The general US MDP meeting, Sept 11, 2019

US MDP epoxy resin research at LBNL – an update

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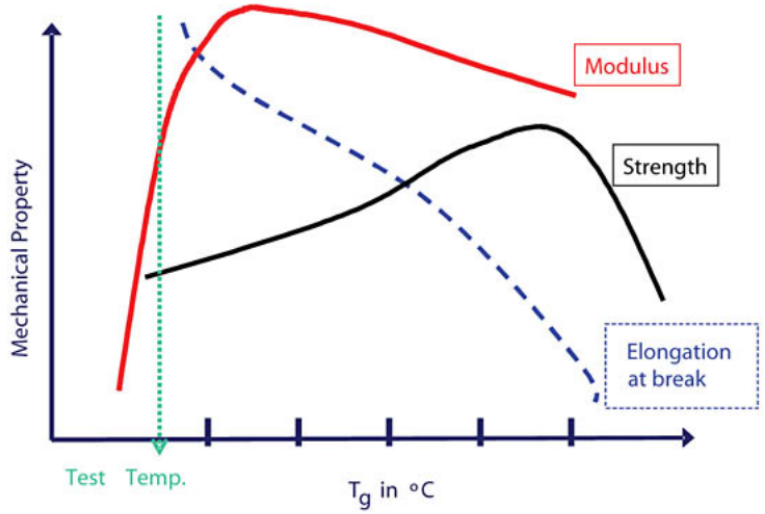
Outline

- A theory of quench training in Nb_3Sn accelerator magnets and implications.
- Some relevant work at LBNL.

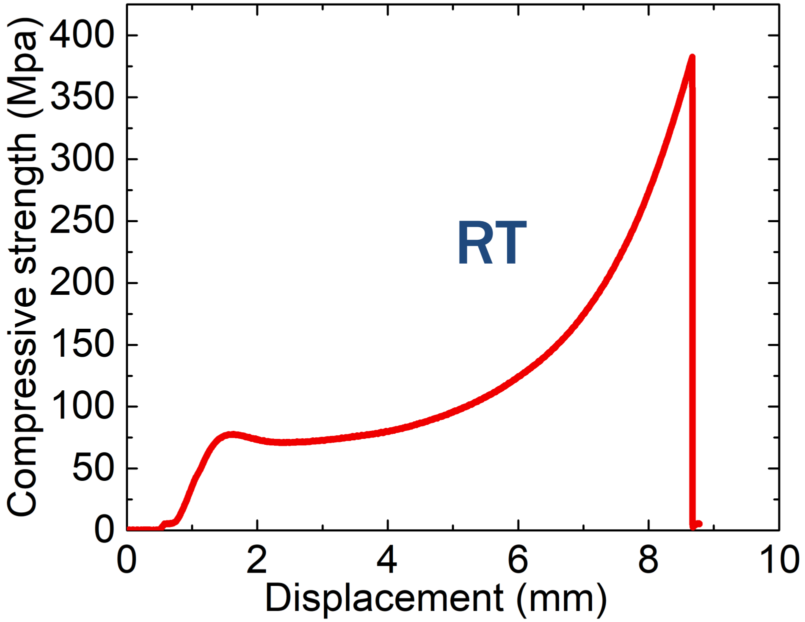
Understanding RT properties of epoxy resins for superconducting magnets

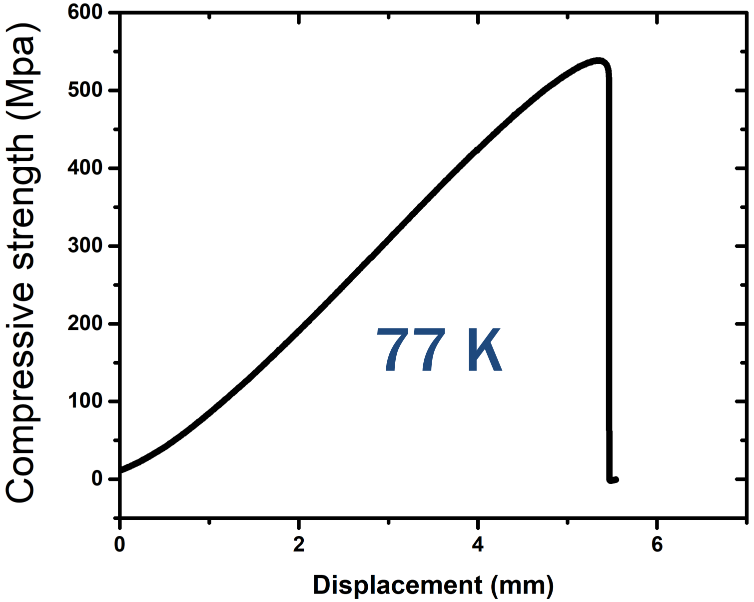
Figure 2 | Room temperature property trends vs. T_g > test temperature for amine-cured epoxy resins.

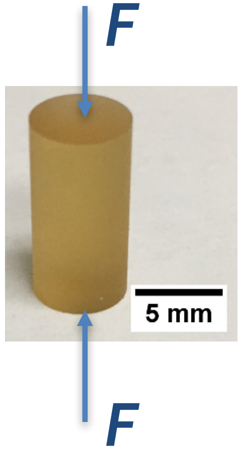


A rather unfortunate truth: Even the high toughness epoxy with large elongation at break at RT becomes brittle at <77 K

The resin tested is a variant of the ATLAS ECT.



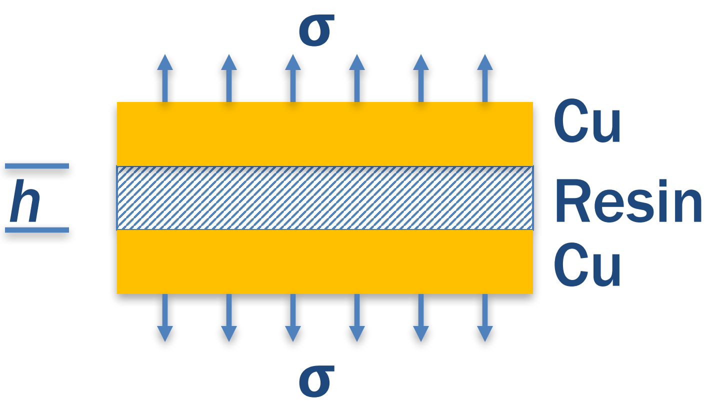




- Compression tests of NHMFL-mix 61, CTD-101K were given by Theo Tervoort at ICMC 2019.
- Plenty of evidence of NHMFL-mix61 cracking at 4 K given by Nb_3Sn CCT subscale magnets and CCT5 (systematic microscopy images by Diego Arbelaez).

Test by Shijian Yin at LBNL.

The work of fracture differs



$$\sigma = \frac{\int_{4\,K}^{RT} E\Delta\alpha dT}{1-2\mu}$$

If $\frac{\sigma^2 h}{4E} > \gamma$,resin cracks.

γ is the work of fracture.

	ITER CS	71 A	ATLAS ECT variants			
	DGEBF/ MTHPA*	DGEBF POPDA	DGEBF/ PPGDGE 50/50	DGEBF/ PPGDGE 60/40	DGEBF/ PPGDGE 80/20	DGEBF/ PPGDGE 100/0
	Work ($\mathrm{J/m^2}$)	Work ($\mathrm{J/m^2}$)	Work ($\mathrm{J/m^2}$)	Work ($\mathrm{J/m^2}$)	Work ($\mathrm{J/m^2}$)	Work ($\mathrm{J/m^2}$)
RT	103	899**	2241**	353	310	205
	± 12			± 53	± 42	± 58
77K	199	525	396	359	249	110
	± 13	± 67	± 27	± 49	± 49	± 25
4.2K	61	178	128	100	93	63
	± 5	± 10	± 9	± 2	± 9	± 3

*MTHPA – methyl tetrahydrophthalic anhydride – typically Huntsman HY 917

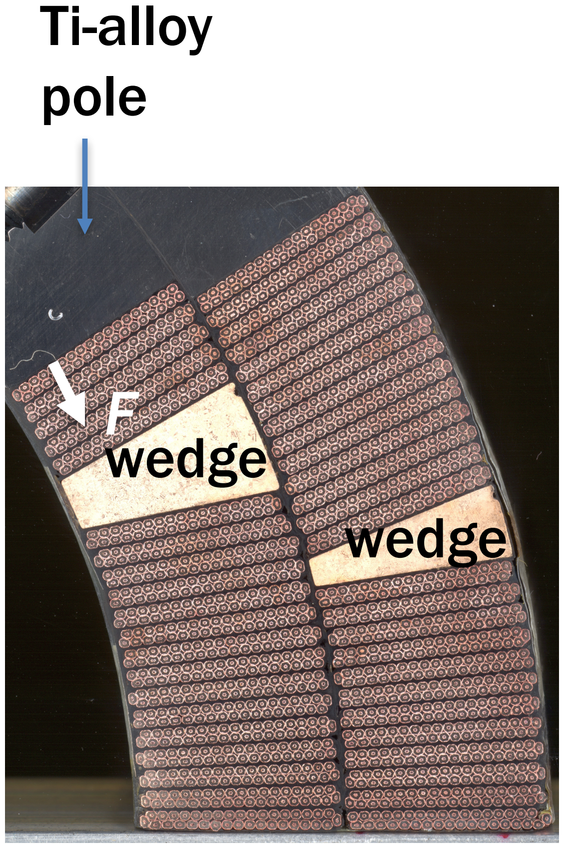
**Results at RT are geometry dependent - no single value for the work of fracture.

Resins		DGEBA / POPDA	DGEBA/ Anhyd.	100/0	60/40
Thermal Stress (MPa)	4K	95	124	124	101
Cracking Index (mm)	4K	0.36	0.07	0.07	0.19

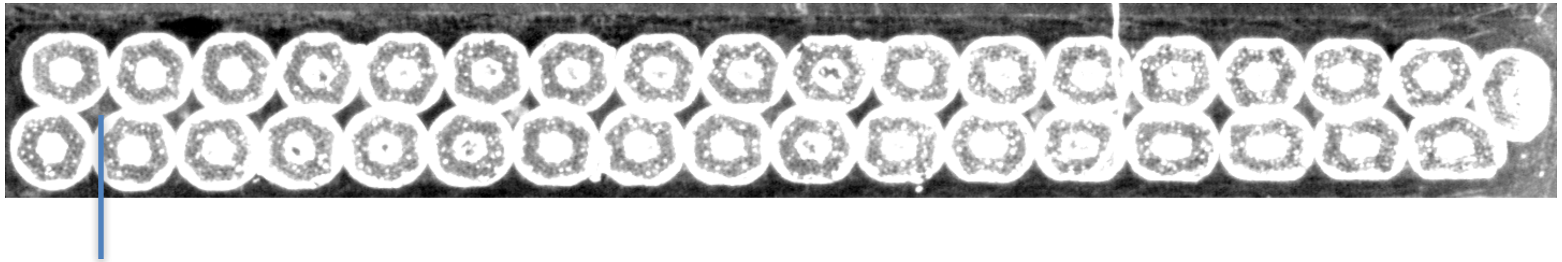
Work of fracture and cracking index data from D. Evans, ICMC2019.
RAL-71A (POPDA hardener – the toughest epoxy for cryogenic uses – David Evans)

Micro-cracking predicted at the pole turn of a cosine-theta Nb_3Sn magnet

, inside the Rutherford cable, between strands, at 4 K and when the magnet is powered.



HQ-C06, Hugh Higley



Neat resin, as large as 0.4 mm

Stored energy in neat resin $\frac{\sigma^2 h}{4E}$

@4K and when excited to >0.7 SSL

- As large as 449 J/m^2, at the pole turn (coil $\sigma_\theta \sim 0$)
- Much smaller (38-81 J/m^2) at the mid-plane (coil $\sigma_\theta \sim -150\ MPa$)

Credits to Akira Yamamoto for initially raising questions about controlling cracking of epoxy between strands inside a Rutherford cable.

Microcracking expected to be more severe in CCT magnets (without prestress).

- Large volumes with neat resins.
- Store energy even larger than 449 J/m^2 (4 K and powered, coil $\boldsymbol{\sigma_\theta \sim 0}$)

Let’s be generous and assume this theory is right, what are implications?

- Training would depend on the amount of prestress coil received, and prestress sequences (in related to cooling).
    - Prestress would help reduce training for CCT magnets?

- Training would depend on the packing factor of the Rutherford cable.

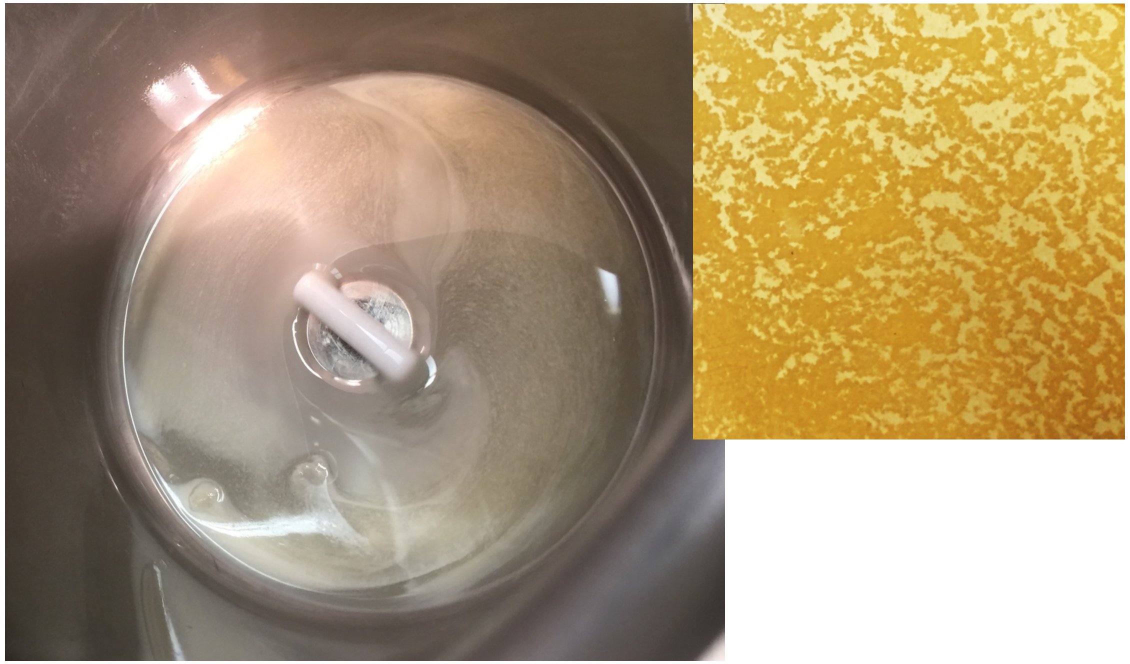
- Even the toughest epoxy Evans knows would still crack (inside the Rutherford cable).

- Beneficial to add inorganic particles inside the Rutherford cable, but what and how?

Reducing CTE of epoxy, and increasing toughness, with inorganic nanoparticles fillers – an issue

Epoxy/BN 2D nanoplates, after mixing

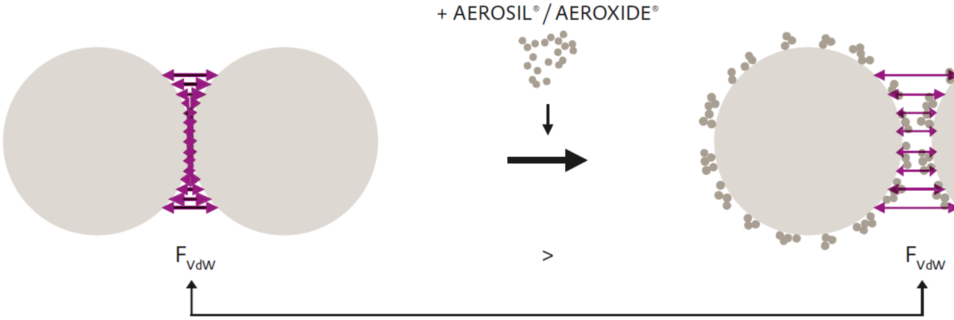
50 ℃/12 h, particle segregation



Work by Shijian Yin at LBNL.

An approach developed to fix the issue: Using aeroxide to reduce Van der Waals forces between particles

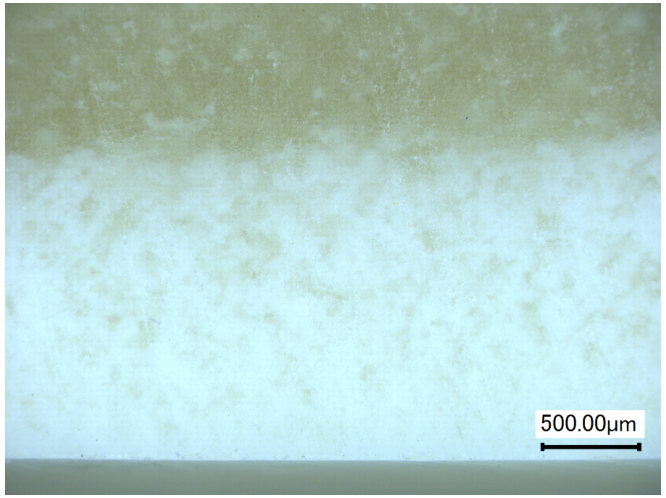
Figure 19

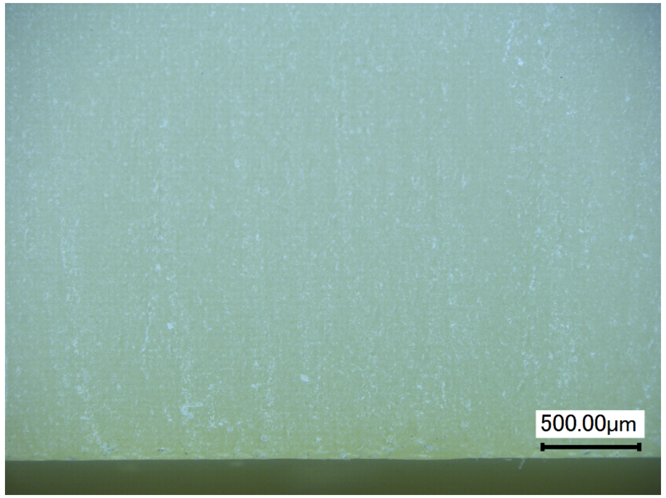


AEROSIL® and AEROXIDE® aggregates act as spacers and reduce Van der Waals forces (F_{VdW}) between powder particles.

Epoxy, BN2D 0.3wt%

Epoxy, BN2D 0.3wt%, Aluc 1 wt%





Work by Shijian Yin at LBNL.

LBNL teams up with CTD with a new phase II SBIR to develop and verify advanced epoxy for superconducting accelerator magnets

COMPOSITE TECHNOLOGY DEVELOPMENT, INC.
ENGINEERED MATERIAL SOLUTIONS

Andrea Haight (PI), Mark Haynes, Nicole Thomas

- **LBNL responsibilities**
  - **(Help select) test and verify three advanced epoxy formulations, using the Nb_3Sn CCT subscale magnet as the primary test bed, and other experimental tests and modeling, for superconducting accelerator magnet uses.**

High toughness epoxy development at CTD, Inc.

- **Toughening approaches based on CTD’s prior work with high strain resin systems for cryogenic tank applications**

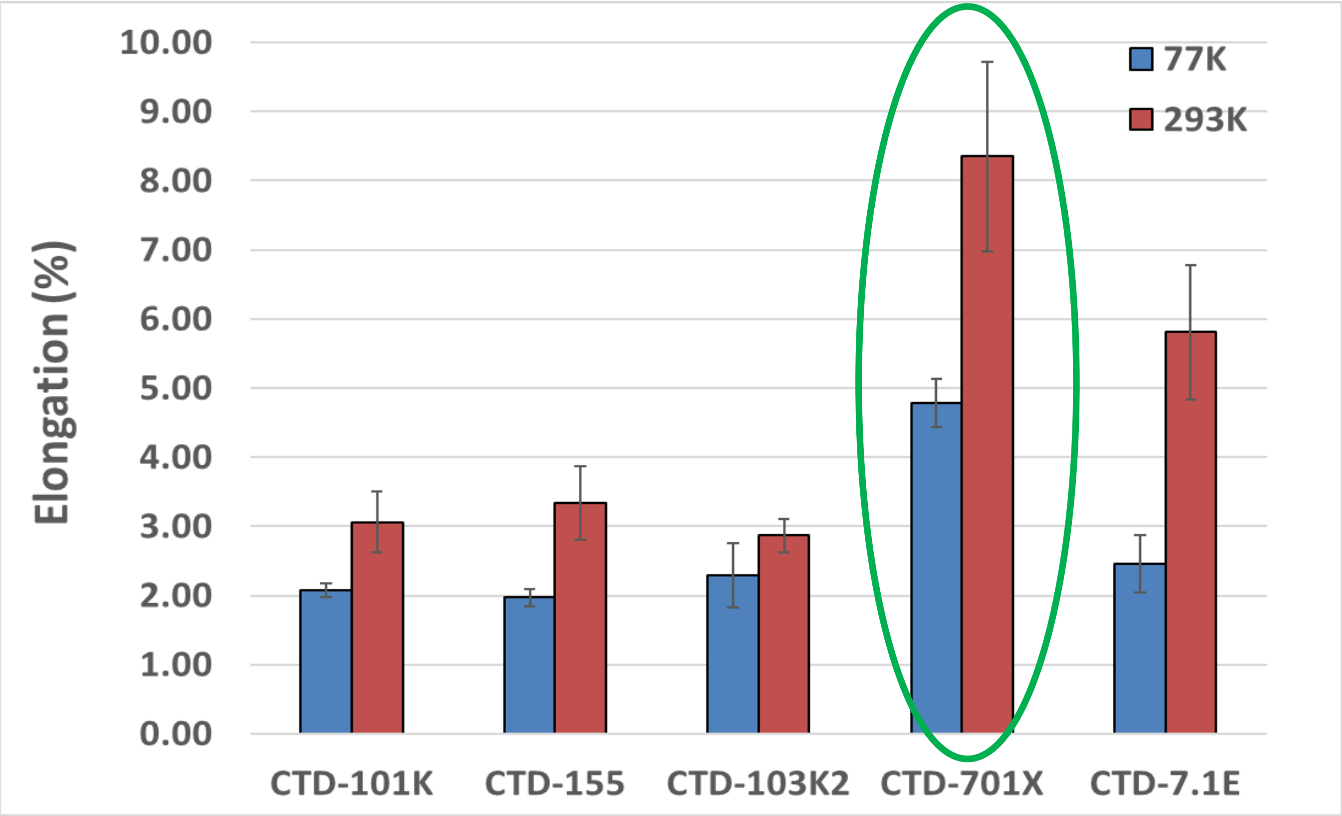
Name	Description
CTD-101K	Baseline anhydride-cured epoxy resin used in HEP magnets
CTD-103K2	Long pot life 2-part formulation with performance similar to CTD-101K
CTD-103LT	Lightly toughened version of CTD-103K2
CTD-155	Anhydride-cured epoxy resin with reactive rubber toughener
CTD-7.1E	Low viscosity, toughened amine-cured epoxy resin
CTD-701X	Extremely low viscosity, tough polyolefin resin system

Processability of Toughened Formulations

- **Toughening approaches based on CTD's prior work with high strain resin systems for cryogenic tank applications**

Description	T_g (tan δ)	Viscosity @ 60 °C (cP)	
		Initial	7 hours
CTD-101K	150 (avg QA)		
CTD-103K2	141	61	61.5 (4.5 hr)
CTD-103LT	126	24	71
CTD-155	147	125	127
CTD-701X	131	25 (@ 25 °C)	70 (90 min, 25 °C)
CTD-7.1E (tank formulation)	72	225	Not determined

CTD-701X shows high elongation at break at 77 K

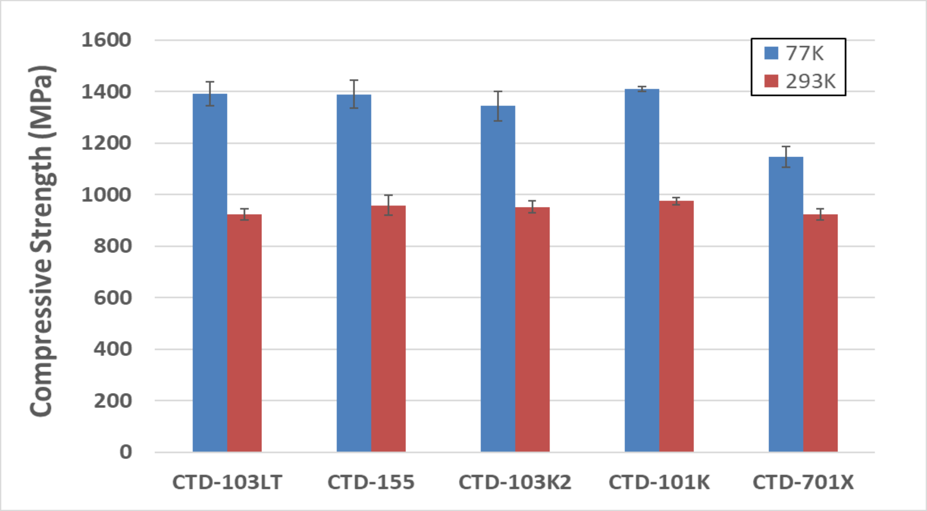


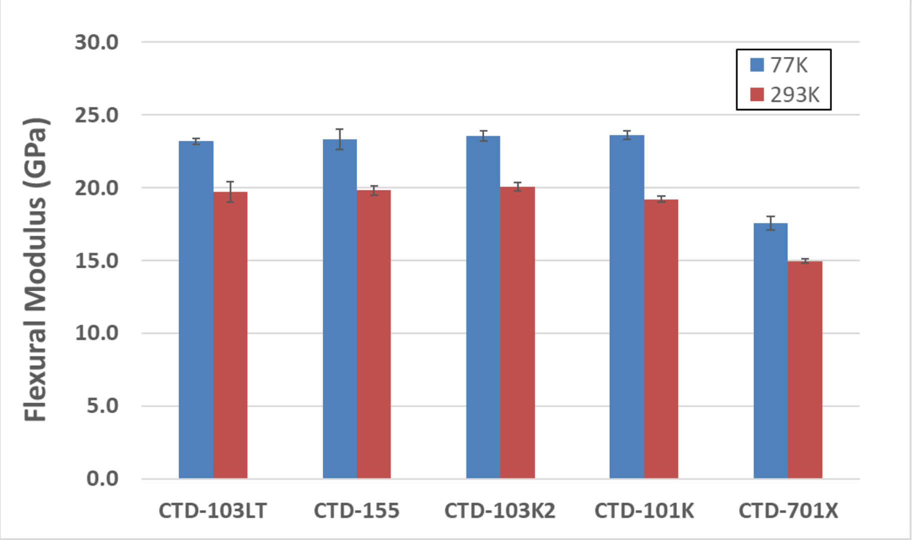
- **All epoxy resins showed similar performance**
- **CTD-701X slightly lower, not unexpected**

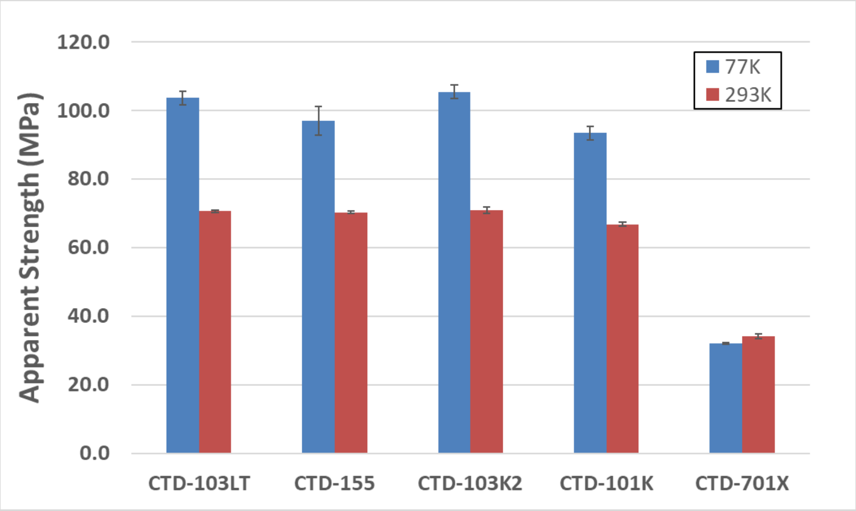
- ASTM D638

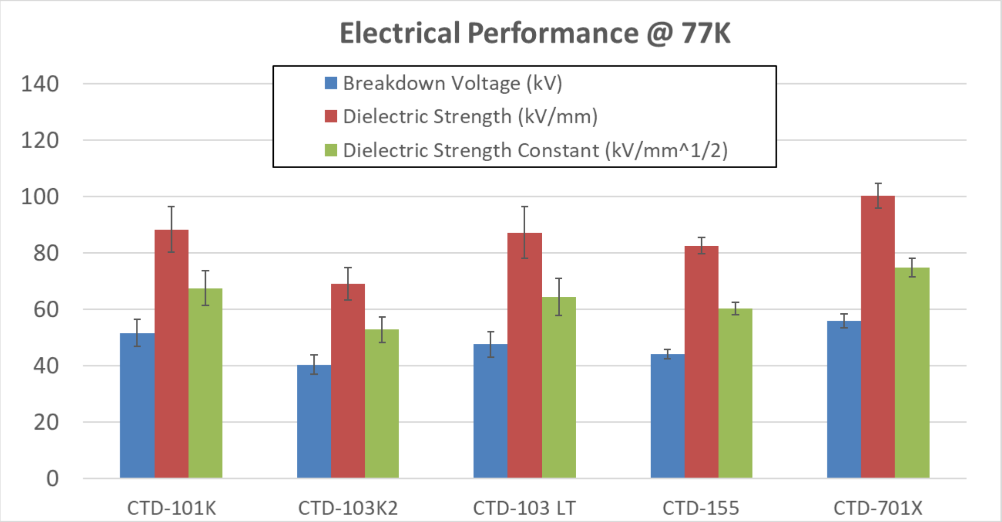
- Toughening approaches in epoxy most effective at room temperature
- CTD-701X shows high elongation, even at 77K

CTD-701x has compressive strength, flexural modulus and dielectric strength comparable to CTD-101K.

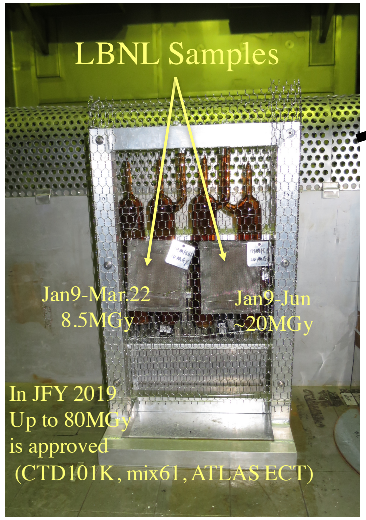




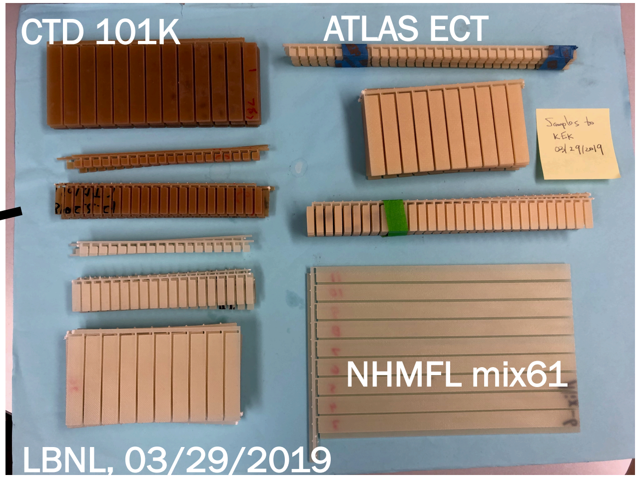




Collaborating with KEK to run Irradiation tests



- 10 MGy and 20 MGy samples at QST.
- 100 MGy experimental time secured for 2019/2020, and samples prepared.



KEK 大学共同利用機関法人
高エネルギー加速器研究機構

Toru Ogitsu

Summary

- **Existing theories of quench training in Nb_3Sn accelerator magnets.**
    - **Serrated yielding behaviors causing jerky strand movement.**
    - **S-2 fiber cracking.**
    - **Bond failures: Insufficient pre-stress leads to pole turn separating from the pole.**
    - **Bond failures: Stick-slip of cable at the end of a CT magnet and a CCT magnet.**
- **A theory of a key contributor to quench training in Nb_3Sn accelerator magnets and implications.**
- **Two collaborations: (with CTD Inc) Verifying advanced epoxy formulations for superconducting accelerator magnets; (with KEK) irradiation toughness of epoxy with gamma-ray irradiation.**

Bond failures – thanks to S. Caspi for discussions.