

*Fiber Optic Based Thermometry  
System for Superconducting RF  
Cavities  
Technical Review*

August 23<sup>rd</sup>, 2011

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President/CEO

MicroXact Inc.

# Agenda:

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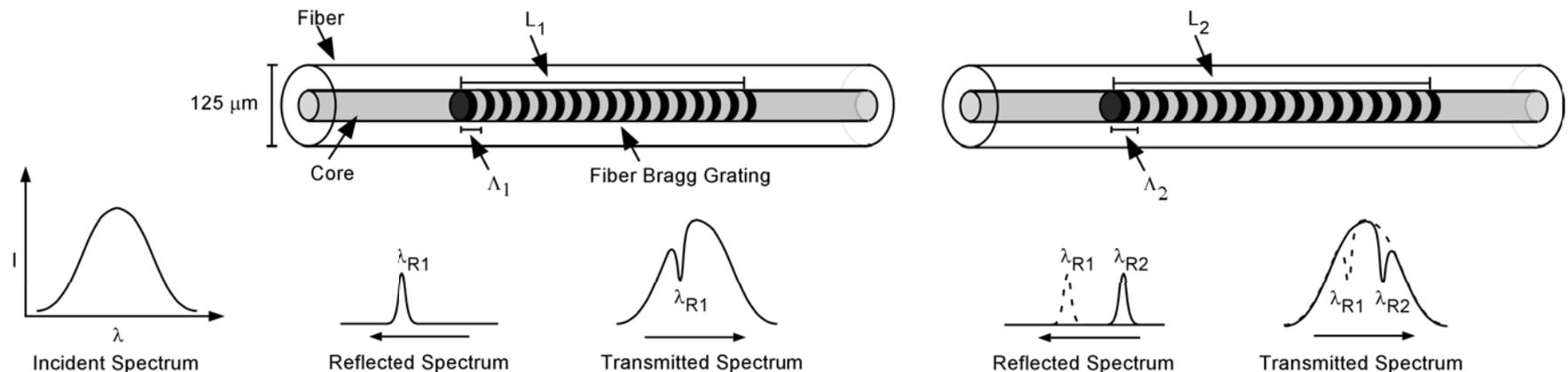
- Addressed Problem
- MicroXact Solution
- Project Goals
- Technical Progress
- Financial Standing

# Addressed Problem



# FBG Temperature Sensors: Principle of Operation

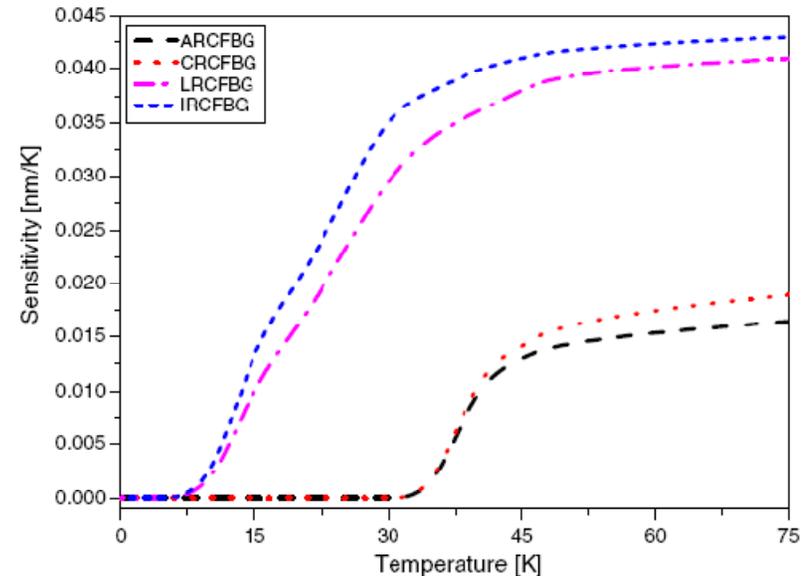
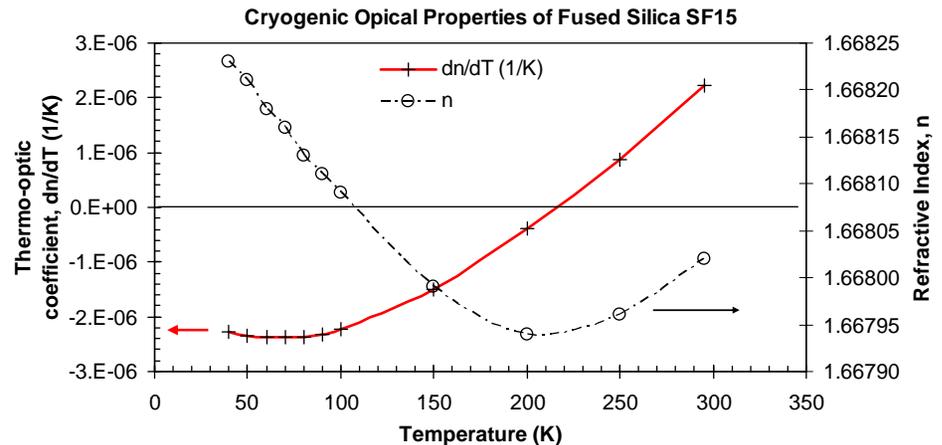
- Spectral position FBG reflectivity maximum:  $\lambda_{FBG} = 2\Lambda n$
- Temperature-induced changes:  $\Delta\lambda_{FBG} = 2\left(n\frac{\Delta\Lambda}{\Delta T} + \Lambda\frac{\Delta n}{\Delta T}\right)\Delta T = 2\Lambda\left(\frac{\partial n}{\partial T} + n\varepsilon\right)\Delta T$
- Two sources of temperature sensitivity: thermo-optic effect and thermal expansion.



# Cryogenic FBG Temperature Sensors: State of the Art

- Both thermo-optic coefficient and thermal expansion coefficients are very small for silica @ cryo-T.
- This leads to very poor accuracy/resolution below ~70K.
- Metal-coated optical fibers were suggested to boost CTE at cryogenic temperatures.
- High stress at silica/metal interface due to dissimilar CTEs causing delamination/fiber breakage at coating thicknesses >200nm, which severely limit T resolution:

$$P(T) = \frac{1}{\lambda_{SiO_2} + \lambda_c} \left( \frac{1}{\Delta T} \int_{T_0}^T \alpha^* d\tau - \alpha_{SiO_2} \right) \Delta T$$

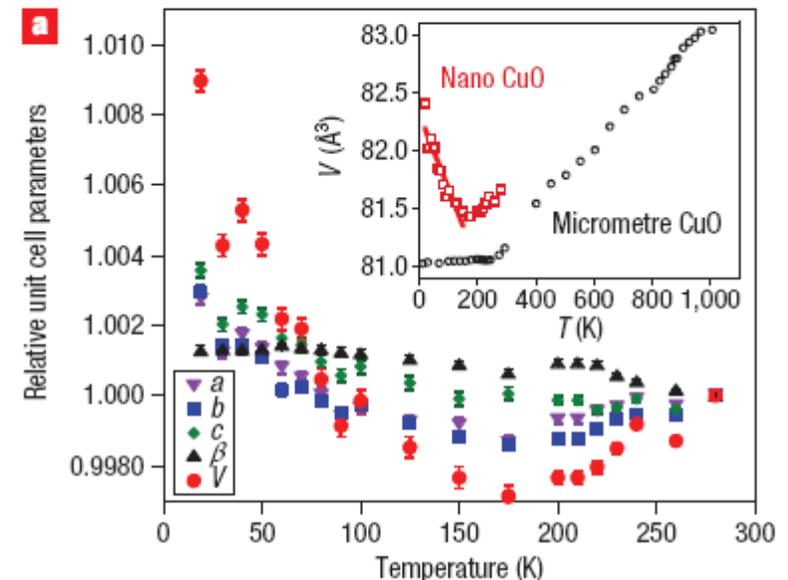


R. Rajini-Kumar et al. Performance evaluation of metal-coated fiber Bragg grating sensors for sensing cryogenic temperature *Cryogenics* 48 (2008) 142–147

# Proposed Solution

## Proposed concept

- CTE of  $-1.1 \times 10^{-4} \text{K}^{-1}$  (1000x higher than that of silica) was reported by Zheng et al. for CuO nanoparticles down to at least 4K.
- If such a material would be embedded into polymer matrix and FBG will be recoated with such material modeling predicted few mK resolution down to 2K and below.
- CuO NP/polymer nanocomposite FBG coating to get mK resolution of FBG sensor @2K.
- Polymer package for convenient mounting of the sensor assembly of SCRF cavity surface.
- Single instrument/single feedthrough required.
- Low cost per sensing point
- If realized, this would represent an ultimate solution for SCRF cavity characterization and multiple other cryogenic applications.



Zheng, X. G. et al. *Nature Nanotech.* 3, 724–726 (2008)

# Project Goals

# Milestone Schedule

**Milestone 1** (End of Year 1): Demonstrate experimentally that the proposed nanocomposite coating enhances the resolution/reliability of single FBG sensors at cryogenic temperatures sufficiently to meet the program goals (10mK or better accuracy at 2K-4.3K range).

**Milestone 2** (End of Year 1): Demonstrate experimentally distributed fiber optic temperature sensing on "dummy" can with Nb flat plate.

**Milestone 3** (End of Year 2): Experimentally demonstrate distributed fiber optic temperature measurements with 10mK or better accuracy at conditions similar to those used in SRF cavity testing.

**Milestone 4** (End of Year 2): Demonstrate the performance of distributed fiber optic cryogenic temperature sensor with a "dummy" can and heaters (10mK or better accuracy at 2K-4.3K range).

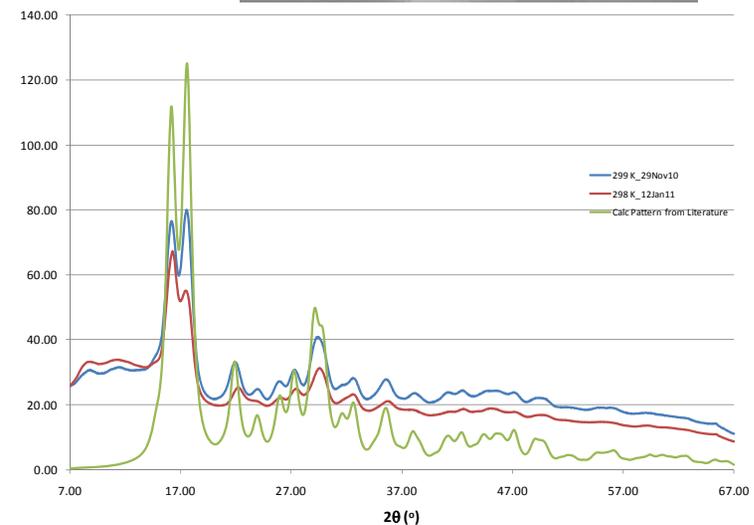
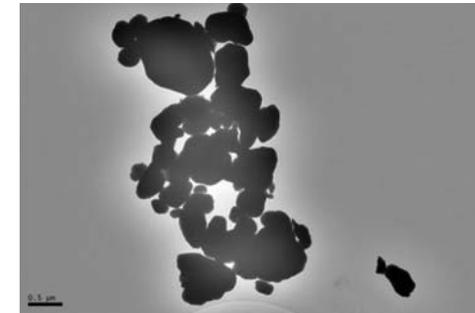
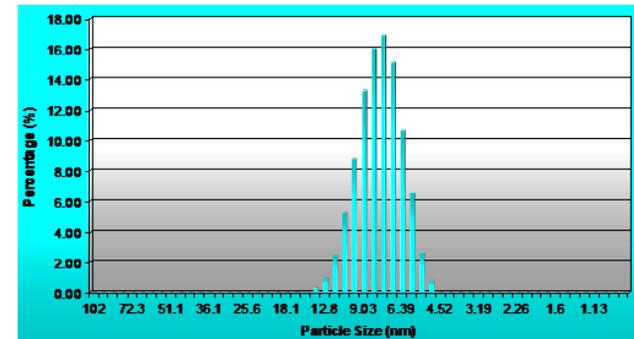
**Milestone 5** (End of Year 3): Demonstrate fiber optic SRF cavity characterization system that will have the following parameters: 1) sensor density at least as high as with any existing systems, 2) temperature accuracy of 10mK, 3) higher cavity surface coverage than what is available with fixed thermometry systems, and 4) temporal resolution of at least 1Hz.

# Technical Progress

# CuO nanoparticle synthesis and post-processing

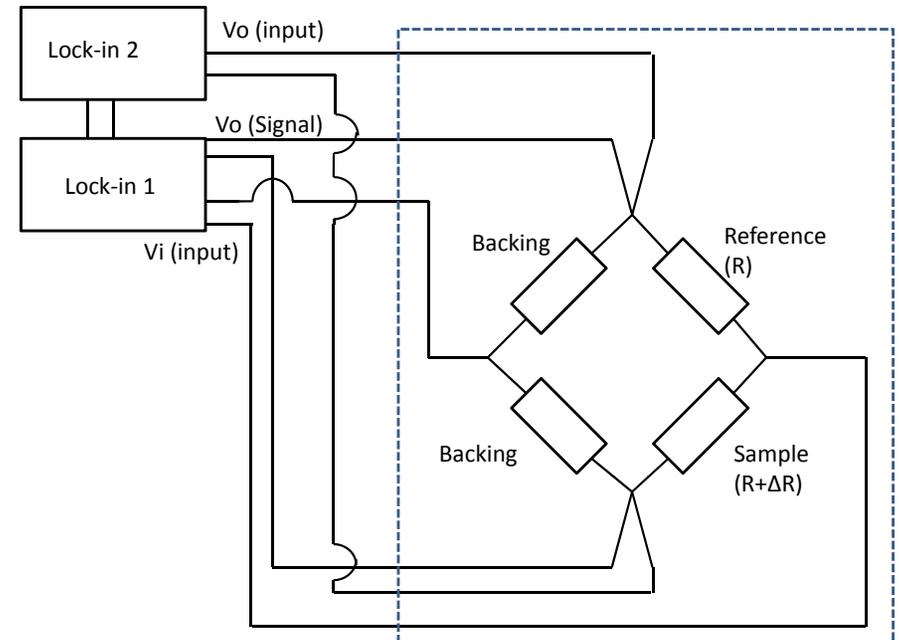
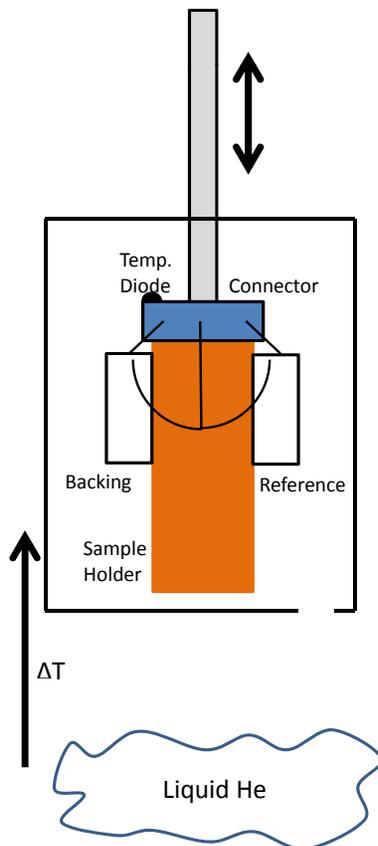
The following CuO nanomaterials were investigated during the course of the project:

- 1) MicroXact-synthesized CuO NPs.
- 2) CuO NPs from American Elements.
- 3) CuO NPs formed by ball milling of Sigma Aldrich CuO material.
- 4) Ball-milled CuO NPs from American Elements
- 5) Ball-milled MicroXact-synthesized CuO NPs
- 6) All of the above with different surface treatments, post processing (electron irradiation, annealing), etc.
- 7) Compacted CuO NPs from Institute of Metal Physics, Urals Branch of the Russian Academy of Sciences, Ekaterinburg, Russia.
- 8) We tried our best to get the sample from Zheng's group, but in-vain.

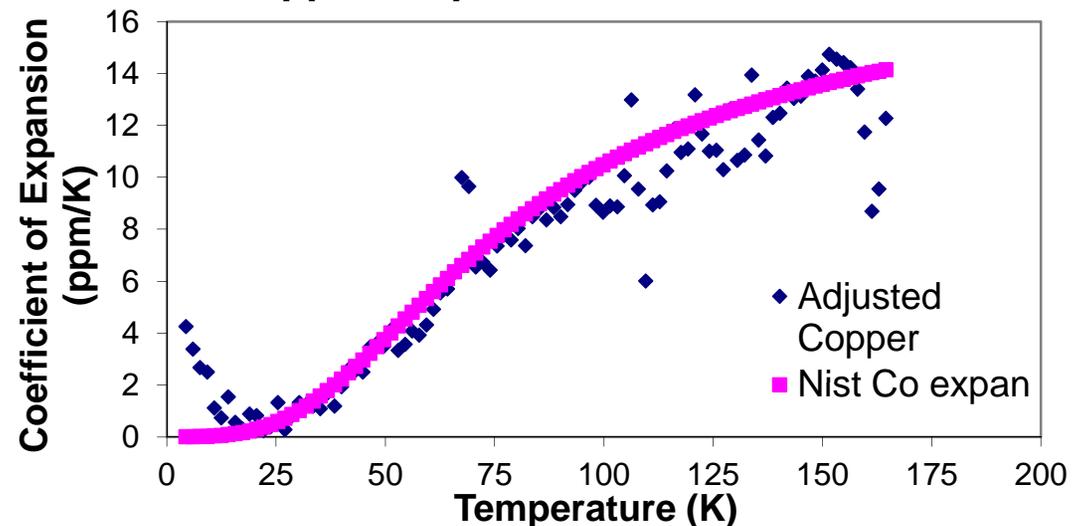


# CTE characterization setup

Error below 1ppm/K was achieved for 15K-150K range for not matched strain gages, with better accuracy for matched strain gages achievable.



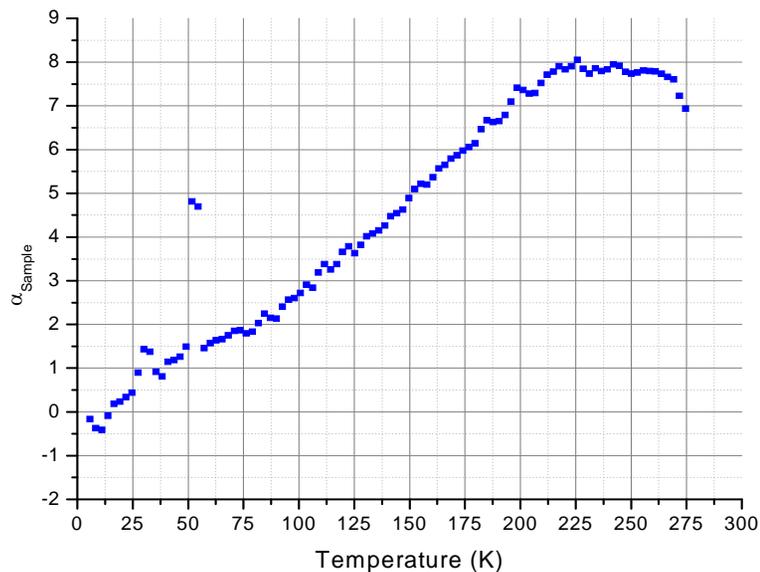
Copper Acquired vs. Known Values



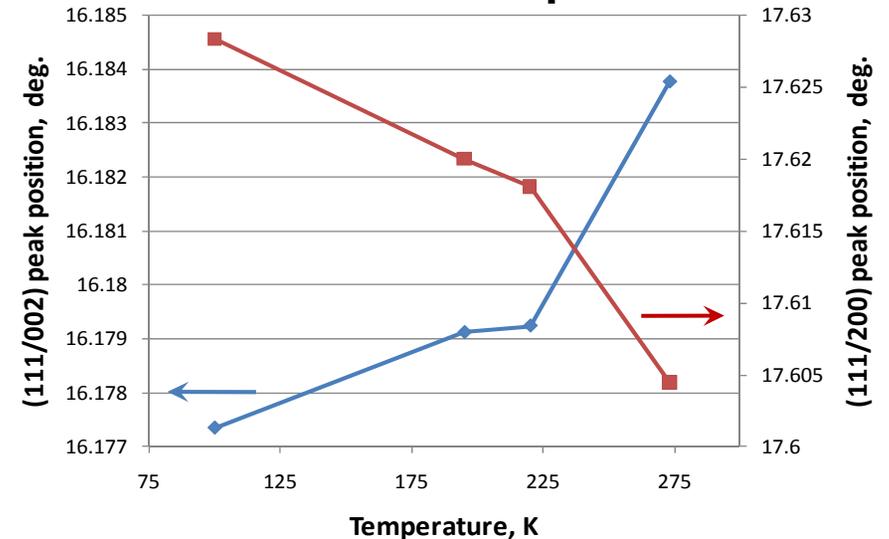
# CuO NP characterization results

We did not observed giant negative CTE of neither CuO NPs nor CuO NP-based bulk materials (nanocomposites) for any type of CuO material and any treatment tried.

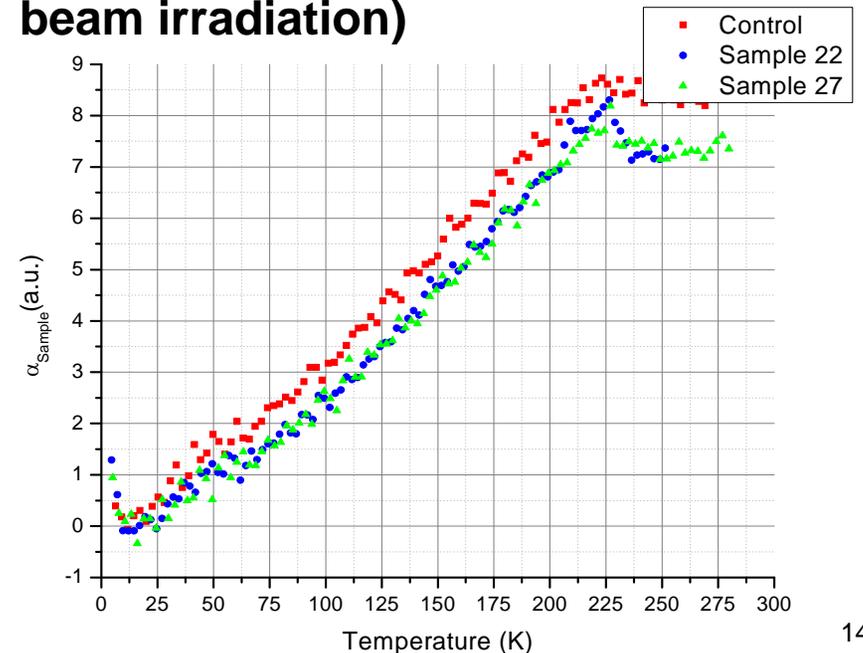
## CTE of CuO from the group of Gizhevskii et al.



## XRD studies of the powder

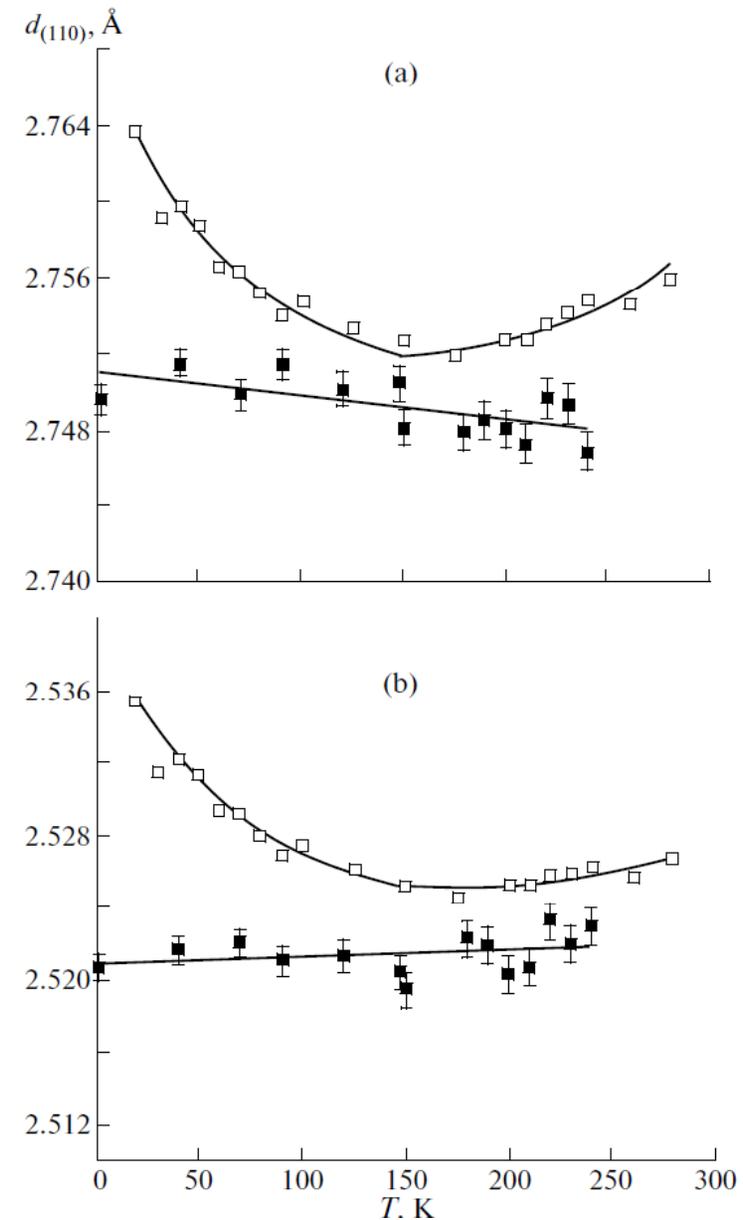
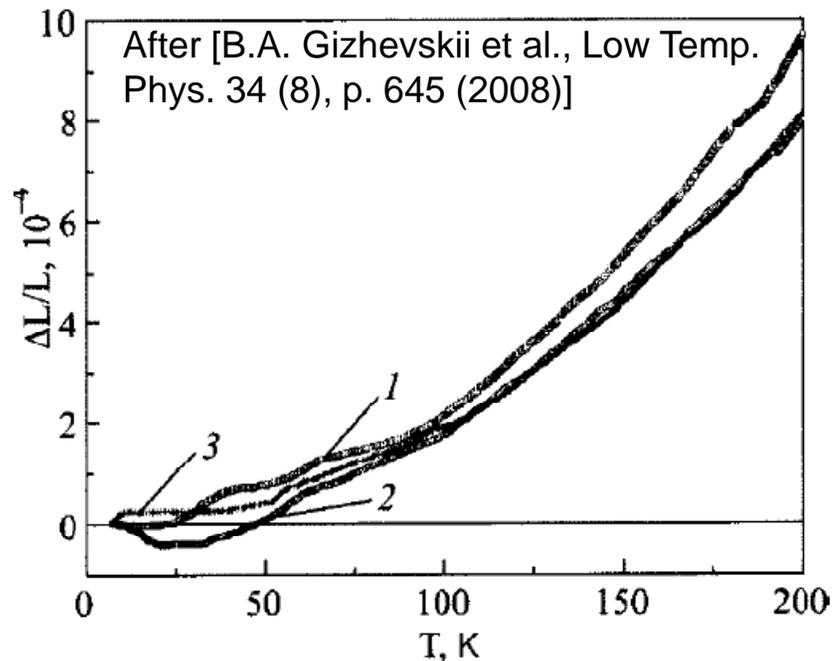


## CTE of CDC CuO (before and after e-beam irradiation)



# CuO NP characterization results by other groups

“No giant negative thermal expansion coefficient was revealed in these samples. An analysis ... showed that the thermal expansion coefficient in the nanoparticles depends strongly on preparation technique” (I.V. Golosovsky, et al., ).



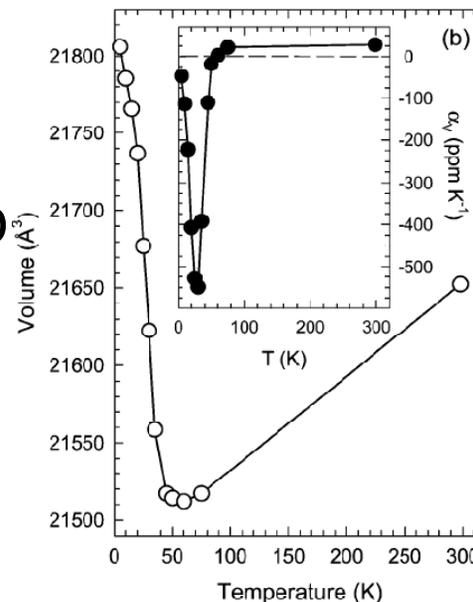
I.V. Golosovsky, et al., 2011, Kristallografiya, 2011, Vol. 56, No. 1, pp. 170–174

# Conclusions on giant negative CTE of CuO nanoparticles

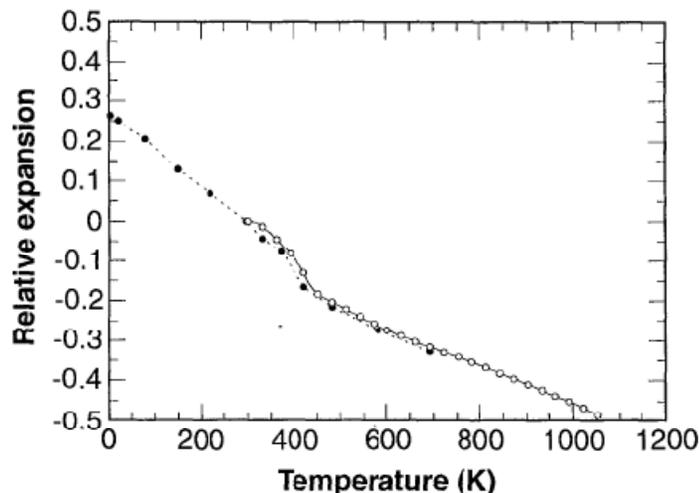
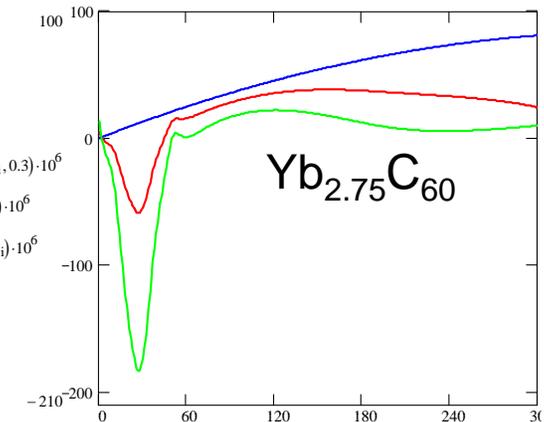
- Our XRD results are in-line with the later results by Golosovsky et al. for all different types of CuO NP fabrication and treatments and are contrary to the results published by Zheng et al.
- Our CTE measurement results are in line with earlier results by Gizhevskii et al. for all different types of CuO NP fabrication and treatments and are contrary to the results published by Zheng et al.
- We were not able to get CuO sample from Zheng et al. so it is impossible to state with certainty that giant negative CTE of CuO nanoparticles at cryogenic temperatures is ambiguous, but all our data and published so far data suggests that this is something we should not rule out completely.
- Based on that we decided to abandon CuO NP/polymer composite concept as fiber coating for cryogenic fiber optic sensors.

# Possible alternatives for fiber coatings:

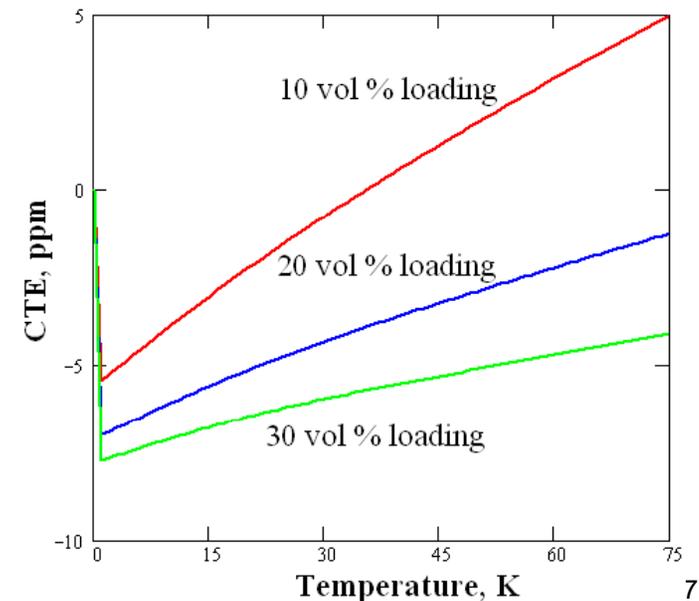
- Yb<sub>2.75</sub>C<sub>60</sub> was reported to have sizable negative CTE at 50K (based on XRD studies). CTE of bulk Yb<sub>2.75</sub>C<sub>60</sub> material is still to be measured. Too risky approach.
- ZrW<sub>2</sub>O<sub>8</sub>/polymer coating should provide ~10mK resolution.



S. Margadonna, Chem. Mater. 2005, 17, 4474-4478



T.A. Mary et al., Science, Vol. 272 (5258) pp. 90-92 (1996)

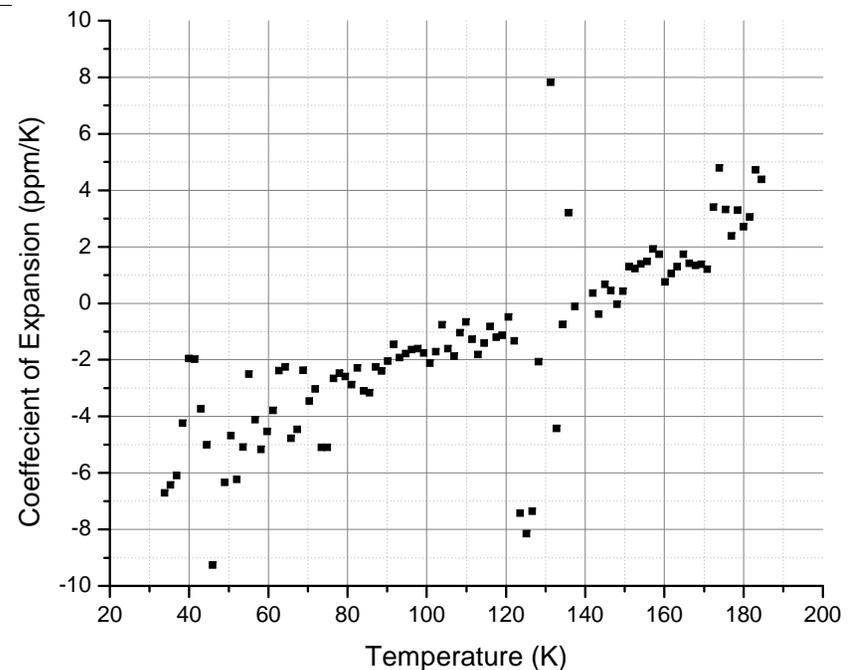
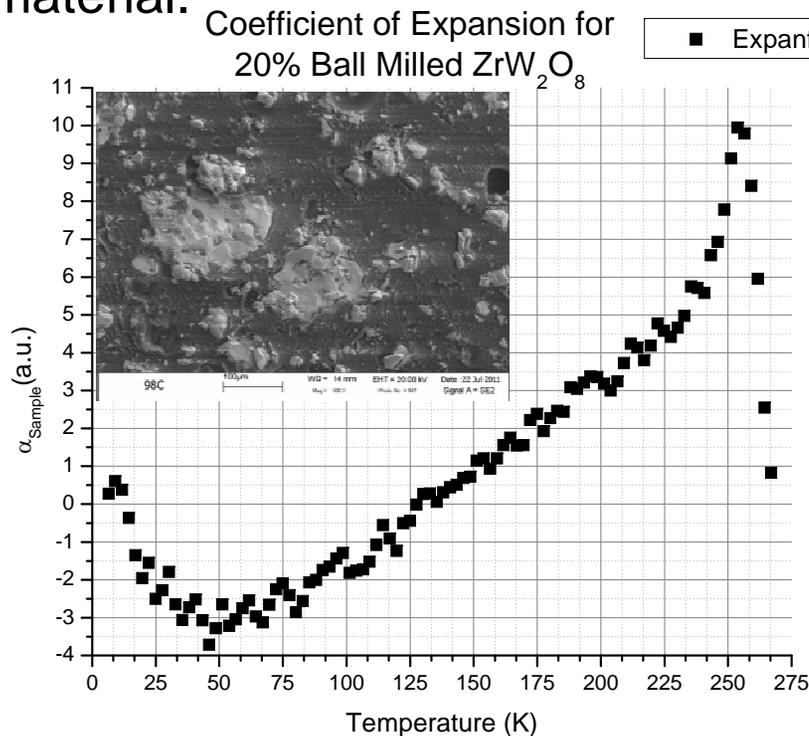


# ZrW<sub>2</sub>O<sub>8</sub> properties

- Three phases of ZrW<sub>2</sub>O<sub>8</sub> exist with only  $\alpha$ -phase sufficient for our purposes. There is a very limited supply of cubic ZrW<sub>2</sub>O<sub>8</sub>.
- With Alpha Aesar ZrW<sub>2</sub>O<sub>8</sub> particles and some post-processing we managed to get negative CTE material.

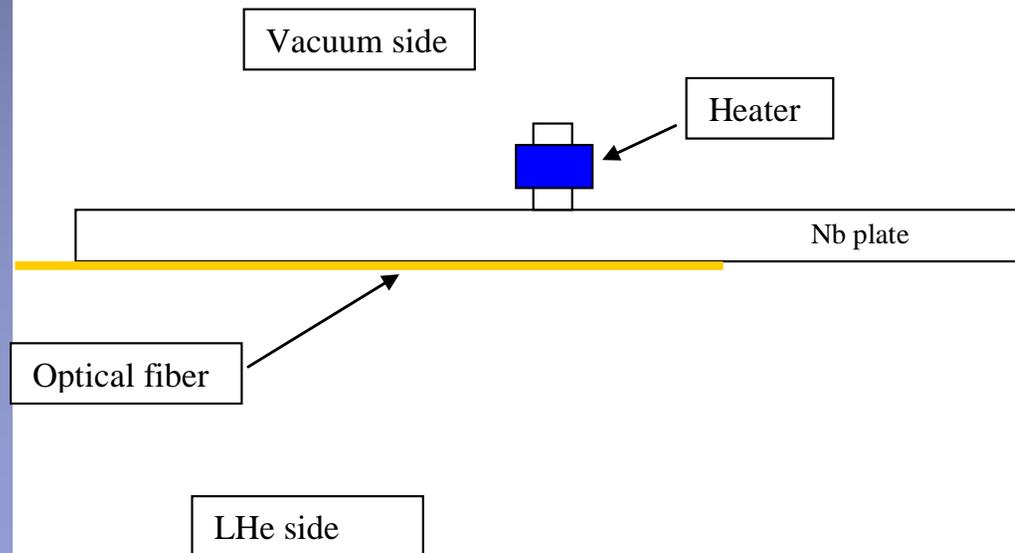
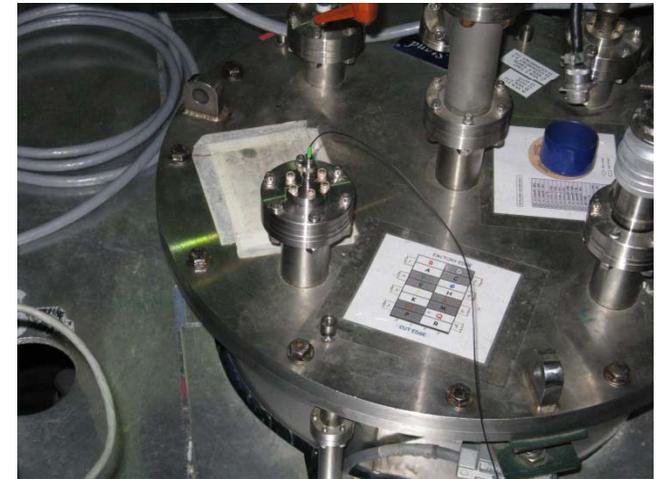
Thermal expansion ( $\Delta l/l$ )

$\alpha$ -ZrW <sub>2</sub> O <sub>8</sub> (20–430 K)	$-8.7 \times 10^{-6} \text{ K}^{-1}$
$\beta$ -ZrW <sub>2</sub> O <sub>8</sub> (430–950 K)	$-4.9 \times 10^{-6} \text{ K}^{-1}$
$\gamma$ -ZrW <sub>2</sub> O <sub>8</sub> (20–300 K)	$-1.0 \times 10^{-6} \text{ K}^{-1}$

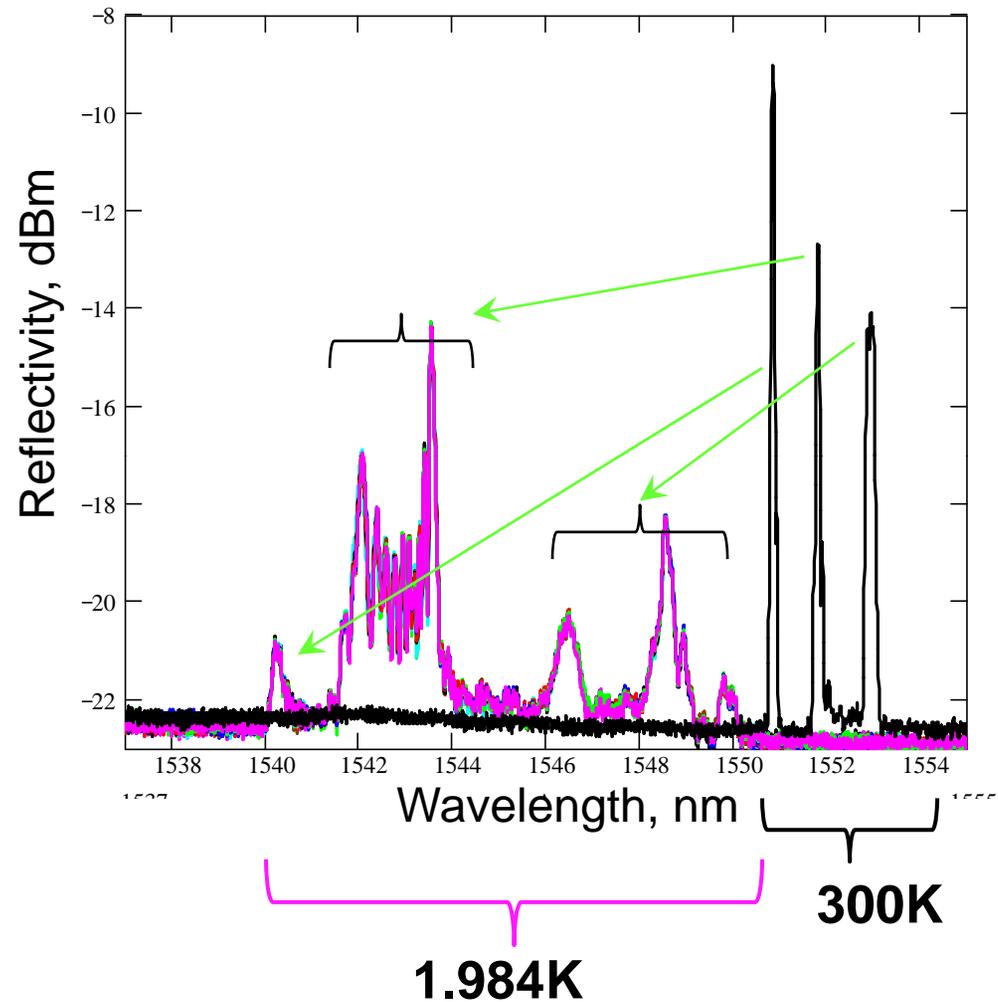
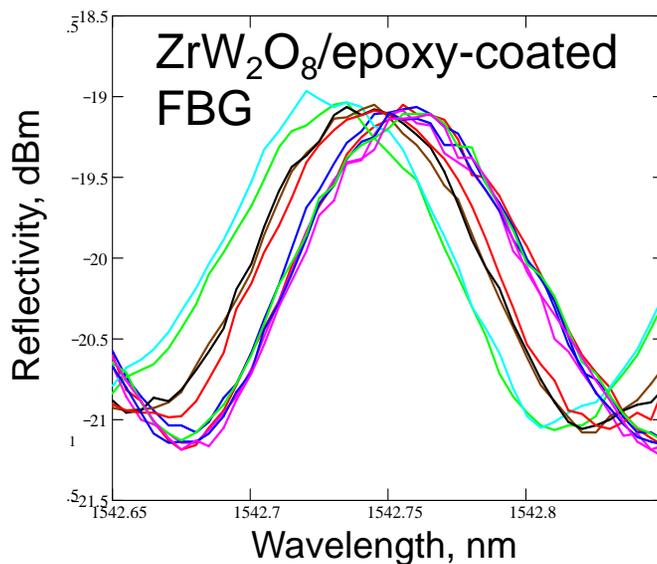
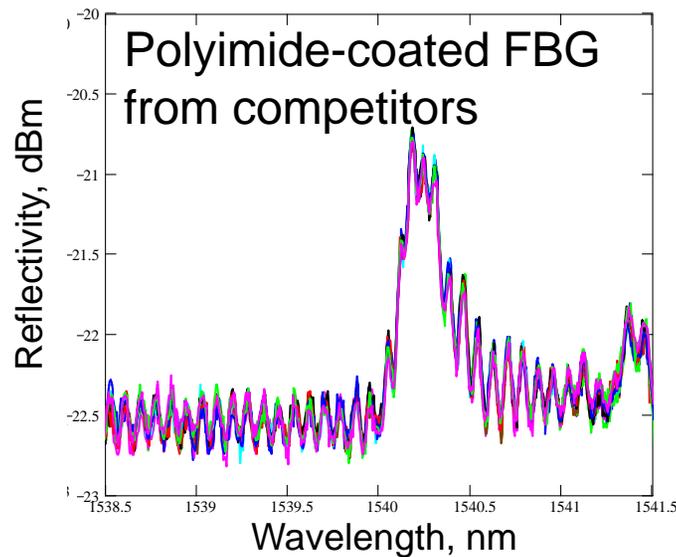


## Demonstration of sensitivity of our sensors at 2K at Jlab.

- Three FBG sensors were taped to the surface of the Nb plate together with reference carbon resistor.
- Heater on the other side of the plate was creating spatially localized heating (up to 1.8K, ~1.5cm in diameter heating area).



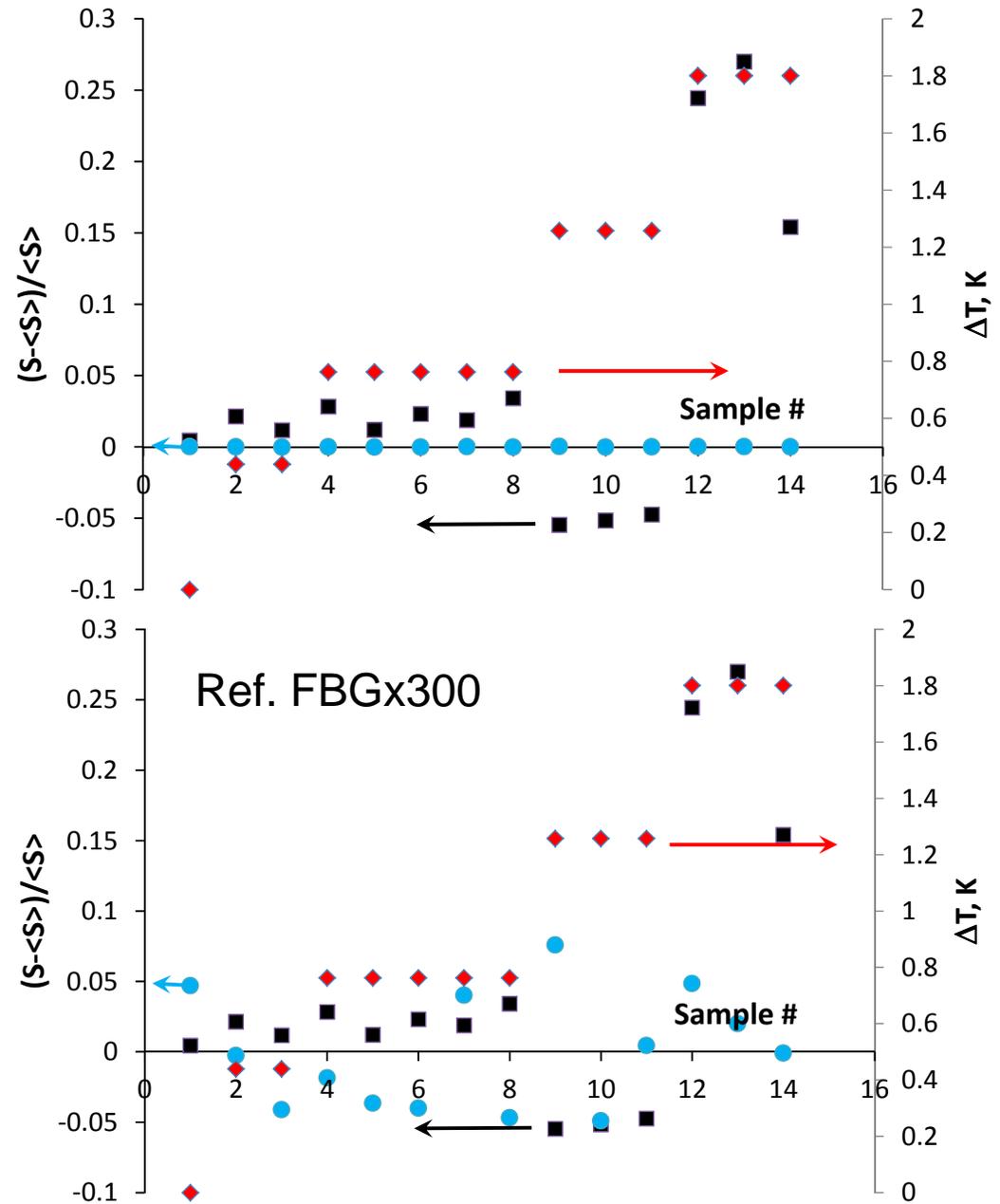
# Demonstration of sensitivity of our sensors at 2K at Jlab (continued).



- Branching of ZrW<sub>2</sub>O<sub>8</sub> /epoxy coated sensors is due to nonuniformity of coating along FBG length.

# Demonstration of sensitivity of our sensors at 2K at Jlab (continued).

- We surely see sizably larger (x300) noise from  $ZrW_2O_8$  /epoxy coated sensors, which is not associated with read-out instrumentation.
- Statistically we can resolve with confidence  $\Delta T = "1.8K"$  (it is not clear how close the difference was to 1.8K since the sensor was quite a bit off-center).
- The increased resolution compared to SoA is clearly demonstrated, but we are long ways to go from reliable sensor fabrication.



## Meeting initially stated milestones:

**Milestone 1** (End of Year 1): Demonstrate experimentally that the proposed nanocomposite coating enhances the resolution/reliability of single FBG sensors at cryogenic temperatures sufficiently to meet the program goals (10mK or better accuracy at 2K-4.3K range).

Mostly met, with 1 year delay

**Milestone 2** (End of Year 1): Demonstrate experimentally distributed fiber optic temperature sensing on "dummy" can with Nb flat plate.

Met, with 1 year delay

**Milestone 3** (End of Year 2): Experimentally demonstrate distributed fiber optic temperature measurements with 10mK or better accuracy at conditions similar to those used in SRF cavity testing.

Not met, but can be met by the end of 3<sup>rd</sup> year

**Milestone 4** (End of Year 2): Demonstrate the performance of distributed fiber optic cryogenic temperature sensor with a "dummy" can and heaters (10mK or better accuracy at 2K-4.3K range).

Not met, but can be met by the end of 3<sup>rd</sup> year

**Milestone 5** (End of Year 3): Demonstrate fiber optic SRF cavity characterization system that will have the following parameters: 1) sensor density at least as high as with any existing systems, 2) temperature accuracy of 10mK, 3) higher cavity surface coverage than what is available with fixed thermometry systems, and 4) temporal resolution of at least 1Hz.

We will be able to meet most technical specifications, but not all

# Suggested path forward

- We believe (based on consultations with JLab) that overall project objectives should be changed from demonstrating the full system at the end of Year 3 toward the demonstration of reliable and repeatable multiplexible fiber optic sensor with accuracy better than 10mK @ 2K, if project to be continued into Year 3.
  - We need to optimize  $ZrW_2O_8$  material (probably synthesizing it ourselves is a better path forward to make sure it is in  $\alpha$ -phase).
  - We need to still optimize polymer/  $ZrW_2O_8$  composite (functionalization of  $ZrW_2O_8$ ).
  - We need to optimize quite a bit fiber coating process.
  - We need significantly more testing at Jlab.
  - OFDR, recoating DSS fiber is clearly out of reach based on where we are at present on the project.

# Financial Standing

# Financial Standing

- We are ~63% spent during ~66% of the project.
- Since significant changes in technical approach occurred, division of spending per initially proposed tasks is very approximate.
- CRADA, related to the project, is ~10% spent on Jlab side.

	FY2009	FY2010	FY2011	Total
a) Funds allocated	\$62,000	\$201,500	109500	\$373,000
b) Actual costs to date	\$56,081	\$184,907	120842	\$361,830
c) Uncosted commitments				
d) Uncommitted funds (d=a-b-c)	\$5,919	\$16,593	-11342	\$11170

WBS or ID #	Item/Activity	Baseline Total Cost (AY\$)	Costed & Committed (AY\$)	Estimate To Complete (AY\$)	Estimated Total Cost (AY\$)
<u>Task 1</u>	Develop CuO/epoxy nanocomposite	17295	20,113		20,113
<u>Task 2</u>	Demonstrate elastic properties of nanocomposite at cryogenic temperatures	32540	27,245		27,245
<u>Task 3</u>	Develop nanocomposite fiber coating	26726	36,799		36,799
<u>Task 4</u>	Demonstrate performance of single-point fiber optic sensor at cryogenic temperatures	59332	59,332		59,332
<u>Task 5</u>	Demonstrate distributed fiber optic temperature sensing on "dummy" can with Nb flat plate	32074	30,008		30,008
<u>Task 6</u>	Develop the preliminary concept of all the sensor packaging/fixturing	35162	27,522		27,522
<u>Task 7</u>	Optimize CuO/epoxy nano-composite coating fabrication	33,000	32,908	92	33,000
<u>Task 8</u>	Develop interrogation instrumentation	49,000	47,066	1,934	49,000
<u>Task 9</u>	Fabricate distributed fiber optic cryogenic temperature sensor	95,000	80,836	14,164	95,000
<u>Task 10</u>	Demonstrate the performance of distributed fiber optic cryogenic temperature sensor with a "dummy" can and heaters	42,000		42,000	42,000
<u>Task 11</u>	Develop/demonstrate packaging/fixturing solution	63,591		56,671	56,671
<u>Task 12</u>	Develop calibration procedures/techniques	58,352		58,352	58,352
<u>Task 13</u>	Demonstrate temperature sensing solution meeting program performance goals	49,057		48,957	48,957
Totals:		584,000	361,830	222,170	584,000

# Additional Information

# Additional Information

- One MS in Physics (2011, Virginia Tech), one MS in Physics (in preparation, Virginia Tech), one PhD (in preparation, Virginia Tech).
- One engineer and one senior technician trained (MicroXact).
- 3 publications in various stages of preparation.