3rd KE4738 Technical Meeting
March 24th, 2021

General information
&
Feedback from the US-MDP

Davide Tommasini, CERN

Newcomers in the CERN Team

Christian Scheuerlein : Staff, material scientist.
Thermo-mechanical characterization, irradiation programme.

Barthi Verma : Fellow, chemist.
Characterization of adhesion strength of impregnation systems.
Parameters to explore: configuration, geometry, materials (copper, stainless steel, polyimide, glass fabrics, mica) as a function of the impregnation resin.

I also remind

Sébastien Clément
Staff, plastic technologist. In charge of the operation of the polymer lab.

Irradiation programme

We intend to explore the radiation ageing (thermo-mechanical and dielectric properties) of different impregnation resins as a function of irradiation type, dose, and environment (air, vacuum, low temperatures). We are preparing the programme, which we intend to present and discuss in a next meeting.

News from the US-MDP Meeting (1-5 March)

Evolution of the US Magnet Development Program

- The formation was initiated by DOE-OHEP as a response to the last Snowmass and P5 process
- The Updated Roadmap for MDP is publicly available
  - https://arxiv.org/abs/2011.09539

2020 Updated Roadmaps

The 2020 Updated Roadmaps for the US Magnet Development Program

Compiled by

Soren Prestemon, Kathleen Amm, Lance Cooley, Steve Gourlay, David Larbalestier, George Velev, Alexander Zlobin

&

With Major Contributions from
Technical Leads and Collaborators
within the US MDP

US Magnet Development Program (MDP) Goals:

GOAL 1:

Explore the performance limits of Nb_3Sn accelerator magnets with a focus on minimizing the required operating margin and significantly reducing or eliminating training.

GOAL 2:

Develop and demonstrate an HTS accelerator magnet with a self-field of 5 T or greater compatible with operation in a hybrid LTS/HTS magnet for fields beyond 16 T.

GOAL 3:

Investigate fundamental aspects of magnet design and technology that can lead to substantial performance improvements and magnet cost reduction.

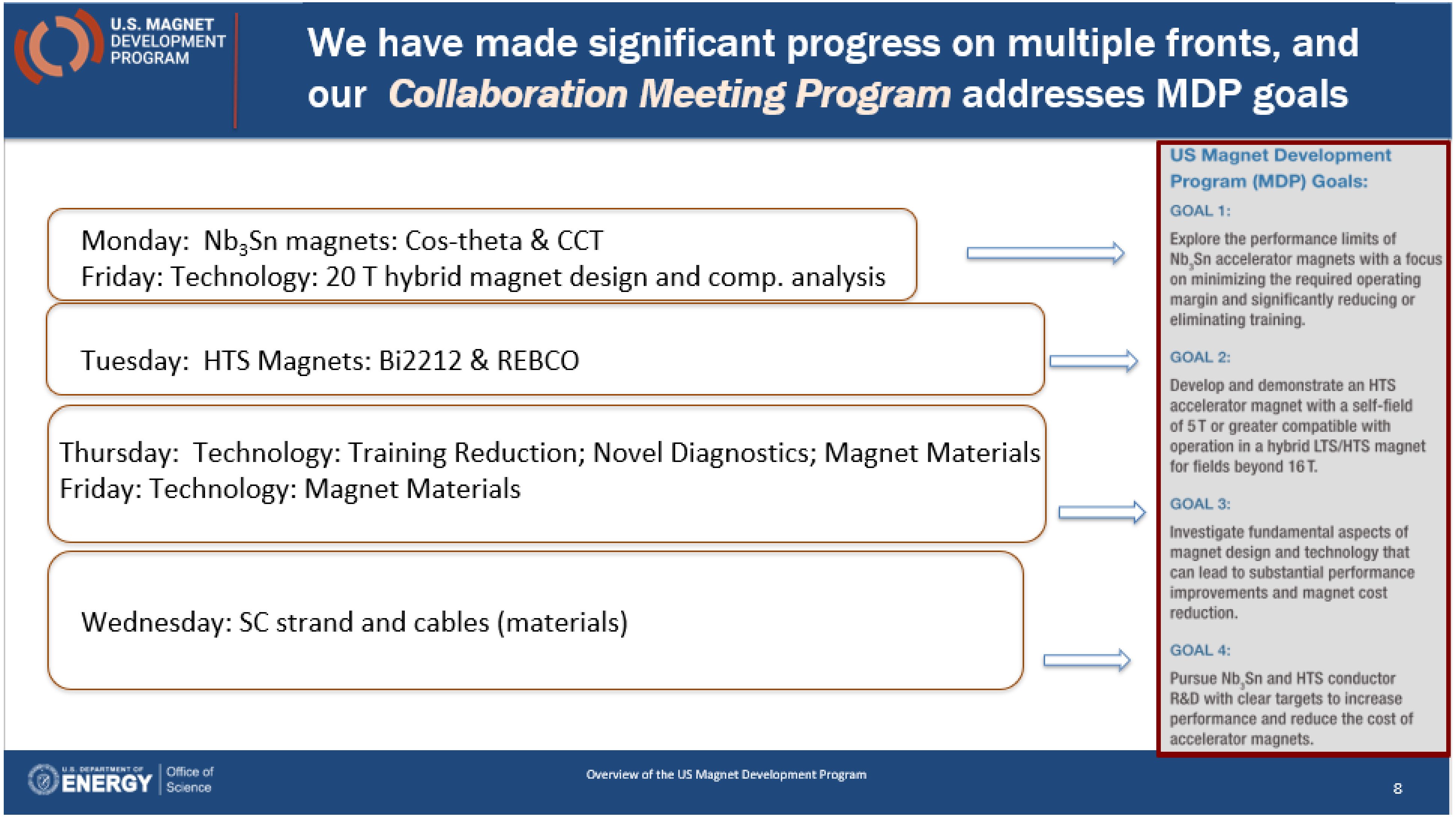
GOAL 4:

Pursue Nb_3Sn and HTS conductor R&D with clear targets to increase performance and reduce the cost of accelerator magnets.

Overview of the US Magnet Development Program

Courtesy Soren Prestemon, LBNL

News from the US-MDP Meeting (1-5 March)



U.S. MAGNET DEVELOPMENT PROGRAM

We have made significant progress on multiple fronts, and our *Collaboration Meeting Program* addresses MDP goals

Monday: Nb$_3$Sn magnets: Cos-theta & CCT
Friday: Technology: 20 T hybrid magnet design and comp. analysis

Tuesday: HTS Magnets: Bi2212 & REBCO

Thursday: Technology: Training Reduction; Novel Diagnostics; Magnet Materials
Friday: Technology: Magnet Materials

Wednesday: SC strand and cables (materials)

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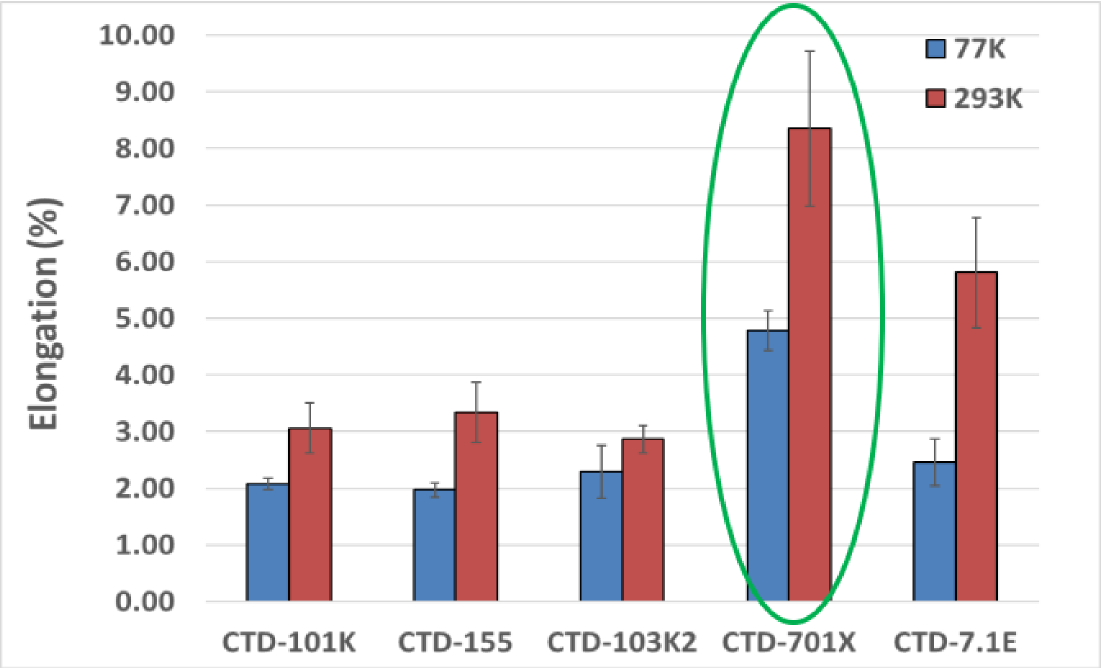
U.S. DEPARTMENT OF ENERGY | Office of Science

Overview of the US Magnet Development Program

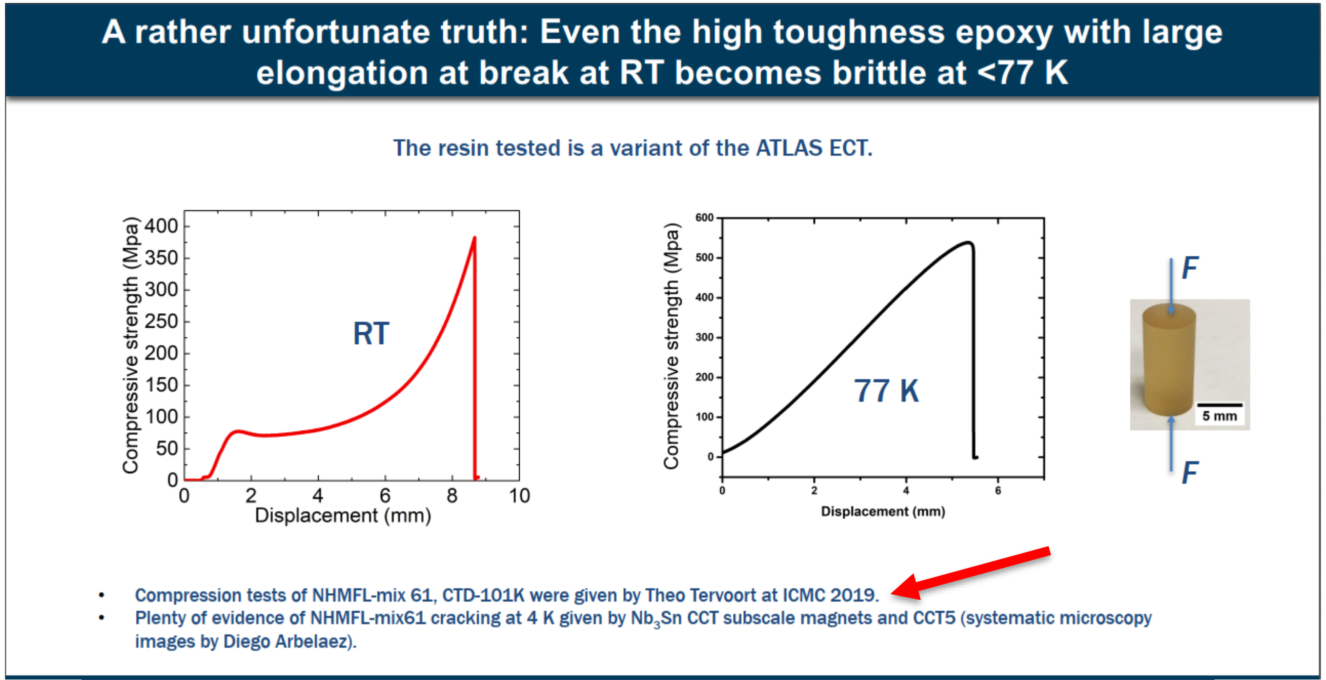
8

Courtesy Soren Prestemon, LBNL

From Tengming Shen, LBNL 2019



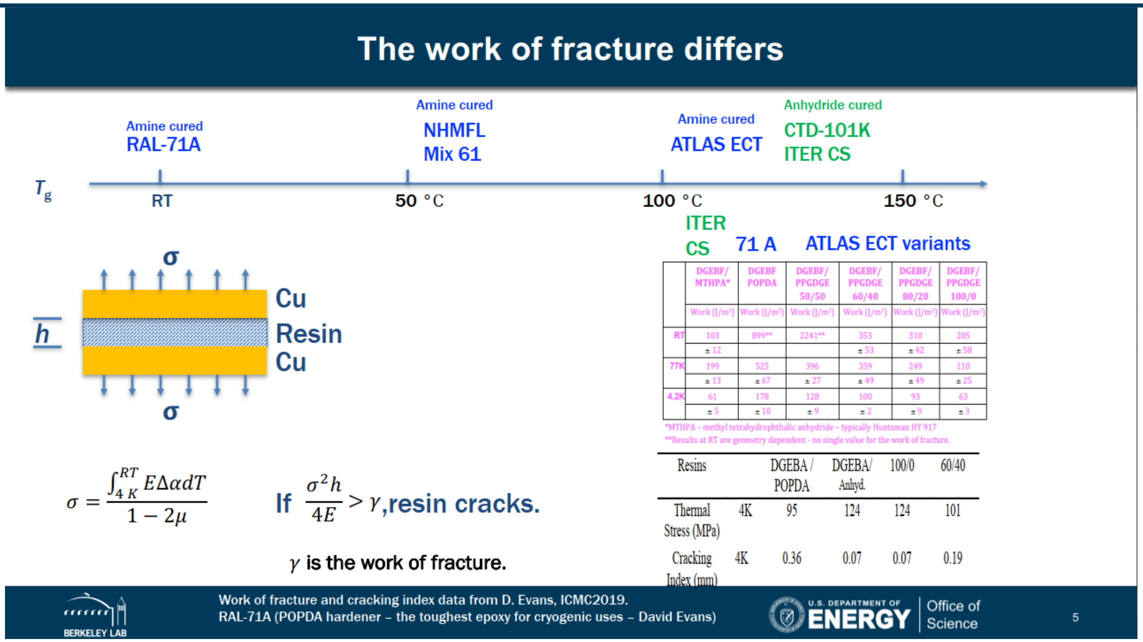
Name	Description
CTD-101K	Baseline anhydride-cured epoxy resin used in HEP magnets
CTD-103K2	Long pot life 2-part formulation with performance similar to CTD-101K
CTD-103LT	Lightly toughened version of CTD-103K2
CTD-155	Anhydride-cured epoxy resin with reactive rubber toughener
CTD-7.1E	Low viscosity,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



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).



The work of fracture differs

	DGEBF/ MTHPA*	DGEBF POPDA	DGEBF/ PPGDGE 50/50	DGEBF/ PPGDGE 60/40	DGEBF/ PPGDGE 80/20	DGEBF/ PPGDGE 100/0
	Work (J/m²)	Work (J/m²)	Work (J/m²)	Work (J/m²)	Work (J/m²)	Work (J/m²)
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

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

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

γ is the work of fracture.

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

Recent studies at LBNL (Tengming Shen)

- **Since 2020, LBNL has been collaborating with Fermilab (S. Krave) and CTD (A. Haight) to test and verify new resin developed with subscale CCT magnets, partly supported by DOE - SBIR.**

- **We have been running three independent studies at LBNL:**
  - **Developing approach of preparing epoxy/filler suitable for superconducting magnets, especially epoxy nanocomposite.**
  - **Explore the possibility of high toughness, or high thermoconductivity (more for HTS), and high specific heat epoxy, and processing parameters.**

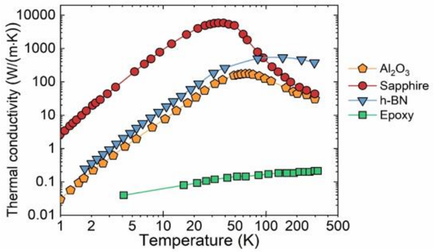
This presentation.

Disclaimer: It is an academic excise. Methods used have no connections to work at industry (CTD).

Design of Epoxy Composite with High Thermoconductivity

High thermoconductivity fillers

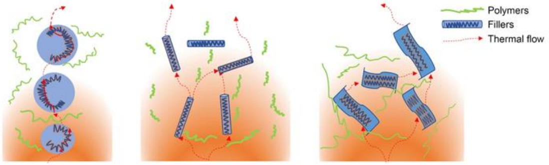
Electrical insulation fillers with high thermoconductivity at low temperature[1-3]



Epoxy resin: CTD 528

- Low Toxicity
- Long Pot-Life
- Low Viscosity, <1000 cP @ 25 ˚C
- Excellent adhesion to fibers and fillers

Filler shapes can make a big difference.

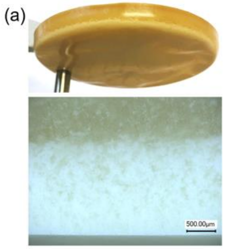


[1] Bagrets N., Otten S., Weiss K. P., et al. Thermal and mechanical properties of advanced impregnation materials for HTS cables and coils[C]. IOP Conference Series: Materials Science and Engineering IOP Publishing, 2015.
[2] Lake Shore Cryotronics Inc. Appendix I: Cryogenic reference tables[EB/OL]. https://www.lakeshore.com/docs/default-source/product-downloads/literature/lstc_appendixi_l.pdf
[3] Sichel E. K., Miller R. E., Abrahams M. S., et al. Heat capacity and thermal conductivity of hexagonal pyrolytic boron nitride[J]. Physical Review B, 1976,13(10):4607-4611.

Preparation of the Epoxy Nanocomposite

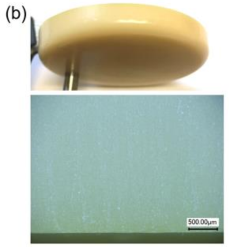
To avoid agglomeration and sediment

(a)



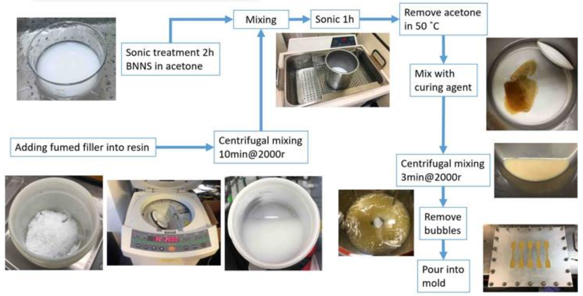
Sediment

(b)



Good Dispersion

Our Preparation Process


Sonic treatment 2h BNNS in acetone
Mixing
Sonic 1h
Remove acetone in 50 ˚C
Mix with curing agent
Adding fumed filler into resin
Centrifugal mixing 10min@2000r
Centrifugal mixing 3min@2000r
Remove bubbles
Pour into mold

Solution used by us:

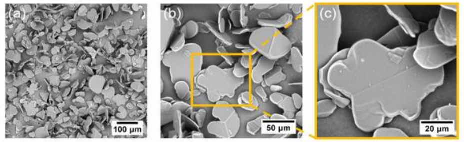
Supersonic treatment

Adding fumed filler

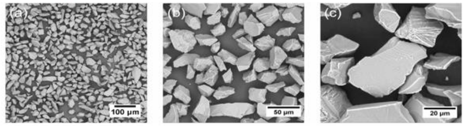
Centrifugal mixing

Filler shapes can make a big difference. We have tried several fillers.

BNmp



Sapphire



Fillers we tried

BNmp

(Boron nitride micro particle)

- Single-crystal filler
- Plate shape
- Mean particle size 45μm

BNnp

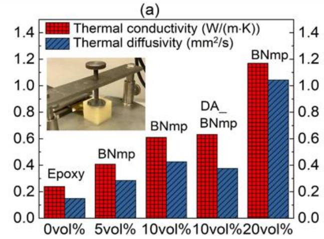
(Boron nitride nanoparticle)

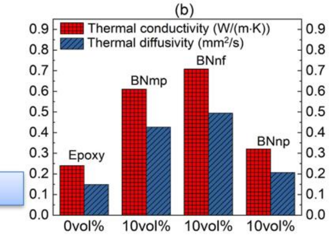
- Diameter 70-80nm

BNnf

(Boron nitride nanoflake)

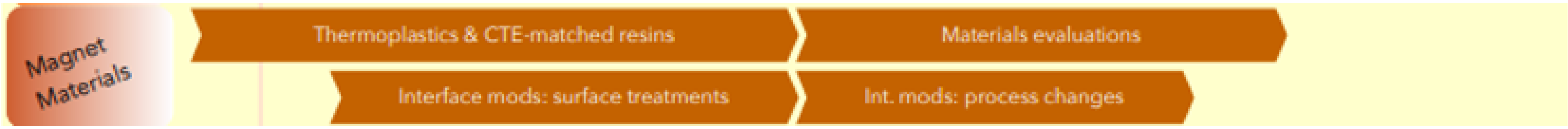
- Lateral size 500-600nm





Enhancement of thermoconductivity 10vol% BNnf > BNmp > BNnp

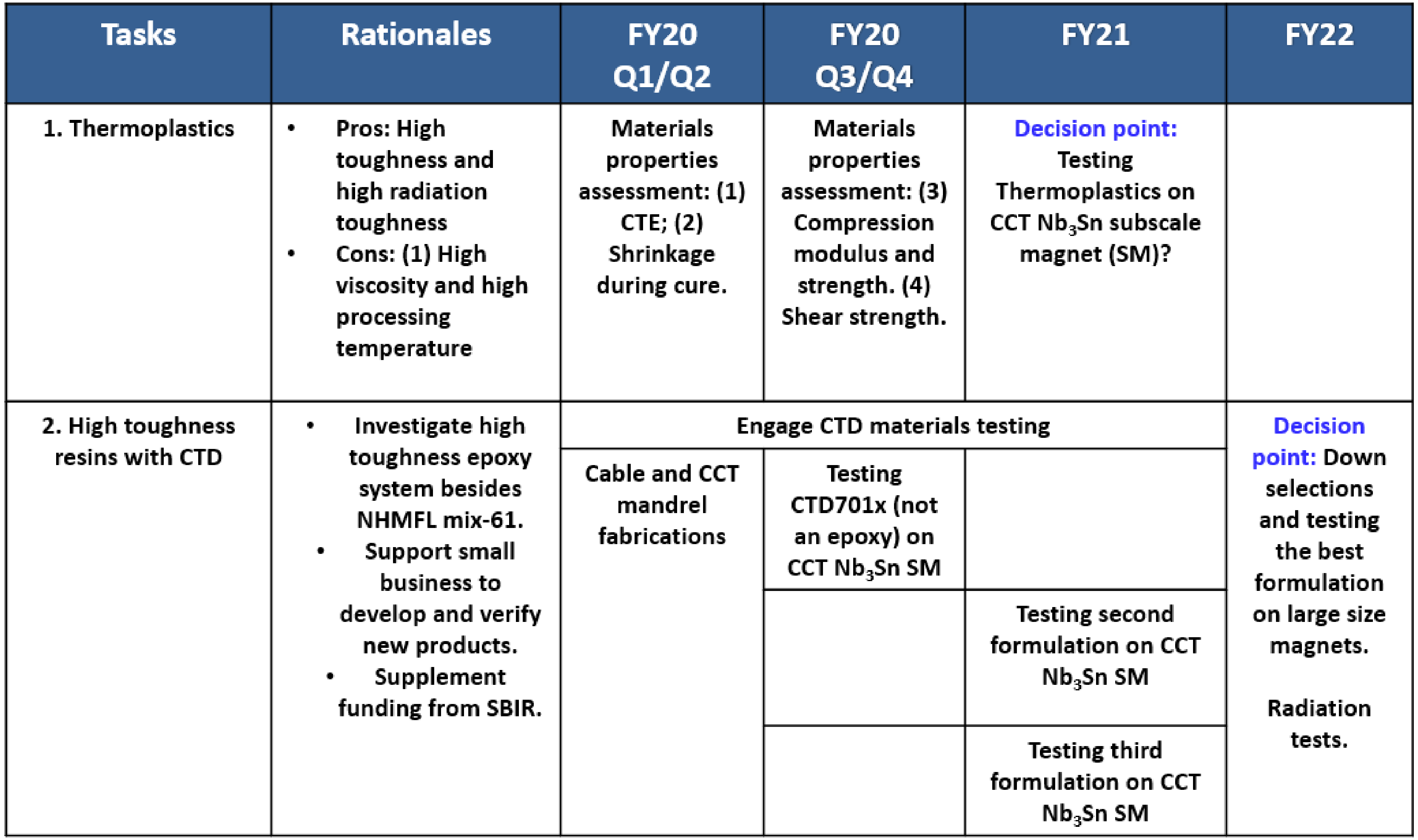
Recent studies at FNAL (Steve Krave)



Driving technical questions:

1. How do magnet materials directly affect magnet training and performance?
2. How can magnet materials be designed to facilitate improved magnet performance?
3. Can improved processes lead to more cost effective and robust magnet designs?

Recent studies at FNAL (Steve Krave)

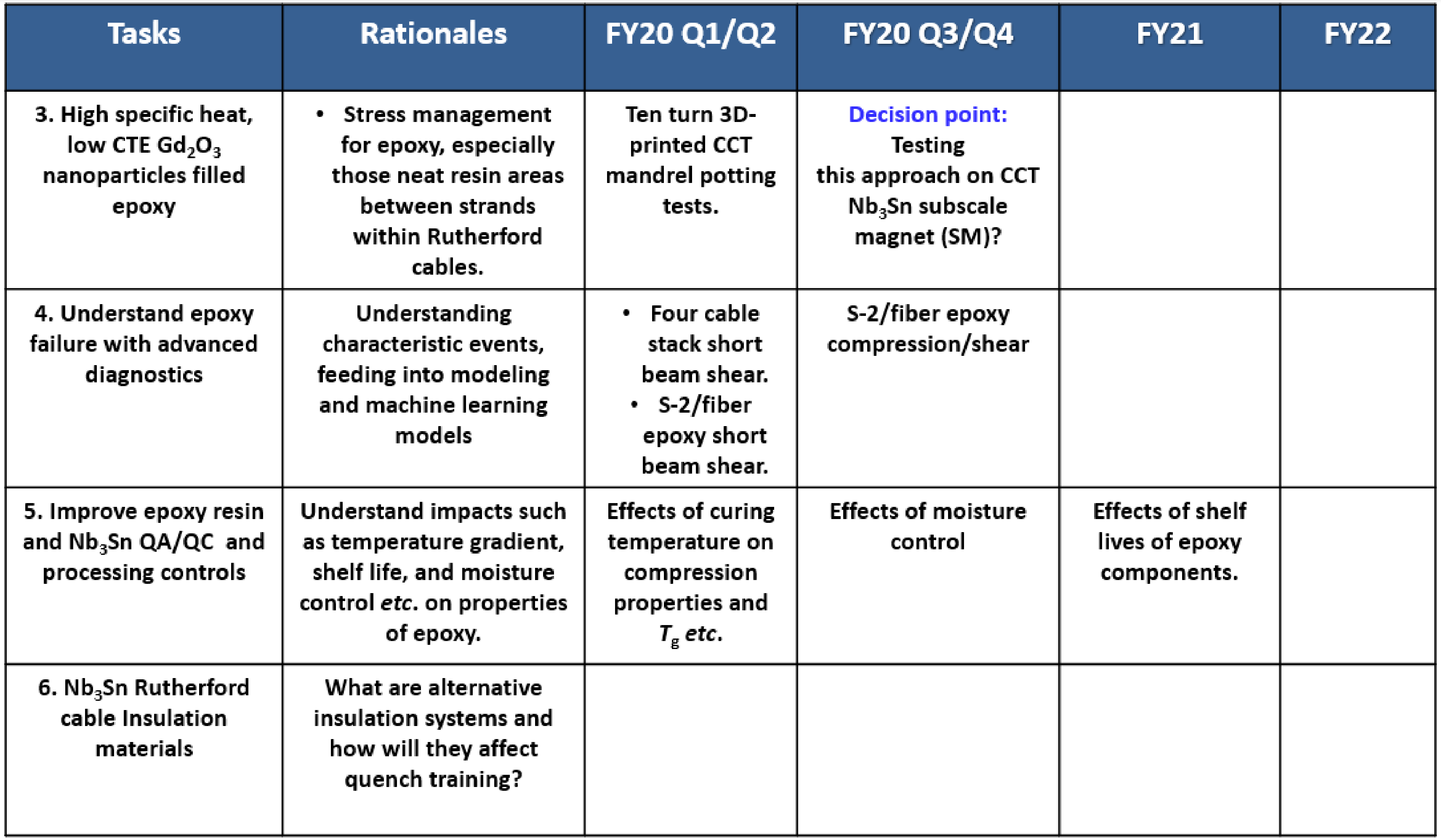


Tasks	Rationales	FY20 Q1/Q2	FY20 Q3/Q4	FY21	FY22
1. Thermoplastics	• Pros: High toughness and high radiation toughness • Cons: (1) High viscosity and high processing temperature	Materials properties assessment: (1) CTE; (2) Shrinkage during cure.	Materials properties assessment: (3) Compression modulus and strength. (4) Shear strength.	**Decision point:** Testing Thermoplastics on CCT Nb_3Sn subscale magnet (SM)?	
2. High toughness resins with CTD	• Investigate high toughness epoxy system besides NHMFL mix-61. • Support small business to develop and verify new products. • Supplement funding from SBIR.	Engage CTD materials testing			**Decision point:** Down selections and testing the best formulation on large size magnets. Radiation tests.
		Cable and CCT mandrel fabrications	Testing CTD701x (not an epoxy) on CCT Nb_3Sn SM		
				Testing second formulation on CCT Nb_3Sn SM	
				Testing third formulation on CCT Nb_3Sn SM	

Still exciting but no hands on time to complete work

Good progress here, ready to impregnate CCT with 701X

Recent studies at FNAL (Steve Krave)



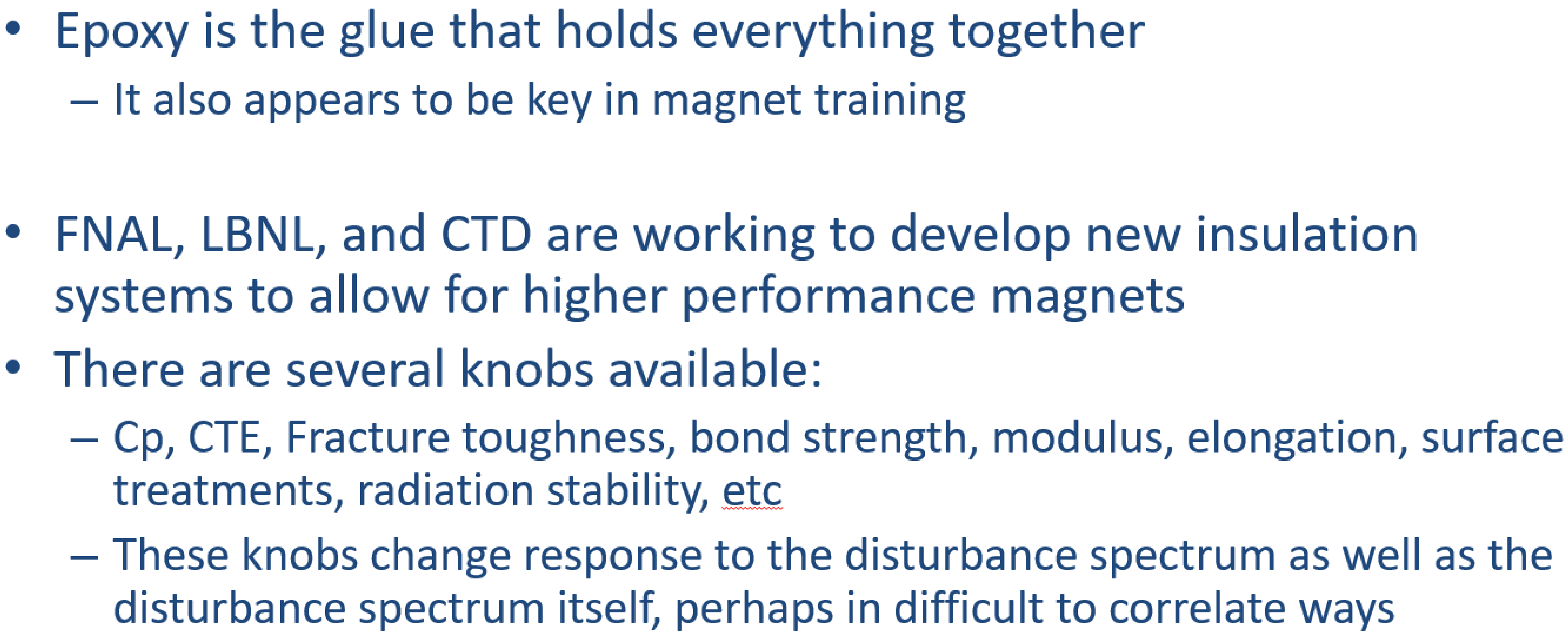
Tasks	Rationales	FY20 Q1/Q2	FY20 Q3/Q4	FY21	FY22
3. High specific heat, low CTE Gd_2O_3 nanoparticles filled epoxy	- Stress management for epoxy, especially those neat resin areas between strands within Rutherford cables.	Ten turn 3D-printed CCT mandrel potting tests.	Decision point: Testing this approach on CCT Nb_3Sn subscale magnet (SM)?		
4. Understand epoxy failure with advanced diagnostics	Understanding characteristic events, feeding into modeling and machine learning models	- Four cable stack short beam shear. - S-2/fiber epoxy short beam shear.	S-2/fiber epoxy compression/shear		
5. Improve epoxy resin and Nb_3Sn QA/QC and processing controls	Understand impacts such as temperature gradient, shelf life, and moisture control *etc.* on properties of epoxy.	Effects of curing temperature on compression properties and T_g *etc.*	Effects of moisture control	Effects of shelf lives of epoxy components.	
6. Nb_3Sn Rutherford cable Insulation materials	What are alternative insulation systems and how will they affect quench training?				

Some progress at LBNL, not enough resources to complete work

Useful data and understanding, not used in ML models

Data from AUP project, exciting info

Recent studies at FNAL (Steve Krave)



- Epoxy is the glue that holds everything together
  - It also appears to be key in magnet training

- FNAL, LBNL, and CTD are working to develop new insulation systems to allow for higher performance magnets
- There are several knobs available:
  - Cp, CTE, Fracture toughness, bond strength, modulus, elongation, surface treatments, radiation stability, etc
  - These knobs change response to the disturbance spectrum as well as the disturbance spectrum itself, perhaps in difficult to correlate ways

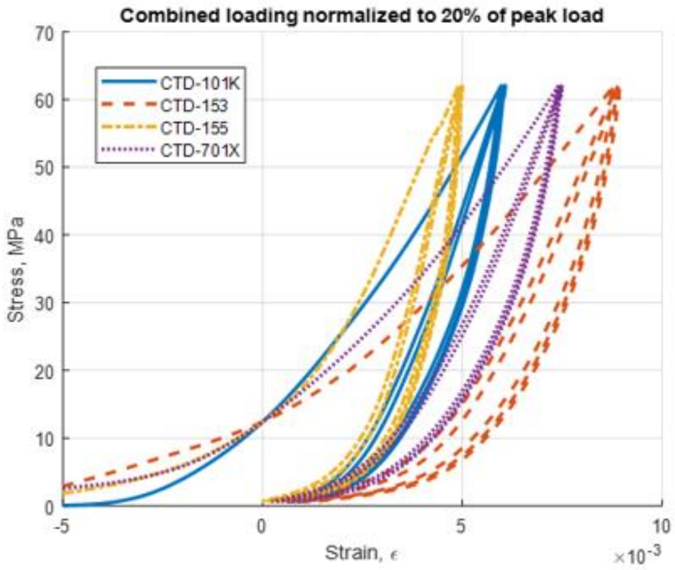
Recent studies at FNAL (Steve Krave)

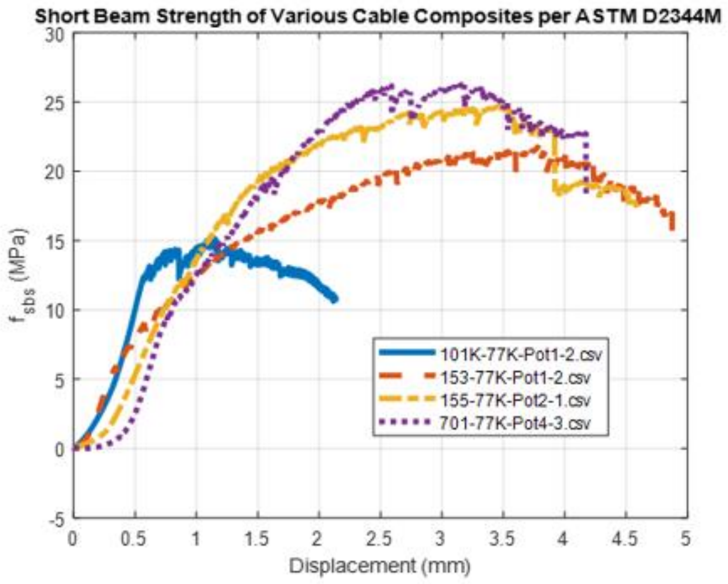
We have measured a handful of candidate resins in collaboration with CTD

- CTD 155 and 153, toughened epoxy resins
- CTD 701 Thermoset polyolefin, extremely tough

LBNL will impregnate a subscale CCT with the 701 system in the near future

Coming up: Filled CTD 155

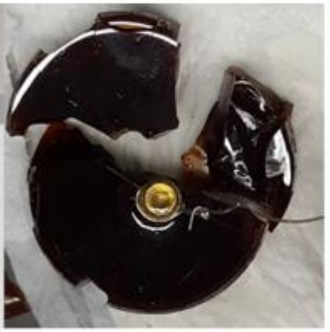




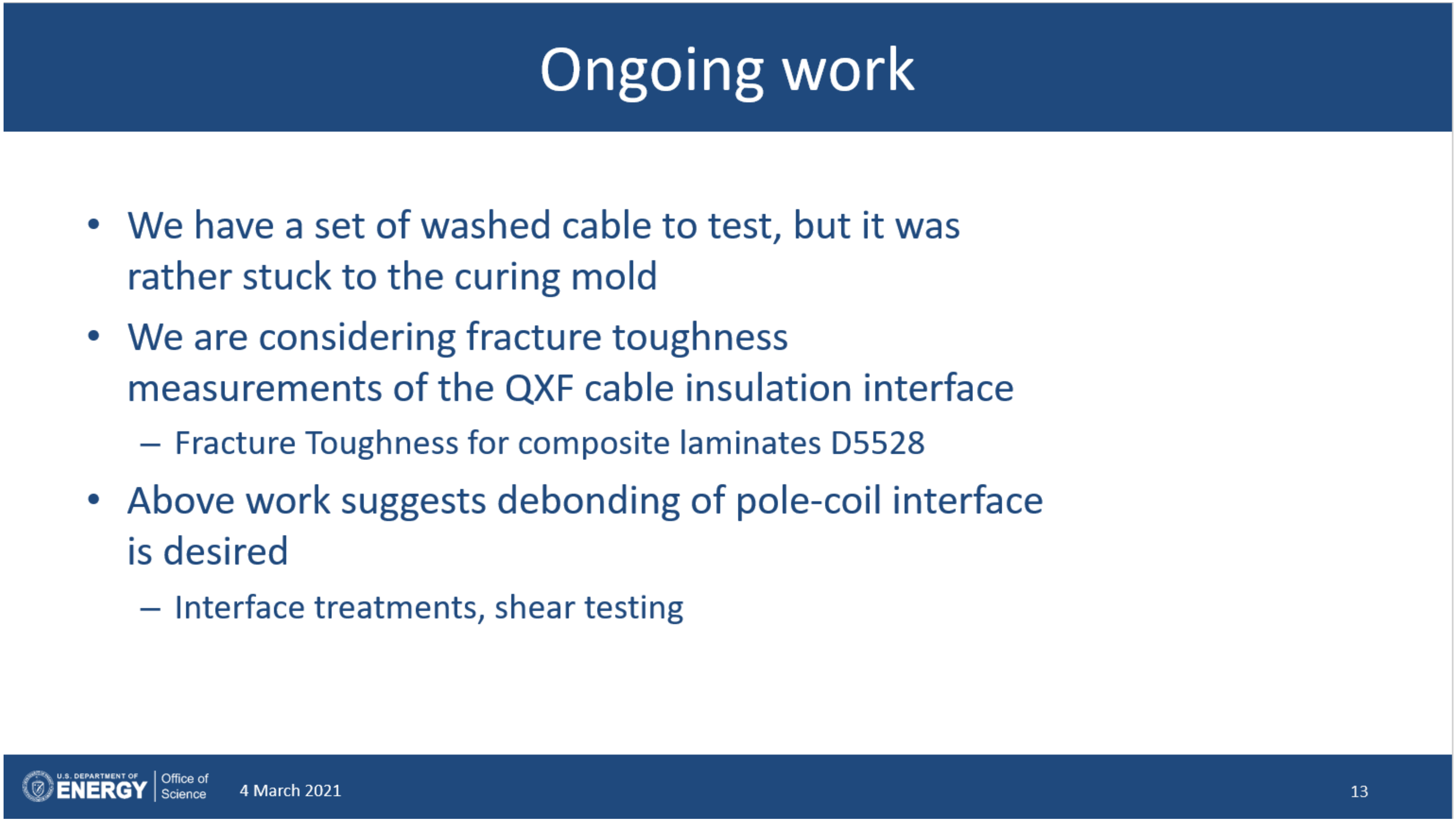

CTD 153 After 5 Thermal Cycles


CTD 155 After 10 Thermal Cycles


CTD 701X After 10 Thermal Cycles


CTD 101K After 10 Thermal Cycles

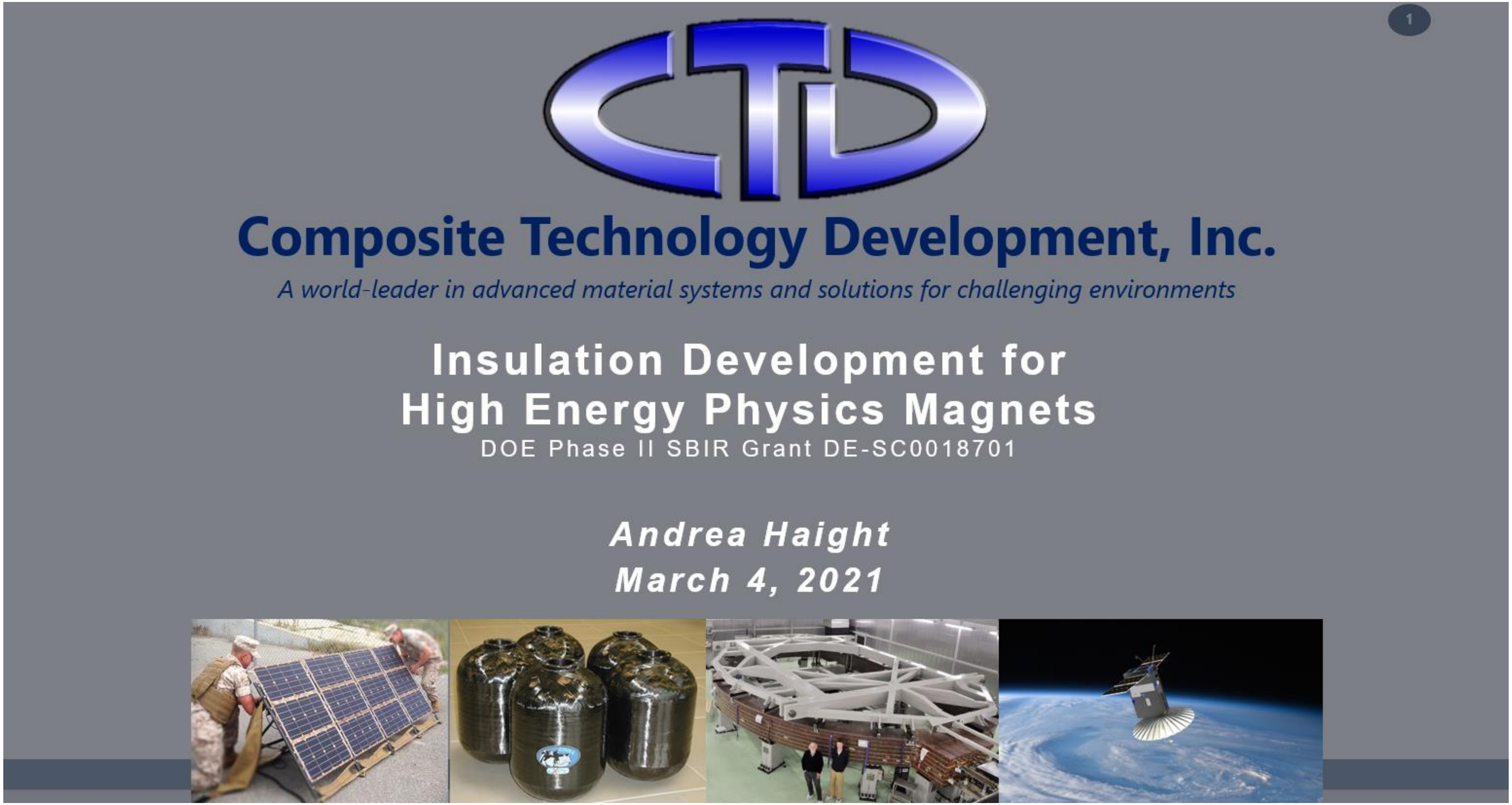
Recent studies at FNAL (Steve Krave)



Ongoing work

- We have a set of washed cable to test, but it was rather stuck to the curing mold
- We are considering fracture toughness measurements of the QXF cable insulation interface
  - Fracture Toughness for composite laminates D5528
- Above work suggests debonding of pole-coil interface is desired
  - Interface treatments, shear testing

Recent studies at CTD (Andrea Haight)



Composite Technology Development, Inc.

A world-leader in advanced material systems and solutions for challenging environments

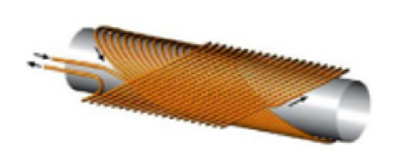
Insulation Development for High Energy Physics Magnets

DOE Phase II SBIR Grant DE-SC0018701

Andrea Haight

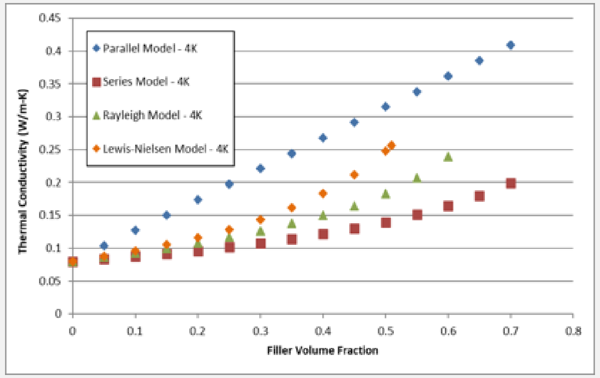
March 4, 2021

Recent studies at CTD (Andrea Haight)

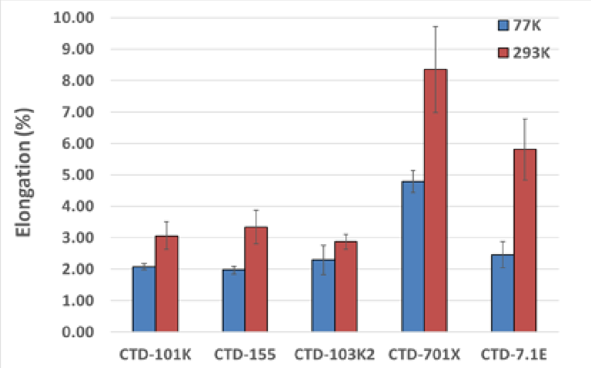


Program Goal:

Address long magnet training cycles through insulation optimization



Formulation & evaluation of thermally conductive insulation



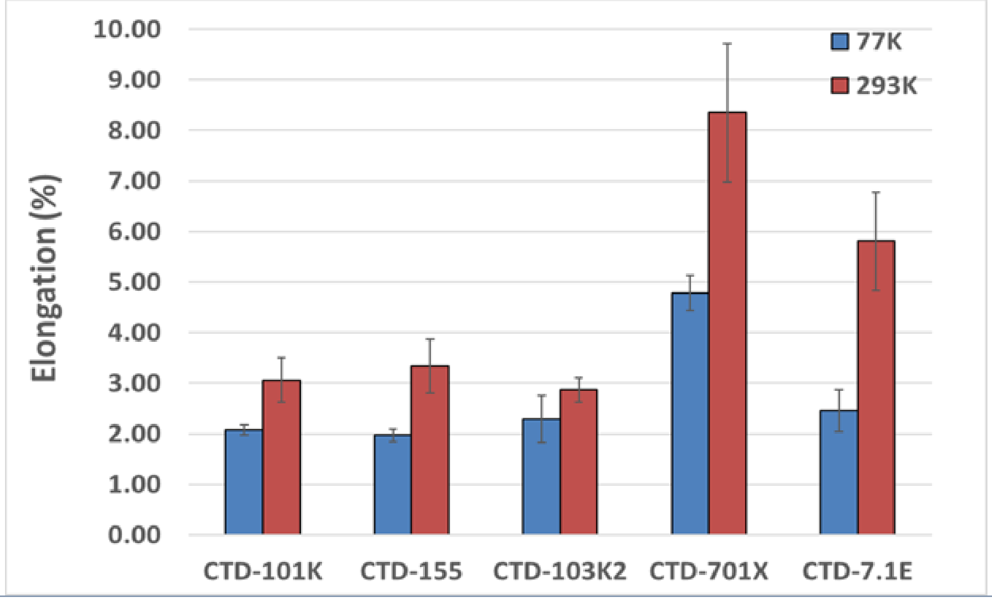
Formulation & evaluation of high strain insulation

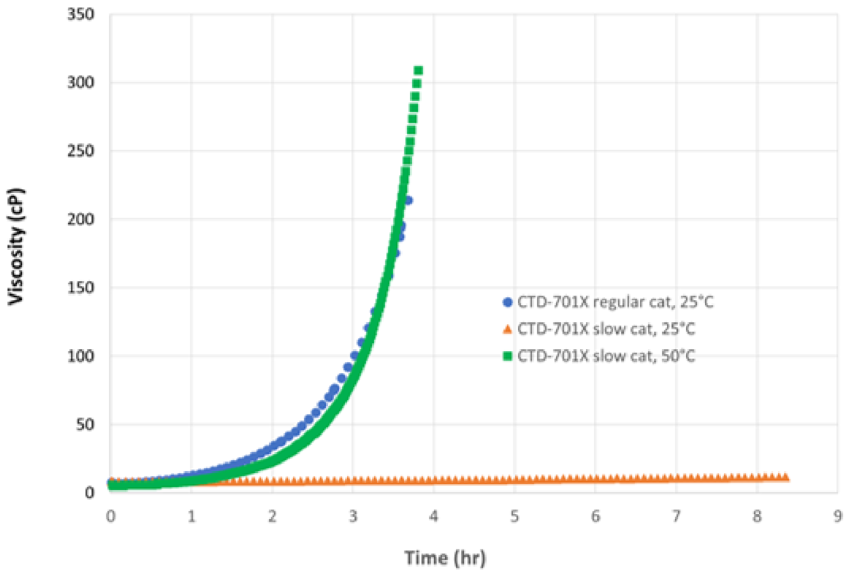
- **Optimization of high strain and thermally conductive insulation options**
- **Stack testing for screening options**
- **Subscale CCT magnet fabrication & testing**

Recent studies at CTD (Andrea Haight)

Phase I Toughened Resin Systems

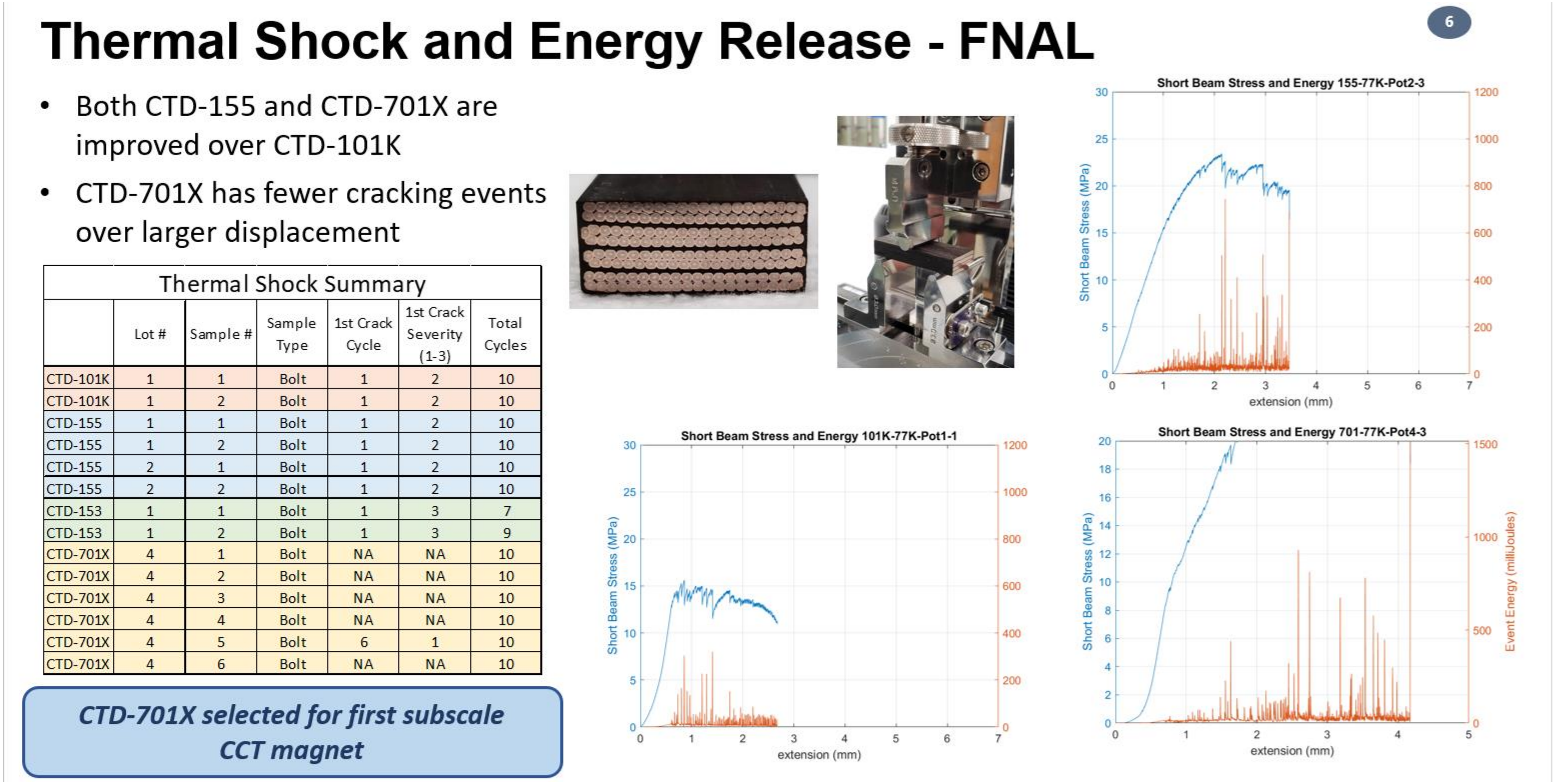
Name	Description	T_g, °C (tan δ)	Viscosity @ 60°C (cP)	
			Initial	7 hours
CTD-101K	Baseline anhydride-cured epoxy resin	150 (avg QA)	70	100
CTD-103K2	Long pot life 2-part formulation similar to CTD-101K	141	61	61.5 (4.5 hr)
CTD-103LT	Lightly toughened version of CTD-103K2	126	24	71
CTD-155	Anhydride-cured epoxy resin with reactive rubber toughener	147	125	127
CTD-701X	Extremely low viscosity, tough polyolefin resin system	131	25 (@ 25°C)	70 (90 min, 25°C)
CTD-7.1E	Low viscosity, toughened amine-cured epoxy resin	72	225	Approx. 2 hr





- Initial CTD-701X formulation showed rapid viscosity increase at 2hr (25°C)
- Catalyst change allowed a significant increase in potlife – heat required to initiate cure

Recent studies at CTD (Andrea Haight)



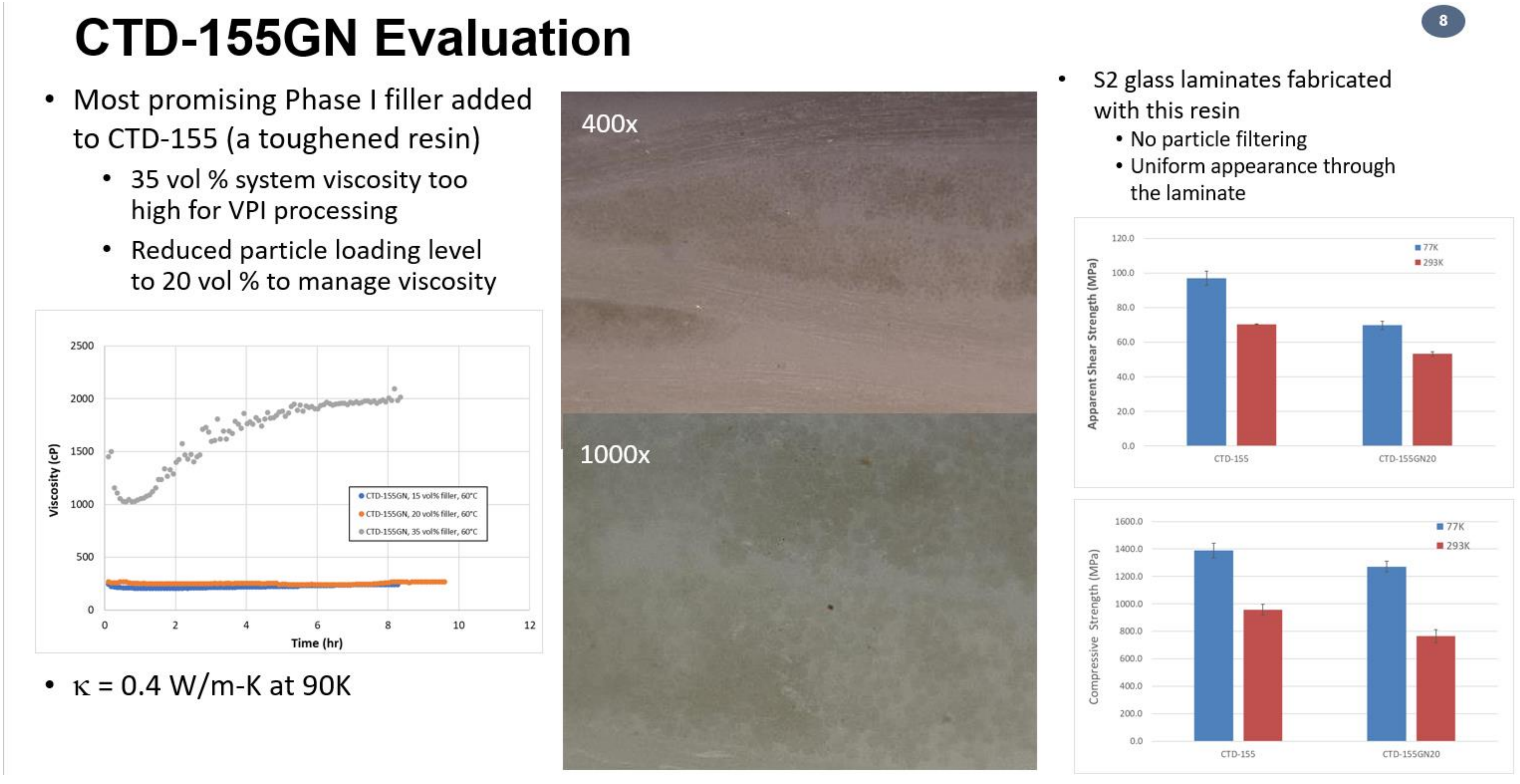
Thermal Shock and Energy Release - FNAL

- Both CTD-155 and CTD-701X are improved over CTD-101K
- CTD-701X has fewer cracking events over larger displacement

Thermal Shock Summary						
	Lot #	Sample #	Sample Type	1st Crack Cycle	1st Crack Severity (1-3)	Total Cycles
CTD-101K	1	1	Bolt	1	2	10
CTD-101K	1	2	Bolt	1	2	10
CTD-155	1	1	Bolt	1	2	10
CTD-155	1	2	Bolt	1	2	10
CTD-155	2	1	Bolt	1	2	10
CTD-155	2	2	Bolt	1	2	10
CTD-153	1	1	Bolt	1	3	7
CTD-153	1	2	Bolt	1	3	9
CTD-701X	4	1	Bolt	NA	NA	10
CTD-701X	4	2	Bolt	NA	NA	10
CTD-701X	4	3	Bolt	NA	NA	10
CTD-701X	4	4	Bolt	NA	NA	10
CTD-701X	4	5	Bolt	6	1	10
CTD-701X	4	6	Bolt	NA	NA	10

CTD-701X selected for first subscale CCT magnet

Recent studies at CTD (Andrea Haight)



CTD-155GN Evaluation

- Most promising Phase I filler added to CTD-155 (a toughened resin)
  - 35 vol % system viscosity too high for VPI processing
  - Reduced particle loading level to 20 vol % to manage viscosity

- κ = 0.4 W/m-K at 90K

- S2 glass laminates fabricated with this resin
  - No particle filtering
  - Uniform appearance through the laminate

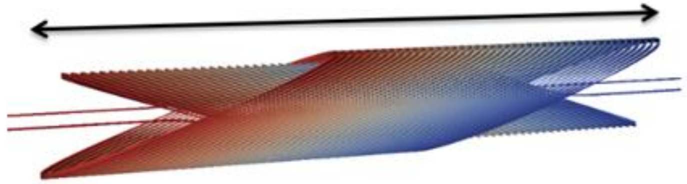
Recent studies at CTD (Andrea Haight)

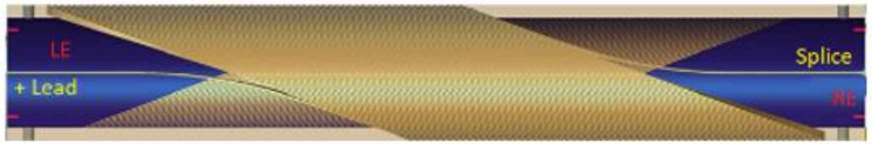
Upcoming Work

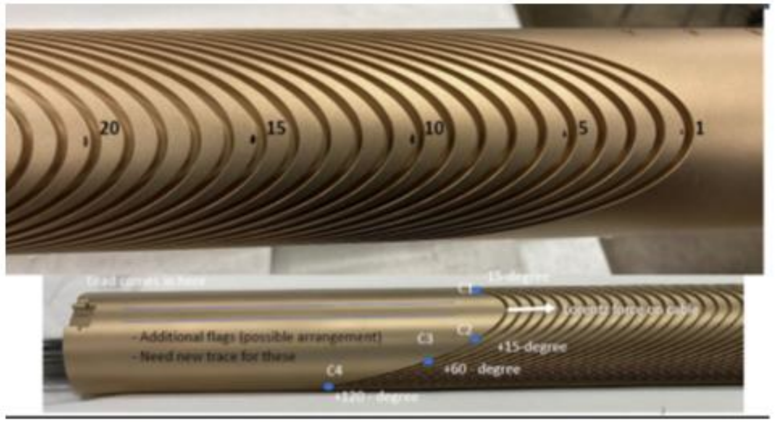
- Subscale CCT impregnation at LBNL
  - CTD-701X
  - Testing to follow shortly thereafter
- Completion of CTD-155GN20 laminate testing at CTD
- Stack evaluation of CTD-155GN20 at FNAL
- Second subscale CCT fabrication at LBNL
  - Planned as CTD-155GN
- Third subscale CCT resin system TBD

CCT sub magnet (D. Arbelaez, US MDP 2021)

45 turns / layer = 500 mm physical length







Acoustic sensors + quench antenna (M. Marchevsky, US MDP 2021)

www.cern.ch