

Sapphire Crystal Development at SIOM for LIGO

Ren-yuan Zhu
Caltech

Snowmass, Colorado
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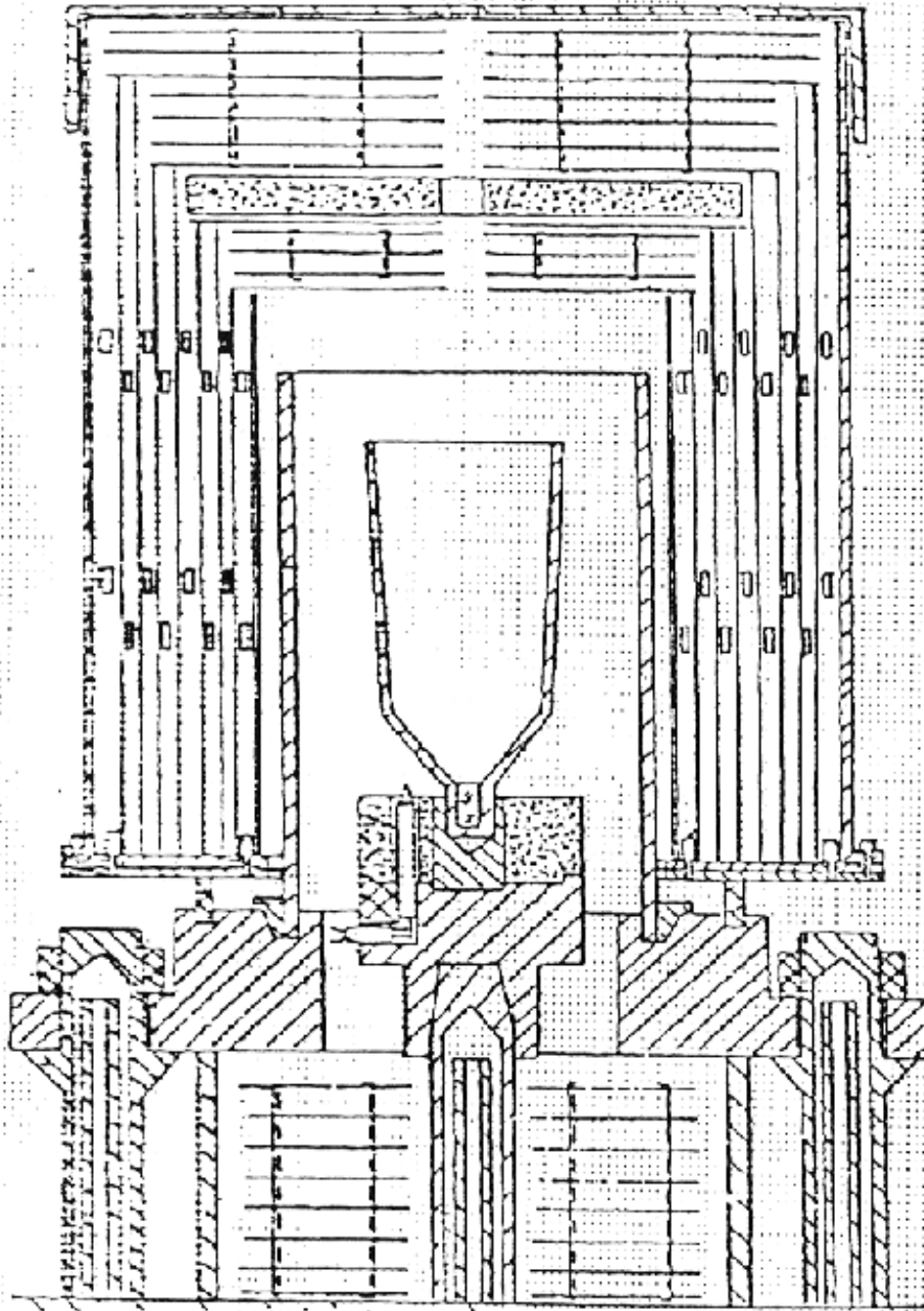
- History of Sapphire Development at SIOM.
- Result of Phase I Program.
- A Discussion on the Phase II program.

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



A Sapphire Growth Furnace at SIOM

Jan 25, 2000, SIOM



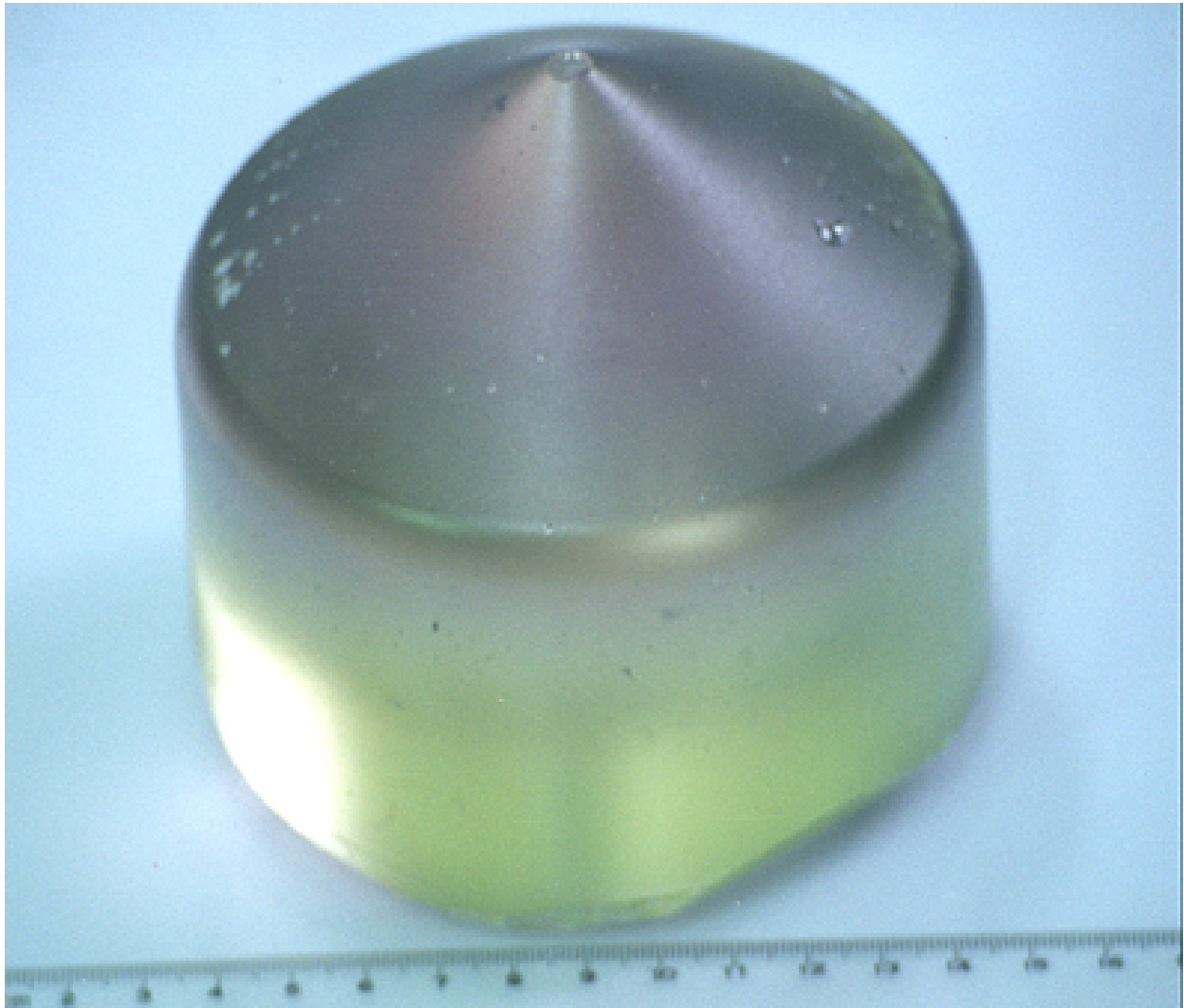
A Sapphire Annealing Furnace at SIOM

Jan 25, 2000, SIOM



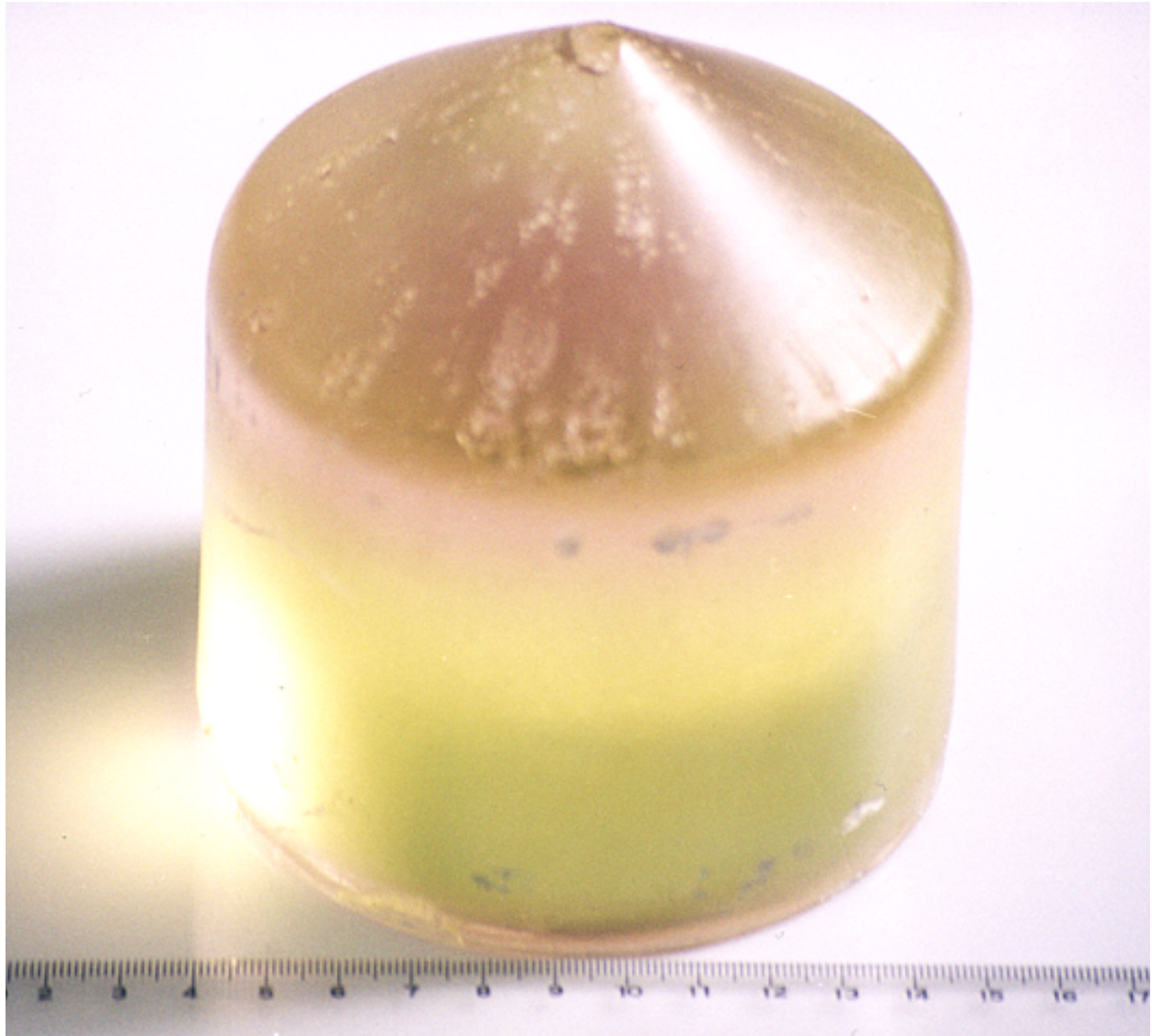
As Grown Sapphire Sample # 28

Jan 25, 2000, SIOM



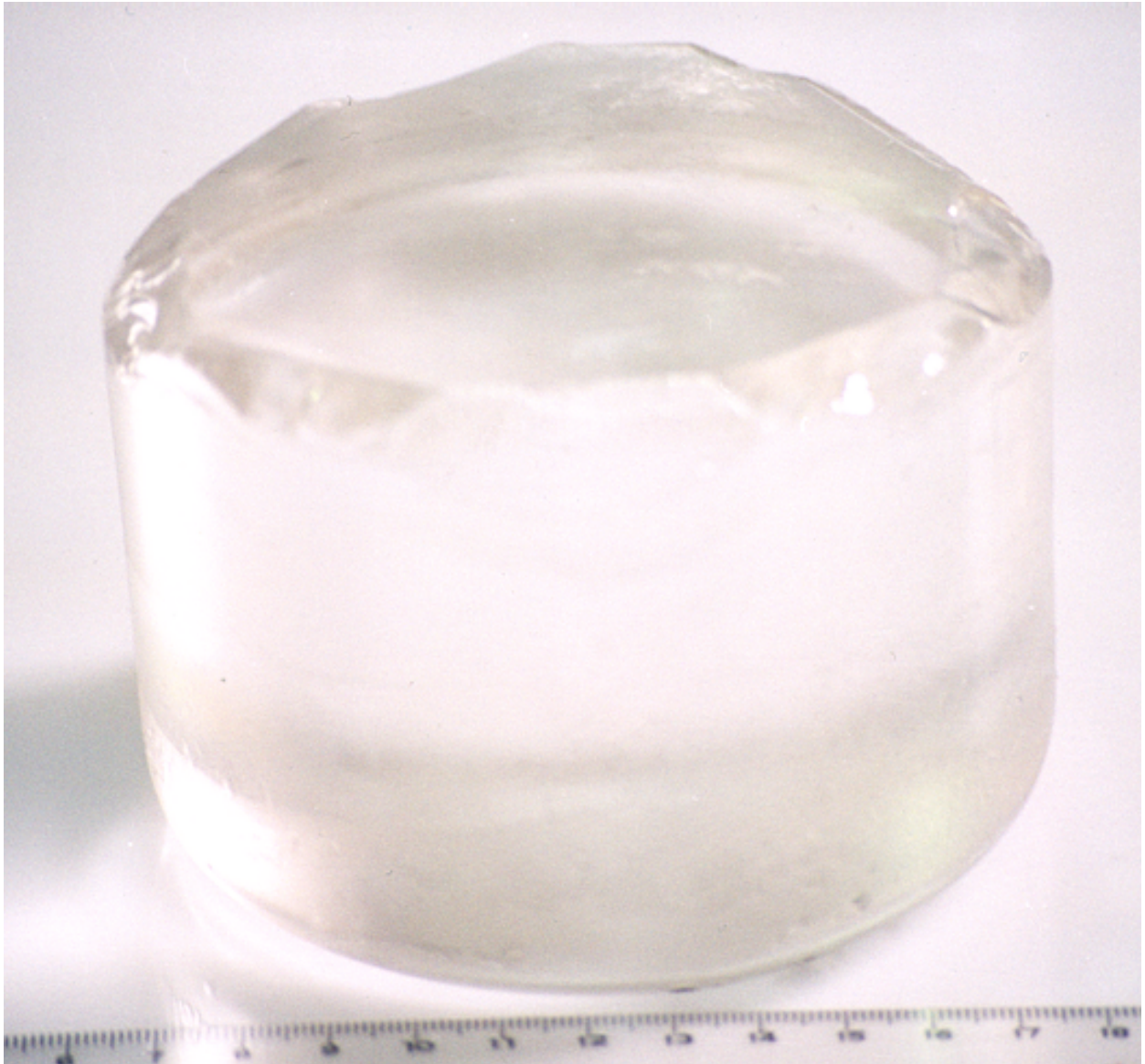
As Grown Sapphire Sample # 30

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

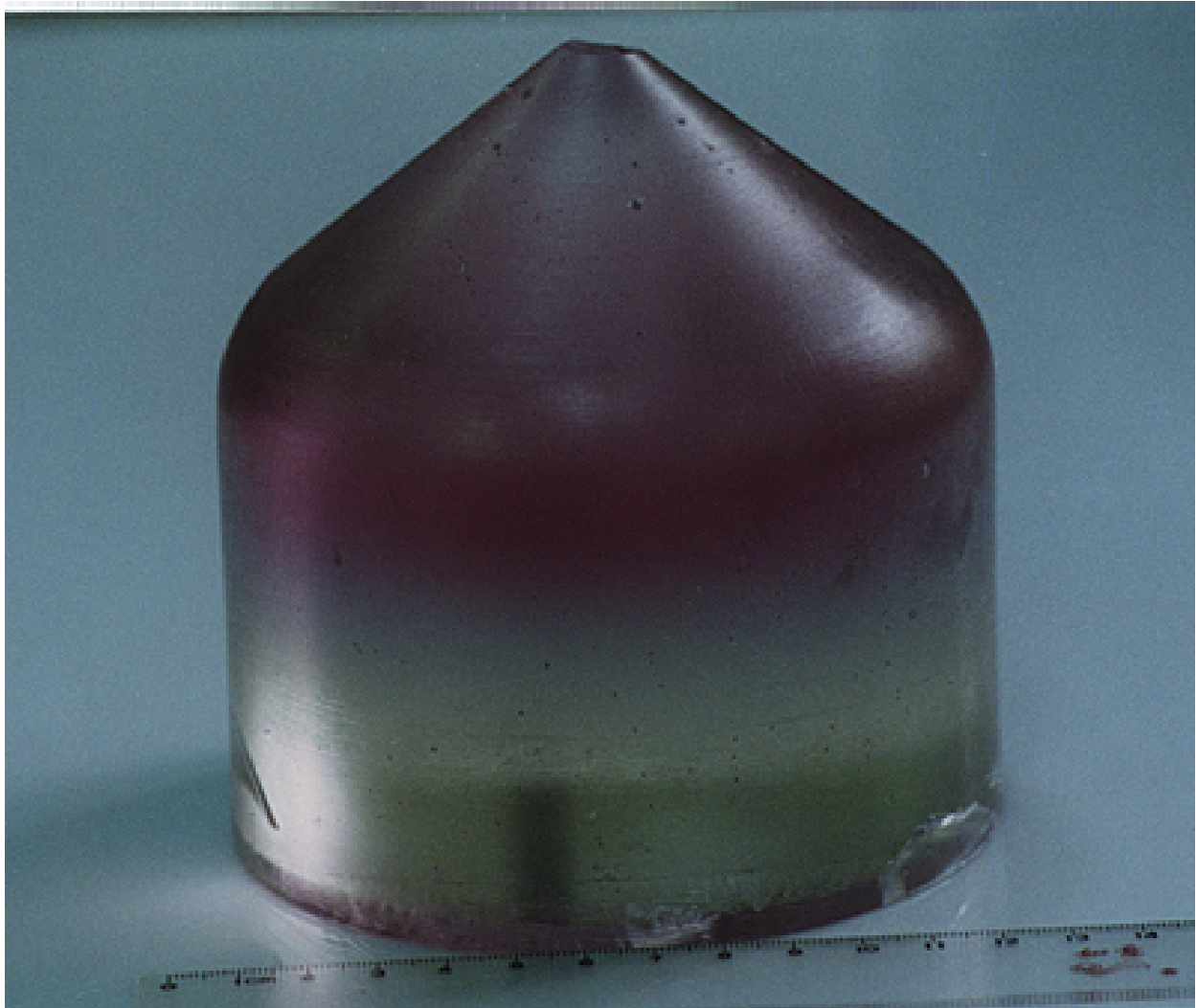
- The best raw material from Biesterfield (Czech).
- The best Chinese raw material from Dalian.
- Raw material from S.E.I. (Swiss) has high Ti contamination.
- Also identified contaminations during growth, such as Fe, Co, Ni and Mo.

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

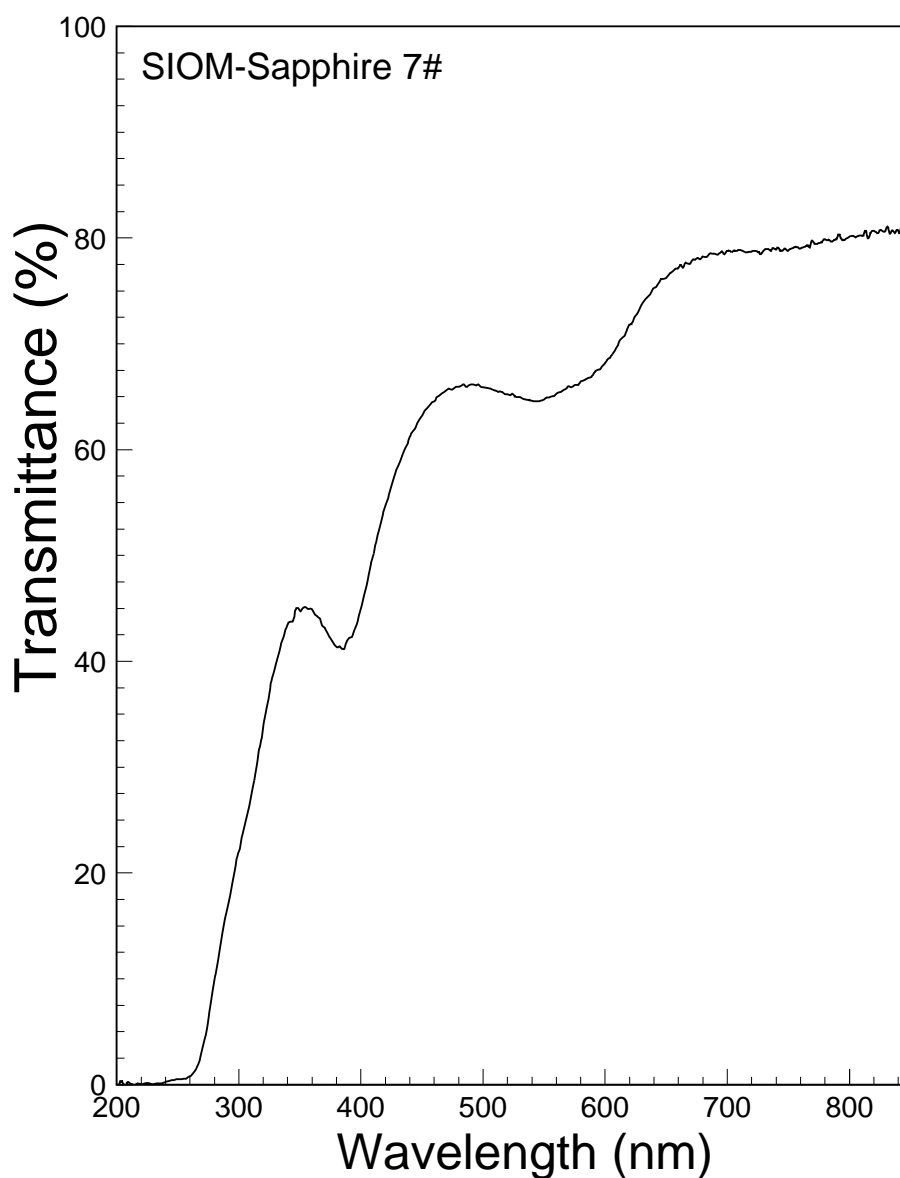


Sapphire Transmission

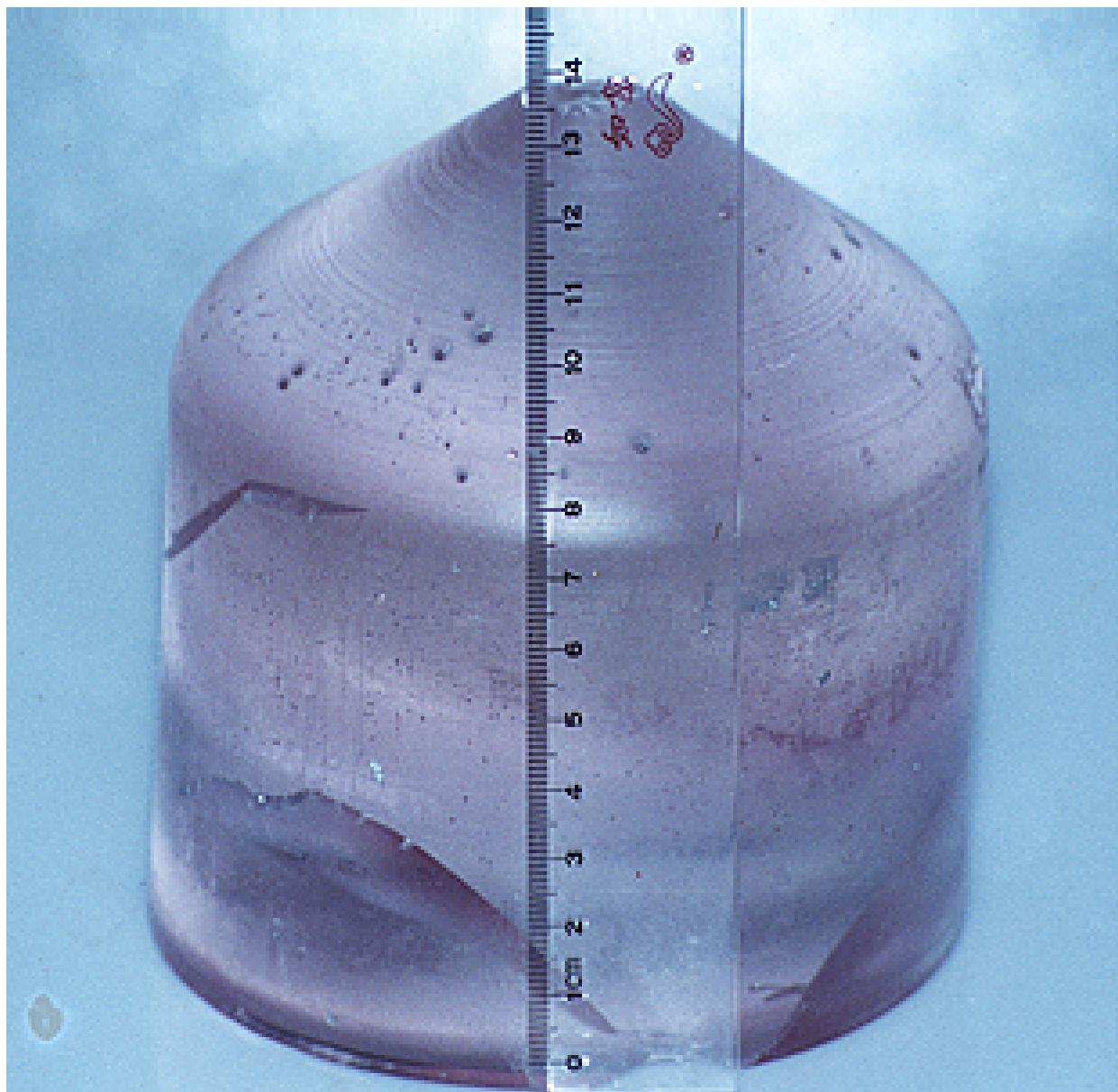
Measured with Hitachi U-3210 Photospectrometer

7.8 cm SIOM-7, Biesterfield (Czech) Material

Identified Contamination of Ti & Cr



As Grown Sapphire Sample # 14

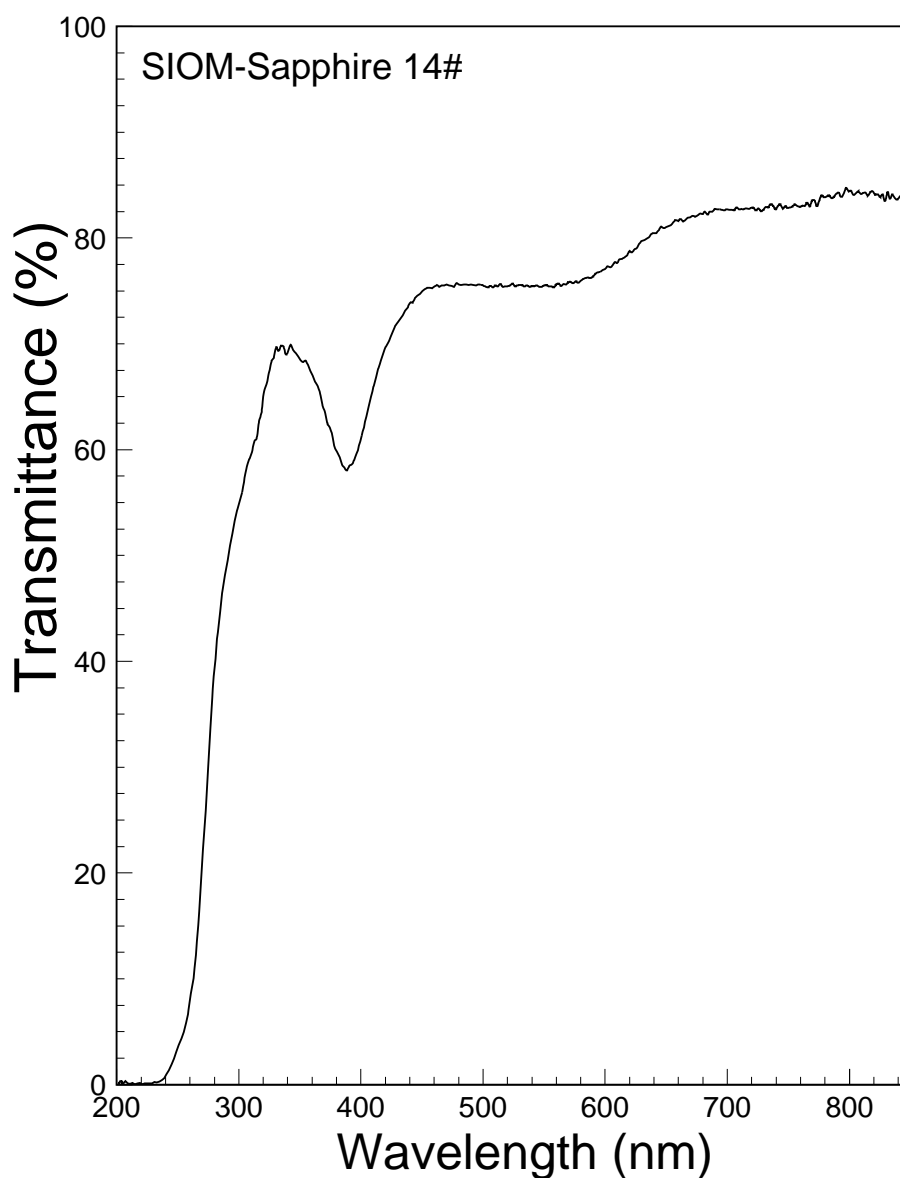


Sapphire Transmission

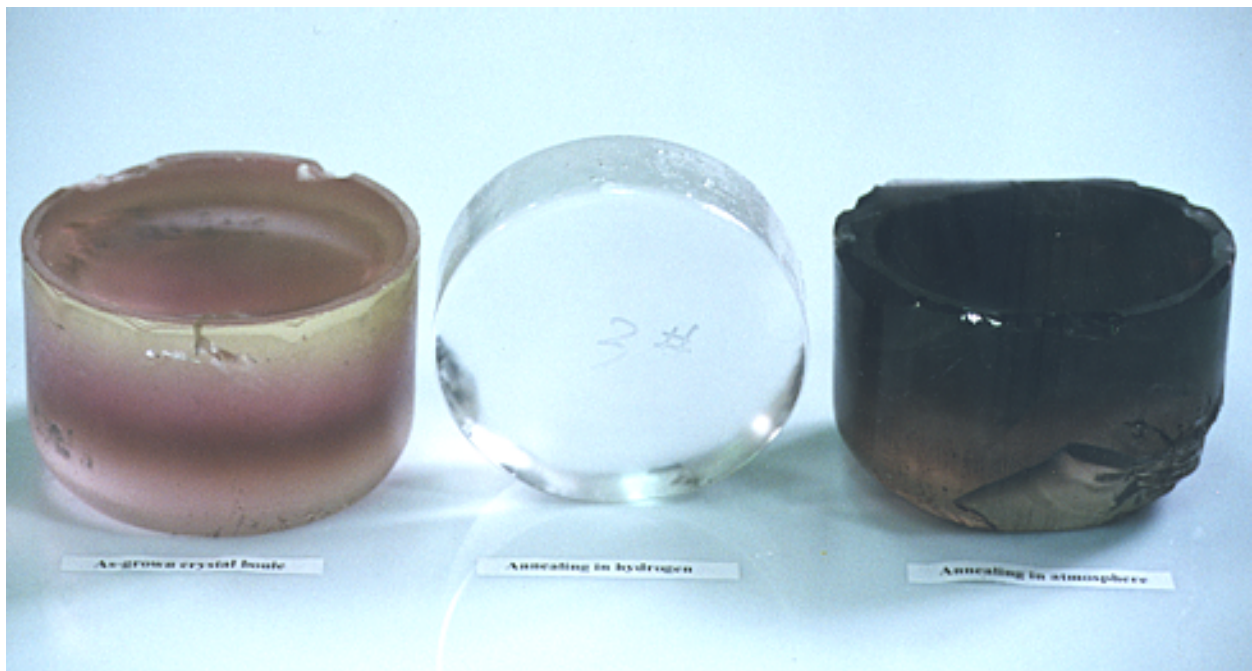
Measured with Hitachi U-3210 Photospectrometer

5.3 cm SIOM-14, Dalian Raw Material

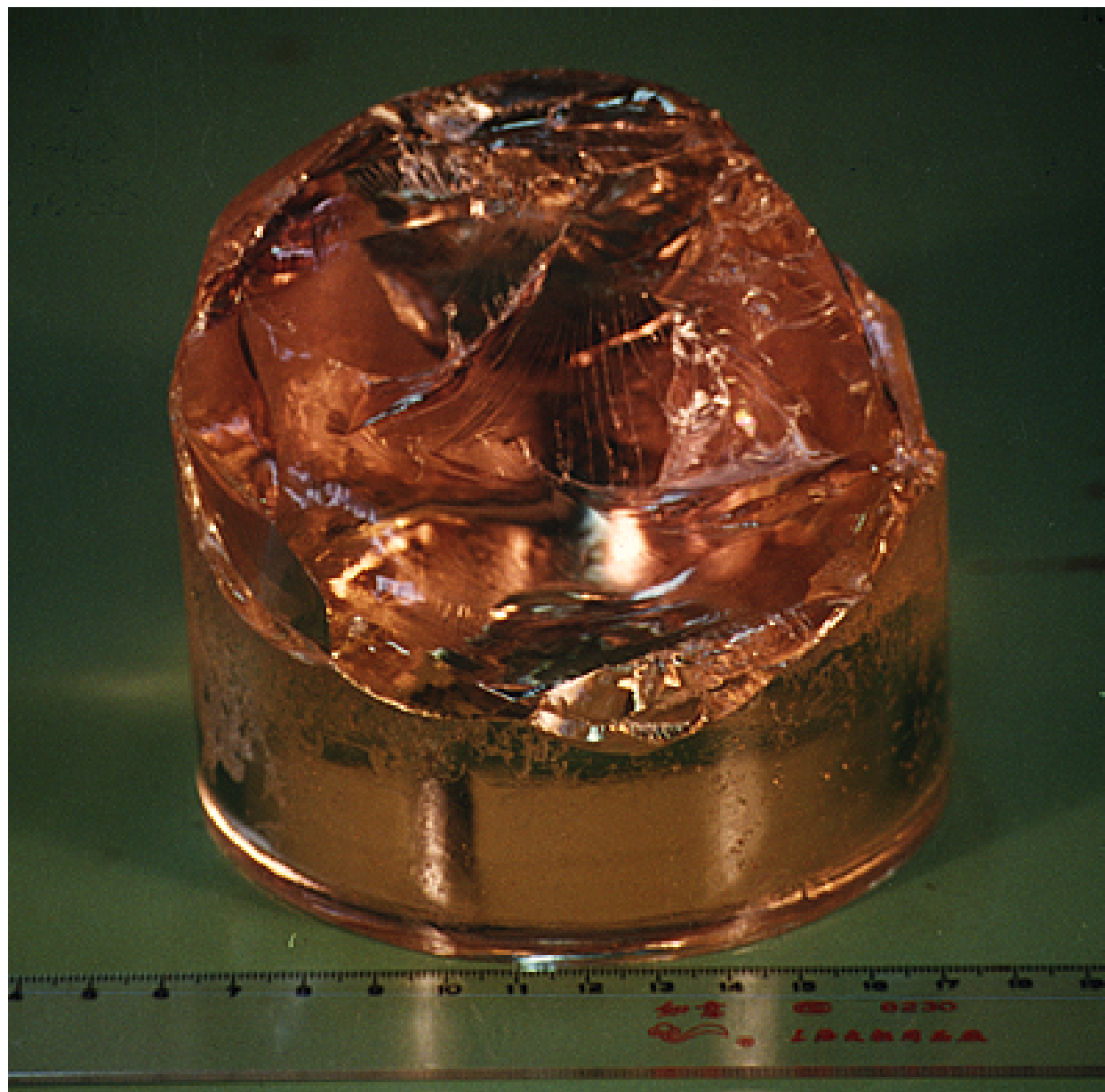
Identified Contamination of Ti & Cr



As Grown, Annealed in H₂ and Air

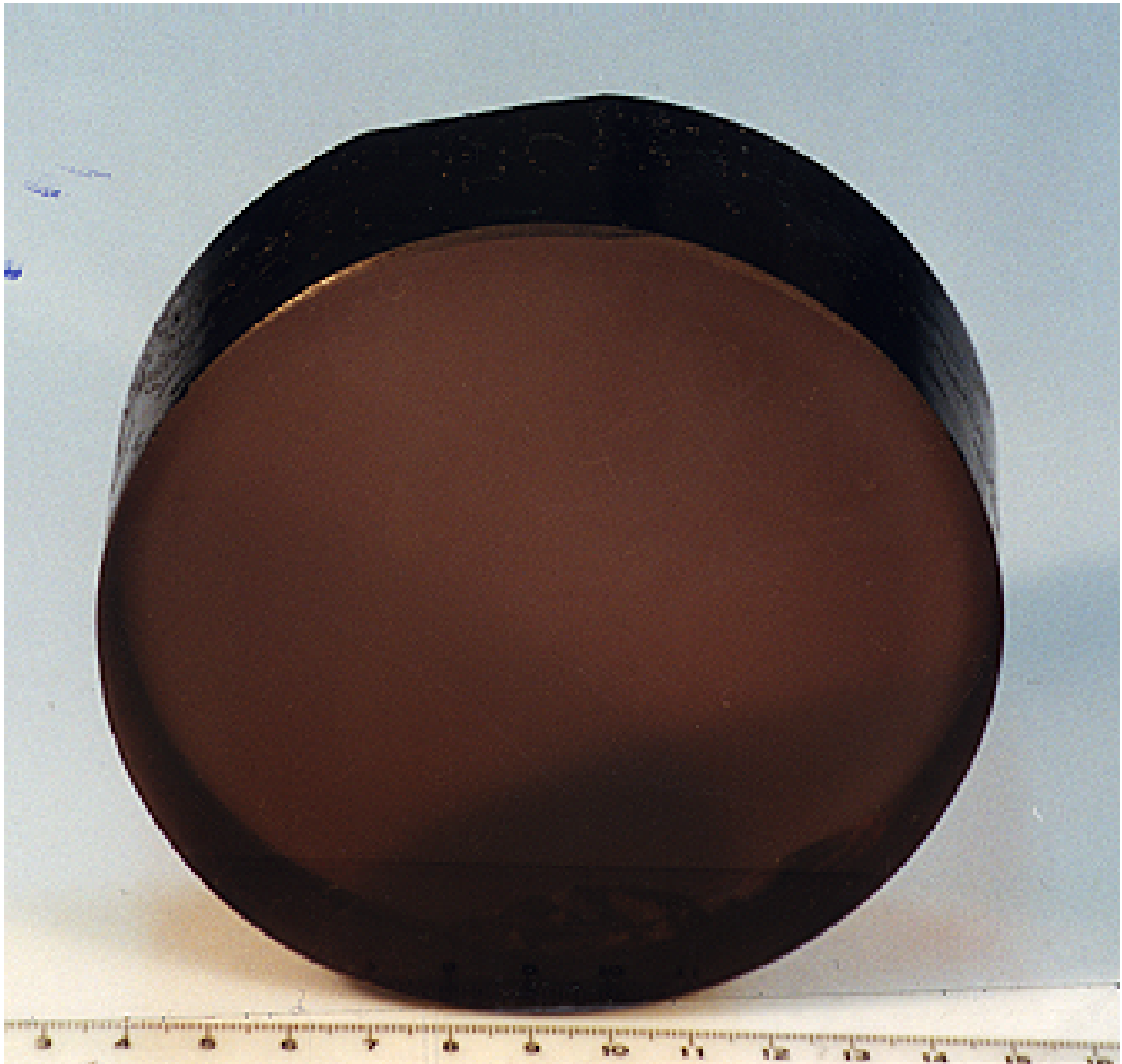


As Grown Sapphire Sample # 3



Annealed in Air Sapphire Sample # 3

ϕ 11 cm Slice, A-Axis



Annealed in H₂ Sapphire Sample # 3

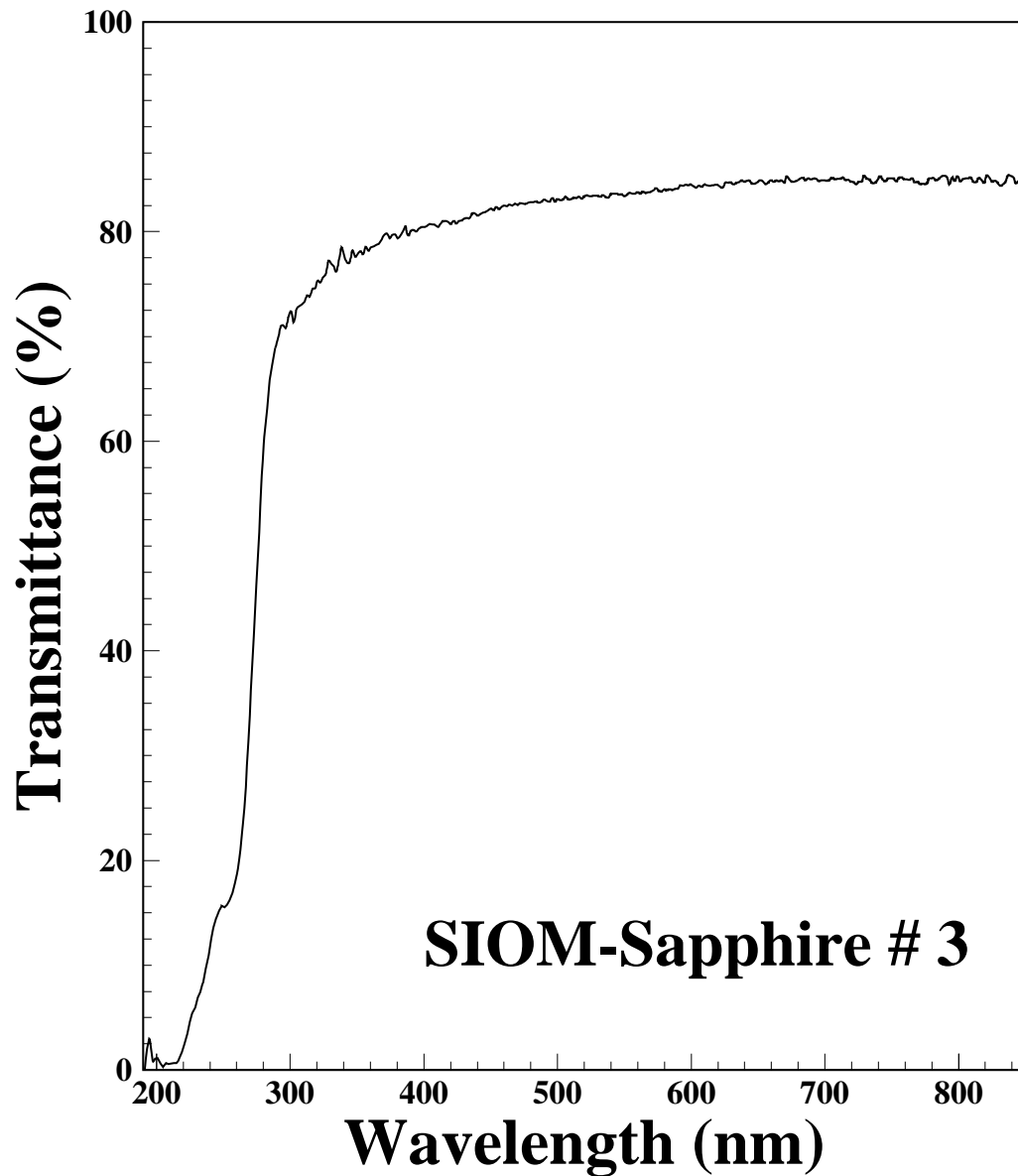
ϕ 11 cm Slice, A-Axis



Sapphire Transmission

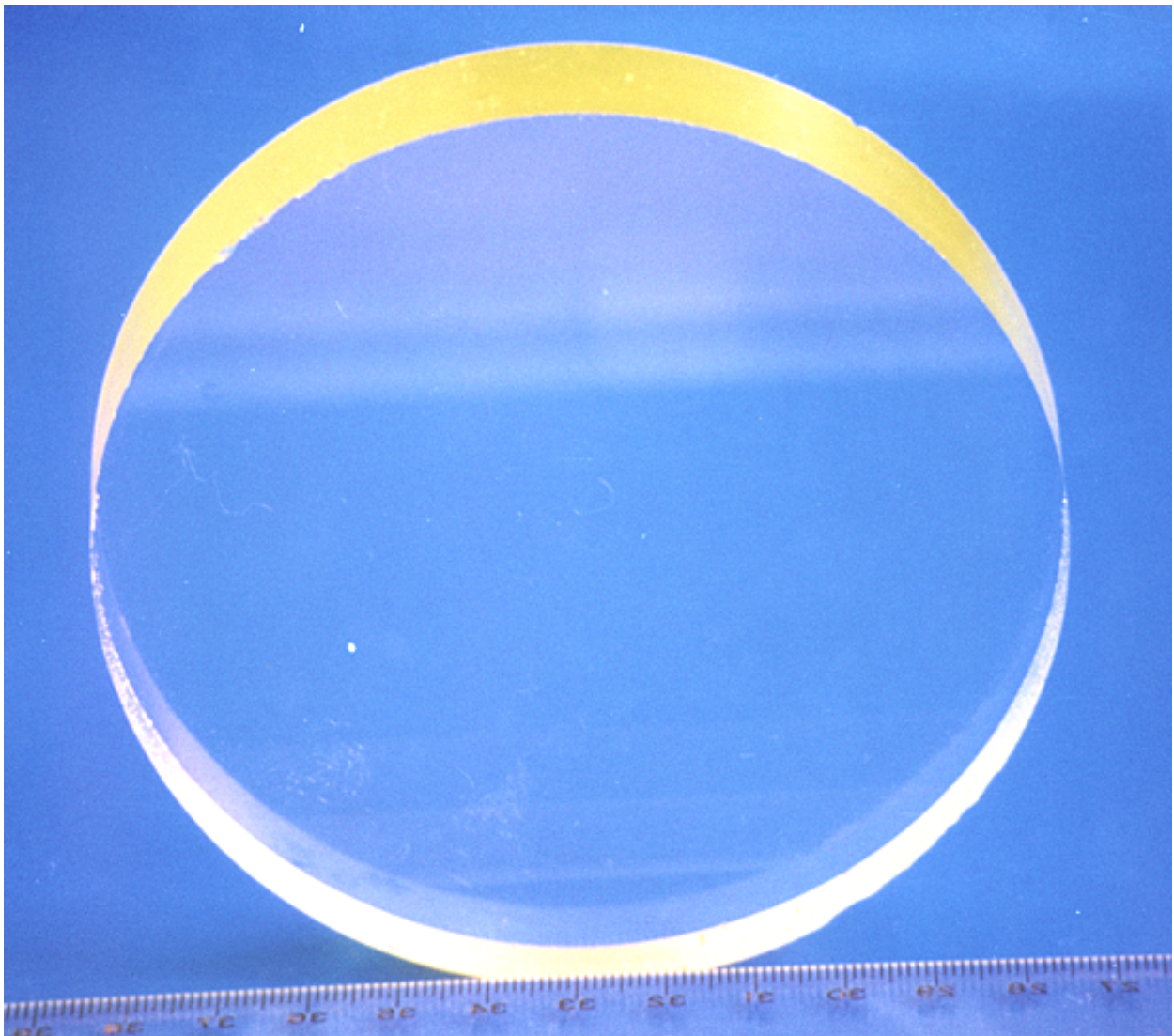
Measured with Hitachi U-3210 Photospectrometer

$\phi 11 \times 3.6$ cm SIOM-3, Annealed in H_2



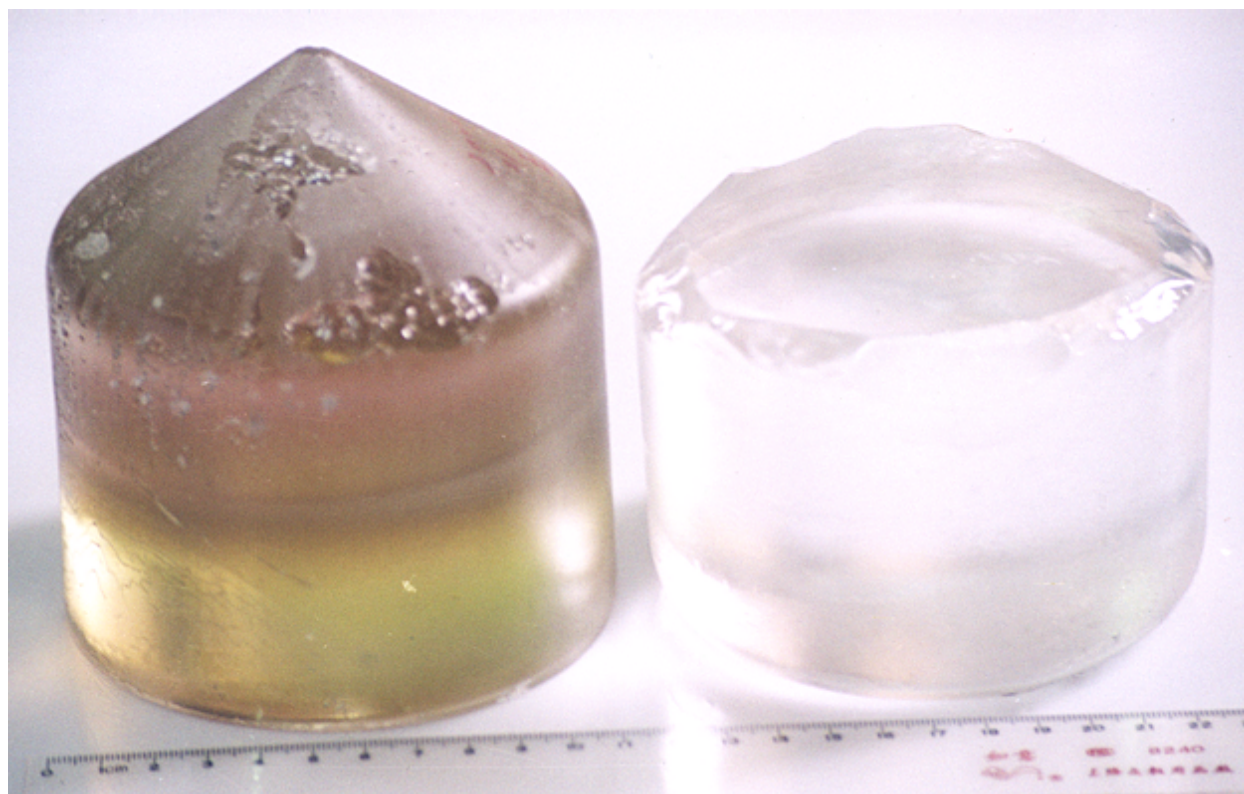
Annealed Sapphire Sample

ϕ 11 cm Slice, C-Axis



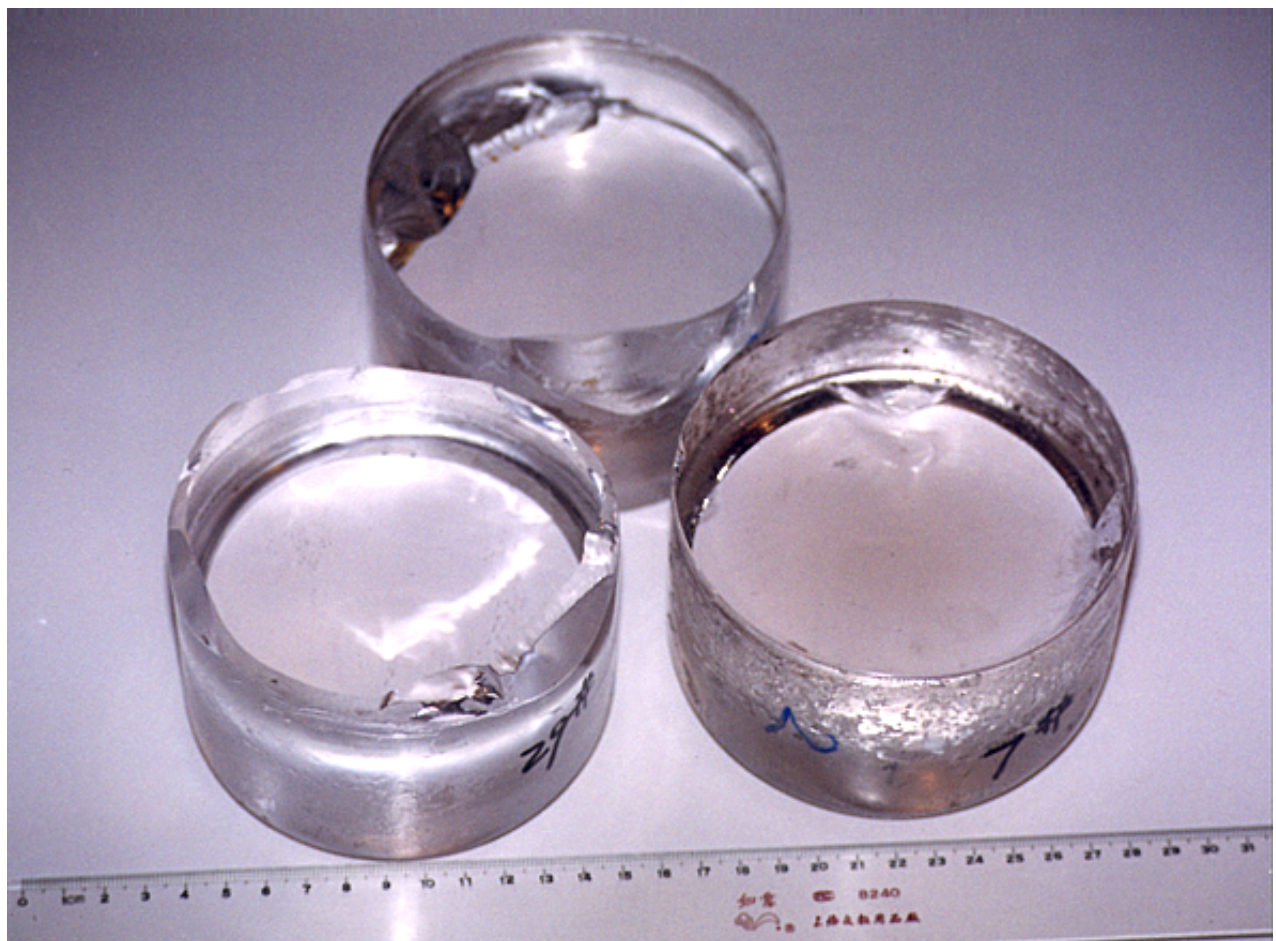
As grown & Annealed Sapphire Sample # 31

Jan 25, 2000, SIOM



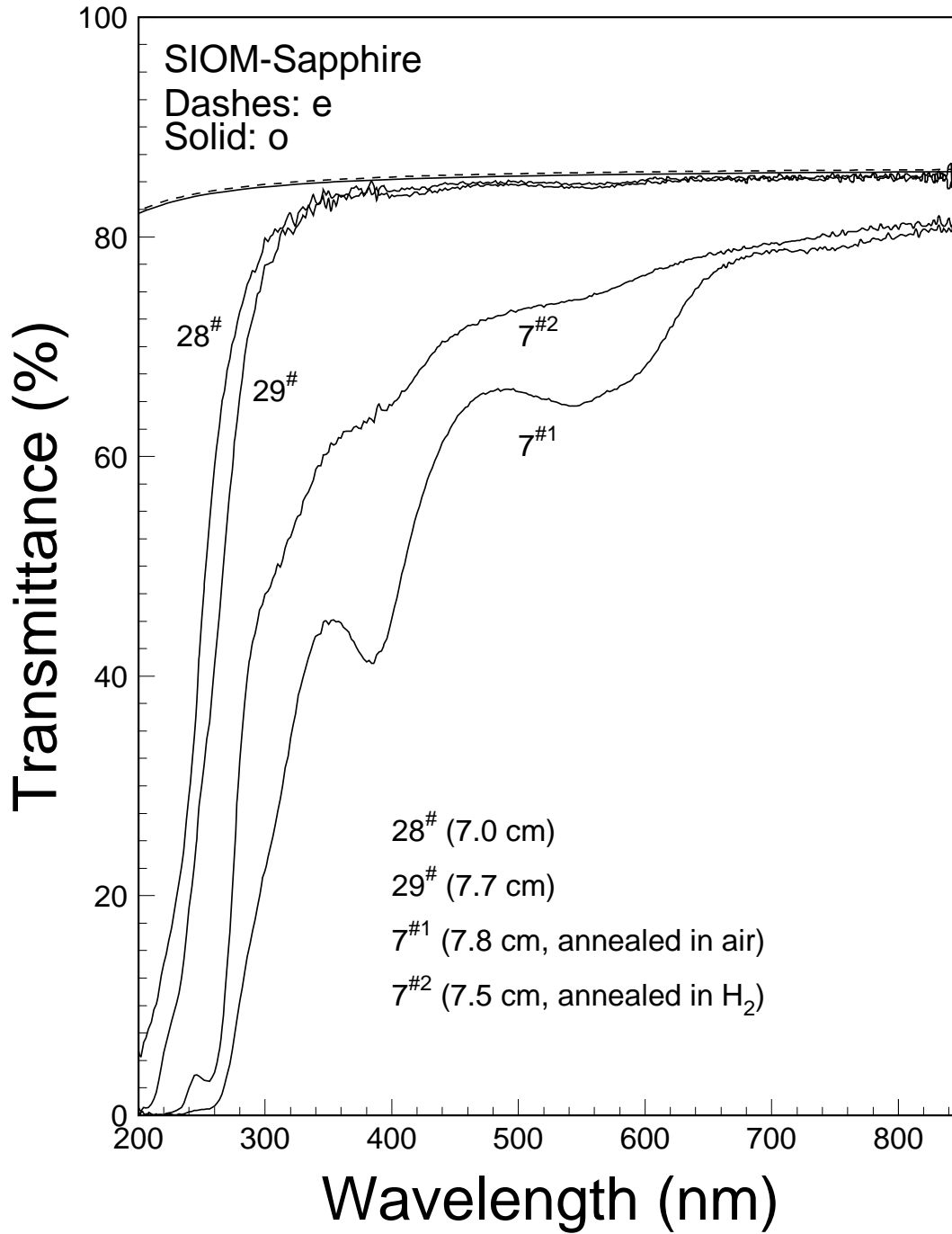
Three Phase I Sapphire Samples from SIOM

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 $R = \frac{(n-n_{air})^2}{(n+n_{air})^2}$



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.

A Brief Discussion on Phase II Program

- LIGO requires sapphire samples of diameter of 32 cm and length of 12 cm with M or A axis along the cylinder axis. The final specification for portion of crystals within the central 8 cm is
 1. Absorption of < 10 ppm/cm at $\lambda = 1.06 \mu\text{m}$;
 2. Homogeneity of $< 5 \times 10^{-7}$ at $\lambda = 1.06 \mu\text{m}$.
- The main issue of SIOM samples is 5 times worse absorption.
- The adapted approaches are
 1. Quality control of raw material.
 2. Material characterization to identify harmful impurities.
- Experience in CsI(Tl) and PbWO_4 development for high energy physics may help.

Summary

Starting July 1, 1997, SIOM has been working on Sapphire development for LIGO. Impurities in raw materials and crystals were identified, and a two step annealing technique was developed, which is effective in eliminating color centers in Sapphire crystals.

LIGO evaluated three Phase I samples in 2000, and concluded that some, sample. e.g. #28, has attractive quality, such as very low absorption. A phase II program will be carried out to see the feasibility of SIOM in providing sapphire samples required by LIGO.