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E. I. DU PONT DE NEMOURS & COMPANY
INCORPORATED
WILMINGTON, DELAWARE 19898

POLYMER PRODUCTS DEPARTMENT

July 2, 1987

CERTIFIED MAIL - RETURN RECEIPT REQUESTED
Mr. Edward L. Jordan, Director
Division of Emergency
Preparedness and Engineering
Response
Office of Inspection and Enforcement
U.S. Nuclear Regulatory Commission
Washington, DC 20555

Dear Mr. Jordan:

Information Notice No. 87-08 issued on February 4, 1987 and Information Nótice No. 87-16 issued on April 2, 1987 have recently been brought to our attention. These information bulletins issued by the Nuclear Regulatory Commission indicate that Du Pont Kapton® polyimide film is being used in nuclear power plant containment areas. Because of its excellent radiation resistance, Kapton® is frequently used in high radiation environments. However, Kapton® tends to degrade when exposed to high temperature steam or certain volatile chemicals such as sodium hydroxide. Accordingly, when Kapton® is used in nuclear power systems that require certification to IEEE-323 and 383, engineered designs which protect Kapton® from direct exposure to loss of coolant accident sprays are required.

Even though Du Pont does not sell Kapton® directly to the nuclear power industry, we have received a number of reports that have provided us with information that the use of Kapton® in nuclear power plant containment areas in an inappropriate manner may be more widespread than the NRC bulletins suggest. For many years, Du Pont has had available for interested parties a "Radiation Resistance Bulletin" outlining the proper procedure for the use of Kapton® in radiation resistant applications. We are prepared to provide copies of this bulletin to you if you should deem it appropriate to communicate this information to nuclear power operators and contractors. A copy of this bulletin is attached to this letter.

If you have any questions or would like to discuss this matter further, please feel free to call upon George Cressman, Sr. Marketing Specialist. His telephone number is (302) 774-9529.

Very truly yours,

G. Robert Mc Kay Jr.

G. Robert Mc Kay
Division Director
Specialty Polymers Division
Polymer Products

Kapton® bulletin
POLYIMIDE FILM

RADIATION RESISTANCE

Industrial Film Division

KAPTON® POLYIMIDE FILM RADIATION RESISTANCE

Because of its excellent radiation resistance, KAPTON is frequently used in high radiation environments where a thin, flexible insulating material is required. In outer space, KAPTON is used both alone and in combination with other materials where radiation resistance at minimum weight is necessary. U.S. Government laboratory test data on gamma, electron and neutron radiation exposure of KAPTON is summarized in Tables I, II and III. KAPTON remains functional after exposure to radiation dosages up to 10^9 rads. Additional testing by CERN, The European Organization for Nuclear Research, confirms this data. Copies of this report are available from Du Pont.

KAPTON is also used in nuclear reactors and linear accelerators. Many of these applications require testing that involves exposure to an adverse chemical environment in addition to radiation. *For example, Loss of Coolant Accident (LOCA) tests for qualification in containment areas in nuclear power plants expose the system to steam and sodium hydroxide; both of which tend to degrade KAPTON.* Accordingly, when KAPTON is used in nuclear power systems that require certification to IEEE-323 and -383, engineered designs which protect KAPTON from direct exposure to LOCA sprays are required.

The excellent ultraviolet resistance of KAPTON in the high vacuum of outer space has been confirmed by NASA's Lewis Research Center. The data is shown in Table IV. In the earth's environment, however, there is a synergistic degrading effect on KAPTON if it is directly exposed to some combinations of ultraviolet radiation, oxygen, and water. Figure 1 shows this effect as a loss of elongation when KAPTON is exposed in Florida test panels. Figure 2 shows the loss of elongation as a function of exposure time in an Atlas Weatherometer. Design consideration should be given to recognize this phenomenon.

Normal room fluorescent lighting has no noticeable degrading effect on KAPTON. It should also be noted that the dielectric strength of KAPTON which has undergone physical property loss is not significantly affected unless material fracture due to system stresses occurs.

For further information on the radiation resistance of KAPTON, please contact your Du Pont KAPTON representative or Du Pont Product Information at (302) 774-2421 or 800-441-7515.

TABLE I

EFFECT OF GAMMA RADIATION EXPOSURE ON KAPTON POLYIMIDE FILM

COBALT 60 SOURCE (OAK RIDGE)

Property	Control 1 Mil Film	10^6 Rads 1 Hr.	10^7 Rads 10 Hrs.	10^8 Rads 4 Days	10^9 Rads 42 Days
Tensile Strength (Mpsi)	30	30	31	31	22
Elongation (%)	80	78	78	79	42
Tensile Modulus (Mpsi)	460	475	490	475	421
Volume Resistivity (Ohm-Cm) @ 200°C	4.8×10^{13}	6.6×10^{13}	5.2×10^{13}	1.7×10^{13}	1.6×10^{13}
Dielectric Constant @ 10^3 HZ @ 23°C	3.46	3.54	3.63	3.71	3.50
Dissipation Factor @ 10^3 Hz @ 23°C	.0020	.0023	.0024	.0037	.0029
Dielectric Strength (Volts/Mil)	6500	5660	5540	5700	6460

TABLE II

EFFECT OF ELECTRON EXPOSURE ON KAPTON POLYIMIDE FILM 2 MEV ELECTRONS (VAN DER GRAAF)

Property	Control 2 Mil Film	1×10^9 Rads	2×10^9 Rads	3×10^9 Rads
% of Initial Tensile and Elongation Retained	100	89	78	75
Volume Resistivity (Ohm-Cm) @ 23°C	6×10^{14}	6×10^{14}	5×10^{14}	3×10^{14}
Dielectric Strength (Volts/Mil)	1700	1610	1720	1695
Dielectric Constant @ 10^3 Hz	3.5	3.4	3.9	4.2
Dissipation Factor @ 10^3 Hz	.0062	.0310	.0259	.0388

TABLE III

EFFECT OF NEUTRON EXPOSURE ON KAPTON POLYIMIDE FILM MIXED NEUTRON AND GAMMA RADIATION (BROOKHAVEN PILE)

	5×10^9 Rads	10^{10} Rads
Temperature 175°C Flux 5×10^{12} Neutrons/Cm²/Sec.	Film Darkened	Film Darkened and Tough

TABLE IV

EFFECT OF ULTRAVIOLET EXPOSURE ON KAPTON POLYIMIDE FILM

VACUUM ENVIRONMENT*

	1000 Hr. Exposure
Tensile Strength, % of Initial Value Retained	100
Elongation, % of Initial Value Retained	74

*2×10^{-6} MM Hg at 50°C. UV Intensity Equal to Space Sunlight to 2500A.

Figure 1

Effect of Florida Aging on KAPTON® Polyimide Film

Ultimate Elongation Versus Exposure Time

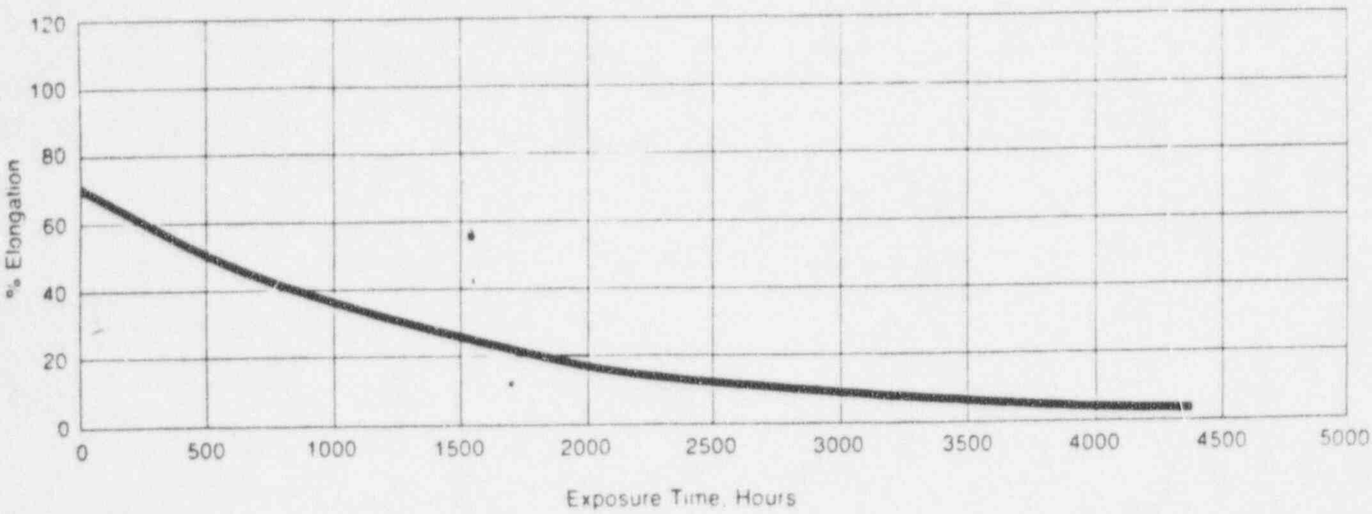
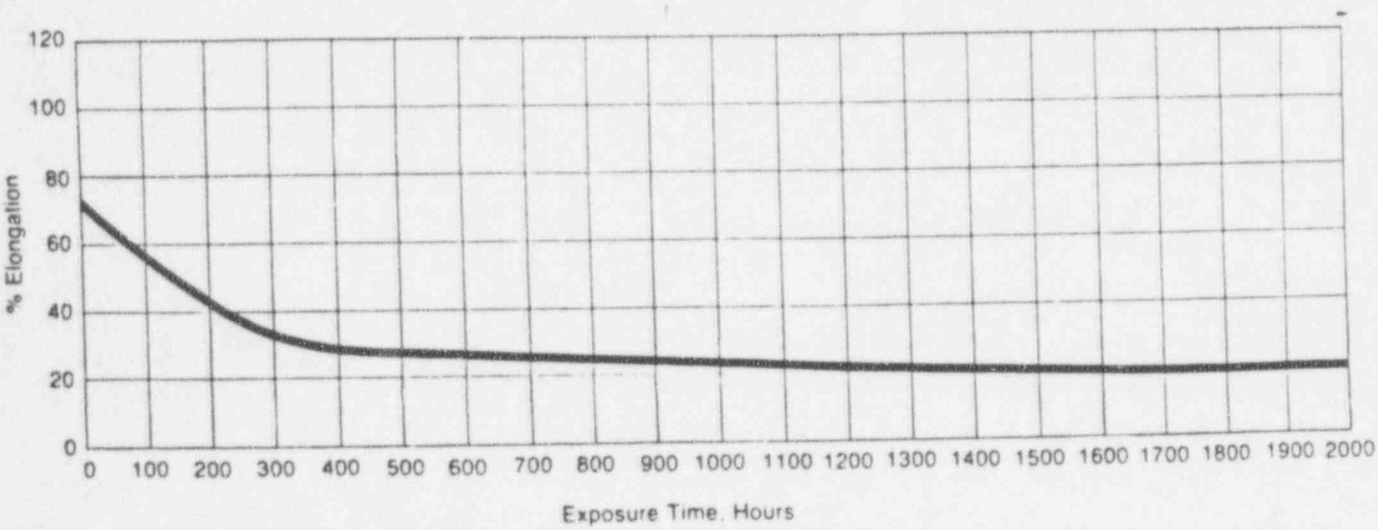


Figure 2

Effect of Weathering on KAPTON® Polyimide Film
Atlas Weatherometer

Ultimate Elongation Versus Exposure Time



CUSTOMER SERVICES ADDRESS

DU PONT COMPANY □ POLYMER PRODUCTS DEPARTMENT □ CUSTOMER SERVICES □ CHESTNUT RUN □ WILMINGTON DE 19898 (800) 441-9494 (800) 441-9498 (West Coast Only)

Industrial Films Division

DU PONT

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Southern California Edison Company
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August, 25 1987

Conax Buffalo Corporation
2300 Walden Avenue
Buffalo, New York 14225
(716) 684-4500

RECEIVED
AUG 2 1987

Attention: Mr. Richard Dulski

Dear: Mr. Dulski:

Subject: Failure Analysis of Containment Penetration Cables
San Onofre Nuclear Generating Station, Unit 1

Enclosure: Hi-REL Report No. FR-067094, Part Number 15-1, 15-3

Enclosed is an information copy of the failure analysis report performed on the Unit 1 Containment Penetration conductor insulation. The report documents all non-destructive and destructive analysis conducted by Hi-Rel Laboratories. We will keep you informed of any development or information that comes out of this ongoing evaluation.

Our evaluation of this event is continuing and any information, comments or suggestions that you may have in regard to the report or our investigation would be welcomed and appreciated.

We appreciate the friendly assistance your organization has provided to SCE. If you have any questions or comments please call Mr. Jim Redmon at (714) 368-9133.

Sincerely,

J. T. Reilly
Station Technical Manager

JRRedmon:JRR
Enclosure

B/5

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HI-REL
LABORATORIES

911 SOUTH MOUNTAIN AVE • MONROVIA • CALIFORNIA 91016 • (818) 357-6083 • TWX 910-585-3242

Southern California Edison Company

Report Number FR-067094

June 17, 1987

Part Number: 15-1, 15-3

Cable Segments

Quantity: Two (2)

Service Period: Approximately One (1) Year.

Service Location: Unit 1, WPC 6.

Submitted by: James Riddle

Approved by: Christopher J. Richards

Southern California Edison Company

Report Number FR-067094

INTRODUCTION

Hi-Rel Laboratories received two (2) wire segments from Southern California Edison Company for failure analysis. The conductors reportedly failed insulation resistance testing performed during a scheduled outage of the Unit 1 Reactor at the San Onofre Nuclear Power Station. The wires were in service at penetration WPC 6 on the outside of the containment. The conductor designations were 15-1 and 15-3. The reported service period was one (1) year.

The purpose of the failure analysis was to determine the cause of the insulation resistance failure of the conductors. If the Kapton insulation was found to be damaged (as it was reported to be) the anomalies were to be characterized as electrically, chemically, or mechanically induced. The analysis should determine if there is a generic problem with the wires and/or a mis-application of the products which render them unreliable in this application.

The samples were #2 gauge (0.2576 in. Dia.) copper stranded wires with Kapton, type F, insulation. Sample 15-1 had a partially intact crimp type splice on one end and bare wire, with 1 inch of the insulation stripped off, on the opposite end. Sample 15-3 had a fully intact crimp connection on one end with a solid copper wire as the other conductor in the splice. These crimp connections were reportedly the factory splices at the penetration. They were both encapsulated in black polyolefin shrink sleeve. The splices were to be cross sectioned and evaluated during the analysis.

ANALYSIS PROCEDURE/RESULTS

External Visual Inspection revealed ruptures in the Kapton insulation on both wires. The rupture on 15-1 was located several inches from the bare wire end (the field splice side of the conductor). The rupture on 15-3 was approximately 1½ feet from the cut, field splice, end of the wire.

Examination of these rupture sites revealed a blackening of the underlying copper wire and a white powder substance in and around the affected area.

A foreign particle was imbedded in the insulation on 15-1. The particle was not in the area of the rupture site.

The partially intact splice of 15-1 had been cut off through the middle of the connection. The stranded wire side of the splice was intact, the end of the wire could be seen recessed from the cut face. There was no evidence of corrosion to the wire ends or to the body of the crimp. The black insulation was intact over the splice. There were no anomalies in this area other than the cut through the splice. Fifteen-three had a fully intact factory splice. The sample had been cut approximately 2 inches into the solid conductor on the penetration side of the splice. There were no anomalies on the insulation over the splice.

The spliced segments were cut from the wires. A minimum segment of wire, up to and including the rupture sites, was cut from the opposite end of the wires. The wire segment from 15-1 with the imbedded particle was also retained for analysis. The remaining wire was returned for possible re-use.

Electrical Examination was not performed on the wire segments with the ruptured Kapton. Any tests would have been at least partially destructive to the insulaation. The loss of evidence at the failure site would have outweighed the benefit of the test.

A piece of insulation was cut from a section of 15-3. The insulation was flattened and placed between two parallel metal plates. A potential of 5000 volts DC was placed across the plates utilizing a HYPOT dielectric withstanding voltage tester. The test voltage was maintained for 24 hours with no breakdown of the insulation. The Kapton insulation did start to break down, however, with approximately 8000 volts AC applied across the sample.

The splices were tested for series resistance with a Kelvin sensing arrangement. A current of 1.00093 Amperes (DC) was passed through the samples and the voltage drop was measured with a precision microvolt meter. Sample 15-3 had an overall voltage drop between the solid conductor and the stranded conductor of 0.000153 volts. The voltage drop between the solid conductor and the splice was 0.000130 volts, and the drop between the splice and stranded conductor registered 0.000031 volts. The voltage drop on 15-1 between the splice and the stranded wires was 0.000060 volts. The data indicates that the crimps to the stranded wires form a lower resistance connection than the crimp connection to the solid conductor.

Cross Sectional analysis was performed on the splices. The black insulation was removed by making one lengthwise cut through it with a razor blade. Visual inspection of the surface of the splice revealed no anomalous conditions. There was no evidence of water on the wire or on the inner surface of the black insulation. There was no evidence of corrosion or chemical attack to the metal splice, the copper wire or the Kapton insulation.

The two samples were suspended in thermosetting epoxy and cross sectioned perpendicular to the axis of the wire/splice system. The half splice from 15-1 was sectioned from one side to an area where the wire was fully compressed. The splice from 15-3 was sectioned from both sides so that the stranded wire connection and the solid wire connection could be evaluated.

Internal Visual Examination of the cross sections revealed no anomalous conditions. The compression of the crimp was uniform and adequate on all the connections. The compression of the stranded wires formed the central strand into an almost perfect hexagon on both samples. There was no evidence of corrosion on any of the metal in the cross sections.

Energy Dispersive X-Ray Spectrum Analysis (EDS) was performed on small pieces of plastic material scraped out of the rupture site on 15-3. The underlying copper was not disturbed.

Examination of the clean wire revealed that silicon and a small amount of chlorine were present on the copper surface. The presence of silicon on the wires is probably not related to the failure mode. It may have been introduced from lubricants on the tools used to cut the wires or during handling prior to the analysis.

Depackaging was performed with a razor blade. The sections of insulation with the rupture sites were cut from their respective wires.

Internal Visual Examination of the wire revealed that corrosion had occurred to the copper underlying the break in the Kapton. The wire was blackened over a large area and there were local green patches in areas of extensive corrosion. The white powder substance, noted during the external examination, was present in localized clumps on the wire. The extent of the corrosion and the presence of byproducts was similar on both samples.

Examination of the insulation revealed the presence of the white powder material on the inner surface and between the delaminated layers of the insulation. The ruptured area was dry and white in color. This indicates the absence of Kapton in the rupture area (Kapton is orange in color). There were no abrasion marks on the insulation surrounding the ruptured area. There were no jagged or sharply cut ends on the insulation in the rupture cavity. There were no mechanically induced nicks or scratches in the copper wire underlying the ruptured insulation. There was no sign of electrically induced damage, such as melted portions of insulation or arc pitting in the copper wire. Both samples were similar in appearance.

EDS was performed on the white powder and the green corrosion products taken from the copper wire underlying the rupture on 15-3. The green spots contained sodium and chlorine on a strong copper background. The white powder produced a very strong sodium peak with a trace of silicon and copper. Note this EDS does not detect elements below atomic number 9 (flourine), thus, carbon and oxygen can not be detected. The sodium, therefore, may have been in the form of a carbonate or hydroxide.

The material was found to contain a number of elements: sodium, magnesium, phosphorus, sulfur, chlorine, potassium, and calcium. Silicon was present in the spectrum as well as the metals aluminum, zinc and iron. EDS does not provide specifically quantitative information, however, the ratio of the peaks is semi-quantative and some conclusions can be drawn. The proportional concentration of the ion species is close to that found in sea water, however, other elements are present which had to be introduced from another source. For example, sulfur is not present in sea water in concentrations above 1000 parts-per-million (the detection limit of EDS). The contamination consisted mainly of sodium chloride. The silicon may have been introduced from a cleaning agent, lubricant or paint. Phosphorus may have been introduced from a cleaning agent. Zinc and iron are the basic constituents of galvanized steel. Aluminum and sulfur are not easily accounted for: these elements will help "fingerprint" the, as yet, unidentified source of the contaminants which could be significant in the understanding of this failure.

EDS was also performed on the foreign particle on 15-1. It was found to be composed of aluminum, silicon, chlorine and iron. The presence of hydrocarbon residues is also strongly indicated. It has been speculated that the particle may be a splatter from a welding operation conducted in the area of the wire. The segment of wire containing the particle was metallographically cross sectioned. Examination revealed that the particle did not penetrate the entire insulation. The Kapton/Teflon insulation around the particle appeared to have melted. This indicates that the particle was injected into the insulation at a high velocity while it was very hot.

EDS was also performed on two wire strands taken from the exposed end of 15-1. One of the copper wires was black in color and the other was a "clean" correlation sample. The spectra revealed a large amount of sodium in one area on the surface of the blackened copper. Silicon, chlorine, and potassium were also present in this spectrum. Another area of the blackened wire was examined where there were dark colored spots on an otherwise clean surface. The spots were found to contain silicon and chlorine. No sodium was present in this spectrum.

Scanning Auger Electron Spectrum Analysis (AES) was performed to further characterize the white powder and the black material from the copper wires. AES provides data on the presence of carbon and oxygen as well as confirming the presence of the ions detected in the EDS.

The spectra confirmed the presence of sodium, chlorine, copper, and oxygen as the major elements in the white powder. Carbon and potassium were present as minor elements in the white powder. Carbon and potassium were present as minor elements. AES of a second area of the white powder revealed no carbon on the surface. Sodium, chlorine and oxygen were present in this area along with a small amount of potassium. The potassium was present on the surface and at a depth of 200 angstroms. Phosphorous was present 200 angstroms into the white material but not at the surface. This data indicates that the white powder consists of sodium and oxygen, most likely in the form of sodium hydroxide.

AES of the black material on the copper wire revealed that copper and oxygen were the major constituents. Small amounts of sodium, chlorine, potassium and carbon were present on the surface. The carbon disappeared at 200 angstroms but the ions remained. It can be concluded that the black material on the copper wires consists mainly of copper oxides.

Infra-Red Spectrum Analysis (IR) was performed on three samples of Kapton from 15-1, 15-3 and a correlation sample obtained from a different source. A sample of Kapton was taken from the area of the rupture (15-3) and another sample was taken from an undisturbed area (15-1). No significant differences were present in the spectra taken from the two areas on the insulation. There was a slight difference between the insulation samples and the correlation sample. The differences appeared as a peak at the 10.2 um wavelength region of the spectrum. The difference may be due to the presence of residual Teflon on the insulation samples. the report from the IR test lab, Spectra Research, is included with this report.

The preliminary conclusion that can be drawn from the IR data is that no major chemical break down is occurring to the Kapton on the insulation. The damage is restricted to the immediate area of the rupture sites. The rupture sites are highly localized damage sites.

Chemical Experiments were performed on two pieces of insulation cut from 15-3. The samples were approximately 2 inches in length. They were cut from the wire by circumferential cuts and slit open by one lengthwise cut.

One sample was placed in a beaker of saturated sodium hydroxide solution at 70 degrees Centigrade for 16 hours. Examination revealed that most of the Kapton had dissolved and disappeared. The Teflon plys remained intact. This experiment proves that sodium hydroxide breaks down the Kapton.

The second sample was placed in a beaker of saturated sodium chloride solution at 70 degrees Centigrade for 16 hours. Two razor cuts were made in the sample prior to the test. One cut was lengthwise, the other was circumferential. The cuts extended completely through the insulation. Subsequent examination revealed that Kapton did not dissolve as in the other experiment, but it did appear to have been chemically attacked at the exposed cut faces. The cuts took on a smooth curved surface instead of the straight face caused by the razor cut. This experiment indicates that the Kapton was slowly attacked by the sodium chloride solution. It shows that if the ruptures were initially abrasive punctures, the evidence of a mechanical rupture would be masked by subsequent chemical attack of the fracture or cut surfaces.

An experiment was conducted where a 2 inch wire segment, with insulation intact, was immersed in a saturated sodium chloride solution at 70 degrees Centigrade for 16 hours. Examination of the wire strands revealed that they were taking on the appearance of the blackened strands underlying the rupture sites. The darkening was uniform on all the strands. The ends of the cut wire segment were not sealed so the solution could travel between all the windings. This experiment indicates that corrosion of the copper wires by sodium chloride can occur and is manifested as a black film on the wire strands.

Other experiments are recommended in order to further characterize the variables involved in this failure mode. Placing wire segments in hot salt water solutions while the wires are conducting current and/or maintaining a voltage is recommended. Consultation with polymer chemists and corrosion chemists may provide ideas for superior, tightly controlled experiments which will help resolve the failure mode for these insulated wires.

SUMMARY

The possible factors contributing to the insulation failure are chemical attack, mechanical damage, and electrically induced insulation failure.

There is no data to support the electrically induced insulation breakdown hypothesis. The location of the failures is an improbable site for transient induced insulation breakdown. The failure sites are too far away from the inductive loads. A collapse of the inductive field when motors were switched off could have initiated spikes, but the loads were on the far side of the wire segments past several splices on the other side of the penetration. There was no evidence of electrically induced damage, such as arc pits or local melting, on the copper wires underlying the rupture sites. Testing revealed that the Kapton could withstand 5000 VDC across it. This performance greatly exceeds the manufacturer's recommended limits.

Chemical attack has obviously occurred to the affected areas. The question is whether the ruptures were initiated mechanically or if chemical attack could account for all the damage. Literature is available which documents the susceptability of Kapton to chemical attack by sodium. If the ph is raised to 11, chemical breakdown is imminent. Teflon coatings are applied to this insulation system in order to protect the Kapton. A mechanism for the chemical breakdown of the Teflon must be provided in order to explain the failures as strictly chemically induced. Another possibility is that the copper wire was initially contaminated during manufacturing and/or handling prior to being covered by the insulation. Mechanical pressure by the corroding copper would rupture the Kapton/Teflon system from within.

Experiments have shown that exposing mechanically damaged insulation to hot sodium/chlorine/water solution, causes changes in the area of the cuts which appear as a "softening" of the edges. This subsequent chemically induced damage masks the evidence of any initial mechanical damage. The mechanical damage will be difficult to substantiate unless samples are obtained with mechanical damage on or around some areas of slight chemical attack. The arguments in favor of initial mechanical damage center on the question of how the chemical agents got through the Teflon layers in the insulation.

It is recommended that an effort be mounted to examine other wires in the Unit 1 system to determine the extent of the problem. If damaged wires are located, it is important to uncover environmental variables which may play a significant role in the onset and development of the failure mode.

An experimental program should be developed which will (1) simulate the failure mode and create ruptures exactly like those in the field failures, (2) isolate the variables involved in the degradation of the insulation, (3) provide good estimates of reaction rates to determine the short term/long term seriousness of the problem and (4) provide data for selection of possible replacement materials to be employed if this system is found to be inadequate for the application requirements.

Firm conclusions cannot be extrapolated from the current set of facts. The insulation is susceptible to chemical attack under the appropriate conditions. It is most important to determine if the insulation can stand up to the environmental stresses in the application location under normal conditions. If the insulation can, in fact, withstand these stresses, then induced or manufactured mechanical imperfection in the insulation system must be present for the problem to develop to the degree where insulation resistance failures start to appear.