Main results of the polymer irradiation studies for the ITER project at ATI Vienna

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Outline

Introduction: ITER-Requirements

Geometry scaling

Temperature Effects / Radiation Environments

Results

Conclusions

Introduction

One of the most critical component of the magnet in a radiation environment

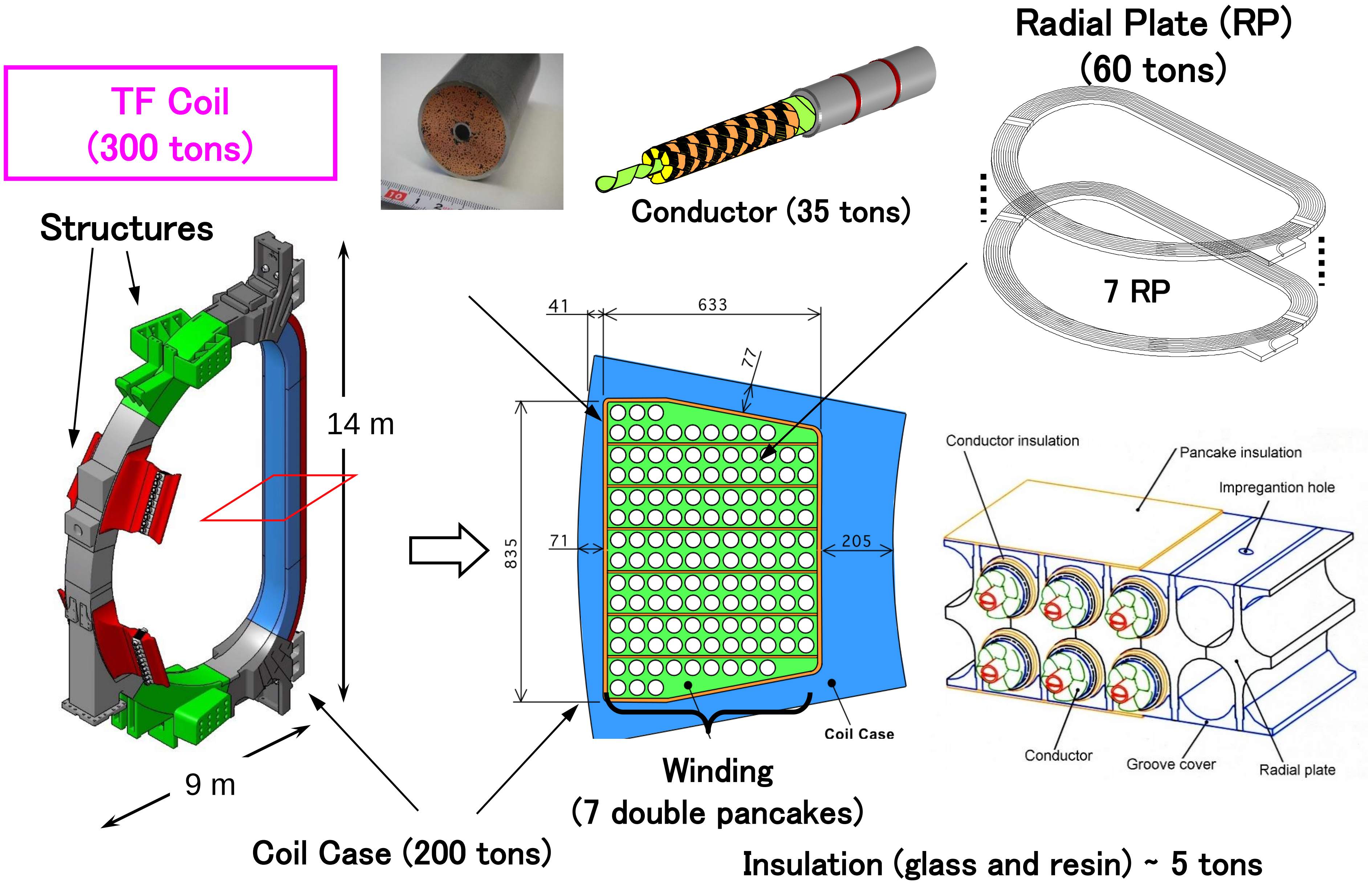
Has to provide electrical insulation

Has to provide mechanical strength and to withstand thermal contraction / expansion and Lorentz forces

Must be suitable for a vacuum-pressure impregnation process – “pot life”

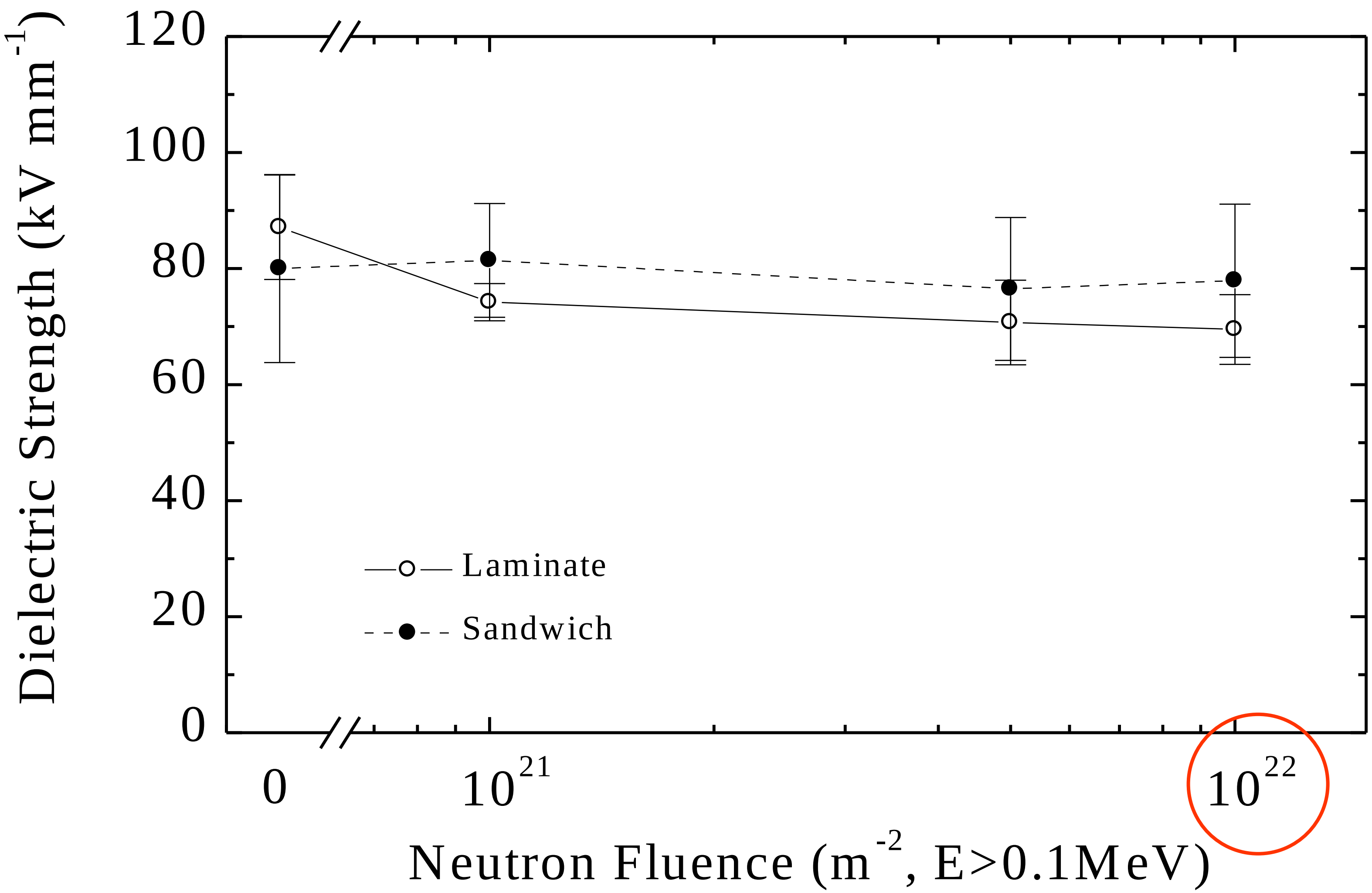
Should not change geometry (gas production, swelling)

TF Coil Design



Electrical properties

Dielectric strength at 77 K

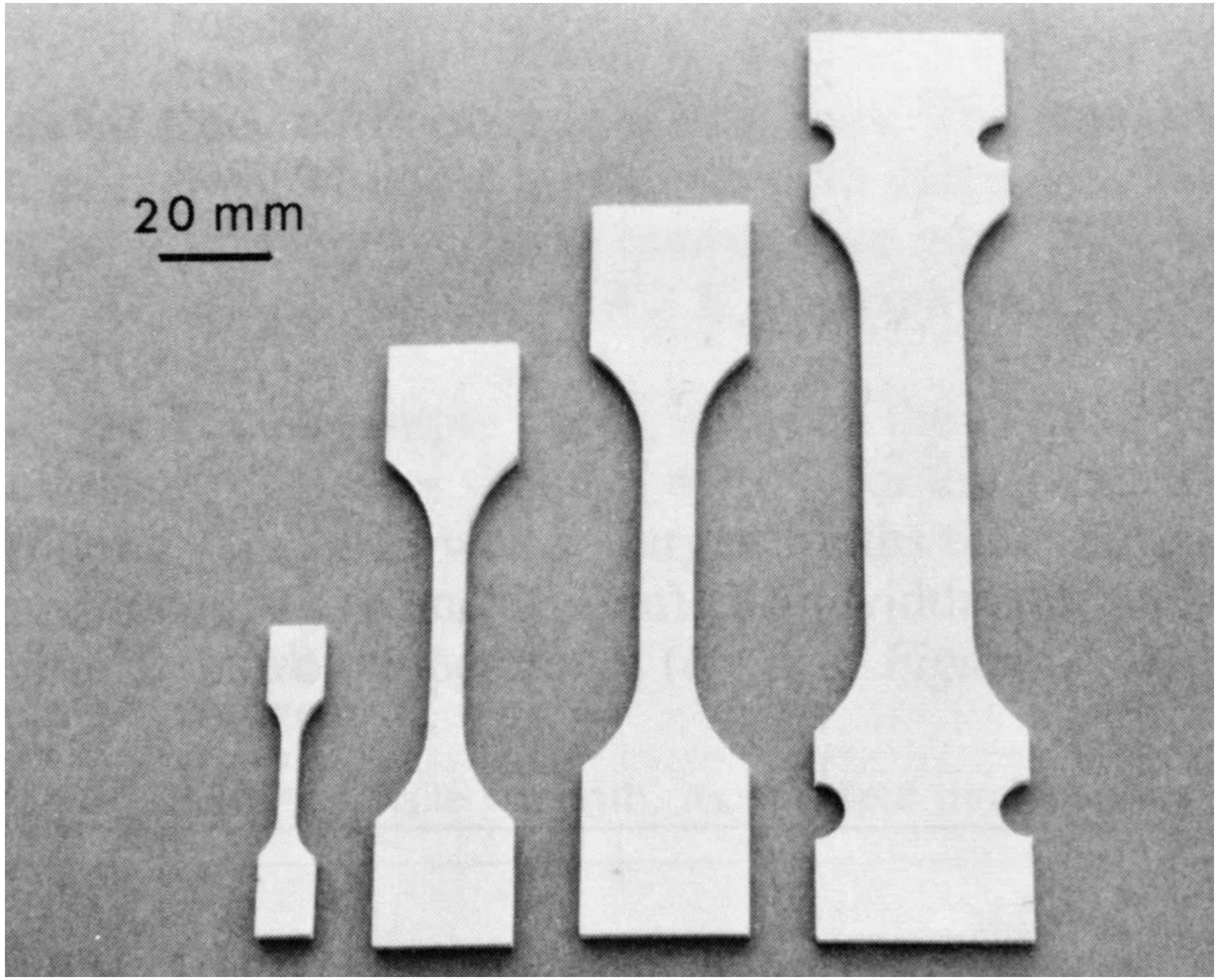


ITER

Araldite F (epoxy)

Mechanical properties

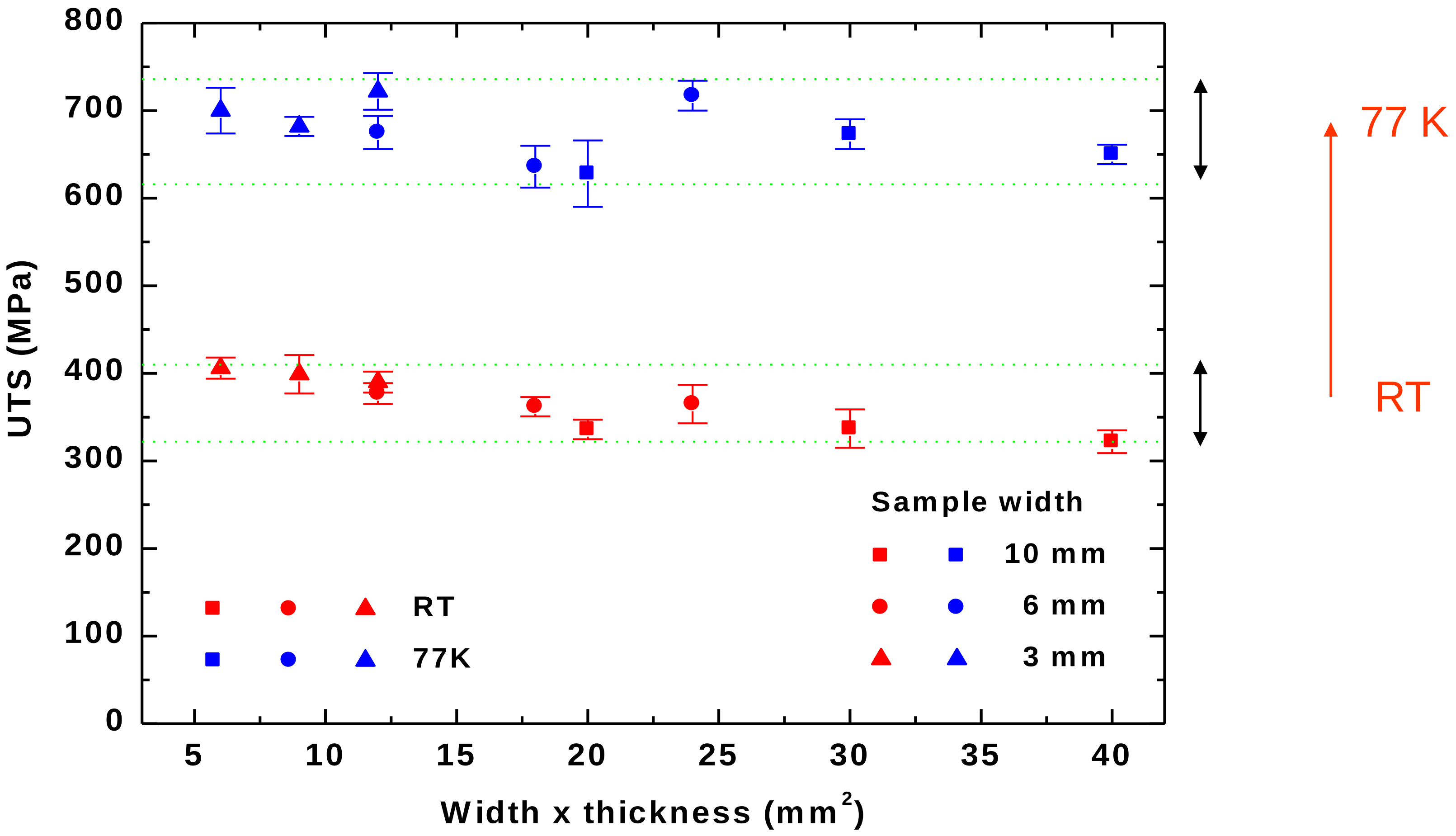
Tensile test specimen geometries (scaling experiments)



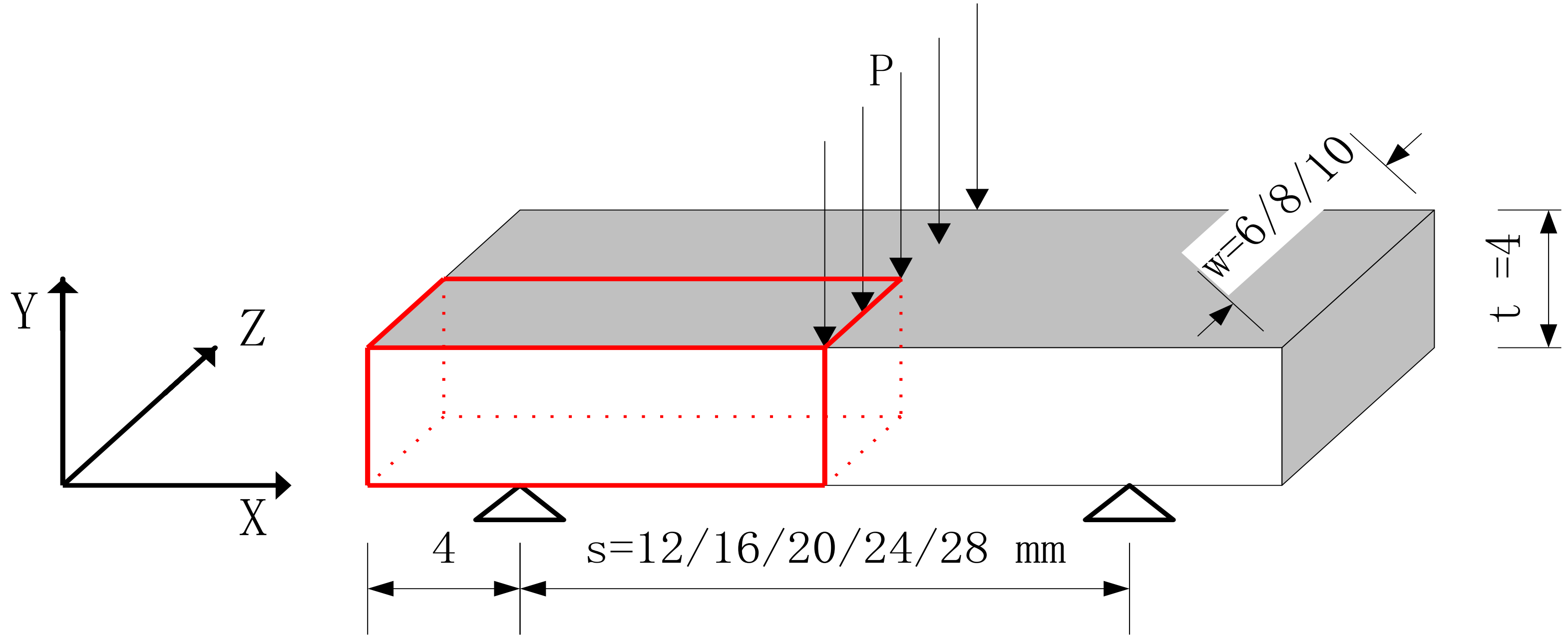
Small sample geometry for irradiation and low temperature experiments

Scaling under static load in tension

(2D E-Glass/Epoxy)



Short-beam-shear test



Important: ILSS depends on span-to-thickness ratio !

Not 100 % pure shear !

➔“apparent ILSS”

➔ FE correction needed!

Radiation effects on insulators

Neutrons — directly deposited energy by the entire neutron spectrum
(via computer codes and damage parameters for
each constituent of resin, i.e. H, C, O, N, ...)
production of H, He → gas production

γ-rays — Dose rate (Gy/h) times irradiation time (h)

⇒ Total absorbed energy (Gy)

⇒ Scaling quantity ?

Element	Total absorbed energy ($\times 10^8$ Gy)	Displacements per atom ($\times 10^{-3}$)	Helium production (at ppb)	Hydrogen production (at ppb)
Hydrogen	9.09	0.89	—	—
Boron[a]	458.66	777.17	1.94×10^6	4.97
Carbon	0.14	2.83	6.33	0.01
Nitrogen	0.37	2.90	248.56	4733.30
Oxygen	0.09	4.21	24.67	0.17
Fluorine	0.09	5.37	63.12	2.95
Sodium	0.08	5.80	2.00	3.87
Magnesium	0.04	5.86	8.53	3.45
Silicon	0.03	5.52	7.04	14.97
Potassium	0.06	2.93	72.02	356.15
Calcium	0.06	3.10	145.73	302.99
Sulphur	—	3.69	179.28	181.09
Iron	0.01	2.90	0.91	16.05
Aluminium	0.06			

[a] 20% ^{10}B, 80% ^{11}B.
[b] Data not available.

For a fast neutron (E>0.1 MeV) fluence of $4 \cdot 10^{22}$ m^{-2}

Damage calculations

Examples:

ZI-003 (Epoxy Resin)
15 wt% H, 75 wt% C, 3 wt% N, 7 wt% O

ZI-005 (Bismaleimide Triazine)
5 wt% H, 73 wt% C, 10 wt% N, 12 wt% O

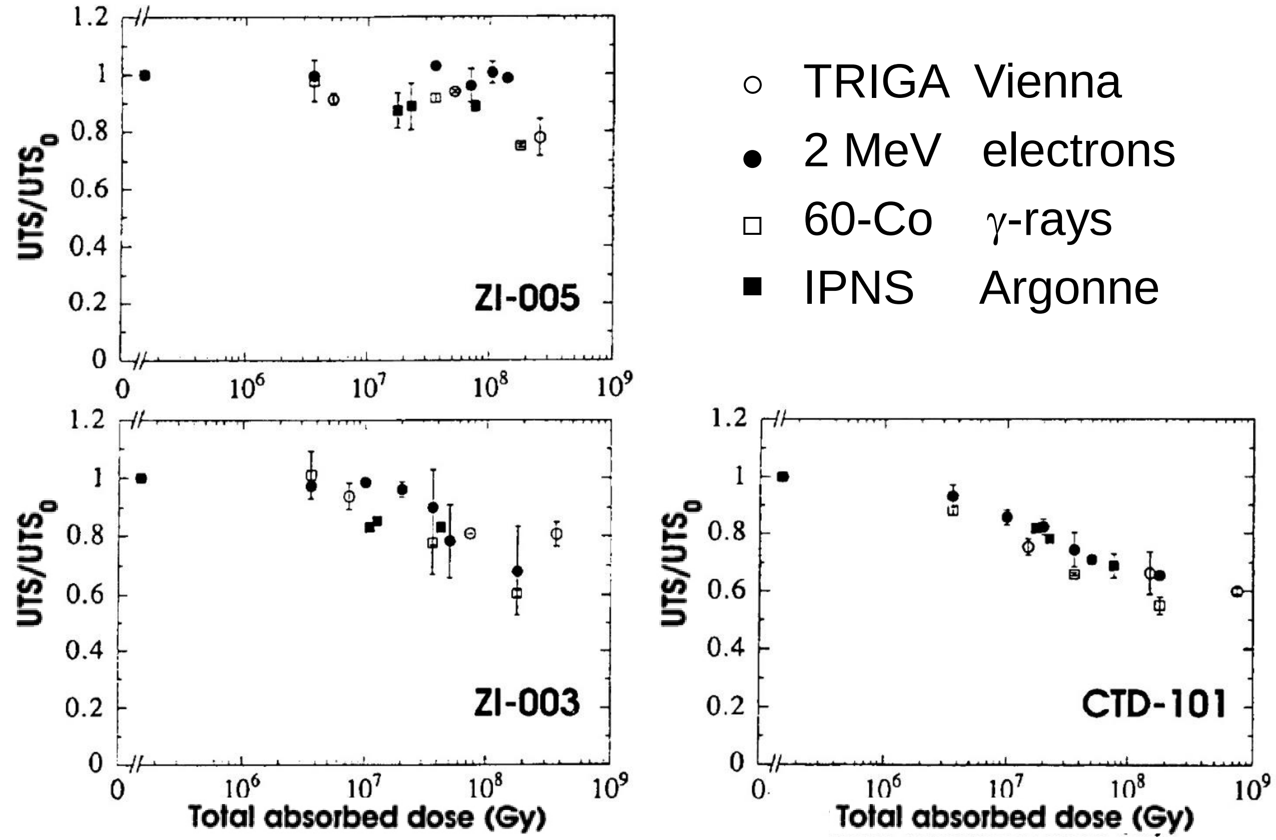
irradiated to 5×10^{22} m^{-2} (E>0.1 MeV) in:

irradiated to 7.8×10^{21} m^{-2} (E>0.1 MeV)

		TRIGA Vienna:		IPNS Argonne:	
ZI-003:	from neutrons:	1.86×10^{8} Gy	(50 %)	16.3×10^{6} Gy	(39 %)
	from gamma rays:	1.82×10^{8} Gy	(50 %)	25.9×10^{6} Gy	(61 %)
		3.68×10^{8} Gy	(100%)	42.2×10^{6} Gy	(100%)
ZI-005:	from neutrons:	0.76×10^{8} Gy	(30 %)	6.5×10^{6} Gy	(20 %)
	from gamma rays:	1.82×10^{8} Gy	(70 %)	25.9×10^{6} Gy	(80 %)
		2.58×10^{8} Gy	(100%)	32.4×10^{6} Gy	(100%)

Insulators: absorbed dose

Tests at 77 K, irradiation at ~340 K (minor influence)



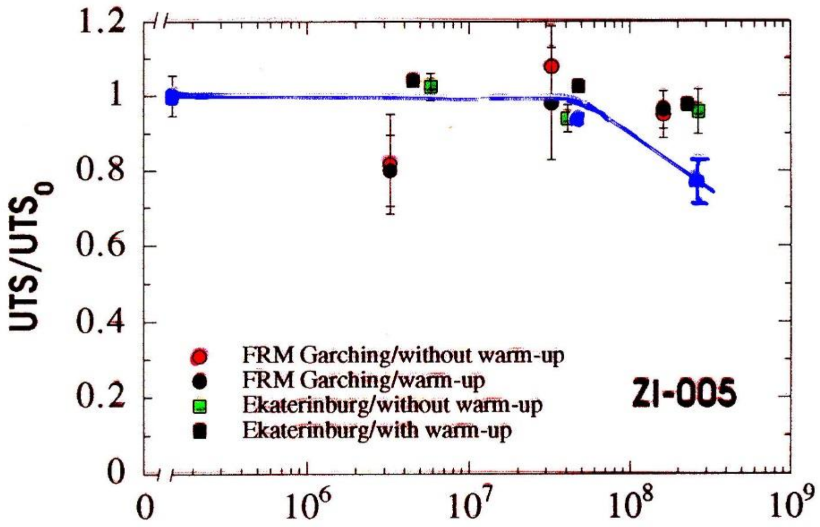
Ionizing radiation breaks chemical bonds.

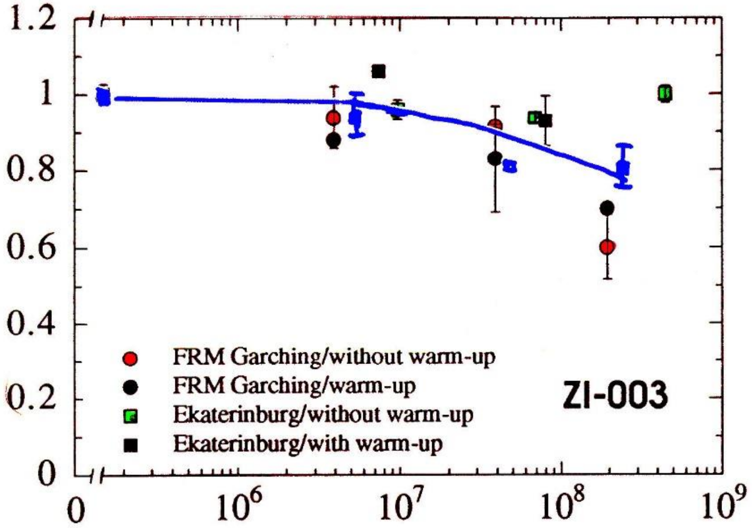
Deposited energy (dose: J/kg) changes chemistry.

K. Humer et al., *Cryogencis* **35** (1995) 871

Reasonable scaling of all data to the absorbed dose.

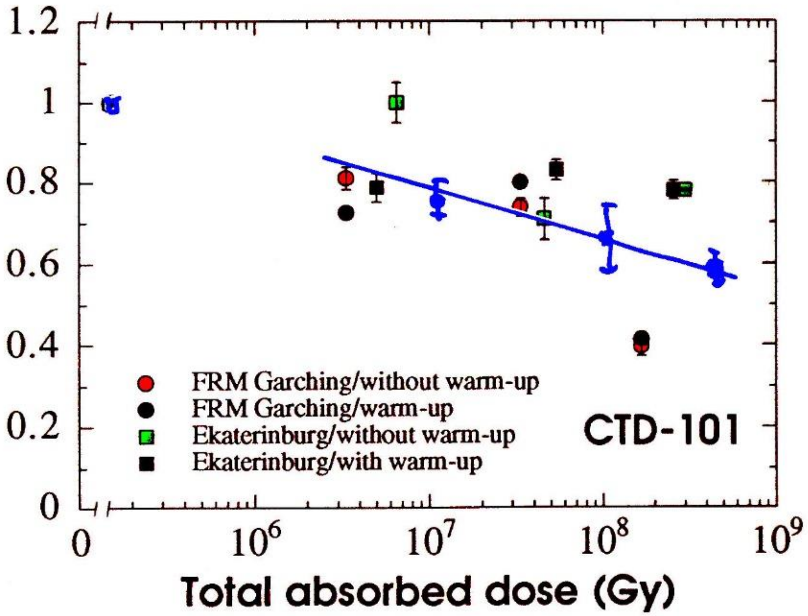
Influence of irradiation temperature and of annealing at RT





Neutron sources:
Garching ~ 5 K
Ekaterinburg 77 K
ATI ~340 K

Tests at 77 K
... Similar results for ILSS !



Summary – Irradiation and test conditions

- Influence of irradiation temperature:

  No or only minor effects for all materials

- Influence of annealing cycles after LT irradiation:

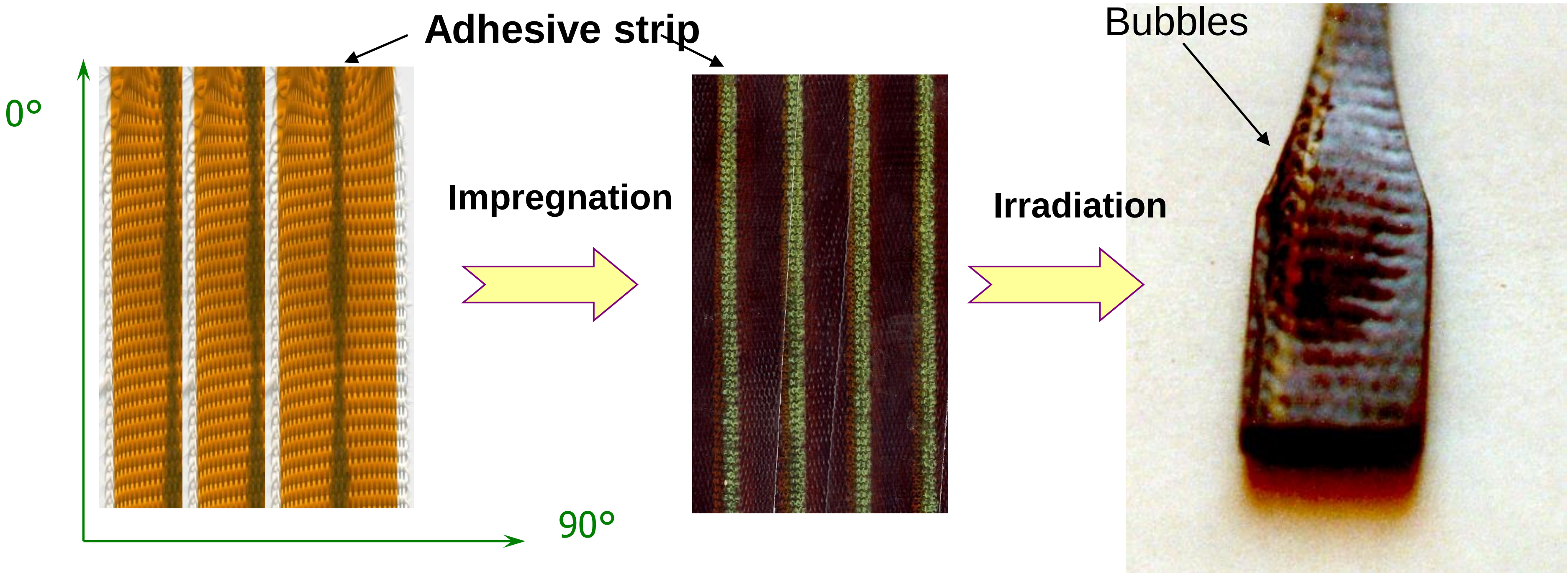
  Only small effects at high doses in *one* 2D GFR epoxy

- Experimental evidence provided for damage scaling with the total absorbed energy

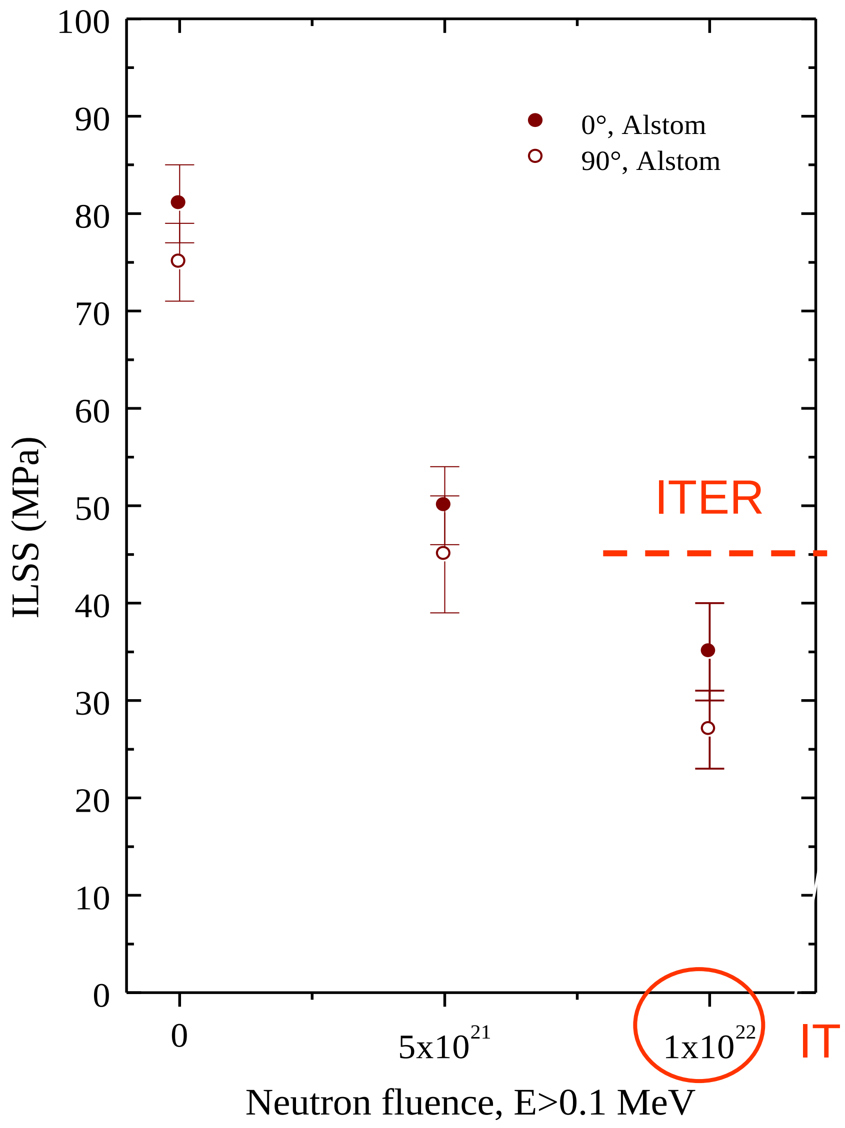
- Predictions of property changes in an unavailable radiation environment are feasible

RESULTS: ITER TF Model Coil - Epoxies

ANSALDO (Italy)	ALSTOM (France)
Combined Kapton-H foil/R-glass fiber reinforced tape	
DGEBA Epoxy (Araldite F type) + Adhesive	DGEBA Epoxy (MY 745-type)
Turn insulation system	Winding-pack insulation system



DGEBA Epoxy (Alstom)



ITER design fluence

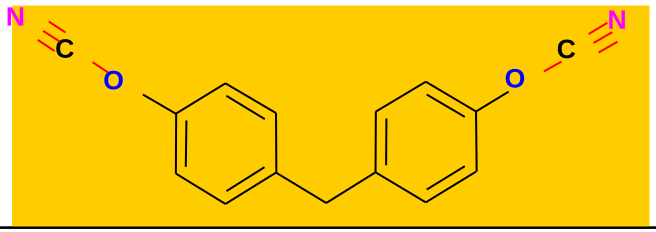
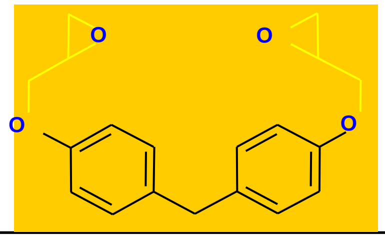
Interlaminar shear strength (ILSS) at 77 K

Material performance drastically affected by irradiation

Mechanical properties fall below the limits of the ITER specification

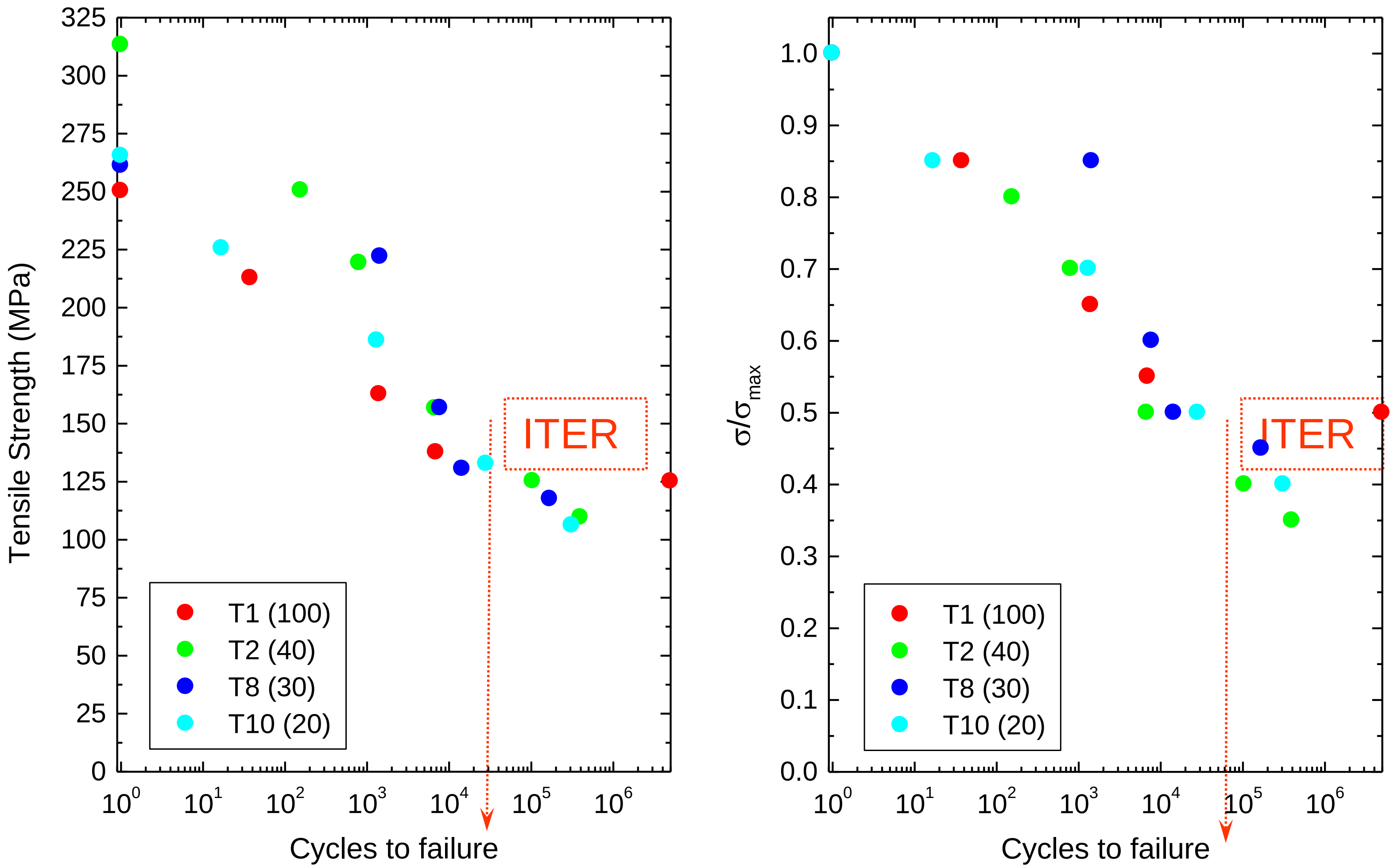
Improvement of the matrix stability urgently required

New materials: Cyanate ester and CE / epoxy blends

	Cyanate ester	Epoxy
	AroCy L-10 	PY 306 
Safety precautions	Avoid local overheating (hot spots) Store in sealed containers in dry rooms Provide sufficient air exchange Take necessary actions to avoid static electricity	Provide sufficient air exchange Take necessary actions to avoid static electricity Avoid strong acids and bases
Viscosity	$\eta_{25\,°C}$ = 120 mPa s $\eta_{60\,°C}$ = 17 mPa s	$\eta_{25\,°C}$ = 1200-1600 mPa s
Pot life at high quantities	Dependent upon type and concentration of co-catalyst and catalyst used	Can be handled

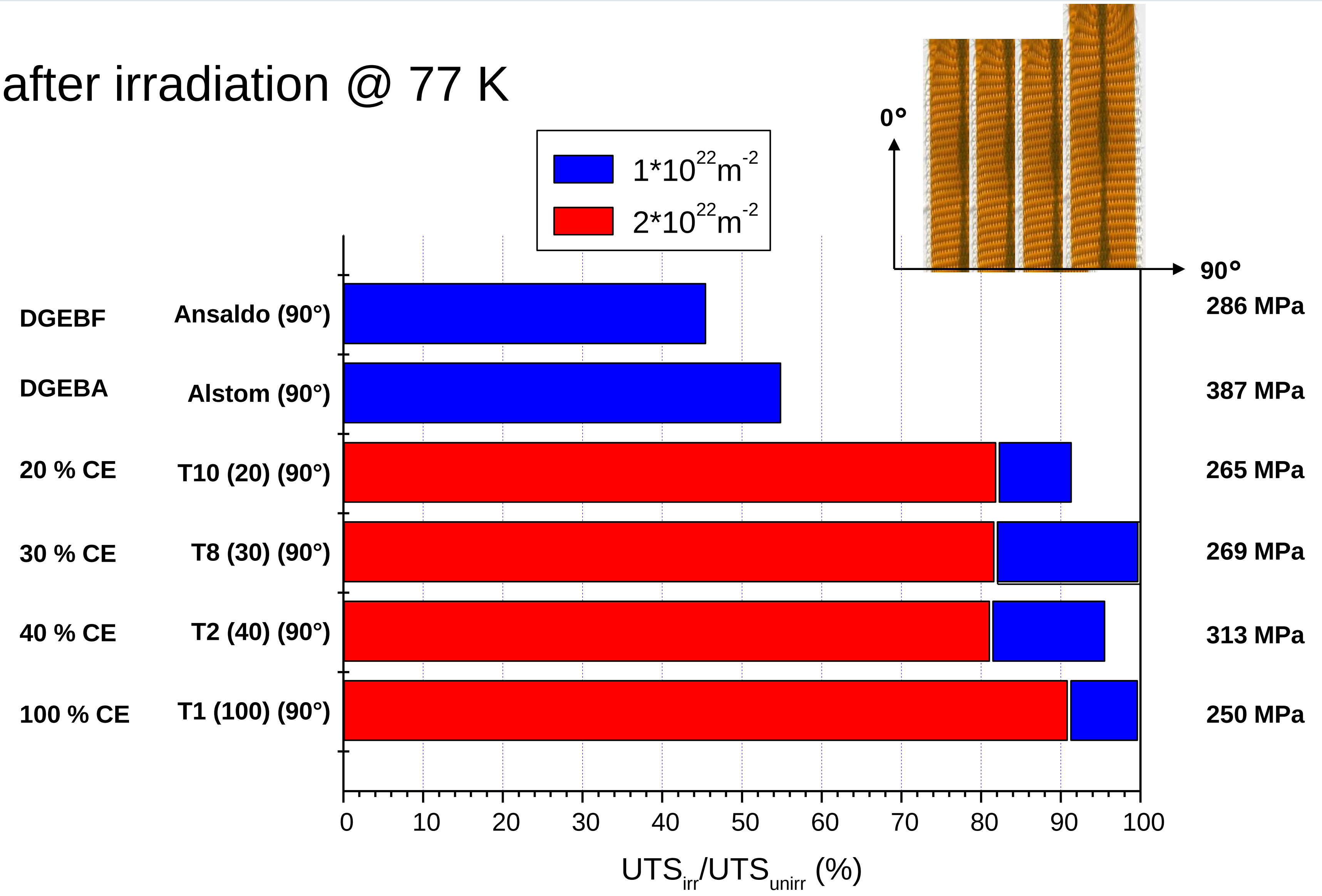
Influence of CE content

Fatigue measurements (90°)



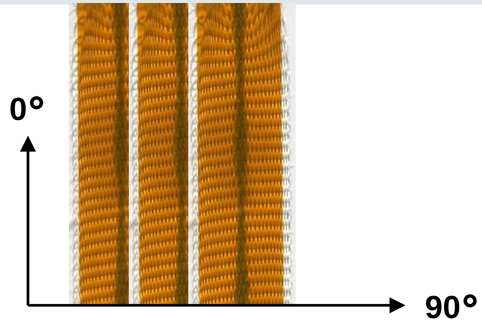
Influence of CE content

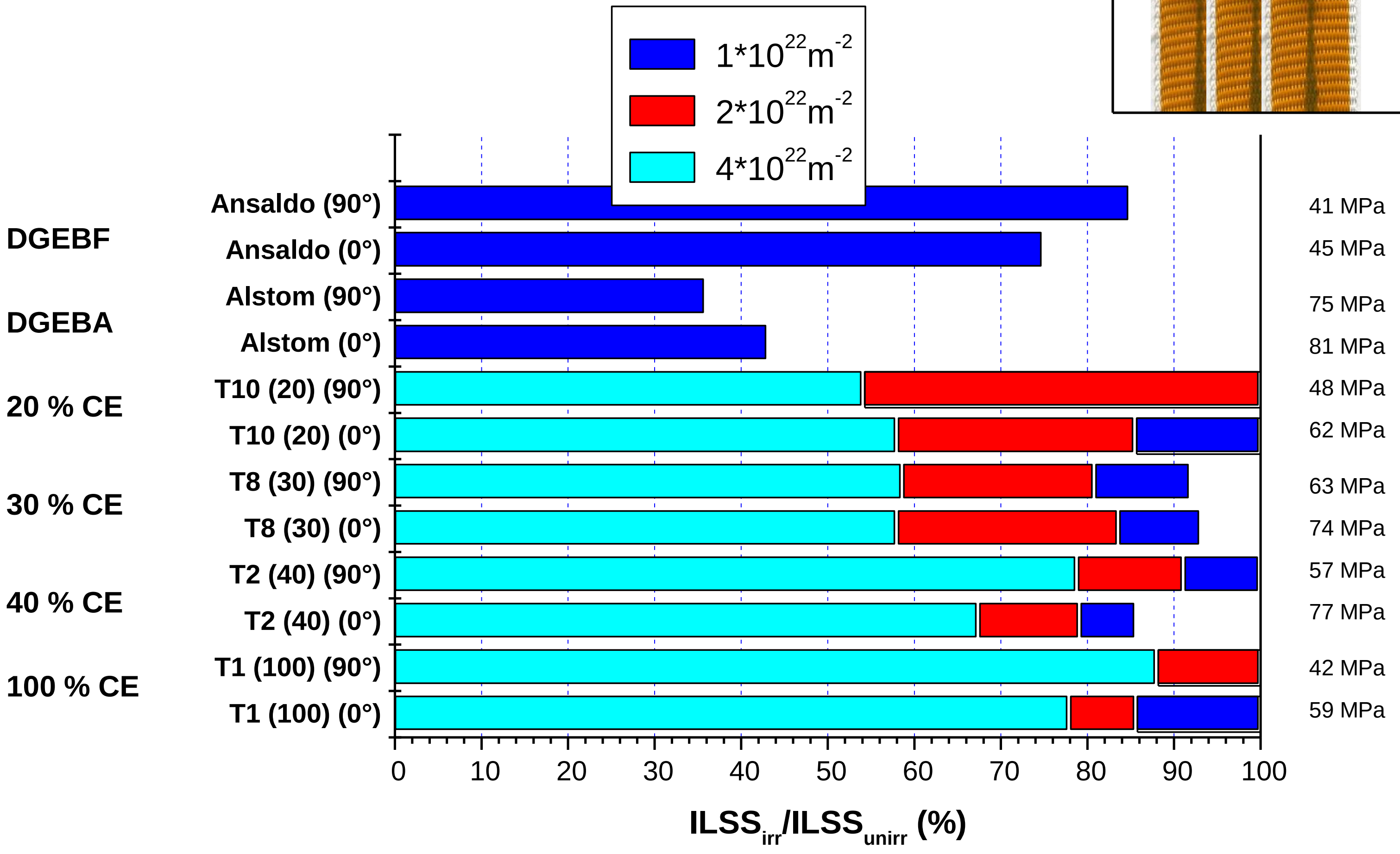
UTS after irradiation @ 77 K



Influence of CE content

ILSS after irradiation @ 77 K





Mechanical properties before and after irradiation

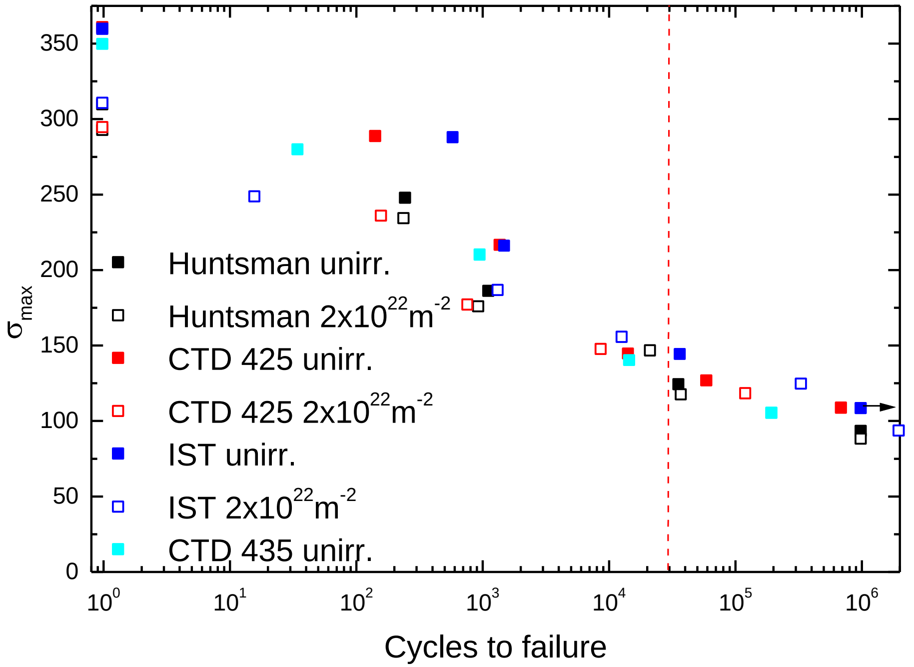
Ultimate tensile strength (MPa)

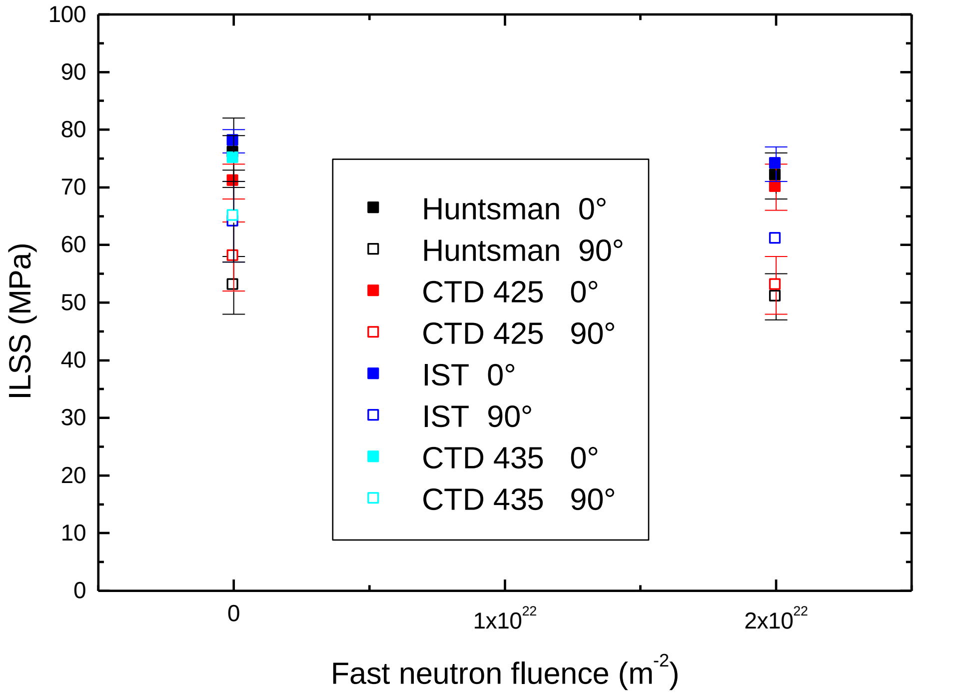
	unirradiated	irradiated
Huntsman	309 ± 5	292 ± 13
CTD-425	360 ± 5	294 ± 13
IST	359 ± 19	310 ± 11
CTD-435	349 ± 9	

Apparent interlaminar shear strength (MPa)

Material	ILSS (MPa)			
	0°		90°	
	unirr.	irr.	unirr.	irr.
Huntsman	76 ± 6	72 ± 4	56 ± 4	51 ± 4
CTD-425	71 ± 3	70 ± 4	58 ± 6	53 ± 5
IST	78 ± 2	74 ± 3	64 ± 7	61 ± 4
CTD-435	75 ± 4		65 ± 2	

Fast neutron (E>0.1 MeV) fluence of $2 \cdot 10^{22}$ m^{-2}





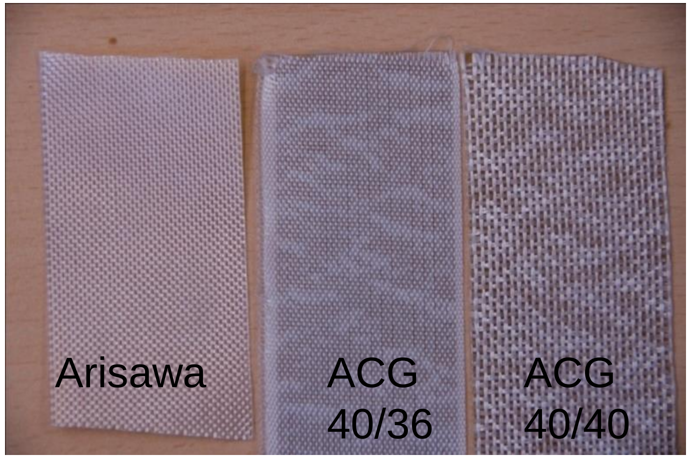
Gas evolution rates

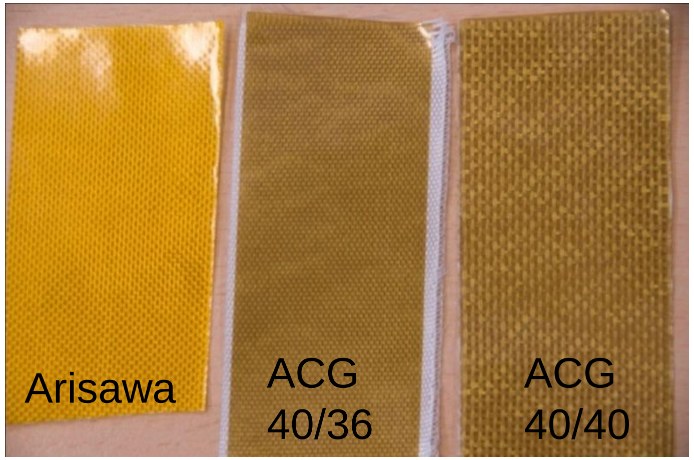
Material	Chemistry	Neutron Fluence (E>0.1 MeV) (10^{21} m^{-2})	Total absorbed Dose (MGy)	Gas Evolution Mean ± Sdev (mm^3)	Gas Evolution Rate Mean ± Sdev ($mm^3g^{-1}MGy^{-1}$)
CTD-422	Cyanate Ester/Epoxy	1	4.19	105 ± 0	68.9 ± 2.4
CTD-10x	Cyanate Ester/Epoxy/BMI	1	4.05	83 ± 11	57.1 ± 6.8
CTD-101K	Epoxy/Anhydride	1	4.14	165 ± 0	108.4 ± 1.9
CTD-7x	Cyanate Ester/Epoxy/PI	1	3.90	75 ± 0	48.2 ± 0.4
CTD-15x	Cyanate Ester/BMI	1	4.46	60 ± 0	38.9 ± 0.3
CTD-101	Epoxy/Anhydride	1	4.11	165 ± 21	114.3 ± 10.3
CTD-HR3	Cyanate Ester/PI	1	4.31	60 ± 0	33.9 ± 0.5
CTD-404 CTD-404	Cyanate Ester	1 5	4.04 20.18	68 ± 11 200 ± 9	47.0 ± 7.4 30.4 ± 1.1
ER Baseline	Epoxy/Anhydride	1	4.15	263 ± 11	176.2 ± 10.3

Bonded glass/polyimide tapes

3 different tapes

- Arisawa, JP: 40/40 mm glass / polyimide tape (Kapton)
- Advanced Composite Group (ACG), UK: 40/40 mm glass / polyimide tape (Upilex)
  40/36 mm glass / polyimide tape (Upilex)





Test material

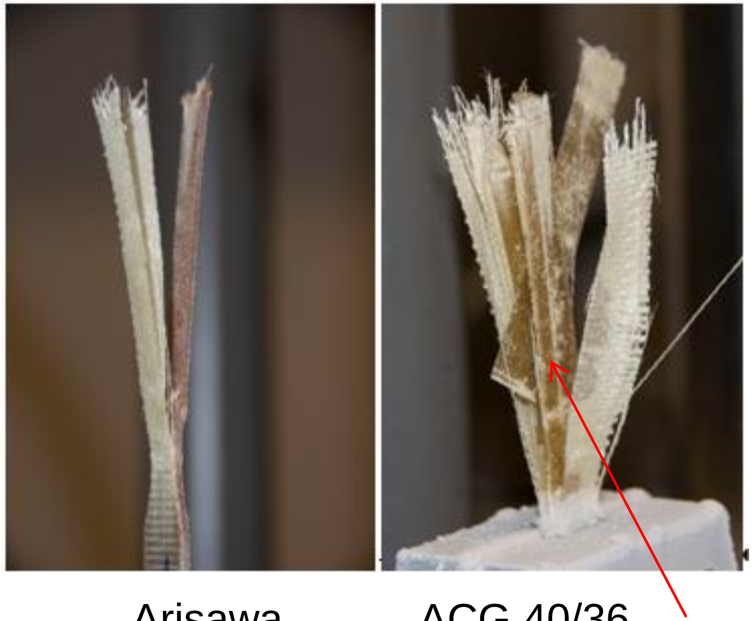
1 layer of S2 glass (0.15 mm)
3 layers of bonded tapes
1 layer of S2 glass (0.25 mm)

} impregnated with Huntsman LMB 6653/ 6622-4

Mechanical properties before and after irradiation

Ultimate tensile strength (MPa)

	unirradiated	irradiated
Arisawa	445 ± 13	407 ± 15
ACG 40/36	332 ± 21	278 ± 6
ACG 40/40	190 ± 11	

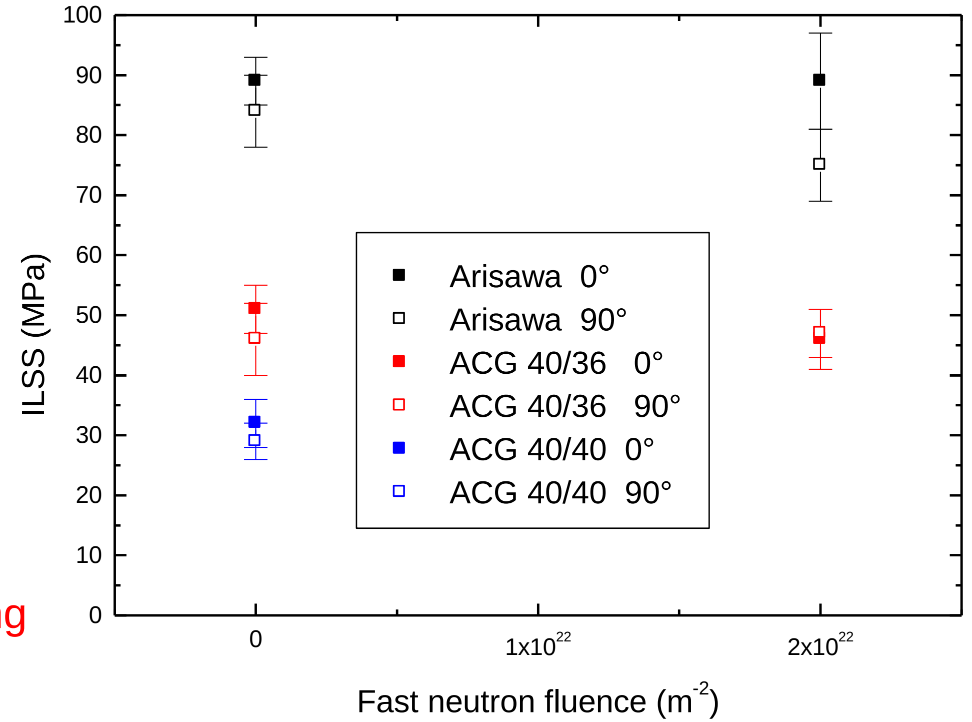


Arisawa ACG 40/36

delamination caused by weak bonding between resin and polyimide

Apparent interlaminar shear strength (MPa)

Material	unirr.		irr.	
	0°	90°		
ACG 40/36	51 ± 4	46 ± 6	46 ± 5	47 ± 4
ACG 40/40	32 ± 4	29 ± 3		
Arisawa	89 ± 4	84 ± 8	89 ± 8	75 ± 6



Summary

CE/epoxy insulation systems

- Poor bonding between *pure* CE and Kapton (delamination)
- Number and thickness of fibers have a stronger effect on the mechanical properties than the CE content
- Excellent mechanical properties after exposure to the ITER design fluence (CE content can be reduced to 20 %)
- Slight degradation at $2*10^{22}$ m^{-2} (~20 %)
- Very favorable gas evolution rates
- Chemistry indicates optimum properties at 40 % CE
- Blends with CE content between 100 % and 40 % seem to have the potential to withstand neutron fluences > $4*10^{22}$ m^{-2} (DEMO)