# ESTIMATION OF RADIATION DOSE TO EPOXY RESIN BY IR SPECTROPHOTOMETRY

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#### ABSTRACT

To estimate the radiation dose to the epoxy resin used for an insulation of the magnet coil, an IR (infrared) absorbance can be an effective method. Carbonyl radicals generated in the epoxy resin reveal an IR absorbance signal in the range of  $1705 - 1720 \text{ cm}^{-1}$  of which magnitude is proportional to the radiation dose. Irradiated samples of the epoxy resin were taken from the TRISTAN-MR magnets and their IR absorbance was compared with the calibrated data obtained from those of samples with controlled irradiation in order to estimate the dose. This method can be applied to the insulated material irradiated more than  $10^4$  Gy and only small amount of resin shaved from the surface of the insulation is enough for the measurement.

#### **1 INTRODUCTION**

Orbital synchrotron radiation of the high energy electron synchrotron is emitted to the tangential direction of the beam orbit. The critical energy is given by the formula  $\varepsilon_c [eV]=2218E^3 [GeV]/R[m]$ , where E is the beam energy and R the bending radius. For the TRISTAN main ring (MR)  $\varepsilon_c=220 [keV]$  at E=29 [GeV]. Photons with this critical energy interact with Aluminum atoms in the vacuum chamber and are scattered in the Compton regime to hit the insulation material of the magnet coil.

To estimate the radiation dose of the epoxy resin, so far we used dosimeters such as TLD (thermoluminescence dosimeter, BeO[UD-170L]) [1], Co glass and radiation sensitive color film (available on the market by the name of Radcolor) [2]. These dosimeters were put on the magnet coil surface during the radiation exposure and the radiation dose was calibrated by a time integral of the beam current at a fixed beam energy. Thus the total dose was estimated multiplying the beam experience of the coil since the beginning of operation. But these measurements aimed at the indirect measurement of the absorbed dose of the coil.

The present method will give a direct method to measure the radiation damage of the epoxy resin which is being used in the radiation environment and enable us to foresee its life. By irradiation, the carbonyl radical is induced in the epoxy resin and it can be revealed by an infrared (IR) spectrophotometry. If the relation between the absorbed dose and the absorbance of the carbonyl band is calibrated under the controlled irradiation, total radiation dose can be obtained by fitting the IR absorbance on the calibration curve [3] [4].

### 2 RELATION BETWEEN ABSORBED DOSE AND IR ABSORBANCE

To calibrate non-irradiated epoxy resin was scraped from the spare magnet and irradiated in the specimen boxes which were specially prepared for the radiation exposure to investigate the material properties under radiation. Radiation levels in boxes were calibrated with Co glass dosimeter whose integrated beam intensity for 30 minutes in TRISTAN-MR at 29 GeV was 6.6 mA.hr, corresponding to  $1.5 \times 10^3 \sim 5.8 \times 10^4$  Gy depending on the place where the boxes were positioned. Radiation dose to Co glass dosimeter was estimated as the increase of optical density by comparing the penetrations of light of 550 nm wave length between the irradiated and nonirradiated Co glass. Average dose of 10 Co glass dosimeters irradiated at the same time and at the same position in the ring showed (1.42x105±7%) Gy for 56.7 mA.hr at 29 GeV (corresponding to  $2.5 \times 10^6$  Gy/A.hr).

Another source of gamma ray is the Co-3kCi irradiation field at the Electrotechnical Laboratory. It has a capacity of  $10^5$  Gy/day.

Epoxy specimens from the unused coil irradiated at two different sources fit on almost the same IR absorbance spectrum line of the carbonyl radicals generated in the epoxy resin by irradiation as shown in Fig.1, where the triangles are for TRISTAN beam and circles for the Co gamma ray.

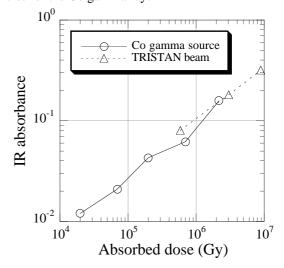


Figure: 1 Calibration curve between the absorbed dose and IR absorbance for the epoxy resin of the main dipole and quad.

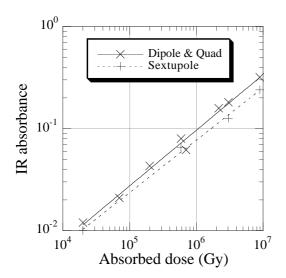


Figure: 2 Calibration curves for dipole & quad and sextupole. Epoxy resin was treated in different way by manufactures, so we distinguish between them.

As shown in Fig.2, calibration curves differ slightly depending on the manufactures. Dipole and quad were manufactured by the company H and sextupole by T. The former adopted amine and the latter acid anhydride as an epoxy hardener. The former has no absorbance spectral line around 1700 cm<sup>-1</sup> for un-irradiated epoxy and it appears at 1713 cm<sup>-1</sup> by irradiation. While the latter gives the spectrum of ester at 1730 cm<sup>-1</sup> before irradiation and this spectrum is affected slightly by irradiation. Difference before and after irradiation can be recognized by taking their difference.

### 3 ESTIMATION OF ABSORBED DOSE BY DOSIMETRY

In December of 1987 the radiation shield of vacuum chamber made of Al was reinforced all around the MR ring attaching 5 mm thick Pb plates and Pb fiber. Lead fiber has only half shielding power of the plate but was used to reduce the radiation from one magnet to the next by filling the both ends of the space between the chamber and the magnet pole. Roughly estimated dose is as follows since the beginning of TRISTAN operation,

1986.11 ~ 1988.2	2.0x10 <sup>6</sup> Gy (~ 10 A.hr)
1988.3 ~ 1990.7	2.5x10 <sup>6</sup> Gy (~ 50 A.hr)
1990.8 ~ 1991.1	none (shutdown for installation
	of mini-β system)

 $1991.2 \sim 1995.12 \quad 4.5 \times 10^6$  Gy (~90 A.hr), where the number in the parenthesis is the integration of

beam intensity during the given period.

Total dose of the TRISTAN magnet for 10 year's operation was  $9x10^6$  Gy. These data were estimated from radiation measurement with TLD. Dose rates were  $2x10^5$  and  $5x10^4$  Gy/A.hr before and after shield reinforcement, respectively. If there was no shield, dose rate was  $5x10^6$ 

Gy/A.hr. As the time integral of the beam intensity was 20 A.hr/year at 29 GeV summed up for both electron and positron, dose in a year amounted to  $10^6$  Gy/year. In 1991, radiation was measured for one of the dipoles with Co glass dosimeter as shown in Fig.3.

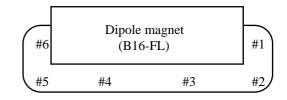


Figure: 3 Arrangement of Co glass dosimeter on the upper surface of the lower coil. Dose rates measured in 1991 were as follows,

#1	3x10 <sup>4</sup> Gy/A.hr	#4	1x10 <sup>4</sup> Gy/A.hr
#2	4x10 <sup>4</sup> Gy/A.hr	#5	4x10 <sup>4</sup> Gy/A.hr
#3	8x10 <sup>3</sup> Gy/A.hr	#6	1x10 <sup>4</sup> Gy/A.hr.

#### 4 ESTIMATION OF ABSORBED DOSE BY IR ABSORBANCE SPECTRUM

If the epoxy resin is irradiated, the carbonyl radical =C=O is generated and absorbs the infrared with the wave length of 1705 ~ 1720 cm<sup>-1</sup>. As the yield of the carbonyl radical is proportional to the absorbed dose, it is estimated by measuring the IR absorbance with the Fourier transform infrared spectrophotometer (FTIR of Perkin Elmer Corp.). Mixed powder of KBr and epoxy resin was pressed to form a thin semitransparent film on the top of the holder and then placed in the spectrophotometer.

Spectrum peaks by the carbonyl radicals are at 1713 and 1708 cm<sup>-1</sup> for H and T, respectively. In either case difference of the IR absorbance between the irradiated and non-irradiated specimens becomes zero at an inherent wave length for epoxy resin,  $1505 \sim 1510 \text{ cm}^{-1}$ .

Sampled magnets distributed along the one curved section (north TRISTAN-MR tunnel) and 10 specimens were sampled at random for each kind of magnets and their IR absorbances were measured. Several samples showed no evidence of irradiation, less than the detectable level. Histogram is given in Fig.4. Sextupoles had rather large dose compared to other magnets. Coils of dipoles and quads were protected by the 10 mm thick Pb plates placed in the coil slot but the sextupole coils would suffer from the intense radiation from the adjacent dipole and quad.

Typical IR spectrum for the sample shaved from the surface epoxy layer of the quadrupole coil darkened by heavy radiation dose is shown in Fig. 5. Its IR absorbance fitted on the calibration curve of Fig.2 showed  $10^6$  Gy for 10 years operation.

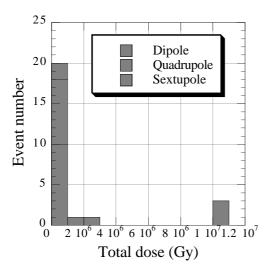


Figure: 4 Distribution of the absorbed dose for three kinds of magnets estimated from the integrated beam current till November of 1993, for 7 year's TRISTAN-MR operation.

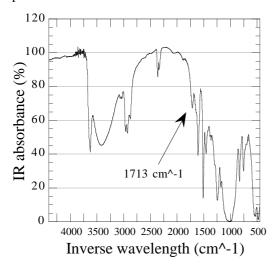


Figure: 5 Typical IR spectrum of the TRISTAN-MR quadrupole coil for heavily radiation-damaged epoxy resin.

## **5** CONCLUSION

The present study was made to investigate whether the TRISTAN magnets can be re-used to KEK-B factory whose construction is now underway. IR spectrophotometry is not widely recognized as a method to estimate the radiation dose. Sensitivity of IR absorbance to the radiation damage of epoxy resin is affected by the chemical treatment and uniformity of the insulation layer because IR method is inevitable to sample only small local part of the insulation material in order not to give damage by scraping the coil in use. However, TRISTAN-MR stopped the service forever and the tunnel is completely evacuated at the beginning of 1996 to prepare for installation of new KEKB rings.

Dipoles and quads are re-used for the high energy ring (HER) but the dipole coils are refurbished by replacing the

ground wrapping with new one leaving the inter-layer insulation as it is. For quads only the severely damaged coils are refurbished by the same method as the dipole coil [5]. Followings are the summary obtained from this study.

- (1) Calibration curve should be prepared by careful estimation of the radiation dose.
- (2) Epoxy resin insulation should be as uniform as possible so as to reflect the whole properties by local specimens.
- (3) Applicable dose range of IR spectrophotometry is  $10^3 \sim 10^7$  Gy.
- (4) Accumulated dose for 10 year's operation of TRISTAN magnets ranges from  $2.6 \times 10^3$  to  $1.1 \times 10^7$  Gy, mostly  $10^4 \sim 10^5$  Gy. Wide dose distribution partly depends on the non-uniform shielding structure reflecting the local shape of vacuum chamber which has many connections with other components such as beam monitor, pumping tube, gate valve and so on.

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