How can we increase the dose limits of organic insulation materials in superconducting accelerator magnets?

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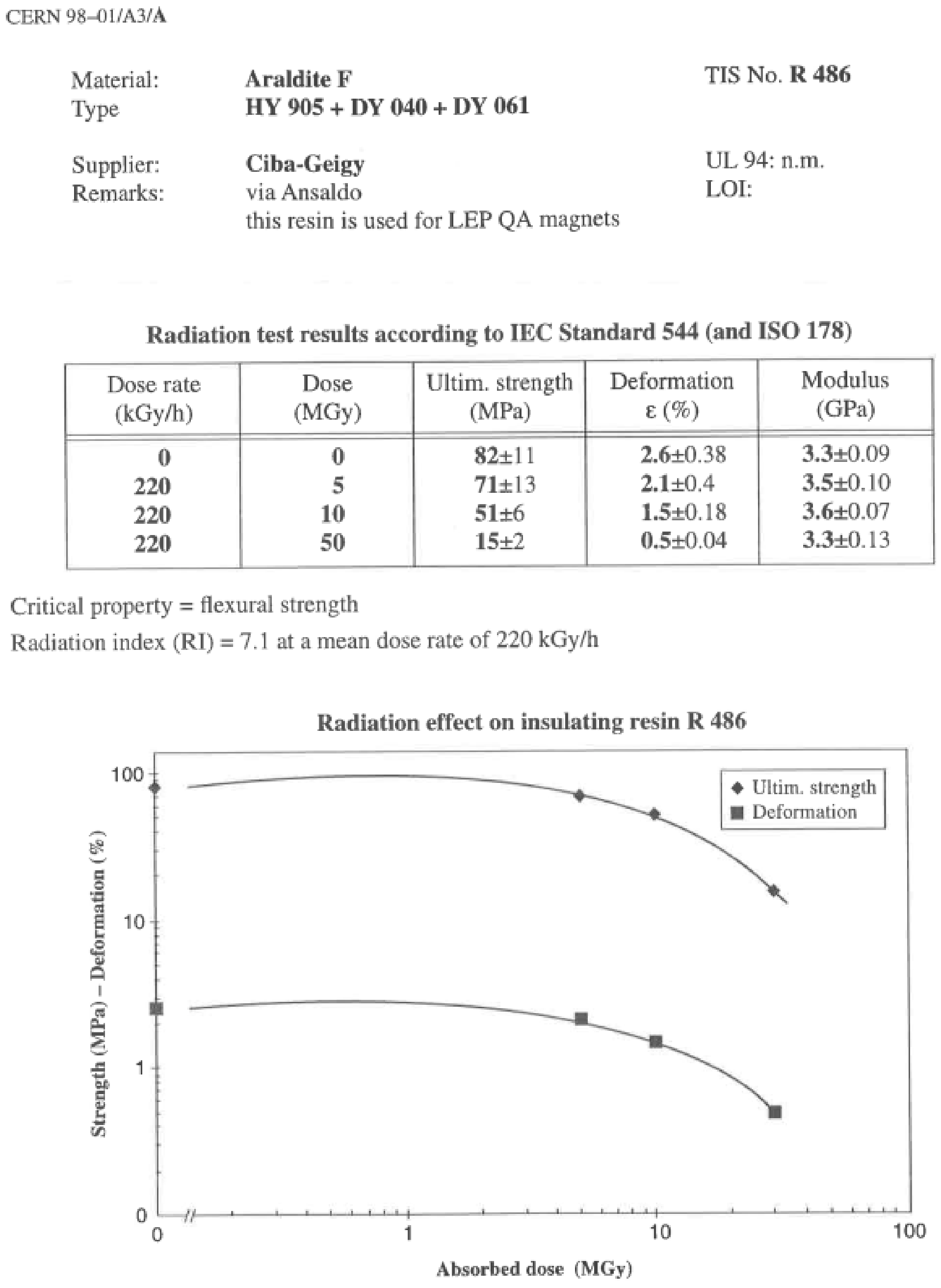
https://indico.cern.ch/event/1450988/contributions/6259757/

Outline

- Previous polymer irradiation damage studies
- Ongoing CERN Polymer Laboratory irradiation damage study
- Possibilities to increase the dose limits of organic insulations systems
- Irradiation sources
- Monitoring polymer irradiation damage
- Results
  - Radiation damage of epoxy resin systems with amine and anhydride-based hardeners during gamma and proton irradiation in ambient air
  - Effect of oxygen
  - Effect of irradiation temperature
  - Effect of mixed neutron/gamma reactor irradiation in air and in vacuum
  - Effect of mixed neutron/gamma irradiation at a spallation source
- Dielectric and mechanical requirements of superconducting magnet insulation systems
- Conclusion and outlook

Previous polymer irradiation damage studies: CERN yellow books

- The so-called CERN yellow books give a comprehensive overview on the irradiation behaviour of organic insulation materials (see for instance [1]).
- Low dose steps by ^{60}Co gamma irradiation with about 4 kGy/h.
- High dose steps in ASTRA fission reactor at Seibersdorf (Austria) with about 200 kGy/h dose rate in air at T<60 °C. Scaling factor is the total dose. The neutron dose is less than 5% of the total absorbed dose.
- Irradiation damage assessment is based on the relative changes of a critical property, for instance the flexural strength, the deformation at break, or the shear strength.
- The acceptable dose limit for rigid polymers like epoxy resins is defined as the dose at which the flexural strength is reduced by 50%.



CERN 98–01/A3/A

Material:	**Araldite F**	TIS No. **R 486**
Type	**HY 905 + DY 040 + DY 061**	
Supplier:	**Ciba-Geigy**	UL 94: n.m.
Remarks:	via Ansaldo this resin is used for LEP QA magnets	LOI:

Radiation test results according to IEC Standard 544 (and ISO 178)

Dose rate (kGy/h)	Dose (MGy)	Ultim. strength (MPa)	Deformation ε (%)	Modulus (GPa)
0	**0**	**82±11**	**2.6±0.38**	**3.3±0.09**
220	**5**	**71±13**	**2.1±0.4**	**3.5±0.10**
220	**10**	**51±6**	**1.5±0.18**	**3.6±0.07**
220	**50**	**15±2**	**0.5±0.04**	**3.3±0.13**

Critical property = flexural strength

Radiation index (RI) = 7.1 at a mean dose rate of 220 kGy/h

Radiation effect on insulating resin R 486

[1] M. Tavlet, A. Fointaine, H. Schönbacher, “Compilation of Radiation Damage Test Data-Thermoset and thermoplastic resins, composite materials”, CERN-98-01, (1998)

Previous polymer irradiation damage studies for the ITER project

- Irradiations have been done in different fission reactors, with very high dose rates in the order of 1 MGy/h.
- Scaling factor is the fast neutron fluence. Design fluence of the insulation system of the ITER Toroidal Field (TF) coils is 10^{22} n/m^2(E>0.1 MeV).
- The total absorbed dose at a certain neutron fluence depends on the simultaneously absorbed gamma dose, which differs in different reactors and irradiation locations. As an example:
  - TRIGA Mark II reactor at TU Vienna, fast neutron flux density was 7.6 x 10^{16} n/m^2 s (> 0.1 MeV) and gamma ray dose rate about 600 kGy/h [2],[3].
  - HR-1 of JRR-3M, Japan Atomic Energy Agency, fast neutron flux density of 1.7 x 10^{16} n/m^2 s (> 0.1 MeV) and a gamma ray dose rate of 2500 kGy/h [4].
- Irradiation damage assessment is mostly based on changes of the mechanical properties of [0°/90°] fibre reinforced composites.

For more information about the ITER irradiation study at TU Vienna see the presentation of Michael Eisterer.

[2] H.W. Weber, "Radiation effects on superconducting fusion magnet components", International Journal of Modern Physics E Vol. 20, No. 6 (2011) 1325–1378
[3] P. E. Fabian, J. A. Rice, N. A. Munshi, K. Humer and H. W. Weber, "Novel radiation-resistant insulation systems for fusion magnets", Fusion Engineering and Design, vol. 6162, pp. 795-799, 2002.
[4] T. Hemmi, A. Nishimura, K. Matsui, N. Koizumi, S. Nishijima, T. Shikam, "Evaluation of Inter-laminar Shear Strength of GFRP Composed of Bonded Glass/Polyimide Tapes and CyanateEster/Epoxy Blended Resin for ITER TF Coils "AIP Conf. Proc. 1574, 154–161 (2014)

The CERN Polymer Laboratory irradiation study

- We study the effect of ionising radiation on functional properties of polymers under irradiation conditions relevant for superconducting and resistive accelerator magnets for different projects:
  - Candidate materials for coil impregnation and electrical insulation of future superconducting High Field Magnets (HFM project) [5], [6], [7]
  - LHC Triplet Task Force (inner triplet magnet wedges and spacers, corrector magnet constituents, superconducting wire insulation, impregnated coil segments, fibre reinforced epoxy ground insulation) [8], [9]
  - HL-LHC (impregnation resin, instrumentation wire insulation, quench heater insulation)
  - PS Booster and SPS MBB magnets (candidate impregnation resins)
  - 3D printed high performance polymers [10]
  - Adhesives
  - Elastomers

[5] D.M. Parragh et al, IEEE Trans. Appl. Supercond., 34 (3), (2024), Art. no. 7800107, https://ieeexplore.ieee.org/document/10319395
[6] D.M. Parragh et al, Polymers 2024, 16(3), 407; https://www.mdpi.com/2073-4360/16/3/407
[7] A. Gaarud et al, Polymers 2024, 16(9), 1287; https://www.mdpi.com/2073-4360/16/9/1287
[8] G. Arduini et al, LHC Triplet Task Force Report, CERN, November 2023, https://cds.cern.ch/record/2882512
[9] C. Scheuerlein, "Irradiation study of MCBC and MCBY LHC corrector magnet polymer constituents", EDMS No. 2861509, March 2023
[10] C. Scheuerlein, D.M. Parragh, J. Vielhauer, A. Gaarud, N. Martin, R. Piccin, "Thermomechanical properties and irradiation induced aging of SLA and FDM 3D printed high performance polymers", accepted for proceedings of ICEC29/ICMC2024; https://indico.cern.ch/event/1296489/contributions/5881837/

Possibilities to increase the dose limits of organic insulation systems

- Choice of most radiation hard insulation materials
- Consider the effect of different irradiation sources, irradiation environment, dose rates and the irradiation temperature to determine the dose limit of the insulation system
  - What are the most suitable irradiation conditions for accelerated irradiation testing of superconducting magnet insulation systems?
- Refine dielectric and mechanical requirements of superconducting magnet insulation systems
  - What minimum mechanical properties are required to prevent critical defects that can lead to dielectric breakdown?

Radiation sources and environments

To verify the effect of the irradiation type and of the dose rate, identical materials have been irradiated with four different sources with dose rates ranging from <1 kGy/h to 900 kGy/h.

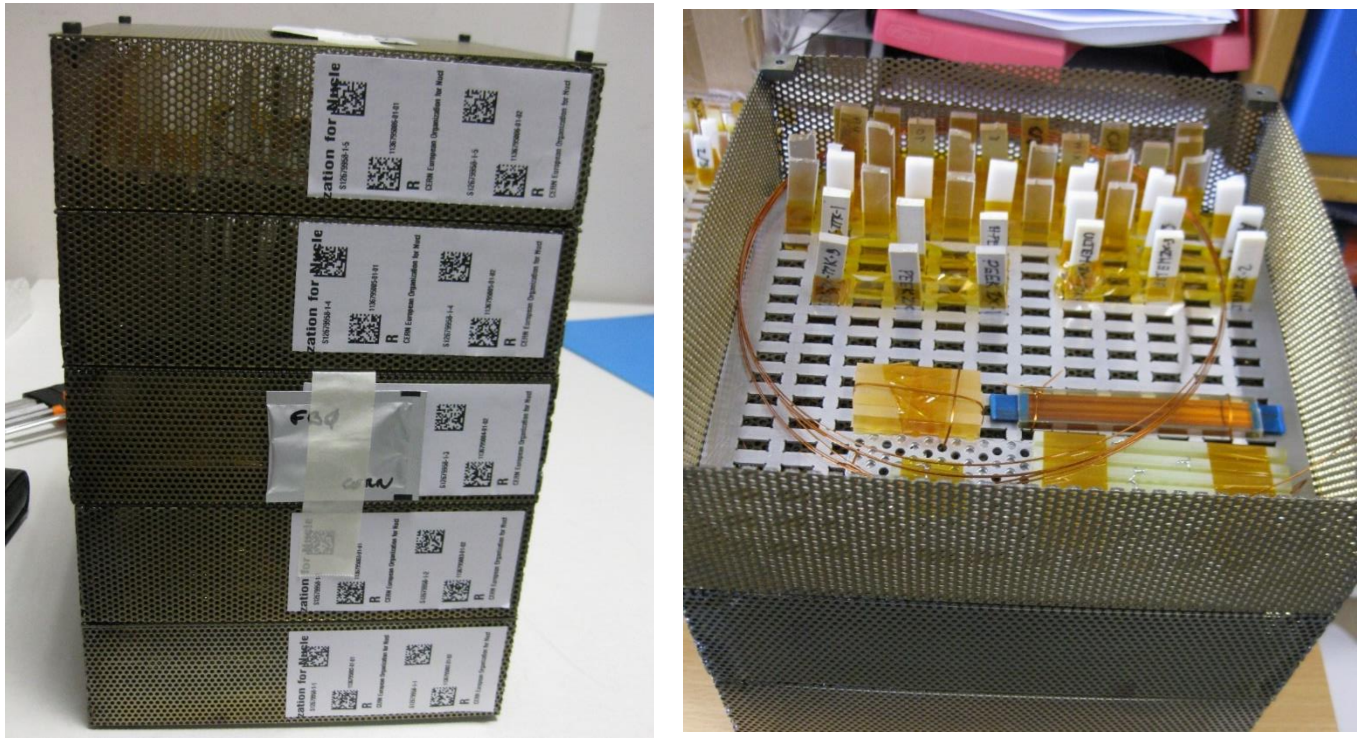
- gamma rays
- protons
- mixed neutron/gamma in a reactor
- mixed neutron/gamma at a spallation source

To verify the effect of environmental oxygen and of the sample temperature during irradiation, identical materials have been irradiated with the same proton beam in three different environments:

- in ambient air
- in inert gas at ambient temperature
- in liquid helium

Gamma irradiation in ambient air

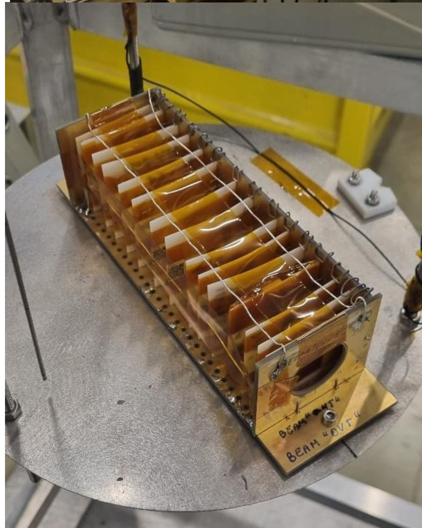
- ^{60}Co source at the Gammatec facility at the Marcoule site of the company STERIS.
- Irradiation in ambient air, temperature is controlled <25°C.
- Dose rate 2 to 3 kGy/h (about 25 MGy per year).
- Large sample volume. Samples for tensile, flexural, DMA and dielectric tests.
- Samples are not activated.
- Irradiation of 5 sample holders up to 30 MGy in 5 MGy dose steps is ongoing.
- Precise dosimetry (Perspex or Alanine dosimeters).
- Homogeneous dose distribution (sample holders rotate during irradiation).



Five sample holders 200 mm x 200 mm x 60 mm, stacked onto each other. Four dosimeters are attached at different locations outside and inside the sample holders.

24 GeV/c proton irradiation at IRRAD

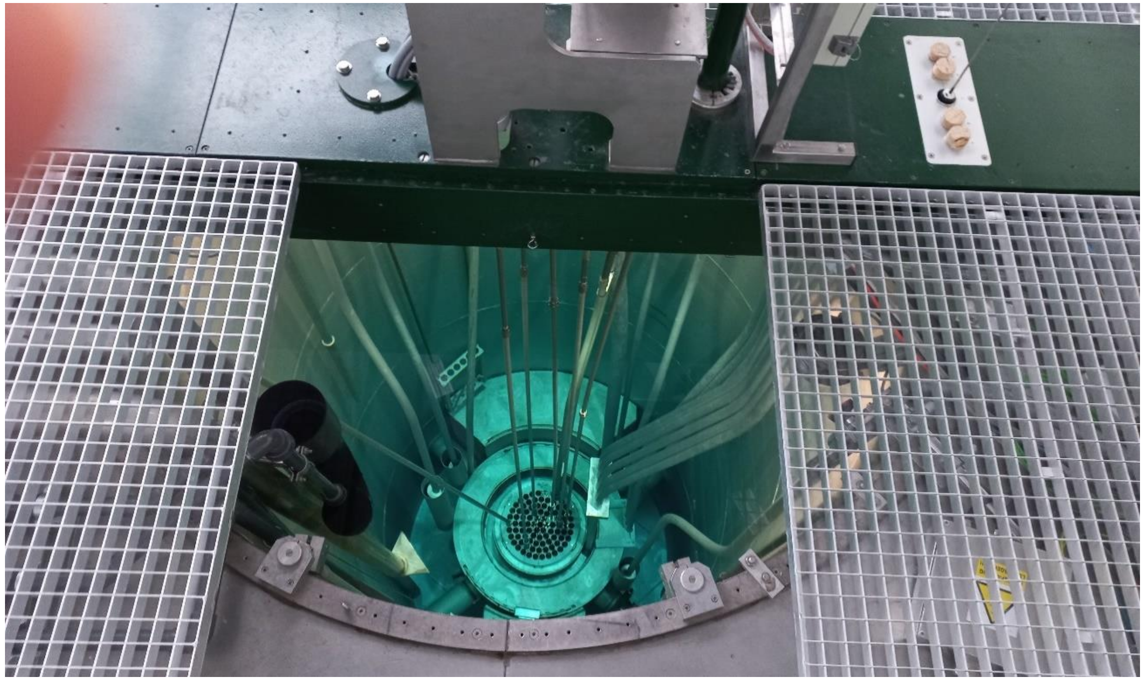
- Pulsed beam from PS accelerator, about 35 MGy per year (since run 2024 about 40% increase of proton fluence per week thanks to increased spill intensity)
- Determination of the proton fluence from the activation of Al witness samples (±7% uncertainty).
- A proton fluence of 3.35×10^{15} p/cm^2 corresponds with a total ionising dose (TID) of 1 MGy in epoxy [11]).
- Irradiated sample cross section is about 10 mm × 10 mm.
- Irradiation in different environments :
    - ambient air
    - inert gas
    - liquid Helium



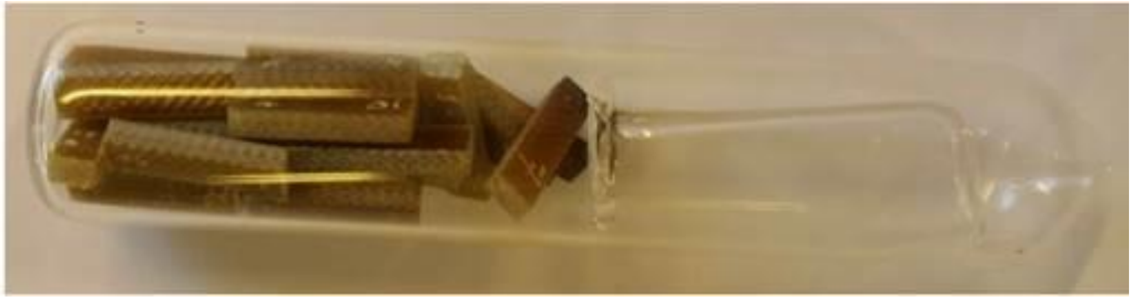
[11] F. Ravotti, "Dosimetry Techniques and Radiation Test Facilities for Total Ionizing Dose Testing", IEEE Transactions on Nuclear Science, vol. 65, no. 8, pp. 1440-1464, (2018)

Mixed gamma/neutron irradiation at the TRIGA Mark II fission reactor of TU Vienna

- Irradiation at the central sample position of the TRIGA Mark II reactor:
  - Gamma dose rate: 600 kGy/h
  - Fast neutron flux density: 3.5×10^{16} m^{-2} s^{-1} (E>0.1 MeV)
  - Total neutron flux density: 1.3×10^{17} $m^{-2}s^{-1}$
- In a typical epoxy resin a fast neutron fluence of 1×10^{22} $m^{-2}s^{-1}$ corresponds with a TID of about 70 MGy (49 MGy from gamma rays and 21 MGy from neutrons).
- Samples are irradiated in sealed quartz capsules. Sample temperature rise to about 70 °C during irradiation is estimated.
- Gas released during irradiation is collected in the capsules and can be analysed afterwards.
- Irradiation conditions are comparable with those of previous studies for the ITER project.
- Fast decay of radioactivity in all epoxy resins and S2 glass composites that we studied. In CTD425 epoxy/cyanate ester formation of ^{60}Co with half live of 5.2 years.



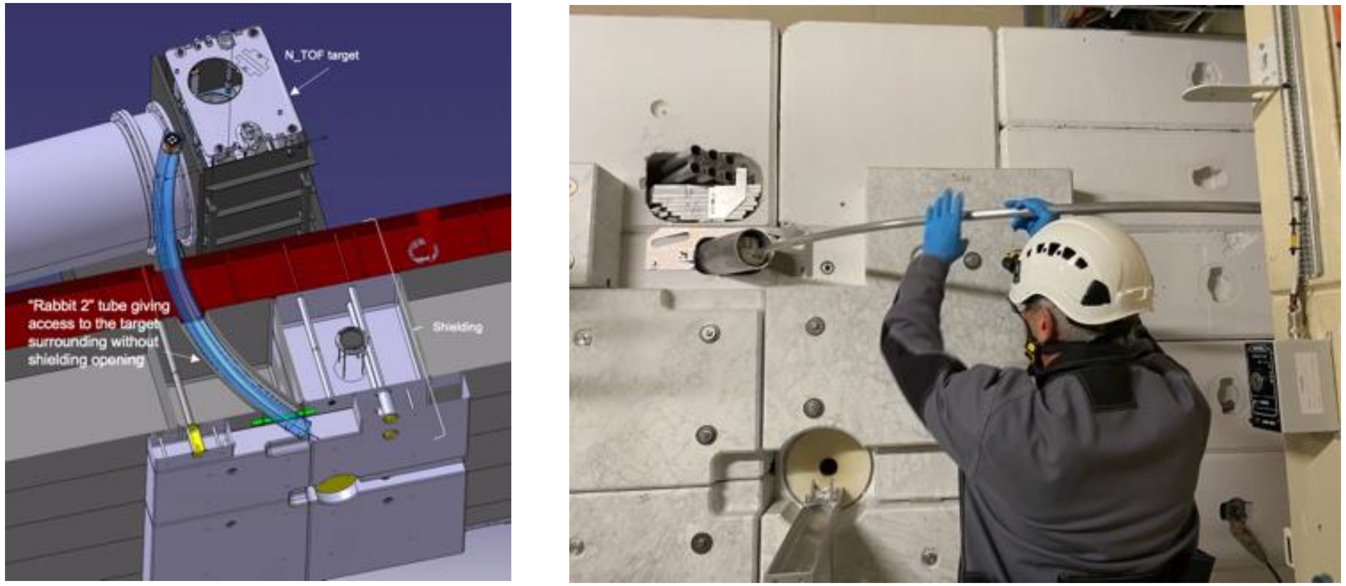
TRIGA Mark II reactor core.



Sealed irradiation capsule with samples.

Mixed neutron/gamma irradiation at NEAR n_TOF

- Irradiations in parasitic mode at the neutron spallation source of the n_TOF facility [12].
- The neutron fluences and simultaneous gamma doses are determined by FLUKA simulations.
- At the Rabbit 2 position of NEAR n_TOF, 0.6 Gy are absorbed in a typical epoxy resin per proton pulse on n_TOF target. TID is dominated by neutrons (75% of TID is due to neutrons) [13].
- A TID of about 2 MGy in epoxy can be achieved during one year operation.



Rabbit 2 location at the n_TOF target.

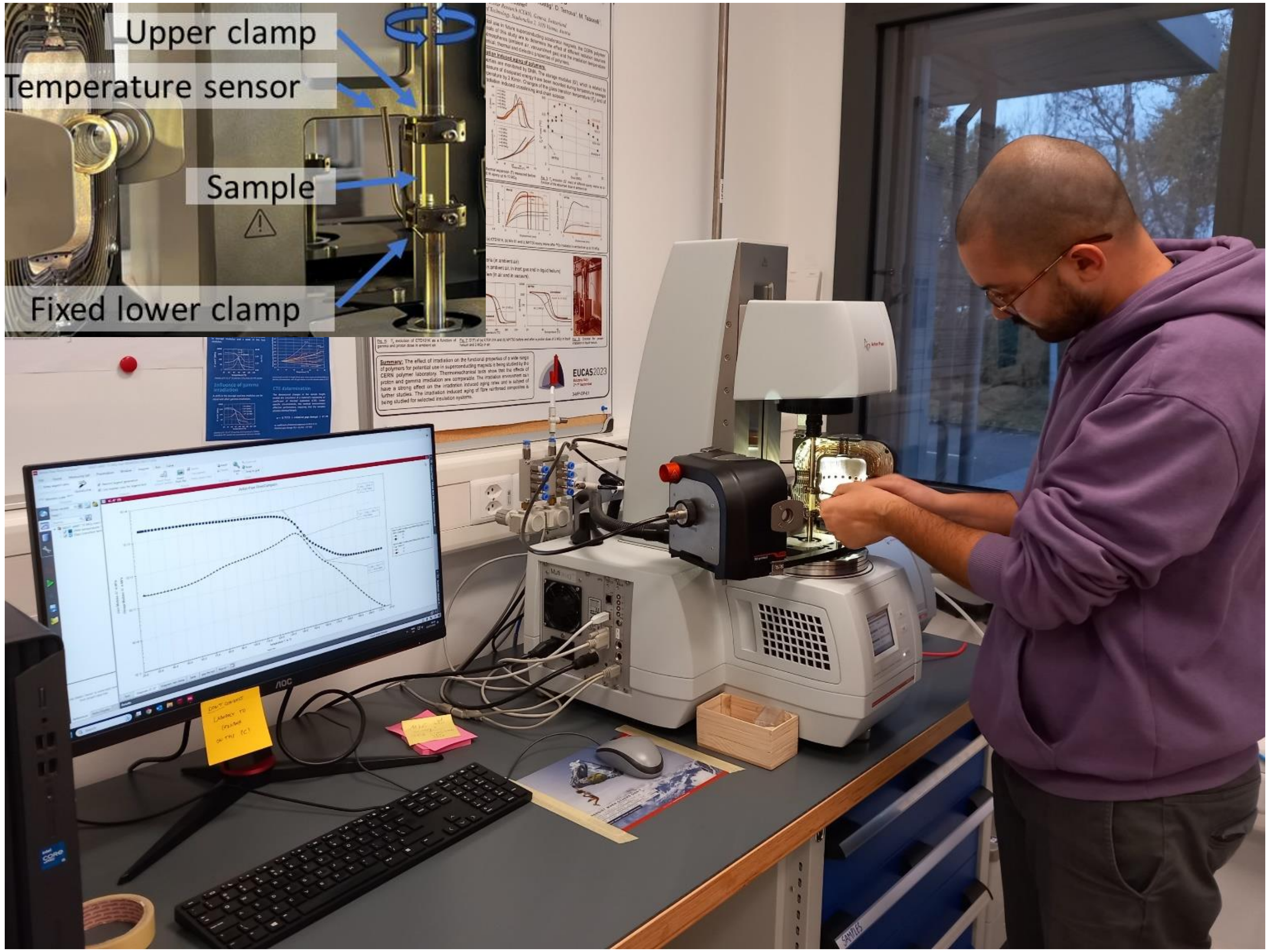
[12] C. Domingo-Pardo, et al, "The neutron time-of-flight facility n_TOF at CERN Recent facility upgrades and detector developments", 2023 J. Phys.: Conf. Ser. 2586 012150
[13] M. Cechetto et al., "High Level Dosimetry in Neutron Environments Compared to Cobalt 60 Gamma Sources"

Dynamic Mechanical Analysis (DMA) for monitoring radiation effects

- DMA is a non-destructive method that can be applied to monitor irradiation damage.
- Irradiation induced cross-linking and chain scission can be revealed.
- DMA test results are very well reproducible and a single sample per material and dose step is sufficient. Only a small sample volume needs to be irradiated. This makes it possible to:
    - Monitor radiation effects in smaller dose steps as was practically possible before
    - Irradiate the same materials with different radiation sources, including small beams
    - Irradiate the same materials in different environments, for instance in a liquid helium cryostat
- All samples of a certain material can be cut from a single plate, largely eliminating uncertainties due to sample preparation.

DMA set-up

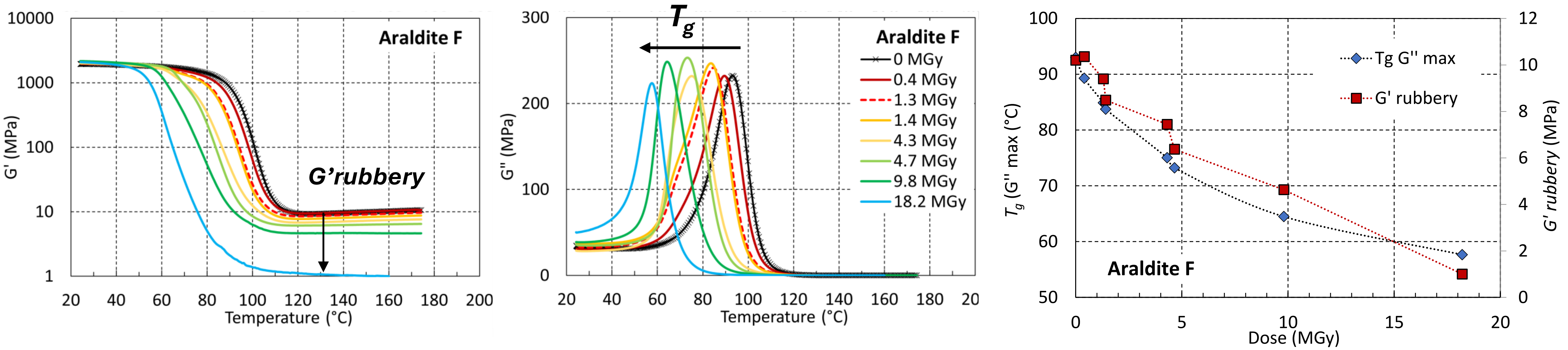
- DMA measures viscoelastic materials properties of the solid resin.
- In torsion mode, DMA measures the response to an applied torque, probing the sample shear properties.
- The storage modulus (G’) is related to sample stiffness, and the loss modulus (G’’) measures the dissipated energy (viscous portion).
- Temperature sweep: Measure G’ and G’’ as a function of temperature.
- Test parameters for irradiation study:
  - Frequency 1 Hz
  - Temperature ramp 2K/min
  - Sample dimensions 4×10×40 mm^3



Anton Paar MCR702e set-up for DMA in torsion mode.

DMA for monitoring cross-linking and chain scission

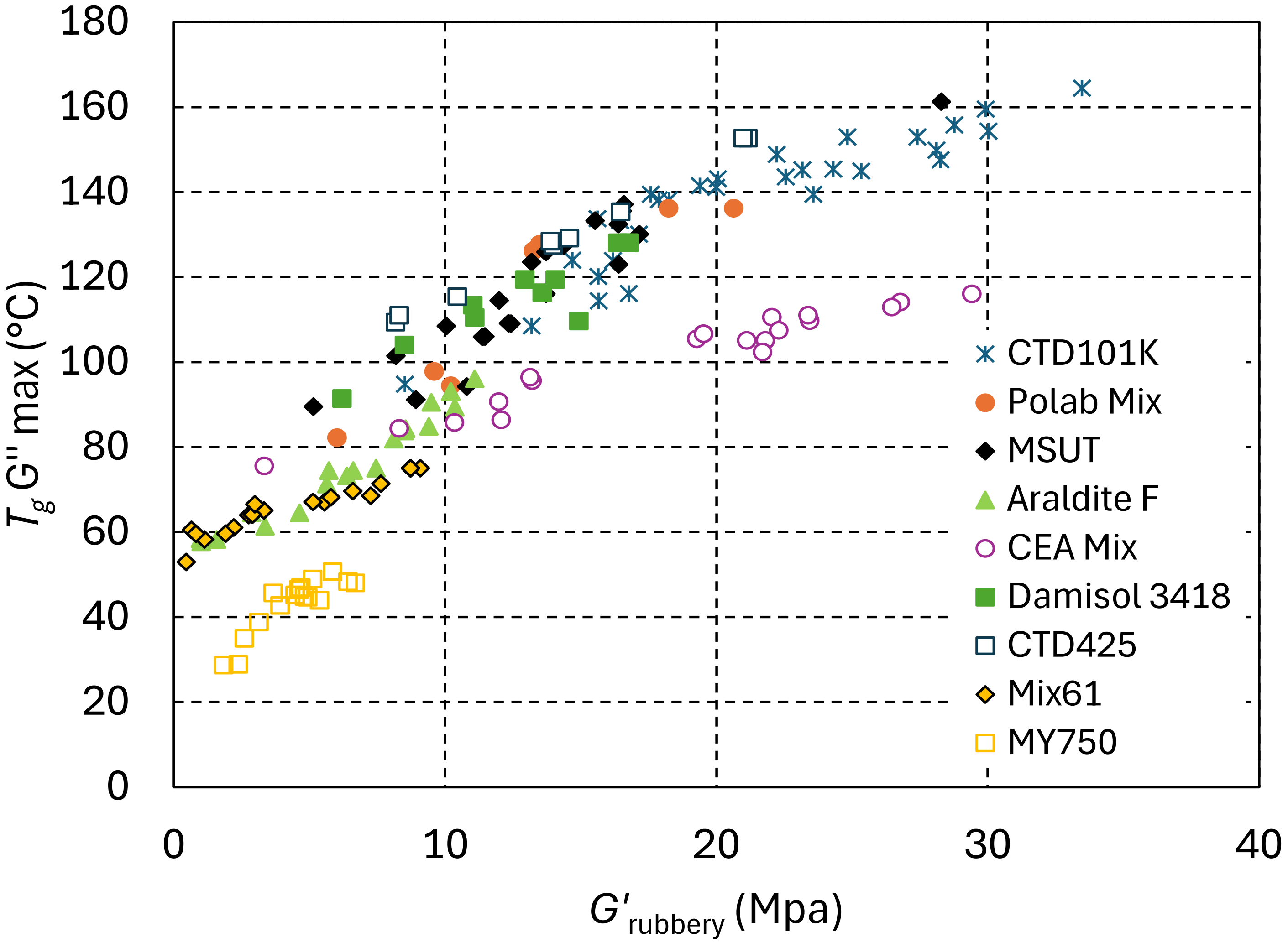
- Irradiation effects like formation of new cross links and chain scission influence the viscoelastic properties, and thus the storage and loss moduli.
- The glass transition temperature (T_g) vs dose evolutions allow to compare aging rates. Increasing T_g indicates that formation of new cross links prevails, decreasing T_g that chain scission rate dominates.
- The storage modulus in the rubbery state above T_g ($G'_{rubbery}$) depends on the molecular weight between cross links.



Araldite F (a) storage modulus G' (T) and (b) loss modulus (G'' (T) evolutions after different proton doses absorbed in ambient air. (c) Chain scission induced T_g and $G'_{rubbery}$ reduction.

T_g and $G'_{rubbery}$ of thermosetting resins

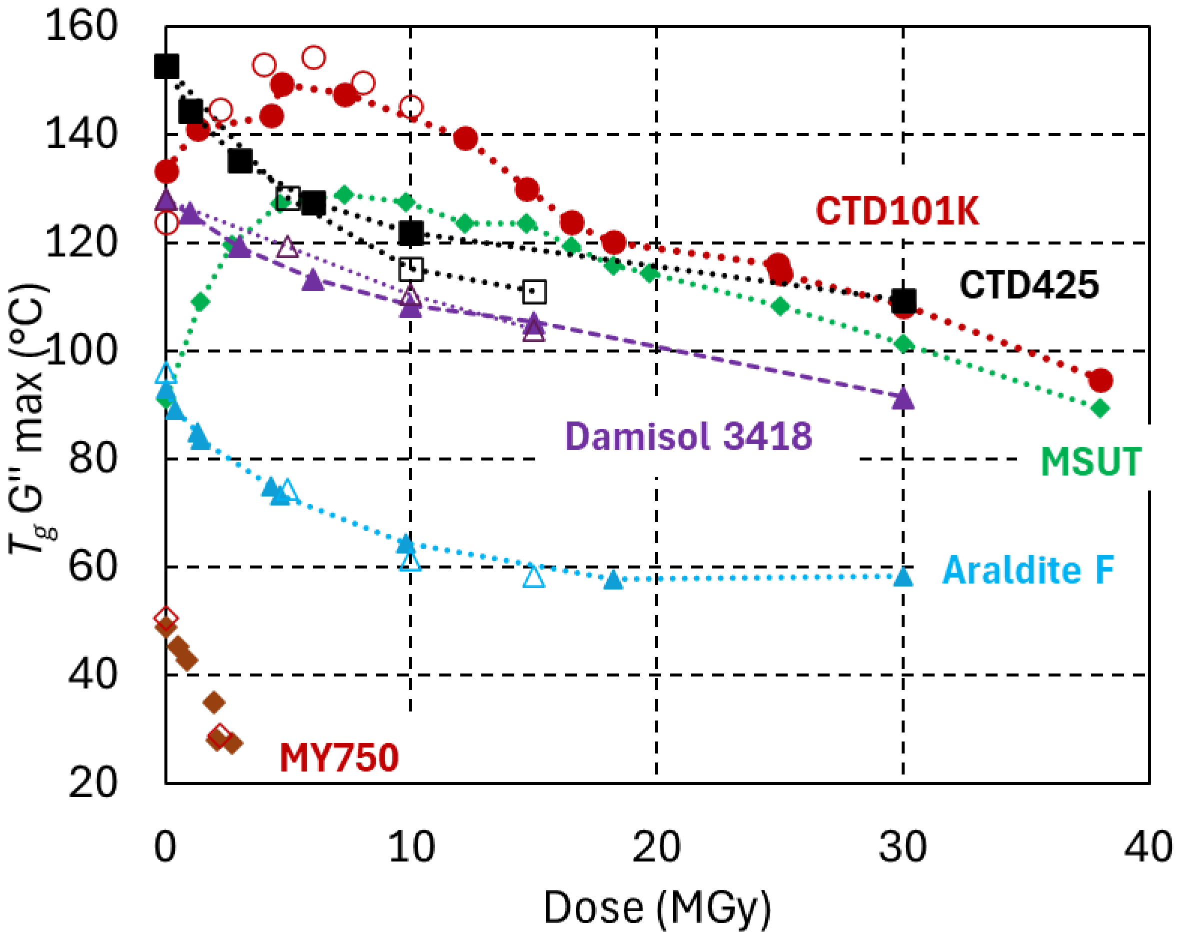
- T_g increases with increasing $G'_{rubbery}$.
- T_g and $G'_{rubbery}$ can be used to monitor irradiation induced chain scission and cross-linking.
- $G'_{rubbery}$ of an ideal rubber is proportional to the molecular weight between cross-links.



T_g (G'' maximum) as a function of $G'_{rubbery}$ of different epoxy rein systems before and after irradiation with different sources in different environments. Mix 61 T_g is determined from tan δ max.

Proton and gamma irradiation in ambient air

Cross-linking and chain scission in selected epoxy resin systems under ^{60}Co gamma and 24 GeV/c proton irradiation



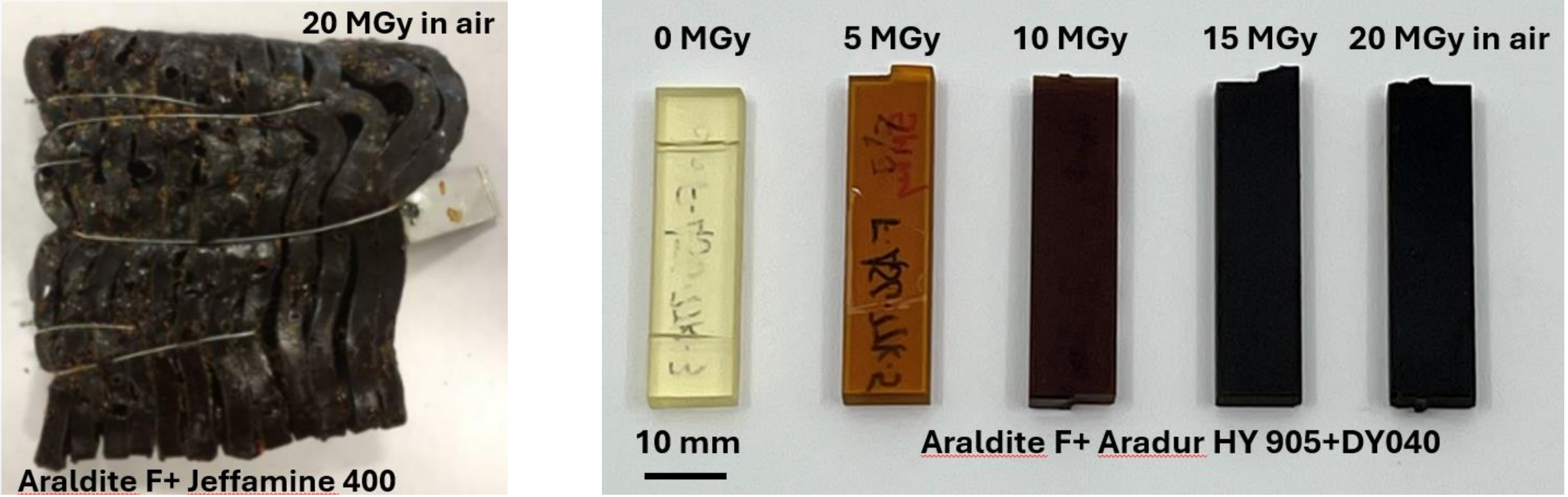
- ^{60}Co gamma and 24 GeV/c proton irradiation have the same effect.
- CTD10K and MSUT: Cross-linking is initially dominating, at higher doses chain scission prevails.
- MY750, Araldite F, Damisol 3418 epoxy systems and CTD425 cyanate ester/epoxy blend: Steady T_g decrease due to chain scission.

T_g evolution of selected epoxy resins and CTD425 cyanate ester-epoxy blend as a function of dose absorbed in ambient air (average dose rate is about 30 MGy per year). Full and empty symbols represent 24 GeV/c proton and ^{60}Co gamma irradiation, respectively.

Effect of hardener type on the radiation hardness of epoxy resin systems

Araldite F: Effect of hardener

- The Araldite F epoxy system with an amine-based hardener is much less resistant to irradiation than the same epoxy resin with the anhydride-based hardener recommended by the supplier.

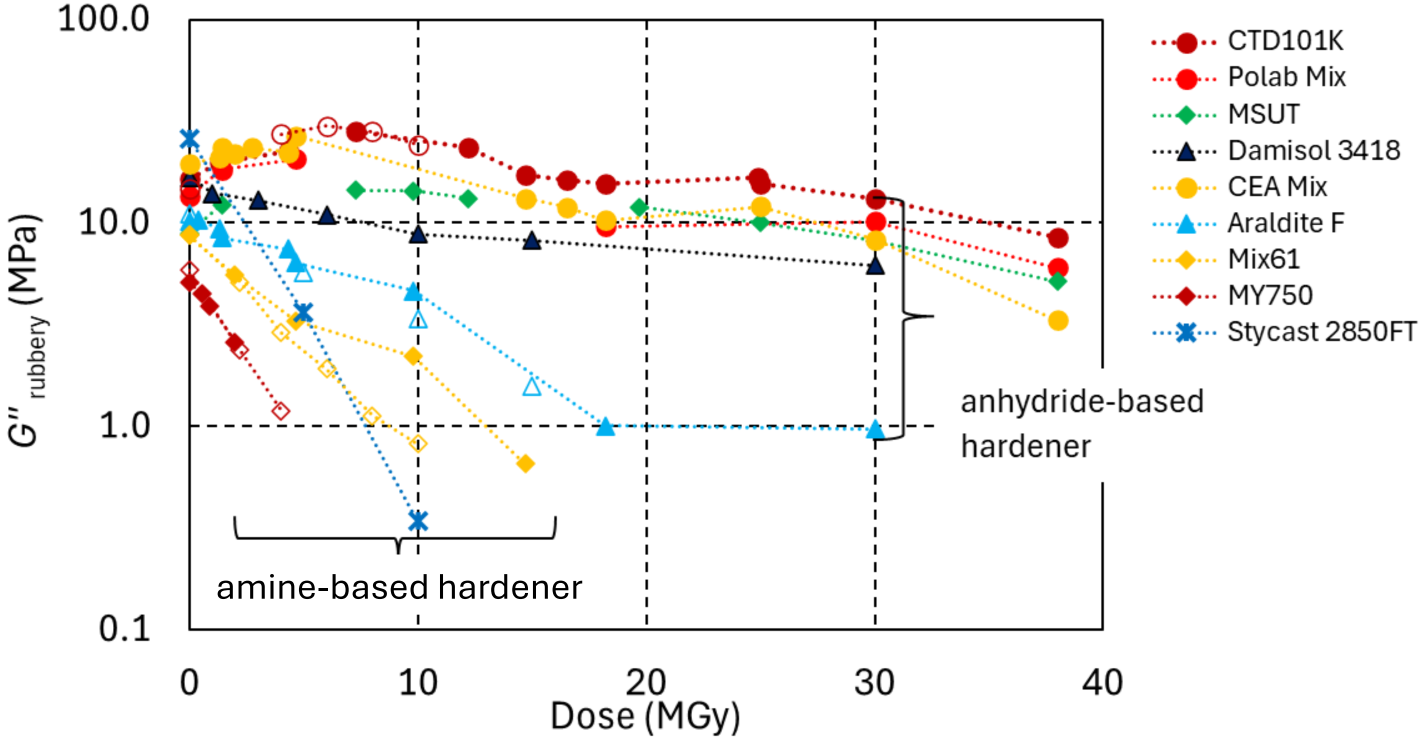


Araldite F with (a) Jeffamine D400 polyetheramine hardener (from [14]) and (b) with Aradur HY 905 carboxylic anhydride hardener *after different doses absorbed in air.*

[14] A. Musso et al, "Characterization of the Radiation Resistance of Glass Fiber Reinforced Plastics for Superconducting Magnets", IEEE Trans. Appl. Supercond. 32 (6), (2022)

Radiation hardness comparison of epoxy systems with anhydride and amine-based hardeners

- Epoxy resin systems with amine-based hardeners degrade at much lower doses than those with anhydride-based hardeners.

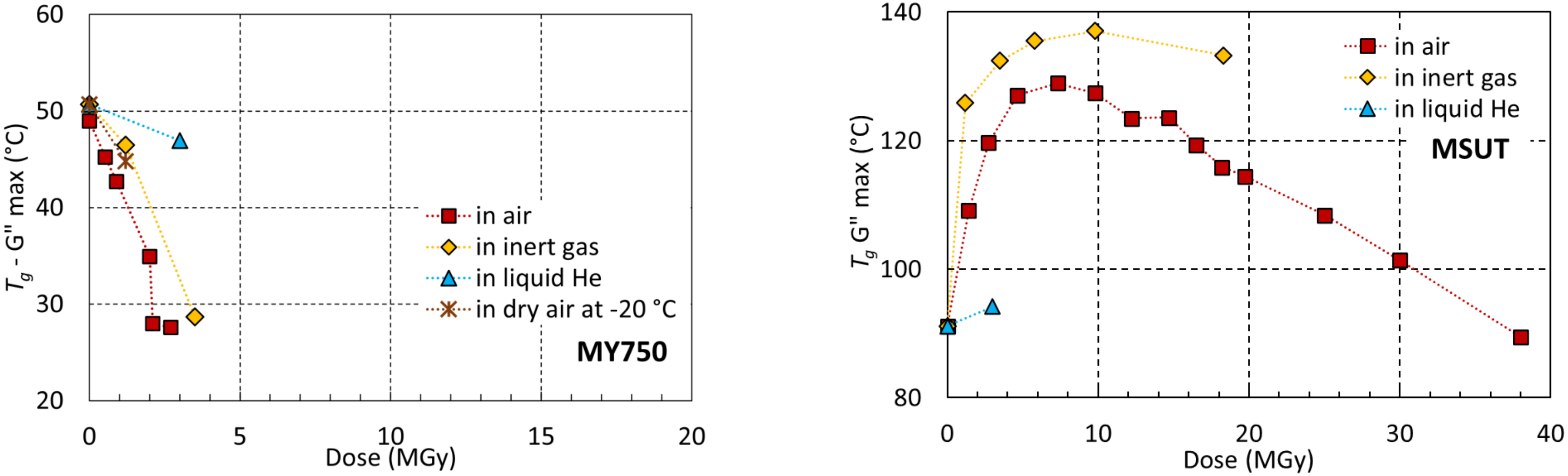


$G'_{rubbery}$ of epoxy resins with anhydride and amine-based hardeners as a function of dose absorbed in ambient air. Full and empty symbols represent 24 GeV/c proton and ^{60}Co gamma irradiation, respectively.

Proton irradiation in ambient air, in inert gas at ambient temperature, and in liquid helium

Influence of irradiation environment and temperature

- Identical materials have been irradiated with 24 GeV/c protons in ambient air, in inert gas, and in liquid helium.
- Compared to ambient air irradiation typically:
  - More cross-linking and less chain scission in inert gas
  - Less cross-linking and less chain scission in liquid He
- To predict the dose limits of organic materials in superconducting magnets, irradiations need to be performed at cold.



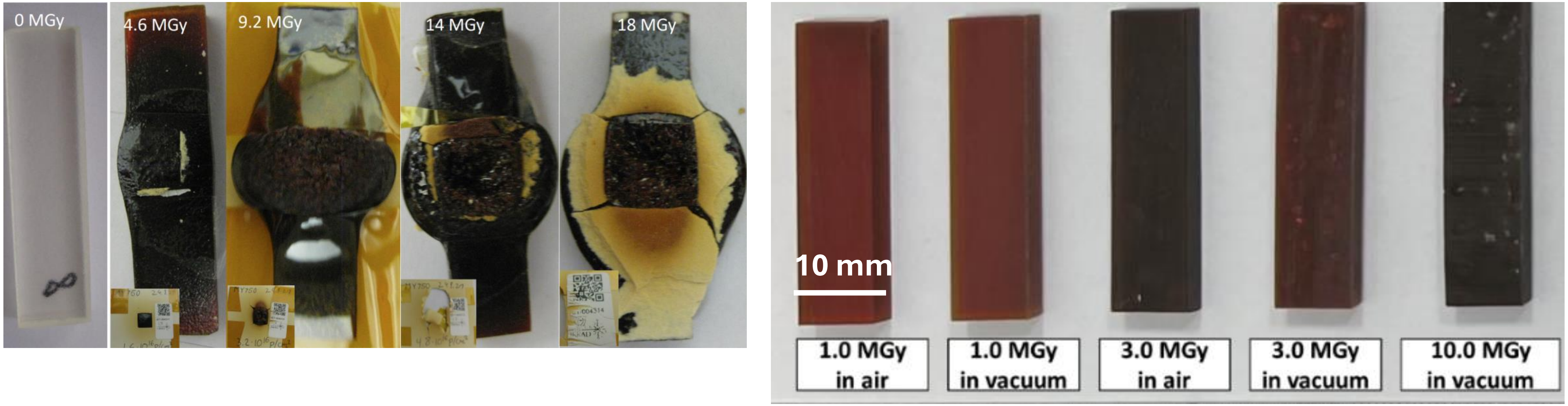
T_g G''$_{max}$ of (a) MY750 and (b) MSUT as a function of proton dose absorbed in ambient air, inert gas and in liquid He.

[15] D.M. Parragh, C. Scheuerlein, N. Martin, R. Piccin, F. Ravotti, G. Pezzullo, T. Koettig, D. Lellinger, "*Effect of irradiation environment and temperature on aging of epoxy resins for superconducting magnets*", Polymers 2024, 16(3), 407

Mixed neutron/gamma irradiation in air and in vacuum

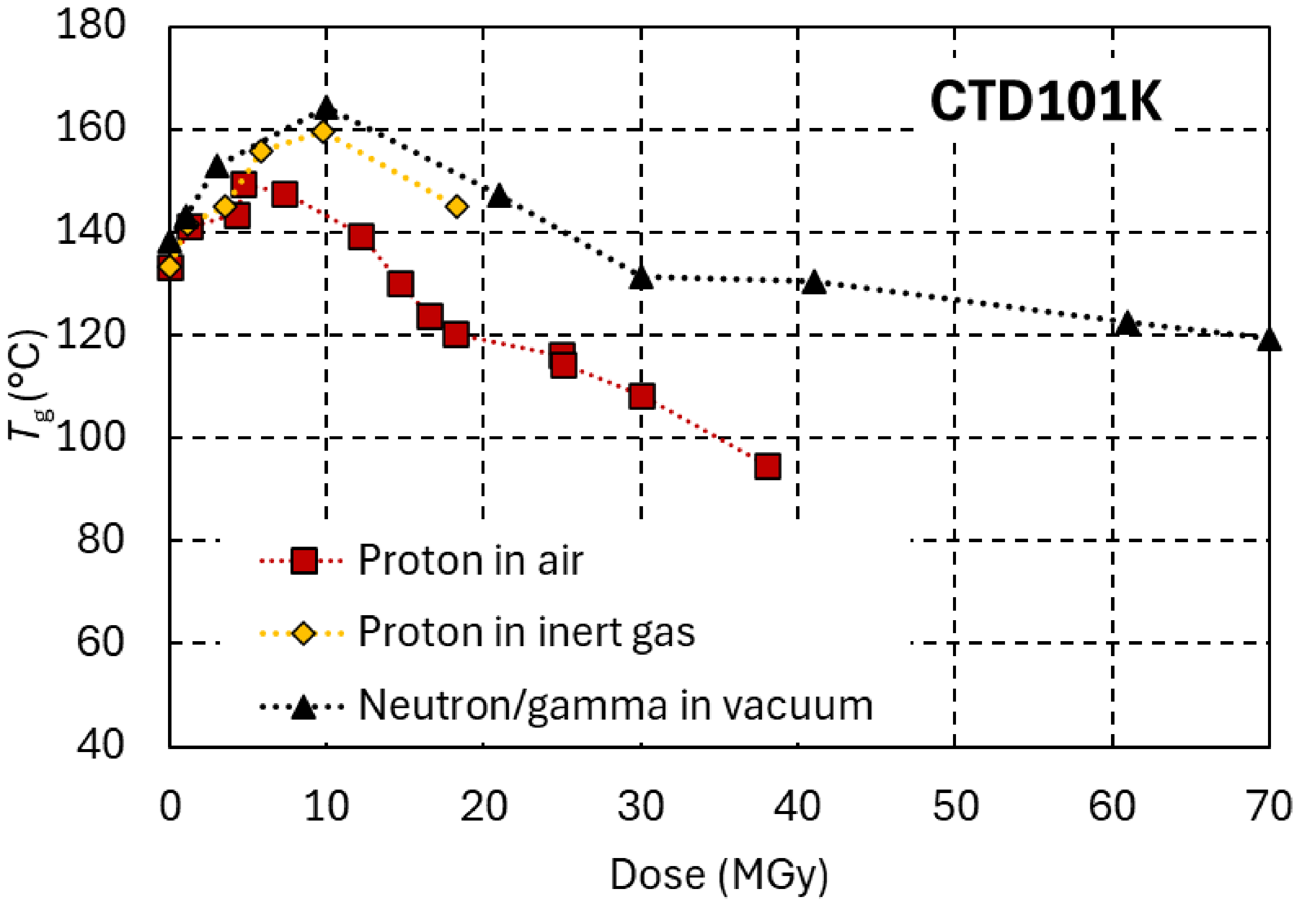
Visual comparison of MY750 epoxy resin system after proton irradiation in air and mixed neutron/gamma reactor irradiation

- The different effect of the same total dose absorbed from the 24 GeV/c proton beam in ambient air (3 kGy/h) and from the mixed gamma/neutron irradiation in the TRIGA Mark II reactor (900 kGy/h) is obvious from the visual comparison of the irradiated samples.



MY750 samples after different doses absorbed (a) from 24 GeV/c protons in ambient air and (b) in the TRIGA Mark II reactor.

Mixed neutron/gamma irradiation in Triga Mark II reactor

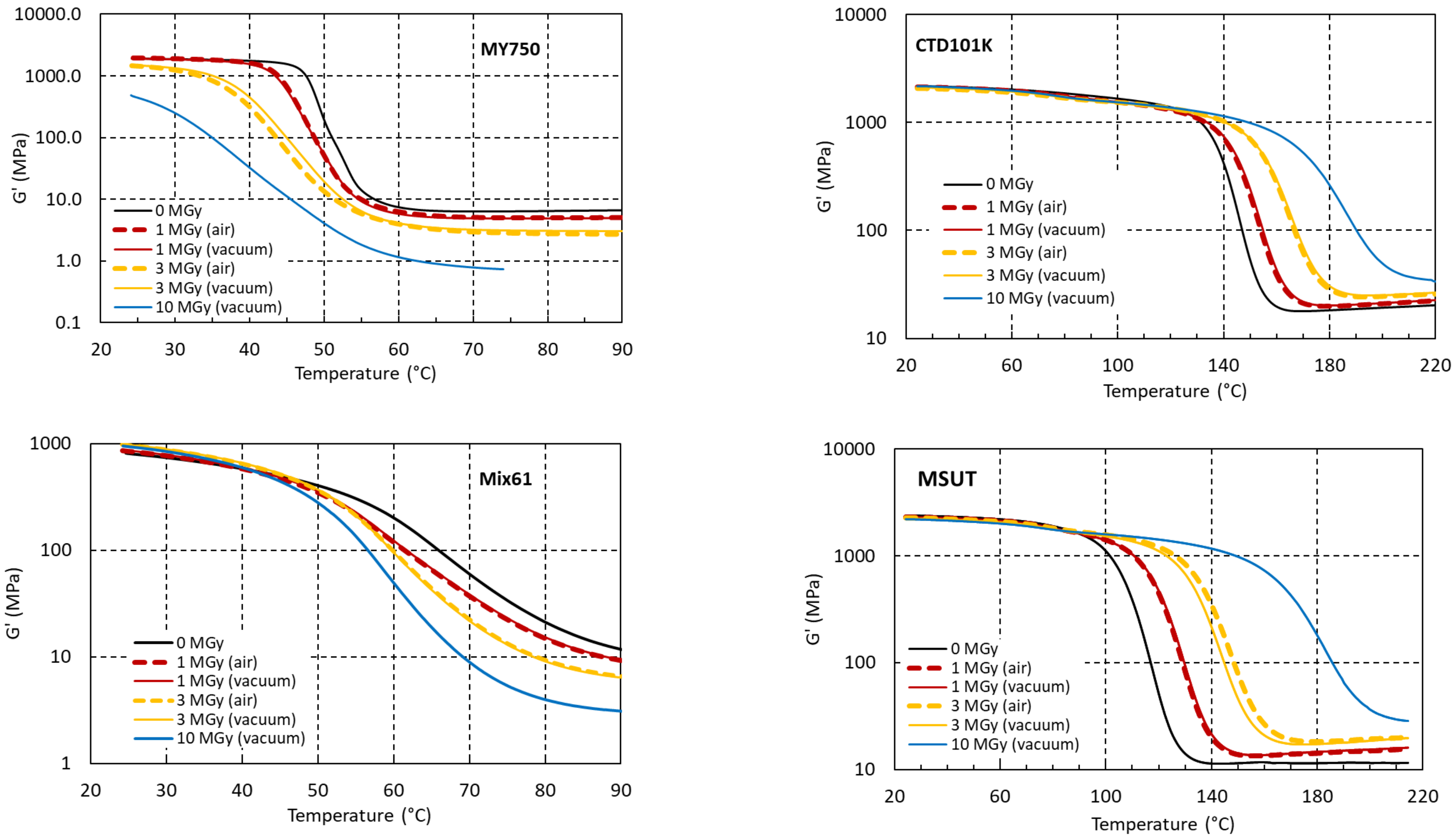


T_g G''$_{max}$ of CTD101K as a function of total dose absorbed during proton irradiation in ambient air and inert gas and, mixed gamma/neutron irradiation in vacuum.

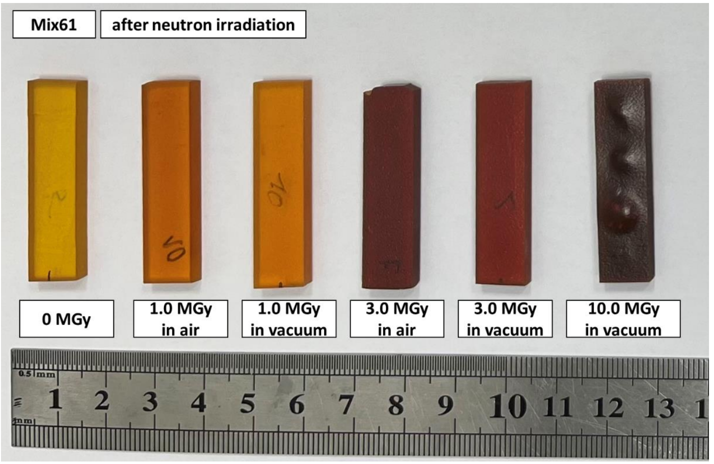
- Mixed neutron/gamma irradiation with about 900 kGy/h at the TRIGA Mark II reactor causes less chain-scission and more cross-linking than proton and gamma irradiation in ambient air with about 3 kGy/h.
- Similar T_g vs dose evolutions during reactor irradiation and proton irradiation in inert gas.
- No significant difference between reactor irradiation in vacuum and in air (see next slide).
- The different effect of proton irradiation in air and reactor irradiation can be explained by the very high reactor dose rate that limits oxygen diffusion.

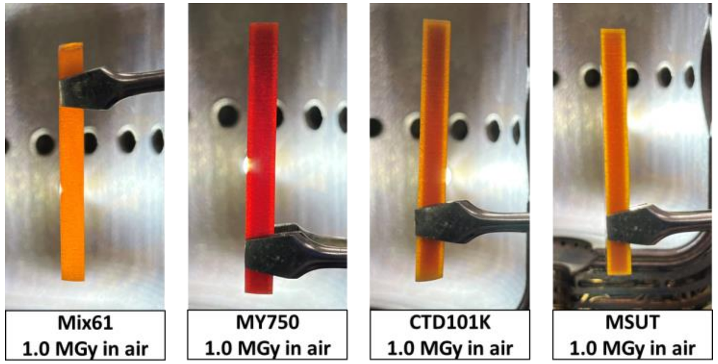
Effect of oxygen during reactor irradiation

- Nearly identical DMA results after reactor irradiation in air and in vacuum of the 4 mm thick samples during 900 kGy/h irradiation to 1 MGy and 3 MGy.
- Visual appearance of the samples after reactor irradiation in air indicates that oxygen diffusion is limited to the sample surface.



G'(T) of different epoxy resins before and after reactor irradiation with 900 kGy/h in vacuum and in air to 1 MGy, 3 MGy and irradiation in vacuum to 10 MGy.

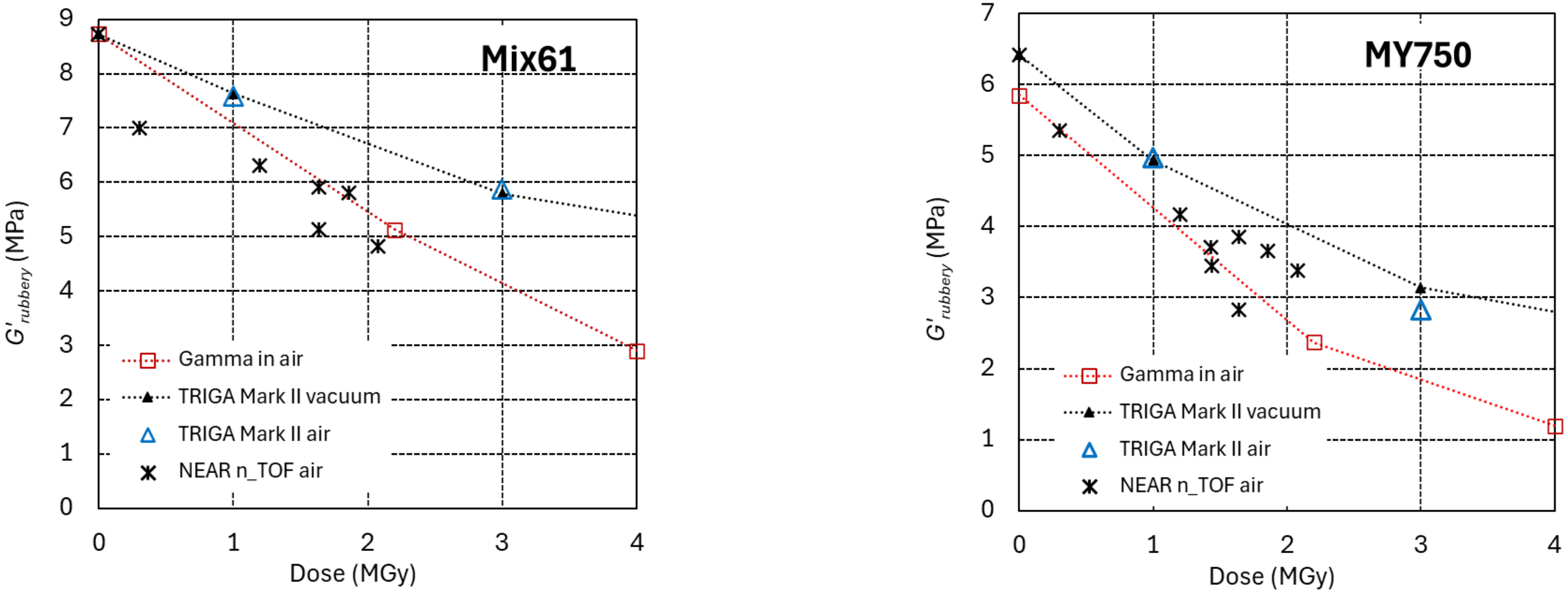




Visual comparison of samples after 1 MGy air irradiation

Effect of neutrons

- At NEAR n_TOF the dose in the epoxy samples is dominated by neutrons (75% of TID from neutrons).
- The comparison between $G'_{rubbery}$ vs dose evolutions during gamma and NEAR n_TOF irradiation indicates that a certain neutron dose causes a similar irradiation damage as the same gamma ray dose.
- Scatter of results is mainly due to dose gradients across the different NEAR n_TOF irradiation locations. Additional NEAR n_TOF data at higher dose levels would be helpful to compare the chain scission efficiencies more precisely.



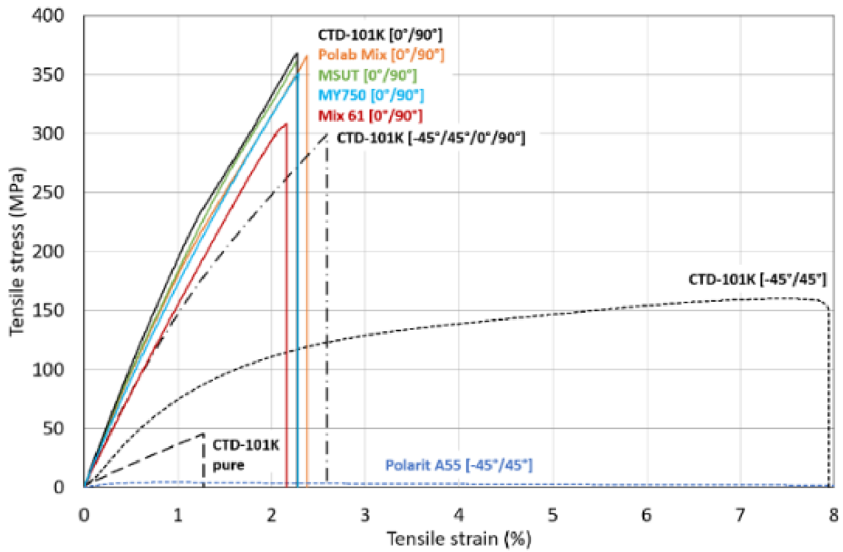
Comparison of the $G'_{rubbery}$ evolution of amine-based epoxy resin systems Mix61 and MY750 as a function of the total dose absorbed in air at a ^{60}Co source and at NEAR n_TOF spallation source, and in air and in vacuum in the TRIGA Mark II reactor.

Dielectric and mechanical requirements of superconducting magnet insulation systems

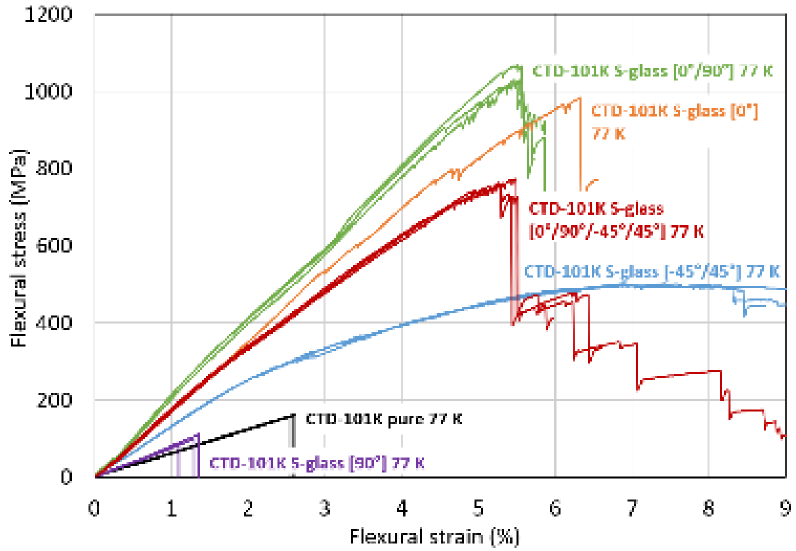
- The most important function of a superconducting magnet insulation system is to maintain a breakdown voltage above the highest voltage that can occur in case of a quench.
- Electrical breakdown caused by irradiation is typically the result of the irradiation induced degradation of mechanical properties.
- Mechanical properties of insulation systems operating in gas or vacuum must assure the absence of cracks in the insulation over the entire live time of the device. Depending on the pressure, breakdown of insulation systems with porosity operating in gas or vacuum can occur at comparatively low voltage (Paschen law).
- In insulation systems immersed in liquid helium cracks and pinholes are filled with liquid helium, which has high dielectric strength.

Mechanical properties of composites for superconducting magnets

- The required mechanical strength of an insulation system depends on the magnet design. In some cases, the mechanical properties of wax impregnated composites are sufficient.
- Defining absolute mechanical strength values in the relevant load directions (instead of a relative change of mechanical strength) that an insulation must maintain to prevent formation of critical defects can help to substantially increase the dose limits.



Tensile stress-strain curves at RT of V_f=33% S2 glass fibre reinforced CTD101K, Polab Mix, MSUT, MY750, Mix61 and paraffin wax [16].



Flexural stress vs strain of S2-glass reinforced CTD101K specimens with fibre orientations [0°/90°], [90°], [0°], [0°/90°/±45°], [±45°], and pure CTD101K at 77K.

[16] J. Bertsch, C. Scheuerlein, P. Wiker, D. Parragh, A. Echtermeyer, R. Piccin, "*Thermomechanical properties of epoxy and wax matrix composites for superconducting magnets*", accepted for publication in ICEC/ICMC 2024 proceedings

Conclusion and outlook

- For the materials of this study, the same dose absorbed from gamma and proton irradiation with the same dose rate and in the same environment has the same effect. Neutron irradiation has a similar effect, within the experimental uncertainties. TID is a good scaling factor to compare radiation damage from different sources.
- Neutron/gamma reactor irradiation of thick samples with dose rates in the order of 1000 kGy/h (regardless if in vacuum or in air) causes less damage than irradiation in ambient air with dose rates in the order of 1 kGy/h.
- The irradiation temperature can have a strong effect on chain scission and cross-linking rates. To determine the dose limits of organic materials in superconducting magnets irradiations at cryogenic temperature are needed.
- It is likely that radiation hard insulation systems in superconducting magnets can be used up to doses exceeding 100 MGy, requiring irradiation testing at cryogenic temperature to similarly high doses.
- Design and construction of a cryocooler cooled set-up for proton irradiations in vacuum at <30 K is ongoing. Installation and test in the IRRAD facility is planned in 2025.

Thank you for your attention

Extra slides

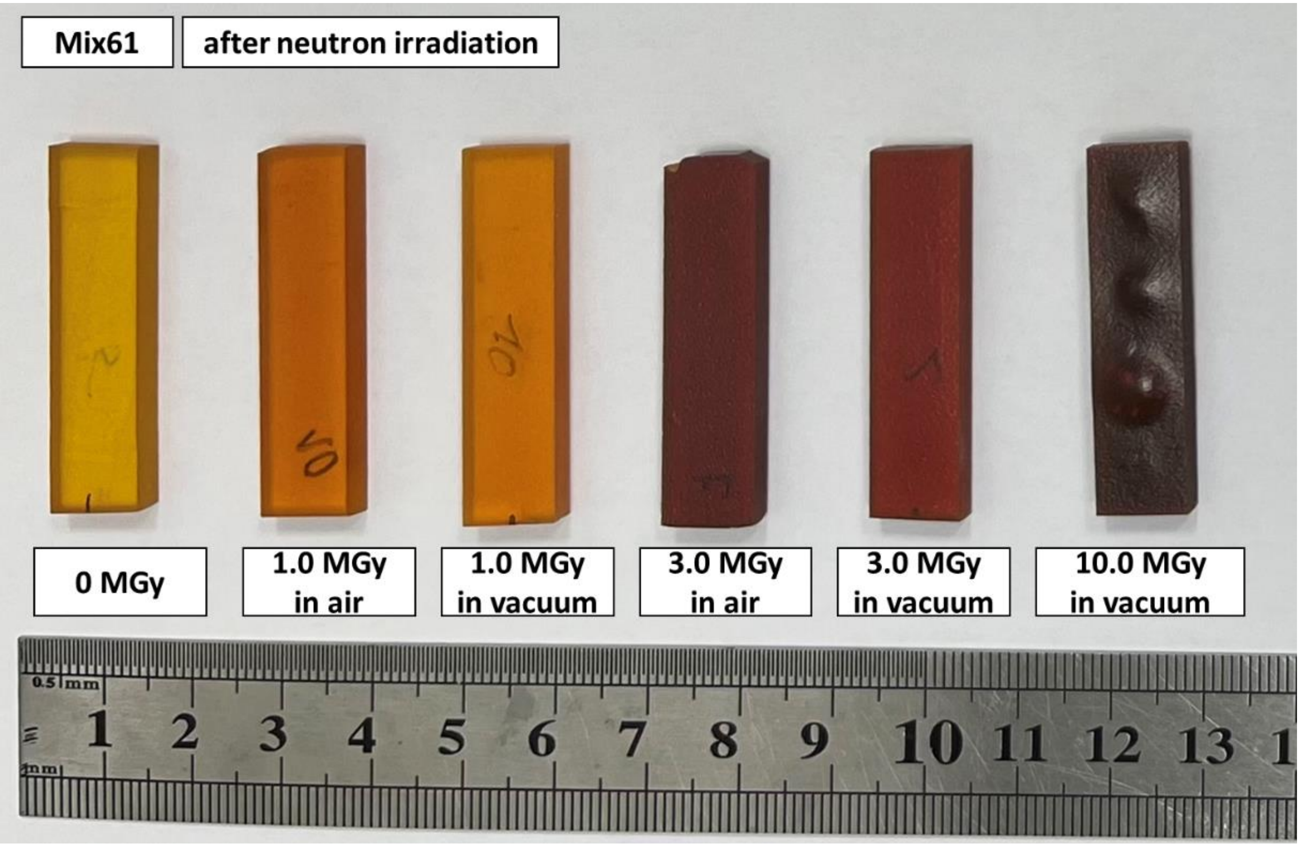
Epoxy resin systems studied

- We study commercially available and widely used epoxy resin systems with curing cycles optimised to achieve highest possible conversion, and epoxy resin systems that are used for superconducting magnet coil impregnation with the processing parameters used by the magnet builders.

1) **CTD101K:** baseline impregnation system for the HL-LHC superconducting magnets, consists of diglycidyl ether of bisphenol-A (DGEBA), a carboxylic anhydride hardener, and an accelerator, all of which are supplied by Composite Technology Development Inc. (U.S.A.). These components are mixed in the proportion CTD101K resin : hardener : accelerator = 100 parts by weight (pbw): 90 pbw : 1.5 pbw. The curing temperature cycle comprises two plateaus 5 h-110 °C and 16 h-125 °C post-curing.
2) **MSUT:** Consists of the Araldite MY740 bisphenol A / epichlorohydrin resin (type DGEBA, Mw < 700 g/mol), cured with the carboxylic anhydride hardener of mixed composition Aradur HY906, and the amine accelerator DY062 in the respective ratio 100 pbw: 90 pbw: 0.2 pbw. The curing temperature cycle comprises two plateaus 4 h-85 °C and 16 h-110 °C post-curing.
3) **MY750:** Composed of the Araldite MY750 bisphenol A / epichlorohydrin resin, type DGEBA, Mw < 700 g/mol (100 pbw) and the aliphatic polyamine hardener Aradur HY5922 (55 pbw) from Huntsman Corporation. The curing temperature cycle comprises two plateaus 6 h-40 °C and 3 h-80 °C post curing.
4) **Mix61:** Composed of a diglycidyl ether of bisphenol-A (DGEBA) resin, an aromatic hardener of the amine type, a high molecular weight co-reactant of the amine type and a liquid low molecular weight additive. The curing temperature cycle comprises two plateaus 16 h-60 °C and 24 h-100 °C post curing.
5) **CEA mix:** Two-component resin system Huntsman Araldite® CY192-1, a cycloaliphatic epoxy resin (100 pbw), and its corresponding anhydride type hardener, Huntsman Aradur® HY 918-1 (100 pbw), has been used by CEA Saclay for the impregnation of Nb3Sn quadrupole coils. The applied curing cycle recommended by CEA Saclay comprises three isothermal plateaus as follows: 80 °C-24 h, 120 °C-34 h+ 130 °C-12 h.
6) **Araldite F:** used by the company ASG Superconductors S.p.A. for impregnation of magnet coils and consists of the bisphenol A / epichlorohydrin resin (type DGEBA) Araldite F, the carboxylic anhydride hardener Aradur HY 905, and the polyglycol flexibilizer DY 040. The resin, hardener, and flexibilizer are combined in the ratio of 100 pbw : 100 pbw : 10 pbw, respectively. The Araldite F/Aradur HY 905/flexibilizer DY 040 system does not contain the accelerator DY 061, and filler that is recommended by Huntsman. The applied curing cycle comprises two plateaus 100 °C-10h+ 135 °C-48 h.
7) **POLAB Mix:** CERN Polymerlab development. Araldite DY040 is a solvent-free and low-viscous hot-curing flexibiliser liquid used for medium- or high-voltage insulators. POLAB Mix is produced with 10 wt.% DY040 relative to CTD101K epoxy resin. First the CTD101K epoxy resin and hardener are mixed and degassed, and then DY040 is added. This mix is again degassed before the accelerator is added, and a final degassing is executed. The curing temperature cycle comprises two plateaus 5 h-110 °C and 16 h-125 °C post-curing.
8) **Damisol 3418**: One component class H high voltage epoxy resin system from Von Roll. It is a solvent free DGEBA epoxy/anhydride hardener system that can be processed at RT. The curing cycle comprised 4 h-120 °C and 8 h-160 °C plateaus.
9) **Stycast 2850FT-23LV**: Charged epoxy resin for cryogenic applications, with 23LV Polypropylene Glycol Diamine, 3,3'-Oxybis(Ethyleneoxy)Bis(Propylamine) hardener. Because of its particle charge it has comparatively high thermal conductivity at cryogenic temperature, and its coefficient of thermal expansion (CTE) is 44×10-6 K-1, which is about 25% lower than the CTE of about 60×10-6 K-1 of pure epoxy resins.
10) **CTD 425**: Two-part system consisting of an epoxy resin (EP) and a cyanate ester (CE) catalyst. 60 parts EP and 40 parts CE were mixed and degassed at 50 °C. The curing temperature cycle comprised two isothermal plateaus 22 h-100 °C and 24 h-150 °C. Because of its comparatively higher irradiation resistance CTD 425 is used for the impregnation of the ITER toroidal field coils.

Gas evolution during irradiation

- Gas evolution was measured after 10 MGy combined neutron and gamma irradiation under vacuum in the TRIGA Mark II reactor of TU Vienna.
- The CTD101K and MSUT epoxy resins exhibit about three times less outgassing as compared to the MY750 and Mix61 epoxy resins.
- After 10 MGy neutron irradiation, bubbles have formed in the Mix61 and MY750 samples (but not in CTD101K and MSUT) [5].



Mix 61 samples after irradiation in air and in vacuum. Bubbles can be seen in the sample after 10 MGy irradiation.

[5] D.M. Parragh et al, https://ieeexplore.ieee.org/document/10319395

Dielectric strength testing

- Pure epoxy resins have typical DC breakdown strength in the order of 100 kV/mm (e.g. CTD101K 199±11 kV/mm, Polab Mix 203±14 kV/mm [17]).
- Fibre reinforced epoxy composites dielectric strength is usually reduced with respect to that of the pure epoxy.
- HV testing in air requires large samples to avoid flash-over.
- HV testing in oil or liquid nitrogen avoids flash-over. Sample size can be reduced (Ø=40 mm disks, 0.5 mm thick).
- Irradiation of pure epoxy and composite Ø=40 mm disks for dielectric testing is ongoing.

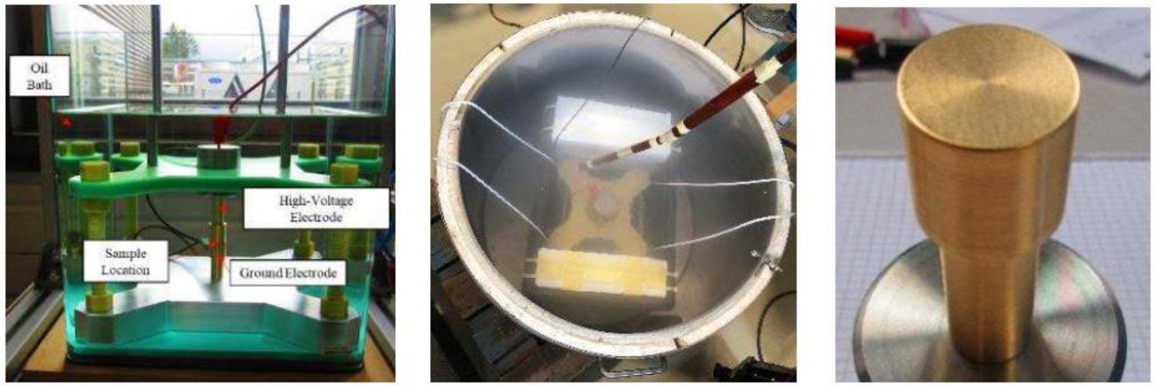
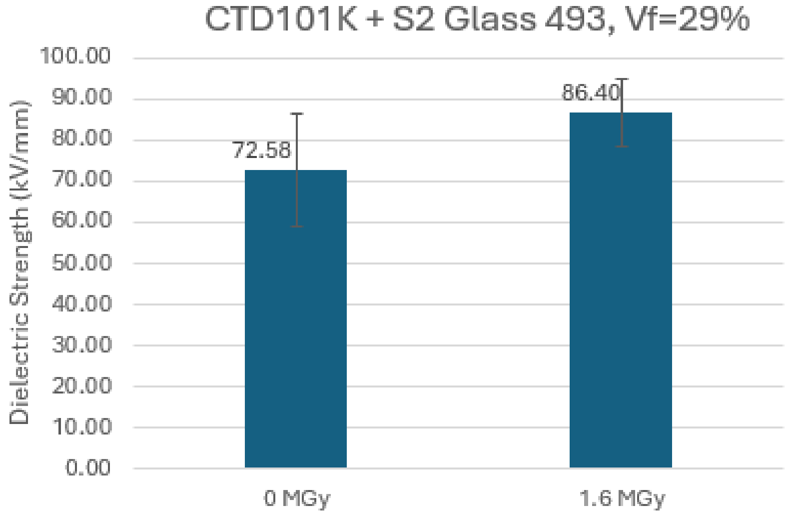


Figure 1: DC Breakdown Test Equipment (a) in Oil and (b) in Liquid Nitrogen. (c) Close-up of Brass Electrode

[17] J. Osuna et al, “Advanced Composite Insulation Systems for Niobium-Tin Superconducting Magnets: Electrical Characterization of Laminates at Cryogenic Temperatures”, accepted for proceedings of ICEC29/ICMC2024.



Comparison of dielectric strength of CTD101K-S2 glass composite before and after irradiation. Courtesy Dante Polvani.

Irradiation damage: comparison epoxy resins vs thermoplastic polycarbonate

- Effect of 3.6 MGy gamma radiation in ambient air with 2 kGy/h on flexural strength:
    - MSUT epoxy resin: negligible effect
    - Araldite F epoxy resin: about 30% reduction of flexural strength
    - Polycarbonate: drastic reduction of mechanical properties

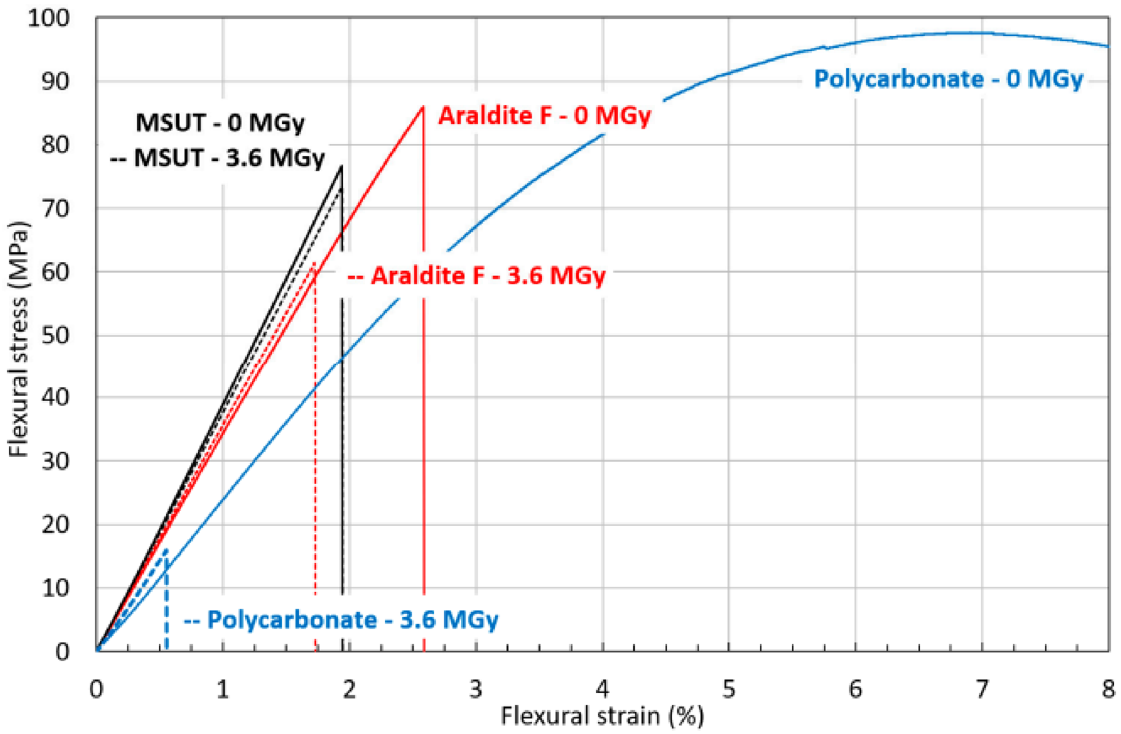
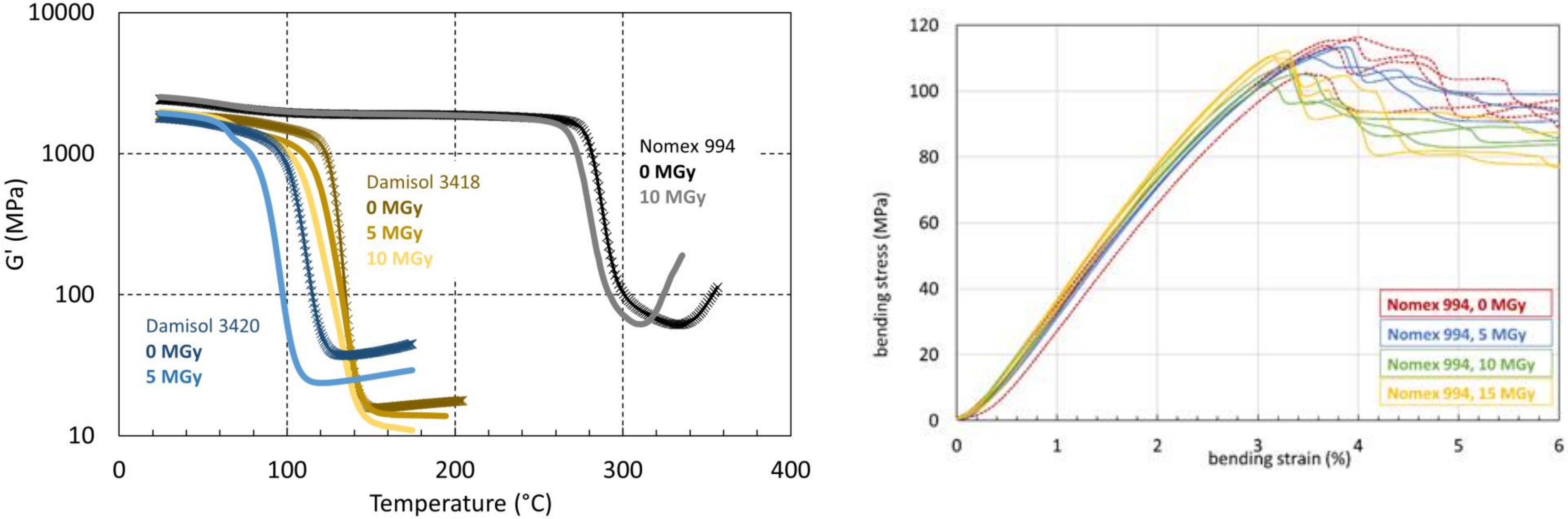


Figure 16. Comparison of RT flexural stress–strain curves of MSUT, Araldite F, and PC before and after 3.6 MGy gamma irradiation. The irradiated material stress–strain curves are represented by dashed lines.

Polymers considered for PSB booster magnets

- Irradiation hardness of the one component low viscosity epoxy resins Damisol 3418 and Damisol 3420 and of Nomex 994 pressboard are studied in the context with the construction of new PCB booster magnets.
- Nomex 994 has outstanding radiation hardness (almost unchanged up to 15 MGy in ambient air). Irradiations up to 30 MGy in ambient air are ongoing.

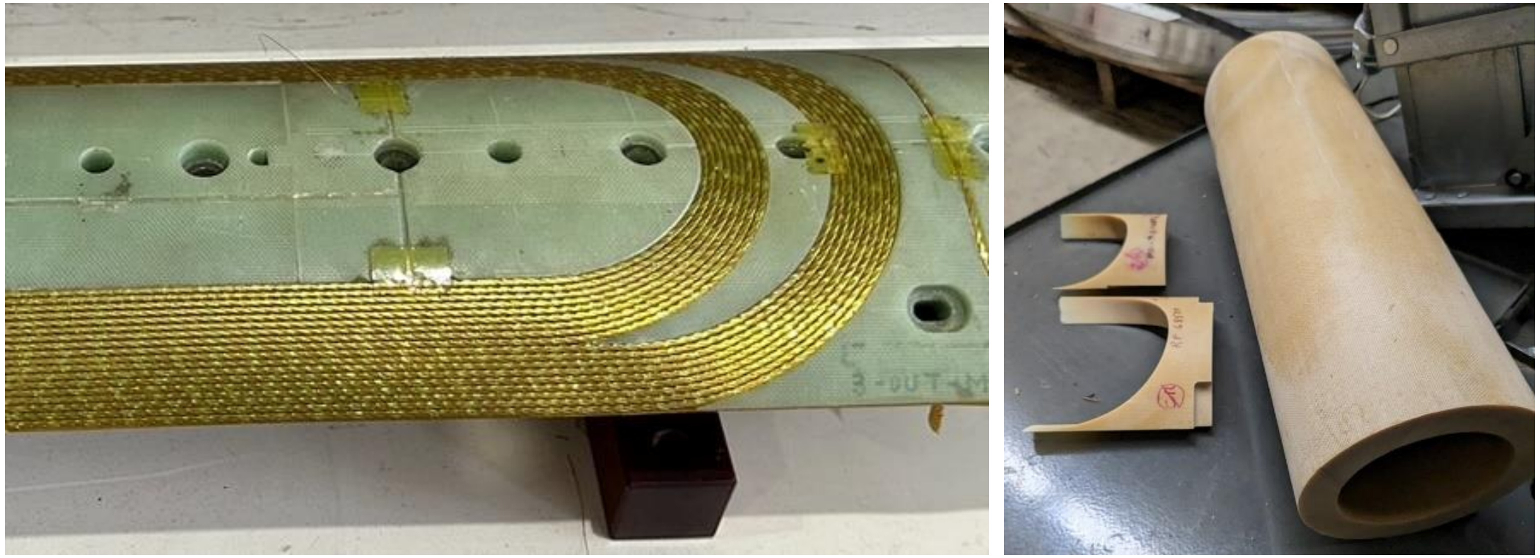


(a) G'(T) of Damisol 3418 and Damisol 3420 of Nomex 994 and (b) flexural stress strain of Nomex 994 before and after irradiation in ambient air.

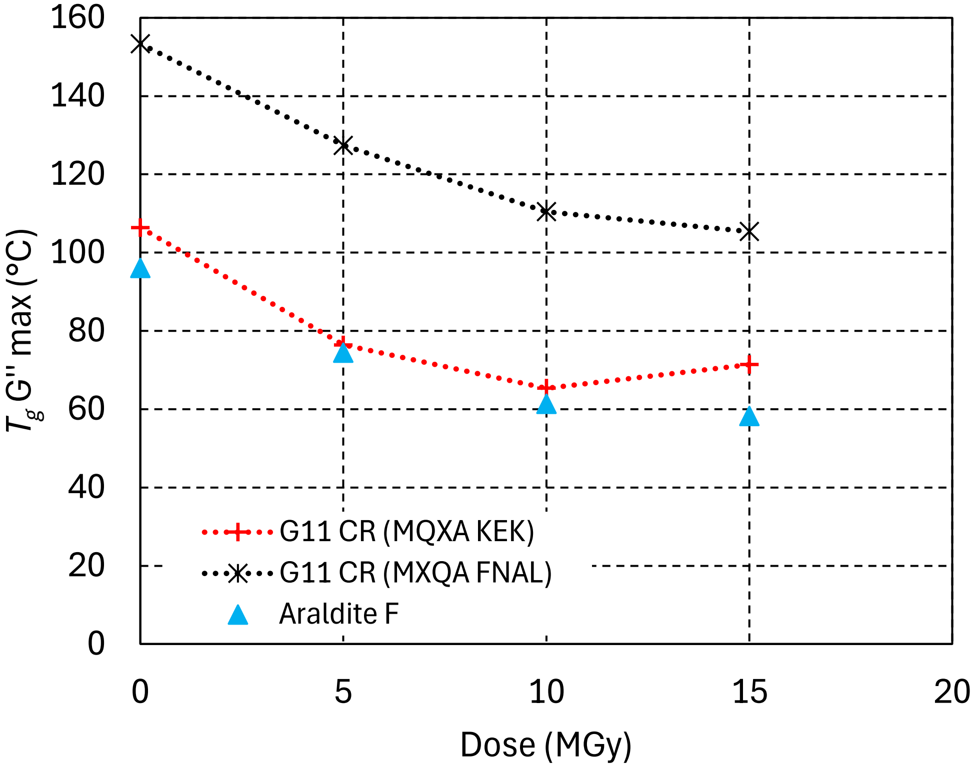
LHC and HL-LHC superconducting magnet constituents

LHC MQXA coil end spacers

- Goal: Confirm the dose limits of the wedges and spacers installed in the LHC MQXA magnets from FNAL and KEK (study is performed in the frame of the LHC Triplet Task Force)
- Up to 15 MGy in ambient air, the chain scission rate in the epoxy matrix of both EP GC3 (G11-CR) composites is comparable to that of Araldite F irradiated in ambient air with the same dose rate.
- Gamma irradiation to 30 MGy is ongoing.



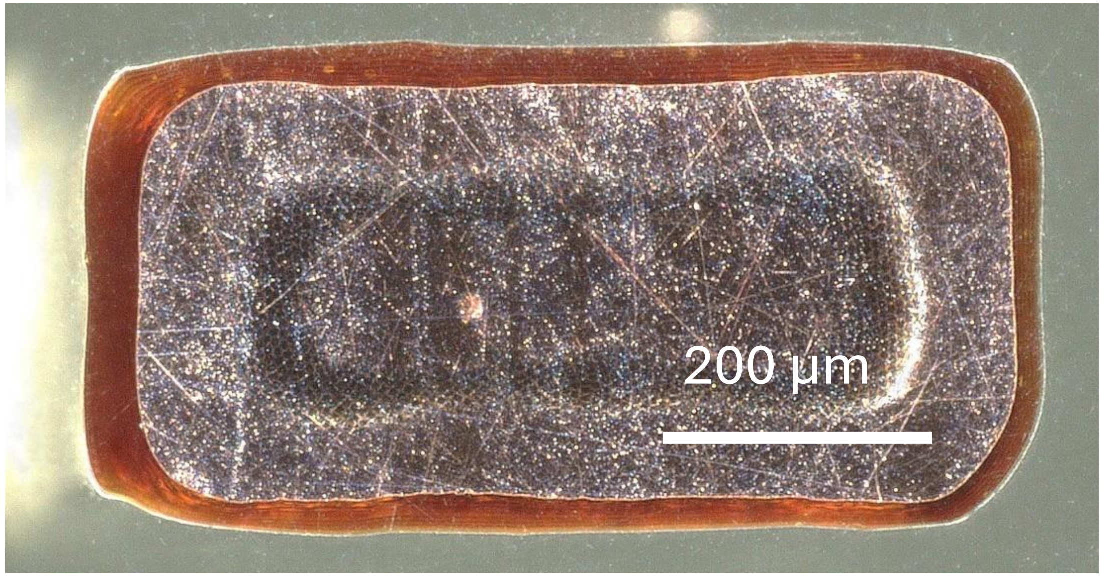
LHC MQXA inner triplet magnet endspacers made of EP GC3 (also known as G11-CR) (a) from KEK and (b) from FNAL.



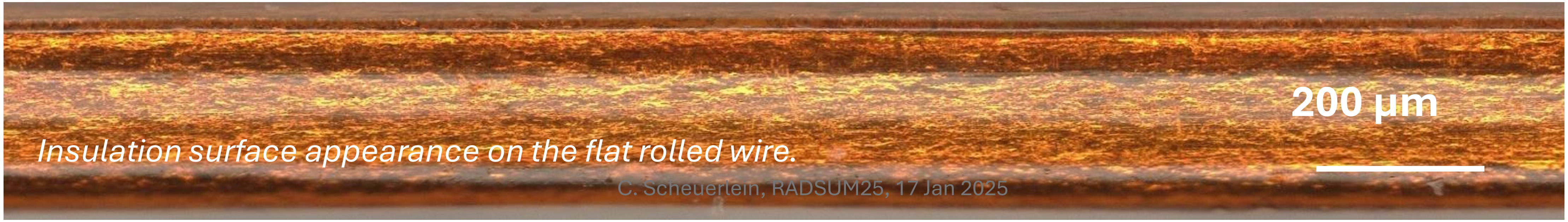
T_g as a function of the gamma dose absorbed in ambient air.

PVA enamel insulated Nb-Ti/Cu wire

- PVA insulation is applied on the round wire and afterwards the wire with insulation is rolled to the final rectangular shape.



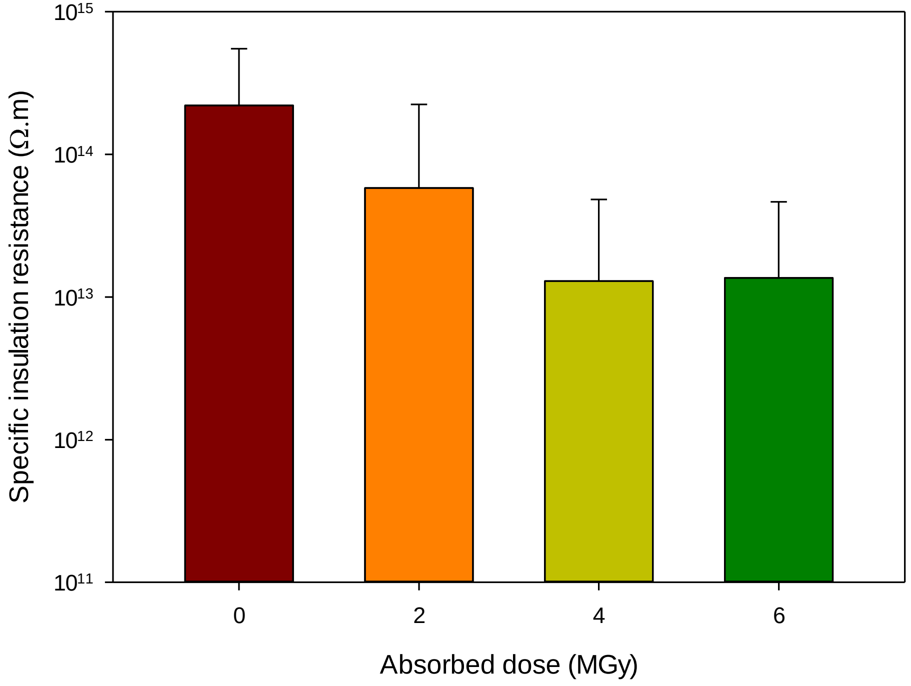
Flat rolled wire cross section. Courtesy of S. Pfeiffer, EN-MME.



Insulation surface appearance on the flat rolled wire.

PVA insulation resistance

- According to the IEC/IEEE 62582-6 part 6 “Insulation resistance standard”
- Set-up consists of picoamperemeter (Keysight B2981A) and a voltage generator Keithley 2290E-5 5kV).
- DC voltage was applied to the conductor while the current was recorded through the external painted electrode.
- Leakage current values are acquired at 500 V until the reaching of the steady-state value or after 24h from the beginning of the test.
- Leakage current measurements indicate a significant reduction of insulation resistance after 2 MGy and a further reduction after 4 MGy.

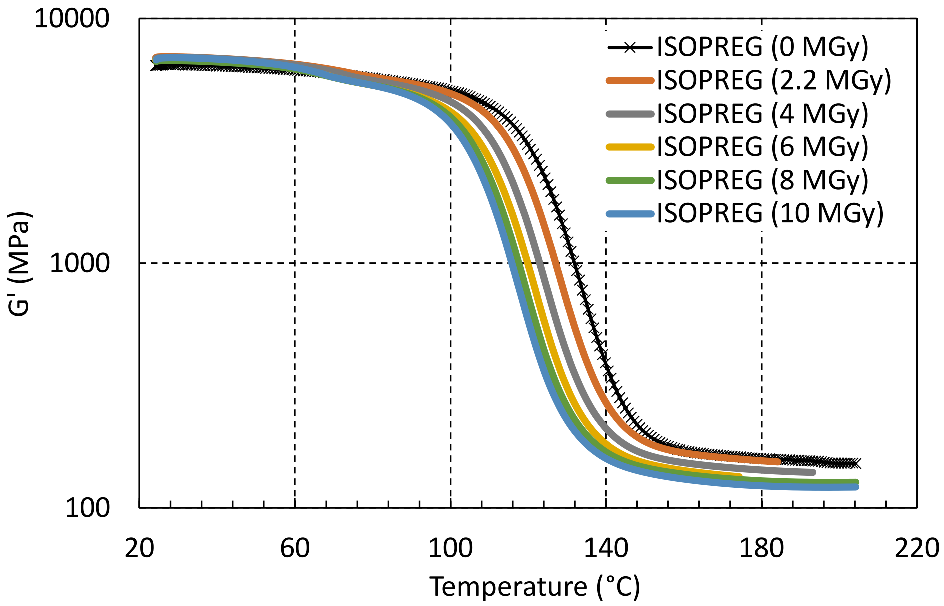


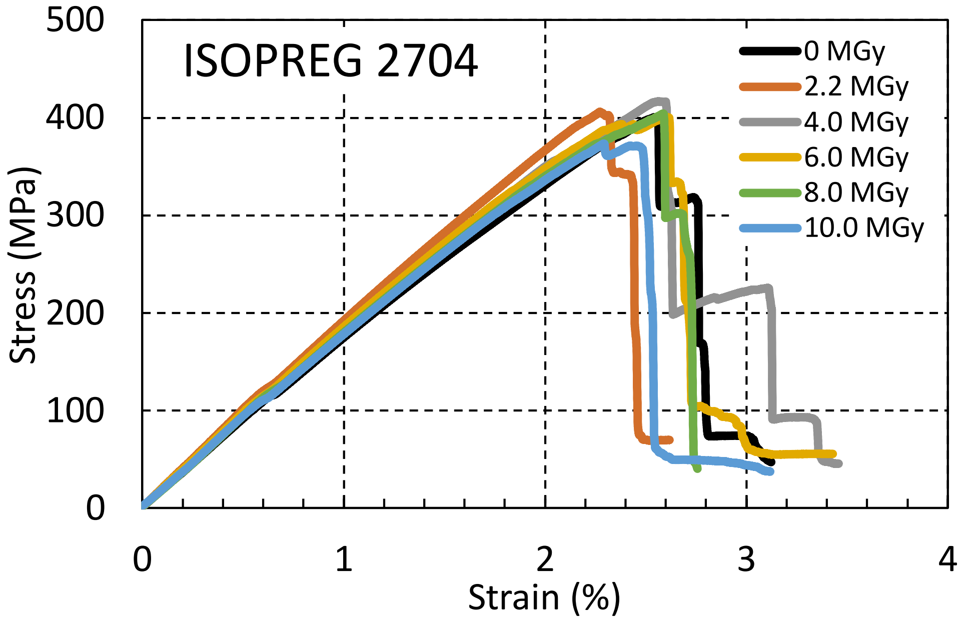
Specific insulation resistance before and after gamma irradiation up to 6 MGy [18].

[18] S.V. Suraci, R. Piccin, J. Osuna, C. Scheuerlein, D. Fabiani, “*Radiation aging effect on electrical properties of superconductive magnet wires*”, 2024 IEEE 5th International Conference on Dielectrics (ICD)

ISOPREG 2704 flexural test results

- ISOPREG 2704 sheet from Isovolta, 15 h-120 °C curing, glass fibre volume fraction V_f=60 %, [0,90] fibre orientation (half of the fibres are oriented in the load direction).





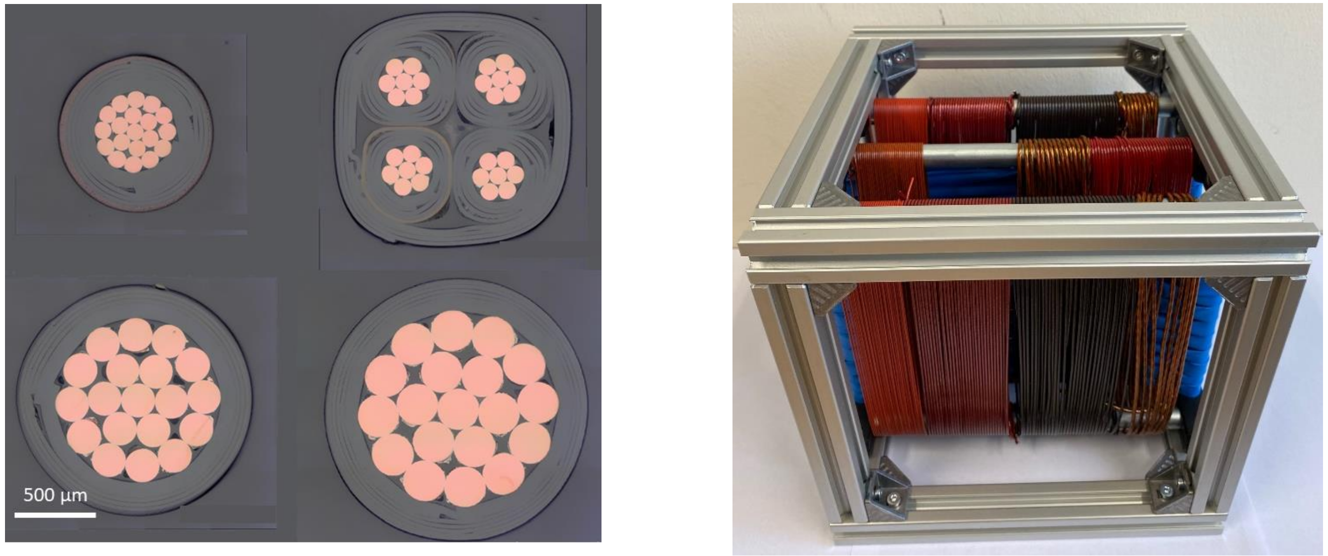
(a) Storage modulus (G') and flexural stress-strain curves of ISOPREG 2704, V_f=60 %, [0,90] *after different dose levels.*

- After 10 MGy T_g is reduced by 16 °C with respect to T_g of the unirradiated sample.
- In the [0,90] test configuration the fibre mechanical properties dominate the composite properties.
- No significant effect of 10 MGy ^{60}Co gamma irradiation on the mechanical properties of ISOPREG 2704, (V_f=60 %, [0,90]) [19].

[19] D.M. Parragh, "Effect of gamma irradiation on the thermomechanical properties of MCBY corrector magnet constituent material ISOPREG 2704, CERN Polymer lab test report, EDMS No. 2816963, (2023)

HL-LHC instrumentation wires

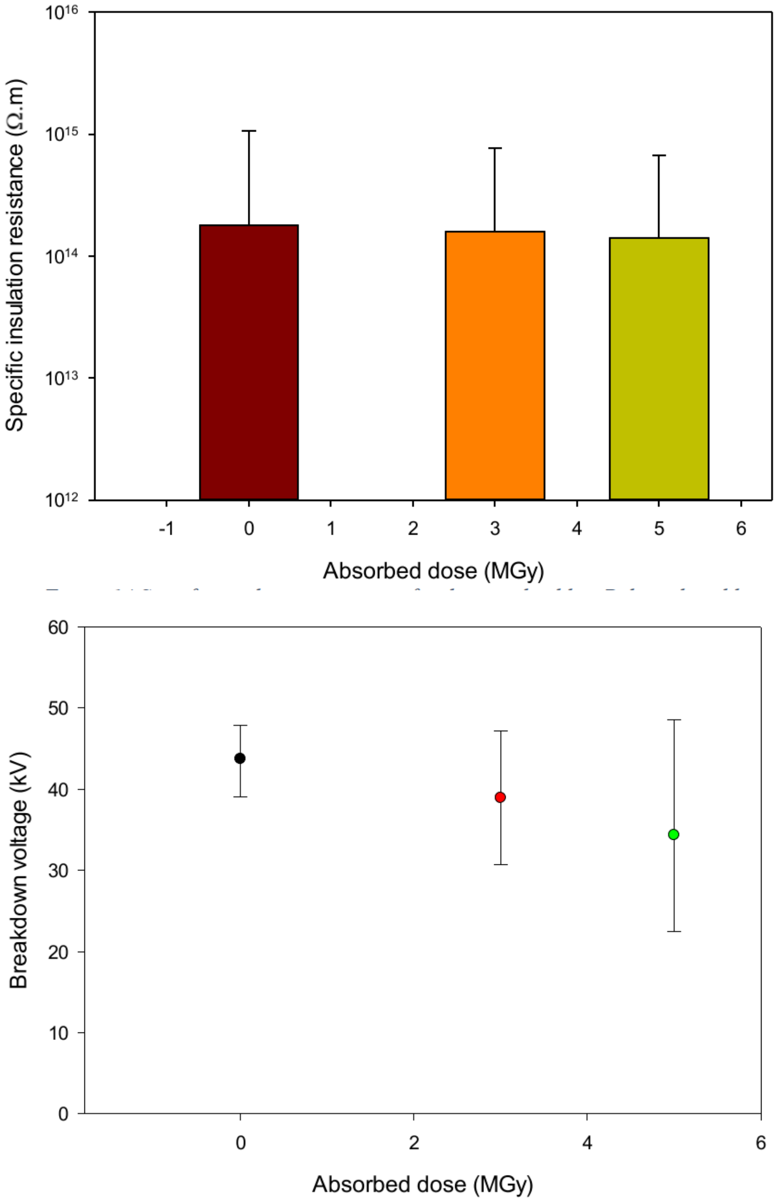
- HL-LHC instrumentation wires have been tested after gamma irradiation up to 5 MGy in ambient air (maximum dose of the wire insulation expected in HL-LHC).
- No significant effect on polyimide insulation resistance
- After 5 MGy average breakdown voltage in air remains >30 kV.
- What is the dose limit of the polyimide wire insulation and its adhesive?



(a) Instrumentation wire cross sections and (b) wires on sample holder for irradiation

[20] Axon, "Equipment wires and cables for high temperatures," Axon Cable, September 2019. [Online]. Available: https://www.axon-cable.com/publications/WIRES-CABLES.pdf

[21] G. Brun, "Supply of Polyimide-Insulated Instrumentation Wires for the LHC Magnet Cold Masses", CERN LHC-MB__A-CI-0009 Technical specification, EDMS No. 304209, (2001), https://edms.cern.ch/ui/file/304209/1/it2904description.pdf



(a) Specific insulation resistance and (b) breakdown voltage before and after irradiation up to 5 MGy. Courtesy S.V. Suraci. University of Bologna.