Search for trace element absorption sources in synthetic sapphire*

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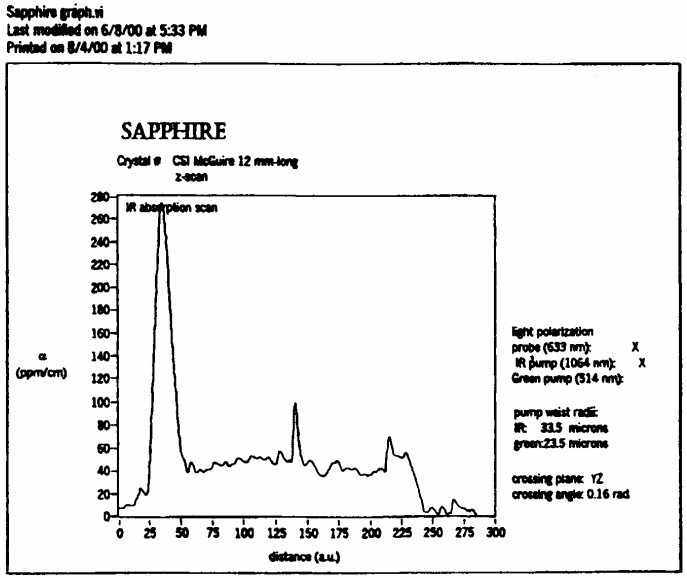
U. S. Department of Energy
Office of Basic Energy Sciences

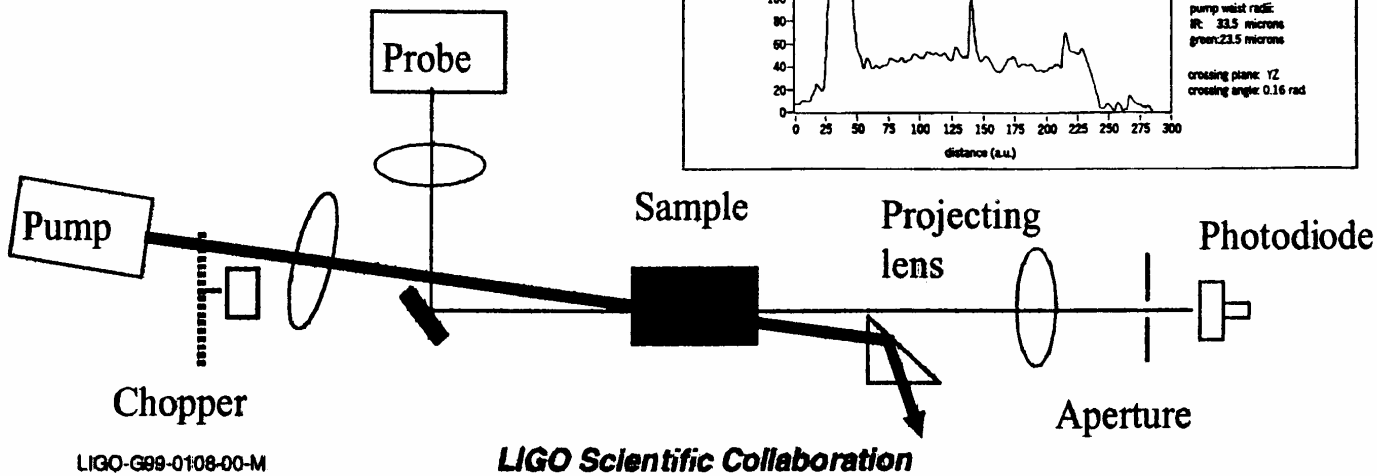
TALK OUTLINE

- **MOTIVATION AND OBJECTIVES**
- **BULK (*INAA*) TRACE ELEMENT MEASUREMENTS**
- **SYNCHROTRON RADIATION STUDIES**
  --- Surface measurements (*TXRF*)
- **SUMMARY**
- **FUTURE WORK**

Absorption at 1064 nm in Sapphire

Starting material
Recycled material
Distribution Function - Position
Oxidation State
Annealing
Lattice Quality





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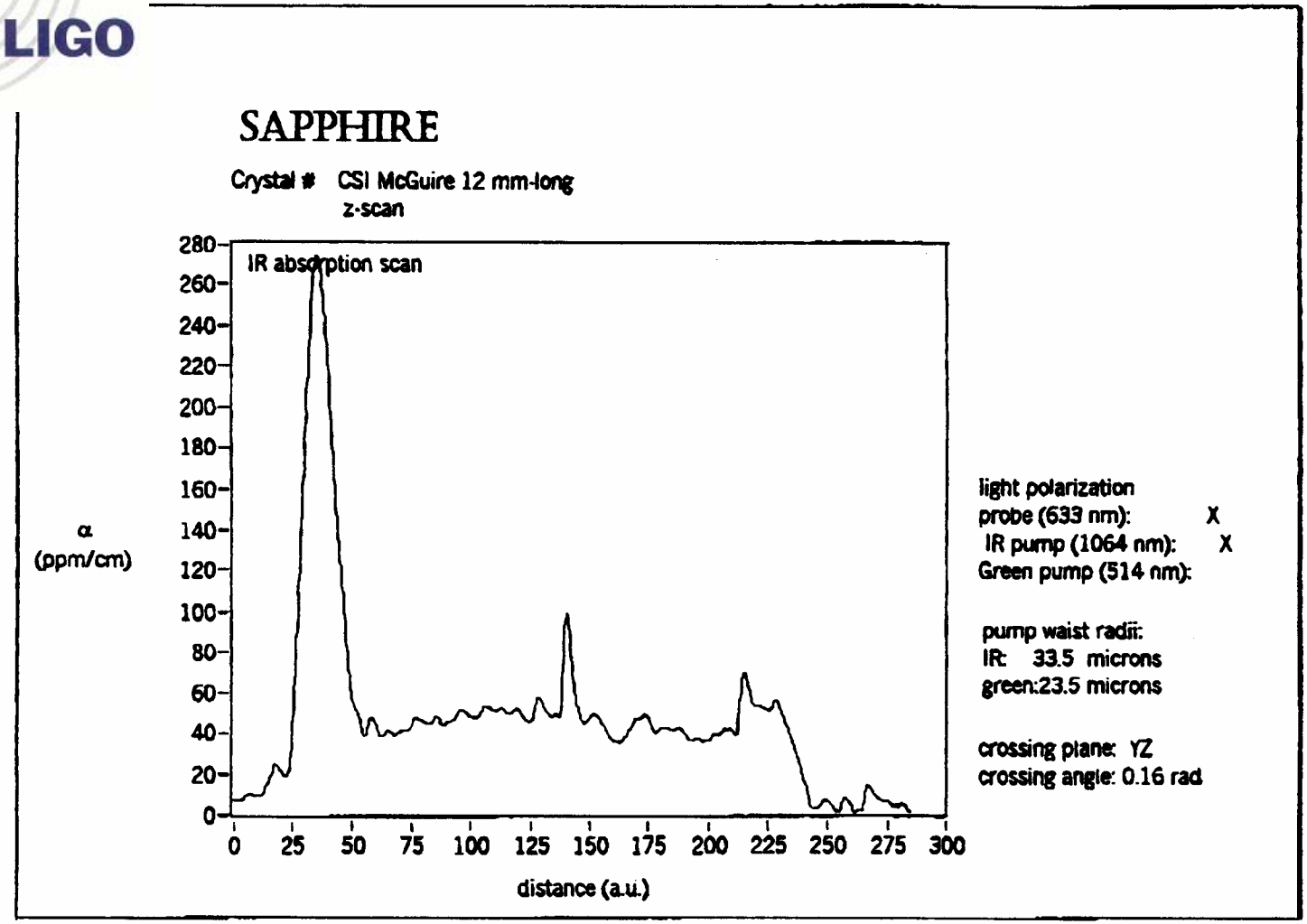
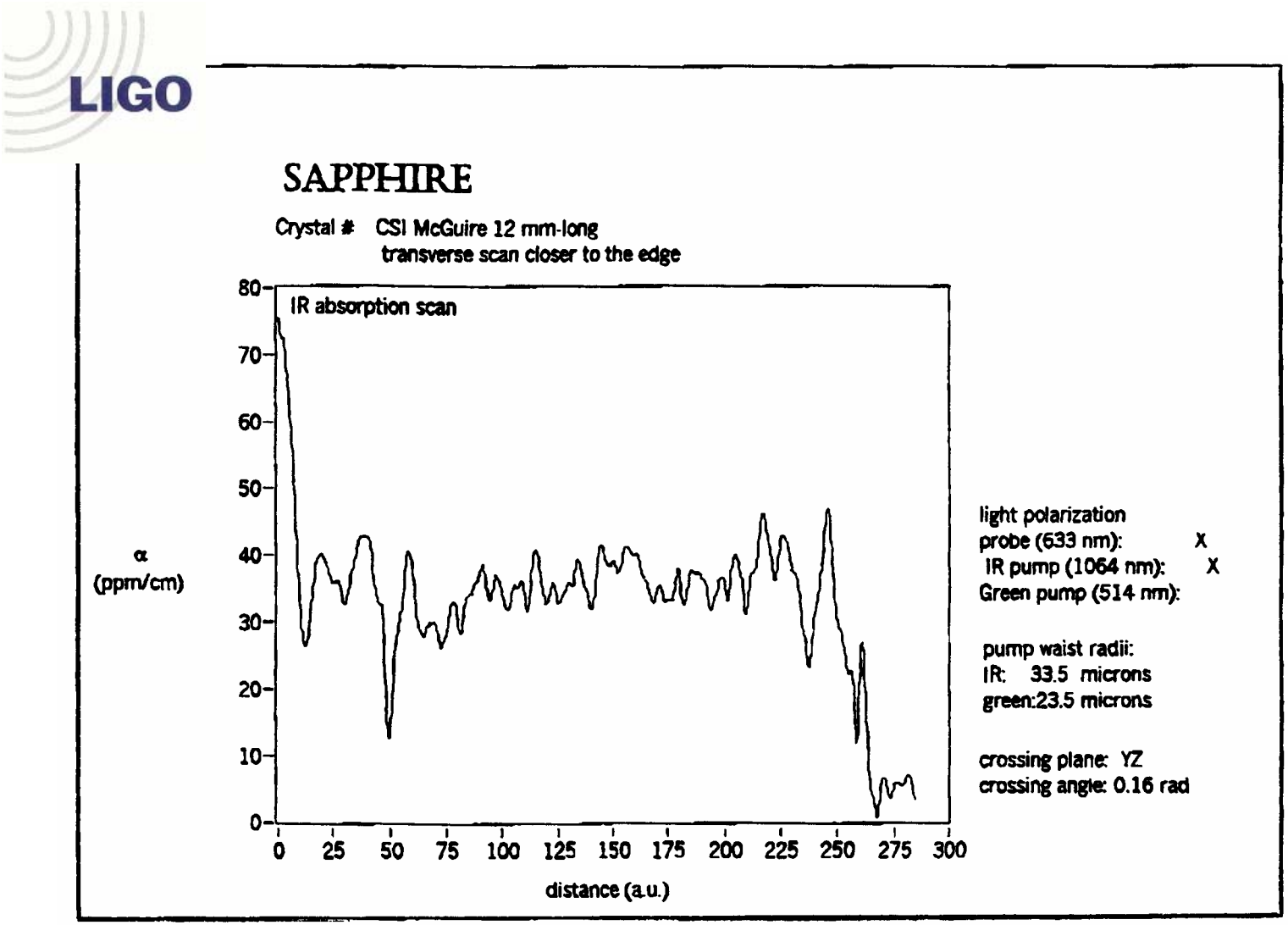
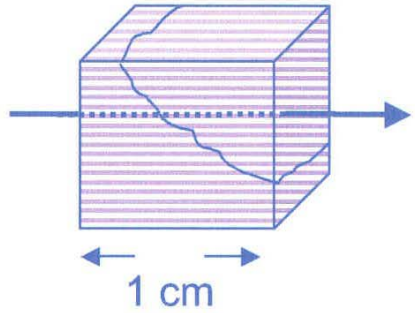
LIGO Scientific Collaboration

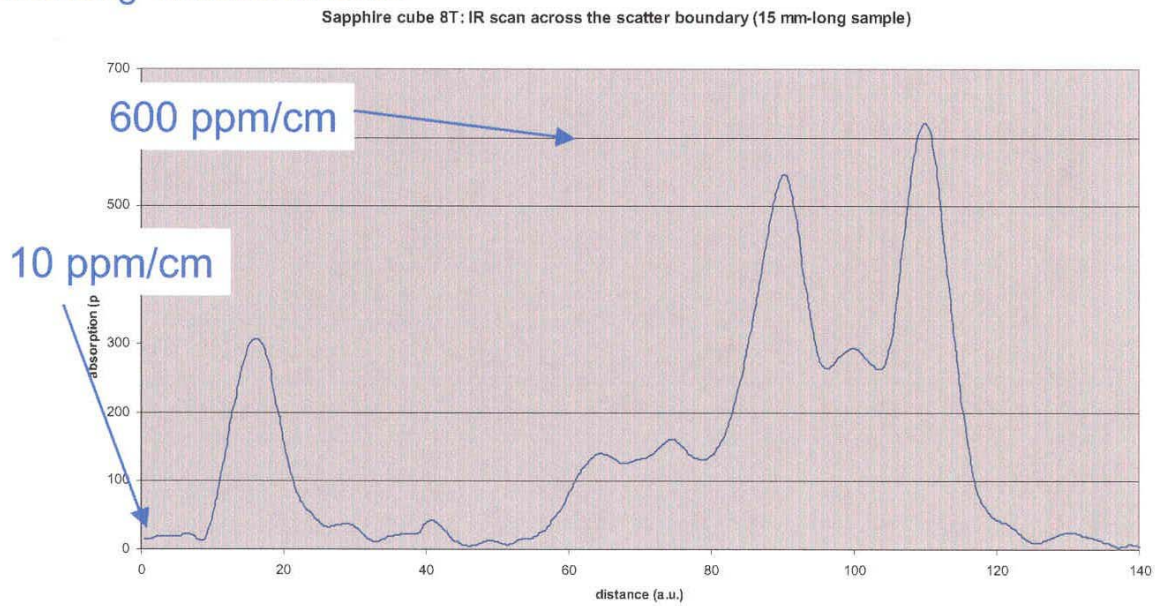

SAPPHIRE
Crystal # CSI McGuire 12 mm-long
z-scan
IR absorption scan
α
(ppm/cm)
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
0 25 50 75 100 125 150 175 200 225 250 275 300
distance (a.u.)
light polarization
probe (633 nm): X
IR pump (1064 nm): X
Green pump (514 nm):
pump waist radii:
IR: 33.5 microns
green:23.5 microns
crossing plane: YZ
crossing angle: 0.16 rad


SAPPHIRE
Crystal # CSI McGuire 12 mm-long
transverse scan closer to the edge
IR absorption scan
α
(ppm/cm)
80
70
60
50
40
30
20
10
0
0 25 50 75 100 125 150 175 200 225 250 275 300
distance (a.u.)
light polarization
probe (633 nm): X
IR pump (1064 nm): X
Green pump (514 nm):
pump waist radii:
IR: 33.5 microns
green:23.5 microns
crossing plane: YZ
crossing angle: 0.16 rad

Curious observation (Rosetta Sapphire)

- Single 1 cm sample
  - » region with 10 ppm/cm
  - » region with 600 ppm/cm
  - » abrupt boundary between
- Preparation unexceptional
- Tantalizing existence proof
- Mechanism not yet clear
  - » suggests "self-normalizing" measurements





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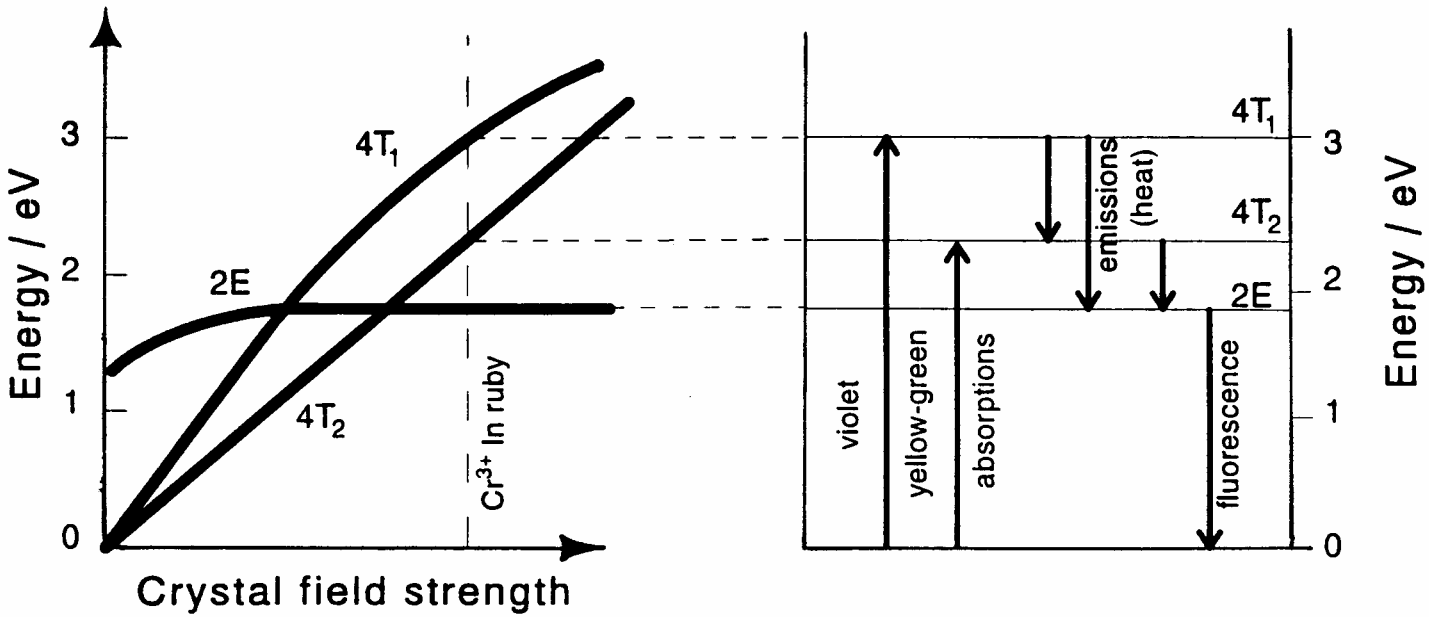


From K. Nassau, Scientific American 1980, 134.

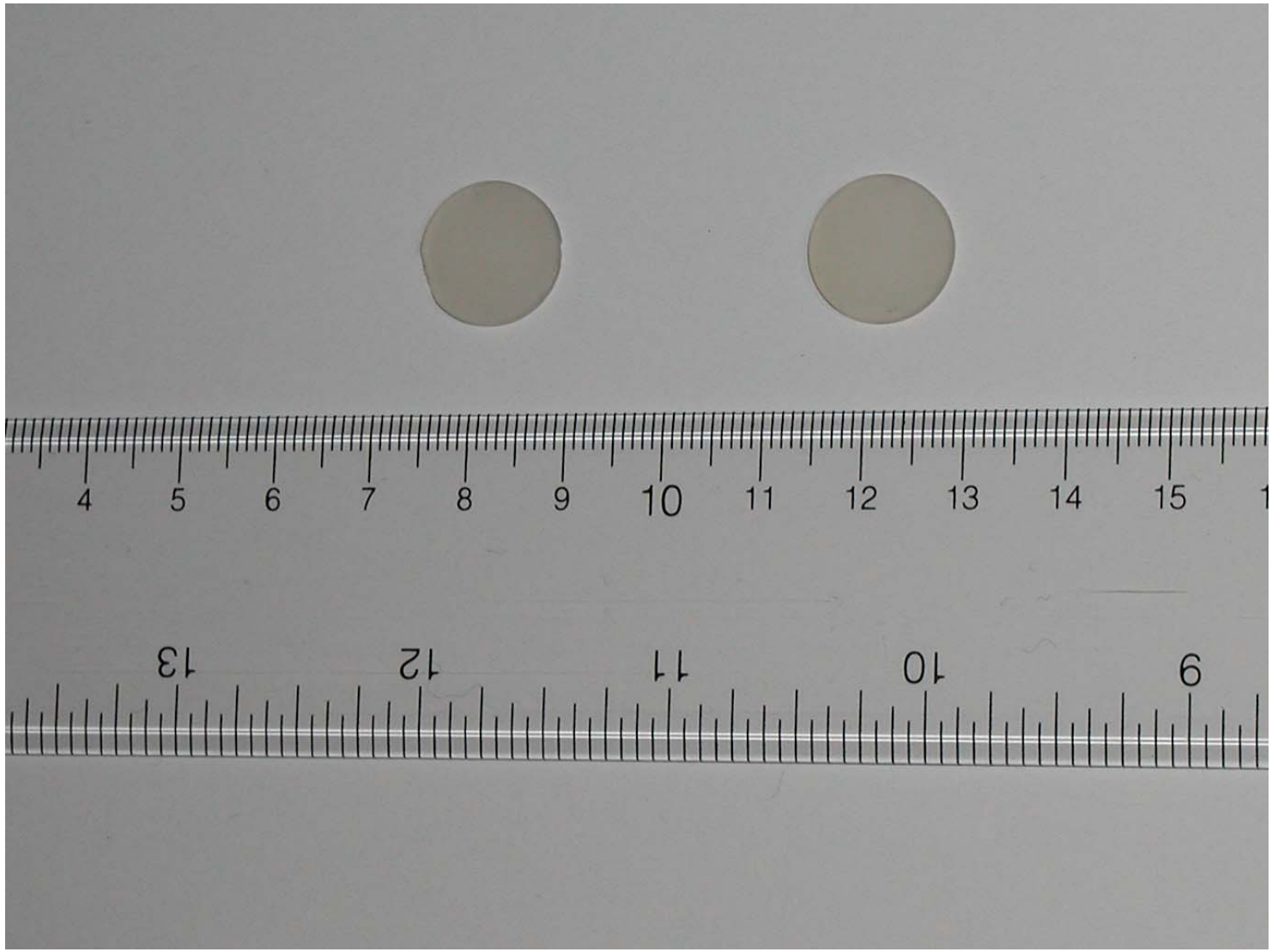
Trace Element Measurements

Objective:

Obtain physical correlations between chemical impurities (Ti, Cr, Fe, Co, etc.) and optical absorption characteristics of materials under consideration for use as test masses and optical coatings in advanced LIGO.

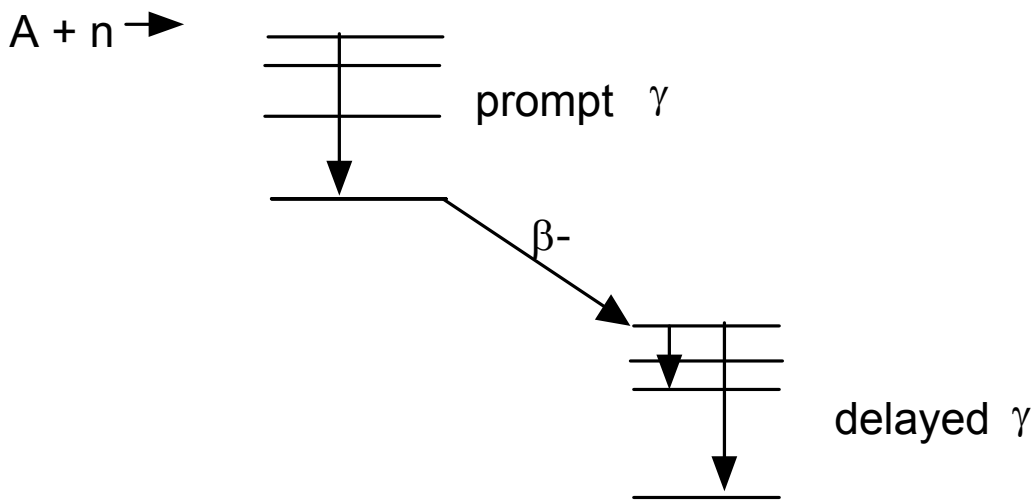
Trace Element Measurement Techniques

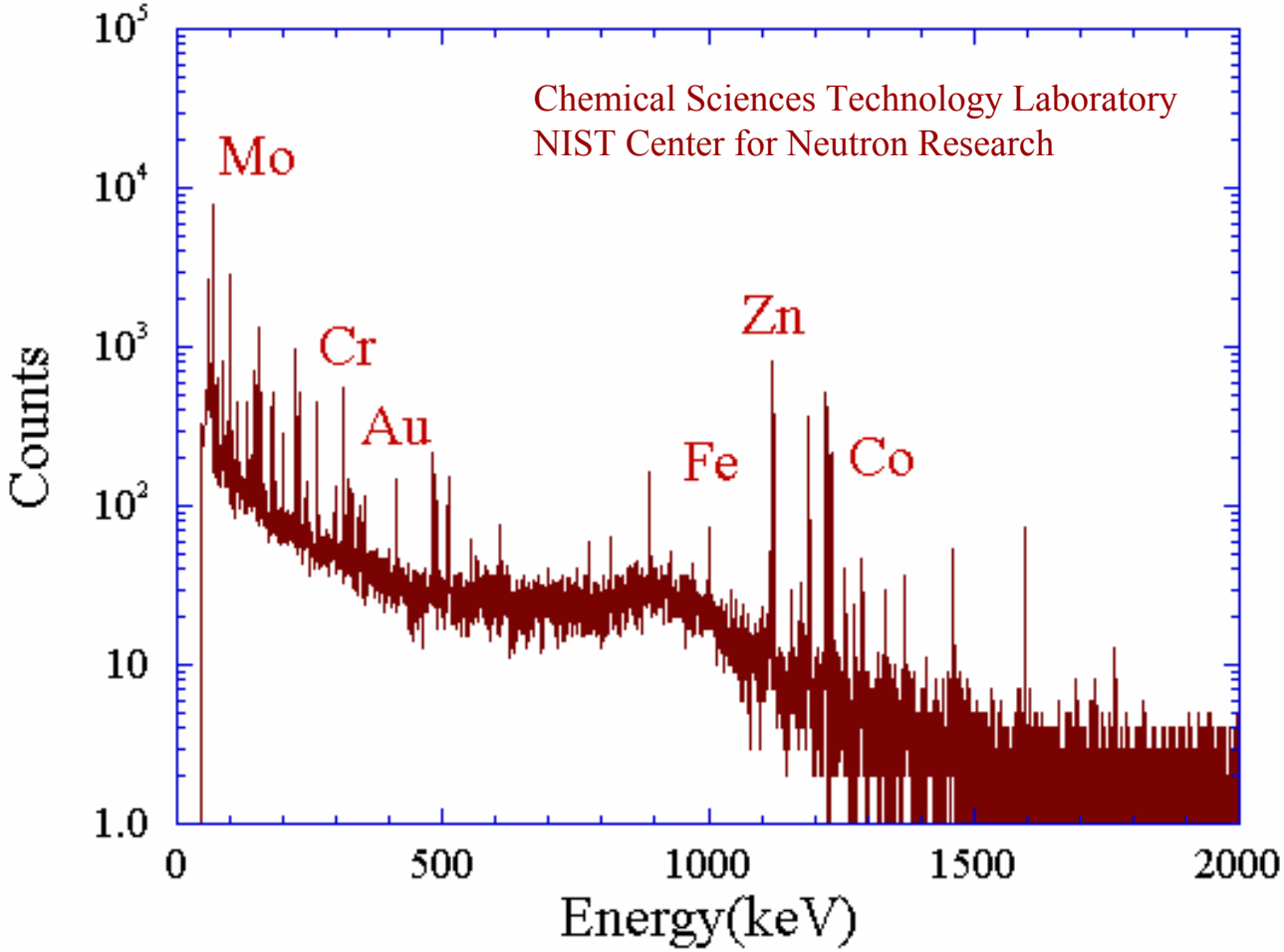
X-ray Fluorescence (XRF)	CAMD, SSRL, ALS
Extended X-ray absorption fine structure (EXAFS)	CAMD
X-ray absorption near edge spectroscopy (XANES)	CAMD, SSRL
Neutron Activation Analysis (INAA)	NIST
Prompt Gamma Neutron Activation Analysis (PGNAA)	NIST
Neutron Depth Profiling (NDP)	NIST
Electron Spin Resonance (ESR)	NIST


4 5 6 7 8 9 10 11 12 13 14 15
13 12 11 10 9

Neutron Activation Analysis (PGAA&INAA)

- Principle: when exposed to a neutron beam, nuclei absorb neutrons and form compound nuclei which de-excite by emission of prompt γ-rays. The often-produced radioactive product nuclei emit delayed γ-rays . The γ-ray energy is used to identify the isotope and the amount of radiation is directly proportional to the amount of element.





Typical gamma-ray spectrum showing the locations of peaks corresponding to the indicated elements.

LIGO

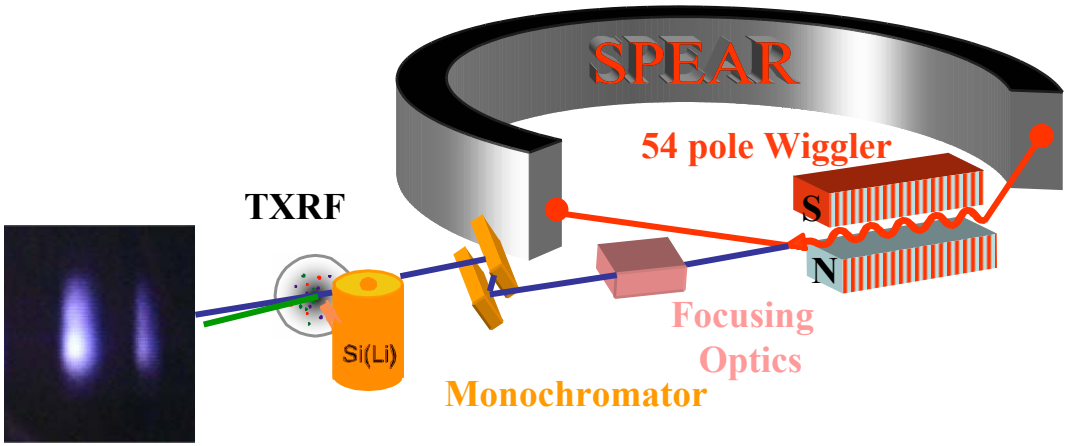
Bulk Analysis Results (INAA at *NIST*)

Table 1. Concentrations of elements observed in two samples of HEM™ grown sapphire*.

Element	Sample #1	Sample #4	Concentration
			Units
Sc	0.4 ± 0.1	0.4 ± 0.1	ppb
Cr	6 ± 2	6 ± 2	ppb
Fe	2.3 ± 0.2	2.7 ± 0.2	ppm
Co	0.5 ± 0.1	0.5 ± 0.1	ppb
Zn	<10	≤ 40	ppb
La	54 ± 10	63 ± 12	ppb
Ta	9 ± 2	11 ± 2	ppb
Ce	85 ± 9	87 ± 9	ppb
As	1.6 ± 0.2	2.8 ± 0.2	ppb
Se	< 3	< 3	ppb
Mo	0.6 ± 0.1	0.6 ± 0.1	ppm
Au	2.3 ± 0.6	3.5 ± 0.7	ppb

*S. C. McGuire, G. P. Lamaze and E. A. Mackey, *Trans. Am. Nucl. Soc.*, **84** (2002), 484.

Synchrotron Radiation TXRF Facility at SSRL



Collaborators:

SSRL:

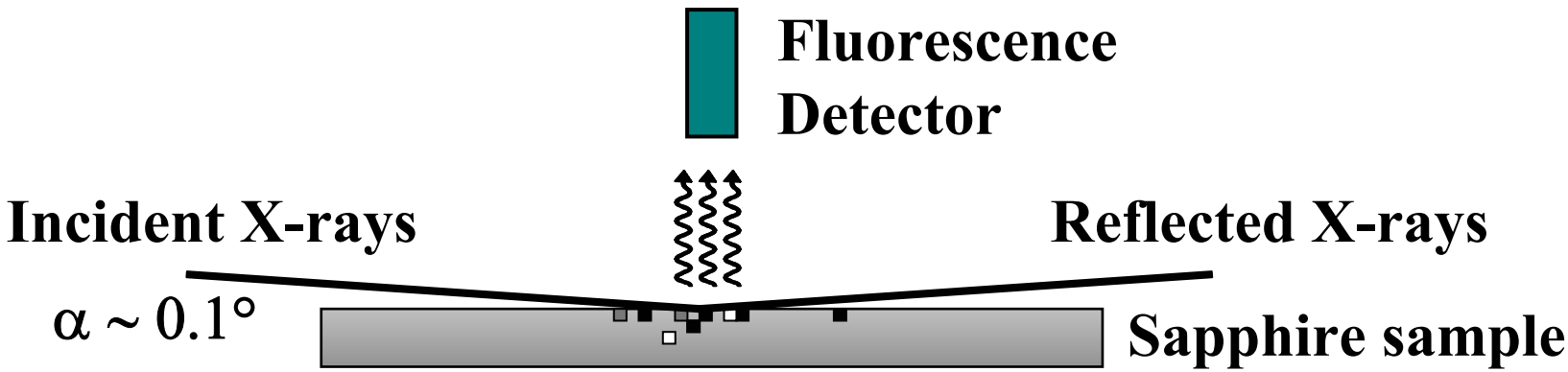
- P. Pianetta
- K. Leuning
- S. Brennan
- A. Singh

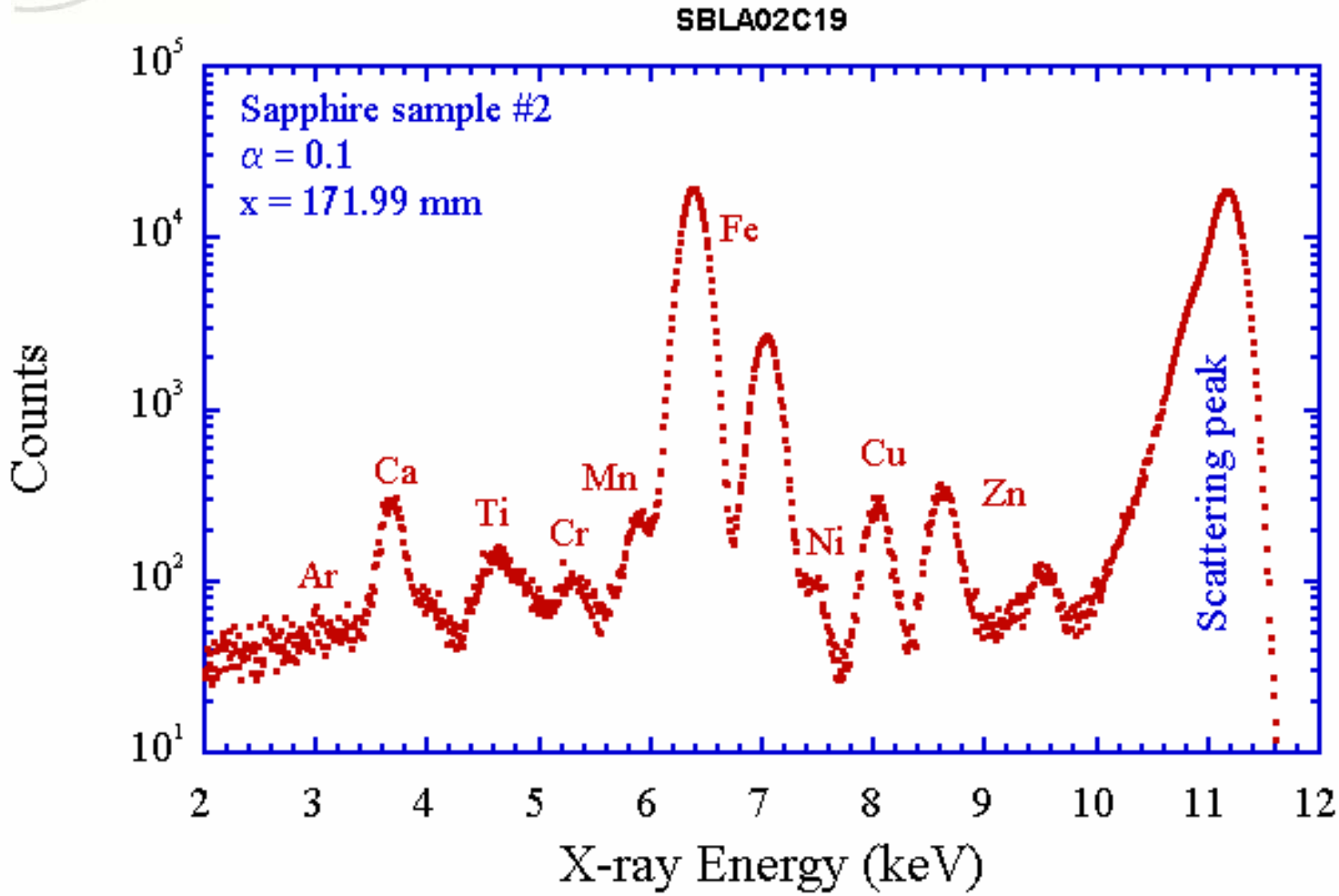
Southern Univ.

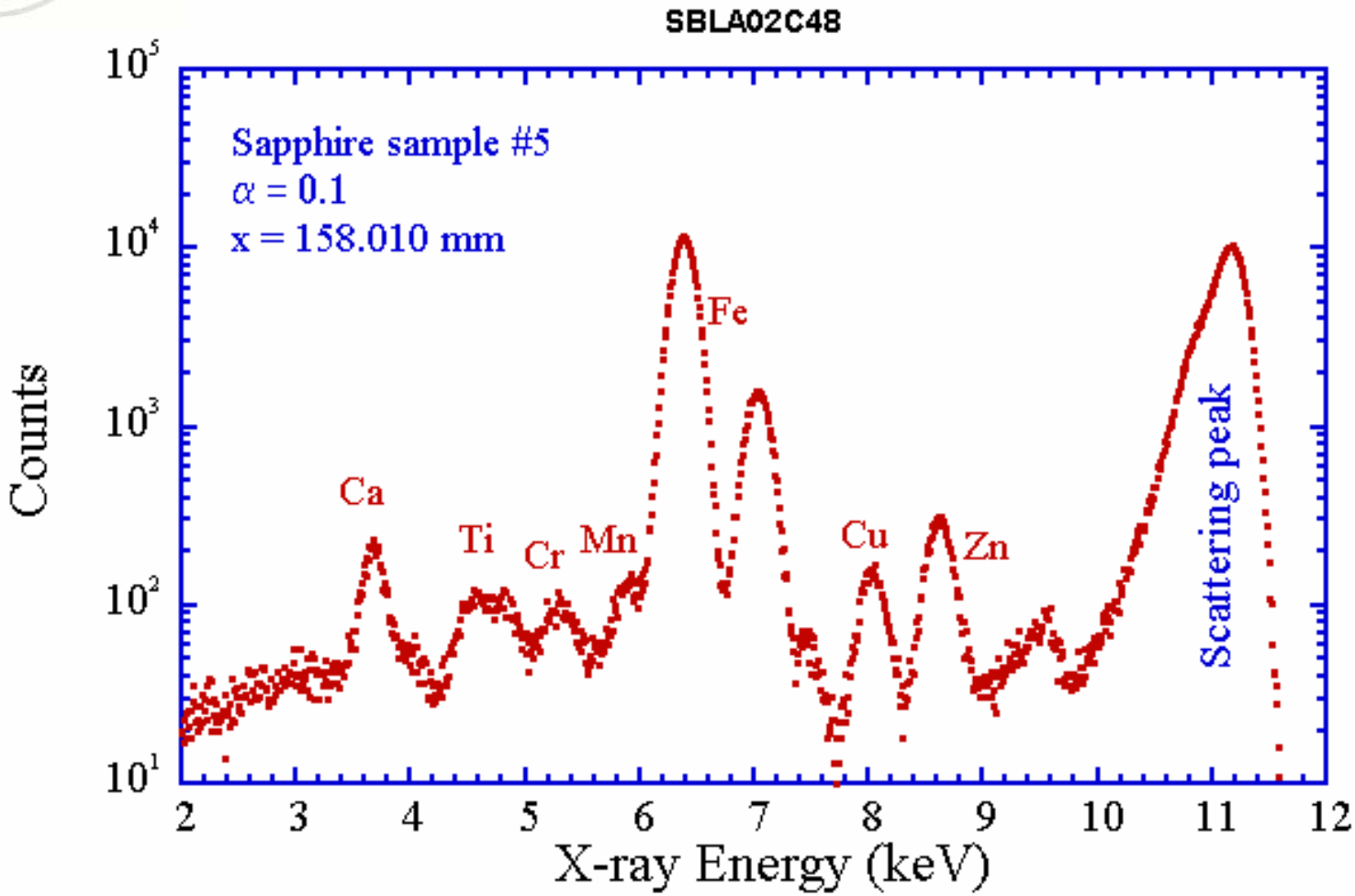
- S. C. McGuire
- M. Baham
- E. Preddie

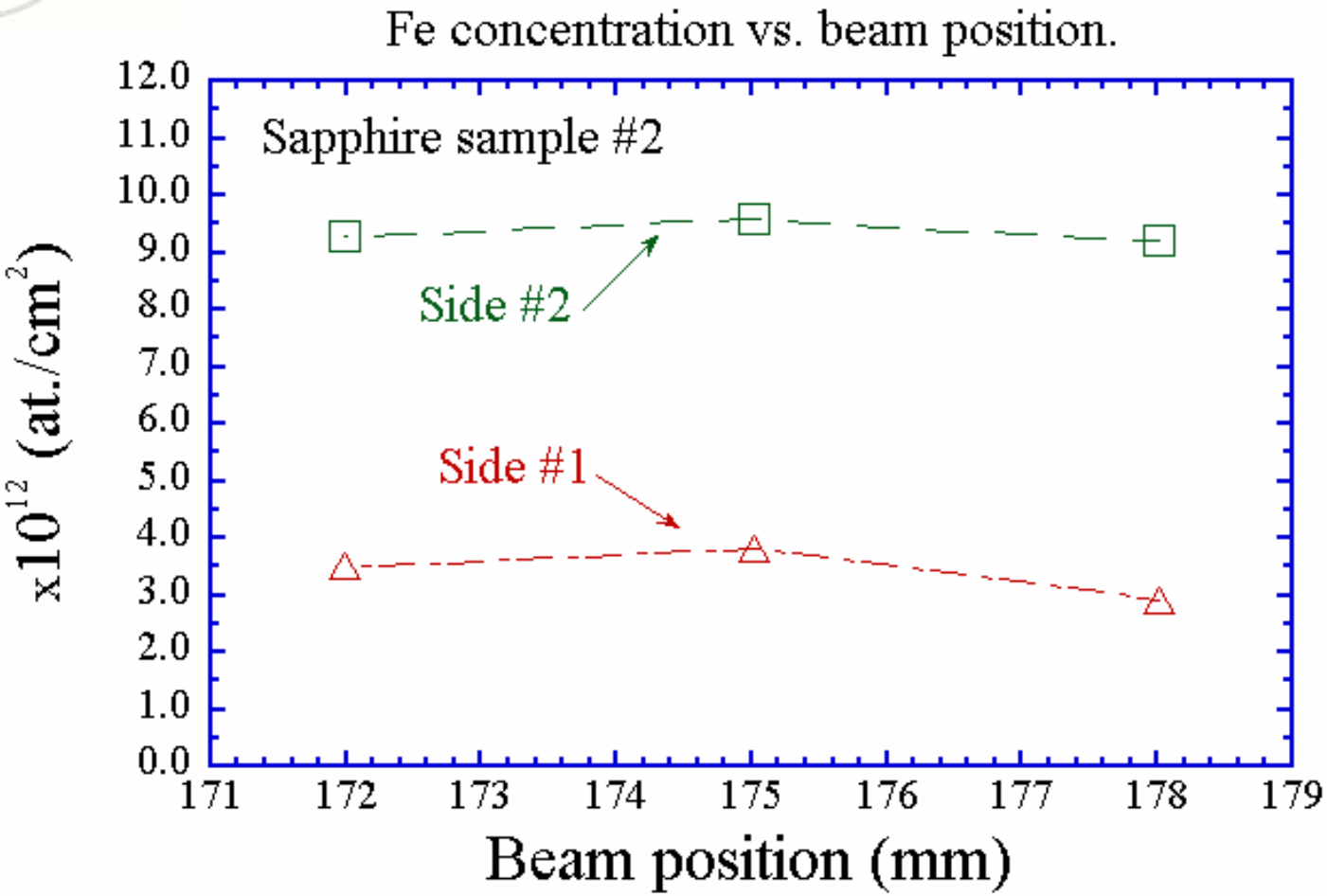
X-ray energy: 11.3 keV
Angle of incidence ~ 0.08°
Detector: Si(Li)—no parasitic peaks
Automatic critical angle measurement
Wafers: Small pieces to 200 mm
Cleanroom mini-environment

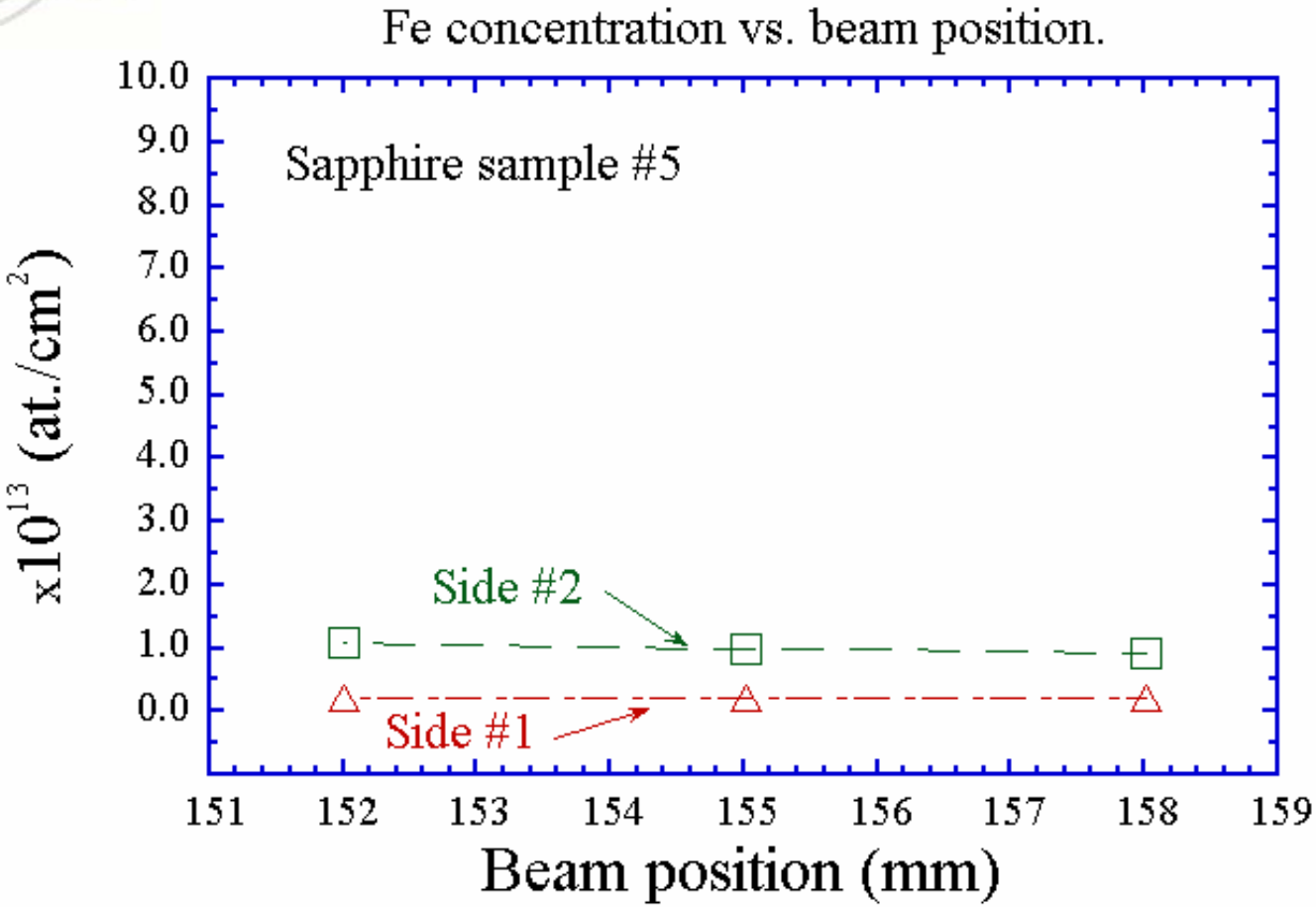
Experimental setup in the total reflection X-ray fluorescence (TXRF) experiments.

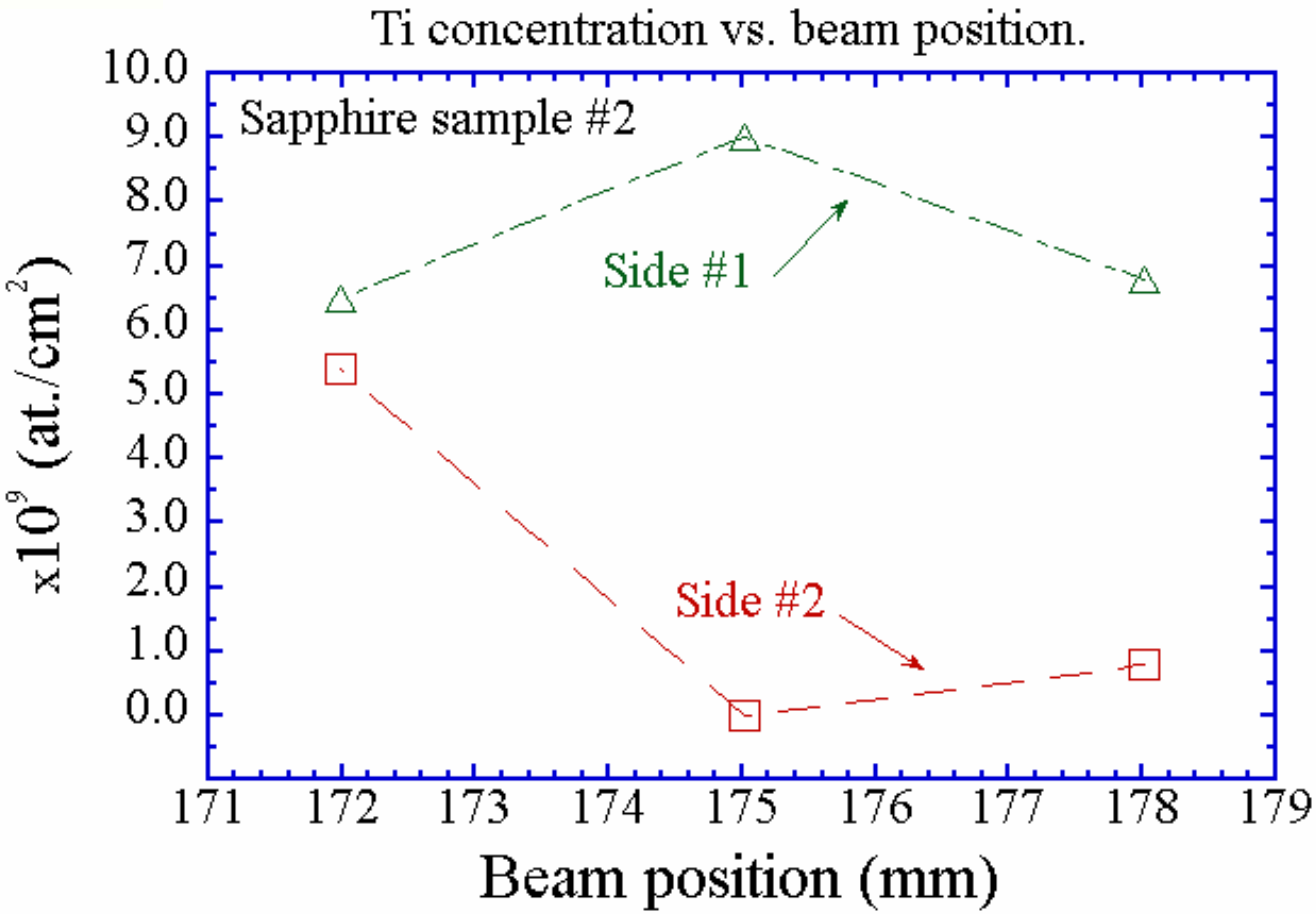


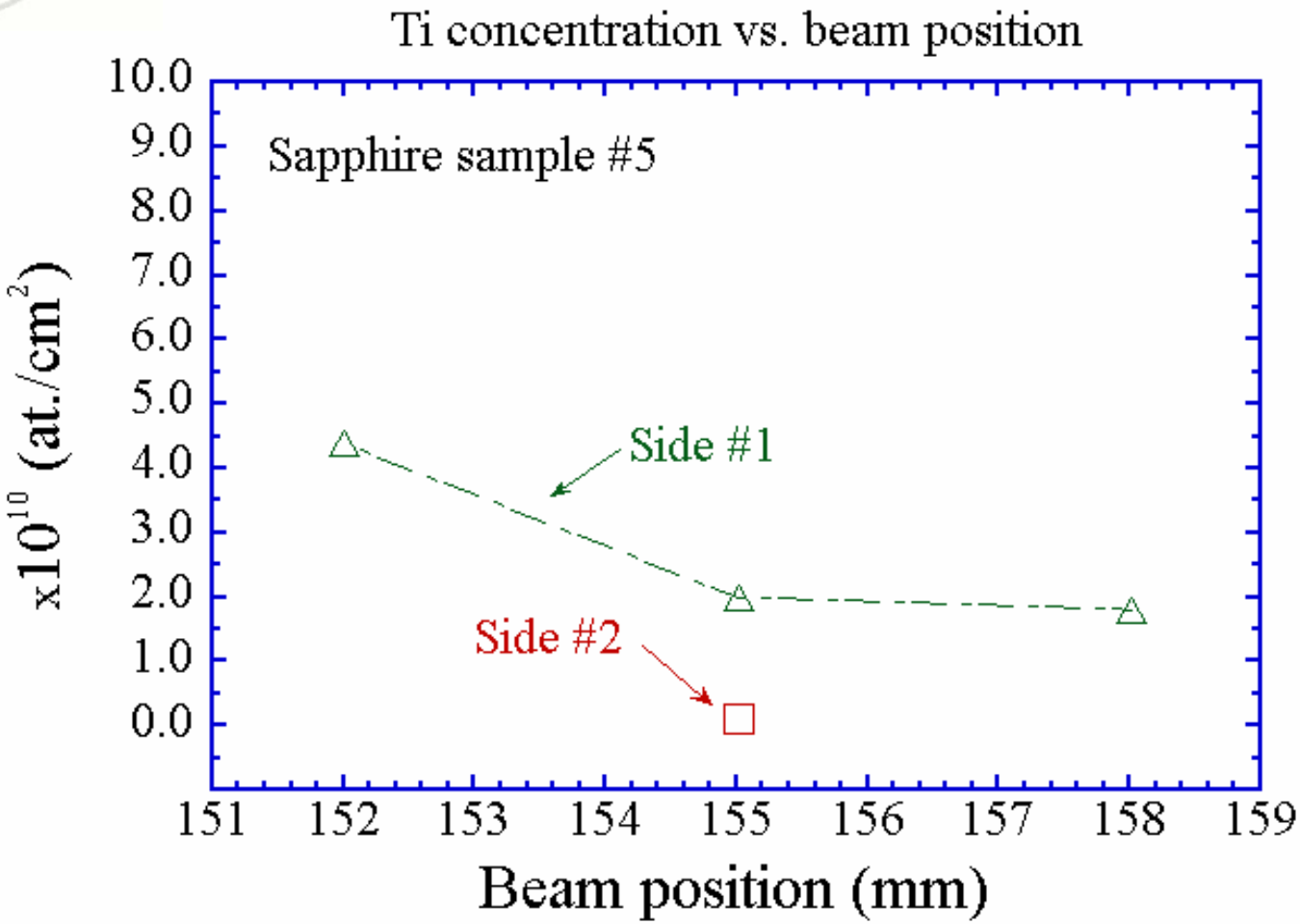

SBLA02C19
Sapphire sample #2
α = 0.1
x = 171.99 mm
Counts
X-ray Energy (keV)
Ar
Ca
Ti
Cr
Mn
Fe
Ni
Cu
Zn
Scattering peak


SBLA02C48
Sapphire sample #5
α = 0.1
x = 158.010 mm
Counts
X-ray Energy (keV)
Ca
Ti
Cr
Mn
Fe
Cu
Zn
Scattering peak


Fe concentration vs. beam position.
Sapphire sample #2
Side #2
Side #1
x10^12 (at./cm^2)
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
171
172
173
174
175
176
177
178
179
Beam position (mm)


Fe concentration vs. beam position.
Sapphire sample #5
Side #2
Side #1
x10^13 (at./cm^2)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
151
152
153
154
155
156
157
158
159
Beam position (mm)


Ti concentration vs. beam position.
Sapphire sample #2
Side #1
Side #2
x10^9 (at./cm^2)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
171
172
173
174
175
176
177
178
179
Beam position (mm)


Ti concentration vs. beam position
Sapphire sample #5
Side #1
Side #2
x10^10 (at./cm^2)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
151
152
153
154
155
156
157
158
159
Beam position (mm)

SSRL TXRF RESULTS

TABLE 2. Surface Trace Element Concentrations in HEM™-grown sapphire.

ELEMENT	Sample#2 (atoms/cm^2)	Sample#5 (atoms/cm^2)
S	10^{11}	10^{11}
Cl	10^{11}	10^{11}
Ca	10^{11}	10^{11}
Ti	10^{10}	10^{10}
Cr	10^{9}	10^{9}
Fe	10^{12}	10^{12}
Co	10^{11}	10^{11}
Cu	10^{10}	10^{10}
Zn	10^{10}	10^{10}

Recent INAA Results from NIST

Table 1. Mass fraction estimates based on comparison with SRM 2709 San Joaquim Soil.**

Element	LL1	HL1	SRM 1575a	Certified Value
Sc	0.06 ± 0.02 ppb	0.20 ± 0.04 ppb	10.8 ± 0.8 ppb	10.1 ± 0.3 ppb
Cr	9 ± 2 ppb	8 ± 1 ppb	0.36 ±0.03 ppm	0.3 - 0.5 ppm range
Fe*	≤ 1 ppm	≤ 1 ppm	45 ± 2 ppm	46 ± 2 ppm
Co	≤ 1 ppb	1.2 ± 0.4 ppb	68 ± 3 ppb	61 ± 2 ppb
Zn	30 ± 3 ppb	40 ± 4 ppb	39 ± 2 ppm	38 ± 2 ppm
Sb	≤ 2 ppb	≤ 2 ppb	10 ± 3 ppb	not certified
La	7 ± 0.4 ppb	4 ± 0.4 ppb	53 ± 7 ppb	not certified

*Longer gamma ray counts are in progress to determine whether Fe is present in these samples.

**Initial trace element determinations for one pair of high loss (HL) and low loss (LL) sapphire samples. A total of four pairs are being examined.

Summary

- Measurements to date show typical broad range of elements at sub-ppm levels.
- Excellent sensitivity for the elements of primary interest.
- Initial measurements on high-loss and low-loss sapphire show comparable trace element content.
- Protocols and procedures established for sample preparation and sequencing of measurements.
- MOA between Southern University and SSRL has been executed.

Future Work

- INAA sapphire standards development is ongoing at NIST.
- Completion of INAA trace element measurements on high-loss and low-loss sapphire specimens.
- Initiation of low-level counting facility at Southern University.
- SSRL Single Experiment/Program proposal to be submitted 11-1-03.
  SUBR (McGuire, Henry, Preddie)
  TXRF/SSRL(Brennan, Leuning, Pianetta, Singh)
  Ginzton Laboratory (Fejer and Route)
- TXRF Facility upgrade, Jan. 2004, to include ~10 μm spatial resolution.