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# A[illegible]d Compatibility of TNF-Doped Mylar

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*Abstract*

*TNF-doped Mylar is a new radiation-hard dielectric that has recently been qualified as a viable substitute for Mylar in capacitors. The advantage of TNF-doped Mylar is that it satisfies both the nuclear safety and radiation hardness requirements of weapons. Mylar is not radiation-hard. Aging and compatibility studies were carried out to insure that 1)TNF does not diffuse from the film during fabrication of the capacitor or during storage; and 2)there are no compatibility problems with aluminum foil (the conductor) or Fluorinert (the secondary dielectric). Losses of TNF were barely detectable during the vacuum bakes used in fabricating capacitors or during accelerated aging tests carried out below $T_g$ (70C) over a two year period in air. In other accelerated tests, no compatibility problems were detected with aluminum or Fluorinert. TNF-doped Mylar is now being used in the MC-4109 capacitor that was called out for use in SRAM II. We anticipate no age-related or compatibility-related problems with TNF-doped Mylar.*

TNF-doped Mylar has been specified as the dielectric of choice in the liquid-filled capacitors (MC-4109) used in SRAM II. TNF-doped Mylar is a new radiation-hard dielectric that is made by incorporating TNF (2,4,7-trinitro-9-fluorenone) into Mylar by techniques that are similar to those used to dye Mylar. TNF-doped Mylar was chosen in preference to mica-paper, a radiation-hard inorganic dielectric, for reasons related to nuclear safety. TNF-doped Mylar, like Mylar, will melt at 250C in the event of an accidental fire. As a result, the capacitor shorts out and the weapons is rendered inoperable, i.e., a nuclear detonation cannot occur. Thus, Mylar capacitors function as what has been termed "weak links" in nuclear safety schemes. Capacitors made from mica-paper cannot serve as weak links; mica-paper does not melt and the melting of the aluminum foils is too high (~650C). Radiation-hardness tests, carried out in the Hermes Facility, showed that the radiation-hardness of TNF-doped Mylar was comparable to that of mica paper; voltage/charge losses brought about by an intense X-ray pulse were about 10% for both dielectrics at equivalent absorbed doses.. TNF-doped Mylar is attractive because it can satisfy both the nuclear safety and radiation-hardness requirements of weapons. The saturation level of the traps is very high and not a concern over the time period of interest.

Pure Mylar has high radiation-induced-conductivity. The X-ray pulse excites electrons to a high energy state which then migrate by a dispersive transport mechanism under the influence of the electric fields of the capacitor. This results in discharge of the capacitor. TNF enhances resistance to radiation by trapping the negative current carriers; it has no effect on the number of current carriers that are generated by the X-rays. This trapping process is not irreversible, however, but sufficiently strong to substantially reduce the mobility of the current carriers in an electric field.

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Before TNF-doped Mylar could be committed to any weapon system, it was necessary to determine if there were any compatibility or age-related problems. Areas of concern included loss of TNF during capacitor fabrication and stockpile strorage and compatibility with aluminum and Fluorinert (FC-40). We describe here the results of accelerated and short term non-accelerated aging and compatibility experiments that were carried out to address these concerns.

During fabrication, capacitors are vacuum baked to remove sorbed water. Typically, the capacitors are placed in vacuum ovens at 80C and 1 Torr for 24 hours. Earlier specifications called for an vacuum bake at 100C and 1 Torr for 24 hours. Doped Mylar which contained 1% TNF (the optimum concentration) lost less than 1% of the total TNF in the film after a vacuum bake under the more severe of the above two conditions.

To address the question of whether TNF would be retained over the stockpile life of any weapon system, free-standing films of TNF doped Mylar having TNF concentrations of 1.1 and 2.2% were aged over the temperature range of 54C to 100C for about two years. Loss of TNF was monitored by absorption in the ultraviolet at 350 nm. No loss of TNF was observed at 54C; at 70C, small decreases (<1%) of absorption were observed. There is a possibility that these small absorption losses were due to annealing effects that resulted in densification and a reduction in the thickness of the film. This possibility is being explored in a separate series of tests. At 85 and 100C, losses of TNF were 7 and 19%, respectively, for the film that contained 1.1% TNF; losses were 9 and 37%, respectively, for the 2.2% film.

Prediction of losses of TNF at the temperatures of service (~25C) using the Arrhenius paradigm is not feasible with this data because Mylar has a glass transtion temperature ($T_g$) at ~72C. $T_g$ can be defined as the temperature of the onset of large scale segmental chain motion. The trends of property changes with temperature are oftentimes altered significantly by this transition. Since the free volume in polymers is greater above the $T_g$ , the transport rates of TNF from Mylar would be expected to be much higher above $T_g$ than below $T_g$. Extrapolation of rates of transport through $T_g$ to lower temperatures where the polymer is in a glassy state would therefore be erroneous and high. In any case, these data are encouraging insofar as little, if any, loss of TNF was observed at temperatures <u>below</u> $T_g$ over a period of two years. This indicates that the physical forces binding TNF to Mylar are fairly strong. This finding is consistent with doping data which indicated that TNF has a strong affinity for Mylar. In doping experiments, it was observed that the concentration TNF in Mylar films was higher by a factor of three than the concentration in the doping solution. This indicates that, thermodynamically, TNF prefers to reside in Mylar rather than the doping solvent.

In general, Mylar capacitors used in weapons have two dielectrics. The primary dielectric being Mylar or TNF-doped Mylar and the secondary dielectric is either air or Fluorinert. The advantage of Fluorinert over air is that it stabilizes the high voltage breakdown characteristics in the end margin regions. Fluorinert is a liquid dielectric that is made by completely fluorinating low molecular weight hydrocarbons. The above described aging experiments are applicable only to those capacitors that use air as the secondary dielectric. When it was learned that the capacitor to be used in the SRAM II was the liquid-filled type, these aging experiments were reiterated with some changes

using Fluorinert as the aging medium. Both undoped and TNF-doped Mylar were aged in FC-40 at 50, 60 and 70C over a period of 12 months. For purposes of comparison, the same materials were also aged in air under the same conditions. Properties monitored were mass, thickness and TNF content (UV). No trends were observed for any of these properties over the 12 month duration of the test. Mass changes were less than 2% and appeared to vary almost randomly as function of time and temperature. These mass changes are probably artifacts related to removal of excess Fluorinert from the film prior to weighing. Within experimental error (1-2%), no changes were discernible in the thickness for materials aged in air or Fluorinert. Furthermore, no change in the TNF content of the aged films was detected. In short, no compatibility or age-related problems appear to exist between Fluorinert and TNF-doped Mylar.

To determine if TNF-doped Mylar and the aluminum foil of the capacitor are compatible, accelerated aging tests were carried out in which TNF-doped Mylar was sandwiched between two strips of aluminum foil and aged for three months at 45, 55 and 71C. The surface of the aluminum was then inspected microscopically for any evidence of corrosion and by x-ray photoelectron analysis (XPS) for evidence of contamination. In the latter analysis we compared the surface elemental composition of aged and unaged samples. Of particular interest was the element nitrogen; the only source of nitrogen is TNF. There was no evidence for corrosion of the aluminum by microscopic examination nor was there any nitrogen detected on the surface of the aluminum. On this basis of these findings we concluded that there are no gross compatibility problems between TNF-doped Mylar and aluminum.