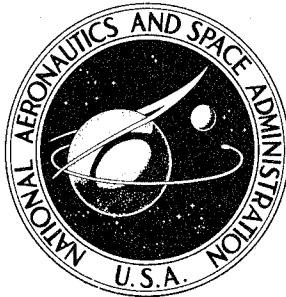


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**OUTGASSING CHARACTERISTICS OF
AN EPOXY-IMPREGNATED MAGNET COIL**

by Ronald J. Schertler

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DEPARTMENT OF DEFENSE
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ABSTRACT

Outgassing tests as a function of temperature were conducted using an epoxy-impregnated magnet coil. Glass tape impregnated with epoxy resin provided the electrical insulation between the conductors. Outgassing data were taken by measuring the rate of pressure rise due to the magnet coil heating in a closed vacuum system. The outgassing rate increased exponentially from 3.05×10^{-8} torr-liter per square centimeter-second at an epoxy temperature of 24°C to 9.0×10^{-7} torr-liter per square centimeter-second at 53°C . The coil assembly had a higher outgassing rate at temperatures above 30°C than small disk-shaped samples of the same epoxy.

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SUMMARY

Outgassing tests as a function of temperature were conducted using a 41.2-centimeter outside diameter by 2.86-centimeter wide epoxy-impregnated magnet coil. Insulation was provided by an epoxy glass tape (National 8A840) and an impregnating epoxy resin (National 2A566). Outgassing data were taken by measuring the rate of pressure rise in a sealed evacuated chamber containing the magnet coil assembly. The starting pressure was 1.0×10^{-6} torr. Measurements showed that the outgassing rate increased exponentially from 3.05×10^{-8} torr-liter per square centimeter-second at an epoxy temperature of 24°C to 9.0×10^{-7} torr-liter per square centimeter-second at 53°C . The coil assembly had a higher outgassing rate at temperatures above 30°C than small disk-shaped samples of the same epoxy.

INTRODUCTION

The thermal outgassing measurements described herein were taken as part of a magnet coil design program for an electron injection research experiment. The research experiment is intended to study the behavior of low-energy electrons in various trapping magnetic fields. A principal requirement of the experiment is that the background pressure must be less than 1.0×10^{-7} torr in order to minimize electron scattering effects caused by the residual background gases.

Magnet coil design and fabrication considerations indicated a water-cooled coil, wound from hollow rectangular copper tubing and insulated with epoxy-impregnated glass tape, was required. Because 14 of these coils would be used in the experiment and might be operated at elevated temperatures, it was anticipated that the combined coil outgassing rate could compromise the facility base pressure that otherwise might be obtained. The elevated temperature would occur because of resistance heating of the coils. Therefore, the outgassing characteristics of the coils had to be determined.

A limited amount of data on various epoxies is available in the literature (refs. 1

to 5). However, because the data were usually obtained from very small simple samples under carefully controlled conditions, it was not certain that they would be adequate for predicting the performance of the complex, multicomponent configuration. This experiment was consequently performed to obtain the necessary data.

EXPERIMENTAL APPARATUS AND PROCEDURE

The magnet coil, consisting of 14 turns of copper tubing in a pancake arrangement, is shown in figure 1. The conductor consists of 0.93-centimeter by 1.25-centimeter rec-

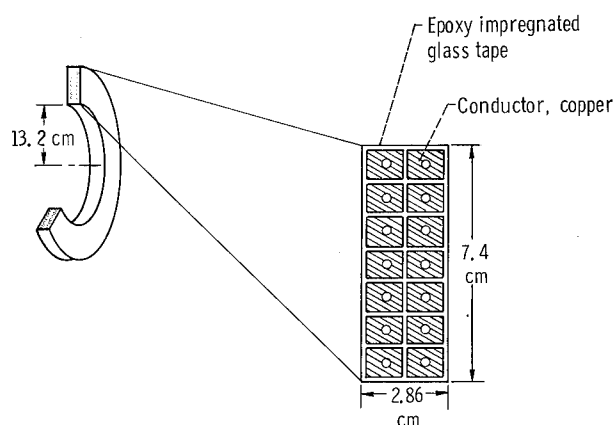


Figure 1. - Sample magnet coil.

tangular, oxygen-free copper tubing with an internal channel 0.445 centimeter in diameter. The tubing was wrapped twice with 0.0203 centimeter thick National 8A840, "B" staged epoxy glass tape before the coil was formed to provide inter-turn insulation. The term "B" staged refers to a partially cured state of the epoxy in which the epoxy is still flexible and workable. Insulation of the total assembly from ground was provided by wrapping the exterior of the formed coil with a double layer of the same tape. The entire coil was vacuum/pressure impregnated with National 2A566 epoxy resin (ref. 6). In this process, vacuum pumping was used to evacuate all trapped air from voids in the epoxy insulation. These voids were then subsequently filled with epoxy resins by a pressurized application. Vacuum/pressure impregnation provided a solid, homogeneous system. After the coil had been impregnated, it was clamped and cured in a metal jig.

In general, "B" staged epoxy glass tapes are suitable for electrical insulation up to 130° C and when used with epoxy impregnants are suitable up to 155° C bulk temperature (ref. 6).

The magnet coil was mounted in a bell jar vacuum facility evacuated by a liquid-nitrogen-cold-trapped 15.2-centimeter diameter oil diffusion pump, backed by appropriate mechanical pumps. Figure 2 is a photograph of the magnet coil mounted in the bell jar. The total internal volume of this facility was 120 liters. The ultimate vacuum attainable with the coil mounted in the test chamber was 7.0×10^{-7} torr. The pressure was monitored with a Bayard-Alpert type ionization gage shielded from the magnetic field.

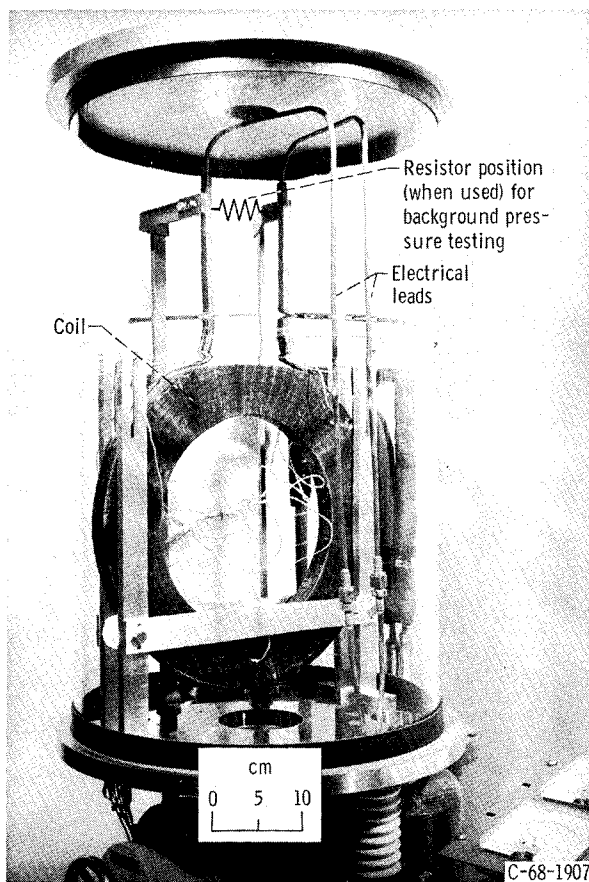


Figure 2. - Magnetic coil in bell jar for outgassing tests.

Six thermocouples located at various positions on the epoxy insulation were used to measure the epoxy temperature. Two additional thermocouples were located on the copper coil leads. All the epoxy insulation thermocouple readings were within 3°C of one another for each thermal run.

Outgassing rates were determined by measuring the rate of pressure rise dP/dt in the closed or blanked-off, evacuated bell jar containing the sample coil. Assuming that the perfect gas law applies, the outgassing rate is

$$Q(t, T, P) = \frac{dP}{dt}(t, T, P) \frac{V}{A} \quad (\text{torr-liters/cm}^2\text{-sec})$$

where t is the time the system has been under vacuum, T is the temperature of the epoxy insulation, P is the pressure at which dP/dt was measured, V is the bell jar volume, and A is the surface area of the coil insulation. The bell jar volume was not corrected for the volume occupied by the magnet coil and coilholder which amounted to approximately 4 percent of bell jar volume.

The first step in using the method was to determine the background outgassing rates of the complete vacuum system without the magnet coil. This system included the bell jar, coilholder, thermocouple wires and insulation, and electrical leads for the coil. This auxiliary material was placed inside the bell jar and the chamber was pumped down to a pressure of approximately 1×10^{-6} torr. At this point the test section was blanked off from the pumping system and the pressure as a function of time was recorded. When the pressure in the chamber reached 1.0×10^{-3} torr, the chamber was opened to the diffusion pump until the next measurement.

The tests were repeated for various electrical lead temperatures. The copper electrical leads (without the magnet coil in place) were connected through a resistor (fig. 2) and heated by a few hundred amperes of current. Iron-constantan thermocouples with teflon insulation were used to measure the temperature of the electrical leads near the resistor. These background measurements were taken after approximately 24 hours of vacuum exposure. For lead temperatures above 38°C the glass bell jar was heated slightly with a heat blower in an attempt to simulate the heat effects of the coil radiating to the bell jar and freeing trapped or absorbed gas. No significant differences were noted in the pressure rise against time curve between runs with and without bell jar heating.

After the background measurements were completed, the magnet coil was mounted into the bell jar and instrumented with the same type of iron-constantan thermocouples used in the background measurements. The coil was exposed to vacuum for approximately 6 hours before outgassing measurements were attempted.

To determine the outgassing rate of the epoxy insulated coil, pressure as a function of time was recorded in a similar manner as before with the coil in place in the bell jar. Data were plotted and all curves were corrected for background outgassing. Since the volume (120 liters) of the bell jar and the approximate surface area ($2.6 \times 10^3 \text{ cm}^2$) of the coil insulation were known, the outgassing rate then could be computed from a slope drawn to a corrected curve of pressure against time. To avoid the effects of adsorption pumping from the bell jar surfaces, the outgassing rates were computed at pressures between 1.0×10^{-4} and 4.0×10^{-4} torr. The process was repeated and the outgassing rates were computed for a series of coil insulation temperatures.

RESULTS AND DISCUSSION

The blanked-off, or closed-chamber technique for measuring outgassing rates has advantages and disadvantages when compared to other techniques such as a method in which the sample is placed in a chamber connected to a high vacuum pump through an orifice (ref. 2).

The main advantage of the closed chamber method is that it involves simple determinations of the rate of pressure rise and of volume and area. Other advantages include the measurement of outgassing rates over both a wide range of pressure in reasonable lengths of time and at any preselected value of system pressure regardless of length of time the sample has been under vacuum (ref. 2).

The prime disadvantage of the closed chamber technique is that adsorption of the outgassed products on the walls of the vacuum chamber can be a source of error below 10^{-4} torr. However, this type of error is easily avoided by making dP/dt determination at a higher pressure level. Generally, this adsorption effect is greatest for low starting pressures in the test chamber and decreases in relative importance as the closed chamber pressure rises. Ion gage pumping can be a source of error with all methods. However, for these tests, ion gage pumping is negligible.

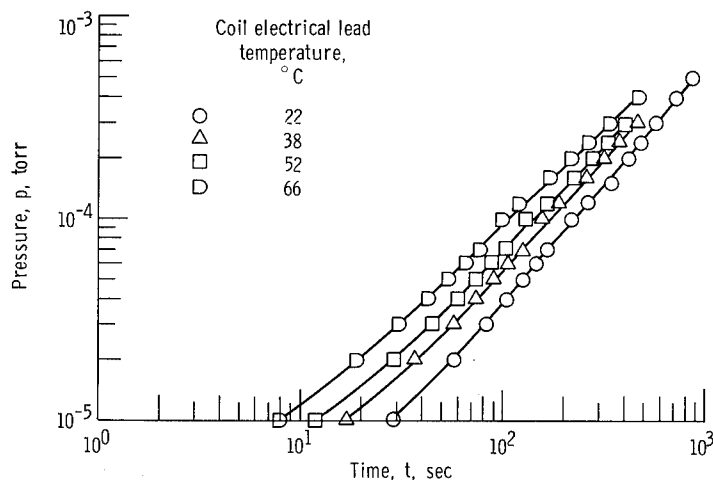


Figure 3. - Background pressure as function of time for range of coil electrical lead temperatures.

Background pressure as a function of time is shown in figure 3 for coil lead temperatures between 22° and 66° C. These curves were used to correct the subsequent data taken with the coil at different temperatures. Figure 4 is a graph of the initial outgassing rate of the magnet coil as a function of time both before and after a heat treatment. This initial test was performed on the coil as received from the manufacturer. Outgassing

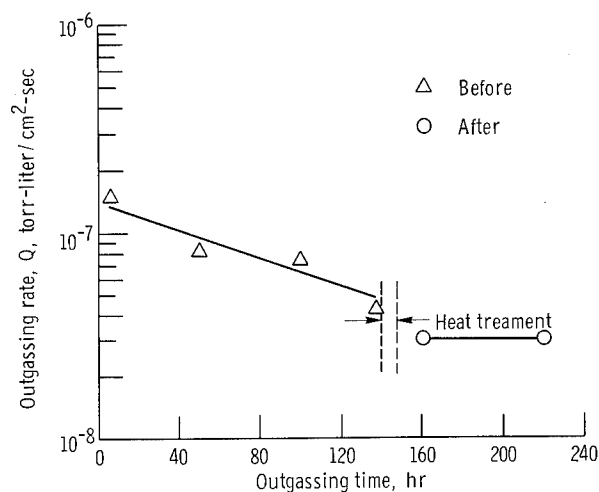


Figure 4. - Room-temperature (24° C) outgassing rate of magnet coil as function of time before and after heat treatment.

measurements were made at an epoxy insulation temperature of 24° C (room temperature). The outgassing rate after 6 hours of pumping was 1.5×10^{-7} torr-liter per square centimeter-second. After 138 hours of continuous vacuum, at a bell jar pressure in the low 10^{-6} torr range, the outgassing rate decreased to 4.14×10^{-8} torr-liter per square centimeter-second. The initially high outgassing rate was attributed to dissolved gases which were present in the liquid form of the original components prior to epoxy cure by the coil manufacturer. In an attempt to remove any volatile material still remaining in the coil insulation the coil was heated in vacuum for approximately 10 hours at temperatures above 100° C. After this vacuum heat treatment and before the coil was exposed to the atmosphere, the outgassing rate of the coil assembly was 3.05×10^{-8} torr-liter per square centimeter-second (fig. 4). However, even after the coil assembly subsequently was exposed to atmosphere, the outgassing rate remained at this low value.

Figure 5 shows the pressure of the bell jar containing the magnet coil as a function of time for epoxy temperatures between 24° and 53° C. The coil was heated by passing increasing amounts of current through it. For temperatures much above 53° C, the pressure rise was faster than could accurately be recorded with the system. No attempt was made to obtain data below room temperature (24° C).

Before each run the bell jar was evacuated and the temperature of the epoxy insulation was stabilized for approximately 10 minutes. However, as the temperature of the epoxy insulation was increased above room temperature, it was no longer possible to evacuate the bell jar to 1.0×10^{-6} torr in a short time. For these temperatures, the starting pressure was between 4.0×10^{-6} and 8.0×10^{-6} torr.

Figure 6 shows the outgassing rate of the epoxy insulation as a function of epoxy temperature for the magnet coil. Also shown on the graph are the thermal outgassing rates

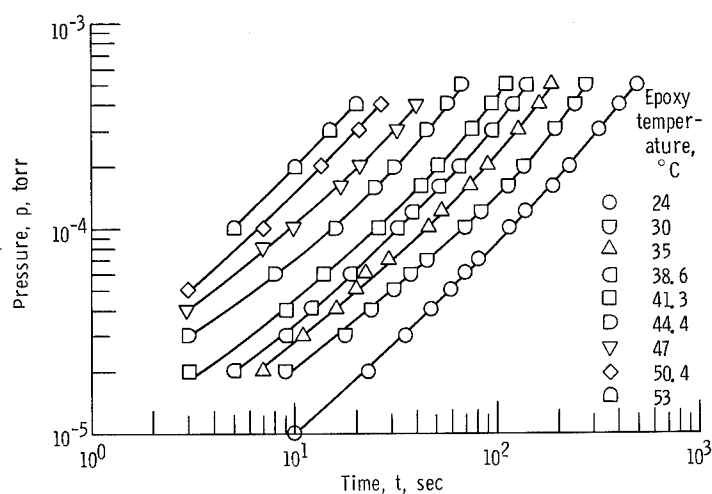


Figure 5. - Pressure as function of time for range of epoxy insulation temperatures.

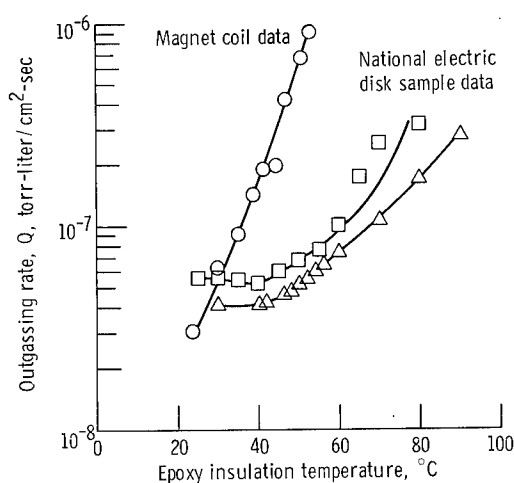


Figure 6. - Outgassing rate as function of temperature for national 2A566 epoxy.

of two small disk shaped epoxy samples, 0.635 centimeter thick by 8.9 centimeters diameter, of the same National epoxy resin as used in the magnet coil. These samples were fabricated and tested under controlled conditions, and the data were provided by Mr. B. Zimmerman of the National Electric Coil-Division of McGraw Edison, Columbus, Ohio. The slope of the thermal outgassing rate of the magnet coil is steeper than that of the sample disks. Also, the outgassing rate of the coil assembly is greater than that of the sample disks for temperatures greater than 30° C. The exact reason for these differences is not known. It is possible that different heating methods and epoxy surface conditions may be contributing factors.

Outgassing rates for metals have been shown to be largely dependent on the surface conditions of the material (ref. 4). Outgassing rates are higher for metals with comparatively rough surfaces. Similar differences may also contribute to the observed difference in the outgassing rate between the magnet coil assembly and the sample disks. The disk-shaped samples had very smooth surfaces. The magnet coil, on the other hand, had a comparatively rough surface, thus the true coil surface area may have been much greater than the macroscopic value used. Difference in sample heating methods may also contribute to the observed differences in the outgassing rates since the outgassing of the epoxies is a bulk property. The magnet coil was heated internally by passing current through the coil. Surface temperature measurements gave a good indication of the bulk epoxy temperature. The disk samples, on the other hand, were heated externally and surface temperature measurements may not have given a proper indication of the bulk epoxy temperature. Therefore, in view of these differences and the lack of any other comparative data, the outgassing rates determined for the magnet coil were felt to be sufficiently accurate to estimate the pumping and cooling system requirements needed for the electron injection experiment design, and no further attempt was made to make a detailed analysis of errors and/or differences in results. The total number of coils (14) needed for the experiment were fabricated and installed in a large vacuum facility and have operated successfully. As far as is known, the data of figure 6 is the first of its kind to be reported and should have application in a wide variety of technologies.

SUMMARY OF RESULTS

Values of thermal outgassing rates for a magnet coil insulated with an epoxy-impregnated glass tape were necessary for the magnet coil design of an electron injection research experiment. Outgassing data were taken from a sample magnet coil by measuring the rate of pressure rise in an evacuated bell jar containing the coil. Outgassing rates were determined to vary from 3×10^{-8} to 9×10^{-7} torr-liter per square centimeter-second for epoxy temperatures between 24° and 53° C, respectively. These thermal outgassing rates were compared with those obtained from small disk shaped epoxy samples. For epoxy temperature of 30° C, the magnet coil outgassing rate was about the same as the disk sample outgassing rate. At 53° C the coil exhibited about one order of magnitude higher outgassing rate than that obtained from the small samples. This difference was attributed to surface roughness effects and different heating methods. The data are be-

lieved to be the first of its kind reported and should be useful for engineering design estimates in which National 2A566 epoxy is used.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, April 8, 1969,
120-26-02-10-22.

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