Sapphire bonding

3rd Cryo-Payload Meeting
ICRR 柏の葉
2011.08.31

T. Suzuki
KEK/ICRR

Mirror suspension

Mechanical support : Enough strength
Heat path for cooling : Large thermal conductivity at cryogenic temperatures.
Low mechanical loss : Suppress thermal noise

Sapphire suspension : as a solution

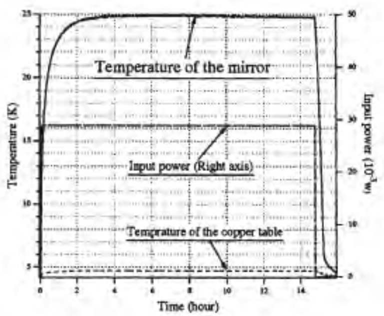


Fig. 2. The mirror temperature changes as a function of time for an input power of 29 mW.

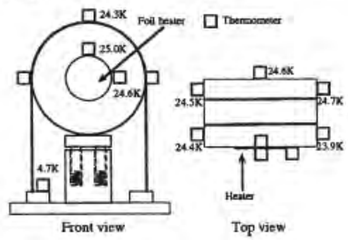


Fig. 3. Distribution of the equilibrium temperature in the case of 29 mW heat power.

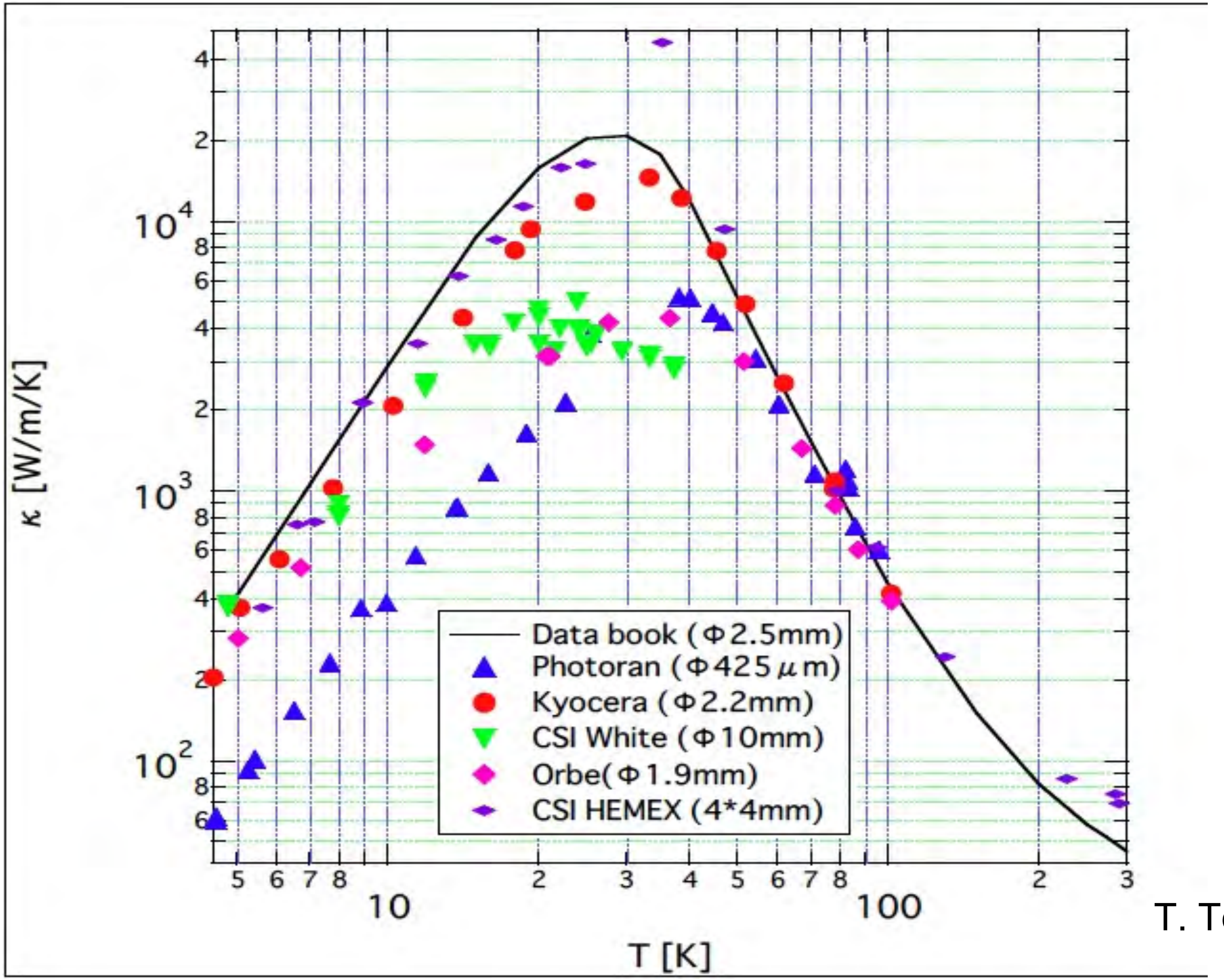
T.Uchiyama et al., PLA242(1998)211

Summary of the parameters for the estimation of Q_{pend}.

Objects	Symbol	Value
mass of the mirror	m	50[kg]
gravity acceleration	g	$9.8[m/s^2]$
length of the pendulum	l	0.25[m]
number of the fiber	n	4(2 loops)
diameter of the fiber	d	1[mm]
Young's modulus	Y	$4.7\times10^{11}[N/m^2]$
dissipation dilution ratio	η^{-1}	2.7×10^{-2}
Q-factor of the sapphire fiber	Q_{fiber}	7.7×10^{6}
Q-factor of the pendulum	Q_{pend}	2.9×10^{8}

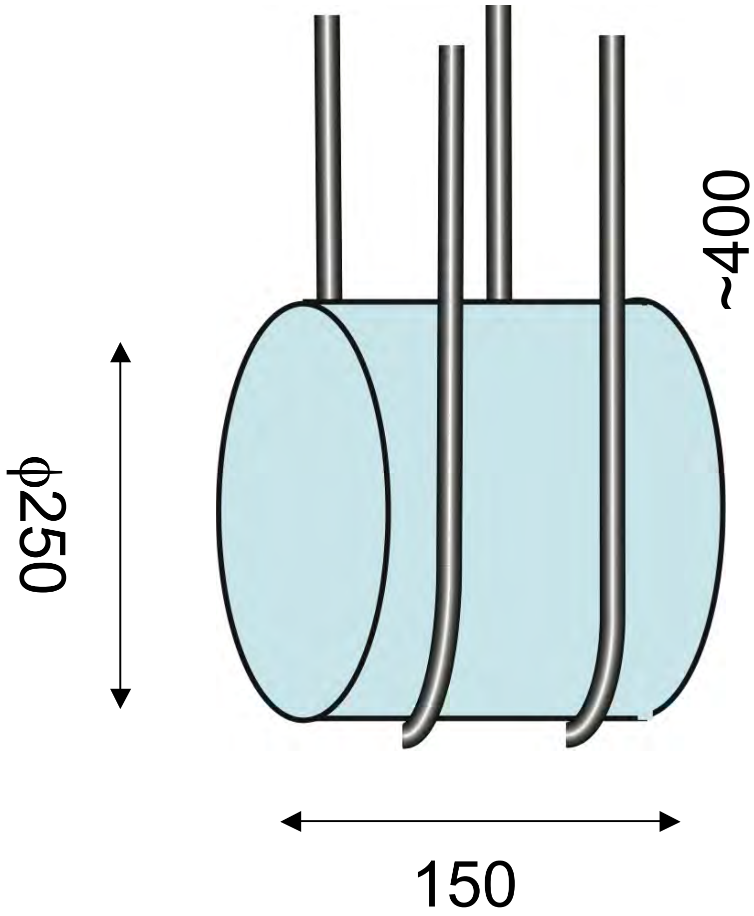
T.Uchiyama et al., PLA273(1998)310

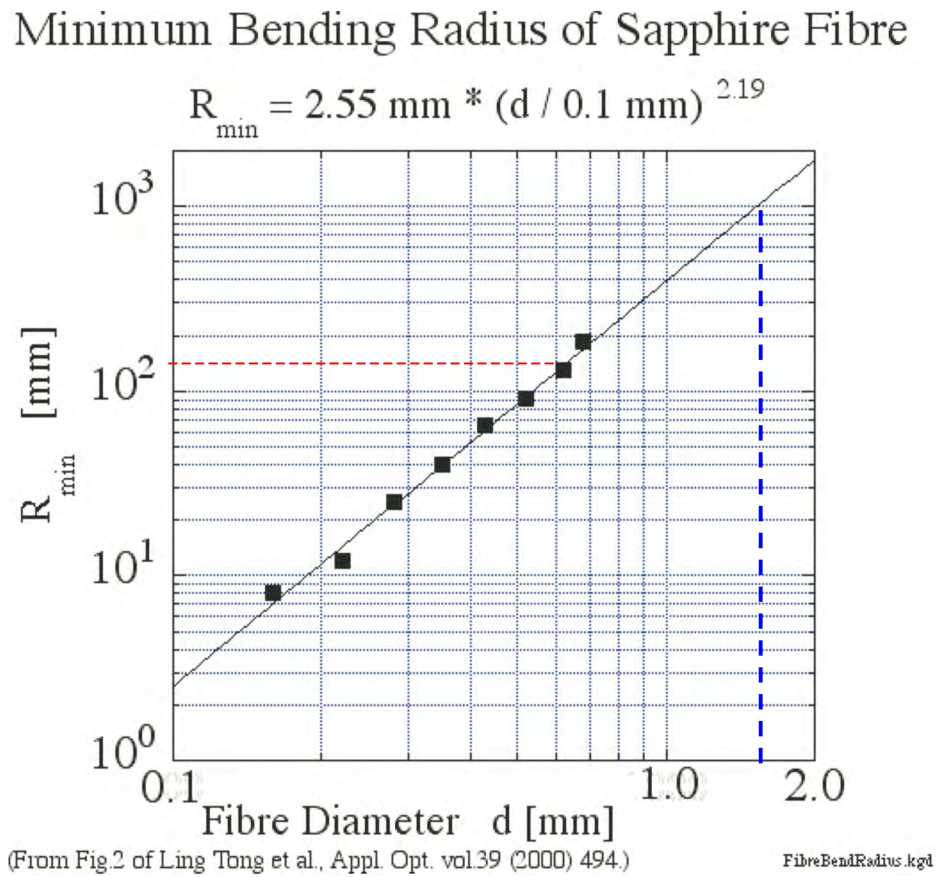
Thermal condctivity of Sapphire



T. Tomaru

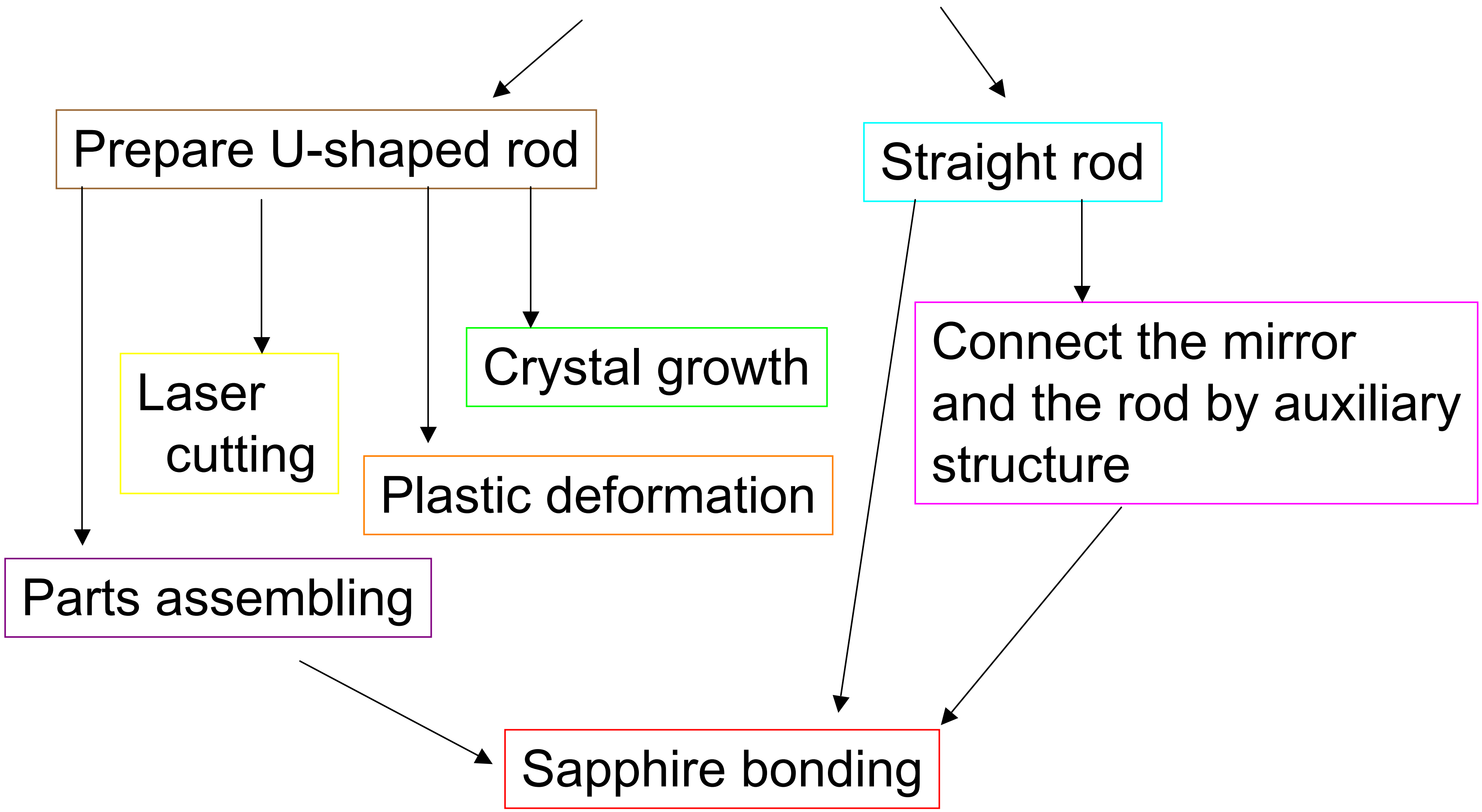
U-shape support and minimum bending radius of fiber



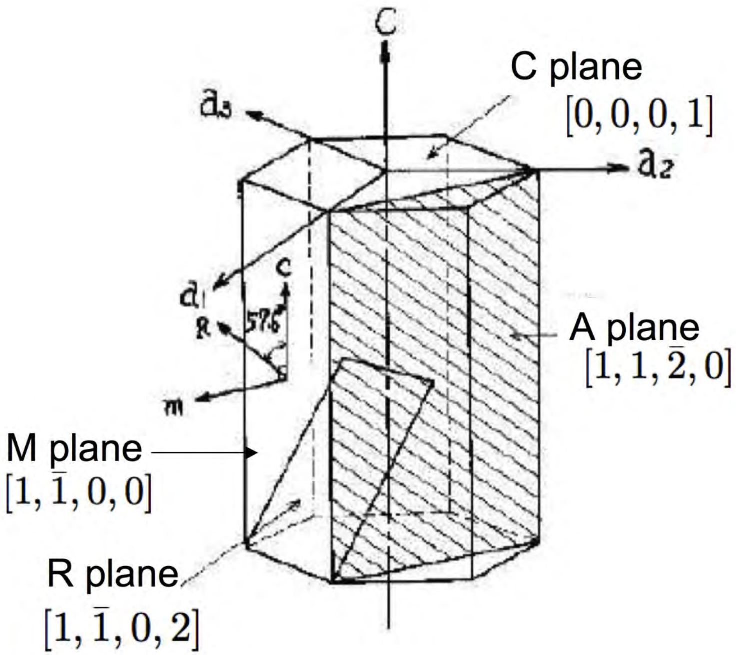


Why bonding？

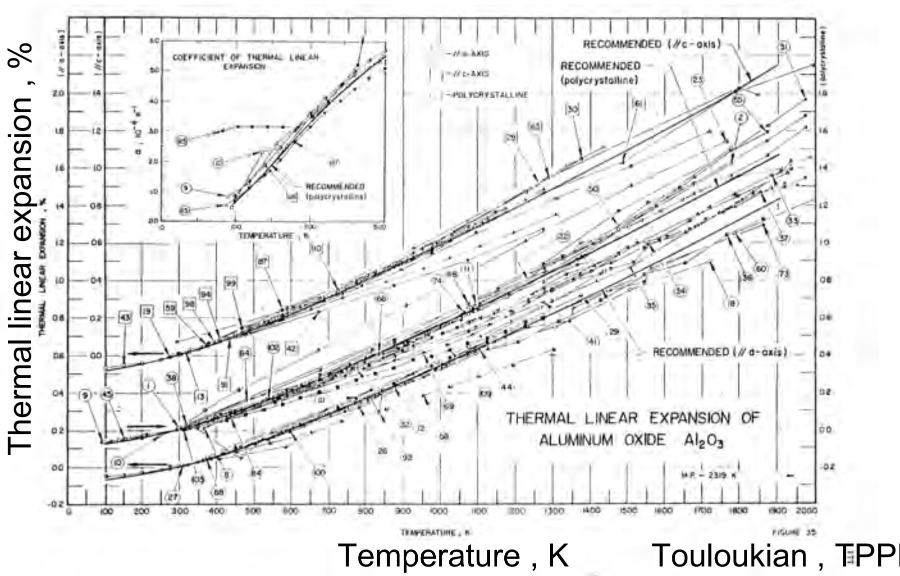
Curvature radius of R=125mm is unavailable by elastic bending for ϕ1.8mm sapphire rod without breaking.



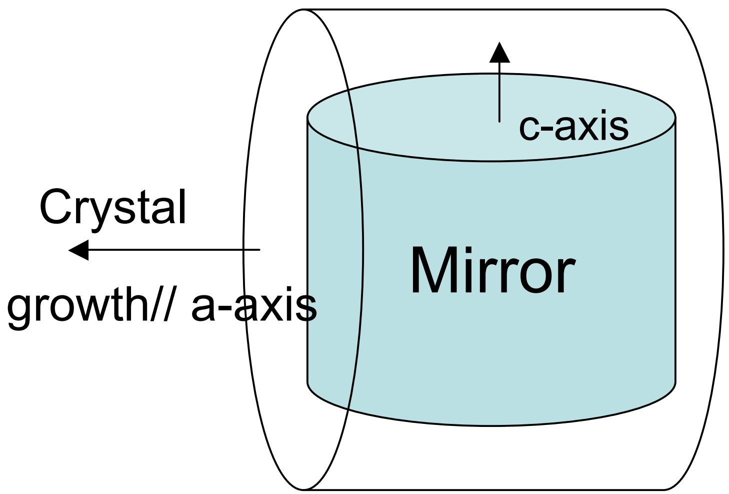
Sapphire : single crystal of α alumina



Thermal expansion



Touloukian , TPPM



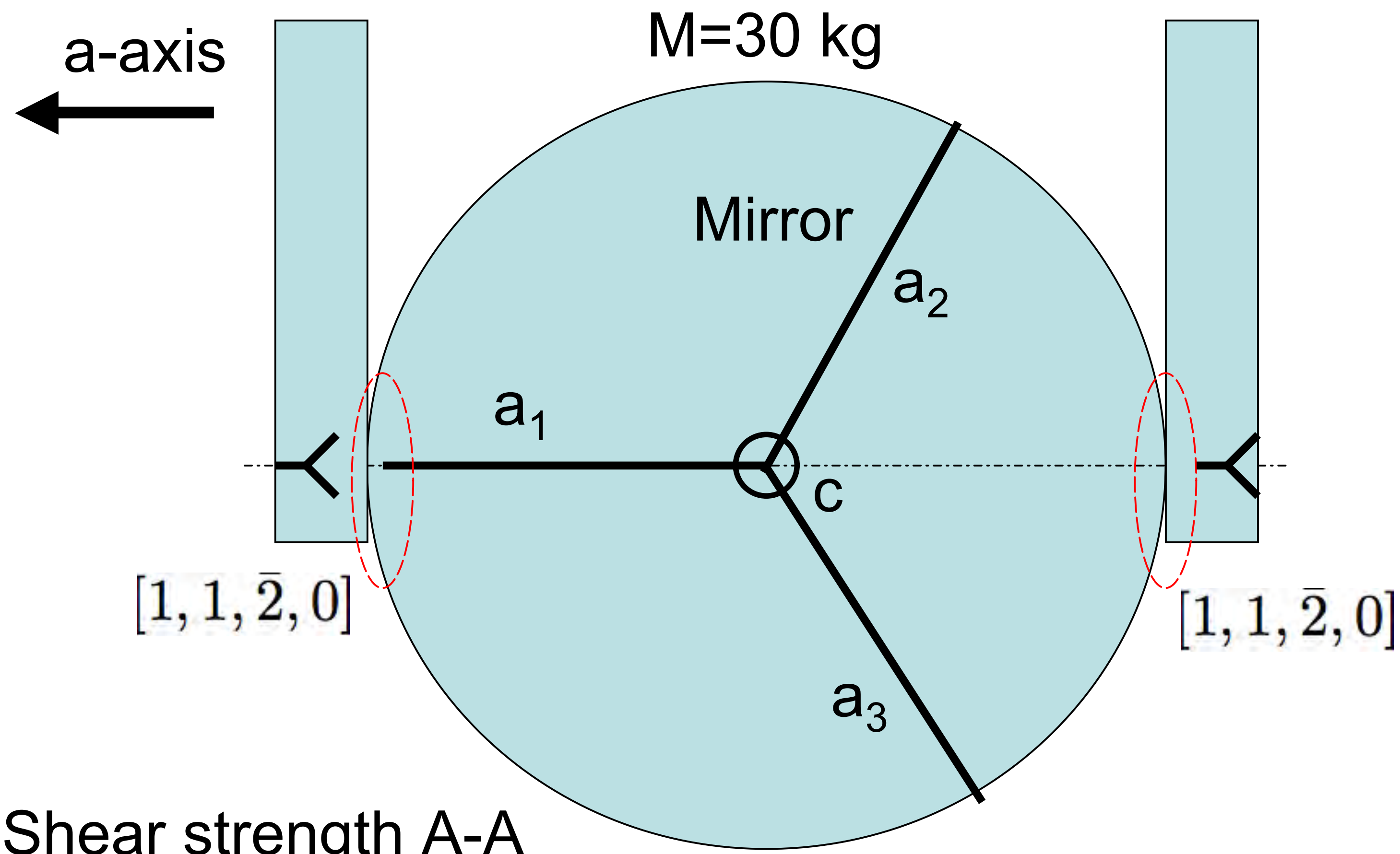
Hexagonal (Rhombohedral)
Unit cell : a=0.4755nm, c=1.2991nm
Density 3.98x10^3 kg/m^3
Melting point 2313 K

Tensile strength 280~410 Mpa

(Young's modulus 345 GPa)
(Poisson's ratio 0.29)

CSI

Candidate of bonding direction A-A



Shear strength A-A

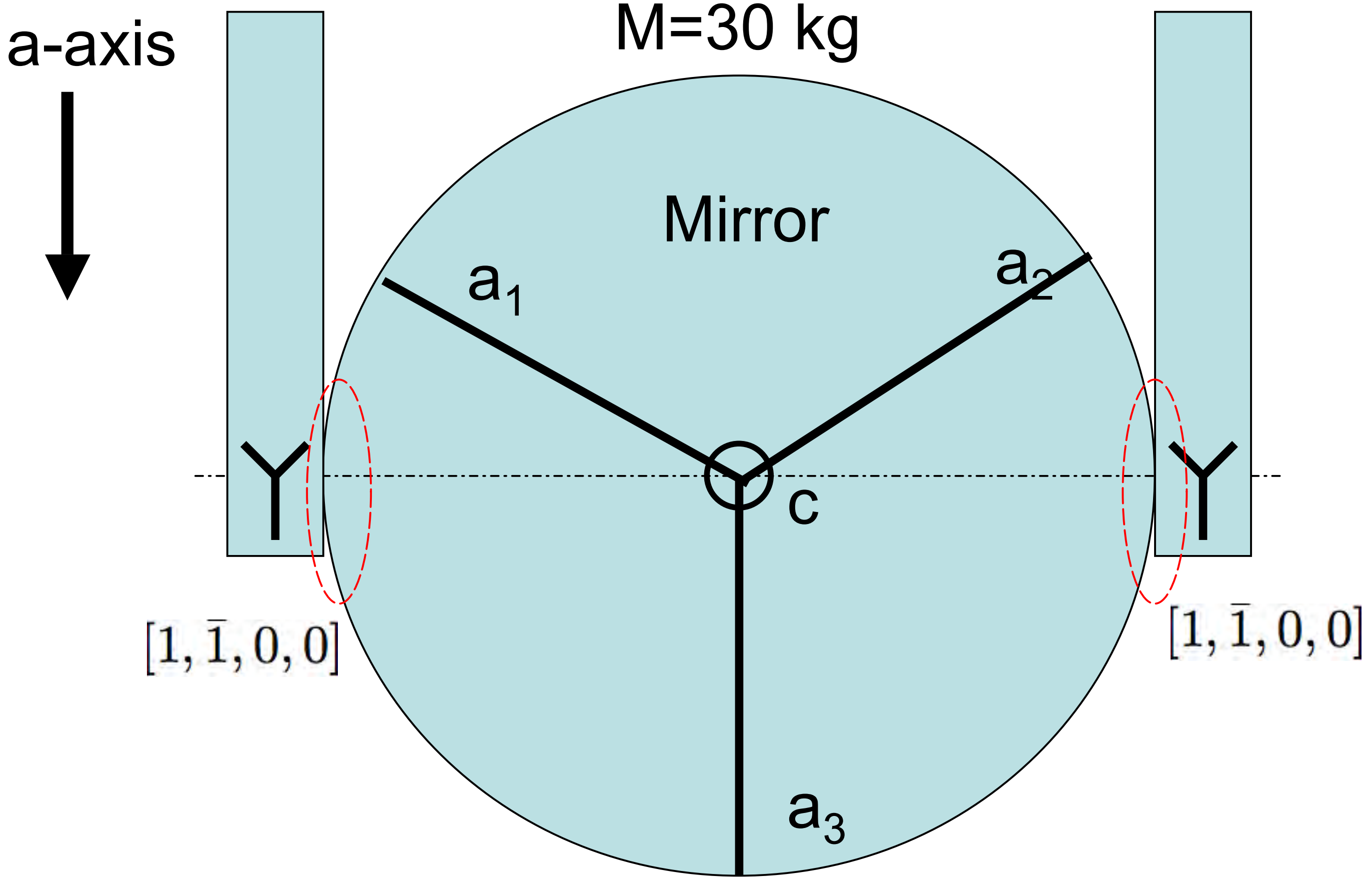
$$\tau_{bond} = \begin{cases} 28\,[MPa] \rightarrow A \approx 10\,[mm^2] \\ 7\,[MPa] \rightarrow A \approx 40\,[mm^2] \end{cases}$$

Diffusion, AFB

Hydroxy-catalysis, silicate

JPCS32(2006)309_314

Candidate of bonding direction M-M



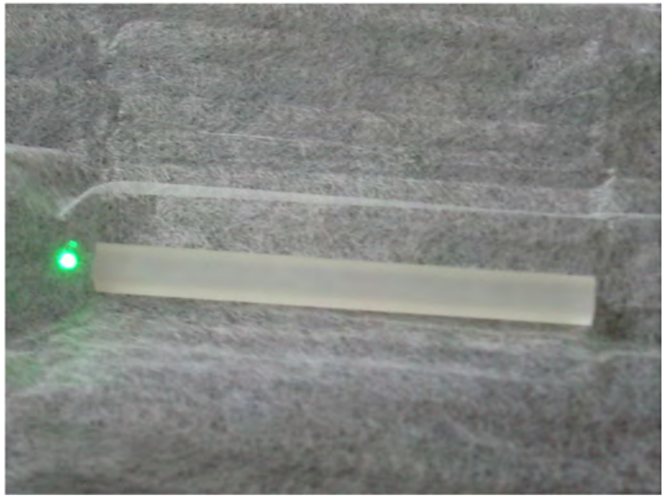
Shear strength M-M 未測定

Known methods of bonding

	Precise polish	Interposition material	Temperature treatment	Sapphire-Sapphire	Thermal conductance	Mechanical loss
AFB, Diffusion	Necessary	none	1300~1400 ℃	Almost same as bulk ~ 28 MPa	~ 4 $W/K/mm^2$	Not yet measured
Direct(1), SAB1 (~ 2000)	Necessary	None (Ar^+ beam)	300 K	-	-	-
Direct(1), SAB2 (2011)	Necessary	Fe, etc (Ar^+ beam)	300 K	Not yet measured	Not yet measured	Not yet measured
Hyroxy-catalysis, silicate	Necessary	KOH, Na_2SiO_3, H_2O	300 K	~ 7 MPa	~ 0.3 $W/K/mm^2$	Not yet measured
Metalize, soldering	(Not required)	Active metal	< 1000 ℃?	Not yet measured	Not yet measured	Not yet measured
Adhesive	Not required	Al_2O_3, $AlPO_4$, H_2O	~ 500 ℃	~20 MPa	Not yet measured	Not yet measured

AFB: Adhesion Free Bonding
SAB: Furface activation Bonding

Bonding



Example of diffusion bonded rod.
Boundary is at the centre.

- Diffusion bonding, AFB: Optical contact + Heat treatment. T=1300℃~1400℃. Strength of bonding is almost sama as that of the bulk.（Onyx Optics、Japan Cell）
- Direct bonding(1), ASB：Clean the surface by Ar ion beam in ultra high vacuum and press to contact. Sapphire-sapphire bonding was difficult. Recently the bonding was done by a nano-scale interpolation layer of metal.
- Direct Bonding(2):After making hydrophilic layer on the surface, contact surface dehydrate by heat treatment.（Appl.Opt. 37(1998)2407）
- Hydroxy-catalysis bonding, Silicate bonding: Put small amount of KOH or Na_2SiO_3 solution. Contact and mature at room temperature. (Phy. Lett. A246 (1998) 471) Strength and thermal resistance are worse than solid bond.(Int. J. Mod. Phys. A20 (2005) 7060)

Need precise polishing

Table 1. Samples considered for the breaking strength analysis. For each combination, orientation and flatness, the number of successful bonded samples over the number of total available samples has been reported, respectively waiting 1, 2 or 3 weeks before the breaking strength test. The diameter of all samples is 5 mm.

			Number of successfully bonded samples		
Bond combination	Flatness	Orientation	First week	Second week	Third week
Si–Si	$\lambda/10$	Random–random	5/7	5/7	3/8
Si–Si	$\lambda/7$	Random–random	0/0	4/5	5/5
Si–Si	$\lambda/4$	Random–random	0/0	2/5	3/5
Si–Si	$\lambda/10$	100–100	5/5	5/5	5/5
Si–Si	$\lambda/7$	100–100	5/5	5/5	5/5
Si–Si	$\lambda/4$	100–100	4/5	4/5	3/5
Si–Si	$\lambda/10$	111–111	5/5	5/5	5/5
Si–Si	$\lambda/7$	111–111	5/5	5/5	5/5
Si–Si	$\lambda/4$	111–111	5/5	5/5	2/5
Si–Al_2O_3	$\lambda/10$	Random–random	3/5	4/5	4/5
Si–Al_2O_3	$\lambda/7$	Random–random	5/5	5/5	5/5
Si–Al_2O_3	$\lambda/4$	Random–random	5/5	5/5	4/5
Si–Al_2O_3	$\lambda/10$	100–C	5/5	5/5	5/5
Si–Al_2O_3	$\lambda/7$	100–C	5/5	5/5	5/5
Si–Al_2O_3	$\lambda/4$	100–C	5/5	5/5	5/5
Al_2O_3–Al_2O_3	$\lambda/10$	Random–random	4/5	2/5	5/5
Al_2O_3–Al_2O_3	$\lambda/7$	Random–random	4/5	4/5	2/5
Al_2O_3–Al_2O_3	$\lambda/4$	Random-random	0/5	5/5	5/5
Al_2O_3–Al_2O_3	$\lambda/10$	C–C	4/5	4/5	4/5
Al_2O_3–Al_2O_3	$\lambda/7$	C–C	0/5	2/5	3/5
Al_2O_3–Al_2O_3	$\lambda/4$	C–C	3/5	3/5	4/5

東京大学 大学院工学系研究科
School of Engineering The University of Tokyo

平成 23 年 5 月 30 日

常温接合の新手法

１．発表者：東京大学大学院工学系研究科精密工学専攻　教授　須賀唯知

２．発表概要：

東京大学大学院工学系研究科精密機械工学専攻・須賀唯知教授、近藤龍一（博士課程；現・太陽誘電株式会社）らの研究グループは、このたび新たに提案した常温接合の新手法を発表します。この方法により、従来困難であった熱酸化膜やガラス、サファイア、圧電単結晶などの無機材料ウエハの接合を常温で実現しました。

３．発表内容：

提案する手法は、具体的には、接合面に、鉄ナノ密着層とシリコン薄膜中間層を組み合わせた表面活性化を行い、常温の接触のみで接合を行う方法です。本手法の実現により、半導体デバイスの３次元積層、MEMS デバイスとの融合、フォトニクスデバイスの開発等が飛躍的に進展することが期待されます。

本手法の開発は、同教授と太陽誘電株式会社との共同研究の成果をもとに、ランテクニカルサービス株式会社、ボンドテック株式会社、および産学連携コンソーシアムである電子実装工学研究所の協力のもとに行われたものです。この成果は、実装における最大の国際会議、第 61 回 ECTC 2011（2011 年 5 月 31 日～6 月 3 日米国フロリダ）にて発表される予定です。

25.4 表面活性化による常温接合

以上の手法では接合面の活性化を機械的な手法に委ねているのに対し，これをよりはっきりと規定され，制御された表面，雰囲気で行なおうとするのがわれわれの提唱する「表面活性を利用した常温接合法」である[10]．この方法は，超高真空中で見られる異種物質間の凝着現象を接合に積極的に利用しようとするものであり，超高真空のようなクリーンな雰囲気でのイオン衝撃により，接合面の清浄化・活性化を実現する．

宇宙空間のような清浄な真空中では，固体表面の活性が非常に高く保たれることが知られている．超高真空中での摩耗・凝着現象はその代表的な例である．機械的な摩擦によって固体表面の酸化皮膜，吸着物質などが除去されて活性な面が露出し，そのまま強固な結合を形成することが，超高真空中で摩耗・凝着が加速される原因と考えられている．この凝着現象は，宇宙空間において使用される機器，器具への異物質の付着，あるいは摺動部材料の摩耗など，宇宙基地利用において直面する基本的な問題であり，1960 年代後半以降 NASA やソ連の科学者によって精力的に研究されてきた．無論，凝着・摩耗はネガティブな側面が強く，研究の重点は，凝着しにくい素材の開発，固体潤滑剤の開発などにおかれていた．冷間圧接という観点から凝着現象を調べたものとしては西ドイツの Ruge のグループ[11] が最初であろう．しかしながら，彼らの実験は，超高真空中で破断させた試料の破面を接合面とするものであり，積極的な接合技術としての意味は薄かった．

これに対し，接合面の活性化を摩擦などの機械的手法によらず超高真空中でのイオン衝撃によって行なう接合手法は，異種物質の接合を対象を限定することなく実現することも可能と考えられ，また，前節の接合法とも異なって接合面の温度上昇も一切なく接合も瞬時に終了するというような際だった特徴を示している．これまでにわれわれの研究グループでアルミニウム-アルミニウム単結晶について詳細な検討を行ない[12]，現在対象をメタル-セラミックスに広げて検討を進めている．また，ほかにも国内数カ所の研究機関で同様の試みが精力的になされている[7,13,14]．

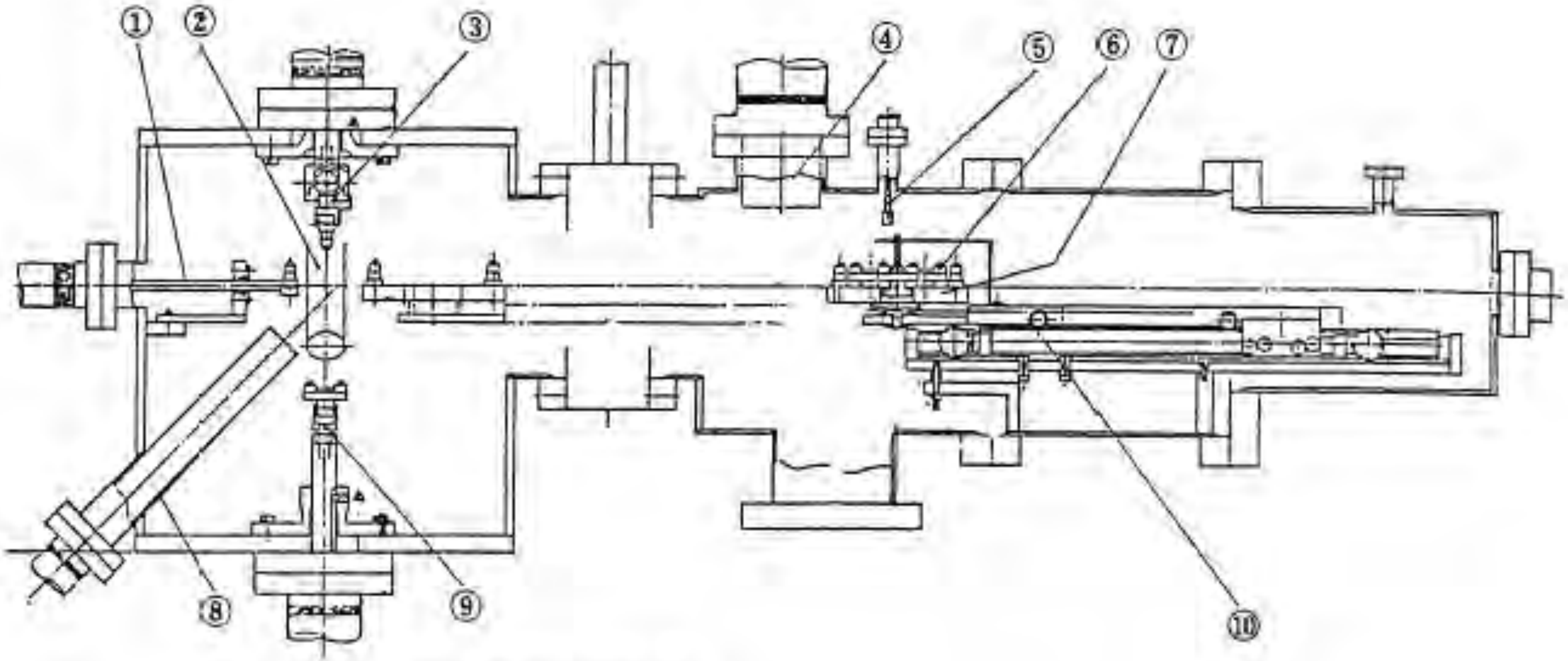
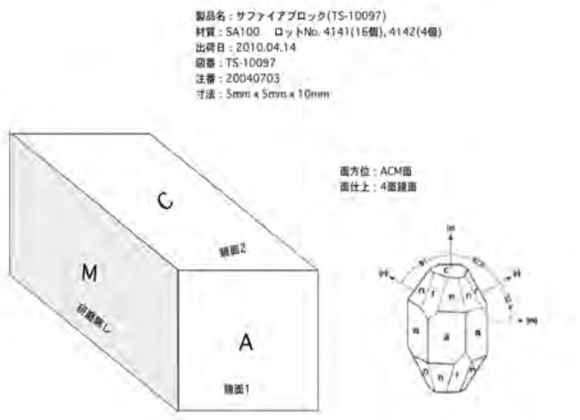


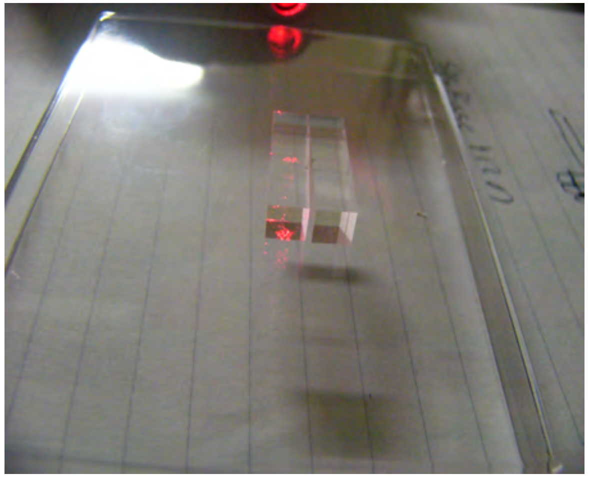
図 25.4　超高真空圧接装置.

①マニピュレーター　②アトムソース 1　③上部圧接軸
④マグネトロン型スパッタ蒸着装置　⑤回転導入端子
⑥試料台　⑦回転台　⑧アトムソース 2　⑨下部圧接軸
⑩ 2 段スライド機構

Bonding test



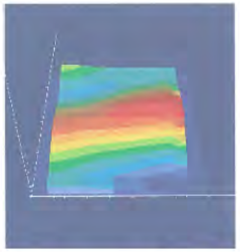
Bonding test



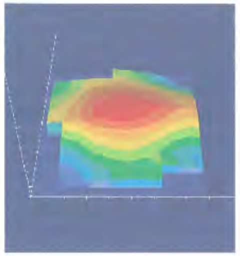
Diffusion bonded samples

FUJINON

6A



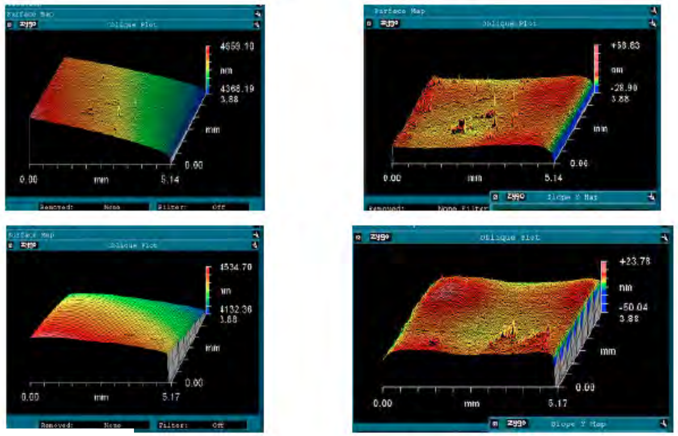
6B



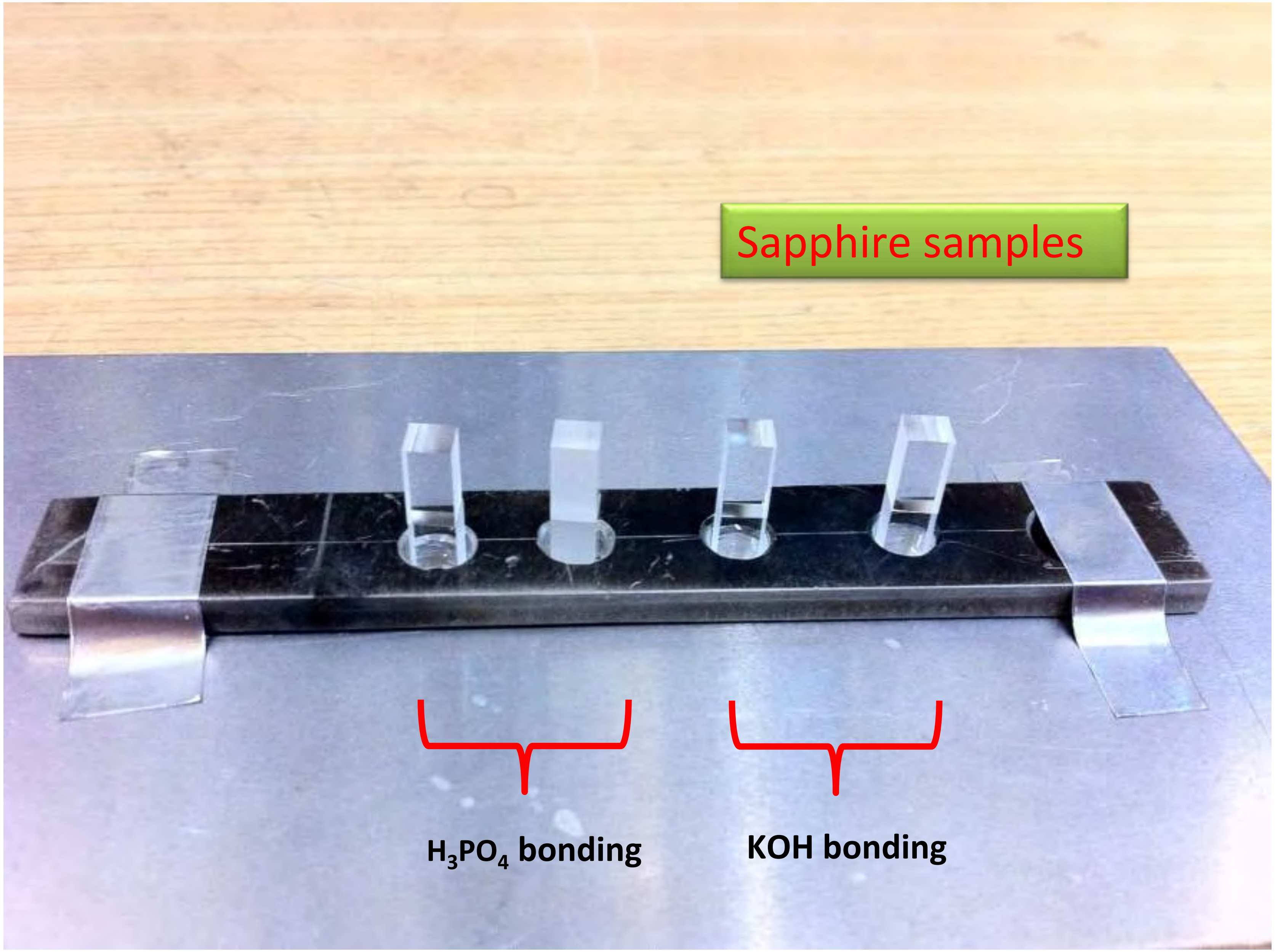
- 測定波長　λ=674.4nm
- 傾き補正：ON　解析点数：378(6A), 310(6B)
- 6A全面PV　86.3 [nm]、6B全面PV　72.2 [nm]
- 6A全面rms 17.5 [nm]、6B全面rms 14.2 [nm]

Zygo New View

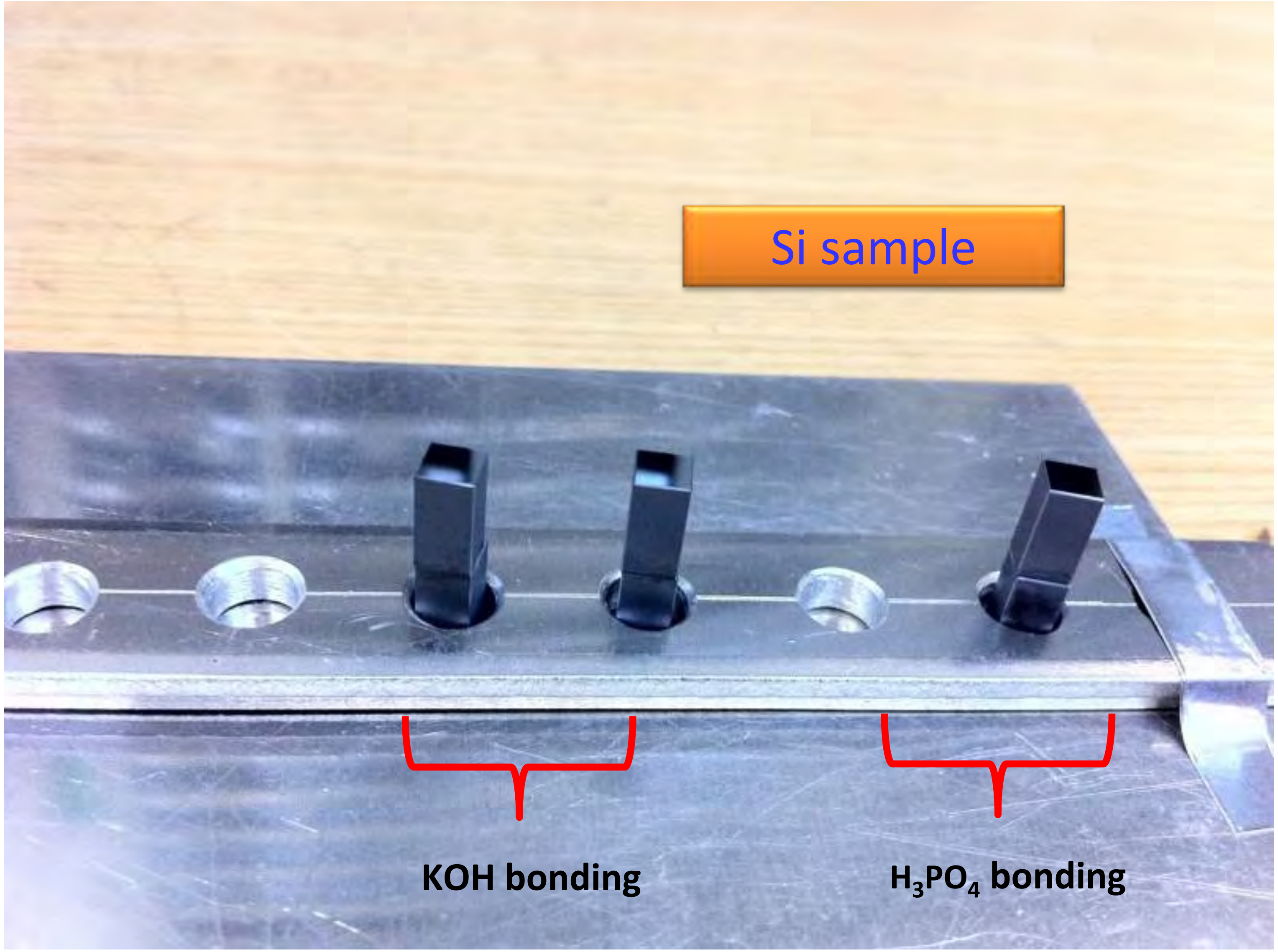
Shape(top)　　Roughness(top)



Shape(bottom)　　Roughness(bottom)



Sapphire block：5×5×10 (mm^3) 5x5 A-plane, polished by CeO, roughness~20nm



Si block : 5×5×10 (mm^3)

Direct bonding (2) , chemical + heat

A. Sugiyama et al., Direct bonding of Ti:sapphire laser crystals, Appl. Opt. 37(1998) 2047

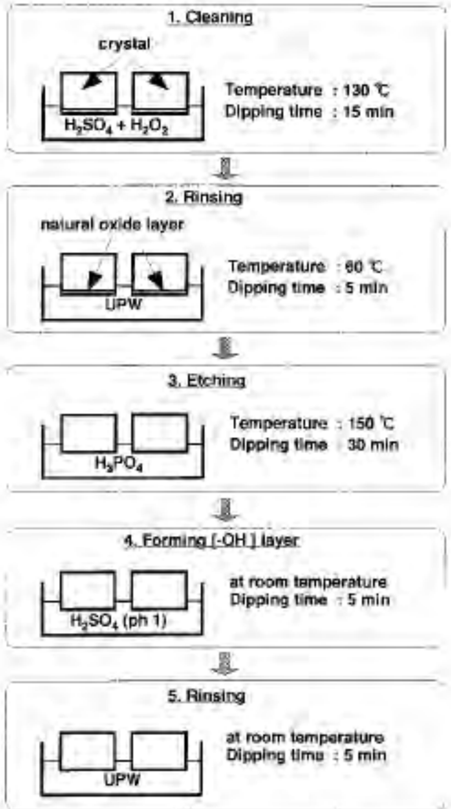
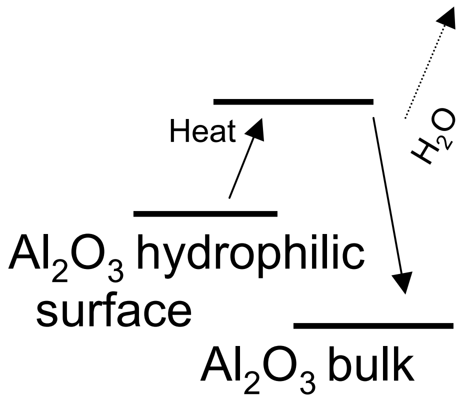


Fig. 1. Procedure for chemical treatments to clean and form an (-OH) layer on bonding surfaces.



$$\lambda \simeq \frac{hc}{k_B T} \qquad ?$$

T≈1100℃ —> λ ≈13 μm

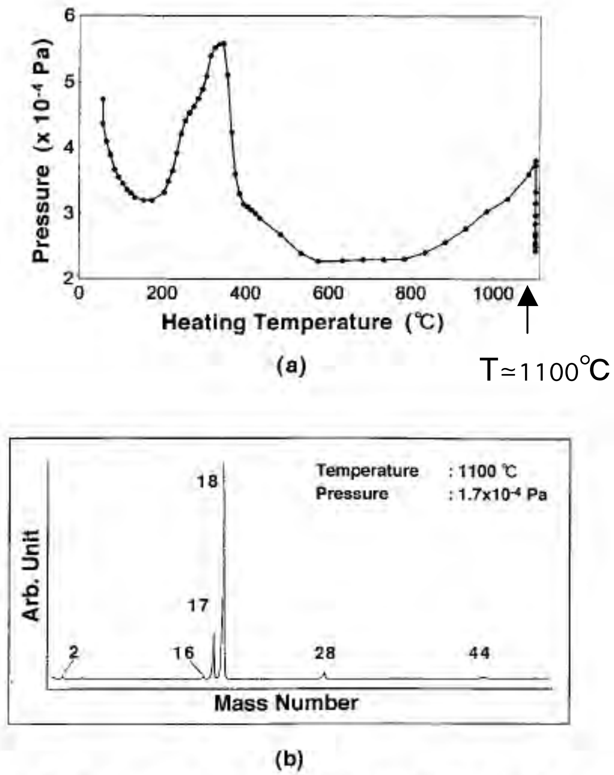


Fig. 2. (a) Pressure variation of the inside furnace during the second heat treatment. For this measurement, the graphite vice was preheated under vacuum. (b) Results of partial pressure measurements by the quadrupole mass filter (ULVAC, MSQ-150A). Each number means the mass number of signals.

Precise polishing, making hydrophilic layer,
Dehyrdoation by heat treatment

Same strength to bulk
Optical homogeniety

Shape controlled crystal growth

Edge-defined Film-fed Growth

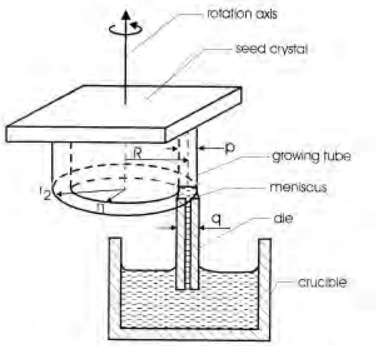
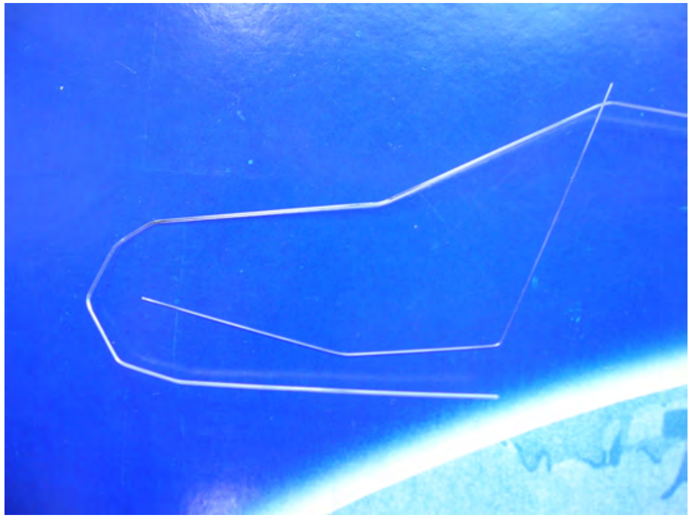


Fig. 2. The scheme of the local shaping technique.

- Forming a shape by crystal growth.
- Plate（Kyocera)、Rod (Photoran）
- Roughness~0.5mm remains for as grown surface.
- It is not certain that U-shape suspension with ϕ 1.8mm and L=300 mm.

j. Crystal Growth 198/199(1999) 201

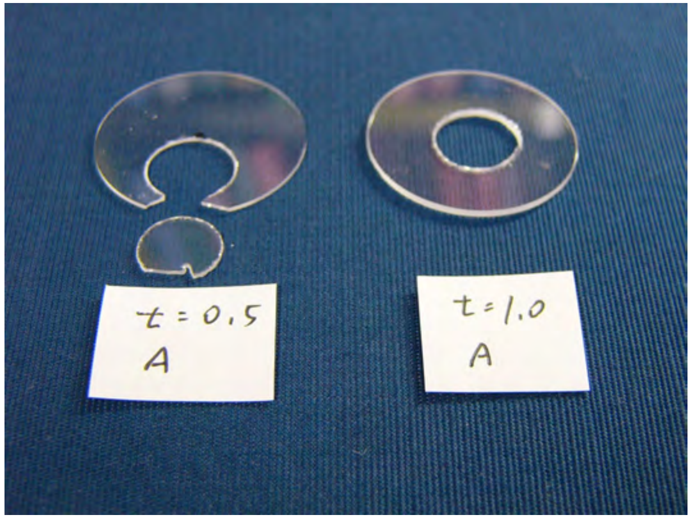
Plastic deformation

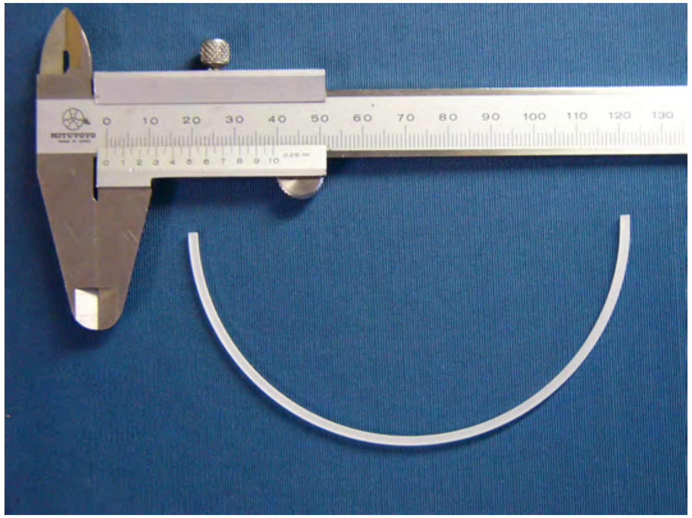


- Heating by CO_2 LASER
- Thin（<ϕ0.8mm）rod can be deformed using 80W class CO_2 laser.
- More powerful laser is necessary for bending ϕ1.8mm rod.
- It is difficult to control thermal stress for using kW-class laser. Also it is hard to stop local melting.

Appl. Opt. 39(2000)404

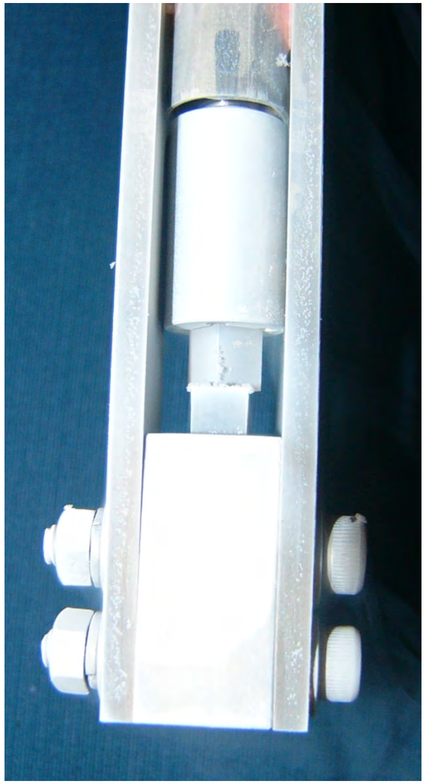
Cutting



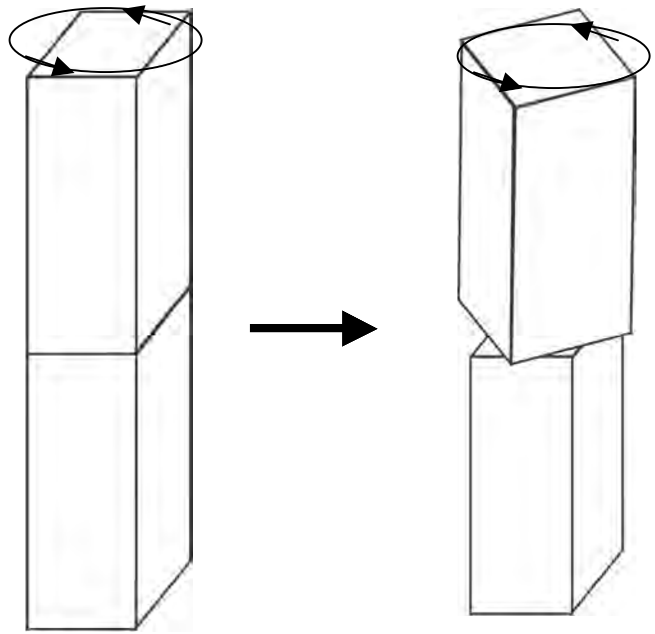


- Laser cutting causes chipping.（Laser cutting works well for alumina ceramics.）
- Mechanical cutting（wire＋diamond powder）seem to be a promising. Shown is a result of mechanical cut from the plate of 2mm thick. Width=2mm, R=50mm.
- Cross sectional area: square, surface : no polish.

Shear strength measurement



Shear strength measurement



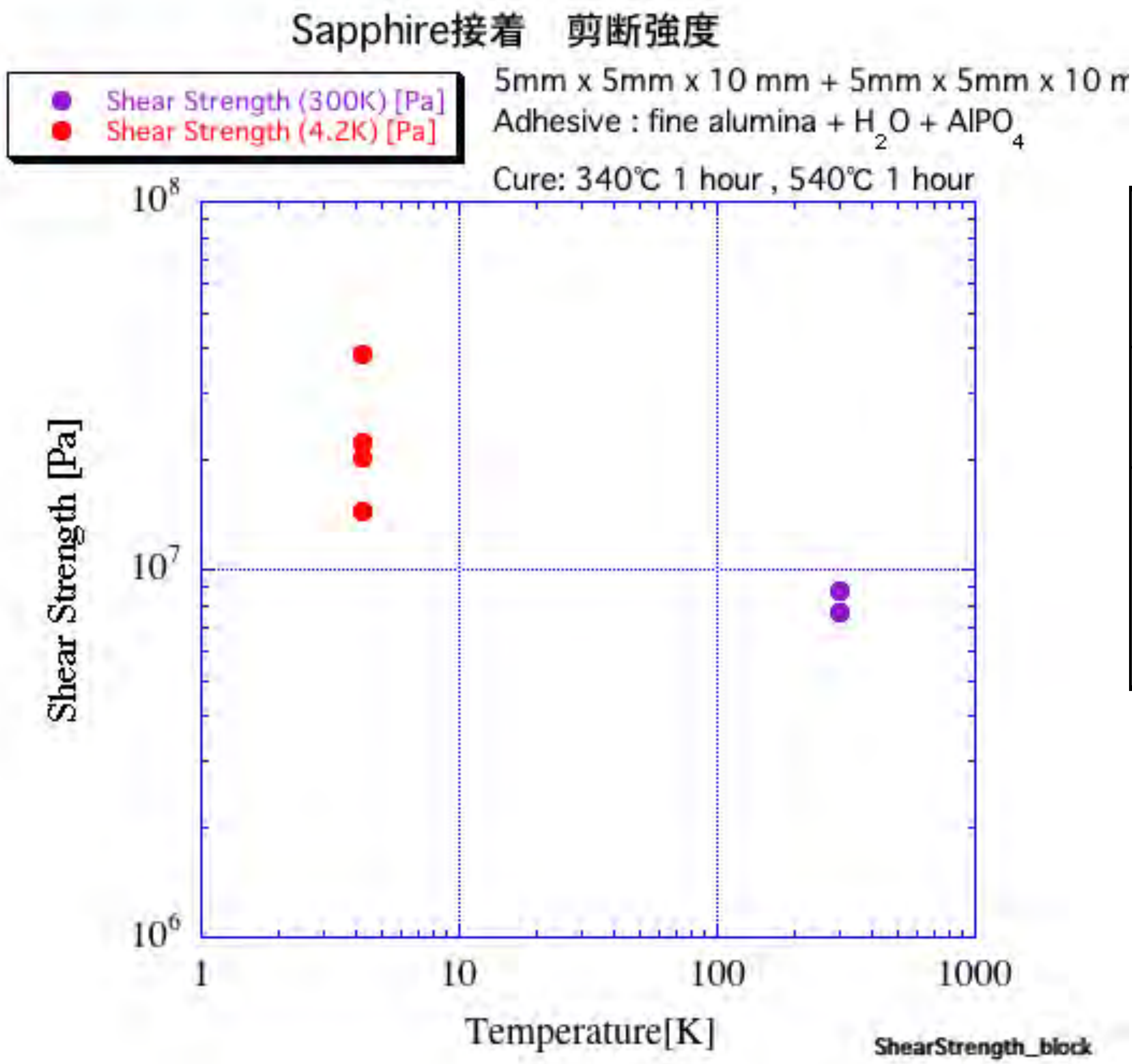
Sapphire block
5mm x 5mm x 10mm

5mm x 5mm -> Bond

Torque meter   peak hold

T=300K , T=4.2K

Shear strength of glued sapphire at 300K and 4.2K



	Shear Strength [MPa]	
T=300K	8.80 7.63	
T=4.2K	38.3 22.2 22.1 20.2	

Issues

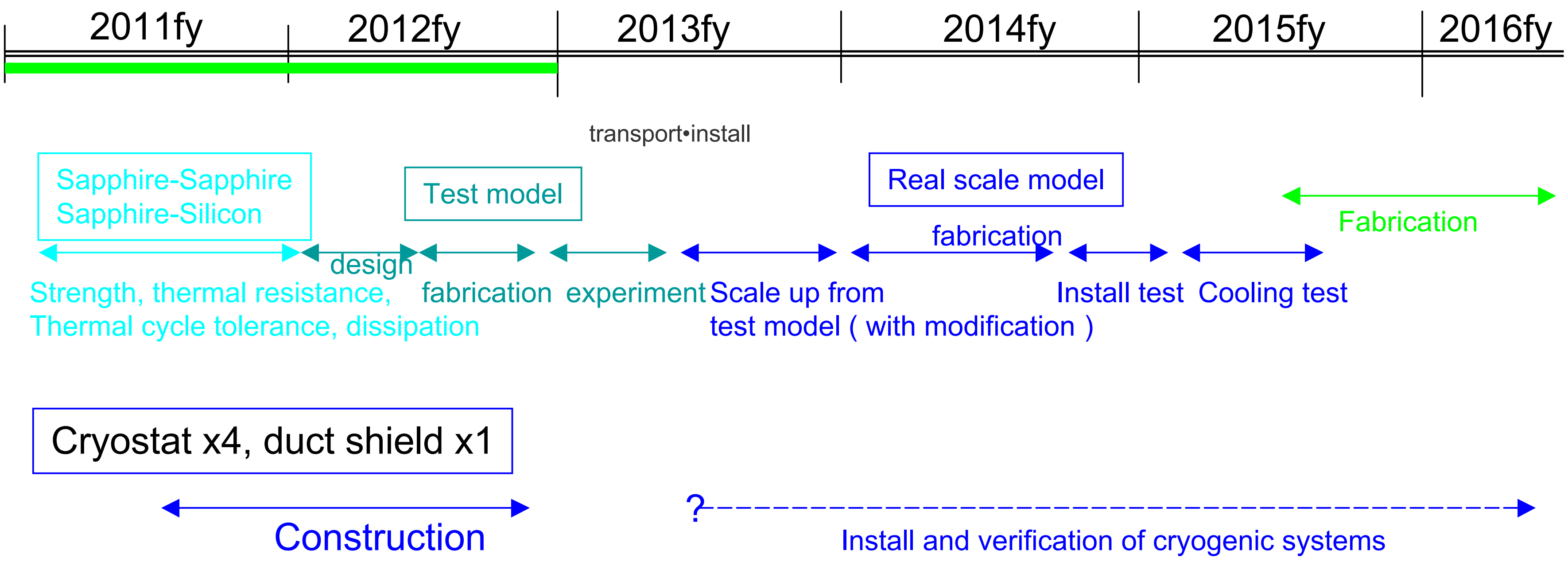
Selection of boding method

- Polishing technique on M-plane
- Confirm bonding strength / thermal stress cycle
- Measure parameters for designing
- Quality control
- Availability of material
- Altenative method

Find a consistent process to construct mirror-suspension system

- Design whole suspension system
- Heat treatment
- Polishing mirror
- Coating
- Assembling / keeping cleanliness
- Installation
- Adjustment

Schedule



Need a small scale test facility for cryogenic suspension

Shorter turning period for test model experiment

Real scale model
- Cryogenics -> small scale facility
- Cryogenics + SAS -> Kamioka

Man power

K.Kimura
T.Uchiyama
Y.Sakakibara
T.Suzuki

K.Yamamoto

R.Takahashi
SAS group

Collaborators

Y.Saito

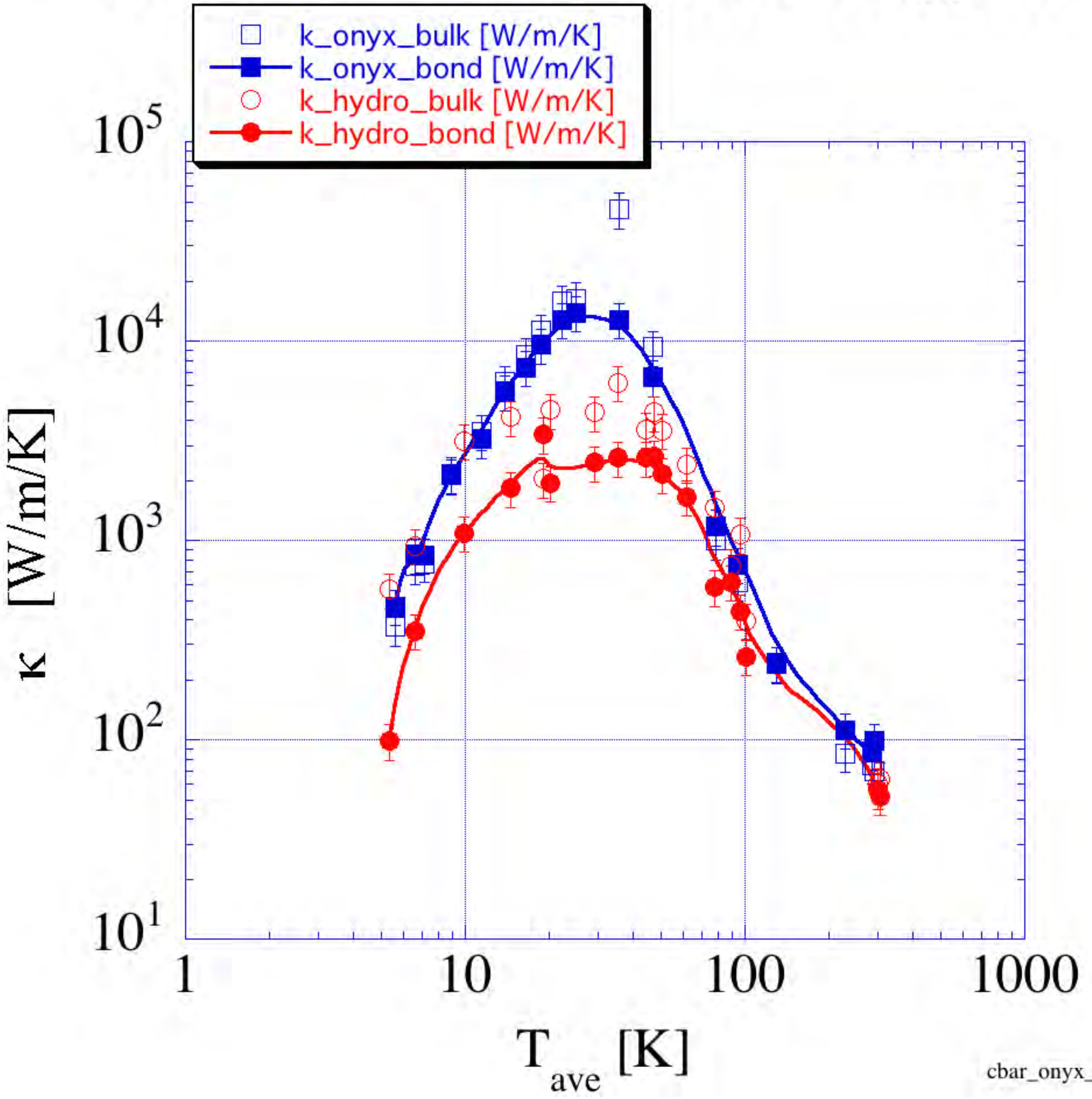
S.Miyoki
Kamiizumi

Ushiba
Shibata

*Effort % is not included

Data

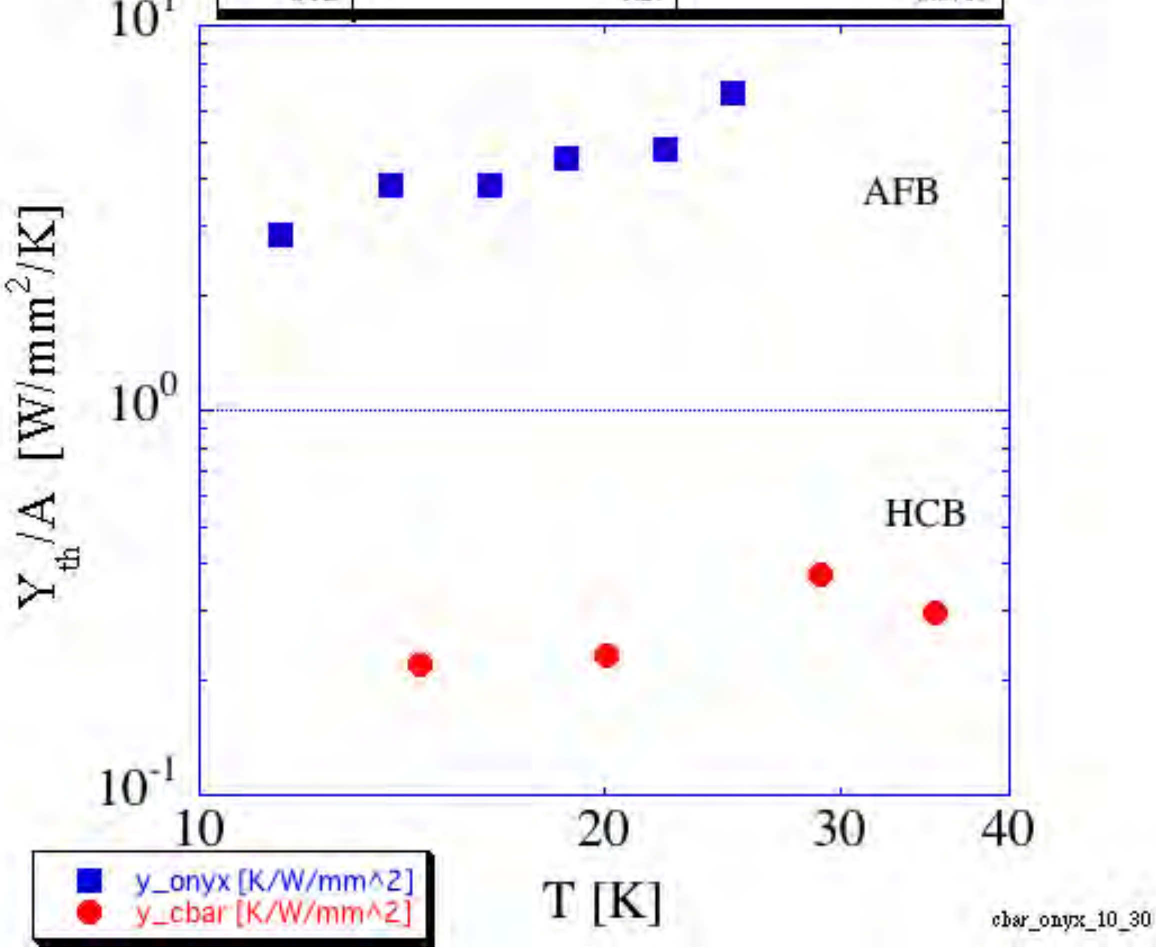
Thermal Conductivity of Bonded Sapphire



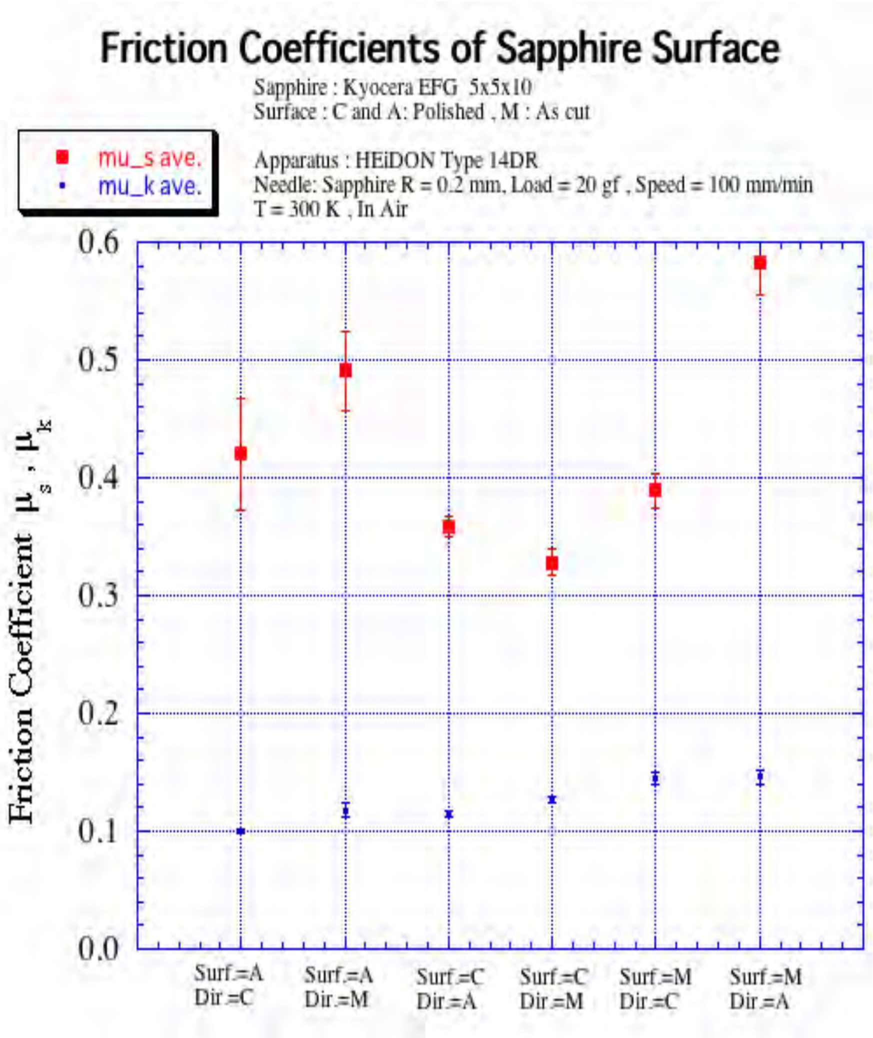
cbar_onyx_tcm.kgd

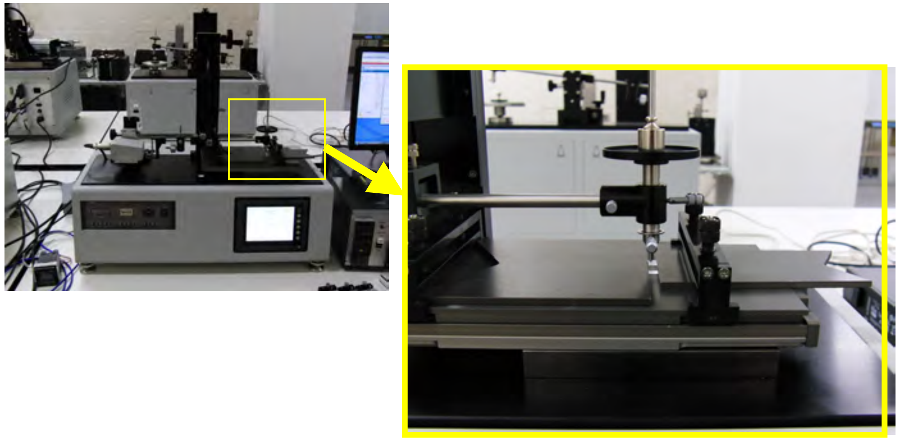
Thermal Conductance of Bonded Boundary

	Y_{th}/A [W/mm^2/K] of AFB	Y_{th}/A [W/mm^2/K] of HCB
Average	4.43	0.280
STD	1.29	0.0719

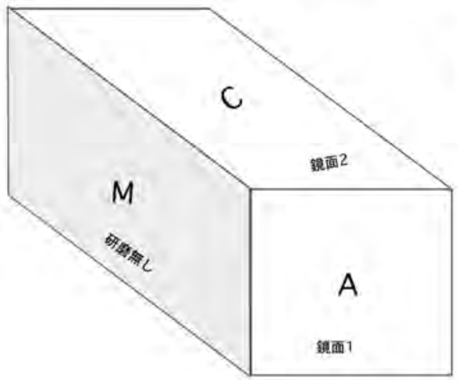


Friction Coefficient of Sapphire Surface

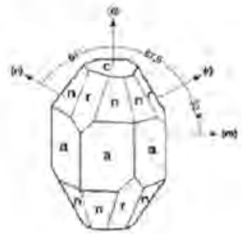




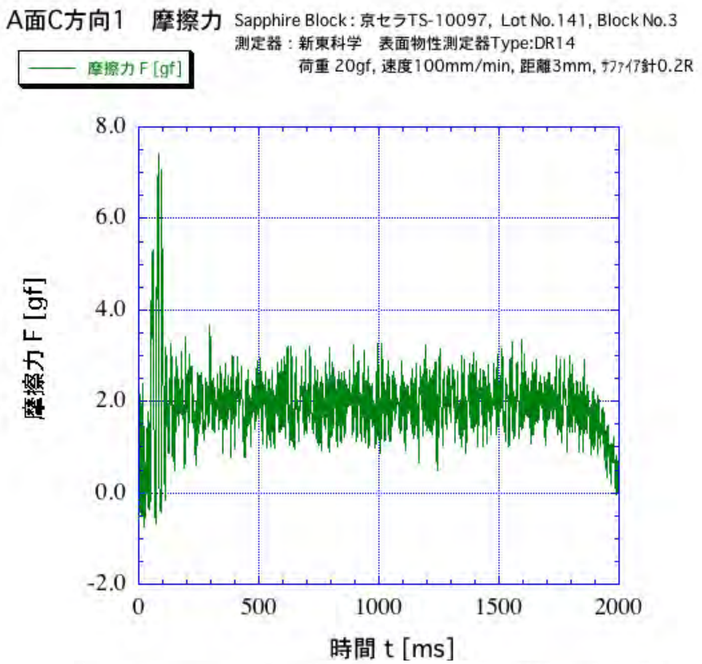
製品名：サファイアブロック(TS-10097)
材質：SA100　ロットNo. 4141(16個), 4142(4個)
出荷日：2010.04.14
図番：TS-10097
注番：20040703
寸法：5mm x 5mm x 10mm

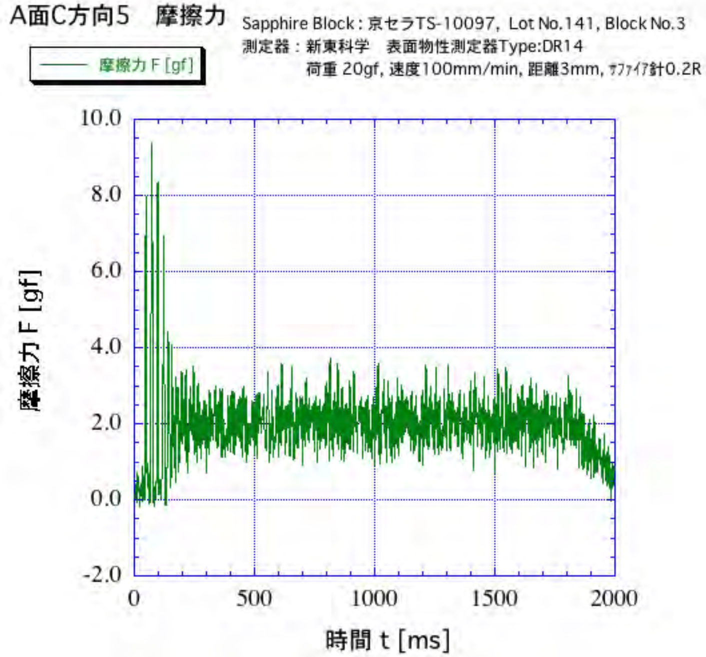


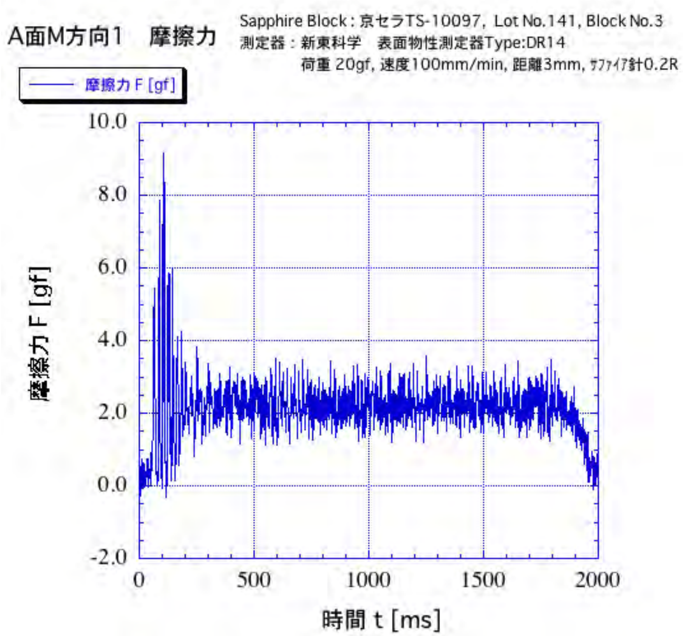
面方位：ACM面
面仕上：4面鏡面

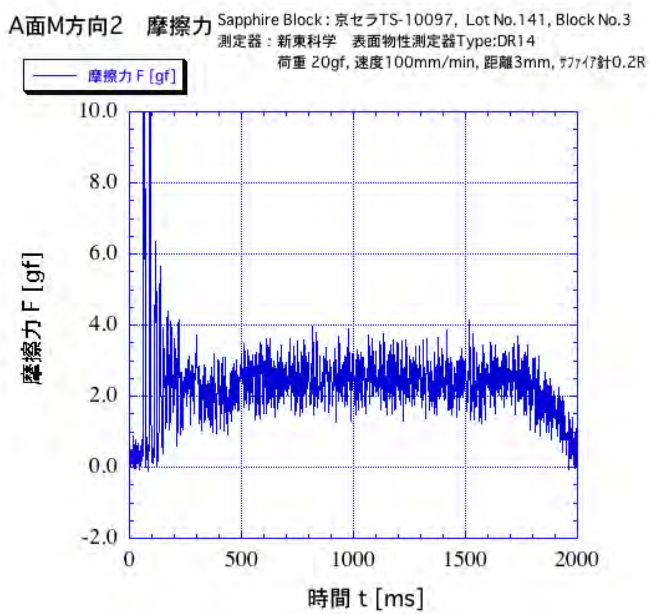


Surface=A , Direction=C, M

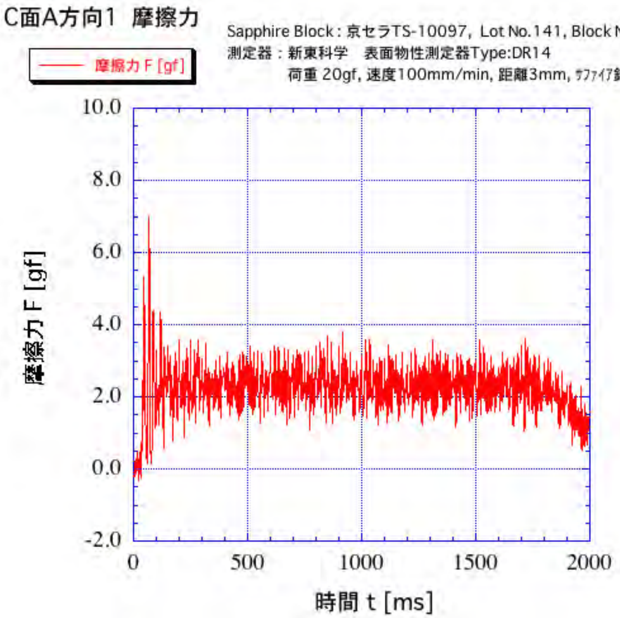


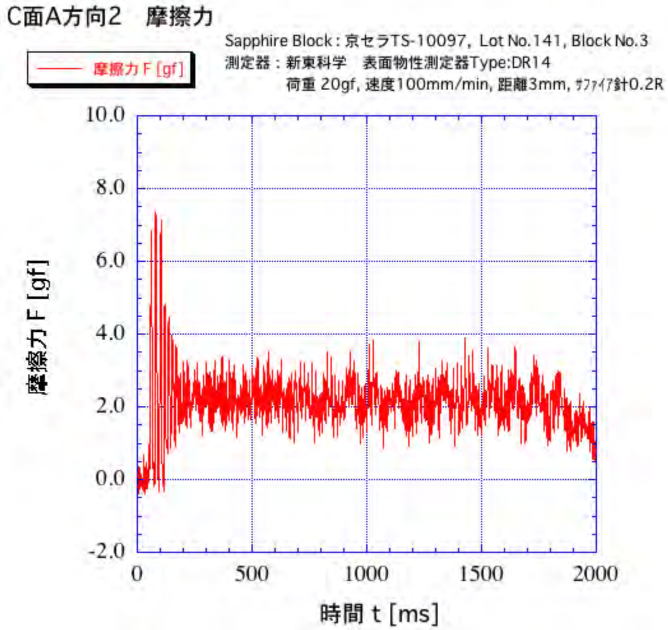


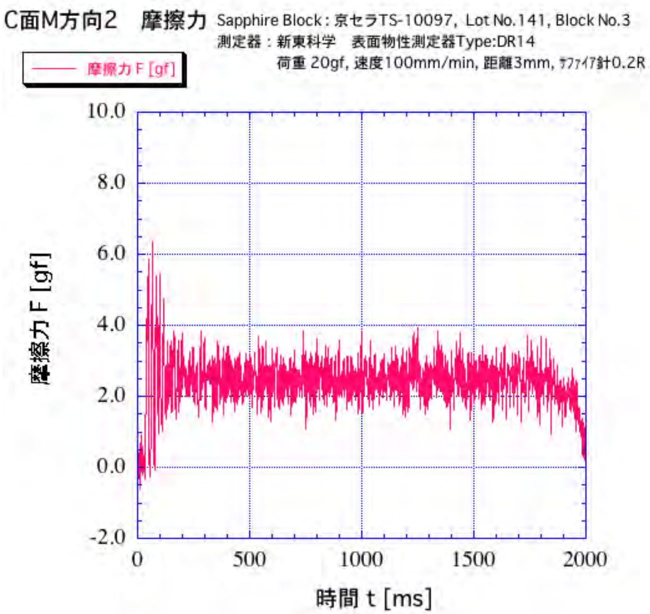


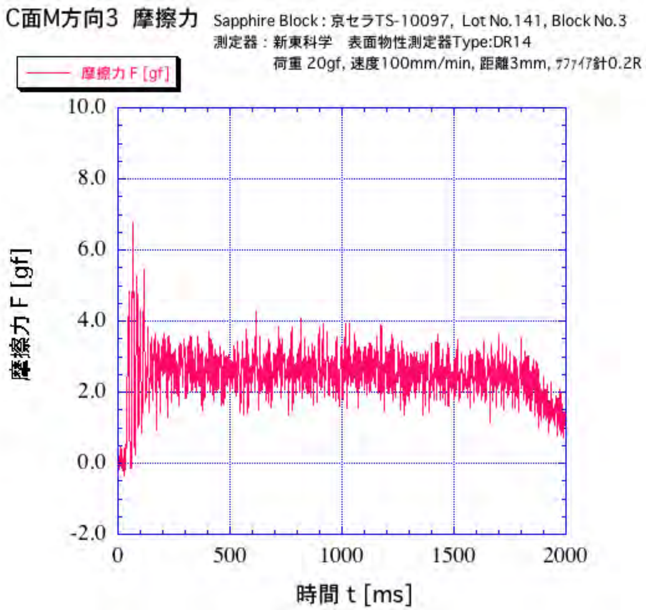


Surface=C , Direction=A , M

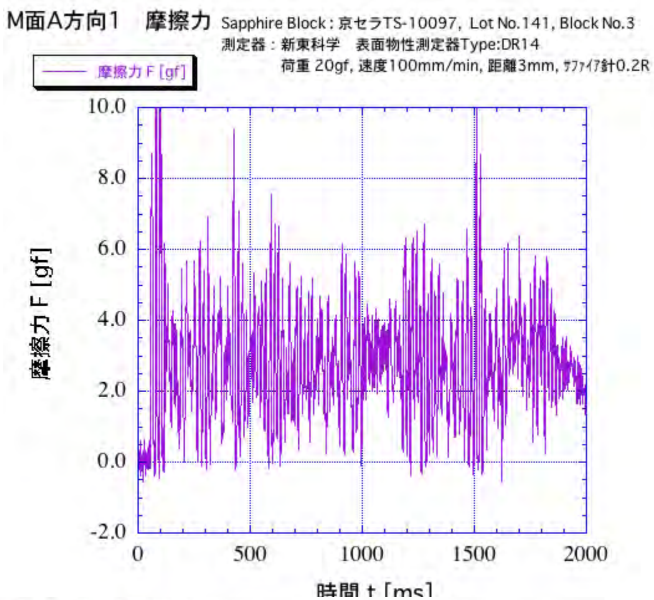


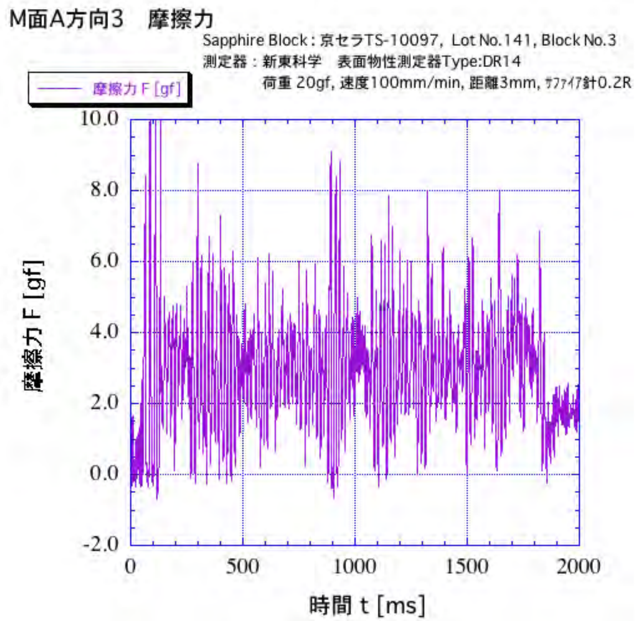


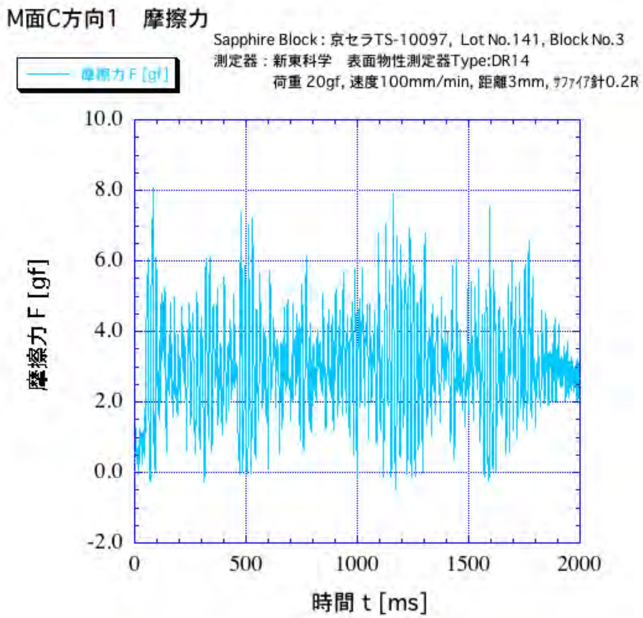


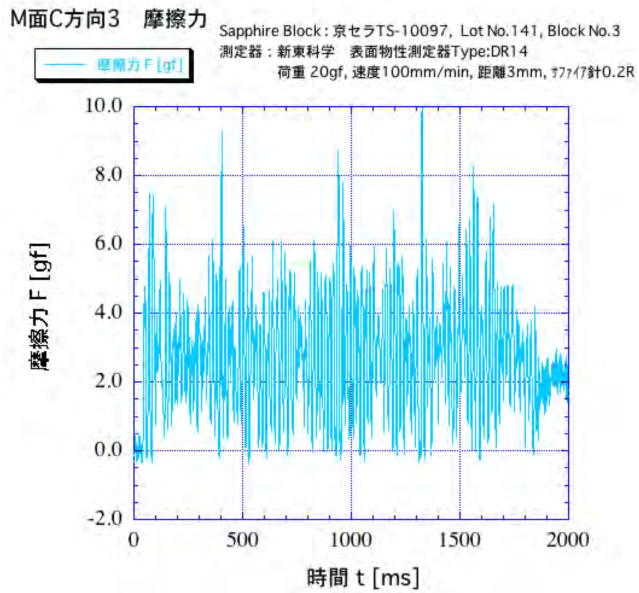


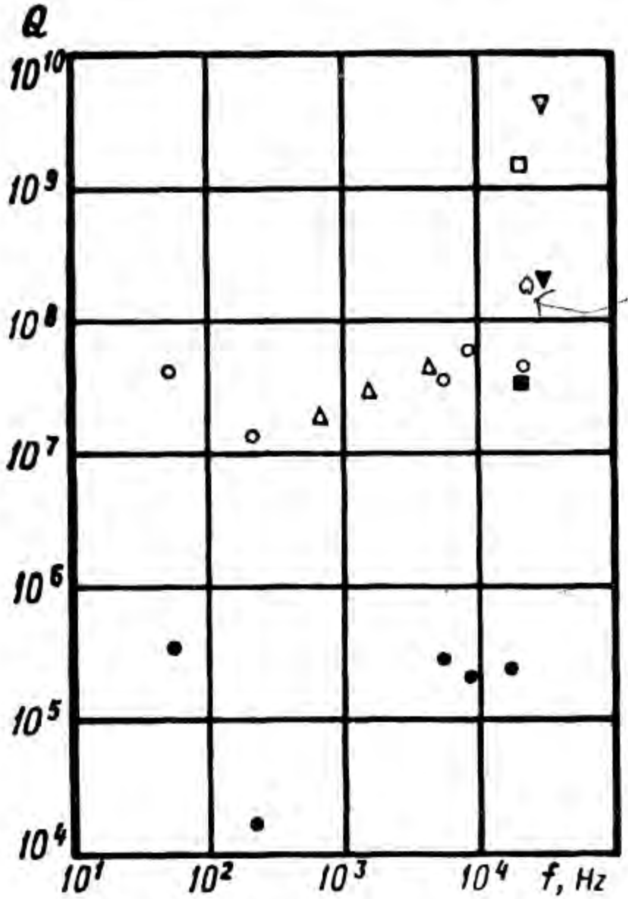
Surface=M , Direction=C , A











coccia et al
PLA 219 (19
35 KHz
40 mK

Fig. 6 Maximum quality factors for mechanical resonators made f
different materials. ○ = Al-5056 ~~(Oide, Tsubono, and Hirakawa 19~~
Δ = Nb (Blair *et al.* 1980b), □ = Si (McGuigan *et al.* 1978), ∇ = A
(Bagdasarov *et al.* 1977). Solid symbols correspond to $T = 300\,\mathrm{K}$,
symbols to $T = 4.2\,\mathrm{K}$. Note added in press: Recently Veitch *e*
(1985) have reported a Q of 2×10^8 at 4 K in a niobium bar.

Braginsky, Systems with small dissipation