Part II

PHYSICAL PROPERTIES EVALUATION OF COMPOUNDS AND MATERIALS

Dielectric Strength and Other Properties of Nitrogen Trifluoride

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GASEOUS dielectrics have been used successfully in transformers and other high voltage electrical equipment. Sulfur hexafluoride (*1–3*) has a relatively high dielectric strength and may be used as a gaseous dielectric, without condensing, under moderate pressures at low temperatures. It might be advantageous to replace sulfur hexafluoride (boiling point −63.7° C.) with a compound which had equivalent dielectric properties, but a lower boiling point. This would permit the use of higher pressures at low temperatures in electrical equipment without causing condensation of the dielectric gas. Some other dielectrics (*6, 14*) have adequate dielectric strengths, but also higher boiling points.

Nitrogen trifluoride has a relatively low boiling point (−125° C.) and critical temperature (−39.3° C.) (*5*) and could be expected to possess high dielectric strength because of its fluorine content. Therefore, a study of the dielectric strength and some other properties of nitrogen trifluoride was undertaken to determine its suitability as a gaseous dielectric.

The oxidizing properties of nitrogen trifluoride (*4, 8, 11*) are similar to those of oxygen—that is, it does no react rapidly at normal temperatures and pressures with most materials. Combustion of an oxidizable material with nitrogen trifluoride can be initiated, however, by the application of an electrical spark or by heat.

EXPERIMENTAL

Preparation of Nitrogen Trifluoride. The gas was prepared by a procedure similar to that described by Ruff (*9, 10, 12, 13*) and Pierce (*7*). The electrolysis was conducted in a Monel cell with a nickel anode operating between 400 and 1000 amp. per square foot. The electrolyte was ammonium bifluoride plus 6.1 % hydrogen fluoride. The product was purified by passing through hydriodic acid, a 10 % sodium hydroxide solution, a drying tube, and a low temperature filter. The yield of purified nitrogen trifluoride was 53 grams, or 64 % when based on the total number of coulombs which passed through the cell. The molecular weight of the product was found from gas density to be 71.1 ± 0.5 grams in agreement with the calculated value.

Mass spectrographic analysis showed that the product had the following identifiable impurities.

N_2	1.55 %
O_2	0.01
N_2O	0.07
NH_2F, SiF_4, and component of mass 54	1

The mixture of hydrogen and nitrogen trifluoride which results during preparation is highly explosive. Consequently, the cell was modified by the addition of a Monel diaphragm which prevented hydrogen from mixing with the product. As a result, the yield was reduced from 64 % to 30 to 40% of the theoretical. There was evidence, however, that the yield was high at first and then decreased as electrolysis proceeded.

Dielectric Strengths. A glass dielectric breakdown cell with electrodes consisting of two nickel hemispheres one-half inch in diameter was used. A 15,000-volt neon sign lighting transformer supplied the 60-cycle potential which was increased manually until breakdown. The test results compare air, sulfur hexafluoride, and nitrogen trifluoride (Figure 1).

Further tests were carried out in an acrylic resin test cell. The cell was a cylinder 4 inches long with an inside diameter of 2 inches. The electrodes were copper spheres 1 inch in diameter and were mounted through acrylic resin end plates. The gap between the spheres was continuously adjustable. The cell was provided with a Bourdon tube-type pressure gage. The test voltage was supplied by a 100,00-volt, 60-cycle alternating current transformer which allowed the voltage to rise at a rate of 300 volts per second from zero until breakdown was reached, at which time the potential was automatically removed. The dielectric breakdown potentials plotted against gap lengths are shown in Figures 2 to 4.

Heat Transfer Properties. To compare the heat transfer properties on nitrogen trifluoride, the following apparatus was prepared. A Type VG-1A Pyrex glass ionization gage tube which had an inside diameter of 32 mm. and a volume at 0° C. of 68.0 ml., was modified by the addition of of a 2-mm. stopcock. A tungsten helix, of 0.38-mm. wire approximately 10 mm. in diameter and 16 mm. long, was located at the center of the tube. This helix had a resistance at 0° C. of 0.112 ohm and at 100° C. of 0.171 ohm. Comparative heat transfer data were obtained after the cell, which had been cooled to 0° C., was filled with the gas to be tested and the stopcock was closed. Sufficient direct current was passed through the helix until its resistance was 0.171 ohm, as determined from measurements of current and voltage. The power necessary to maintain the helix resistance at 0.171 ohm under a pressure of 0.15 micron of mercury was 0.25 watt. This figure represented heat losses other than heat transfer through the gas and was subtracted from the total power values calculated from the measurements of current and voltage to obtain the power dissipated by the gases. The relative heat transfer

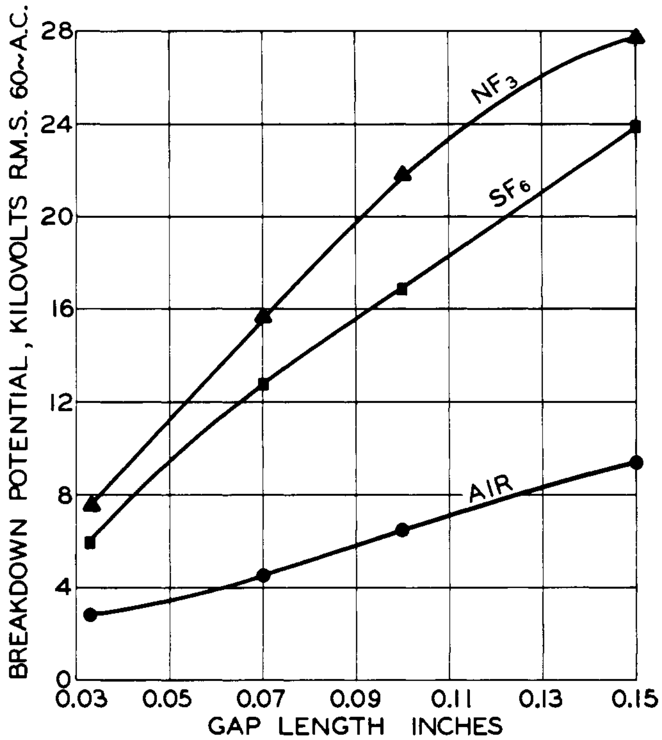

Figure 1. Dielectric breakdown of air, sulfur hexafluoride, and nitrogen trifluoride at atmospheric pressure between nickel hemispheres, one-half inch in diameter

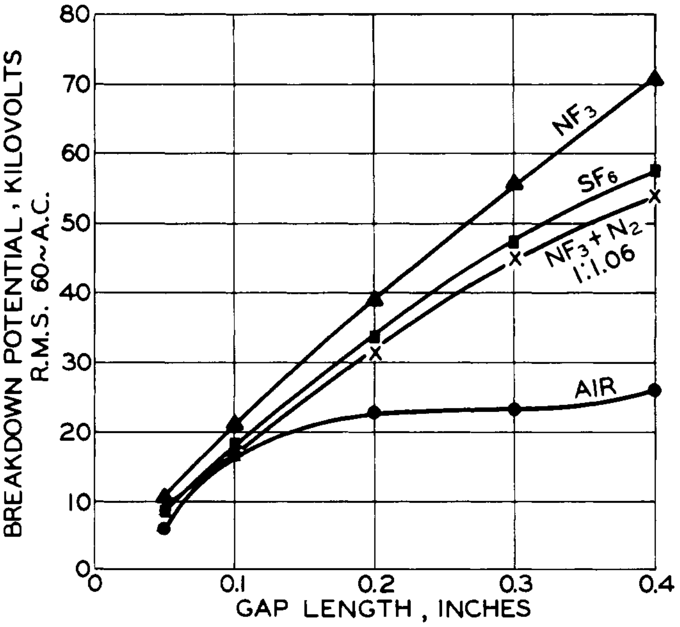

Figure 2. Dielectric breakdown of air, sulfur hexafluoride, nitrogen trifluoride and a nitrogen–nitrogen trifluoride mixture at atmospheric pressure, between copper spheres, 1 inch in diameter

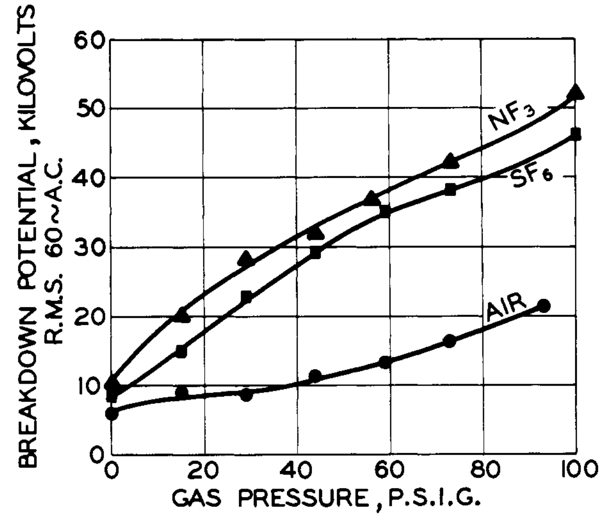

Figure 3. Dielectric breakdown of air, sulfur hexafluoride, and nitrogen trifluoride at various pressures, between copper spheres, 1 inch in diameter, 0.05-inch gap

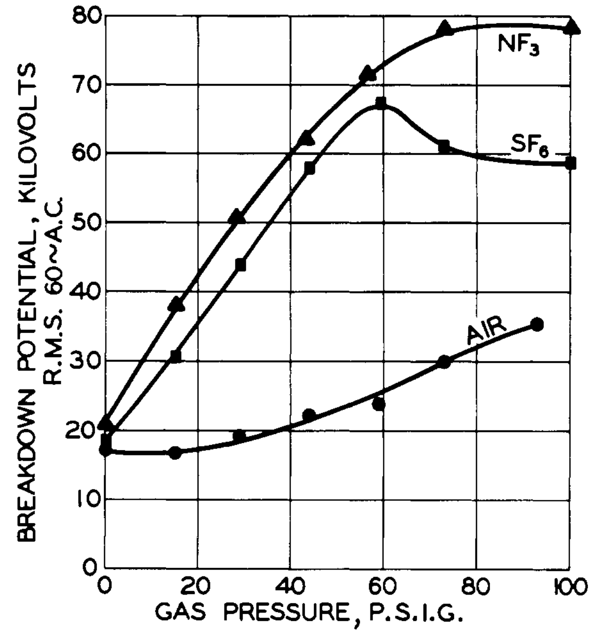

Figure 4. Dielectric breakdown of air, sulfur hexafluoride, and nitrogen trifluoride at various pressures, between copper spheres, 1 inch in diameter, 0.10-inch gap

Table 1. Heat Transfer Data for Various Gases

Gas Tested	Gas Pressure at 0° C., Atm.	Relative Heat Transfer Properties
Dry air	1	1.00
Dry air	2	1.37
N_2	1	1.07
N_2	2	1.38
SF_6	1	1.22
SF_6	2	1.54
NF_3	1	1.14
NF_3	2	1.45

properties were calculated from the power dissipated by the gases by making the power dissipated by air at 1 atm. (1.62 watts) equal to 1 (Table I).

Chemical Reactivity. The reaction of nitrogen trifluoride with water was investigated. A 300-ml. borosilicate glass bulb with a 2-mm. stopcock sealed to it was evacuated and filled with 23 ml. of water and 0.40 gram of nitrogen trifluoride. The bulb was then heated to 100° C. and held at that temperature for 7 days. The partial pressure of nitrogen trifluoride at that temperature was calculated to be 470 mm. The reaction was assumed to be

$$NF_3 + 2H_2O = 3HF + HNO_2$$

The amount of acid which was formed was found by titration with sodium hydroxide. The percentage of nitrogen trifluoride which had reacted was calculated on the basis of the equation and was 1.5%.

The effect of nitrogen trifluoride on paper was also investigated. Several pieces of Whatman No. 30 filter paper were put into a borosilicate test cell, 32 mm. in internal diameter and 110 mm. long, which was then evacuated and filled with nitrogen trifluoride to a pressure of 560 mm. of mercury. For comparison purposes, a few pieces of paper were put into an open test tube. Both the test cell and the test tube were placed in an oven at 100° C. for 40 days. Examination of the paper from the nitrogen trifluoride tube showed that it had turned brown and had become brittle. The paper in the open test tube appeared unaffected.

DISCUSSION

The dielectric strength of nitrogen trifluoride appears to be substantially greater than that of sulfur hexafluoride. The increase in strength is more noticeable in nonuniform as compared with uniform fields. At a narrow gap spacing (Figure 2) and, therefore, relatively uniform field, the superiority of nitrogen trifluoride over sulfur hexafluoride remains almost constant. As the gap spacing is increased (Figures 3 and 4), the field becomes more nonuniform, and the superiority of nitrogen trifluoride over sulfur hexafluoride is much greater. Although an increase in pressure appears to add to the effect of nonuniform fields, it is apparent even at atmospheric pressure (Figure 5). Since nitrogen trifluoride can be used at higher pressures than sulfur hexafluoride without liquifying, its already superior performance at high pressures becomes even more impressive.

The ability of nitrogen trifluoride to transfer heat is 5 to 6 % poorer than that of sulfur hexafluoride. It is, however, 8 to 14 % better than air, so that it should function as a satisfactory coolant in electrical equipment.

The chemical reactivity at moderate temperatures of nitrogen trifluoride must be taken into consideration in any intended application in electrical equipment. The equipment would have to be maintained moisture-free, and conventional organic insulation could not be used. If it were used, the nitrogen trifluoride would probably slowly react with such insulation. The impurities in the gas probably would not cause the reactions at room temperature because of the chemical method of purification employed in the preparation. Most important, a spark inside the equipment could ignite the insulation, causing a fire. Therefore, in any proposed application, the reactions of nitrogen trifluoride with the materials of construction of the unit must be tested.

SAFETY

The physiological characteristics of nitrogen trifluoride have been discussed (11). Apparently, greater danger exists from crude nitrogen trifluoride than from the purified material.

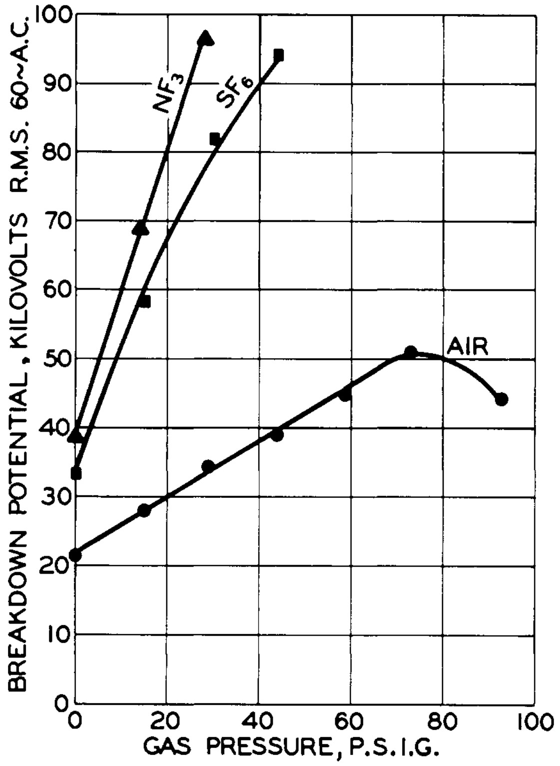

Figure 5. Dielectric breakdown of air, sulfur hexafluoride, and nitrogen trifluoride at various pressures, between copper spheres, 1 inch in diameter, 0.20-inch gap

Nitrogen trifluoride is a relatively stable gas at room temperature and has been stored in this laboratory at pressures up to 600 p.s.i.g. in small steel cylinders without incident.

Valves, hoses, and other equipment which were recommended for handling oxygen were used for nitrogen trifluoride with no apparent ill effects.

While experimental work with nitrogen trifluoride proceeded in this laboratory without an unsafe condition developing, additional studies should be made to determine the extent of the hazards involved.

ACKNOWLEDGMENT

The suggestion of John Dzimianski that nitrogen trifluoride might have dielectric strength was instrumental in initiating this work and is greatly appreciated. The assistance of J.F. Britt with heat transfer measurements is gratefully acknowledged.

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