LIGO-G000414-00-I

Sapphire Development at SIOM for LIGO

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LIGO Seminar, Caltech
November 10, 2000

- A Brief History of Sapphire Development at SIOM.
- Result of Phase I Program.
- Discussion on Future Development.

A Brief History of Sapphire Effort at SIOM

- January 8, 1997: B. Barish suggested to investigate possibilities of Sapphire development in China or Russia.
- March 27-28, 1997: R.Y. Zhu visited SIOM and found TGT technology could produce Sapphire sample along C-Axis.
- April 3 and June 25, 1997 : R.Y. Zhu met C.L. Bai (VP of CAS) and other CAS leaders, suggested CAS support SIOM effort on Sapphire development for LIGO.
- June 30, 1997: W. Kells and R.Y. Zhu visited SIOM. MoU was signed by Q.Q. Wu, P.Z. Deng and J. Xu for SIOM and B. Barish, W. Kells and R.Y. Zhu for LIGO: LIGO provided 50k USD at front. SIOM was required to deliver 3 $\phi 11 \times 8$ cm samples in Phase I program by the end of 1998.
- SIOM carried out **36** test runs. LIGO did GDMS analysis and provided 20 kg each of raw materials from Biesterfield (Czech) and S.E.I. (Switzerland). R.Y. Zhu paid SIOM 12 visits (3/27, 6/30 and 9/29/97, 2/18, 5/6, 8/27 and 11/27/98, 5/25, 7/6 and 9/22/99, 1/25 and 6/27/00).
- Jun 1999: CAS identified Sapphire project and provided additional 2M yuan (0.5M yuan since July 1997). SIOM procured an annealing furnace and a used Swiss cutting machine.
- Jan 2000: LIGO extended Phase I program to the end of 2000. SIOM delivered three phase I samples in May (#7), June (#28) and October (#29).

TGT Sapphire Growth at SIOM
Directional Temperature Gradient Technique

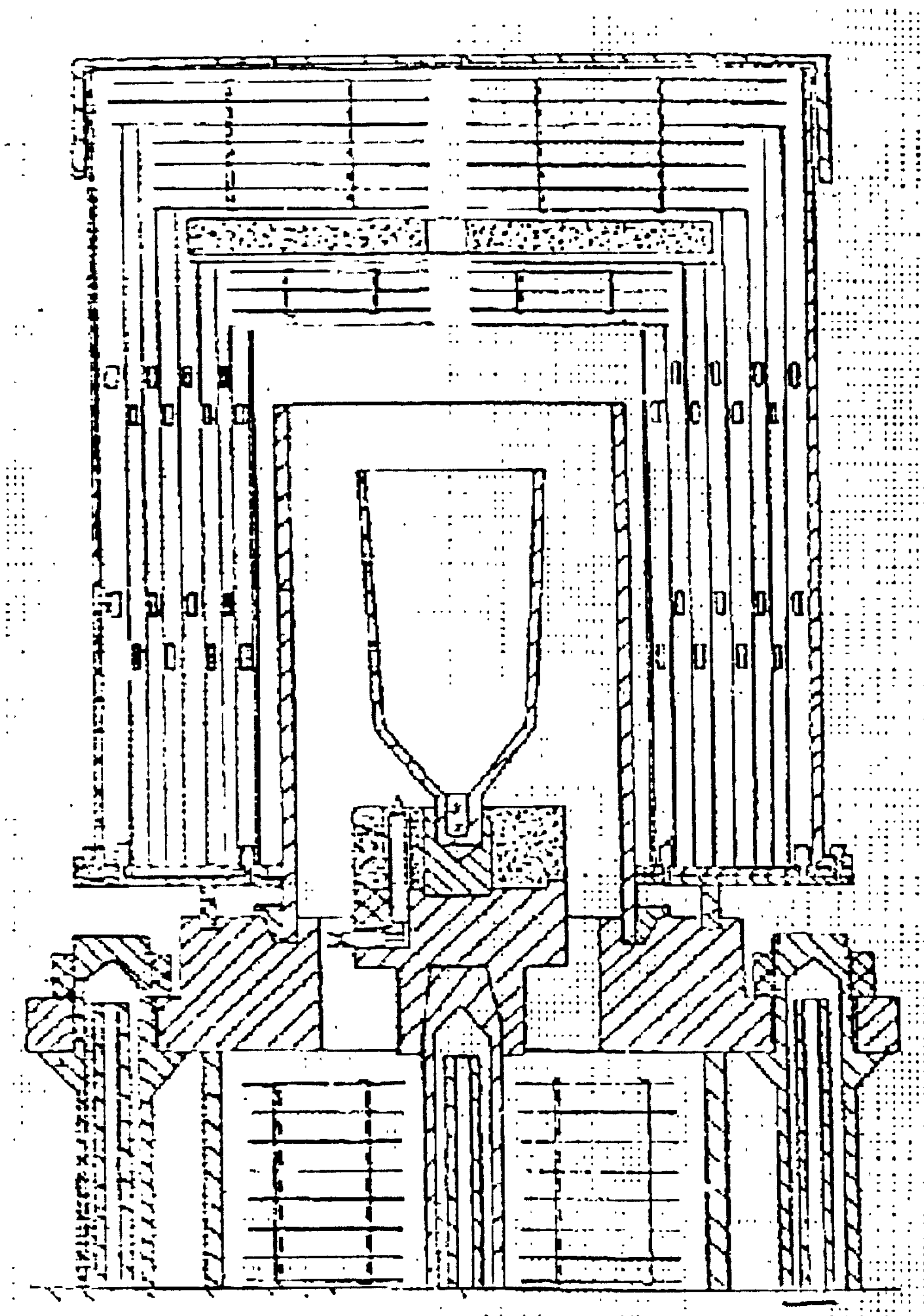
- SIOM started Sapphire growth by using the Verneil method in sixties, and switched to the Czochralski method later.

- Prof. Yongzong ZHOU *et al.* invented the **Directional Temperature Gradient Technique (TGT)** for Sapphire growth in 1978, and SIOM obtained a patent on TGT in 1985.

- TGT uses molybdenum crucible, molybdenum shielding and graphite heater. The difference between TGT and HEM of Crystal Systems Corp. is that TGT does not use helium, or any, gas as heat exchange medium, and provides temperature gradient by the resistance of the heater and water cooling for graphite electrodes.

- Starting 1989 a research group lead by Prof. Peizhen DENG has been concentrating on large size Nd:YAG and Ti:Sapphire crystal growth by using TGT. New furnaces were constructed in nineties, and up to $\phi 120 \times 80$ mm Ti:Sapphire crystals with orientation of (0001), (1120) and (1010) were successfully grown.

Schematic of TGT Furnace



Sapphire MoU between LIGO and SIOM

June 30, 1997, SIOM

Sapphire MoU between LIGO and SIOM

June 30, 1997, SIOM

Sapphire Growth Furnace at SIOM

July 1, 1997, SIOM

MoU between SIOM and LIGO

- Purpose: to establish/define a collaborative relationship between SIOM, CAS, and LIGO project.

- Final Goal: to produce Sapphire crystal blanks with $\phi 25 \times 10$ cm with c-axis along the cylinder axis with an alignment of $< 100\mu$rad, and absorption of < 5 ppm/cm and homogeneity of $< 5 \times 10^{-7}$ at $\lambda = 1.06\mu$m within the central 8 cm.

- Phase I Deliverables:

  1. SIOM will deliver three Sapphire blank of $\phi 11 \times 8$ cm with c-axis along the cylinder axis, and low absorption ($<$ 10 ppm/cm) and good homogeneity ($< 5 \times 10^{-6}$) at $\lambda = 1.06\mu$m within the central 5 cm between Dec 97 to Jun 98.

  2. SIOM will provide a feasibility study of Sapphire test mass required by LIGO.

  3. LIGO will provide technical evaluation of Sapphire samples.

- Cost: LIGO will provide US$50,000 for Phase I R&D to SIOM.

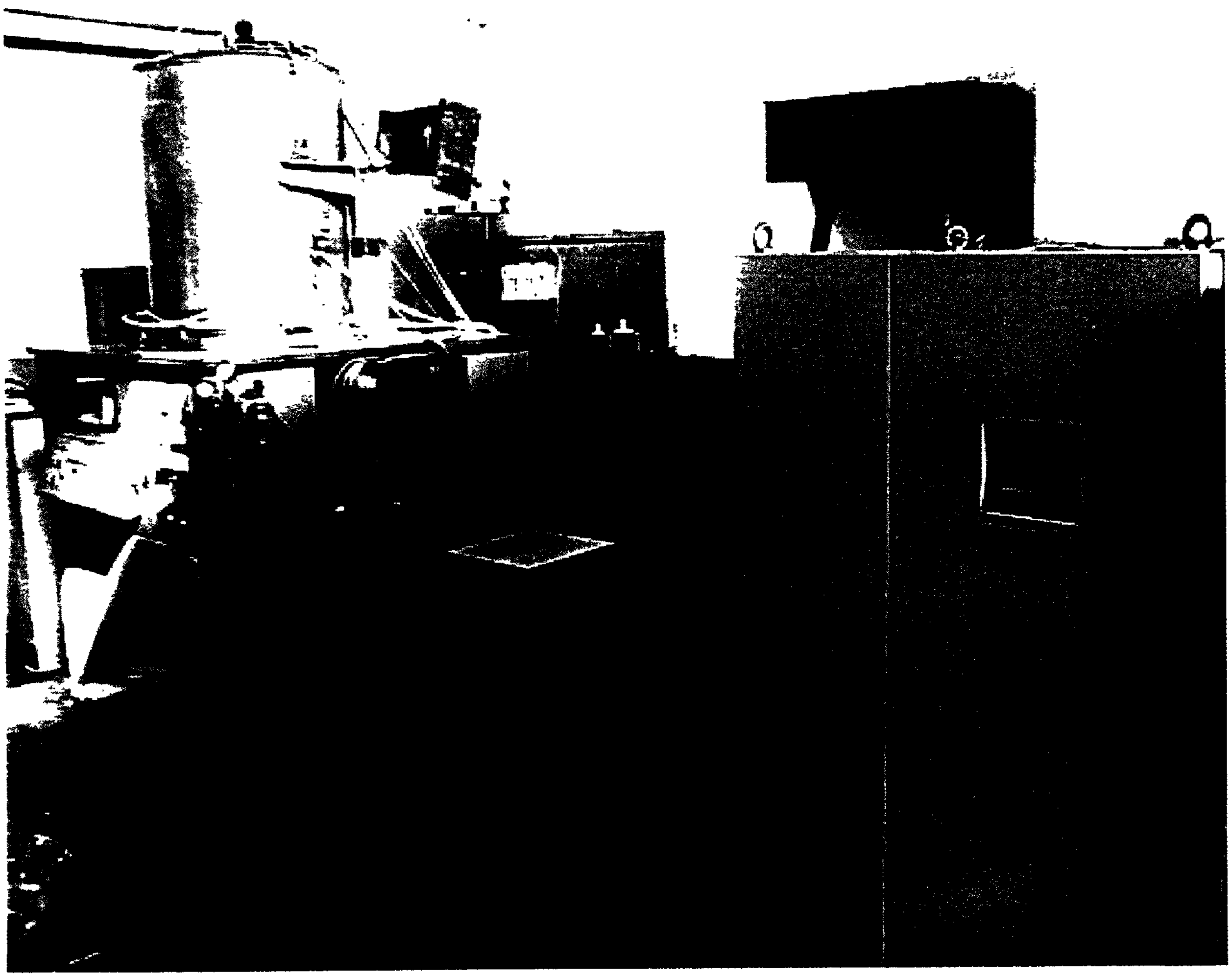
- Schedule:

  1. Phase I Review and Phase II Plan: June 1998.

  2. Phase I Final Report and Phase II MoU: December 1998.

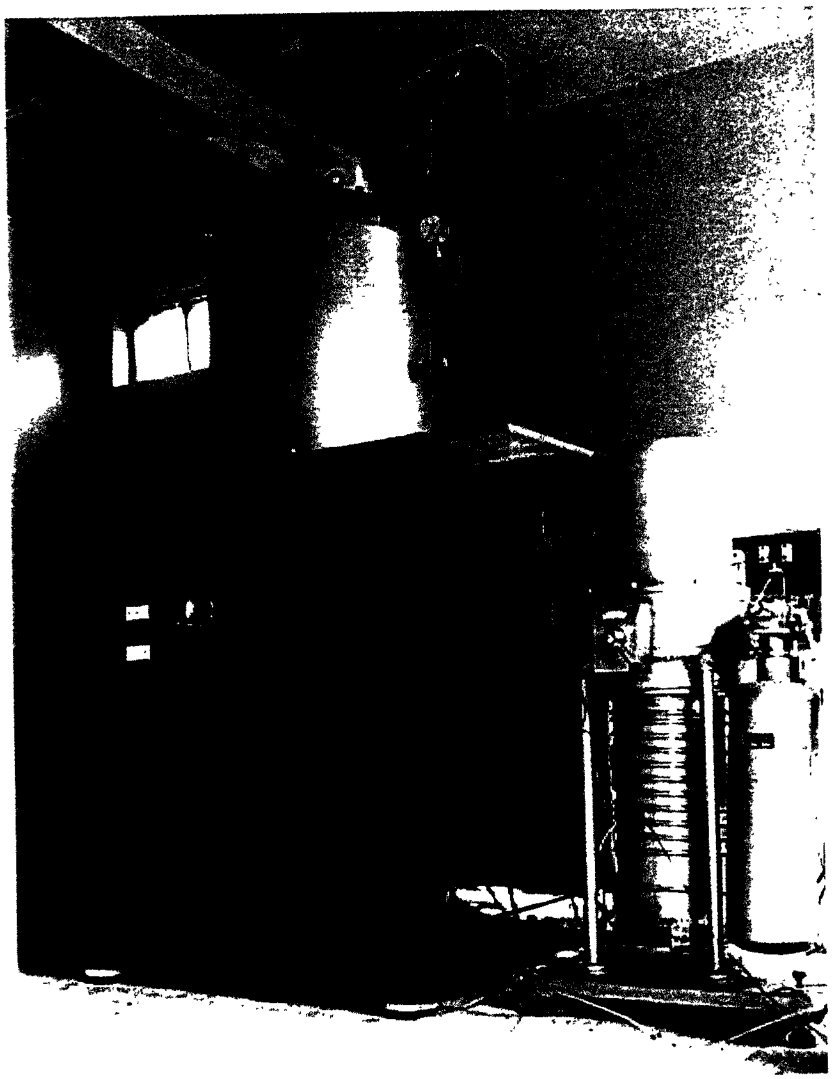
A Sapphire Growth Furnace at SIOM

Jan 25, 2000, SIOM



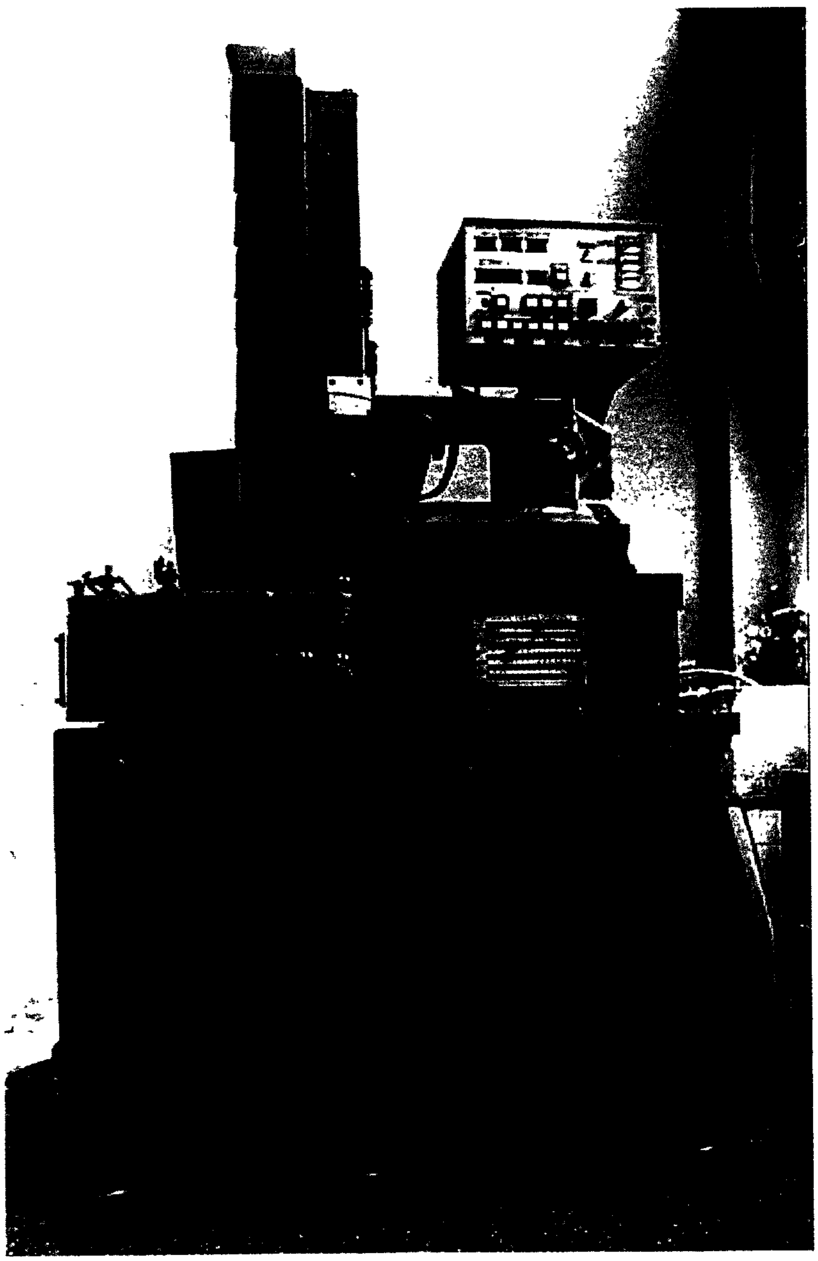
A Sapphire Annealing Furnace at SIOM

Jan 25, 2000, SIOM



A Used Swiss Cutting Machine at SIOM

Jan 25, 2000, SIOM



As Grown Sapphire Sample # 28

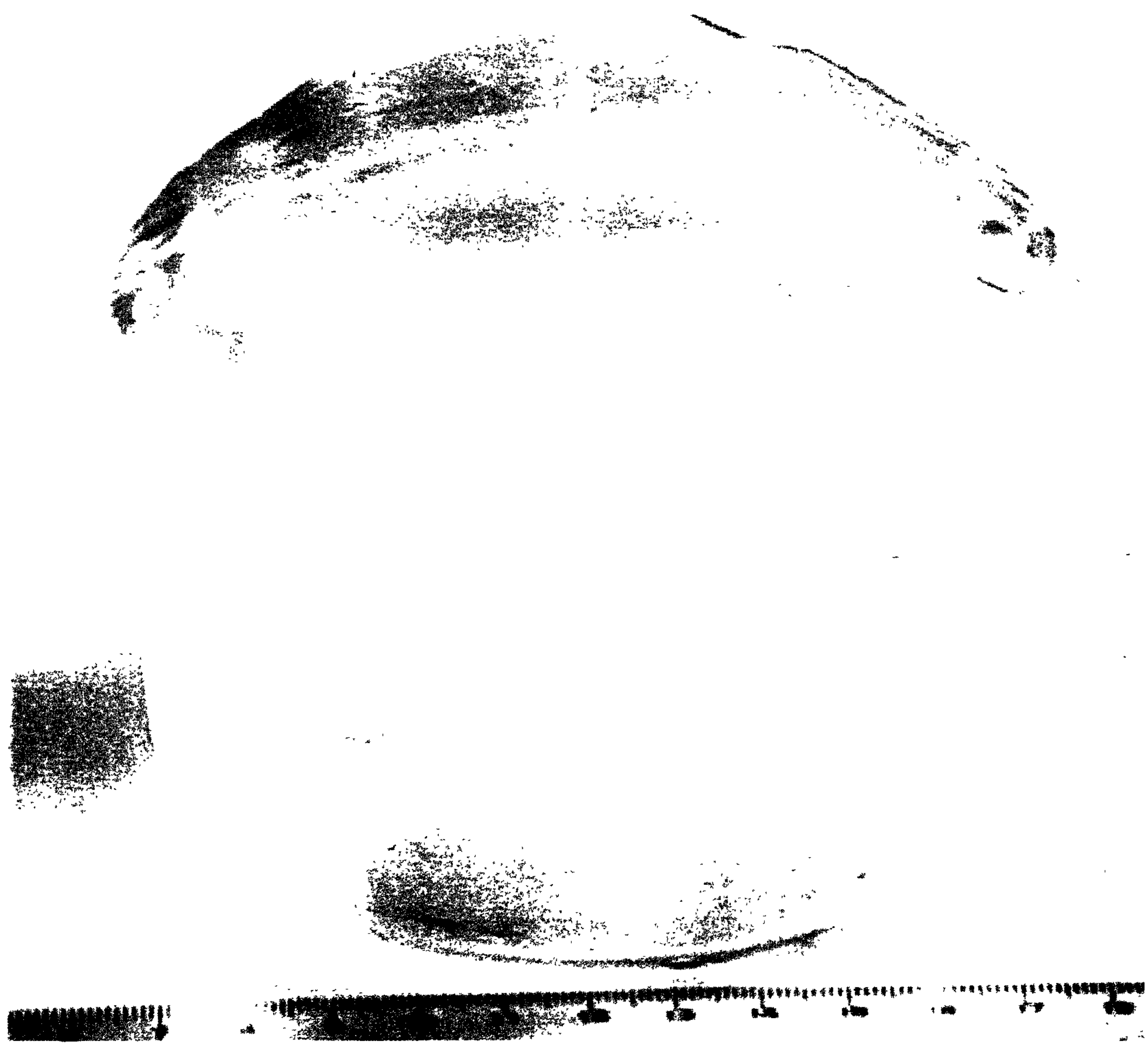
Jan 25, 2000, SIOM

As Grown Sapphire Sample # 30

Jan 25, 2000, SIOM

Annealed Sapphire Sample # 31

Jan 25, 2000, SIOM



GDMS Analysis for Sapphire Development

by Shiva Technology West (8/5, 12/12/97 & 5/28/98)

Element	Japan	Zhejiang	Dalian	Biesterfield	S.E.I
B	0.4	10	1.0	<0.05	<0.05
F	0.4	10	1.0	<0.05	<0.05
Na	<1	18	16	<0.2	<0.2
Mg	11	2.1	7.6	0.5	0.2
Si	17	15	2.9	0.5	1.0
Ca	20	5.6	1.5	<0.05	<0.05
Ti	0.6	1.1	0.5	0.3	6.0
Cr	<10	18	10	<1	<1
Fe	15	20	<10	<1	<1
Co	3.2	1.1	0.5	<0.005	<0.005
Ni	19	<5	7.5	<0.05	<0.05
Cu	1.4	1.9	0.9	<1	<1
Purity(%)	99.9897	99.9891	99.9948	99.9998	99.9988

- The best raw material from Biesterfield (Czech).
- The best Chinese raw material from Dalian.
- Raw material from S.E.I. (Swiss) has high Ti contamination.
- Also identified contaminations during growth, such as Fe, Co, Ni and Mo.

By Shiva Technology West, Inc.

GDMS Analysis for Sapphire Samples

For Ren-yuan Zhu of Caltech

Customer	Caltech	Caltech	Caltech	Caltech	Caltech	Caltech	Caltech	Caltech	Caltech	Caltech	Caltech	Caltech	Caltech
Date	05. Aug 97	08. Aug 97	05. Aug 97	08. Aug 97	05. Aug 97	05. Aug 97	05. Aug 97	12. Dec 97	12. Dec 97	12. Dec 97	28. May 98	28. May 98	02. Jun 98
Customer ID	Al2O3 Crystal 6	Al2O3 Crystal 6	Al2O3 Crystal 7	Al2O3 Crystal 7	Al2O3 Powder 8	Al2O3 Powder 9	Al2O3 Powder 10	Al2O3 Crystal 1	Al2O3 Crystal 2	Al2O3 Crystal 3	Al2O3 Crystal 1	Al2O3 Crystal 2	Al2O3 Ceramic
Shiva ID	W97072413	W97072413	W97072414	W97072414	W97072415	W97072416	W97072417	W97120109	W97120110	W97120111	W98051910	W98051911	W98052110
Description	Japan	Japan (R)	Zhejiang	Zhejiang (R)	Japan	Zhejiang	Dalian	Old SIOM	Dalian, Tail	Dalian, Head	Biesterfield	S.E.I.	Stan
P.O. #	PV 233897	PV 233897	PV 233897	PV 233897	PV 233897	PV 233897	PV 233897	PV 233917	PV 233917	PV 233917	PS 256566	PS 256566	PV256469
Job #	WB0498	WB0498	WB0498	WB0498	WB0498	WB0498	WB0498	WB0852	WB0852	WB0852	WC0563	WC0563	WC0573
Element	Concentration [ppmwt]	Concentration [ppmwt]	Concentration [ppmwt]	Concentration [ppm wt]	Concentration [ppmwt]	Concentration [ppmwt]	Concentration [ppmwt]	Concentration [ppmwt]	Concentration [ppmwt]	Concentration [ppmwt]	Concentration [ppmwt]	Concentration [ppmwt]	Concentration [ppmwt]
Li	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.15	< 0.01	< 0.01	35
Be	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.15	< 0.05	< 0.05	< 0.05
B	2	3	1.4	0.6	0.4	10	1	3	2.5	17	< 0.01	< 0.01	4
O	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix
F	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 0.5	< 0.5	< 1
Na	3.2	20	2.8	5	< 1	18	16	< 1	< 1	< 1	< 0.2	< 0.2	150
Mg	36	79	14	11	11	2.1	7.6	7	2	4	0.5	0.2	55
Al	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix	Matrix
Si	130	390	37	51	17	15	2.9	1	8	< 1	0.5	1	950
P	< 3	4	< 3	< 1	< 3	< 3	< 3	< 3	< 3	< 3	< 0.1	< 0.1	< 0.5
S	2.3	2	< 0.5	< 0.5	12	13	1.2	3	< 0.5	< 0.5	< 0.05	< 0.05	0.3
Cl	< 0.5	< 0.5	0.7	< 0.5	2.9	2.3	0.8	< 0.5	2.5	3	< 0.05	< 0.05	0.5
K	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.2	< 0.2	< 0.5
Ca	2.1	5.3	27	21	20	5.6	1.5	9	1	17	< 0.05	< 0.05	3000
Sc	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Ti	240	240	2.9	3	0.6	1.1	0.5	2.2	< 0.5	4	0.3	6	25
V	0.2	0.5	< 0.05	0.06	< 0.05	0.2	0.1	0.1	< 0.05	0.1	< 0.01	< 0.01	1.5
Cr	140	200	< 10	35	< 10	18	10	80	< 10	< 10	< 1	< 1	8
Mn	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.05	< 0.05	4
Fe	32	24	< 10	6	15	20	< 10	< 10	< 10	< 10	< 1	< 1/3	180
Co	2.9	0.1	0.3	< 0.1	3.2	1.1	0.5	< 0.1	< 0.1	< 0.1	< 0.005	< 0.005	0.07
Ni	< 5	< 2	< 5	< 2	19	< 5	7.5	5	<5	30	< 0.05	< 0.05	0.8
Cu	0.7	< 0.2	< 0.5	0.9	1.4	1.9	0.9	< 0.5	< 0.5	1.5	< 10	< 10	80
Zn	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.5
Ga	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.5
Ge	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.5
As	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 0.05	< 0.05	< 0.5
Se	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.01	< 0.01	< 1
Br	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.5
Rb	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.05
Sr	2.3	2	< 0.05	< 0.05	< 0.05	0.4	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Y	1.4	4	< 0.05	0.1	0.1	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	<0.05	< 0.05
Zr	4.4	5	0.4	0.1	0.2	0.09	1.1	0.5	0.2	0.8	0.8	0.2	8.5

Nb	< 20	< 10	< 20	< 10	< 20	< 20	< 20	< 20	< 20	< 20	<20	<20	<20
Mo	< 5	**9**	< 5	0.2	< 5	< 5	< 5	< 5	**120**	< 5	< 5	<5	< 5
Ru	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Rh	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Pd	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.1	< 0.1	< 0.1
Ag	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	**5**	< 0.1
Cd	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.5
In	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.01	< 0.01	< 0.1
Sn	**1.9**	0.2	0.1	0.3	0.3	0.4	0.8	0.8	0.3	0.4	< 0.05	< 0.05	< 1
Sb	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.1
Te	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.1
I	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.1
Cs	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Ba	**1.7**	**4**	0.5	0.1	< 0.01	0.3	0.09	0.09	0.09	0.09	< 0.05	< 0.05	**2**
La	0.3	< 0.01	0.08	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.05	< 0.05	< 0.05
Ce	0.2	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Pr	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Nd	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.1	< 0.1	< 0.5
Sm	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Eu	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Gd	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Tb	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Dy	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Ho	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Er	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Tm	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Yb	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Lu	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Hf	< 0.5	< 0.1	< 0.5	< 0.1	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Ta	Binder	Binder	Binder	Binder	Binder	Binder	Binder	Binder	Binder	Binder	Binder	Binder	Binder
W	< 10	**20**	< 10	< 1	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Re	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.1
Os	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.05
Ir	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.05
Pt	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.5	< 0.5	< 0.5
Au	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 100	< 100	< 100
Hg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.5	< 0.5	< 0.5
Tl	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.05	< 0.05	< 0.05
Pb	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Bi	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Th	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
U	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
TMI	603.6	1012.1	87.18	134.36	103.1	109.49	52.49	111.69	136.59	78.19	2.1	12.4	4504.67
Purity	99.94	99.9	99.99	99.99	99.99	99.99	99.99	99.99	99.99	99.99	100	100	99.55

Two Step Annealing to Eliminate Color Centers

SIOM's Patented Technology

- As grown Sapphire crystal is pinkish because of contaminations of Ti^{3+} from raw material and carbon from oven, which causes oxygen vacancies and thus form F and F^{+} centers.

- After annealing in O_2/Air, sample turns brownish because of strong oxidation of carbon, but $Ti^{3+} \rightarrow Ti^{4+}$;

- After annealing in H_2, the carbon contamination is eliminated as hydrocarbon and Ti ion remains as Ti^{4+}, which compensate oxygen vacancies, so sample is transparent.

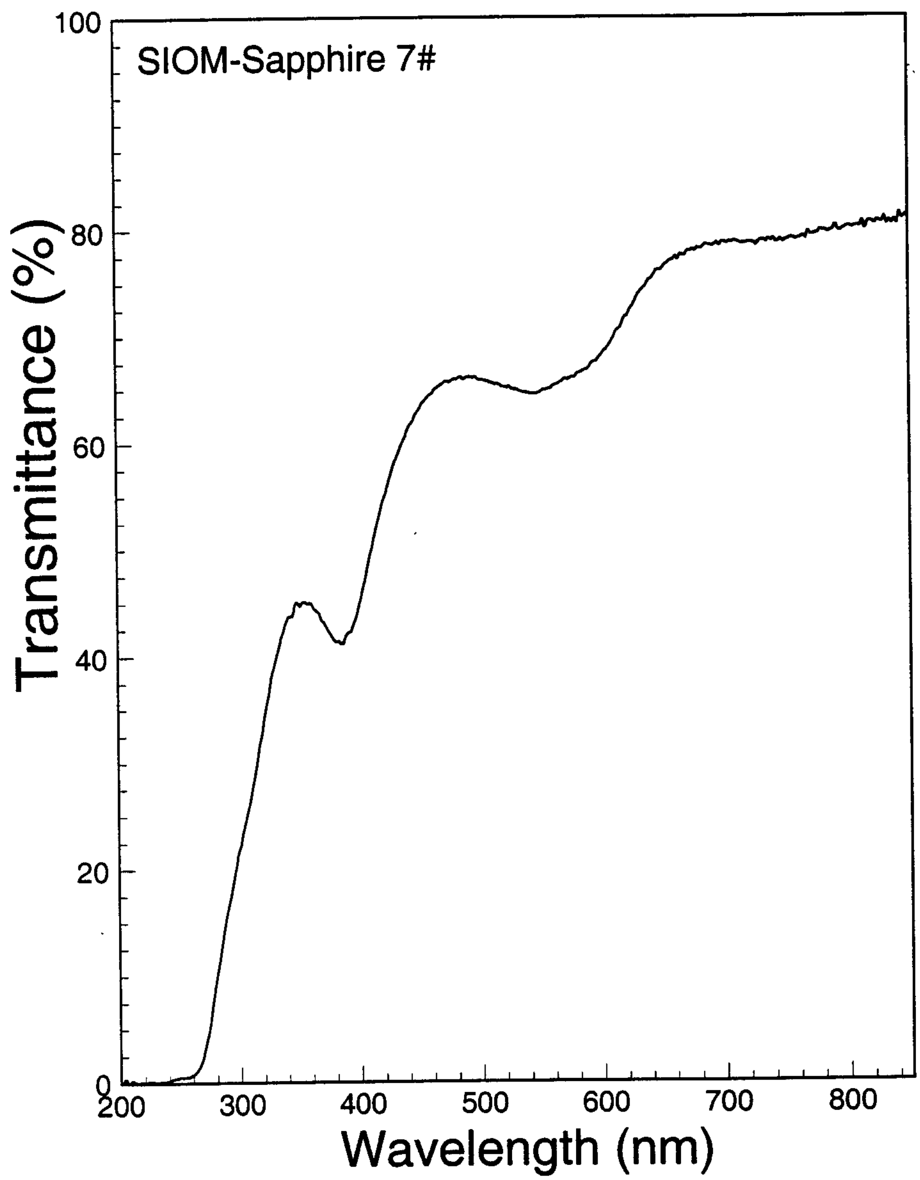
As Grown Sapphire Sample # 7

Sapphire Transmission

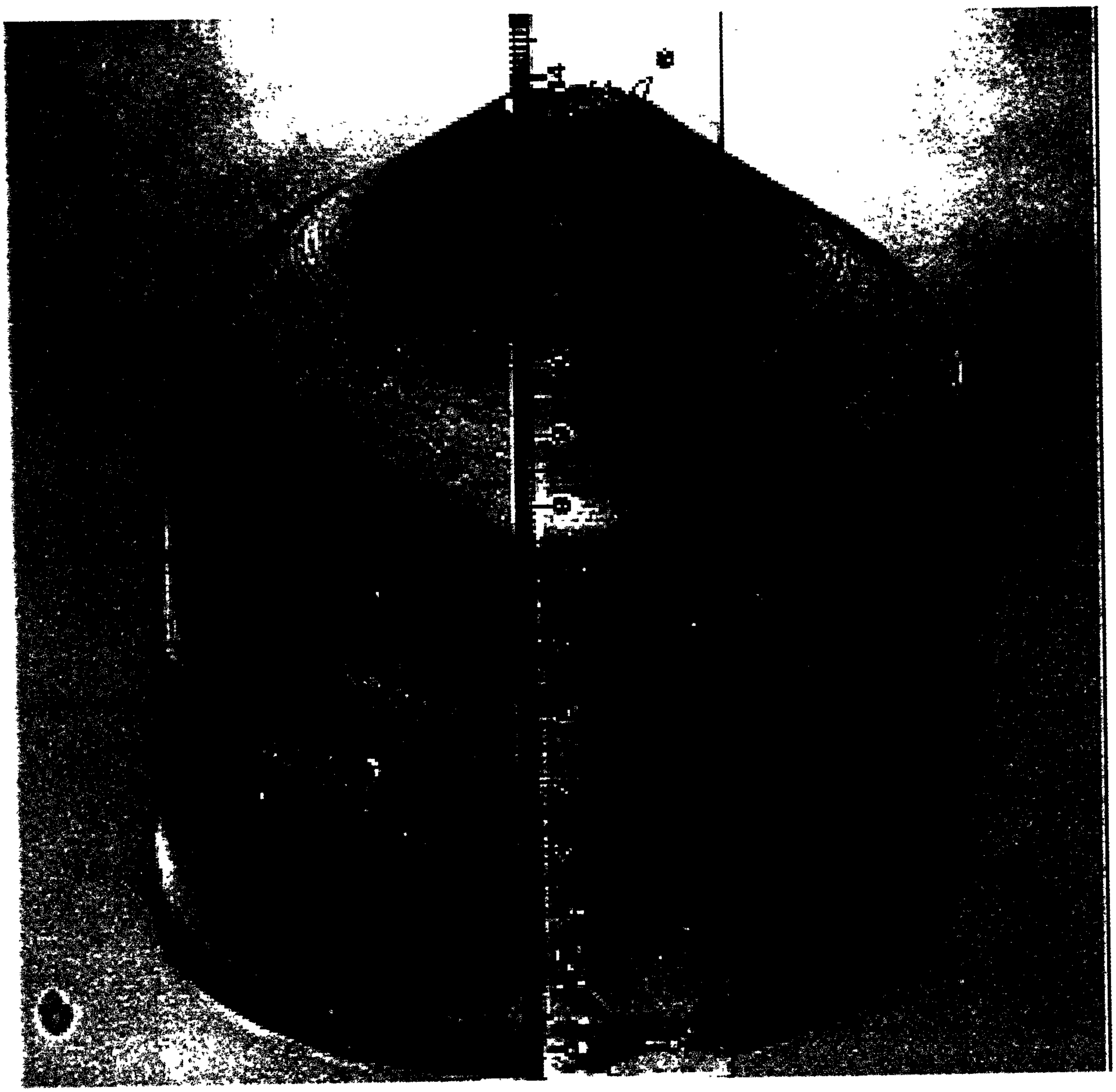
Measured with Hitachi U-3210 Photospectrometer

7.8 cm SIOM-7, Biesterfield (Czech) Material

Identified Contamination of Ti & Cr



As Grown Sapphire Sample # 14

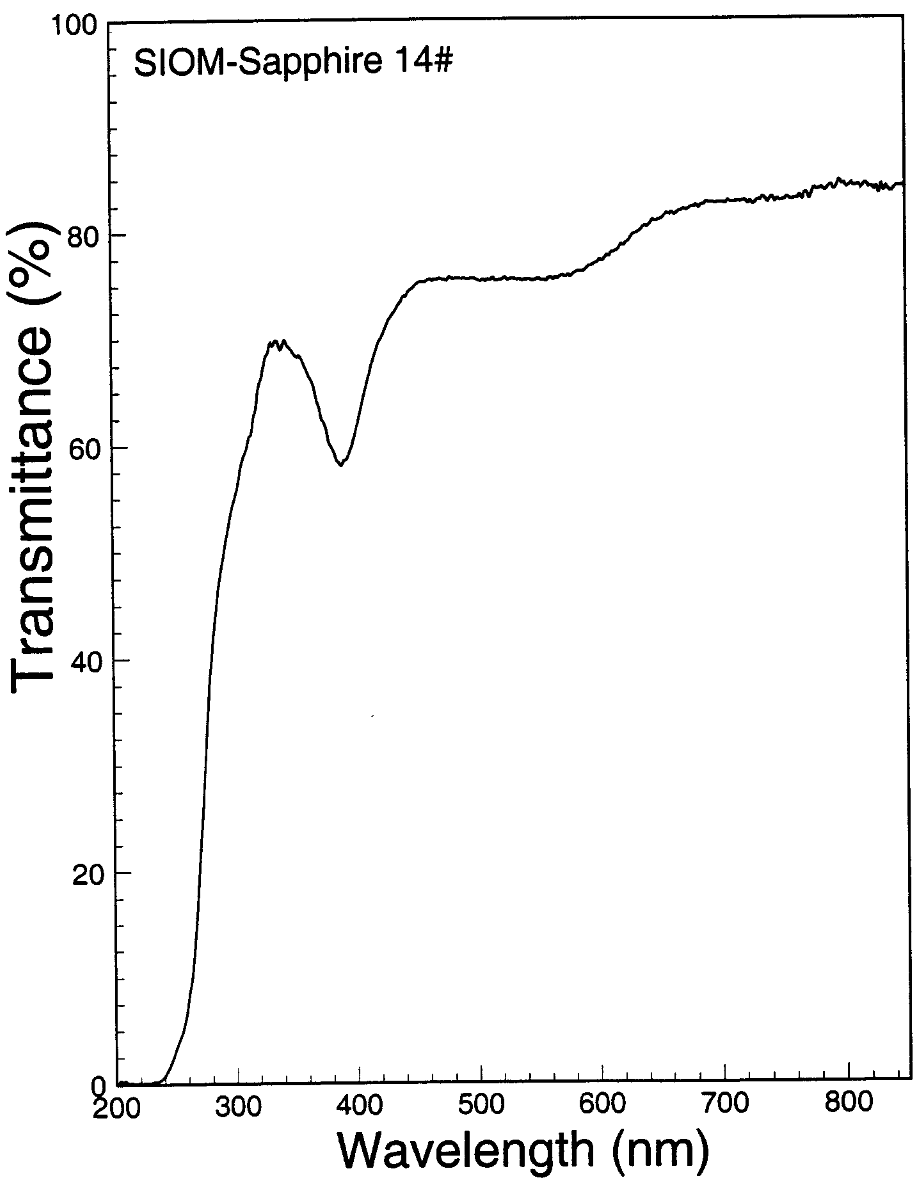


Sapphire Transmission

Measured with Hitachi U-3210 Photospectrometer

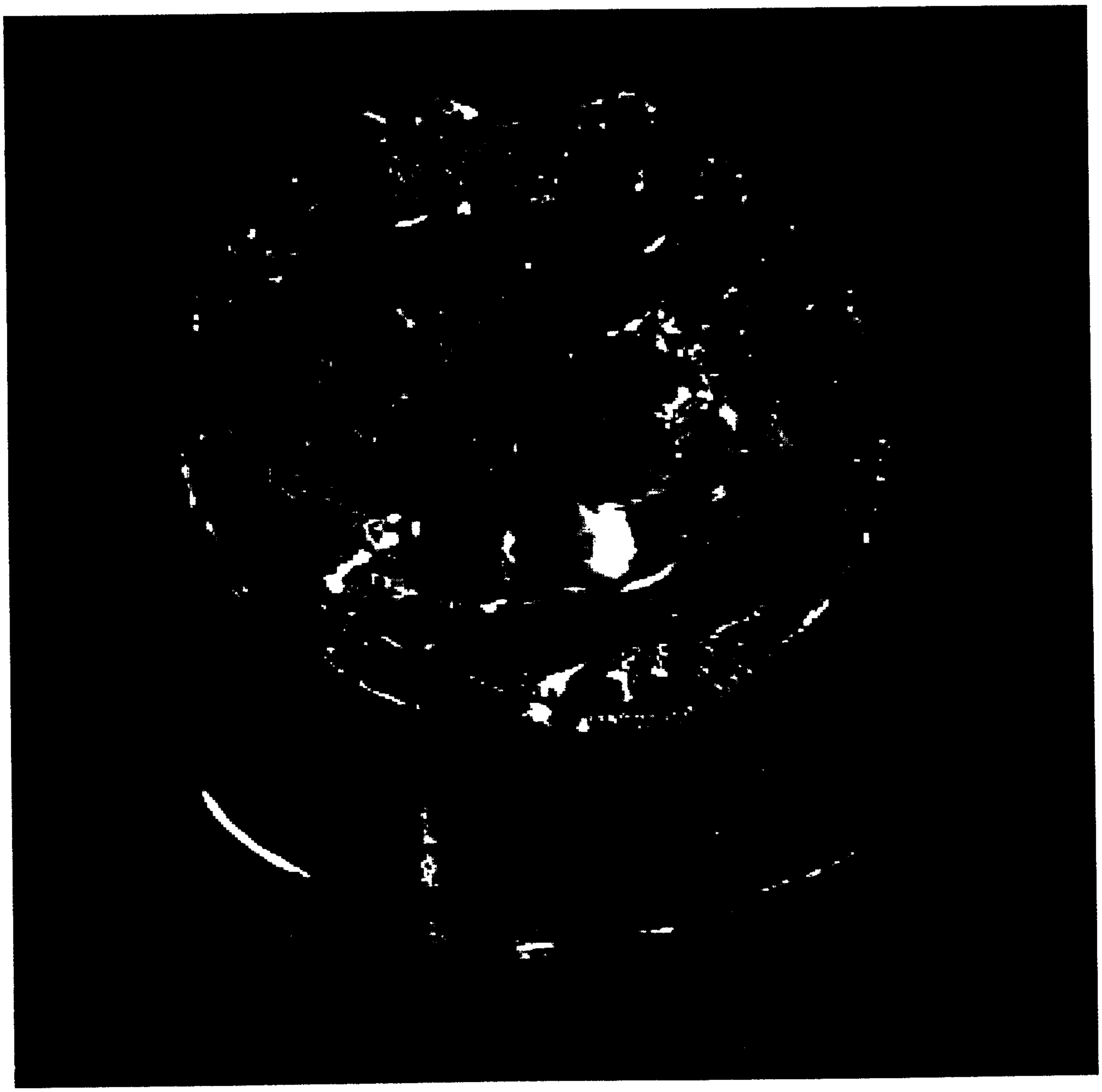
5.3 cm SIOM-14, Dalian Raw Material

Identified Contamination of Ti & Cr



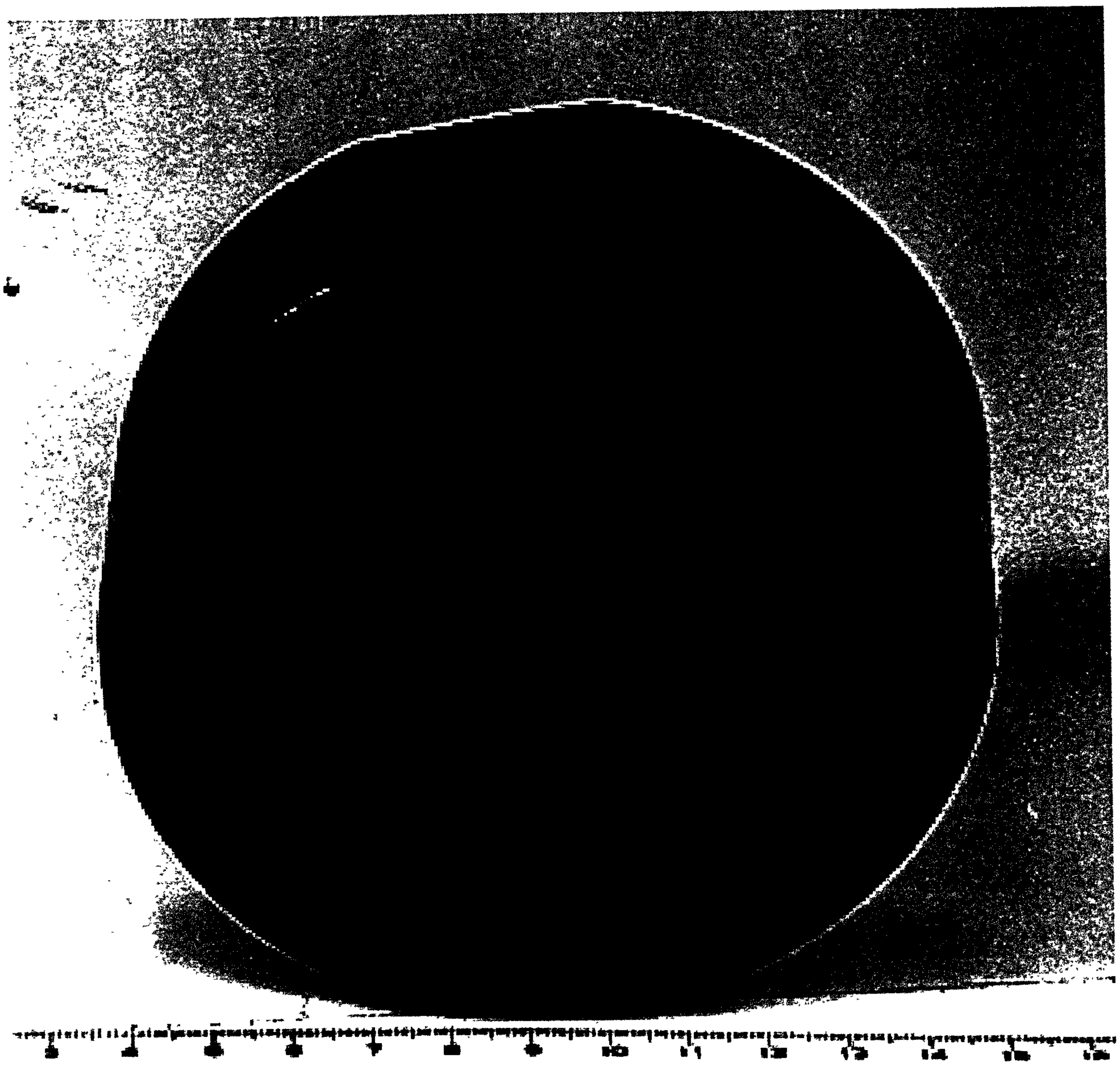
As Grown, Annealed in H_2 and Air

As Grown Sapphire Sample # 3



Annealed in Air Sapphire Sample # 3

ϕ11 cm Slice, A-Axis



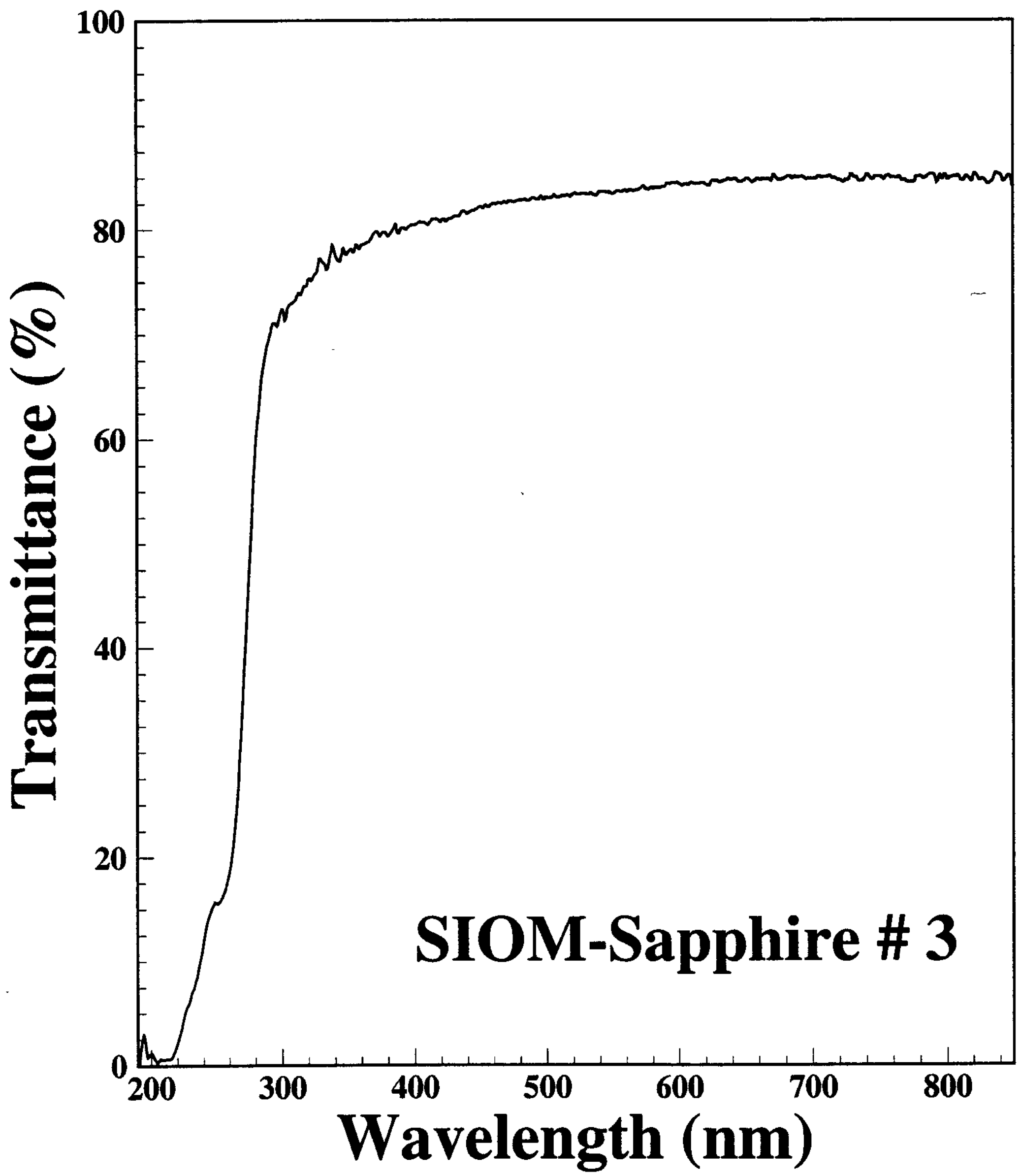
Annealed in H_2 Sapphire Sample # 3

ϕ11 cm Slice, A-Axis

Sapphire Transmission

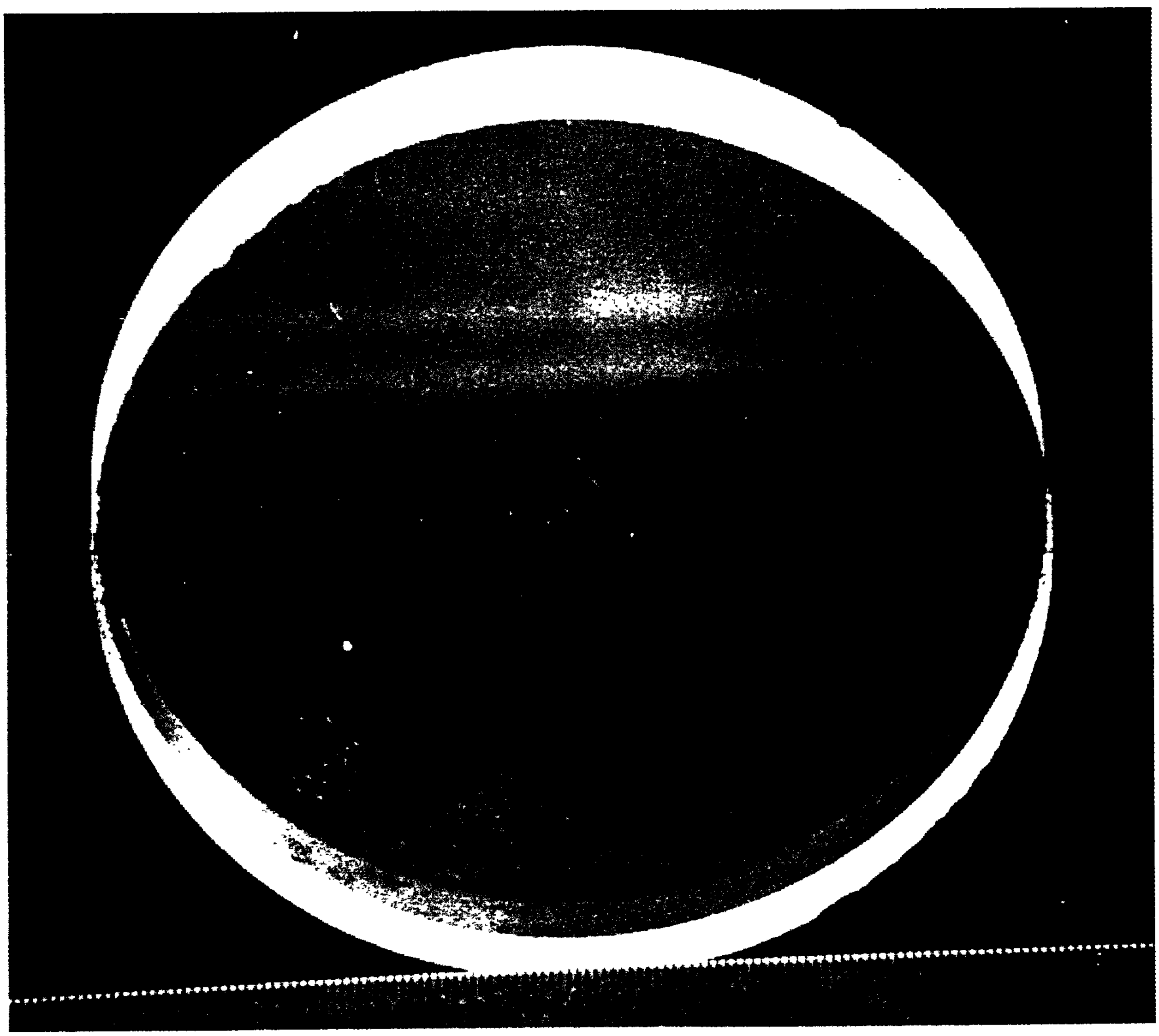
Measured with Hitachi U-3210 Photospectrometer

ϕ11 × 3.6 cm SIOM-3, Annealed in H_2



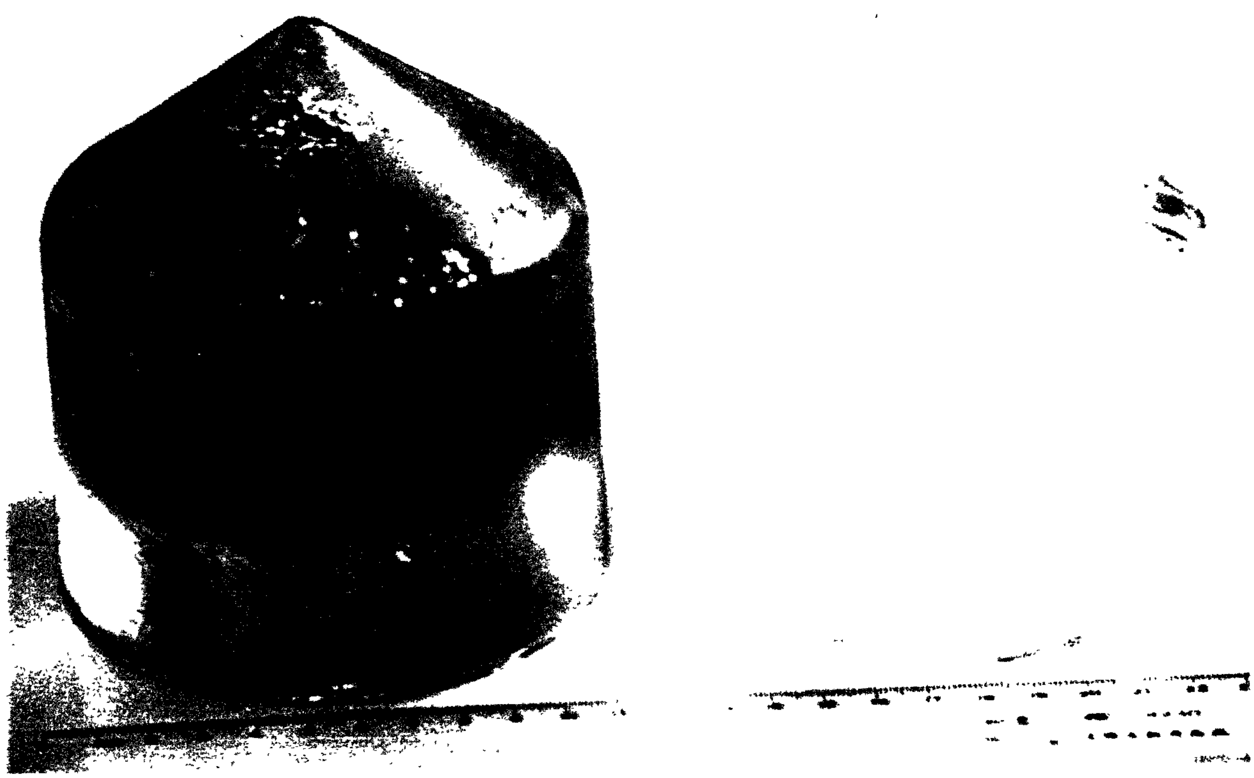
Annealed Sapphire Sample

ϕ11 cm Slice, C-Axis



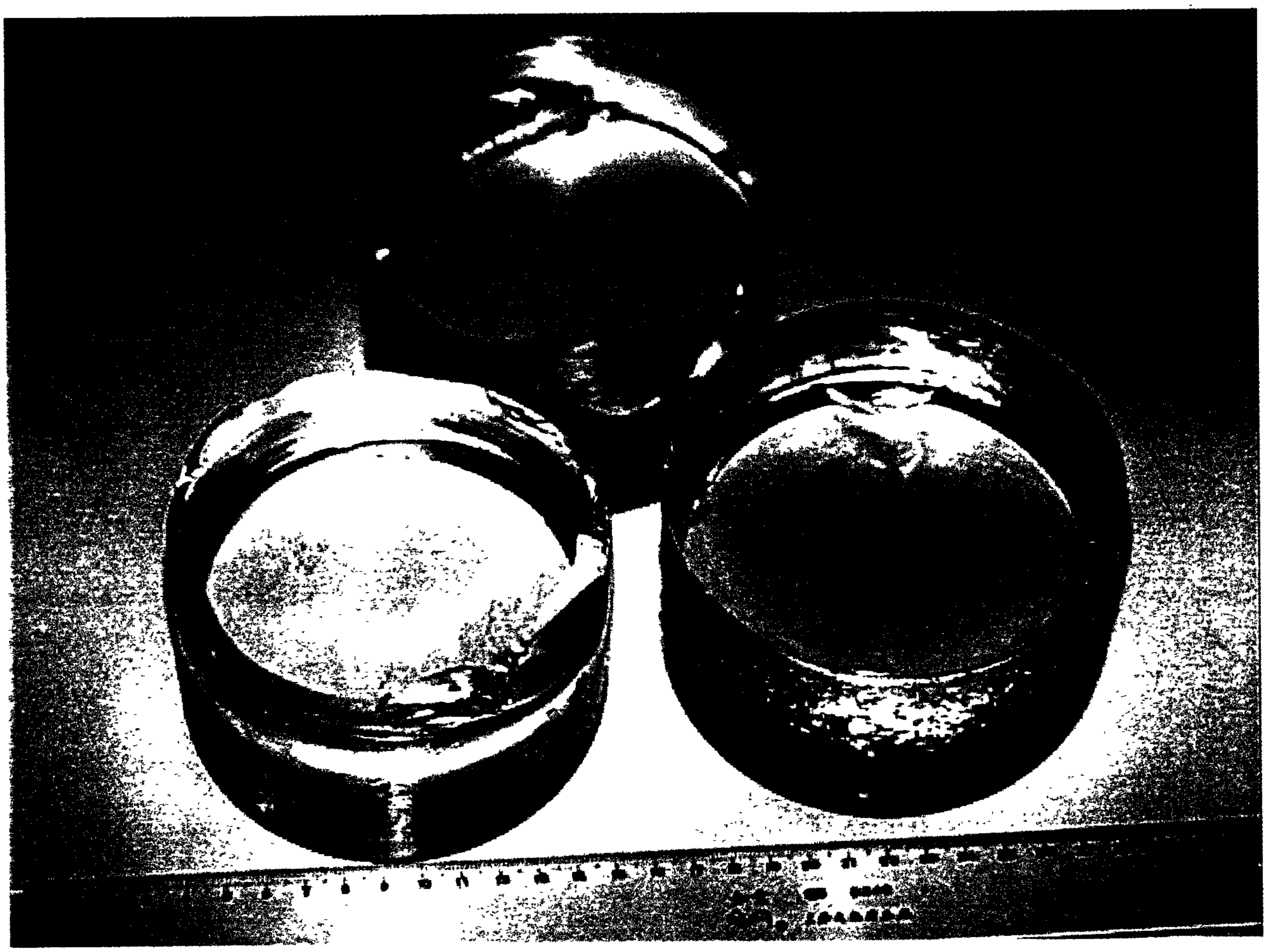
As grown & Annealed Sapphire Sample # 31

Jan 25, 2000, SIOM



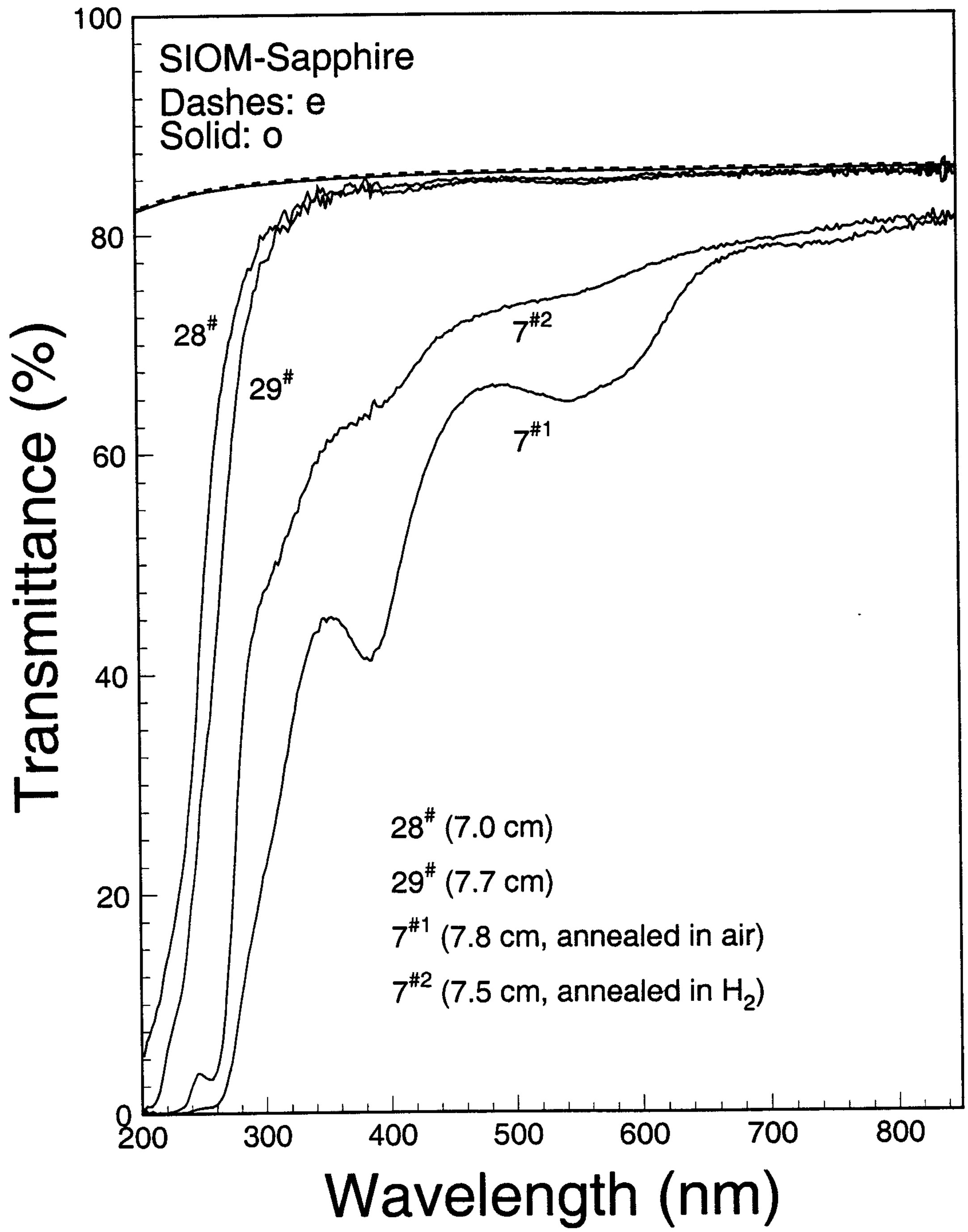
Three Phase I Sapphire Samples from SIOM

Delivered to LIGO in 2000



Transmittance of Sapphire Sample #7, #28 and #29

#28/ & #29 Approaches Theoretical limit: $T = \frac{1-R}{1+R}$, where $R = \frac{(n-n_{air})^2}{(n+n_{air})^2}$



SIOM Sapphire Sample # 28

Delivered in June, 2000

SIOM Sapphire Sample # 29

Delivered in October, 2000

LIGO Evaluation of Sapphire Samples

Data from J. Camp, Sept 14, 2000

Sample	#7	#28	#29	CSI	Quartz
IR Absorption (ppm/cm)	–	35–65		80	
Absorption @514 nm (ppm/cm)	–	280–350		1,200	
Homogeneity: PV (nm)	165	107		177	52
rms (nm)	28	17		30	9
SIOM Data: PV (nm)	422	369	362	–	–
rms: (nm)	48	32	52	–	–

- The best SIOM sample #28 is about a factor of two better than typical CSI sample, but its absorption is about a factor of ten worse than LIGO final specification (< 5 ppm/cm).

- The key technical difficulty is the absorption. The homogeneity seems satisfying LIGO requirement.

- Sample #28 and #29 are both grown with Chinese raw material, while sample #7 with raw material from Czech.

- Measurements of homogeneity by LIGO at 1,064 nm and SIOM at 632.8 nm are more or less consistent.

Issues for Future Sapphire Development at SIOM

- SIOM should start construction of new growth and annealing ovens for ϕ25 cm Sapphire, and refine TGT to reduce inclusions.

- SIOM may use raw material from Biesterfield (originally from Czech), which has less contamination (5 nines) than that from China (4 nines).

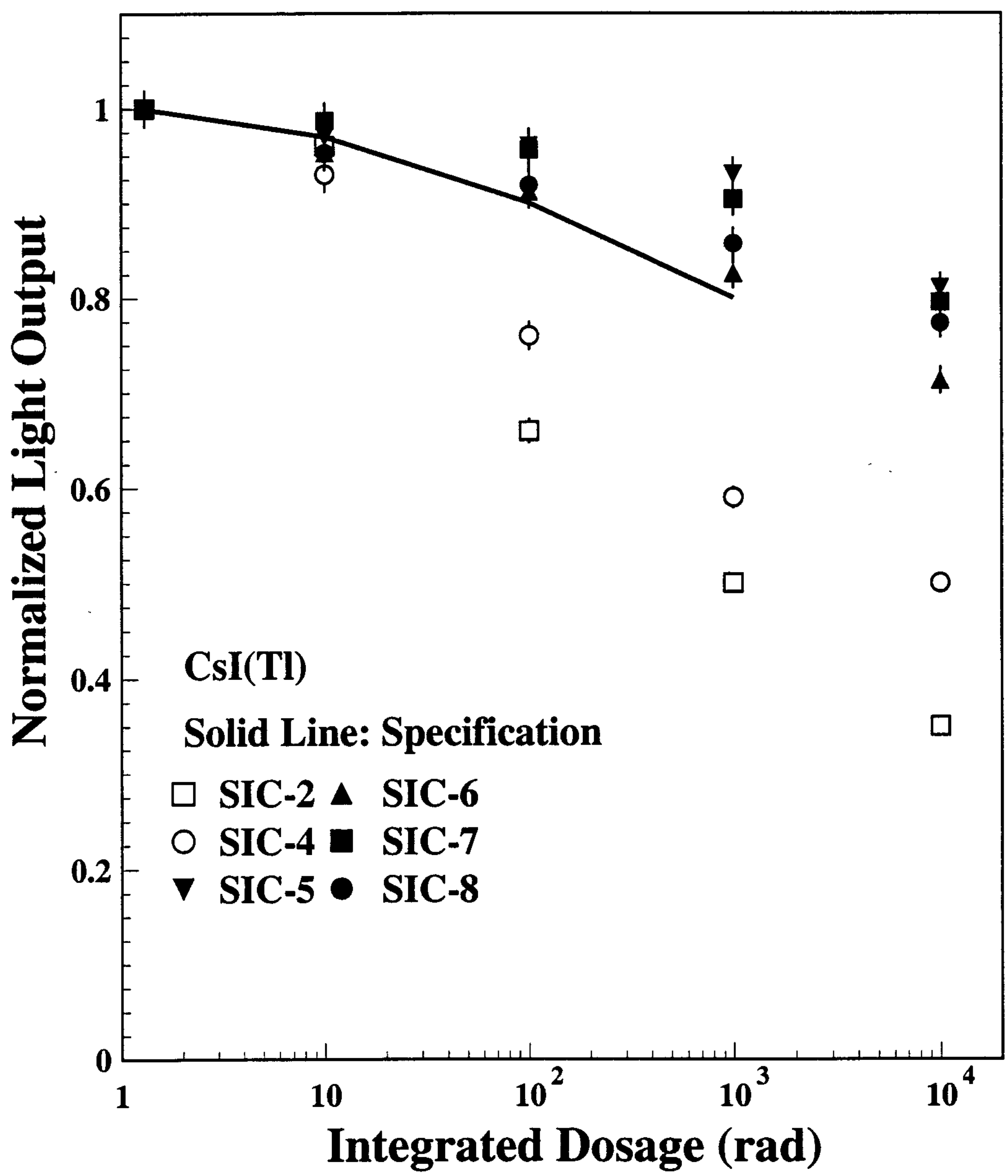
- LIGO should provide ϕ25 cm Mo crucible and cutting/polishing machine.

- LIGO may do more material characterization to help reducing color centers if necessary, as done in CsI(Tl) and $PbWO_4$ development for high every physics.

CsI(Tl) Radiation Hardness Progress

Measured with 2×2744-08 Si PD and 2μs Shaping

30 cm CsI(Tl) Samples from SIC



CsI(Tl) Damage Mechanism

Oxygen Contamination is known to cause radiation damage for other alkali halide scintillators. In BaF_2, for example, hydroxyl (OH^-) may be introduced into crystal through a hydrolysis process, and latter decomposed to interstitial and substitutional centers by radiation through a radiolysis process,

$$OH^- \rightarrow H_i^0 + O_s^- \ \ or \ \ H_s^- + O_i^0$$

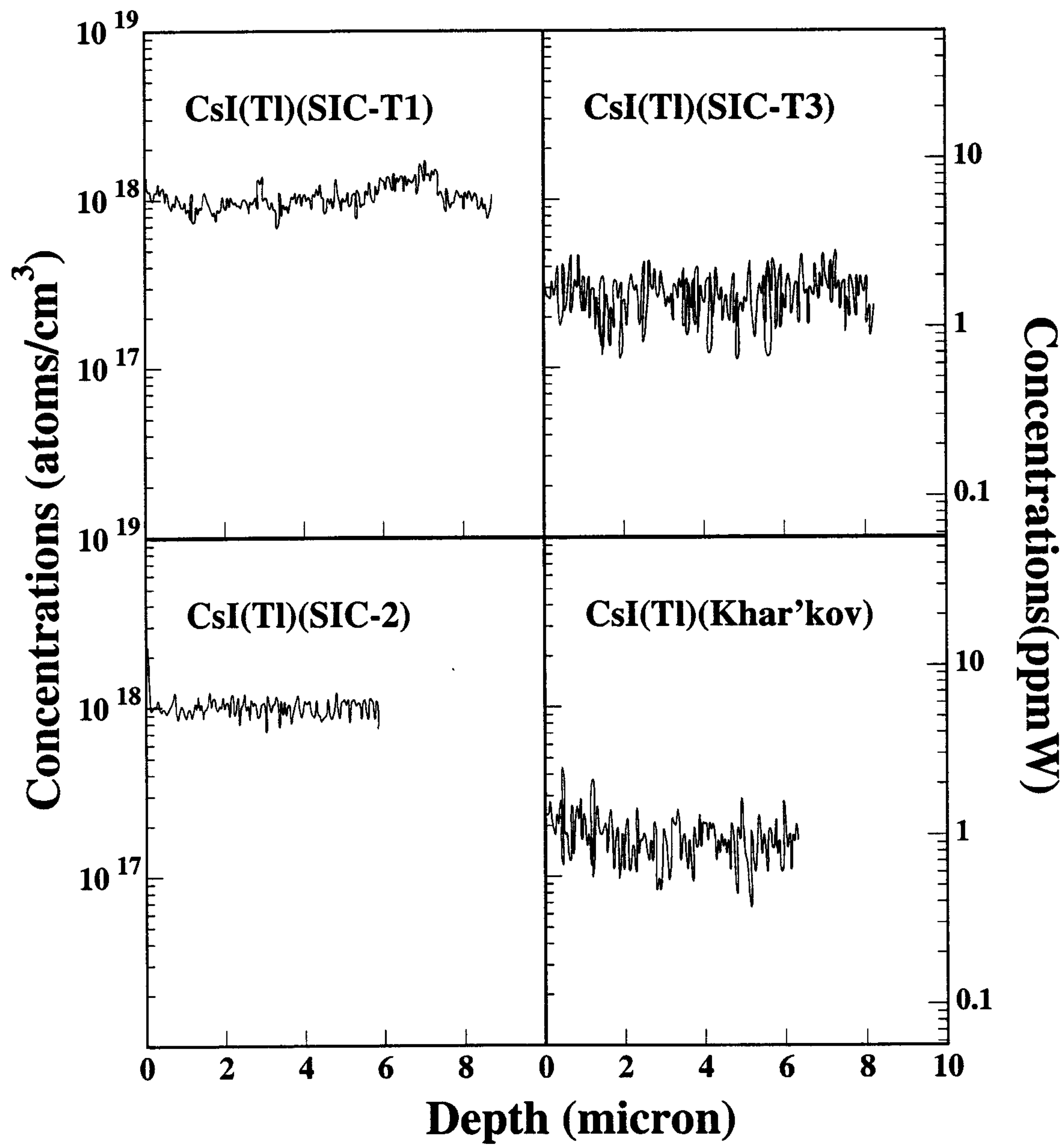
where subscript i and s refer to interstitial and substitutional centers respectively, as discussed in *Nucl. Instr. and Meth.* **A340** 442 (1994).

Possible means for trace oxygen identification: (1) Secondary Ionization Mass Spectroscopy (SIMS); (2) Gas Fusion (LEGO); and (3) Energy Dispersive x-Ray (EDX).

Depth Profile of Oxygen in CsI(Tl)

Secondary Ion Mass Spectrometry Analysis

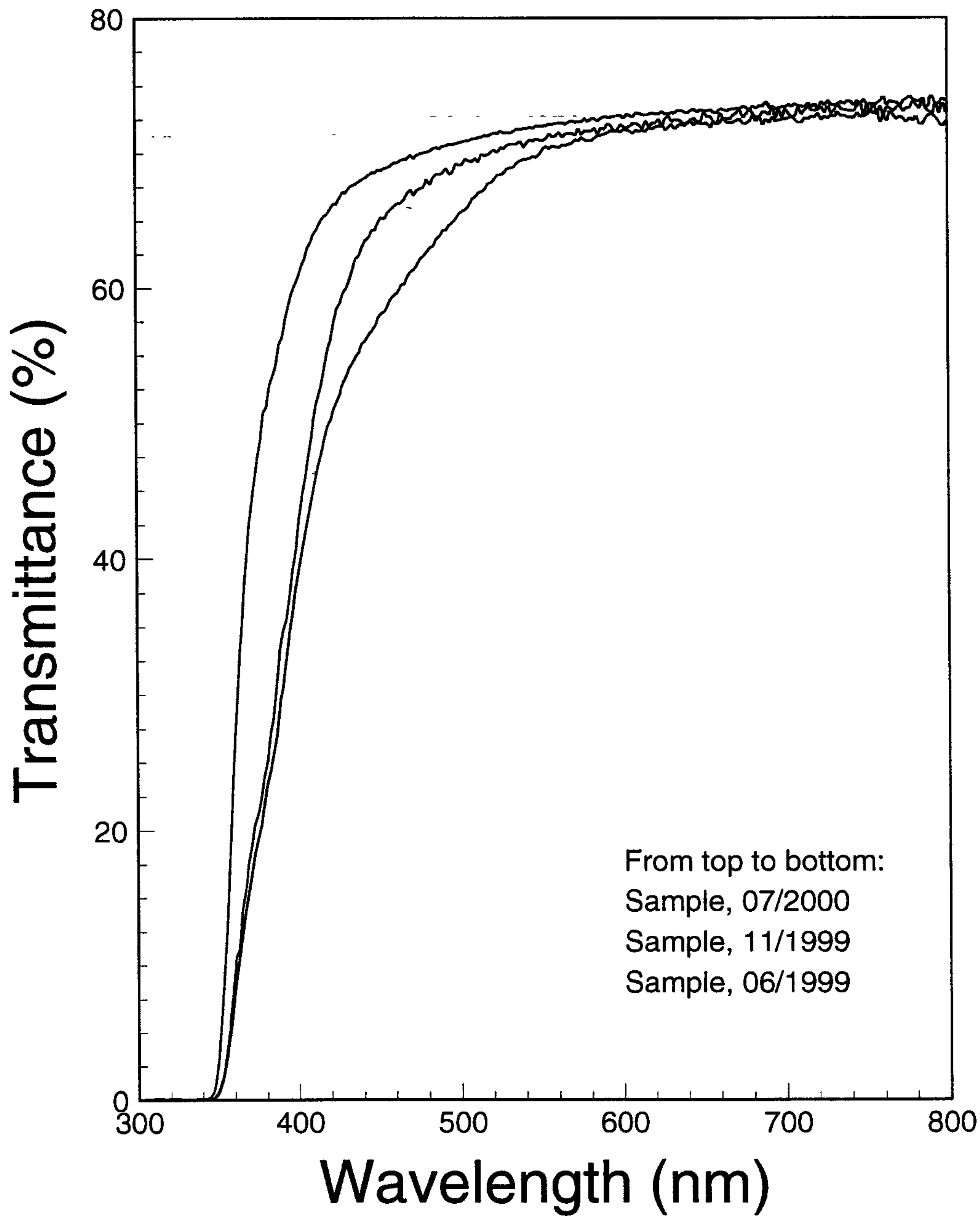
by Charles Evans & Associates



Progress of $PbWO_4$ Transmittance

Measured with Hitachi U-3210 Photospectrometer

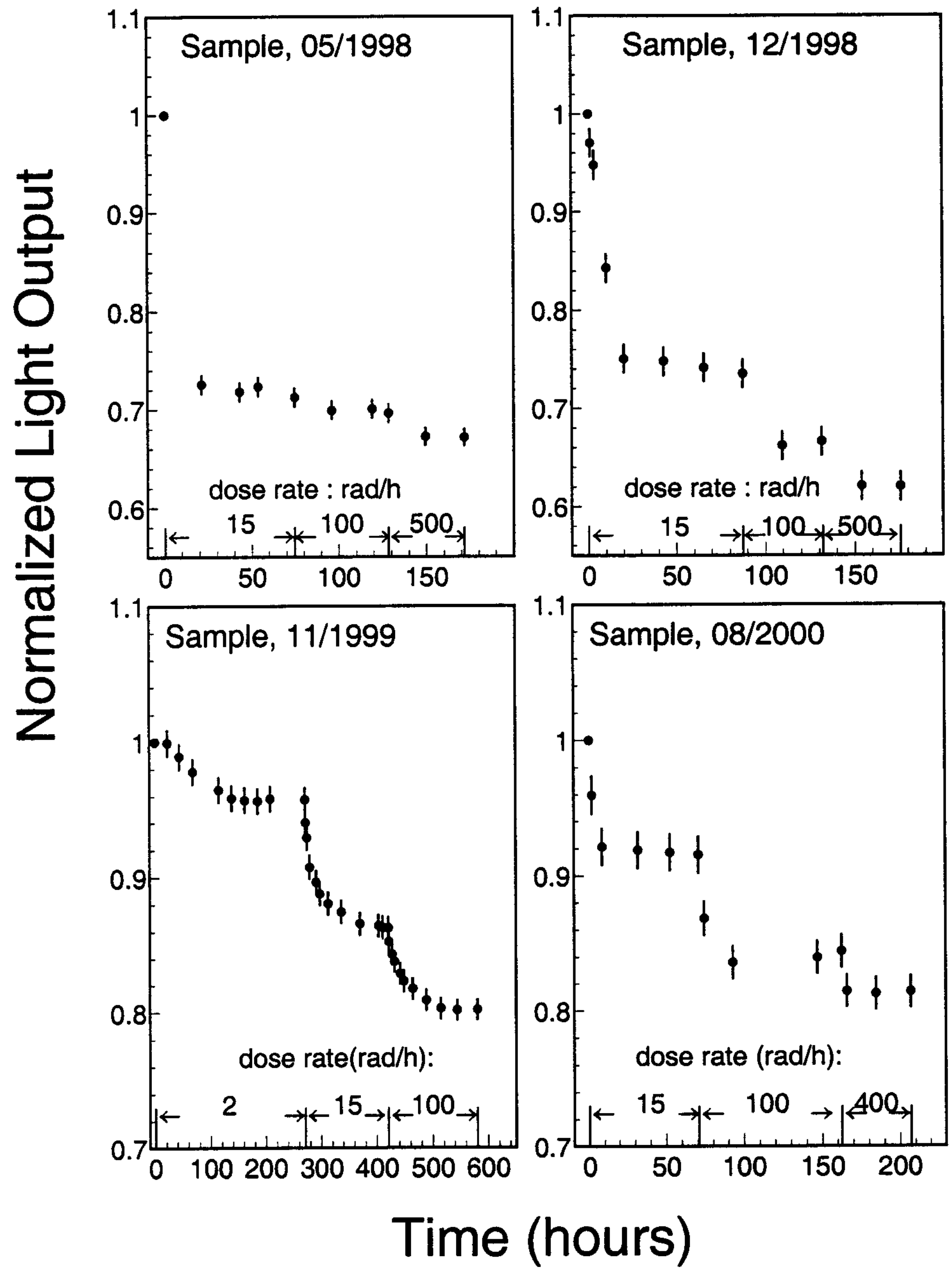
Full size (23 cm) Samples



Progress of $PbWO_4$ Radiation Hardness

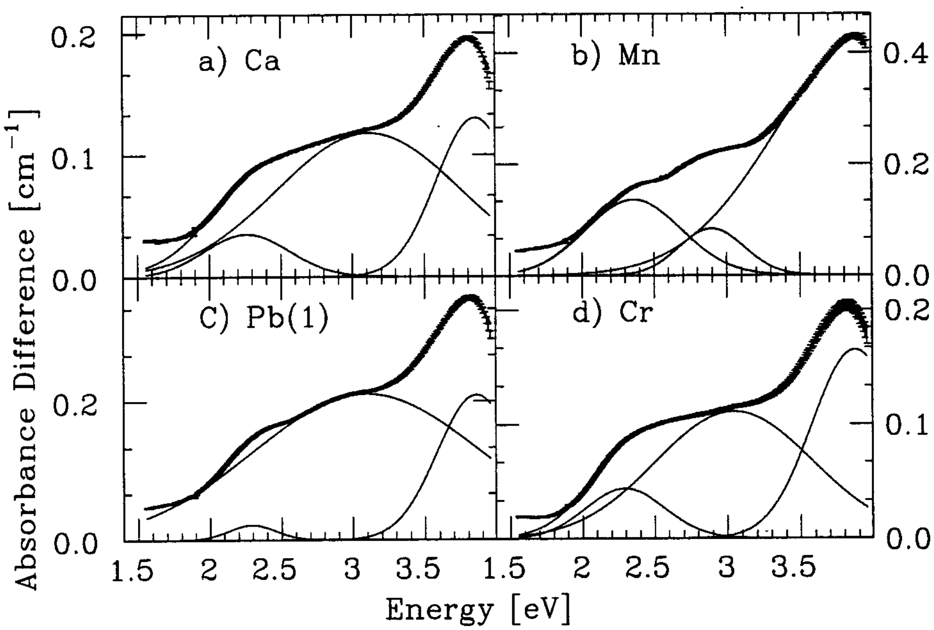
Normalized Light Output Measured with R2059 PMT, 200ns

Full size (23 cm) Samples



$PbWO_4$ Damage Mechanism

Crystal defects, such as Oxygen Vacancy, are known to cause radiation damage for other oxide scintillators. In BGO, for example, three common radiation induced absorption bands at 2.3, 3.0 and 3.8 eV were found in a series of 24 doped samples, as discussed in *Nucl. Instr. and Meth.* **A302** 69 (1991), indicating defect-related color centers.



Possible means for oxygen vacancy identification: (1) Electron Paramagnetic Resonance (ESR) and Electron-Nuclear Double Resonance (ENDOR); (2) Transmission Electron Microscopy (TEM)/Ener Dispersion Spectrometry (EDS); and (3) a pragmatic way: Oxygen Compensation by Post-Growing Annealing in Oxygen Rich Atmosphere.

TEM Study on $PbWO_4$ Crystals

TOPCON-002B Scope, 200 kV, 10 μA

Scale: 1 cm (——) $\Rightarrow$ 20 nm

ϕ5–10 nm **Black Spots Identified**

TEM/EDS Study on $PbWO_4$ Crystals

JEOL JEM-2010 Scope and Link ISIS EDS

Localized (ϕ0.5 nm) Stoichiometry Analysis

Z.W. Yin *et al.*, in SCINT97, Shanghai (9/97)

Oxygen Vacancies Identified

Atomic Fraction (%) in $PbWO_4$

As Grown Sample

Element	Black Spot	Peripheral	$Matrix_1$	$Matrix_2$
O	1.5	15.8	60.8	63.2
W	50.8	44.3	19.6	18.4
Pb	47.7	39.9	19.6	18.4

The Same Sample after Oxygen Compensation

Element	$Point_1$	$Point_2$	$Point_3$	$Point_4$
O	59.0	66.4	57.4	66.7
W	21.0	16.5	21.3	16.8
Pb	20.0	17.1	21.3	16.5

Summary

Starting July 1, 1997, SIOM has been working on Sapphire development for LIGO. LIGO spent 50,000 USD according to the Phase I contract, and identified impurities in raw materials and crystals. SIOM developed a two step annealing technique, which eliminates color centers in Sapphire crystals, and delivered three Phase I samples by October, 2000.

LIGO evaluated samples from SIOM and concluded that sample #28 is one of the samples of the best achieved quality. There are, however, significant technical difficulties in future development at SIOM, such as equipment and infrastructure. With successful completion of Phase I program, a decision for future program should be made by the end of 2000.