



# Preliminary Evaluation of Polyarylate Dielectric Films for Cryogenic Applications

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# PRELIMINARY EVALUATION OF POLYARYLATE DIELECTRIC FILMS FOR CRYOGENIC APPLICATIONS

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## ABSTRACT

Polymeric materials are used extensively on spacecraft and satellites in electrical power and distribution systems, as thermal blankets and optical surface coatings, as well as mechanical support structures. The reliability of these systems when exposed to the harsh environment of space is very critical to the success of the mission and the safety of the crew in manned-flight ventures. In this work, polyarylate films were evaluated for potential use as capacitor dielectrics and wiring insulation for cryogenic applications. Two grades of the film were characterized in terms of their electrical and mechanical properties before and after exposure to liquid nitrogen ( $-196\text{ }^{\circ}\text{C}$ ). The electrical characterization consisted of capacitance and dielectric loss measurements in the frequency range of 50 Hz to 100 kHz, and volume and surface resistivities. The mechanical measurements performed included changes in tensile (Young's modulus, elongation-at-break, and tensile strength) and structural properties (dimensional change, weight, and surface morphology). The preliminary results, which indicate good stability of the polymer after exposure to liquid nitrogen, are presented and discussed.

## INTRODUCTION

Dielectrics play a key role in the performance and reliability of most electrical and other systems. System survivability is in many cases governed by the endurance limit of the dielectric employed within such a system. Polymers are the most commonly used dielectrics because of their reliability, availability, ease of fabrication and cost [1]. The selection of the proper polymer dielectric for a desired application depends on the requirements and the operating conditions of the system. The key fundamental requirements are electrical, mechanical, thermal, chemical, and environmental. While it is highly desirable to employ dielectrics with superior performance in all properties, it is not feasible due to the fact that materials behave differently under exposure to certain environmental or operational stresses. In fact, while some of the properties of the same material excel under certain conditions, others do not fare as well.

An issue currently of major concern is the impact of the space environment on the performance and life of the electrical insulation and dielectrics [2]. These materials are usually sensitive to varying degrees of the severe stresses, which are typically encountered in space-based applications, including atomic

oxygen, vacuum, high energy radiation, low temperature and thermal cycling. For example, the material may experience different alterations in its physical, chemical, electrical and other properties due to stress-induced changes. These include chain scission and cross-linking, outgassing, and embrittlement, to name a few. As a result, degradation occurs in the materials properties that could prematurely terminate the useful life of the dielectric before electrical failure. It is important to note that the space environment may tend to induce changes in some materials or systems, while others are not affected. In addition, some of these changes are transitory while others may be permanent [3]. A correlation often exists between electrical, mechanical, and chemical properties [4]. For instance, changes in electrical properties are usually associated with chemical reactions or mechanical degradation. These, in turn, are intimately connected with the physical state of the material that is governed by its chemical structure [5]. Thus, the properties that are important for a material in a certain field will depend, in particular, on the type of application and how the material is being utilized.

Power systems planned for future space missions emphasize compactness, light weight, reliability, and highly efficient operation. Exposure of the power components and systems, such as capacitors and wiring constructions, to temperature excursions during their lifetime is anticipated in a wide span typical of space missions, as well as terrestrial applications. Included are deep space exploration, communication satellites, cryogenic instrumentation, superconducting magnetic energy storage systems, and magnetic levitation transportation systems [6]. For example, electronic instrumentation deployed near Pluto will encounter temperatures as low as  $-229\text{ }^{\circ}\text{C}$  [7]. The development of electrical systems capable of extreme temperature operation represents a key element to meeting the technological challenges and to fulfilling the requirements of advanced space power systems.

Polyarylate polymer film, a fully aromatic amorphous polyester, was evaluated for use as capacitor dielectric and wiring insulation for cryogenic applications. The recently developed polymer has been reported to display outstanding performance in radiation and high temperature environments [8]. The material, which has also good electrical and mechanical properties at cryogenic temperatures, has low specific weight and moisture absorption and may be used in electrical and electronic circuit boards, solar cells, superconductors, detectors, and in aerospace and automotive industries. The material was characterized in terms of its electrical and mechanical properties before and after exposure to liquid nitrogen. The electrical properties included the capacitance and dielectric loss in the frequency range of 50 Hz to 100 kHz, and volume and surface resistivities. The mechanical properties investigated comprised the Young's modulus, elongation-at-break, and tensile strength. The effect of liquid nitrogen aging on surface morphology and structural integrity was also investigated.

### EXPERIMENTAL PROCEDURE

ISARYL<sup>®</sup> polyarylate films, manufactured by ISONOVA [9], were used in these experiments. Two grades of the solvent-cast films, namely 15F and 25F with thicknesses of 120  $\mu\text{m}$  and 100  $\mu\text{m}$  respectively, were investigated. Some of the properties of the films are given in Table I.

The experiments were performed on as-received (control) samples as well as on samples soaked in liquid nitrogen ( $-196\text{ }^{\circ}\text{C}$ ). All samples were initially cleaned with isopropyl alcohol prior to handling. Aging of the samples was done by soaking them in a liquid nitrogen-filled dewar for 24 hours.

Table I. Properties of the ISARYL<sup>®</sup> films [9]

Film grade	15F	25F
Density (g/cm <sup>3</sup> )	1.21	1.22
Dielectric strength (kV/mm)	220	320
Dielectric constant @ 1 kHz	3.5	3.2
Dissipation factor@ 1 kHz	$7 \times 10^{-3}$	$3.1 \times 10^{-3}$
Surface resistance ( $\Omega$ )	$5 \times 10^{12}$	$5.5 \times 10^{16}$
Surface resistivity ( $\Omega \cdot \text{cm}$ )	$7 \times 10^{15}$	$2.5 \times 10^{17}$
Tensile strength (MPa)	76	100
Tensile modulus (Mpa)	2100	2750
Elongation-at-break (%)	115	70

<sup>®</sup>Trademark of ISONOVA Corp.

The capacitance and dissipation factor (dielectric loss) were obtained for control and aged samples at room temperature using a GenRad Model 1689 Precision RLC Digibridge, in conjunction with a Hewlett-Packard HP 16451B Dielectric Test Fixture, at six different frequencies ranging from 50 Hz to 100 kHz. Volume and surface resistivities of the films were measured using a Keithley Model 6105 Resistivity Adapter which conforms to the ASTM Standards for measurement of electrical resistance of insulating materials. A Keithley Model 247 High Voltage Supply was used as the power source, and a Keithley Model 614 Electrometer was used as the current sensing device.

An Instron tensile strength tester, Series IX, was used to measure the tensile properties of control and aged films. Samples to be tested were held between the grips of the Instron tester and stretched at a constant rate until fracture occurred. The reported value of the tensile properties was taken as the average of 4 readings per each data point. A Metalloplan Leitz optical microscope was used for surface examination of the test specimen.

## RESULTS AND DISCUSSION

Physical characterization of the polyarylate film, in both grades, revealed no effect of exposure to liquid nitrogen for 24 hours. Properties that were investigated on the 2.5 by 2.5 cm samples included changes in the sample dimensions, thickness, weight loss, and surface texture. No shrinkage, brittleness, swelling, color change, or weight loss was detected. Also, optical microscopy observation revealed no distinctive difference between the control and the aged films in terms of wrinkling, cloudiness, roughness, or alteration of the sample surface.

The capacitance, C, and the dissipation factor, DF, of grade 15F and 25F samples are shown in Tables II and III, respectively. These properties of the polyarylate films were obtained at six different frequencies for both the as-received as well as those of aged samples in liquid nitrogen for 24 hours.

It can be clearly seen that both the capacitance and the dissipation factor of both grades exhibited very slight change with aging. These variations are mostly attributed to instrumentation alignment as these properties were measured using a pressure-sensitive electrode test set-up. It is interesting to note that the dissipation factor of the polyarylate films attain high values at the two extreme frequencies regardless of aging. Such a trend is common to some polymers where they exhibit a bathtub-like characteristic with frequency.

Table II. Capacitance and dissipation factor of grade 15F polyarylate film as a function of frequency

Frequency (Hz)	Control		Aged	
	C (pF)	DF	C (pF)	DF
50	209.77	0.0068	202.17	0.0159
400	207.90	0.0061	200.43	0.0055
1 K	206.90	0.0063	200.02	0.0045
20 K	206.40	0.0209	199.39	0.0069
50 K	204.90	0.0111	197.98	0.0084
100 K	205.38	0.0165	198.50	0.0113

Table III. Capacitance and dissipation factor of grade 25 F polyarylate film as a function of frequency

Frequency (Hz)	Control		Aged	
	C (pF)	DF	C (pF)	C (pF)
50	225.36	0.0510	224.47	0.0186
400	221.73	0.0001	222.17	0.0052
1 K	220.63	0.0031	222.51	0.0039
20 K	222.15	0.0082	222.30	0.0049
50 K	219.15	0.0076	220.95	0.0061
100 K	221.22	0.0138	221.82	0.0087

The volume and surface resistivities of the films were obtained by applying a DC potential of 500V and a circular size sample of 6 cm in diameter. The results of both control and aged samples are shown in Table IV. The only appreciable change observed in these measurements is the increase in the surface resistance of the grade 25F film with aging. This change is not believed to be inherent to the dielectric film or indicative of the effect of aging as other properties, such as surface morphology, did not undergo any significant variation. It is probable that surface preparation and any contamination might have contributed to this behavior. The limited availability of test specimens did not allow these tests to be repeated.

Table V depicts the mechanical properties of the control and aged polyarylate films. The values of these properties, which included the tensile strength, Young's modulus, and elongation-at-break, were obtained as the average of 4 tests for each sample. It is evident that, while the tensile strength and elongation-at-break did not change much with aging, the Young's modulus seemed to exhibit slight increase for both films. In general, the experimental values of the mechanical properties investigated in this work are somewhat lower than those reported in the literature [8]. This is due to the fact that, because of limited availability of samples, test specimens used were smaller than desired for more accurate measurements. Nonetheless, exposure of the film to liquid nitrogen did not seem to greatly influence its mechanical properties.

Table IV. Volume and surface resistivities of polyarylate films

	Grade 15F		Grade 25F	
	Control	Aged	Control	Aged
$\rho$ ( $\Omega$ .cm)	$1.1 \times 10^{16}$	$5.5 \times 10^{15}$	$6.2 \times 10^{15}$	$7.6 \times 10^{15}$
$\sigma$ ( $\Omega$ )	$9.2 \times 10^{13}$	$1.25 \times 10^{14}$	$6.3 \times 10^{13}$	$1.1 \times 10^{17}$

Table V. Mechanical properties of polyarylate films

	Grade 15F		Grade 25F	
	Control	Aged	Control	Aged
Tensile strength (MPa)	81	80	74	76
Young's modulus (MPa)	803	906	1035	1058
Elongation-at-break (%)	82	76	47	48

### CONCLUSION

Preliminary investigation of the effect of short-term aging of polyarylate films in liquid nitrogen indicated good stability in their electrical and mechanical properties. Further research and experimental studies are required to fully characterize these and other materials for potential use as low temperature capacitor dielectrics, wiring insulation, optical surface coating, and thermal blankets in space-based applications. In particular, the effect of long term and simultaneous stressing under low temperature, atomic oxygen, radiation, and other environments will be of utmost importance for the development of reliable and efficient space power systems.

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