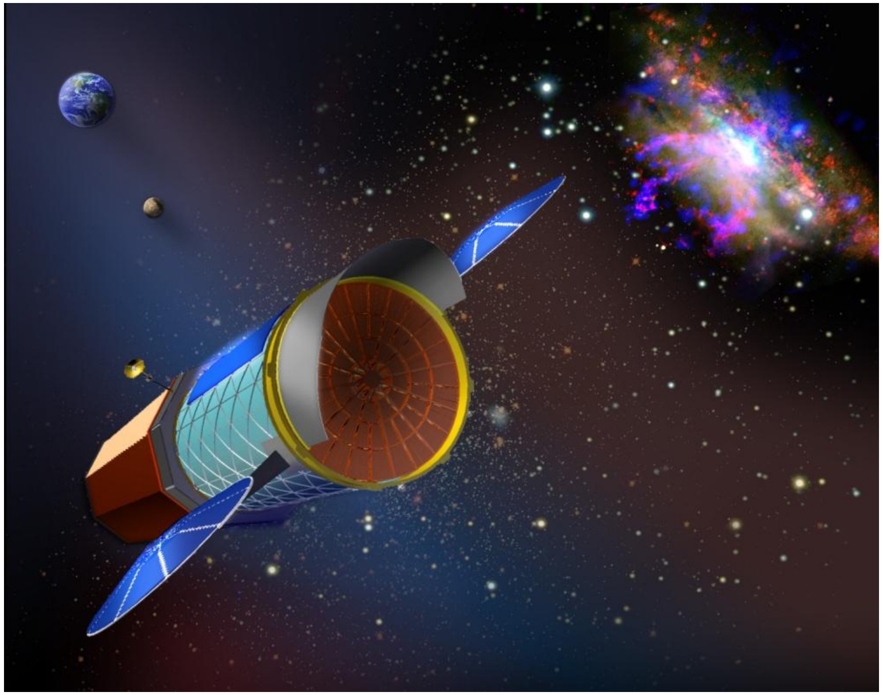
Back-Up Technologies for IXO



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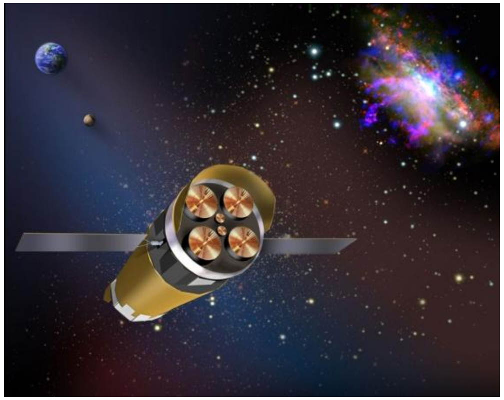
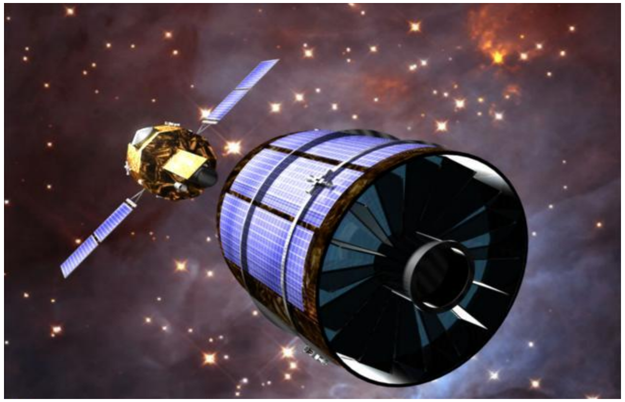
X-ray Optics with
Silicon Wafers

Why Space X-ray Optics with Si wafers and/or slumped glass?

- Future space X-ray telescope such as IXO (International X-ray Observatory) require novel technologies:
  - Light-weight (large apertures, multiply nested)
  - Precise (angular resolutions better than 5 arcsec)
  - Low-cost, mass-production (need for huge geometrical area)
- Old technologies cannot be used as they are either too heavy (thick glass ceramics, e.g. Chandra) or not enough accurate, or both (electroformed Ni, e.g. XMM Newton).



Constellation-X
and
XEUS (now IXO).



Si wafers & X-ray Optics

- The production of Si wafers is a complex process. The recent Si wafers are optimized for semiconductor industry, not for X-ray optics.
- Si wafers parameters need to be optimized for X-ray optics application already at the production stage.
- Si wafers should be shaped stress-free to precise optical shapes.

Si wafers – our approach

- Si wafers are optimized at the production stage.
- Si wafers are bent to precise optical surfaces/geometries.
- Bent Si wafers are stacked into the modules.

What is what in definition of parameters of Si wafers?

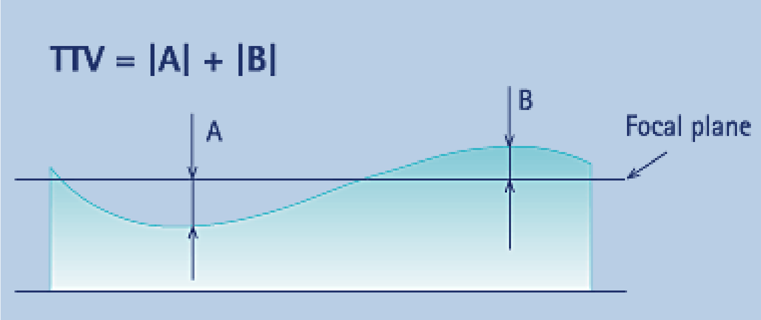
Primary Flat is the flat of longest length appearing in the circumference of the wafer. The primary flat has a specific crystallographic orientation relative to the wafer surface.

Surface flatness is typically measured from an imaginary plane across the centre of the wafer. Data points are then taken from that imaginary plane to the top surface of the wafer. The specification is given as GTIR (Global Total Indicated Readout) which covers multiple data points across the wafer.

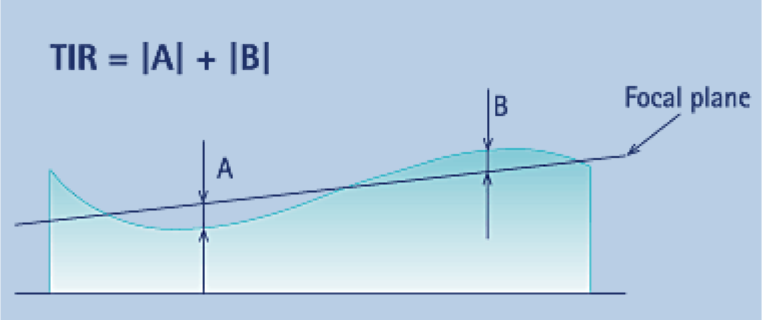
Global parameters: TTV, TIR, WARP, BOW

Local parameters: LTV, LFPD

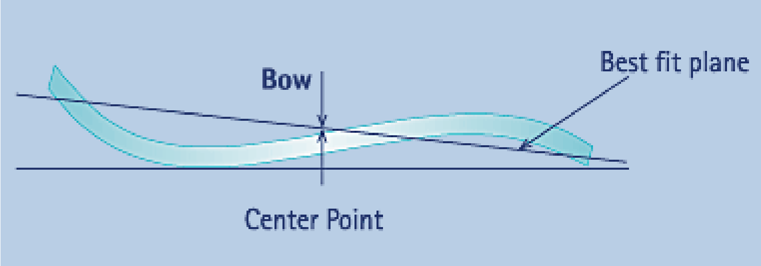
Global parameters



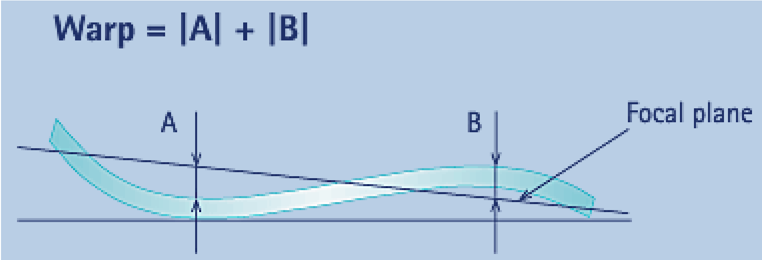
Total Thickness Variation (TTV) represents the difference between the minimum and maximum thickness measured on the wafer.



TIR (Total Indicated Reading) Sum of the maximum positive and negative deviation from the focal plane.

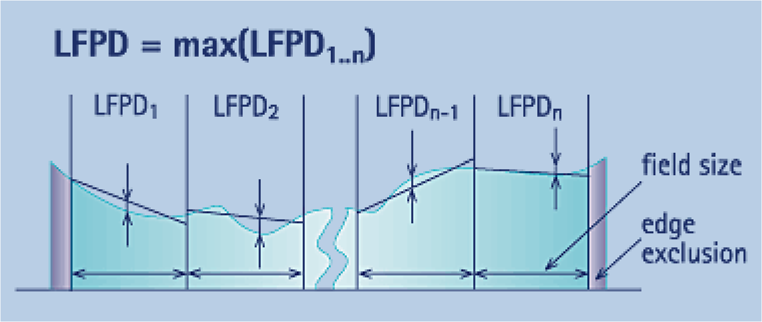


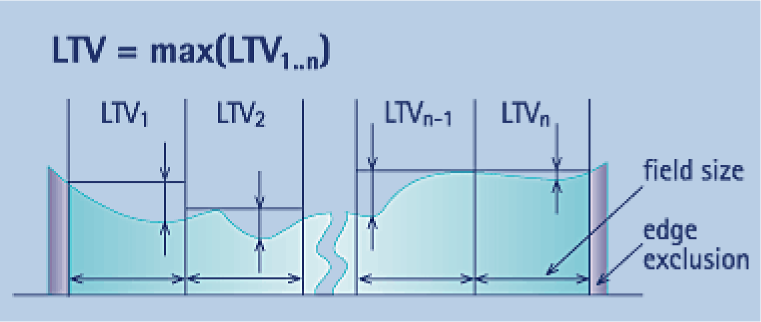
Bow is how concave or convex the deformation of the median surface of the wafer is at its center point, separate from any thickness variations.



Warp is the difference between the maximum and minimum deviations of a wafer's median surface with respect to a reference plane.

Local parameters





LFPD (Local Focal Plane Deviation)
The greatest distance either above or below an established focal plane in a field of special size (e.g. 15x15 mm2).

LTV (Local Thickness Variation)
Distance between the highest Local Thickness Variation and lowest point of the surface in a field of special size (e.g. 15x15 mm2).

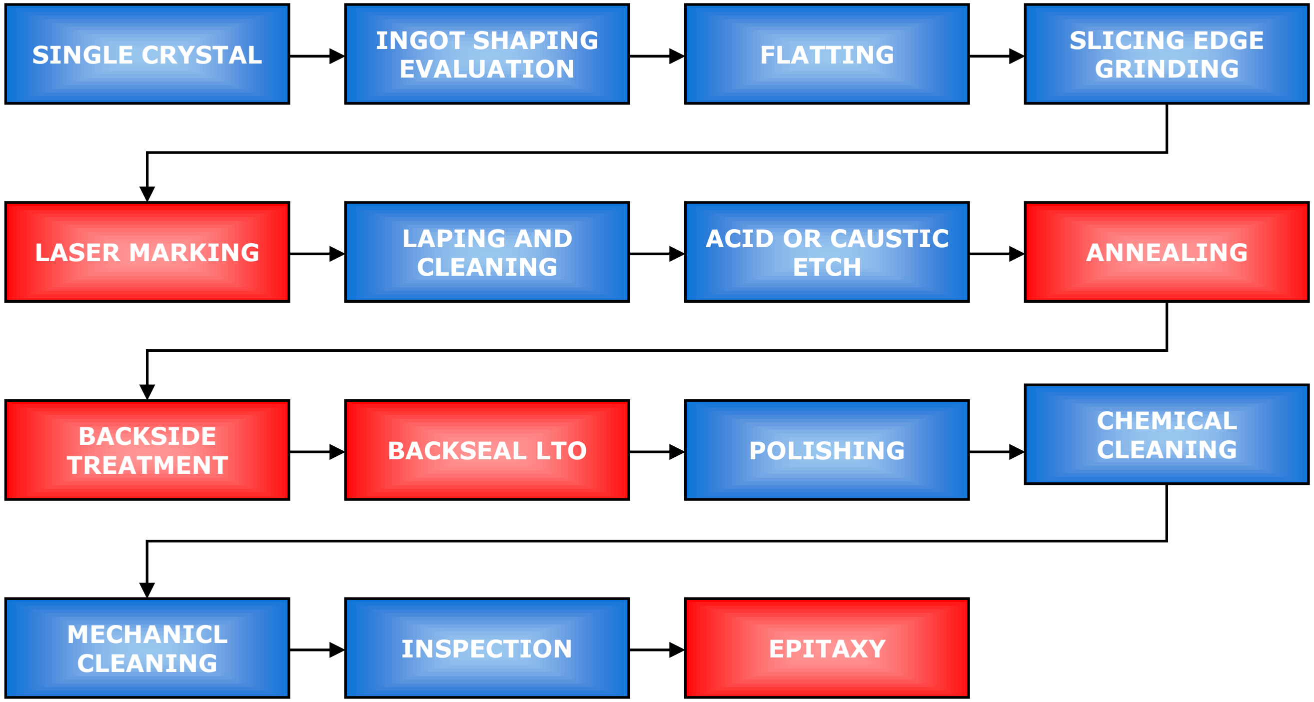
Unfortunately, the float glass manufacturers do not use analogous definitions, hence the direct comparison Si wafers vs. glass is difficult

Back-up technology for IXO

- **There is an urgent need for back-up technology for primary XEUS-IXO technology (MPO, Multi Pore Optics = Mosaic Optics).**
- **The technology described here represents such an alternative.**
- **An obvious option might be the merger of both approaches: i.e. the precisely pre-shaped Si wafers with no internal stress will be used for MPO.**

Improving parameters of Si wafers for X-ray optics applications

Basic chart of silicon wafer manufacturing



SINGLE CRYSTAL

INGOT SHAPING EVALUATION

FLATTING

SLICING EDGE GRINDING

LASER MARKING

LAPING AND CLEANING

ACID OR CAUSTIC ETCH

ANNEALING

BACKSIDE TREATMENT

BACKSEAL LTO

POLISHING

CHEMICAL CLEANING

MECHANICL CLEANING

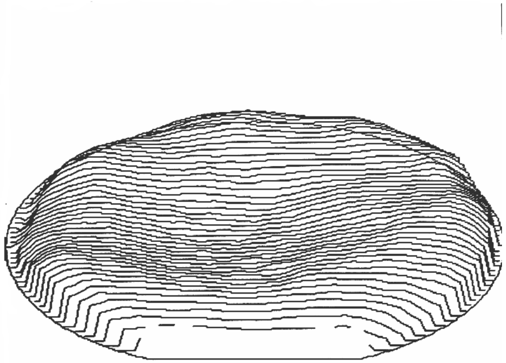
INSPECTION

EPITAXY

🟥 optional

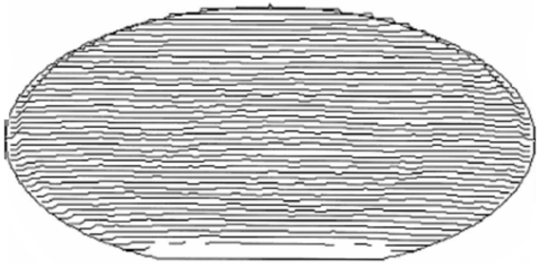
Complex process – has to be optimized for X-ray optics applications.

Development of improved Si wafers for X-ray optics applications



Standard

Flatness of standard silicon wafer used for technologies with photolithographic detail ~ 5 µm, 150 mm diameter. Thickness in the wafer center: Cen. THK 628.81 µm, minimal measured thickness: Min. THK 630.40 µm, maximal measured thickness: Max. THK 632.50 µm. Total thickness variation: TTV = (Max. THK) – (Min. THK) = 2.10 µm. TIR: 1.76 µm.



Improved

Flatness of highly flat silicon wafer developed for sub-micron technologies in ON Semiconductor, 150 mm diameter. Thickness in the wafer center: Cen. THK 610.92 µm, minimal measured thickness: Min. THK 610.58 µm, maximal measured thickness: Max. THK 611.03 µm. Total thickness variation: TTV = (Max. THK) – (Min. THK) = 0.45 µm. TIR: 0.29 µm.

Measurement of flatness

	Standard Si wafer	**Improved Si wafer**
Diameter [mm]	150	150
Cen. THK [µm]	628.8	610.9
Min. THK [µm]	630.4	610.6
Max. THK [µm]	632.5	611.0
TTV [µm]	2.10	0.45
TIR [µm]	1.76	0.29

Flatness of standard silicon wafer is used for technologies with photolithographic detail ~ 5 µm.

Method for high flatness of silicon wafers has been developed by ON Semiconductor Czech Republic: improvement by factor of 5!

Influence of dopants

Dopants used in semiconductor technology.

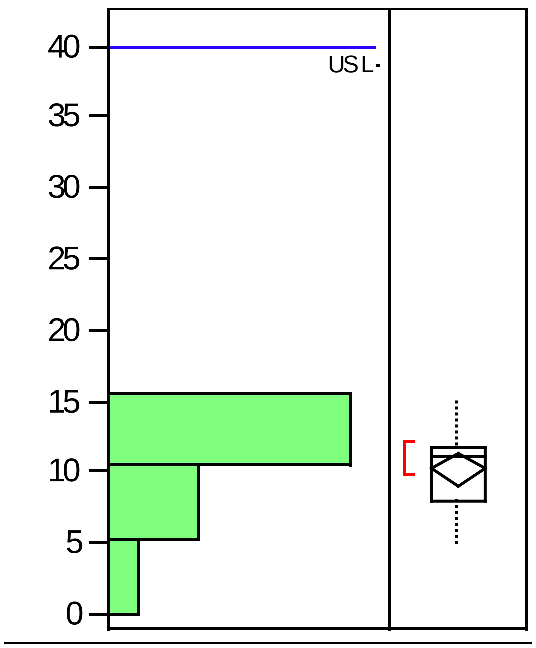
Dopant	**k_0**	**c_s^m (cm^{-3})**	**g(cms^{-1})**	**δ_r (%)**
B	0.800	$6.0x10^{20}$	$8.0x10^{-6}$	-25
As	0.300	$1.8x10^{21}$	$8.0x10^{-3}$	0
P	0.350	$1.3x10^{21}$	$1.6x10^{-4}$	-7
Sb	0.023	$7.0x10^{19}$	$1.3x10^{-1}$	+15

Segregation coefficient, i.e., dopant concentration in crystal/melt k_0, maximal solubility *csm*, evaporation speed *g* and length of the bond dopant-silicon compared to silicon-silicon *δr* are main parameters of Czochralski process.

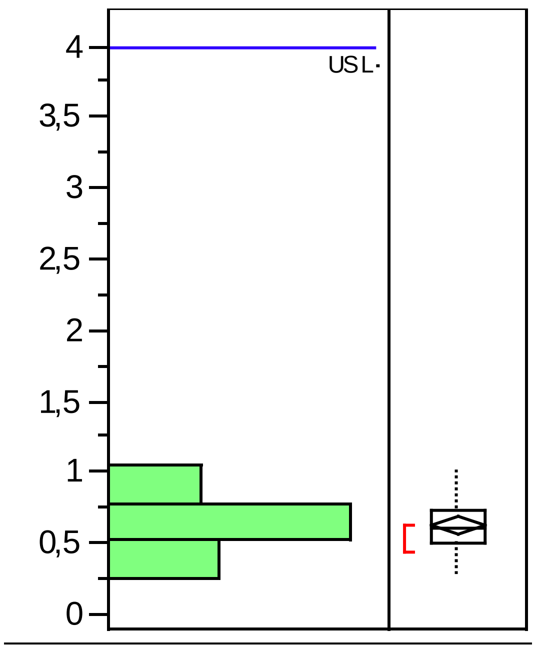
The dopant must be optimized for the X-ray optics application, as the dopant has influence on microrougness and other parameters

Improved Si wafers

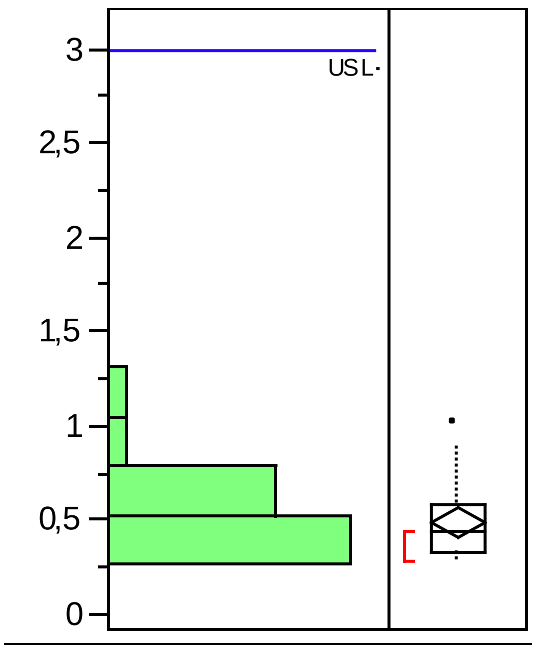
Measurement of 24 silicon wafers flatness, upper specification limit (USL) for semiconductor application is indicated. Wafers were manufactured with novel method for high flatness.



WARP [µm]

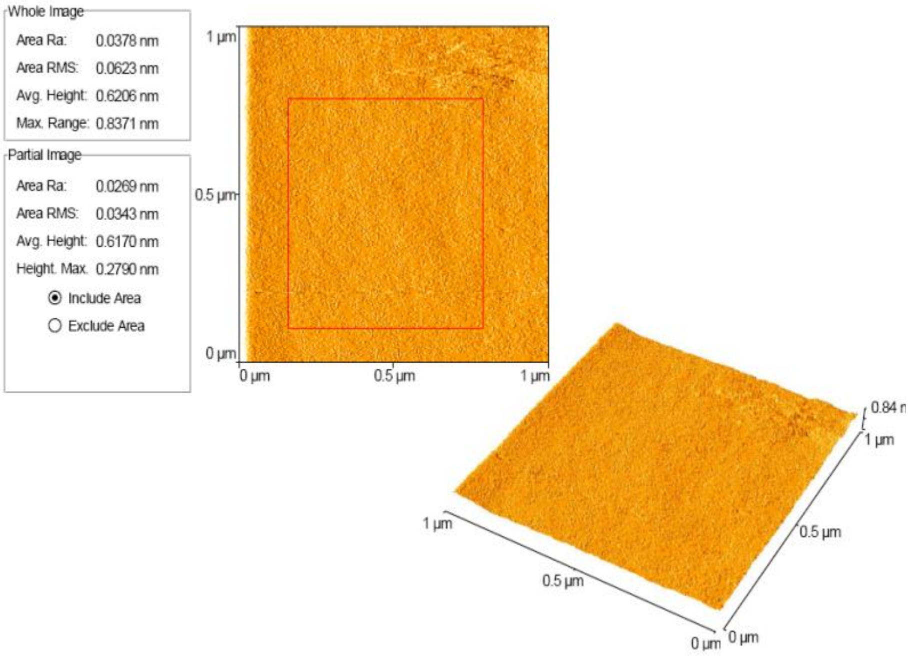


TTV [µm]



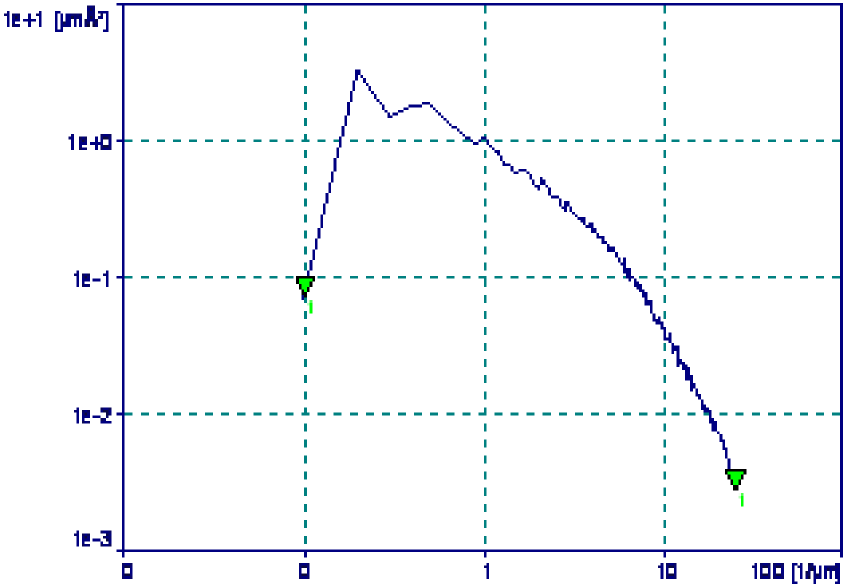
TIR [µm]

Improved Si wafers



Whole Image
Area Ra: 0.0378 nm
Area RMS: 0.0623 nm
Avg. Height: 0.6206 nm
Max. Range: 0.8371 nm

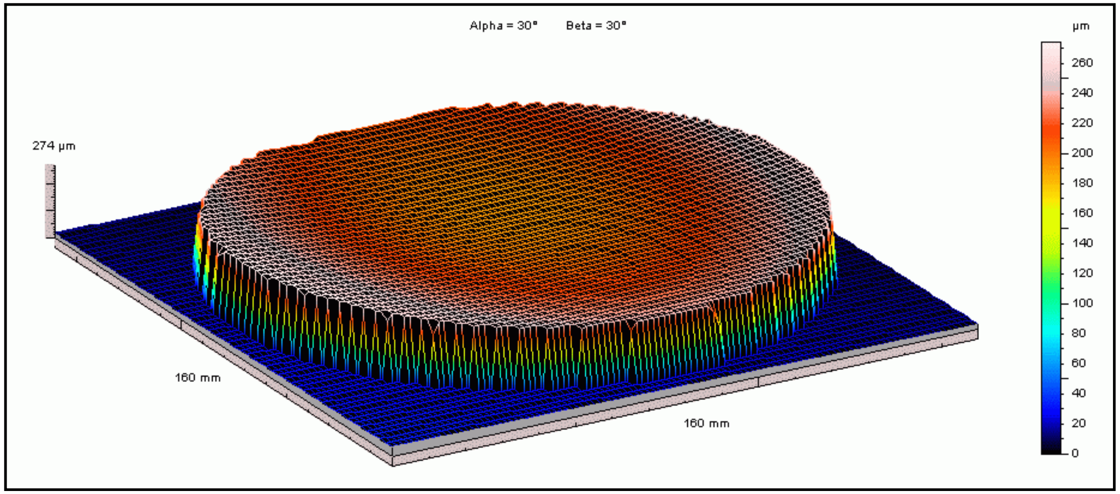
Partial Image
Area Ra: 0.0269 nm
Area RMS: 0.0343 nm
Avg. Height: 0.6170 nm
Height. Max. 0.2790 nm
● Include Area
○ Exclude Area



Surface roughness of polished Si wafer measured with AFM microscopy (left). Crystallographic orientation (100), CZ wafer is heavily doped with arsenic. Measured area 1 µm x 1 µm, Ra = 0.04 nm, RMS = 0.06 nm. Power Spectral Density (PSD) function was calculated for these data.

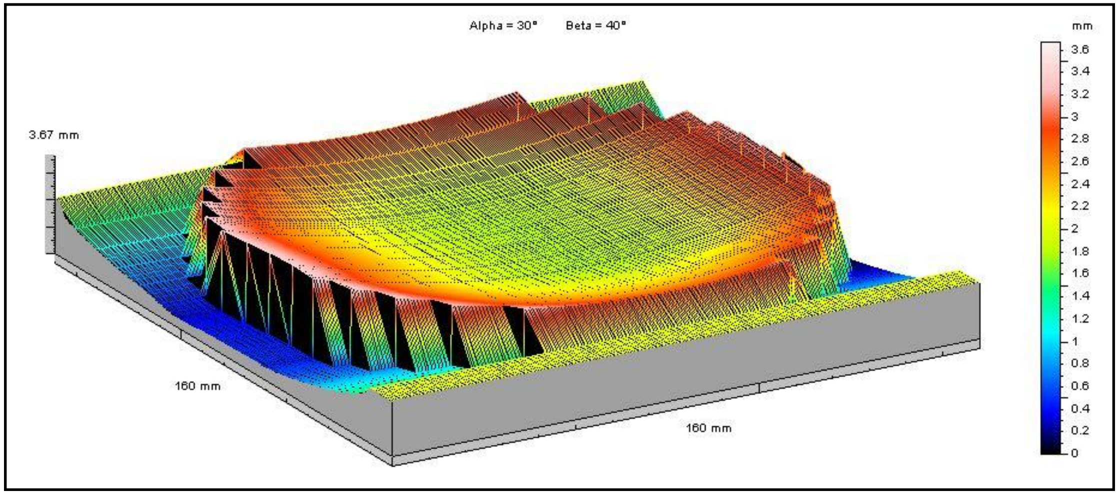
Precise shaping of Si wafers

Measuring of shapes: flat and bent Si wafers



flat Si wafer
(dopant P)
D = 150 mm
t = 0.625 mm

bent Si wafer
(dopant P)
D = 150 mm
t = 1.3 mm
R = 1650 mm



SHAPING METHOD I - INTRINSIC THIN FILM STRESS FORMERLY TECHNOLOGY I

Thin film with residual stress σ_f on the top of silicon wafer deform wafer according stress value and stress type *[S.Timoshenko, J. Opt. Soc. Am., 11, 233 (1925)] (compressive or tensile)*

$$\boxed{\sigma_f = \frac{E}{6(1-\nu)} \cdot \frac{t_s^2}{t_f} \cdot \left(\frac{1}{R} - \frac{1}{R_0} \right)} \qquad (5)$$

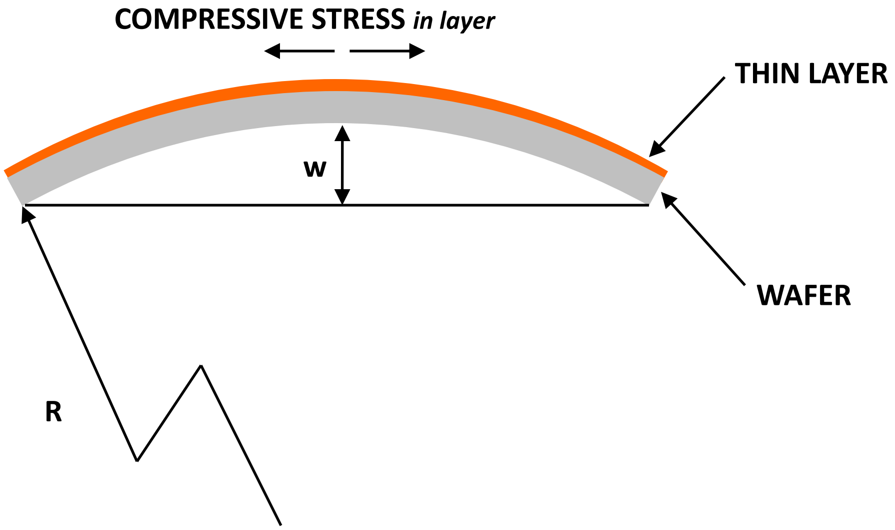
E Young's modulus ; Silicon (100) – $1.3 \cdot 10^{11}$ N/m^2

ν Poisson's ratio; Silicon (100) – 0.28

t_s Wafer thickness

R Radius of curvature after film depo

R_0 Radius of curvature before film depo



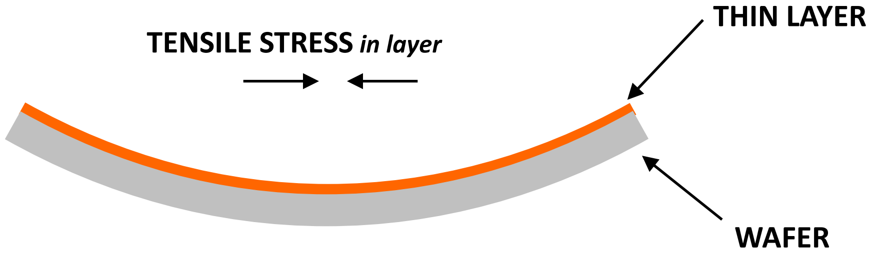
COMPRESSIVE STRESS ***in layer***

THIN LAYER

w

WAFER

R



TENSILE STRESS ***in layer***

THIN LAYER

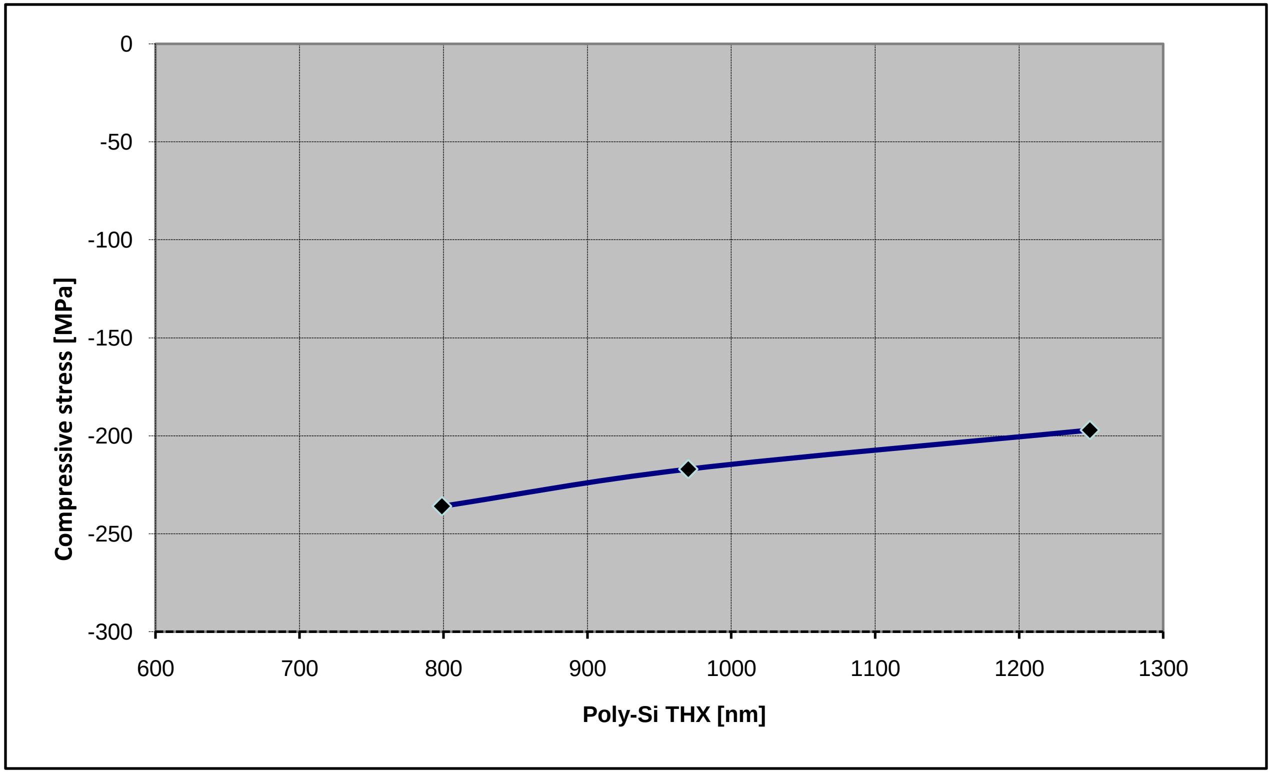
WAFER

Therefore the warp is proportional to the residual stress and film thickness and inversely proportional to the wafer thickness squared.

LPCVD Poly-Si FILMS

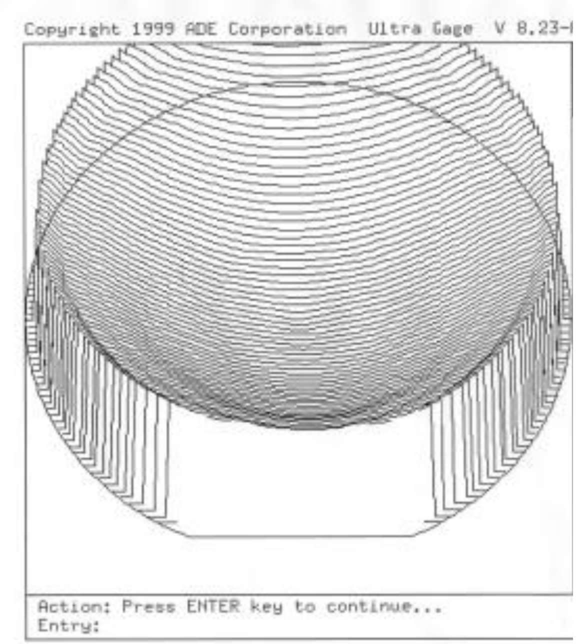
Heat treatment of poly-Si films can cause the atoms to move to low-energy positions. Poly-Si thickness (THX) is proportional to the depo time, which can impact the stress in poly-Si films.

Deposition temperature 615°C



BACK SIDE LAYER

After depo of poly-Si (THX 1436 nm at temperature 615°C) and for wafer thickness 507 µm the warp 110 µm (R = 25.6 m) was achieved.



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Copyright 1999 ADE Corporation  Ultra Gage  V 8.23-
Action: Press ENTER key to continue...
Entry:
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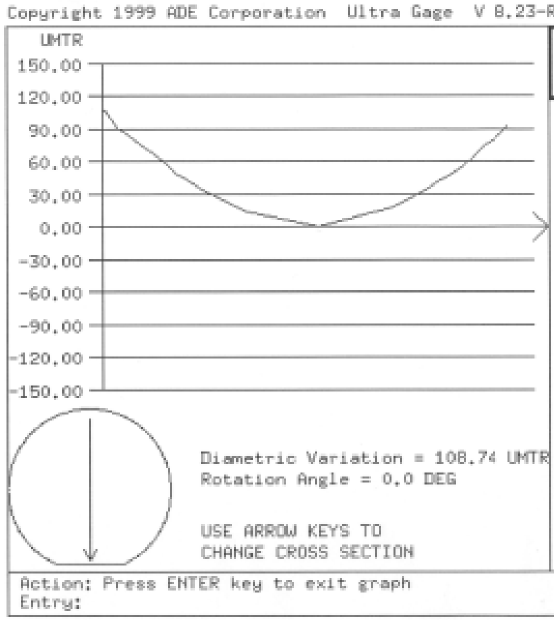
Wafer deformation map



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Copyright 1999 ADE Corporation  Ultra Gage  V 8.23-R
Diametric Variation = 108.74 UMTR
Rotation Angle = 0.0 DEG

USE ARROW KEYS TO
CHANGE CROSS SECTION
Action: Press ENTER key to exit graph
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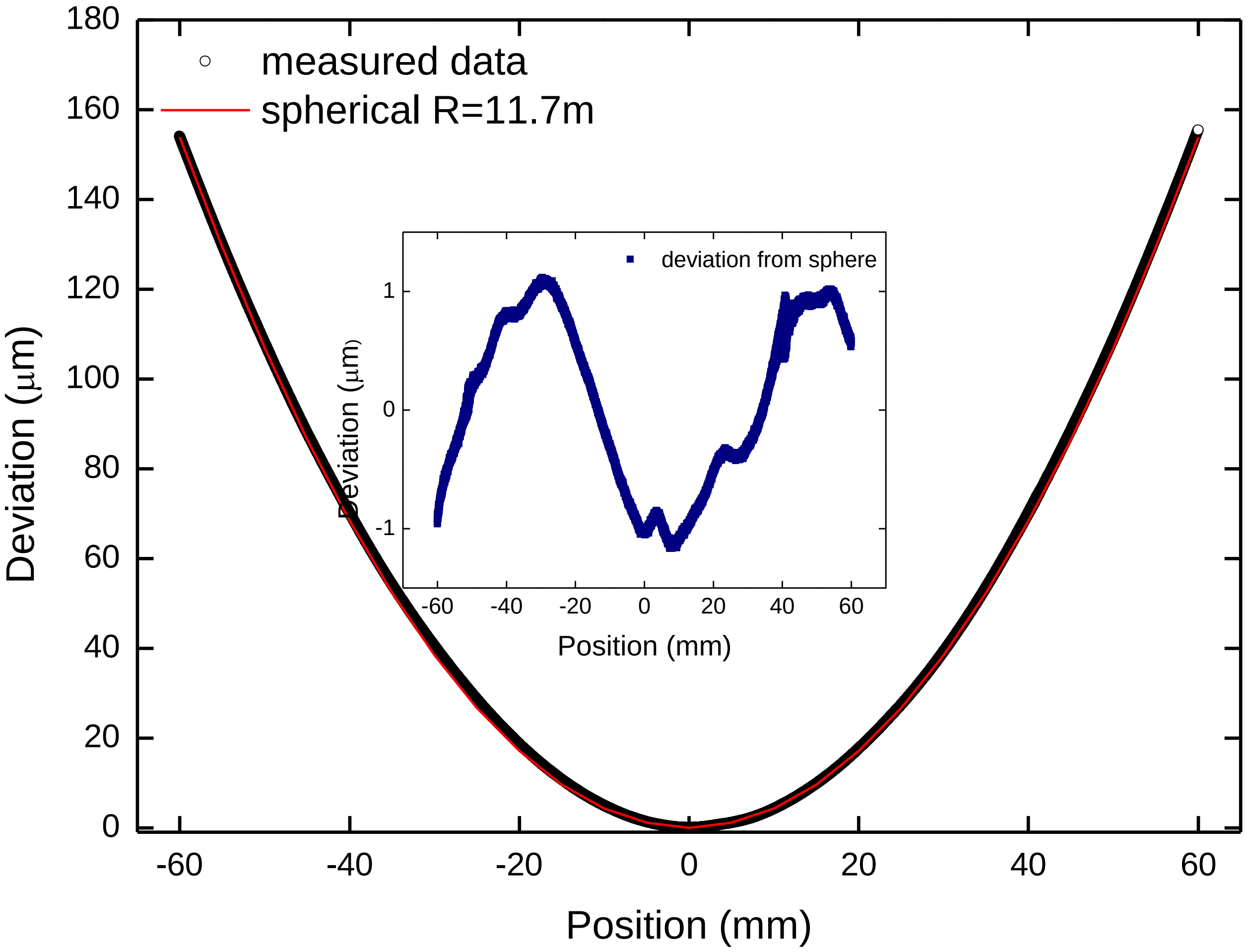
Warp profile perpendicular to the facet

WAFER SHAPE

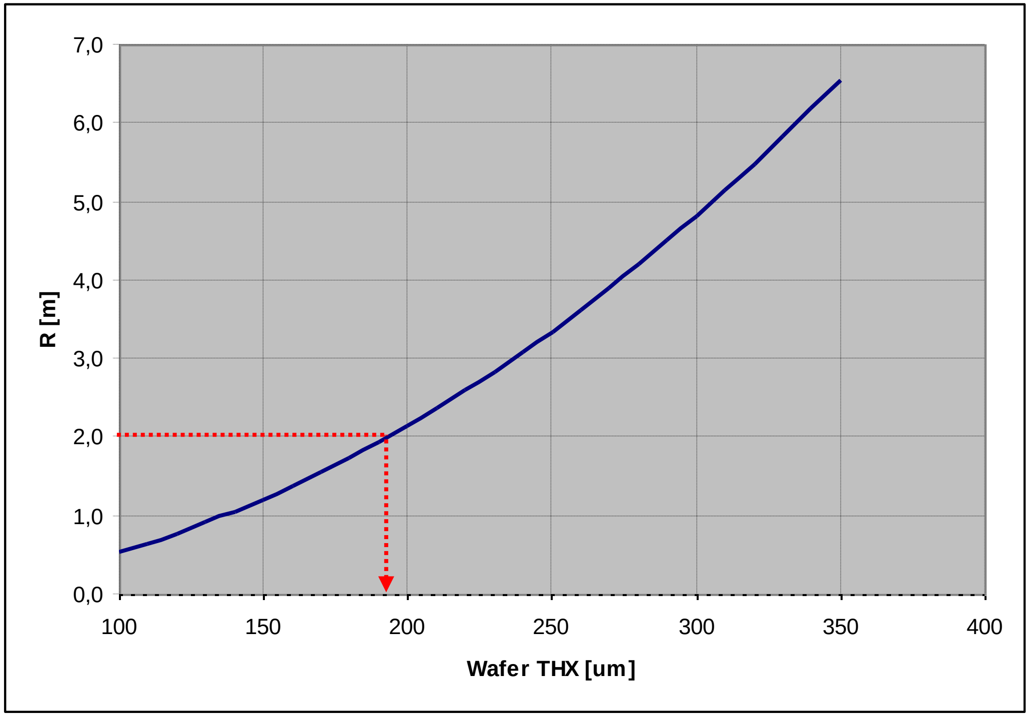
Squared wafer has spherical shape. Deviation from ideal sphere is within 1 μm.



AXRO Prague 2009

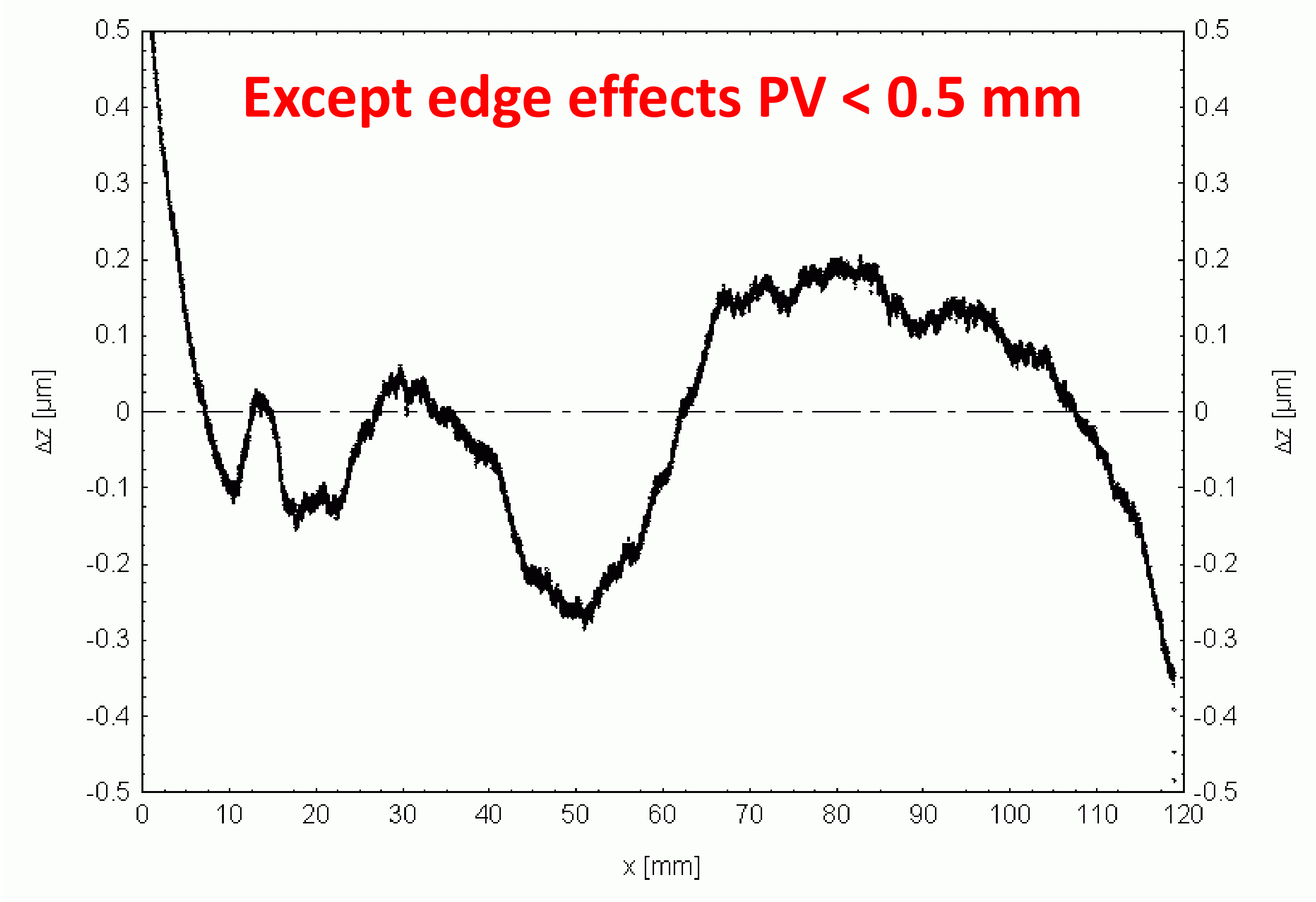
LAYER STACK AND WAFER THICKNESS

- For designed stack we can calculate the wafer thickness to achieve expected radius of curvature.
- As we can see in chart the wafer thickness 195 μm would be needed for R ~ 2 m.
- That thin wafer is sensitive for handling and also it is affected by gravity sag.



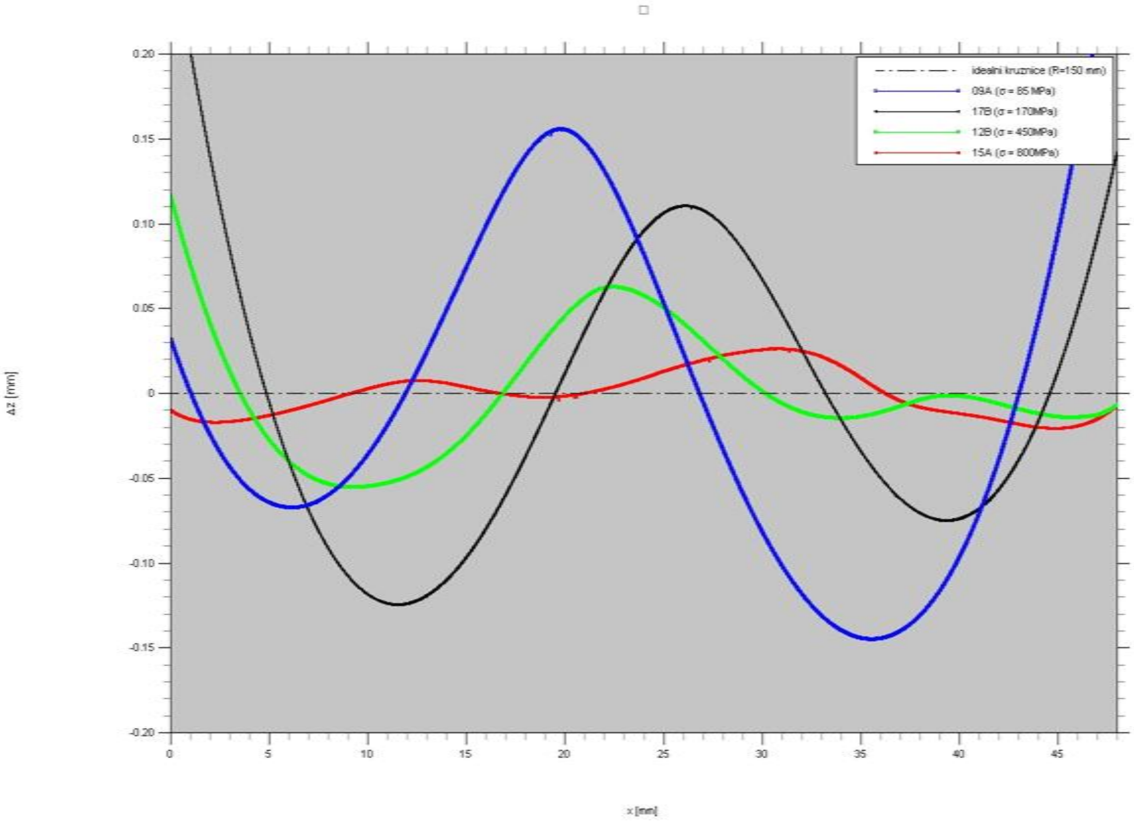
Bent Si wafers - Technology II

Taylor-Hobson profilometer – deviation from ideal shape
D = 150 mm, t = 0.625 mm, parabolic shape


Except edge effects PV < 0.5 mm

Bent Si wafers – Technology III
Thermal Forming

Optimizing parameters of thermal forming of Si wafers

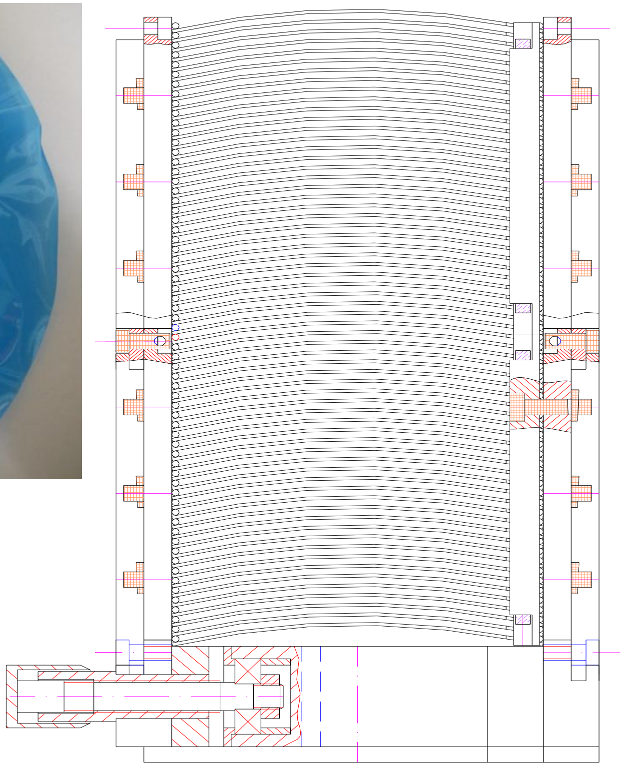


thermally formed Si wafer to test cylinder (R = 150 mm, 72 x 23 x 0.325 mm)

The effect of elastic tension on deviation from ideal surface (thermal forming of Si wafers).

Alternative Si wafer X-ray MFO

Stacked module based on (before) precisely shaped Si wafers



Multi-Foil Optics (MFO)

Three methods for Si wafer cutting tested
Wafers cutted to Squares 10 x 10 cm

How to increase the size of Si wafers?

Motivation:

The size of Si wafers has been limited to about 300 mm diameter recently.

Some X-ray optics applications (such as active X-ray optics for Gen X) require larger sizes.

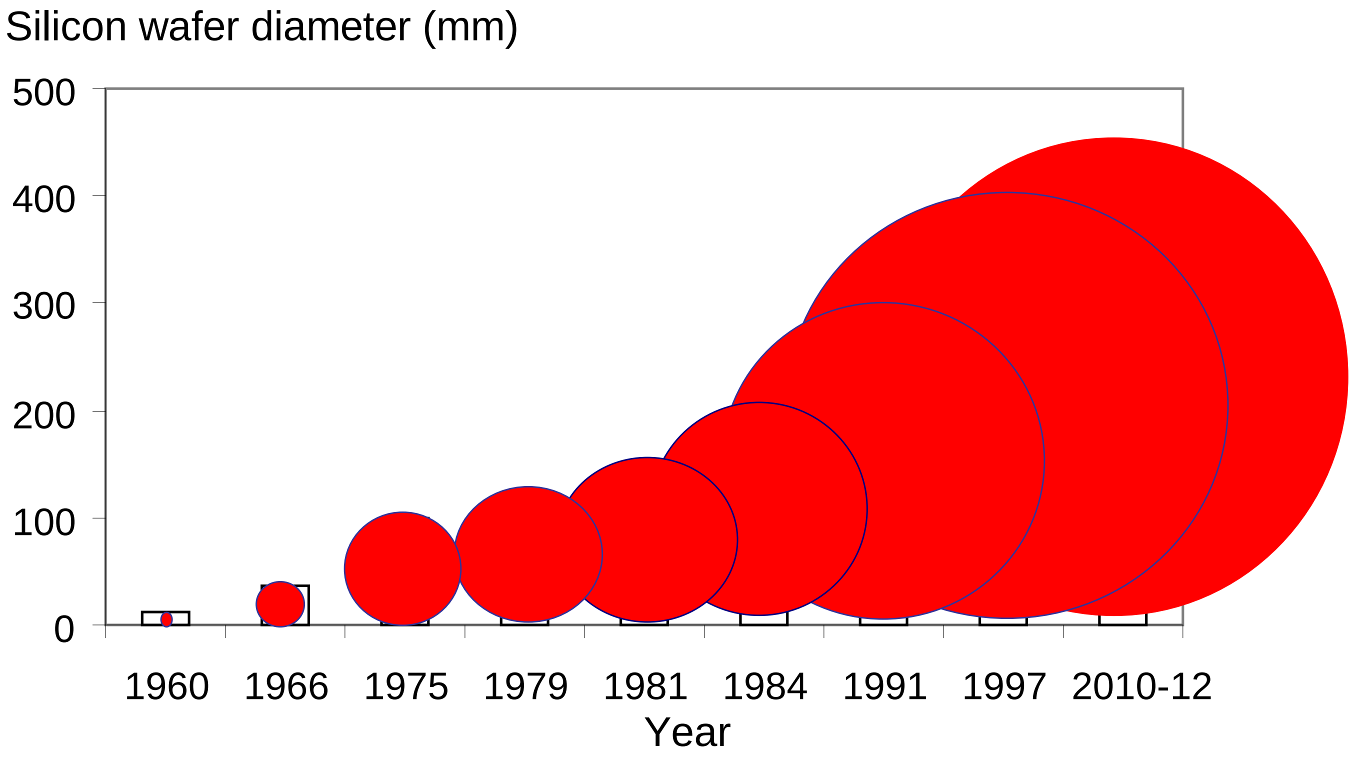
Parameters of Si wafer

The table consists from four blocks – for 100 and 125 mm, for 150 mm wafer diameter with different THK, for large-scale wafer diameters up to 450 mm with standard THK and for thin 450 mm wafer with THK decreased down to 150 µm.

Diameter	THK	Area (A)	Weight (W)	W/A	length (h)	rel. area (SQh)	weight of square (WSQh)
(mm)	(µm)	(sqcm)	(g)	(g/sqcm)	(mm)	(sqcm)	(g)
100	525	78,5	9,6	0,12	70,71	50,00	6,1
125	625	122,7	17,9	0,15	88,39	78,13	11,4
150	625	176,7	25,7	0,15	106,07	112,50	16,4
150	525	176,7	21,6	0,12	106,07	112,50	13,8
150	380	176,7	15,6	0,09	106,07	112,50	10,0
150	150	176,7	6,2	0,03	106,07	112,50	3,9
200	725	314,2	53,1	0,17	141,42	200,00	33,8
300	775	706,9	127,6	0,18	212,13	450,00	81,3
400	825	1256,6	241,6	0,19	282,84	800,00	153,8
450	825	1590,4	305,7	0,19	318,20	1012,50	194,6
450	150	1590,4	55,6	0,03	318,20	1012,50	35,4

Time development

Silicon wafer diameter (mm) – time development overview based on several public sources (Sematech, MEMC, EEtimes).



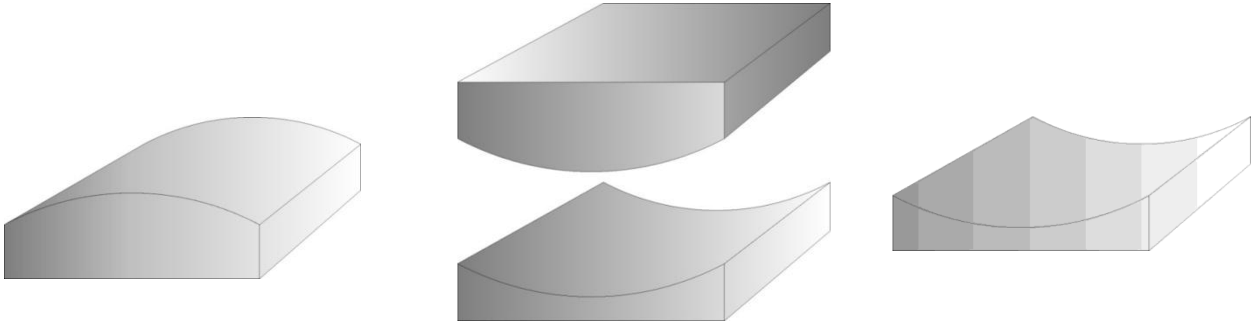
X-RAY OPTICS BASED ON GLASS THERMAL FORMING (GTF)

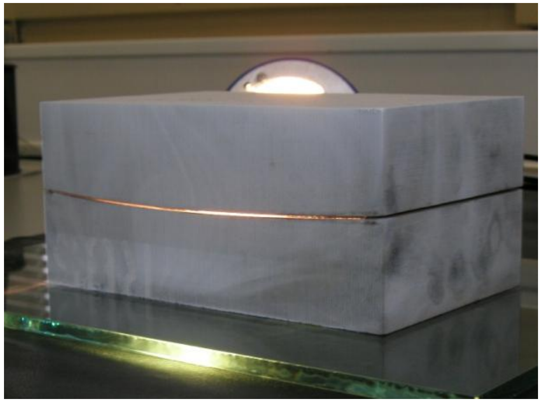
alternative glass technologies represent glass forming

which avoid heating

More details in the aftenoon talk

Various approaches in GTF

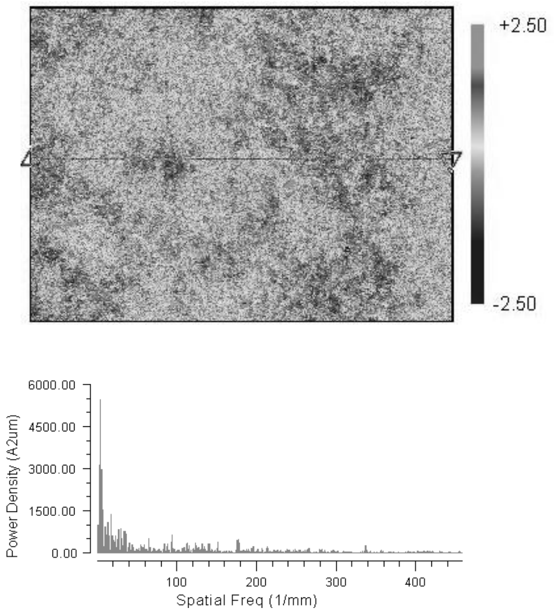




- low-cost design needed (the goal is to produce very large number of shells at a low cost)
- expensive production/material are to be avoided
- the mandrel material/design is important
- recent design: proprietary technology (composite)

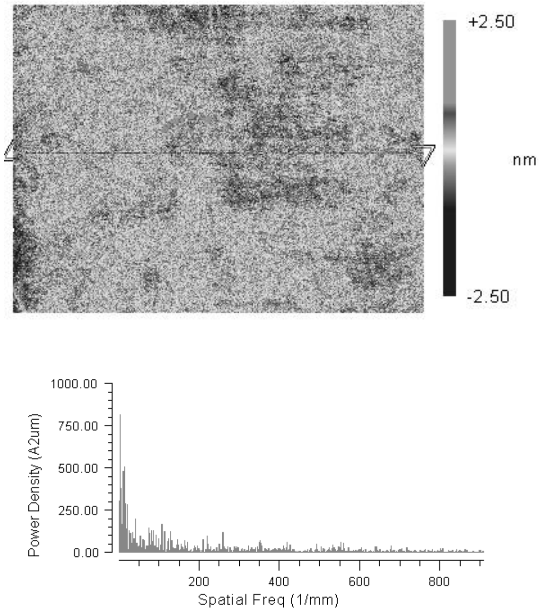
Glass thermal forming

Measuring of roughness – Zygo interferometer



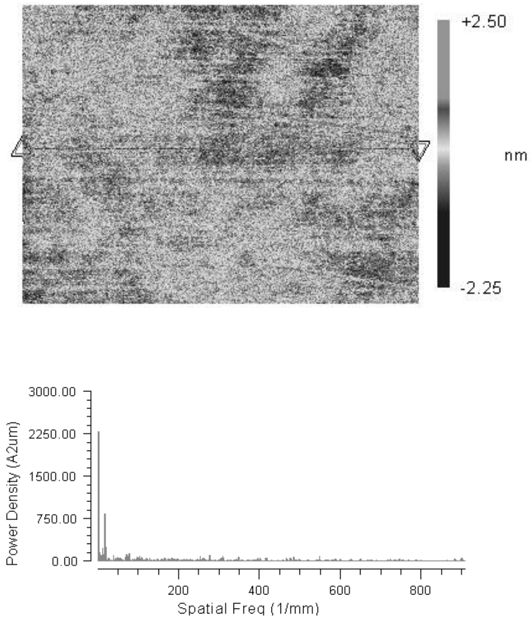
0.70 x 0.52 mm

RMS	1.425 nm
Ra	0.498 nm



0.35 x 0.26 mm

RMS	0.371 nm
Ra	0.282 nm



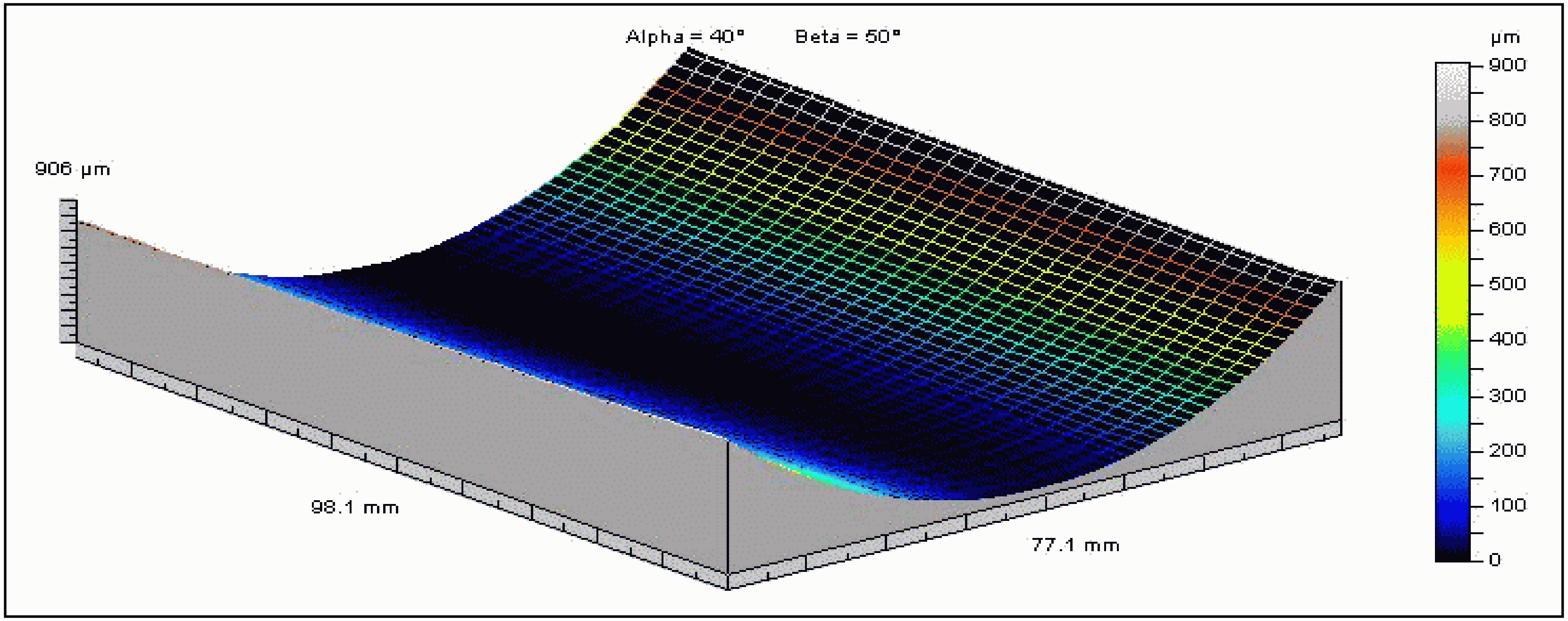
0.35 x 0.26 mm

RMS	0.393 nm
Ra	0.296 nm

Glass thermal forming (GTF)

Measuring of shape

Still optical profilometer – 3D chart



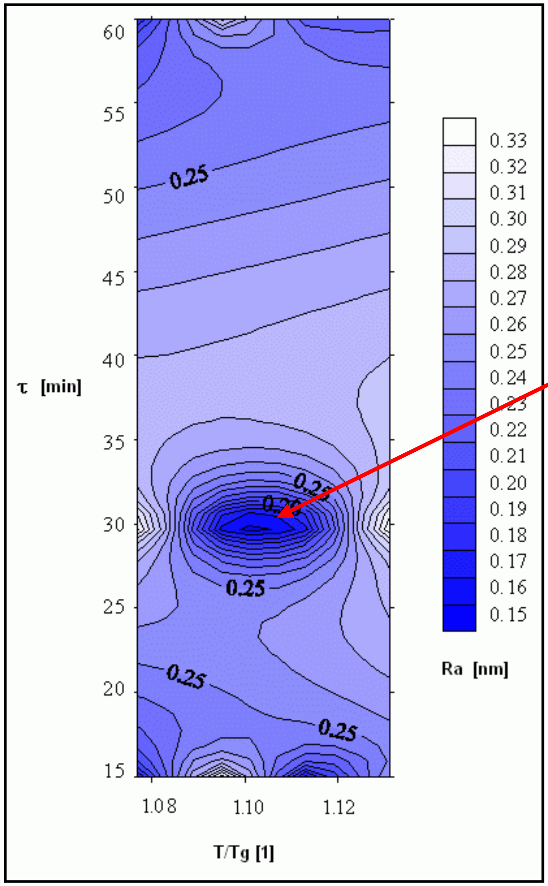
thermally formed glass, parabolic profile

R = 150 mm, 100 x 150 x 0.75 mm, PV from ideal shape

~0.7 mm in the best case recently

Measuring of the roughness after slumping – Optimizing GTF parameters

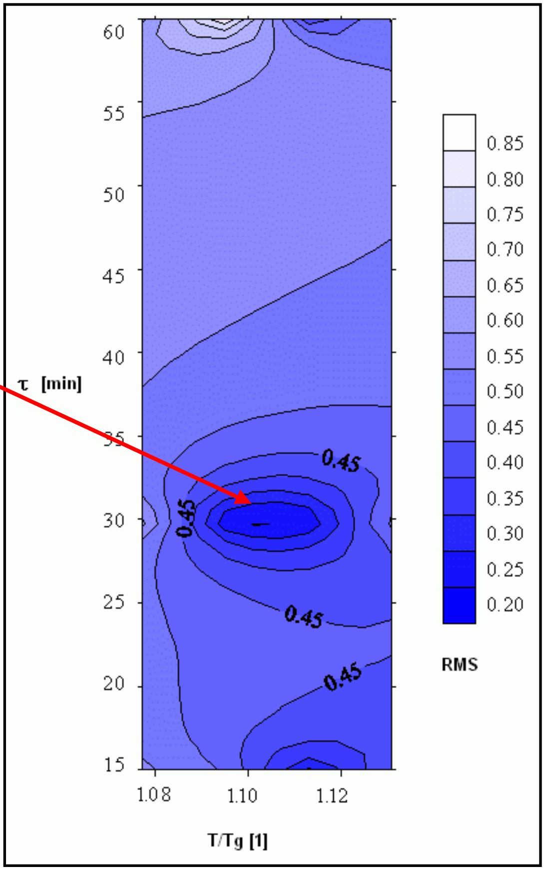
Interferometer Zygo, bent glass, 75 x 25 x 0.75 mm, optimization using > 100 samples formed at different conditions



Ra [nm]

minimal value

Optimizing the parameters of GTF

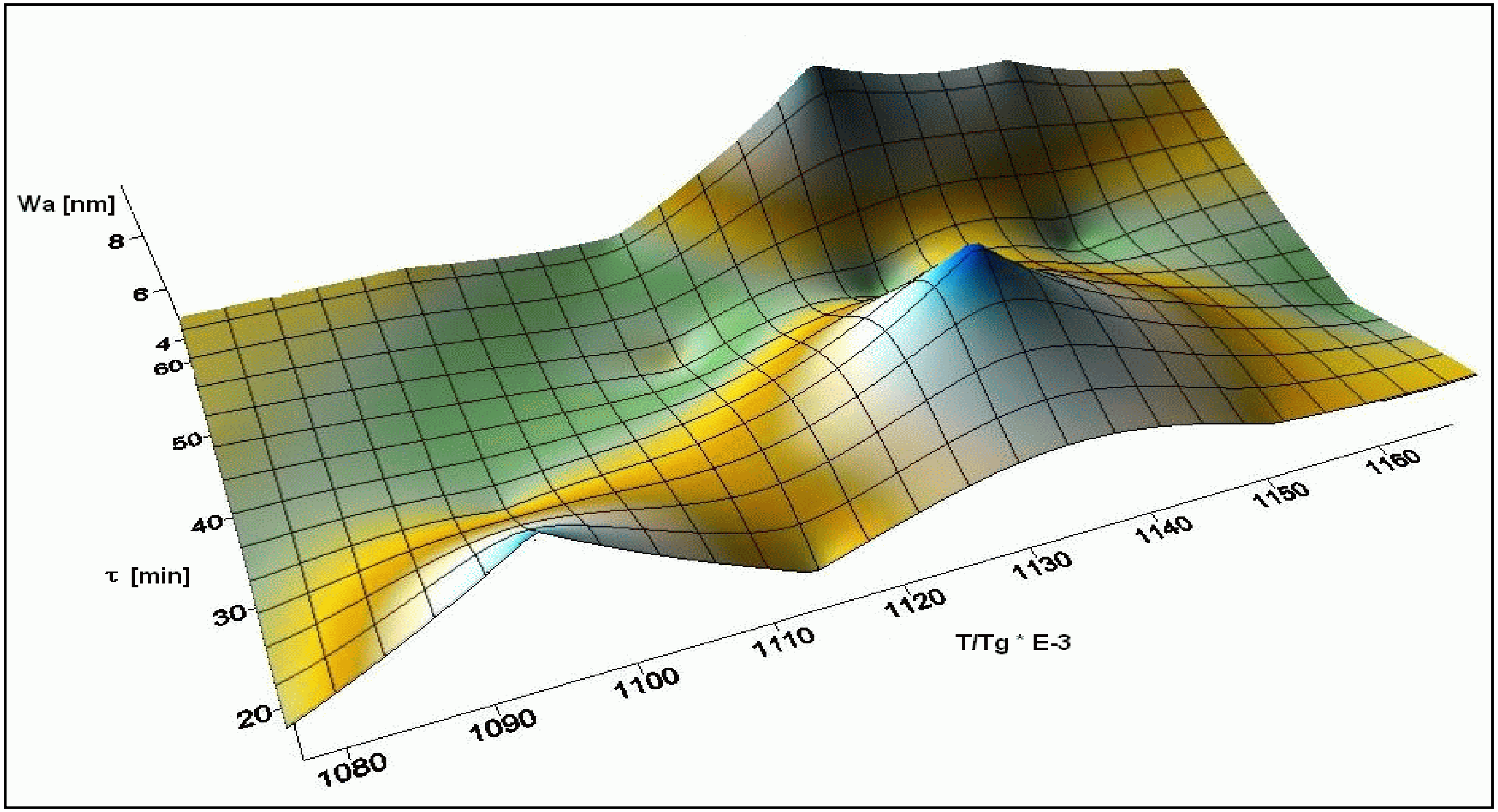


RMS[nm]

Waviness of the surface as function of time and temperature of GTF

based on TH profilometer measurements of numerous samples

(75 x 25 x 0.75 mm, R = 150 mm) - optimization



Comparing Glass and Si wafers

Motto:

Neither recently used standard Si wafers nor borosilicate glass foils are optimized for X-ray optics applications.

No high-quality X-ray optics without superior substrates.

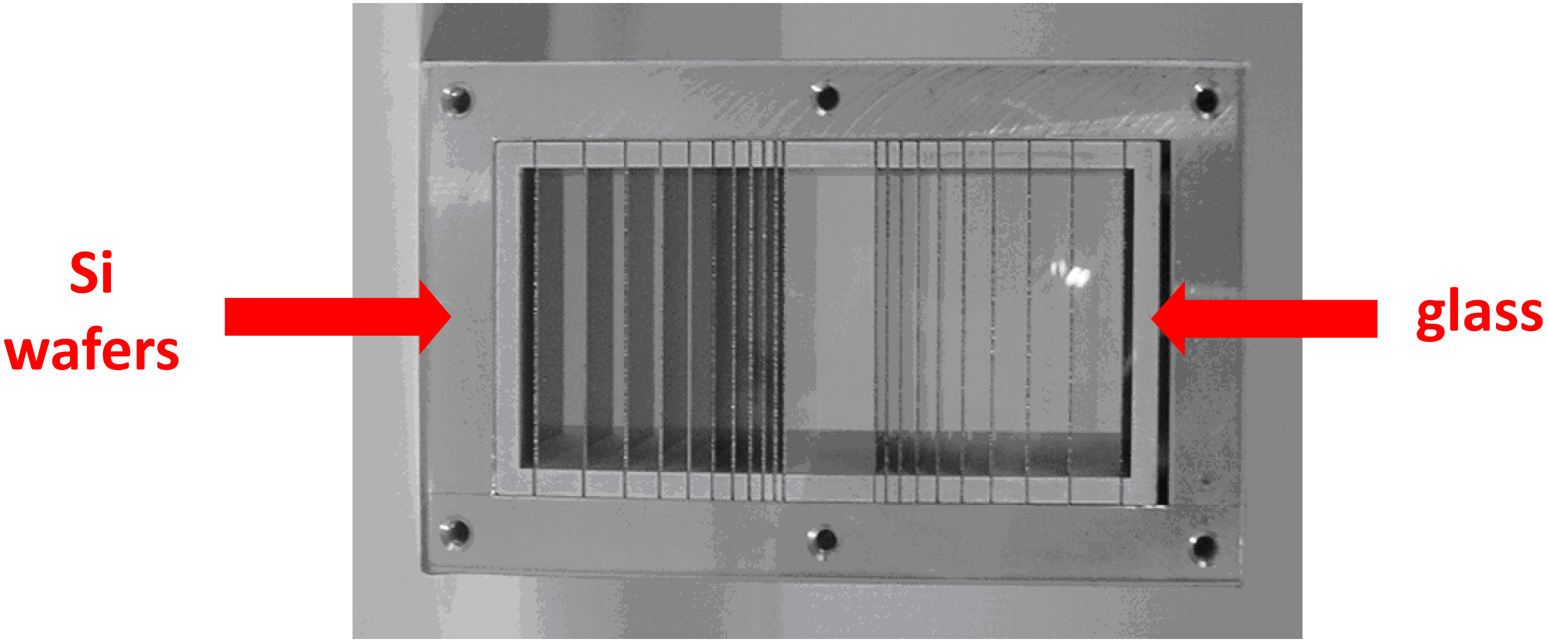
Si & glass substrate

	Borosilicate glass	Soda lime glass	Silicon wafer
density [g/cm3]	2.51	2.51 (2.72)	2.35
stress-optical coefficient [m^2/N]	$3.44 * 1.02 * 10^{-12}$	$3.2 * 1.02 * 10^{-12}$	x
Young's modulus [GPa]	72.9	69	131
$T_{transformation}$ [°C]	557	555(662)	1418
lin. ther. coef. of expansion [K^{-1}]	$7.2 * 10^{-6}$	$9.6\ (4.5) * 10^{-6}$	$2.6 * 10^{-6}$

Si vs. Glass substrate

	Si	Glass
Price per unit substrate	-	+
Range of available thicknesses	-	+
Surface microroughness&waviness	+	-
Possible irradiation damages	+	-
Bending to precise surfaces	-	+
Volume density	+	-
Thermal expansion	+	-
Long-term stability	+	-
Stiffness	+	-

Si vs. Glass Test Module



Test module for tests performance of glass foils vs. shaped Si wafers. Test elliptical Kirkpatrick-Baez optical system, focus 0.5 m, 58 x 50 x 100 mm, glass foils 40 x 40 x 0.3mm, Si wafers 40 x 40 x 0.4 mm

MPO (Multipore/Mosaic Optics) vs. MFO optic

	MPO	MFO
ML deposition	-	+
Gold coating	-	+
Need of bonding	-	+
Mechanical stability	+	-
Long-term stability	-	+
Stress propagation	-	+
Need for supporting structure	+	-
Loss of area by ribs	-	+
Need for complicated treatment of the Si wafers (ribs, conical polishing, lithography etc.)	-	+

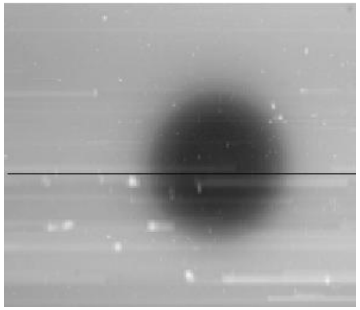
Irradiation tests

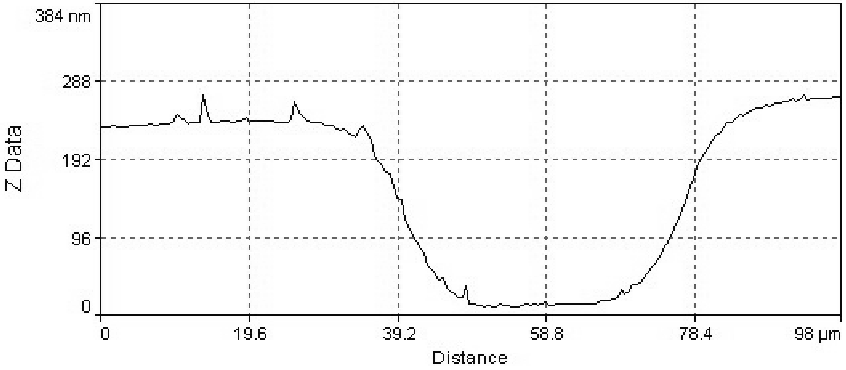
Irradiation Tests

- **The mirrors based on thin Si or glass substrates will work in space environment with influences that can damage the superior shape and surface quality.**
- **Irradiation by electrons and protons may play the main role. Depends on satellite orbit (radiation belt passages).**
- **Detailed tests on various substrates are necessary to exploit the possible damage.**

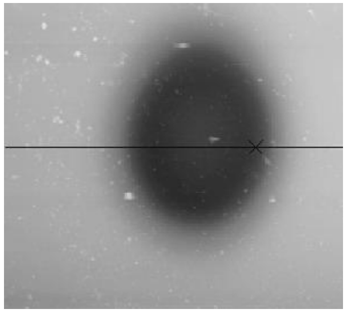
Preliminary Results

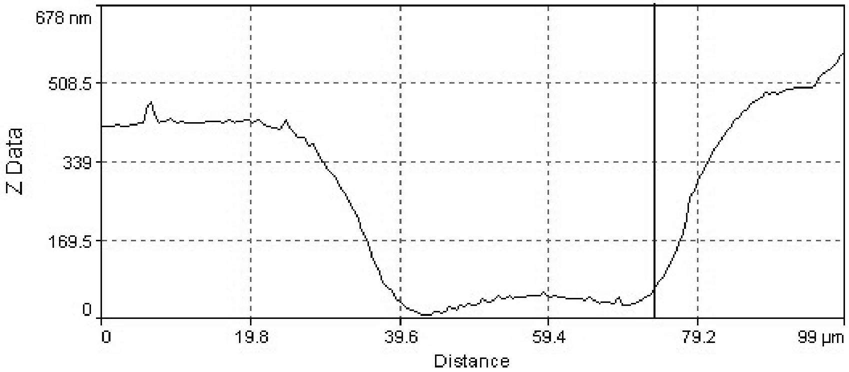
vitreous silica





borosilicate glass





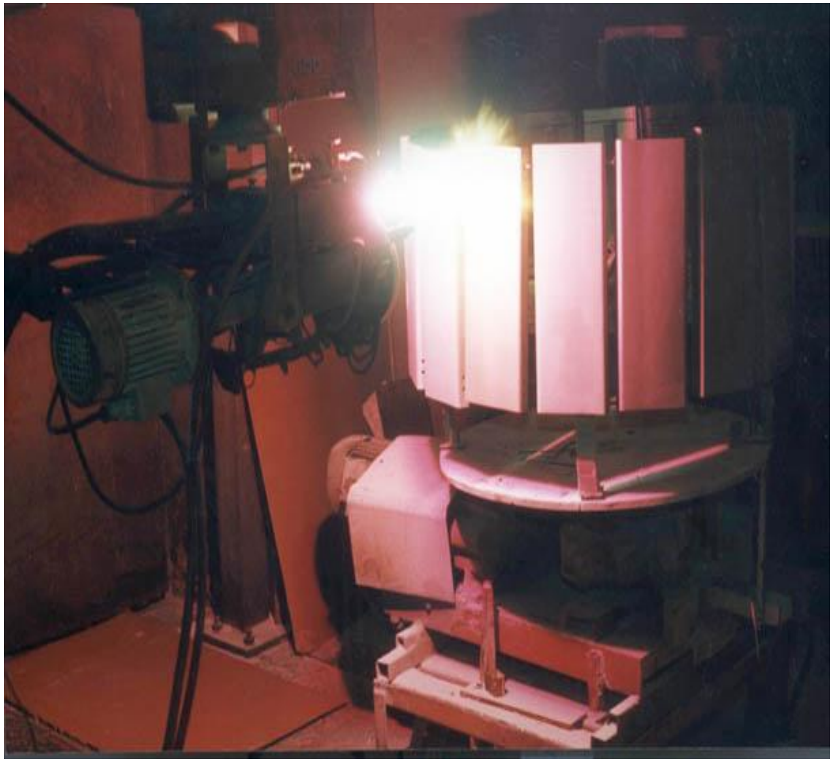
Electron beam energy	50 keV
Time	10 min
Diameter of beam	60 µm
Absorbed current	50 nA

A typical AFM picture of the irradiated glass surface spot (left). The line shows the track of the following scan, results of which are on right.

Irradiation Tests – Preliminary Results

- The mirrors based on thin glass substrates may be negatively influenced.
- Irradiation by electrons and protons may cause changes in shape and in microroughness.
- Si wafer substrates seem to be more resistant than glass.
- More detailed tests on various substrates are necessary to exploit the possible damage in detail.

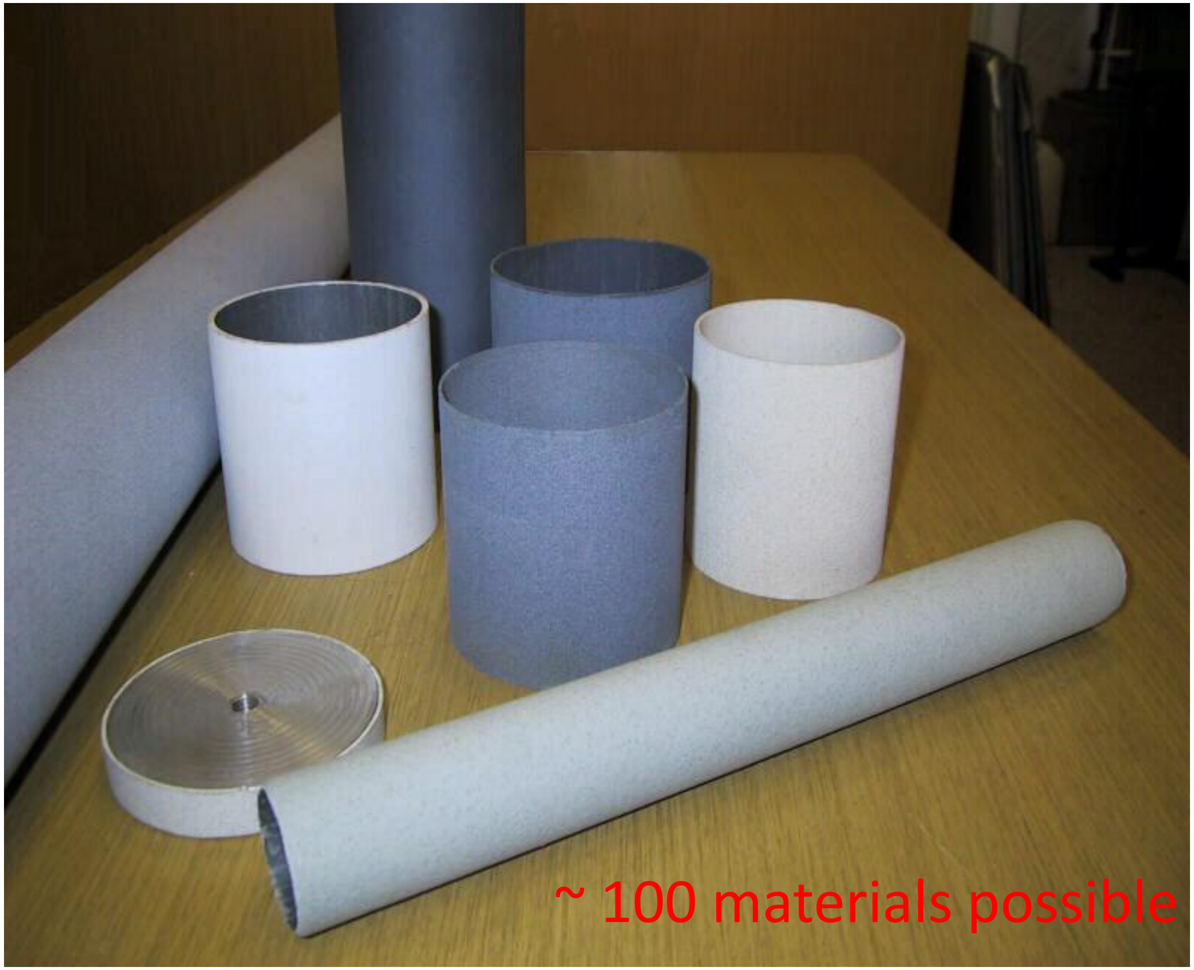
Light ceramics replication by plasma spraying



Plasma spraying facility at the IPP of the AS CR Prague

- Alternative to electroforming replication.
- Lighter than electroforming
- Volume densities 1.5….3.5.
- Under study for future large space X-ray telescopes.
- Promising and innovative: Li (volume density 0.5) based materials .
- Very large number of possible materials (~ 100) , needs optimization.
- Alumina tested in the past is NOT the optimal choice!

The replicated light ceramics shells with thickness 0.2 … 5 mm



~ 100 materials possible

Amorphous-glossy metal alloys

- so far most applications because magnetic properties
- little exploited for X-ray mirrors
- excellent mechanical properties if compared with crystalline materials
- mechanical stiffness 4 times better .... weight reduction by 4
- three available technologies
- only one useful for X-ray mirrors (electrodeposition)

Why amorphous alloys?

Material	Elasticity limit [kg/mm^2]	Tensile strength [kg/mm^2]	Young's modulus [kg/mm^2]	Crystaliz. Temperat[K]
$Fe_{80}P_{13}C_7$ Amorphous	235	310	12 400	420
$Fe_{72}Cr_8P_{13}C_7$ Amorphous	342	385		
$Fe_{72}Ni_8P_{13}C_7$ Amorphous	210	270		410
$Fe_{60}Ni_{20}P_{13}C_7$ Amorphous	190	250		390
$Fe_{40}Ni_{40}P_{14}B_6$ Amorphous		175	14 700	
$Fe_{32}Ni_{36}Cr_{14}P_{12}B_6$ Amorphous		195	14 700	
$Ni_{49}Fe_{29}Al_2P_{14}B_6$ Amorphous		→ 200	9 100	
$Ni_{1-x}P_x$ Amorphous				~ 560
$(Fe_{0.5}Ni_{0.5})_{1-x}P_x$ amorphous				~ 590
$(Co_{0.5}Ni_{0.5})_{1-x}P_x$ amorphous				~ 620
Ni crystalline		→ 50	18 000	
Cu Crystalline	3	20	10 000	
Fe Crystalline	5	35	19 000	
Carbon steel	33	75	20 000	
Molybdenium Steel	60	150	22 000	

The amorphous alloys exhibit ~ 4 times better stifftness ... the shells may be 4 times thinner

Glassy carbon

- completely new material for X-ray mirrors
- thick layers of GC difficult, thin layers feasible
- resin- silica glass plate-curing-peeling-carbonizing
- bulk density ~ 1.5 g/cm^{-3} (and even 0.6 for layers with large porosity) - less than any other material for X-ray mirror production
- the bending strength 50-200 MPa
- the Young modulus 20-32 GPa
- CTE ~ 1 x 10^{-6} C^{-1}
- group with expertise in GC available in Prague
- First samples expected early 2010

Summary

- **Samples of test X-ray mirrors have been produced by using novel technologies.**
- **Shaped thin glass mirrors and Si mirrors have been successfully produced.**
- **Both approaches show promising results with PV values around 1 mm justifying further efforts in these directions.**
- **Improved Si wafers with parameters better suited to meet the X-ray optics applications developed and tested.**
- **Alternative technologies still exist little exploited so far**

The End

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