Surface studies of dielectric materials used in spark gaps

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G. Jackson, L. Hatfield, M. Kristiansen, M. Hagler, J. Marx, A. L. Donaldson, G. Leiker, R. Curry, and R. Ness
Physics, Electrical Engineering, and Chemistry Departments, Texas Tech University, Lubbock, Texas 79409

L. Gordon
Lawrence Livermore National Laboratory, Livermore, California 94550

D. Johnson
IAP Research, Incorporated, Dayton, Ohio 45459

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Dielectric materials commonly used as insulators in spark gaps (lexan, nylon, lucite,macor, boron nitride, delrin, and G-10) have been exposed to the byproducts of arcs in three different spark gap experiments. The first was a 60-kV, 0.05-C/shot spark gap using copper-tungsten or graphite electrodes at various pressures of N_2 and SF_6 gas. The second was a 5–30-kV, 4–25-kA, 0.1–0.6-C/shot, unipolar, pulsed spark gap using graphite, copper-graphite, copper-tungsten, brass, and stainless steel electrodes in N_2 gas or air. The third was a 45-kV, 0.009-C/shot surface discharge switch. Surface analysis of these insulators indicates that most become coated with a thick layer of electrode material depending upon the type of gas, electrode, and insulator material used, and the conditions of the arc. However, lucite insulators inserted in the second spark gap using graphite electrodes and air showed no indications of deposited electrode material on the surface but did show small particles of graphite imbedded in the surface. The self-breakdown voltage (V_{sb}) statistics for spark gaps with insulator inserts show significant deviations from the V_{sb} statistics for spark gaps without insulator inserts, which may be due to deposits of insulator material onto the electrodes.

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INTRODUCTION

The purpose of this study is to investigate the chemical and physical processes that lead to degradation of insulator materials used in high voltage spark gaps, and to determine how these processes vary with different combinations of electrode material, filler gas, insulator material, and spark gap operating parameters. Various insulating materials were exposed to the byproducts of discharges in three different, high power spark gaps. The synergism between the electrode, gas, and insulator materials used in the spark gap can be very important in repetitively operated gaps. Each successive arc induces chemical, mechanical, and thermal processes which affect all parts of the gap. These processes depend on the materials in the gap and the gap operating parameters, such as, peak current, repetition rate, charge transfer, etc.

EXPERIMENTAL ARRANGEMENT

The spark gap (from this point on referred to as Mark I), shown schematically in Fig. 1, was used to expose lexan and blue nylon insulators to 5000 shots each in a spark-gap-switched, critically damped RC circuit.[1] The insulators were inserted as $6\times6\times1/4$ in. flat plates located approximately 1.5 cm away from the 5-cm-diam hemispherical electrodes. The electrode material and the gas in the gap were varied in order to understand better how these parameters affect an insulator. During these experiments the insulators were subjected to discharges in either 2 atm absolute of N_2 gas or SF_6 gas with either graphite or K-33 (tungsten-copper composite) electrodes. The spark gap self-breaks at 40–45 kV and switches approximately 1 kJ of energy in 2 μs at a maximum rep-rate of two pulses per second. The maximum current in the spark gap is 50 kA with a charge transfer of about 0.03 C per shot.

The dielectric plates were cut from standard commerically available sheets using a specially cleaned band saw, cleaned with cyclohexane, and inserted in the chamber. New electrodes were used for each experiment. The electrodes were machined using carbide tipped tools and no cutting fluid. Before insertion in the chamber the electrodes were cleaned with ethynol. The chamber was evacuated to 1×10^{-4} Torr using a turbomolecular pump and then flushed with the gas to be used before filling to the desired pressure. No other preconditioning of insulators or electrodes was done.

The spark gap (from this point on referred to as Mark II), shown schematically in Fig. 2, was used to expose Lucite insulators to 50 000 shots from a unipolar discharge.[2] The insulators were inserted as cylinders located approximately 6.25 cm from the discharge region between two 2.5-cm-diam hemispherical electrodes. The electrode materials used were graphite, copper-graphite composite, two different tungsten-copper composites (K-33 and Elkonite), brass, and stainless steel. The spark gap normally operates at a voltage that is less than 30 kV and switches 9 kJ of energy in 25 μs at a maximum rep-rate of five pulses per second. The maximum current in the spark gap is 25 kA and the maximum charge transfer is 0.6 C per shot. The chamber is continuously flushed so that one chamber volume flows through every 5 s with either air or N_2 gas, while the pressure is maintained at 1 atm absolute.

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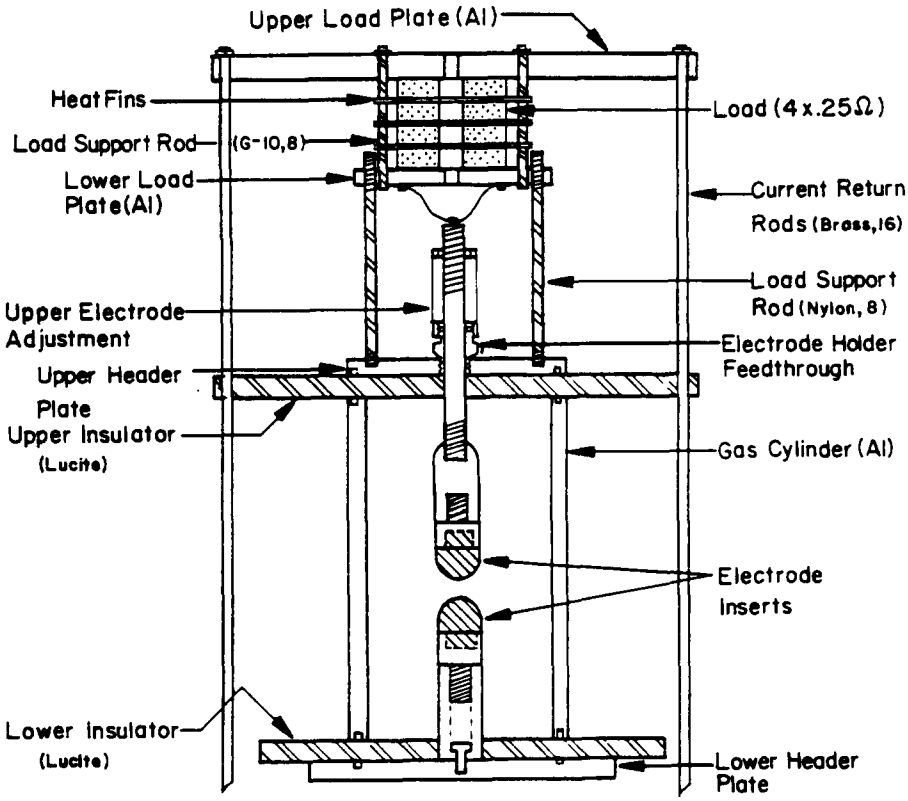

FIG. 1. Mark I spark gap.

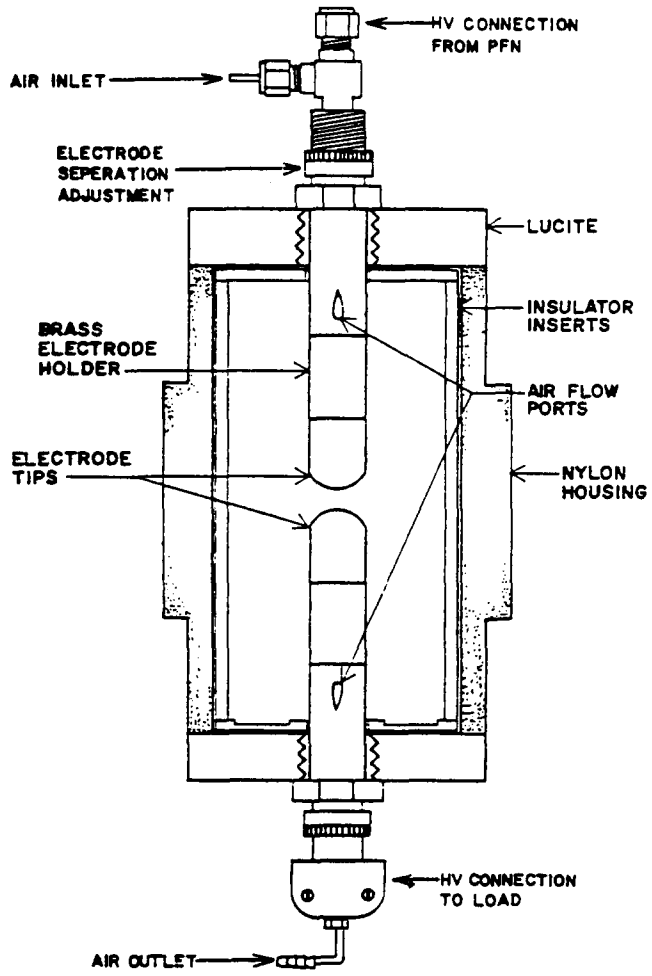

FIG. 2. Mark II spark gap.

For each experiment new electrodes were machined as for the Mark I gap and a new lucite cylinder, cleaned with cyclohexane, was inserted. The cylinders were cut from commercially available lucite tubing. No kind of preconditioning was attempted.

Since the gas is flowing through the Mark II gap during the experiment, no evacuation was necessary. Ample time for flushing was allowed before starting each 50 000 shot experiment.

A surface discharge switch shown schematically in Fig. 3 was used to expose lexan, blue nylon, lucite, macor, boron nitride, G-10, and delrin insulators to 10 000 shots from a "surface" discharge across the insulator surface.[3] The switch operates at 45 kV in the ambient atmosphere (lab air) and transfers 180 J per shot at a rate of 1.2 pulses per second.

The dielectric plates, except for the boron nitride, were cut using clean saws from commercially available sheets. The boron nitride was purchased already cut to the required size. All samples were cleaned with cyclohexane before inserting in the switch. There was no preconditioning of the switch.

It was observed that after a few shots the switch stopped multichanneling. However, when an air jet of "dirty" lab air was blown across the surface of the insulator the sample continued to multichannel until the air jet was turned off. It is suggested that this phenomenon is due to an accumulation of surface charge which deters multichanneling and that the air jet somehow manages to alter this distribution of surface charge in such a way that multichanneling can still occur.[4]

After the insulators were exposed to the byproducts of these arcs they were visually examined for any gross features such as crazing, metal vapor deposition, cracks, or large particles and then studied with several surface analysis tech-

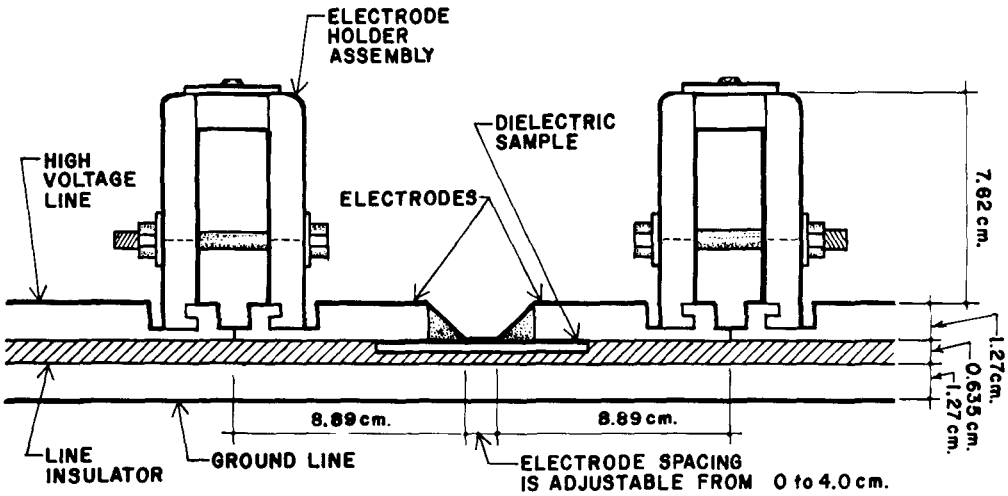

FIG. 3. Surface discharge switch.

niques including electron spectroscopy for chemical analysis (ESCA),[5] scanning electron microscopy (SEM),[6] and x-ray fluorescence (XRF).[7]

Standard methods of clean handling were employed for cutting specimens for analysis to avoid contamination due to fingerprints, etc.

RESULTS

Insulators exposed in Mark I

Analysis of an unexposed lexan insulator with ESCA shows the surface to be composed of 86% carbon and 14% oxygen. This stoichiometry is in good agreement with that of lexan ($C_{16}O_3H_{18}$). It should also be noted that lexan exhibits negative surface charging when exposed to the x-ray beam used in the ESCA spectrometer (as observed by the negative shift in the binding energy of the C_{1s} line). This phenomenon is rare in ESCA.[8] However, for polymers such as lexan which have an aromatic benzene ring it is possible to observe a negative charge accumulation on the surface.

A lexan insulator which was exposed to 5000 shots in the spark gap with graphite electrodes in 2 atm of N_2 also exhibits negative surface charging in the ESCA spectrometer. The ESCA spectrum for this particular sample shows a marked decrease in the amount of carbon and a large increase in the amount of oxygen present on the surface. An expanded scale spectrum of the C_{1s} peak shows it to be considerably broadened compared to the expanded scale spectrum of the virgin sample, probably due to some form of C-N or C-O bonding, since there is some nitrogen and oxygen seen on the surface of this sample. However, the resolution of this peak is not adequate to identify the specific forms of carbon bonding present on the surface. Figure 4 shows a scanning electron microscope (SEM) micrograph of this sample which shows the surface to be imbedded with small microparticles (the largest of which is approximately 2 μ in size). Since the analysis area of ESCA is a circle with a diameter of about 4 mm, these microparticles cannot be identified through the use of ESCA because they are too few and too small. The use of XRF to determine the composition of these particles gave an indeterminate result. Therefore, it is probable that these microparticles are carbon from the graphite electrodes.

Another lexan insulator was inserted into this gap with 2 atm of SF_6 and graphite electrodes. The ESCA scans of this sample showed some surprising results. The amounts of carbon and oxygen seen on this insulator are extremely small compared to the virgin sample, and there is a large amount of aluminum and fluorine seen on the surface. In addition, the surface no longer accumulates a negative surface charge as did the virgin sample but instead exhibits positive charging. The expanded scale spectrum of the Al_{2p} peak shows a shift in the binding energy of this line, which corresponds exactly to AlF_3, and the stoichiometry of the aluminum to fluorine in the spectrum is almost exactly 3:1. Figure 5 is an SEM micrograph of this sample showing a heavy coating of powdery material on the surface. An x-ray dot map of the surface showed that this powder contains aluminum, consistent with the ESCA indication of aluminum fluoride. The fact that the surface was covered with this material explains why the measured concentration of carbon and oxygen is so small on the surface and why the surface of this particular sample exhibits positive surface charging, whereas the two previous samples show the opposite effect. The presence of the aluminum on the surface of this sample was the result of misfires

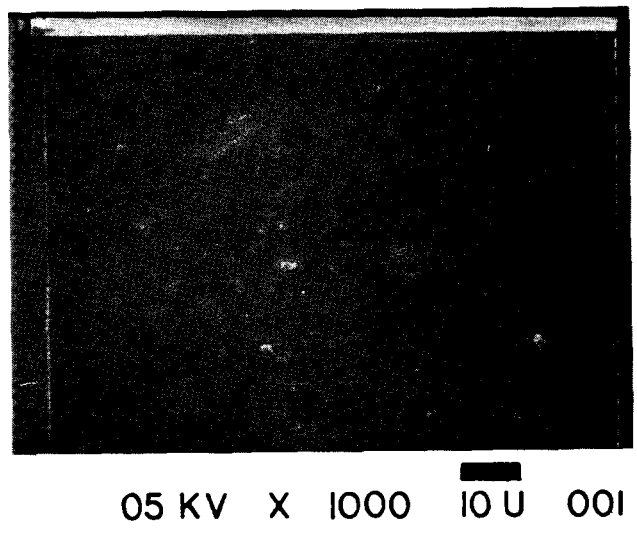

FIG. 4. SEM micrograph of a lexan insulator, which was inserted into Mark I with graphite electrodes in 2 atm of N_2 for 5000 shots.

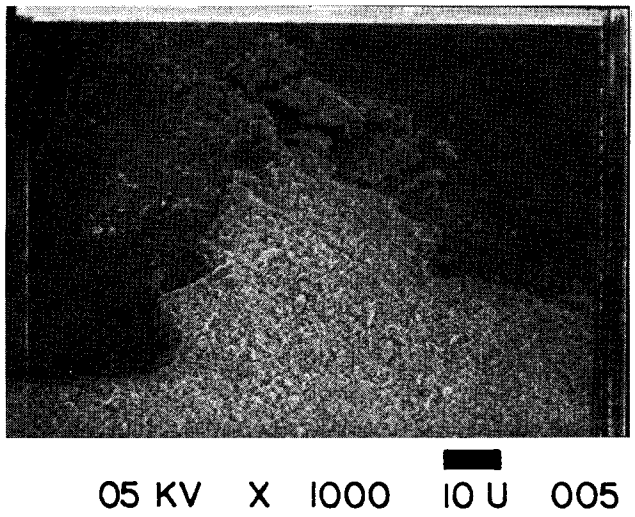

FIG. 5. SEM micrograph of a lexan insulator, which was inserted into Mark I with graphite electrodes in 2 atm of SF_6 for 5000 shots. The powdery substance on the surface is AlF_3.

(arcs) to the aluminum walls of the chamber or arcing between the electrode and the aluminum electrode holder which liberated metallic aluminum. The metallic aluminum then reacted with the fluorine in the gas to form AlF_3 which subsequently deposites inside the spark gap. As a result of these measurements, the arcing between the electrode and the aluminum electrode holder was discovered and eliminated.

For a lexan insulator exposed to 5000 shots in this same spark gap, with 2 atm of N_2 and K-33 electrodes, the ESCA spectrum indicates that the surface of the sample is covered with a very thin layer of copper and tungsten. An expanded scale spectrum of the C_{1s} peak indicates that there is more than one form of carbon present on the surface; however, the resolution of this peak is not sufficient to identify the forms of carbon present. Expanded scale spectra of the copper and tungsten peaks indicate that the tungsten exists in some form of oxide; however, the copper spectrum shows pure metallic copper on the surface. Figures 6 and 7 are micrographs of the surface showing that it is covered with a thin, discontinuous layer of a metallic-looking substance. X-ray dot maps on several large conglomerations apparently formed from molten material indicate that they are composed mainly of copper with a tungsten background. It has been suggested that this means that the copper and tungsten from the electrodes are deposited on the insulator with similar deposition rates.[9] The tungsten, which has a rapid rate of reduction compared to copper, is oxidized on the way to the insulator (by the oxygen in the spark gap in the form of water). Most polymers will absorb anywhere from 3% to 8% by volume of water. This could provide a source of water in the system to account for the presence of the oxygen necessary to oxidize the tungsten present on the insulator. This process yields a deposited layer of copper covered with tungsten oxide on the insulator, so that the copper in this layer never has an opportunity to oxidize even after the insulator is removed from the spark gap.

ESCA analysis of lexan insulators exposed to 5000 shots in 2 atm of SF_6 with K-33 electrodes shows a significant increase in the amount of tungsten (from 4% to 13.3%) present on the surface, as compared to the previous situation in N_2. There is also a large decrease in the concentration of carbon on the surface. However, this may be attributed to a masking of the constituent carbon of the lexan insulator by a surface coating of copper and tungsten, which are both present in large amounts on the surface. Another alternative explanation for the reduced concentration of carbon on the surface, is that the observed carbon is due to hydrocarbon contamination from the air (after the sample is removed from the gap) and not derived from the insulator. The SEM micrographs of the surface indicate that it is covered with a large concentration of powdery material, which x-ray copper dot maps indicate to be some form of copper. These dot maps cannot be used to determine the chemical form of the copper; however, a large concentration of fluorine is also

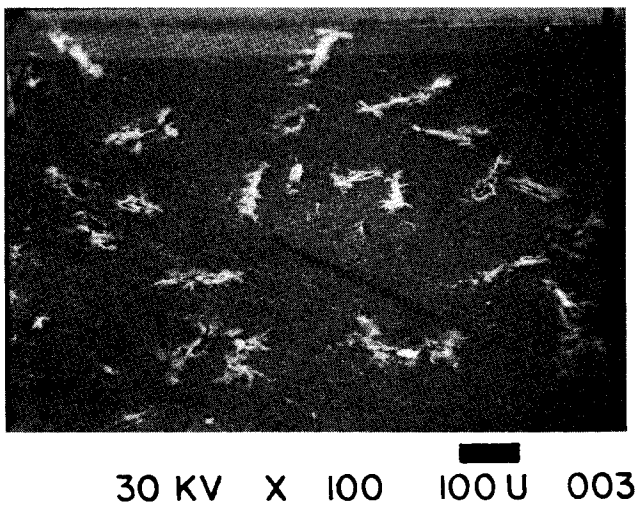

FIG. 6. SEM micrograph of a lexan insulator, which was inserted into Mark I with K-33 electrodes in 2 atm of N_2 for 5000 shots. The "splatts" on the surface are metal vapor deposits from the electrodes.

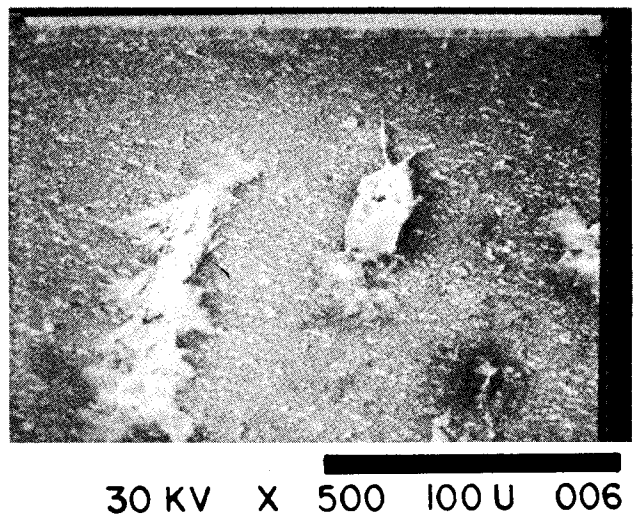

FIG. 7. SEM micrograph which shows a magnified area of the lexan insulator shown in Fig. 6.

observed (using ESCA) on the surface. It is possible that this powder is a copper fluoride. Since fluorine has an atomic number less than 13 it is difficult to observe it using XRF and therefore ESCA was employed. The detailed scan of the copper by ESCA gives an ambiguous answer. The broadness of the detail scan of the F_{1s} peak, however, seems to indicate that there is more than one form of fluorine on the surface (either a copper fluoride or a carbon fluoride). Some other technique needs to be used to understand better the composition of this powdery material.

Similar experiments were performed in this spark gap using a "blue nylon" insulator. The ESCA spectrum of the virgin sample indicates that the insulator is composed of carbon, oxygen, nitrogen, and silicon. Blue nylon is an insulator which is cast in a mold with a blue dye (and not extruded as are most of these polymers). This casting process leads to a larger percentage of cross linking in the polymer and consequently a higher tensile strength. The usual composition of nylon is $C_6H_{11}ON$; however, for the blue nylon insulator the ESCA data gives a concentration of nitrogen in the material that does not agree with this stoichiometry and the presence of silicon in the material was somewhat of a mystery. However, the silicon on the surface of the blue nylon is probably a constituent of a mold release used in the manufacturing process. For a blue nulon insulator inserted into the spark gap with graphite electrodes and 2 atm of N_2, the ESCA spectrum shows that the nitrogen on the surface has disappeared after 5000 shots. Probably the surface is covered with a thin continuous layer of carbon which is masking the nitrogen in the blue nylon. Since the electrodes used are graphite and blue nylon is a carbon based material it is very difficult to verify this hypothesis with ESCA.

The blue nylon insulator inserted in the spark gap with graphite electrodes and 2 atm of SF_6 also appears to be covered with a powdery substance. Figure 8 is an SEM micrograph of this surface. An ESCA spectrum of this sample indicates a large concentration of aluminum and fluorine.

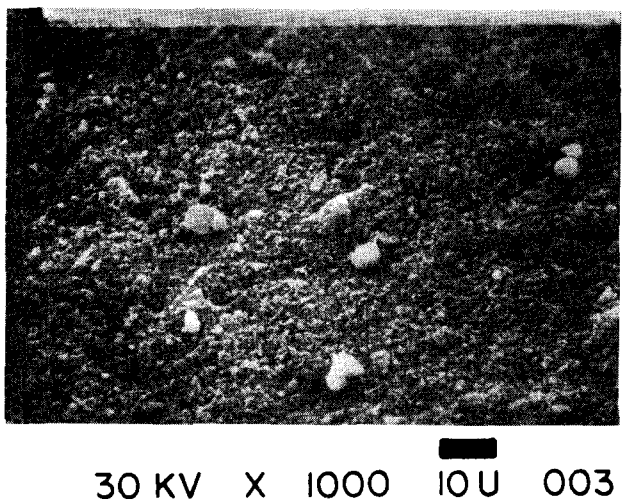

FIG. 8. SEM micrograph of blue nylon insulator, which was inserted into Mark I with graphite electrodes in 2 atm of SF_6 for 5000 shots. The powdery substance on the surface is probably AlF_3.

An expanded scale spectrum of the Al_{2p} peaks indicates that the aluminum is in the form of AlF_3, which is consistent with results discussed above. SEM micrographs and aluminum x-ray dot maps support this result. The C_{1s} peak indicates some form of CF bonding on the surface, however, the resolution of this peak is not sufficient to determine which CF compound is responsible.

The ESCA spectra of the blue nylon insulator from the spark gap with K-33 electrodes and 2 atm of N_2 indicate that the amount of carbon on the surface has been greatly reduced (from 78% to 47%) and that the amount of oxygen and silicon have been increased considerably. The data indicate that there is no copper or tungsten on the surface. The SEM micrographs of this sample also indicate that copper and tungsten are not present on the surface. This difference, compared to results with the lexan insulator, could possibly be due to the silicon mold release, which leaves the surface "slick" so that metal vapor will not deposit and adhere to the surface. The data for the blue nylon insulator in the spark gap with K-33 electrodes and 2 atm of SF_6 show results similar to those for blue nylon with K-33 electrodes and N_2. This indicates that the lack of metal films on the surface is not related to the use of SF_6. Further test are necessary to determine the role of the silicon on the surface.

Analysis of the electrodes used in these experiments indicates that the surfaces of the electrodes are significantly affected by the presence of an insulator. In particular, when a blue nylon insulator is inserted into the gap with either graphite or K-33 electrodes, silicon is found on the electrode surfaces after operation. This silicon may greatly affect the distribution of voltages at which the gap discharges (self-breakdown voltage distribution, V_{sb}). When the electrode surfaces were analyzed using SEM and XRF it was determined that the silicon deposits appeared on the surface in a nodule structure. The heights of these nodules is such that the local electric field may be enhanced causing the gap to discharge at a much lower voltage than expected, as observd in the V_{sb} distributions when blue nylon insulators are inserted into the gap. Also, the standard deviation of the V_{sb} distributions is much larger in these systems indicating an increased number of dropouts, (breakdown events with voltages which are considerably lower than the mean breakdown voltage).[1] Another example of how insulators affect the V_{sb} distribution is seen when lexan insulators are inserted into this gap with graphite electrodes in N_2 gas. In this system the V_{sb} distributions become narrower, giving a smaller standard deviation from the mean, than for the system without a lexan insulator present. The SEM micrographs of the graphite surface indicate that the surface is widely contaminated with some sort of insulating material, which may increase the probability of electron emission from the surface during discharges. This would cause a V_{sb} distribution with a lower mean value and possibly a smaller standard deviation. This did occur for this particular system relative to the system without an insulator present.

Insulators exposed in Mark II

Lucite insulators were inserted as cylinders concentric with the electrodes such that the insulator wall was located

approximately 6.25 cm from the actual discharge. When graphite was used as the electrode material with air flowing through the gap, the lucite insulator showed no visible (naked eye) signs of electrode coating. However, there were signs of crazing on the insulator in the region which was nearest to the discharge and small microparticles were embedded in the surface. Surface analysis of this insulator using ESCA showed no apparent change of the insulator from a virgin sample. The SEM micrographs show a very low density of microparticles on, and embedded in, the surface. A typical area, $1000 \times 1000\ \mu$, contained 39 particles, four of which measured to be 70–80 μ in diameter while the rest were about 10 μ in diameter. This is consistent with the ESCA data if the particles are assumed to be graphite. Since ESCA analyzes a large area (2 – 4 mm diameter) the low density of graphite particles would not produce a significant increase in the carbon percentage detected (71% for virgin lucite). Figure 9 shows a typical SEM micrograph. Note that the large particles appear to be stuck on the surface while most of the smaller ones are embedded in the surface as if the lucite melted on impact.

In contrast, the combination of a lucite insulator, flowing N_2 gas, and graphite electrode used for 50 000 shots in this spark gap produced a heavy coating of carbon on the lucite. Visual inspection shows the coating to be some tenths of a millimeter thick, with easily discernible cracks. This coating might eventually lead to gap failure through insulator flashover. The ESCA data show a significant increase in the carbon concentration of this sample (90%) compared to a virgin sample (71%), indicating a thick carbon coating on the surface.

Apparently, when flowing air is used with graphite electrodes the oxygen in the air combines with the carbon to create CO_2 and/or CO gas. Consequently, the lucite insulator then receives less of a carbon coating than in the N_2 gas, graphite electrode combination.

The ESCA data for the combination of lucite insulator, flowing N_2 gas, and copper-graphite electrodes shows the surface to be coated with a layer of copper which is partially oxidized. The insulator also has a large concentration of adsorbed nitrogen on it. The oxygen in the system probably comes from the disassociation of H_2O in the chamber.

Figure 10 is an SEM micrograph of a lucite insulator which was inserted into this spark gap with flowing air and copper-graphite electrodes. This micrograph shows that a granulated deposit has formed on the surface of the insulator. The ESCA data from this surface showed carbon present, but no copper. Either there is no copper on the surface (which is doubtful) or the surface is masked by a hydrocarbon layer.

A lucite insulator was inserted in this spark gap with K-33 electrodes and another lucite insulator was inserted in this spark gap with Elkonite electrodes. Elkonite and K-33 are both tungsten-copper composites in approximately the same relative ratios, (66% tungsten, 33% copper). For both of these combinations the gas used was flowing N_2 and both were exposed for 50 000 shots. Both insulators are coated with tungsten and copper. In both instances the copper is oxidized and the tungsten is in the form of tungsten-nitride. However, the amount of the two elements on the surfaces of these two lucite insulators is surprisingly different. For the lucite insulator used with K-33 electrodes there is a 6% concentration of copper and a 4% concentration of tungsten and for the lucite insulator used with Elkonite electrodes there is a 3% concentration of copper and a 3% concentration of tungsten. Similarly, the concentration of nitrogen on the surface of the insulators is different for the two electrode combinations, 33% and 10% for the K-33 and Elkonite, respectively. These differences can possibly be related to the way in which the two different materials are formed. The K-33 is formed by sintering tungsten and infiltrating molten copper, whereas in Elkonite tungsten and copper are mixed together in their molten form and allowed to cool.

Another example of electrode material coating the insulator arose in the combination of lucite insulator, stainless steel electrodes, and flowing N_2 gas. The ESCA data indicate that the surface is coated with a thin layer of Cr, Fe, and Ni in relative concentrations of 1.6%, 2.0%, and 1.4%, respec-

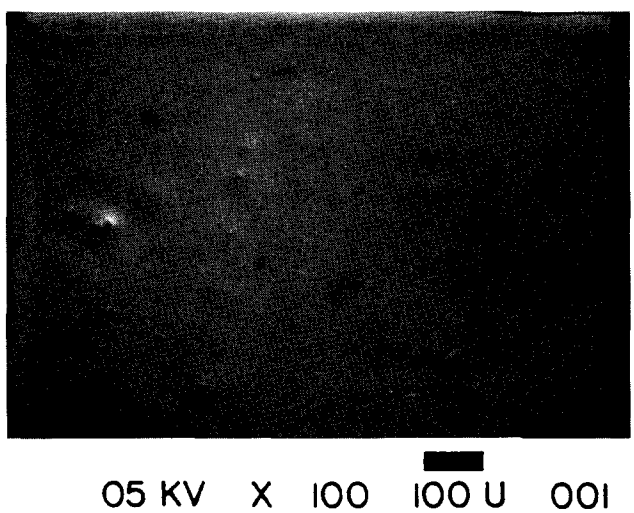

FIG. 9. SEM micrograph of a lucite insulator, which was inserted into Mark II with graphite electrodes in flowing air for 5000 shots.

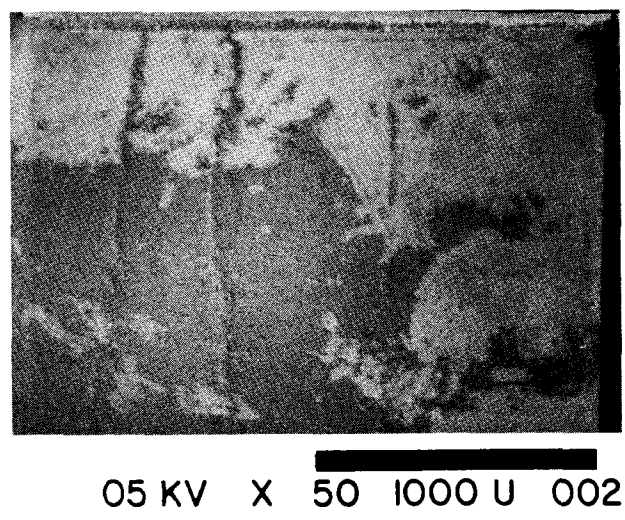

FIG. 10. SEM micrograph of a lucite insulator, which was inserted into Mark II with copper-graphite electrodes in flowing air for 5000 shots.

tively. However, visual inspection of the insulator showed no signs of coating or damage.

Insulators exposed in the surface discharge switch

Visual inspection, with the naked eye, of a boron nitride sample used as the substrate material in this switch showed signs of a considerable amount of erosion along its surface. The surface also had large black tracks on it which bridged the gap from one electrode to the other. In addition, there were large deposits of copper colored material in a line, along the insulator, where the edges of the electrodes were located. Surface analysis of this insulator, using ESCA, indicated that the actual boron nitride structure was not altered but did show a large concentration of carbon and oxygen on the surface, neither of which is a constituent of boron nitride. Perhaps, during the arc discharge plasma chemistry in the ambient atmosphere causes contamination of the surface with hydrocarbons.

When delrin, blue nylon, and lucite insulators were inserted as substrates in the surface discharge switch, each of them showed a large amount of erosion in the regions were discharges occurred. Surface analysis of these insulators using ESCA indicates that these insulators are all coated with a hydrocarbon layer. Whether this hydrocarbon layer is the result of the interaction of the arc with the surface or the interaction of the arc with the surrounding atmosphere, or both, is not known. The only noticeable difference between any of these insulators is that, on blue nylon, there is also a deposit of metallic copper and copper oxide whereas on lucite and delrin there is no detectable concentration of copper. The self-break field for all of these insulators is about the same, 9.7, 10.2, and 10.3 kV/cm for blue nylon, lucite, and delrin, respectively. This self-break field does not change very much for any of these insulators even after 10 000 shots.

SUMMARY

It is apparent from studies of insulators inserted into high voltage spark gaps that the insulator plays a significant role in the operation of the spark gap. Not only does the insulator serve as a high voltage standoff between the cathode and anode but it can also influence the behavior of the switch. The insulators become coated with electrode material and the electrodes become coated with insulator material. The coating of the insulators with electrode material may eventually lead to failure of the gap through insulator flashover. The coating of the electrodes with insulating material significantly affects the self-breakdown voltage statistics of the gap.

The studies with insulators used in spark gaps with graphite electrodes have shown that when air is used as a filler gas the insulators show no signs of gross carbon coating of the insulator. However, when N_2 gas is used as the filler gas the insulators receive a thick coating of graphite material which may eventually cause a surface flashover. The self-breakdown voltage for the system of graphite electrodes and lexan insulator in N_2 gas, however, shows a narrower distribution; consequently, there may be a tradeoff between insulator coatings and self-breakdown voltage distributions.

In the surface discharge switch it appears that plasma chemistry in the air close to the surface of the insulator keeps the insulator from playing a significant role in the discharge process. When lucite, lexan, delrin, and blue nylon insulators were used in the switch no significant differences in the breakdown voltage of the switch were observed. However, the amount of erosion seen on the surface of these insulators probably indicates that the surface must play some role in the flashover process.

ACKNOWLEDGMENTS

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