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Title Page

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H. C. Miller E. J. Furno	Improved Electrical Insulators for Vacuum	No. 78NDD002 Date 03/16/78
Title The Effect of Mn/Ti Surface Treatment on Voltage Holdoff Performance of Alumina Insulators in Vacuum		GE Class 1 Govt Class Unclassified
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Summary

The treatment of the surface of an alumina insulator with a Mn/Ti coating significantly increases its voltage holdoff capability. Insulators treated with this coating had vacuum holdoff voltages about 25% higher than did untreated insulators. During processing (quasimetallizing) the coating penetrates into the alumina, so is fairly insensitive to damage by abrasion or electrical breakdown. The quasi-metallized coating is also compatible with subsequent metallizing and brazing of the alumina insulator. We conclude that the coating (1) decreases the surface resistivity of the insulator, (2) decreases the insulator's secondary electron emission yield, and (3) makes the surface of the insulator dielectrically more uniform.

Key Words

Vacuum breakdown, Electrical insulators, Surface Coatings, Ceramics

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INTRODUCTION

The high voltage holdoff capability of ceramic insulators is a subject of considerable practical and theoretical interest. In general, the voltage holdoff capability of a solid insulator in vacuum is less than that of a vacuum gap of similar dimensions. The vulnerable area is the surface of the insulator, since its bulk voltage holdoff capability normally is better than that of a similar-sized vacuum gap.

Voltage breakdown along the surface of an insulator is usually initiated by electrons produced by field emission at the interface where the insulator meets the cathode.[1-4] This region where insulator, conductor and vacuum are in close proximity is frequently referred to as the triple junction. Some of these field-emitted electrons then impact upon the surface of the insulator producing additional electrons by secondary emission. Some of these secondary electrons will again strike the insulator surface, producing tertiary electrons. Continuation of this process results in a cascade along the surface of the insulator which develops into a secondary electron emission avalanche (SEEA).[3-6] This SEEA in turn leads to a complete breakdown. Such avalanches normally operate so as to produce a significant surface charge on the insulator surface. This surface charge can intensify the field at the triple junction and thus allow a subsequent breakdown at a lower applied voltage. The existence and importance of such surface avalanches and surface charging has been shown both theoretically and experimentally.[4-11]

Considerable work has been done on improving the voltage holdoff capability of insulators in vacuum. The techniques of shielding the triple junction,[2] shaping the insulator and electrodes so as to reduce the field at the triple junction,[12-13] or changing the angle of the insulator in order to prevent the secondary electrons from returning to the insulator surface,[14] have proven helpful. Changing the insulator angle in the reverse direction, so the secondaries return to the surface before they can build up enough energy to have a secondary yield greater than one, also has been shown to improve the voltage holdoff capability. The influence of these parameters is also shown in the observed strong effect of the insulator shape on the holdoff voltage.[3,12-18] The choice of insulator material has an obvious large effect upon the holdoff voltage, though different investigators disagree concerning the order of rank of various materials.[1-3,17-24] Roughening the surface of the insulator, especially the part of the surface near the cathode, raises the breakdown voltage significantly.[1] Application to the insulator surface of a more conductive (or semiconductive) coating also has been found helpful.[25-27]

Our goal at the General Electric Neutron Devices Department (GEND) was to improve the voltage holdoff capability of vacuum diodes. The plain hollow cylinder insulator geometry possesses the advantages of simplicity and compactness in such an application. Alumina is well suited as a material for use in ultra-high vacuum diodes, and improvements in alumina insulators offered the potential of application to more complicated devices. Therefore, we chose the goal of the work reported herein to be the improvement of the voltage holdoff capability of alumina insulators constructed in the form of hollow cylinders and used in vacuum diodes.

EXPERIMENTAL PROCEDURE

Previous investigators[25-30] have modified the surface of ceramics by the application of various coatings. Such coatings have improved the voltage holdoff capability of ceramic insulators. However, some coatings suffer from the deficiency that if the insulator does break down, the resultant damage can permanently decrease the insulator's voltage holdoff capability. Our goal was to find a coating method which would improve the voltage holdoff capability of ceramic insulators, but would not suffer such permanent damage from a breakdown. Other required characteristics were compatibility with the brazing process often used to attach the insulator to other components, and suitability for use in ultra-high vacuum apparatus.

In making metal-to-ceramic seals it is necessary to metallize the ceramic. A suitable formulation for use with alumina ceramics is a mix of Mo/Mn/Ti. When properly applied, a conductive layer of molybdenum is left on the surface. Brazing, soldering, etc., may then be done either directly to the molybdenum layer or to another metal which may be plated over the molybdenum. H. F. Zuhr, GEND, suggested that a coating for alumina ceramic insulators satisfying our requirements might be obtained by using a standard Mo/Mn/Ti metallize mix, but omitting the molybdenum. The resultant Mn/Ti mix would penetrate into the surface layer of the alumina reducing the resistivity, but would not form a metallic layer on the surface which would effectively short out the insulator and destroy its usefulness as an insulator. This suggestion proved most fruitful and was the basis on which the work described in this paper was begun.

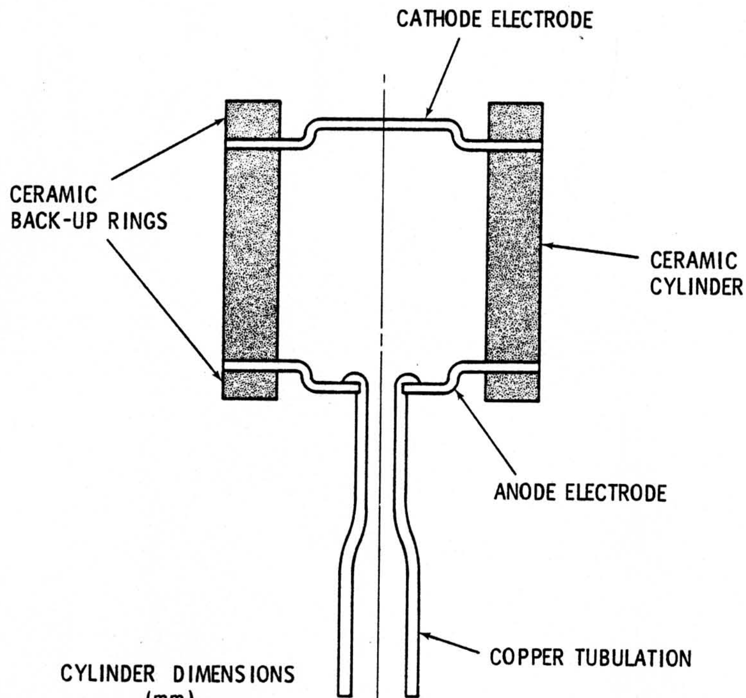
Two formulations have been used. The first was composed of four parts manganese and three parts titanium plus a suitable carrier.[31] This 4/3 ratio is that used in the standard metallize mix. Later a 7 Mn/1 Ti mix was used because it offered improved application characteristics. The coating was applied to the ceramic's surface, then fired in by heating at 1768 K (1495°C) for 45 minutes in an atmosphere of wet hydrogen. Since the processing is similar to that used in the normal Mo/Mn/Ti metallizing of alumina, differing only in the initial formulation of the coating mix, we refer to the process as "quasimetallizing" and the resultant modified alumina surface as a quasimetallized (QM) surface.

The basic alumina insulators used were fabricated by Diamonite Corp., and had a composition of 94% alumina, 1% Cr_2O_3, and 5% other ingredients.

The ceramic insulators tested were fabricated in the form of hollow cylinders. These cylinders were then assembled into vacuum diode test devices. Most such devices appeared as shown in Figure 1. No particular effort was made to shield the negative ceramic-metal-vacuum interface, (triple junction), since we were primarily interested in relative comparisons between insulators. The devices were evacuated, baked out at 450°C, and pinched off after cooling to room temperature. Pinch-off pressure was the mid 10^{-8} torr scale, or less.

The vacuum diodes were subjected to dc and pulsed high voltages. The pulsed voltage was a trapezoidal pulse (rise time of 3 μs, level top for 16 μs, fall time of 7 μs). In order to minimize external breakdowns, especially during the dc tests, the devices were encapsulated in an epoxy semi-rigid inspectable resin (SRIR) compound. This allowed us to be sure that most breakdowns occurred across the ceramic's vacuum surface. (External breakdowns were taken as yielding a lower limit for the insulator's vacuum holdoff voltage on that particular operation.)

Most of the testing was done with the vacuum diodes shown in Figure 1. This configuration has the electrically stressed vacuum surface on the inside of the cylinder. One set of vacuum diodes was constructed using the outside of the insulator as the vacuum surface, with breakdown across the inside non-vacuum surface being discouraged by filling the inside of the cylinder with an organic insulator, Adiprene[32] polyurethane rubber. This "inverted design" set of vacuum diodes also had the end electrode plates shaped to reduce the electrical stress at the triple junctions.



CYLINDER DIMENSIONS
(mm)

MODEL	O.D	I.D.	LENGTH
HVB I	19.15	14.94	14.28
HVB II	19.15	14.94	6.60
HVB III	18.80	13.97	19.05

TOLERANCE ± 0.12 mm

Figure 1. Geometry of HVB Vacuum Diode Test Devices

Three main groups of devices were tested: design HVB I , design HVB III, and the inverted design using the HVB III insulator. The cylinder dimensions of these designs are shown on Figure 1. Some devices of design HVB II were built and tested, but they seemed limited in their voltage holdoff performance more by the interelectrode gap than by the insulator used, and thus were not particularly useful in investigating differences between insulators

RESULTS

Most of the measurements were made on either untreated insulators or insulators quasimetallized with a 7 Mn/1 Ti mix. We found that best results were obtained when both inside and outside surfaces of the insulator were treated. Insulators quasimetallized with a 4 Mn/3 Ti mix also showed improved voltage holdoff performance over plain alumina, but sufficient numbers have not yet been tested to yield quantitatively reliable data.

The results of our tests are shown in Table 1. The pulsed voltage values are the voltage levels to which the devices could be conditioned. The dc voltage values are the initial dc level at which breakdown occurred. Dc holdoff was defined in this manner because many of the devices dc conditioned to values in excess of 190/200 kV, thus attaining holdoff voltages beyond the capability of our equipment. As may be seen, the quasimetallized insulators show improved voltage holdoff characteristics as compared to the untreated insulators. Our experience indicates that the differences are real and significant, though the large experimental scatters and modest sample sizes (28 HVB I's, 12 HVB III's, and 21 inverted design devices), result in only the inverted design group pulsed voltage results and the HVB III group dc voltage results being statistically significant (at the 95% and 80% levels, respectively).

Table 1. Effect of Surface Treatment on Voltage Holdoff of Alumina Insulators

	Insulator Surface			
	Plain		QM 7:1	
	Holdoff Voltage (kV)			
Group	Pulsed	dc	Pulsed	dc
HVB I	72 ± 19	122 ± 49	94 ± 34	157 ± 47
HVB III	75 ± 30	144 ± 28	96 ± 35	186 ± 6
Inverted Design	137 ± 11	-	174 ± 7	-

Histograms of the pulsed voltages attained by the two largest groups (HVB I and inverted) are given in Figures 2 and 3 as support for our conclusion.

The higher pulsed holdoff voltages for the inverted design may be partially caused by the vacuum wall being the exterior, but probably mainly reflect the emphasis in this design on minimizing the electrical stress at the ceramic-metal-vacuum interface (the triple junction).

The percentage increases in voltage holdoff produced by quasi-metallizing are given in Table 2. The excellent agreement between the separate sets of measurements is probably somewhat fortuitous, considering the scatter in the experimental data, but one can reasonably conclude that the 7 Mn/1 Ti quasimetallized ceramic shows an increase of 25% to 30% in voltage holdoff over the untreated ceramic.

Table 2. Increases in Voltage Holdoff of Quasimetallized (7 Mn/1 Ti) Insulators Over Untreated Alumina Insulators

	Holdoff Voltage Increase	
Test Group	Pulsed	dc
HVB I	+31%	+29%
HVB III	+28%	+29%
Inverted Design	+27%	-

Some interesting measurements were made on other characteristics of the quasimetallizing. The quasimetallize penetration depth was measured by J. E. Pearson, (GEND), who used electron beam scanning across the insulator wall thickness to obtain relative measurements of Mn (Ti) intensities. He found that the QM coating penetrated into the alumina to depths of 0.7 to 1 mm. The concentrations of Mn and Ti decreased smoothly from the surface inwards.

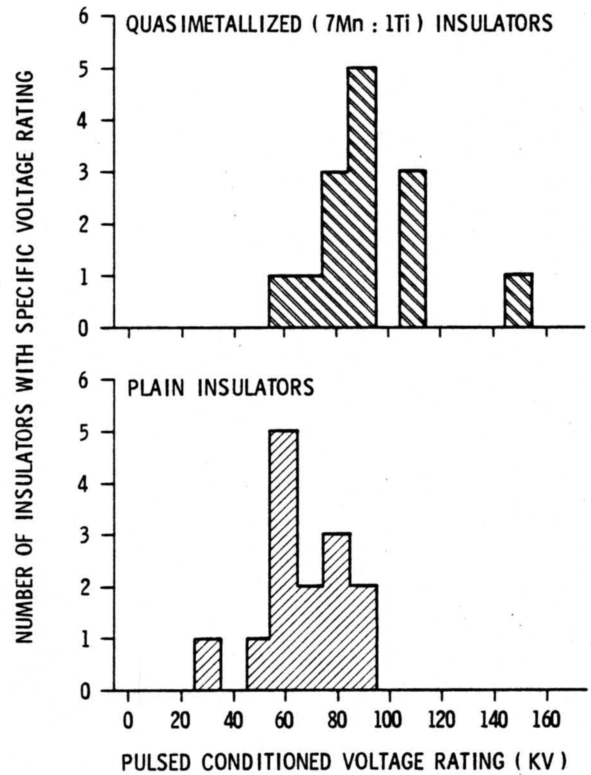


Figure 2. Histograms of Maximum Holdoff Voltages to Which HVB I Vacuum Diodes Conditioned When Subjected to Trapezoidal Voltage Pulses (3/16/7 μs)

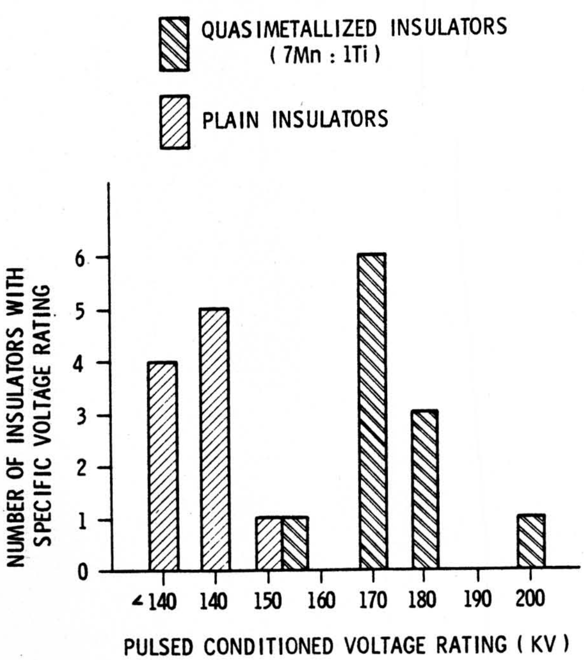


Figure 3. Histograms of Maximum Holdoff Voltages to Which Inverted-Design Vacuum Diodes Conditioned When Subjected to Trapezoidal Voltage Pulses (3/16/7 μs)

The QM treatment significantly lowers the resistance of the insulator. The normal room temperature resistivity of 94% alumina is too large to be directly measured by our equipment, but extrapolation of measurements at higher temperatures indicates it to be at least 10^{18} ohm-cm. The quasimetallized insulators have room temperature resistances in the range 10^{14} to 10^{15} ohm. Measurements of resistances at various temperatures indicate that the resistances of the plain and the quasimetallized insulator follow the normal equation for non-metals:

$$R = A\, e^{B/T} \quad (\Omega)$$

The resistivity of the untreated ceramic (94% alumina) is best expressed as a volume resistivity, so the appropriate parameter values are: $A = 2 \times 10^6$ (70% confidence limits are 5, 0.7×10^6) ohm-cm and $B = 12900 \pm 600$ K. The QM ceramic's resistivity is probably best expressed as a surface resistivity, with values of $A = 9 \times 10^6$ (14, 6) ohm/square and $B = 6600 \pm 70$ K. Thus the effect of quasimetallizing on the resistivity of an alumina insulator is to simultaneously decrease the exponential factor (from 1.11 eV to 0.57 eV if units of kT are used), and increase the pre-exponential factor, but the overall effect is to significantly lower the resistivity of the ceramic at near room temperatures.

ANALYSIS

We conclude that the application of quasimetallize coating to an alumina ceramic insulator significantly increases the voltage holdoff capability of the insulator. The experimental confirmation of this improvement is apparent, the exact mechanism(s) whereby the quasimetallizing operates is less obvious. The lowered surface resistivity is undoubtedly important in alleviating the possibility of charge buildup on the insulator surface over times of the order of seconds or minutes. However, the surface resistivity is too high to allow much charge bleedoff over the milli to microsecond times involved in much pulsed voltage work. Still, the lowered surface resistivity probably helps improve the evenness of charge distributions appearing on the surface during these shorter time intervals and thus reduces the resultant electric stress on the insulator. The quasimetallizing treatment also helps by reducing secondary electron emission from the insulator.[33] A probable significant factor in the increase of voltage holdoff is the improvement of dielectric uniformity of the insulator surface.[34]

Our work is in reasonable agreement with the work of other investigators. Fryszman, Strzyz, and Wasinski[25] showed that applications of a layer of semiconducting Fe_2O_3 to the surface of a ceramic rod (especially near the negative electrode) significantly raised the vacuum breakdown voltage. They also pointed out the importance of having a surface with a secondary emission coefficient <1.

Cross and Sundarshan[26] investigated the effect of cuprous oxide coatings on surface flashover of alumina insulators in vacuum. They found that the Cu_2O coating significantly improved the voltage holdoff capability of the alumina for pulsed (μs) voltages, but had no effect on the flashover strength for dc and 60 Hz voltages. The Cu_2O coating also eliminated the conditioning effect. They postulated that the improvement produced by the Cu_2O coating was due to a reduction in the secondary electron emission yield. The lack of conditioning with a Cu_2O surface was attributed to the elimination of patch charges (uneven concentrations) on the surface by the relatively low (10^6 ohm-cm) resistivity of the Cu_2O.

Sundarshan and Cross[27] also investigated chrome oxide coatings. A coating of green Cr_2O_3 significantly improved the voltage holdoff capability of alumina insulators subjected to pulsed (μs), 60 Hz or dc voltages. Unlike the Cu_2O coating, the Cr_2O_3 coated cylinders showed significant conditioning for both dc and 60 Hz voltages, though no conditioning was apparent for pulsed voltages. Their coated specimens showed a surface resistivity of ~10^{11} ohm/square. They measured the secondary electron coefficient for Cr_2O_3, finding

$$\sigma_{max} = 0.98.$$

This would preclude the insulator surface charging positively and thus minimize secondary electron emission avalanches. Our results differ from those of Cross and Sundarshan in that the quasimetallized insulators showed significant conditioning for pulsed voltages, more so than for dc voltages.

SUMMARY AND CONCLUSIONS

The application of a 7 part Mn/1 part Ti coating to the surface of a 94% alumina insulator significantly increases its voltage holdoff capability. Insulators treated with this coating (quasimetallized) conditioned, when subjected to pulsed voltages, to vacuum holdoff voltages about 25% higher than untreated insulators. The quasimetallized insulators also withstood 25% higher dc voltages before breaking down initially than did untreated insulators. Equipment limitations precluded our measuring final conditioned dc voltages. We concluded that the coating probably (1) decreased the surface resistivity of the alumina, (2) decreased the alumina's secondary electron emission yield, and (3) made the surface of the insulator dielectrically more uniform.

During processing, the coating penetrates into the alumina, so is fairly insensitive to damage by abrasion or electrical breakdown. This process possesses the distinct advantage of being compatible with subsequent metallizing of the alumina insulator and its further assembly (by brazing, etc.) into various devices incorporating metals and other ceramics.

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