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Vacuum evaluation of a new perfluoroelastomer

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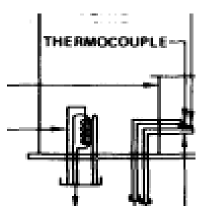
INTRODUCTION

The elastomeric O-rings used in high vacuum valves and flanges generally limit system bake temperatures to a maximum of 200°C and, typically, to below 150°C. Above these temperatures most elastomers have poor mechanical properties and outgas excessively. In order to bake a system at 300°C, gold, oxygen-free high conductivity (OFHC) copper, or polyimide seals are required. The advantages of metal seals are well documented.' Polyimide has excellent outgassing properties at elevated temperatures if properly cured2; however, it does not have the desirable mechanical properties of an elastomer like Viton. In fact, polyimide is relatively brittle, subject to chipping, and typically requires considerable stress to effect a seal.

High temperature, atmospheric pressure experiments conducted at this laboratory on a new fluorocarbon elastomer indicated that this elastomer might be a suitable replacement for metal and polyimide seals in systems bakable to 300°C. This elastomer, developed by E. I. duPont de Nemours & Co., is sold under the name ECD-006 perfluoroelastomer. It possesses many of the mechanical properties of Viton but with a significantly superior high temperature limit. Most of sit mechanical and chemical properties at atmospheric pressure and high temperatures have been documented.'' However, there has been no effort, to date, to investigate its high vacuum properties. This brief communication presents some data on these properties.



Fm. 1. Outgassing measurement apparatus.

EXPERIMENTAL PROCEDURES AND RESULTS

Two types of measurements were made. The first was a simple comparison of the outgassing rates of Viton and ECD-006. The second was an evaluation of ECD-006 as an O-ring in a bakable valve.

A. Outgassing

Figure 1 shows the apparatus used to compare the outgassing rates of Viton and ECD-006. The sample was placed in an enclosed volume on a square-shaped stainless-steel (Type 304) plate which was 1.27 cm (0.5 in.) on a side and 0.38 mm (0.015 in.) thick. The temperature of the plate was monitored with a chromelalumel thermocouple. The heater was constructed to minimize temperature gradients in the center of the plate. Heating of the plate was accomplished by radiation from tungsten filaments. Pressure within the enclosed volume was measured by a nude ionization gauge. A shield was interposed between this gauge and the sample plate to reduce charged particle bombardment of the sample. The only orifice connecting the sample volume with the main chamber was a 1.91-cm (0.75-in.) orifice through a 0.38-mm (0.015-in.) stainless-steel removable cover plate. The volume of the sample enclosure was 2.5 1. The pressure in the main chamber was also determined by a nude ionization gauge. Thoriated iridium filaments were used on both gauges. Two separate ionization gauge control units

Each sample was **0.32 X 0.64 X 0.16 cm (g X'TX I~fin.). After** the sample was placed on the plate and secured with a tungsten ribbon, the chamber was evacuated, and both ionization gauges were turned on. The radiation from the ionization gauge within the sample volume was sufficient to quickly bring the temperature of the sample to 120°C. The samples were baked at this temperature for four hours. After this bake, the pressure in the sample volume was typically 9.3-10.7 X 10-5 Pa (7-•8 X 10-7 Torr) while the pressure in the main chamber was 6.7-8.0 X 10-5 Pa (5-6 X 10-' Torr). The temperature of the sample plate was then elevated from 120° to 400°C over approximately a 13-min interval. Though the precision of the plate temperature measurement was estimated to be t 10°C, the bulk temperature uncertainties are significantly greater due to poor conductive heat transfer across surface boundaries, the low thermal conductivities of the samples, and radiative transfer processes. However, since the materials have similar surface and heat transfer properties, each sample should have experienced approximately the same heating rate because of the use of the programmable supply. Since the rate of temperature rise with time was slow relative to the flow-related equilibrium times for the essentially incondensable gases, the outgassing rate qo was computed in terms of nitrogen equivalents using the relationship

$$qo=S(P2-Pi)'$$

where P2 was the pressure in the sample volume, Pl was the pressure in the main chamber, and S was the speed of the pump out orifice. A pumping speed of 11.6 1/s cm2 was assumed, which for this orifice yielded a speed of 33 1/s. In Fig. 2 are shown the outgassing rates as a function of plate temperature for ECO-006, Viton, and a stainless-steel blank which was used as a reference.

B. Valve test

In order to check the mechanical properties of ECD-006 at elevated temperatures in a typical high vacuum application, the following test was performed. A 1.5-in. (3.8-cm) right-angle, high-vacuum valve (Varian 951-5072) was attached to a 30-1/s VacIon pump. The valve was initially provided with polyimide gaskets on both the bellows and main seals so that the valve could be baked to 300°C. Because the VacIon pump had seen extensive service during which it was

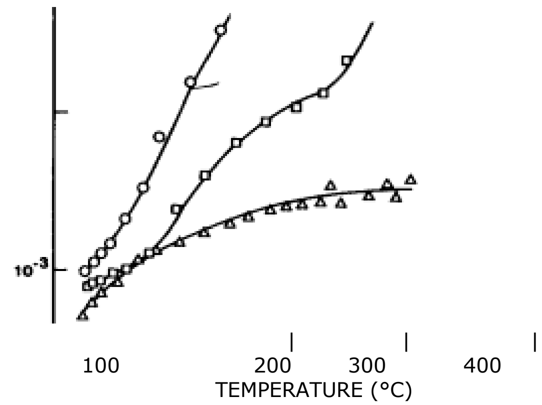


FIG. 2. Outgassing rates as functions of temperature after 4-h bake (120°C).

exposed to large quantities of hydrogen and noble gases, the terminal pressure after a 48-h bake at 300°C was 5.3-6.7 X 10-6 Pa (4-5 X 10-8 Torr), even though the pump was covered and the only load was the polyimide valve. The pressure was determined from the operating current of the pump. After this base pressure was established, the polyimide main seal was replaced with an O-ring cut from ECD-006 sheet stock (Compound 01018). The valve was reassembled and then baked at 300°C for 48 h. The terminal pressure after this bake was again 5.3-6.7 X 10-8 Pa (4-5 X 10-8 Torr). The valve was then disassembled, and the O-ring was inspected. The cracking and thermal set normally encountered with Viton when operated at temperatures in excess of 200°C were not present. The ECD-006 was still pliable. The valve was opened and closed several times with no apparent leaks across the main seal.

RESULTS AND DISCUSSION

Despite the uncertainties associated with the outgassing measurements, e.g., the nitrogen equivalence assumption, the limited sample preparation, etc., Fig. 2 indicates that ECD-006 has an outgassing rate at least comparable if not superior to Viton in the temperature range up to 200°C. It also shows that ECD-006 has an acceptable outgassing rate at 300°C where Viton literally decomposes, as indicated by violent pressure bursts observed on the gauges. ECD-006, even when heated at 400°C, did not decompose as violently as Viton at 300°C. It is also interesting to note that the initial outgassing rate of ECD-006 at 300°C is only 3-5 times greater than that of the stainless-steel blank.

The valve test that was performed used a valve that was designed to have a maximum bake temperature of 125°C in the closed position and 200°C in the open position if Viton-A was used for the main seal. A later design of this valve (Varian 951-5012) can be baked to 200°C in both open and closed positions because of a mechanical modification to reduce the problems of expansion and thermal set. With the ECD-006, bakeout at 300°C in the old style valve was accomplished without difficulty. Also, within the precision of these measurements, the room temperature outgassing rate of ECD-006 was comparable to polyimide.

The evidence of this study indicates that the vacuum properties of ECD-006 are such that it canbe used as a substitute for polyimide in the room temperature to 300°C operating regime. The measurements also indicate that more accurate tests should be made of the outgassing rates of ECD-006 and that its helium permeability, water absorption, and radiation resistance should be determined along with chemical character of the outgassing.

ACKNOWLEDGMENT

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6ECD-006 has been given the trade name Kalrez.