycle is clearly not sufficient to remove some chemical species from the surface. Shallow trapping and detrapping properties of charge carriers may be considerably affected.

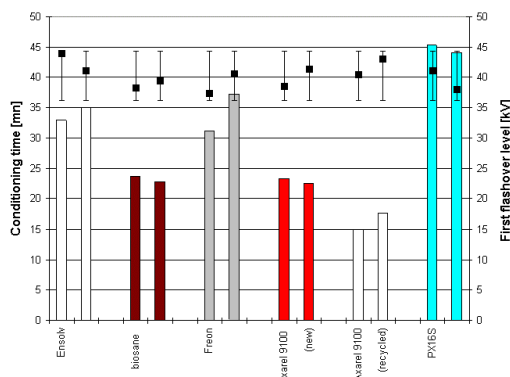


Figure 6 : Cleaning effects : first flashover level (points) and conditioning time (bars).

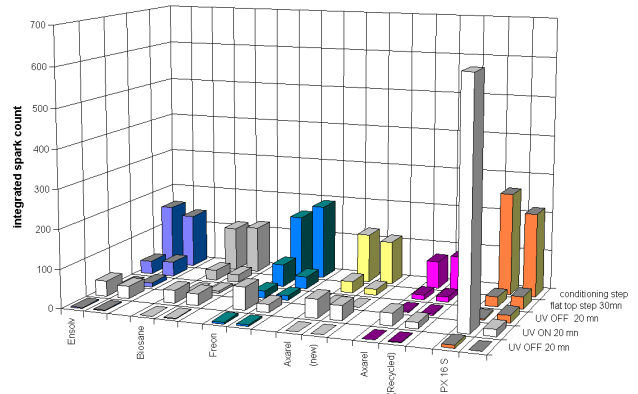


Figure 7 : Cleaning effects : integrated number of sparks during the global test sequences.

Further, Coaker et al. [11] proposed a surface defects-initiated model of surface flashover which can be summarized as follows : under DC stress, significant electron trapping occurs at defects (impurity and oxygen vacancies) within the pore. A local field enhancement across the pore allows trapped electrons to tunnel out of these defects. At a given threshold field, explosive relaxation polarisation with material jets takes place across the pores. As a result for the HV holdoff of alumina, changes of surface properties owing to macrofinishing and chemical cleaning may be advantageous or detrimental.

Residual gas analysis : The residual gas after a baking cycle mainly consists of 94% of H_2 , 3% of H_2O , 2% of CO and 1% of other species (CH_4 , Ar , CO_2 , C_3H_8 , C), with a total pressure of 6.10^{-10} T. A residual gas analysis was performed after a testing a complete set that corresponds to a given cleaning agent. The total pressure increased then to $\sim 2.10^{-9}$ T. The distribution slightly changed giving 90% of H_2 , 3% of H_2O , 4% of CO , 2% of CH_4 , and 1% of other species (Ar , CO_2 , C_3H_8 , C). No signature of a cleaning agent used here could be detected, within the sensitivity of our mass spectrometer.

4 Conclusions and future actions

Improving the HV holdoff of an insulator bridged vacuum gap still remains a technological challenge for both industrial and scientific communities. This need motivated the construction of a new ultra high vacuum HV test stand at CERN. It has been shown that the cleaning procedures could significantly influence the occurrence of flashovers with or without UV stimulation. Alumina samples cleaned with Biosane and Axarel exhibit low spark rates, owing probably to their non polar nature. The production of Axarel will be stopped in the near future but it could be replaced by Biosane. However the cleaning efficiency has to be confirmed with contaminated samples (e.g. with finger prints).

The influence of the mechanical preparation, thermal history and particular cleaning techniques will be investigated, with measurements of surface characteristics and topography, of the secondary electron emission curves and analysis of the HV performance.

The HV facility will be upgraded by improving the current monitoring resolution, and implementing a video recording apparatus.

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