Microwave Characterization of Gamma Ray Irradiated Thin Film Embedded and Non-embedded Nb Resonators

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Abstract. Gamma radiation effects on superconducting microwave transmission line structures, which may find use in future radiation challenging environments, such as satellites or accelerators have been investigated. Two versions of weakly coupled through-type Nb microstrip transmission line resonator material stack-ups were explored. In one version, the Nb signal trace was encapsulated with 20 µm of HD-4110 and in the other version the top Nb was not encapsulated. Exposure of the resonators to gamma radiation was performed for 28 days at room temperature in a sealed vacuum chamber using Co-60 as the gamma radiation source. The quality factors of the resonators were extracted at various cryogenic temperatures below the critical temperature of Nb and resonant frequencies up to 20 GHz. A large dose of gamma radiation used in this work showed a small change in the Nb superconducting properties.

1. Introduction
A crucial challenge when engineering a component for next-generation spacecraft is weight optimization. Reducing weight of the spacecraft allows for more functional equipment to be used in it, resulting in significant cost savings per launch. Given the possibility of taking really powerful equipment into space, we might be able to accomplish processing and compression of complex raw data reducing the data flow between space and ground stations. We believe that thin, flexible and superconducting cables can be an alternative to bulky coaxial cables, reducing the overall weight. For the use of these flexible cables in space, it is important to explore the impact of harsh environments such as high radiation up to 60 Mrad (600kGy) on the conductor and dielectric material. Earlier experiments on the influence of gamma rays on YBCO superconducting properties (critical temperature and critical current) have been reported in [1, 2]. As previously reported, the critical temperature and critical current density of thin superconducting films were not affected by gamma ray exposure up to a cumulative dose of 1.5 Mrad [3].

Various studies have been performed on the mechanical property degradation of polyimide films exposed on the surface of the spacecraft in space radiation environments such as electron, proton and UV rays [4, 5, 6] and extreme temperature conditions [7]. HD-4100 series polyimide products have found their greatest applicability in packaging applications. They have excellent mechanical properties to handle thermal and chemical extremes of post application processing, excellent elongation to prevent cracking, good adhesion and smooth via sidewalls to serves as a dielectric material.
Detailed study of the loss mechanisms is difficult due to the minimal loss of superconducting transmission lines of reasonable lengths. Instead, we looked at the quality factors of structurally identical resonators, which provide a sensitive probe of combined dielectric and superconductor losses. In this paper, we investigated gamma radiation effects on thin, flexible superconducting embedded and non-embedded resonators. The dielectric and conductor loss changes after 4 weeks of irradiation have been reported that show promising results to use these cables in high radiation conditions. We describe the design and fabrication procedure in section 2 followed by irradiation and measurement details in sections 3 and 4. Results are discussed in section 5 followed by conclusion in section 6.

2. Design and Fabrication Procedure

Half-wavelength capacitively-coupled microstrip resonators were designed and fabricated comparably to those in our prior work [8, 9, 10, 11]. The width of these thin transmission line resonators were designed for a 50 Ω characteristic impedance. Two versions were investigated in this work, embedded version, where signal layer was enclosed with another layer of polyimide and non-embedded version, where Nb signal layer was not enclosed. Fabrication of these resonators started with a deposition of 25 nm thick Cr and 200 nm thick Al as a sacrificial layer on a couple of oxidized silicon wafers, which act as temporary substrates during fabrication. A photodefined polyimide from HD Microsystems, HD-4110 was spun on the wafers and cured at 375 °C for one hour to acquire a desired thickness of 20 µm. Wafers were then patterned for signal layer deposition using standard photolithography process and 250 nm thick Nb was sputter deposited followed by a typical lift-off process. Nb was sputter deposited for 30 min with an Ar pressure of 4 mTorr (5.33 mbar) [12]. As a subsequent fabrication process, Ti(50 nm)/Cu(500 nm)/Au(10 nm) deposition was done on the wafers for metallization of connector contacts, followed by lift-off. After this step, the wafer with non-embedded version of samples was put in a dry box until the embedded version wafer had another layer of HD-4110 polyimide spin coated and cured at a lower temperature of 225 °C to protect the superconductivity of embedded Nb, resulting in a 20 µm thick layer after curing. Kapton dots were placed on the connector contact metallization area before spinning the second layer of polyimide on the embedded version samples and were removed before curing. The samples on both the wafers were then protected with a layer of photoresist before releasing in a salt....
solution with 0.5 V applied to the Cr/Al release layer. After releasing and stripping photoresist, the samples were inverted and mounted onto a new Si wafer and was followed by a deposition of 250 nm thick Nb ground plane layer. Fig. 1 shows a schematic drawing of the steps involved in fabrication of embedded and non-embedded Nb microstrip transmission line resonators fabricated using HD-4110 polyimide.

3. Irradiation Details

The irradiation of resonator samples was carried out using cobalt-60 as radiation source. The radiation source consisted of eighteen 43.2 cm tall rods. Each rod was encased in stainless-steel and was kept at a radial distance of 10 cm with 20° arc separation between centers of adjoining rods. The samples were under constant exposure of 1.17 Mev and 1.33 Mev gamma photons during the irradiation, commonly employed in total-dose testing for space research [13]. The sample holder with resonator samples mounted radially normal to the gamma source was lowered into a cylindrical chamber and sealed. The radiation source was lifted from its containment pool to surround the resonator samples in the cylindrical vacuum chamber at ambient temperature. During irradiation, a constant vacuum pressure of 1E-6 Torr was maintained in the cylindrical chamber. Sodium sulfide ion chamber dosimetry system was used to measure the dose rate to air at the chamber center, which was 23.6 rad/s. This dose rate was corrected to different materials based on their mass energy-absorption coefficients for 1.25 Mev photons from National Institute of Standards and technology [14]. Absorbed dose or dose rate to any material can be calculated from the absorbed dose to air as:

\[ D_{material} = D_{air} \frac{(\mu_{en}/\rho)_{material}}{(\mu_{en}/\rho)_{air}} \]  \hspace{1cm} (1)
4. Measurement Details

The resonator samples were baked at 90 °C for 2 hours before each measurement to reduce any humidity effects below detectable levels, which cause shifts in resonance frequency and decrease in measured quality factors at cryogenic temperatures as seen in our previous studies [10]. Southwest Microwave’s edge launch SubMiniature version A (SMA) connectors were used to assemble the resonator samples. These heavy SMA connectors may cause damage to the thin flexible cables, so as a prevention and to provide stability, they were mounted on to a support board. A cryogenic sample probe with assembled resonator sample and cryogenic semi rigid RF coaxial cables, which were used to interface the resonator sample through SMA connectors, was lowered into a pulse tube based closed-cycle cryostat system to carry out the measurements at various cryogenic temperatures. The sample temperature was measured using a temperature diode located near the sample on the sample holder. The scattering parameters were measured using a Keysight N5227A performance network analyzer across a substantially wide frequency range around the resonant peaks. Fig. 3 shows the resonator sample with edge launch SMA connectors and support board, cryogenic probe used for measurements inside a pulse-tube based close-cycle cryostat and Keysight PNA. An incident power of -28±2 dBm at the sample was used in this work.

where $D_{\text{material}}$ is dose rate to specified material, $D_{\text{air}}$ is dose rate to air, $(\mu_{\text{en}}/\rho)_{\text{material}}$ and $(\mu_{\text{en}}/\rho)_{\text{air}}$ is mass energy-absorption coefficient at photon energy of interest for specified material and air, respectively. Table 1 gives the total absorbed dose in each material. The absorbed dose values are very high compared to the radiation levels absorbed by electronics on satellites in a geosynchronous orbit in their typical lifetime, which is 3 kGy for 10 years [15]. Fig. 2 shows the gamma radiation source, sample holder with the resonator samples and vacuum chamber. After 4 weeks of irradiation, the radiation source was lowered back into its safe water storage pool, the sample holder was pulled out and the resonator samples were removed.

Figure 3. (a) Assembled resonator sample with edge launch SMA connectors and support board, (b) Cryogenic sample probe with resonator sample and RF coaxial cables used for measurements in a (c) pulse tube base closed-cycle cryostat using a Keysight performance network analyzer.
Figure 4. Comparison of $1/Q$ vs. resonant frequency for non embedded and embedded resonators before and after 4 weeks of irradiation. Plots show a slightly larger difference in $1/Q$ values indicating increase in loss at higher temperatures (4.2 K and 3.0 K) compared to difference in values at 2.0 K and 1.2 K after 4 weeks of irradiation.

Based on the measurement of similar resonators from our previous work [10], which showed reduction in measured quality factors and non-linearity effects for microwave powers above -25 dBm incident at the sample. After four weeks of irradiation, the resonator samples were reassembled with edge launch SMA connectors within an hour of being removed from the radiation chamber and re-measured using the same measurement approach and set-up.

5. Results and Discussion

The scattering parameters $S_{12}$ and $S_{21}$ were measured up to 20 GHz at various cryogenic temperatures (4.2 K, 3.0 K, 2.0 K, 1.2 K) below critical temperature ($T_c$). Loaded quality factors ($Q_l$) of the resonators, which were obtained by fitting the measured S-parameters using a Lorentzian function [16], were compared before and after four weeks of irradiation. Fig. 4 shows $1/Q_l$ versus measured resonant frequencies of the embedded and non-embedded resonators before and after 4 weeks of irradiation. An average of 10 measurements at each frequency were taken and $3\sigma$ error bars were added to consider the measurement errors and temperature drifts inside the closed-cycle cryostat. To offer an estimate of the sufficiently low coupling loss at each resonant frequency, ADS simulation data with zero conductor loss and zero dielectric loss have been added. According to BCS theory, there is a reduction of quasi-particles and an exponential increase in superconducting charge carriers as the sample is cooled further below its $T_c$, leading to a reduction in microwave losses. This is consistent with continuous reduction of slope, which corresponds to quasiparticle-induced losses as temperature reduces from 4.2 K to 1.2 K. At 1.2 K, the slope nearly approaches zero indicating that the loss is mostly influenced by the dielectric loss, though not completely. Variance in the plots of embedded and non-embedded resonator samples before irradiation is likely due to the additional embedding fabrication process. The authors believe that solvent and moisture may still remain in lower temperature cured HD-4110, which could explain the variance seen in the embedded samples.

Comparing the plots of extracted $Q_l$ values before and after four weeks of irradiation, it’s evident that there is a slight increase in losses at 4.2 K compared to other lower temperatures for both resonator versions indicating no detectable changes in dielectric properties of polyimide at lower cryogenic temperatures. A reasonable approximation of the loss contributors for microstrip resonators can be given as:
Figure 5. 1/Q vs. temperature for non-embedded and embedded resonators before and after 4 weeks of irradiation at different frequencies. Plots show decrease in $T_c$ for both resonator versions after 4 weeks of gamma irradiation. The $Q_l$ values decrease close to $T_c$ as a result of increase in normal electron density in the superconductor.

$$\frac{1}{Q_l} = \frac{1}{Q_c} + \frac{1}{Q_d} + \frac{1}{Q_r} + \frac{1}{Q_{coupling}}$$

(2)

where $Q_c$, $Q_d$, $Q_r$ and $Q_{coupling}$ are the Q-factors associated with conductor loss, dielectric loss, radiation loss and coupling loss, respectively. The radiation loss for these microstrip resonators using equations from [17, 18] was negligible and the coupling loss extracted from the ADS simulations with zero conductor and dielectric loss was also negligible. Thus, equation 2 can be reduced to:

$$\frac{1}{Q_l} = \frac{1}{Q_c} + \frac{1}{Q_d}$$

(3)

It is feasible to separate conductor loss and dielectric loss contribution by inspecting the extracted $Q_l$ as a function of temperature and frequency. The $Q_l$ measurement of the resonators was continued above 4.2 K to estimate the $T_c$ of Nb conductor. Control samples, which were fabricated alongside the irradiated resonator samples, were used to estimate the $T_c$ before irradiation. Fig. 5 shows 1/$Q_l$ versus temperature for non-embedded and embedded control samples and four-week irradiated samples. At temperatures close to $T_c$, the normal electron density increases, thus leading to a decrease in the $Q_l$.
Figure 6. (a) Loss from dielectric ($1/Q_d$) and conductor ($1/Q_c$) vs. frequency for non-embedded (top) and embedded (bottom) resonator samples. (b) Percent increase in dielectric loss and conductor loss after 4 weeks of irradiation. Plots indicate higher relative conductor loss contribution for the difference in the extracted $Q_l$ values at 4.2 K after 4 weeks of gamma irradiation and non-irradiated resonator samples.

Values. Measurements were carried out until the $Q_l$ values of the fundamental resonance frequency peak dropped below a reasonably extractable range. It can be seen that the estimated $T_c$ of the embedded sample is less than the non-embedded sample before irradiation. This may be due to the slight degradation on the Nb surface at the conductor and embedded polyimide layer interface, even with a lower temperature curing process. Using the $T_c$ information and characterizing the losses as a function of temperature and frequency under the influence of an external force, we have come up with a way to separate the conductor and dielectric loss in our superconducting resonators, which is part of a pending publication. Fig.6 shows the extracted conductor and dielectric losses and percent increase in the losses for all resonator samples versus frequency at 4.2 K. The plots show a higher increase in conductor loss compared to dielectric loss indicating a slight degradation in thin conductor film for both resonator versions. The results point to a small change in $Q_d$, which is directly related to dielectric loss factor ($\tan \delta$) indicating small changes in the dielectric properties even after subjecting to very high gamma radiation dosages (608 kGy).
6. Conclusion
For large radiation doses, up to 50.7 Mrad (507 kGy) for Nb superconductor and 60.8 Mrad (608 kGy) for polyimide film, show an average of 50% increase in conductor loss and very small increase in the dielectric losses at 4.2 K for both embedded and non-embedded resonator samples. The $Q_l$ value measurements as a function of temperature assisted us to estimate the $T_c$ of various resonator versions. At lower temperatures, undetectable change in the extracted $Q_l$ values was noticed indicating minimal changes in the dielectric properties of the polyimide (HD-4110). Experimental data presented here provide a good encouragement to use these thin, flexible, low mass superconducting cables in radiation challenging environments, such as space.

7. References
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