Seminar

What Material Scientists Do With Optics:
In-situ Ellipsometry Mesurements

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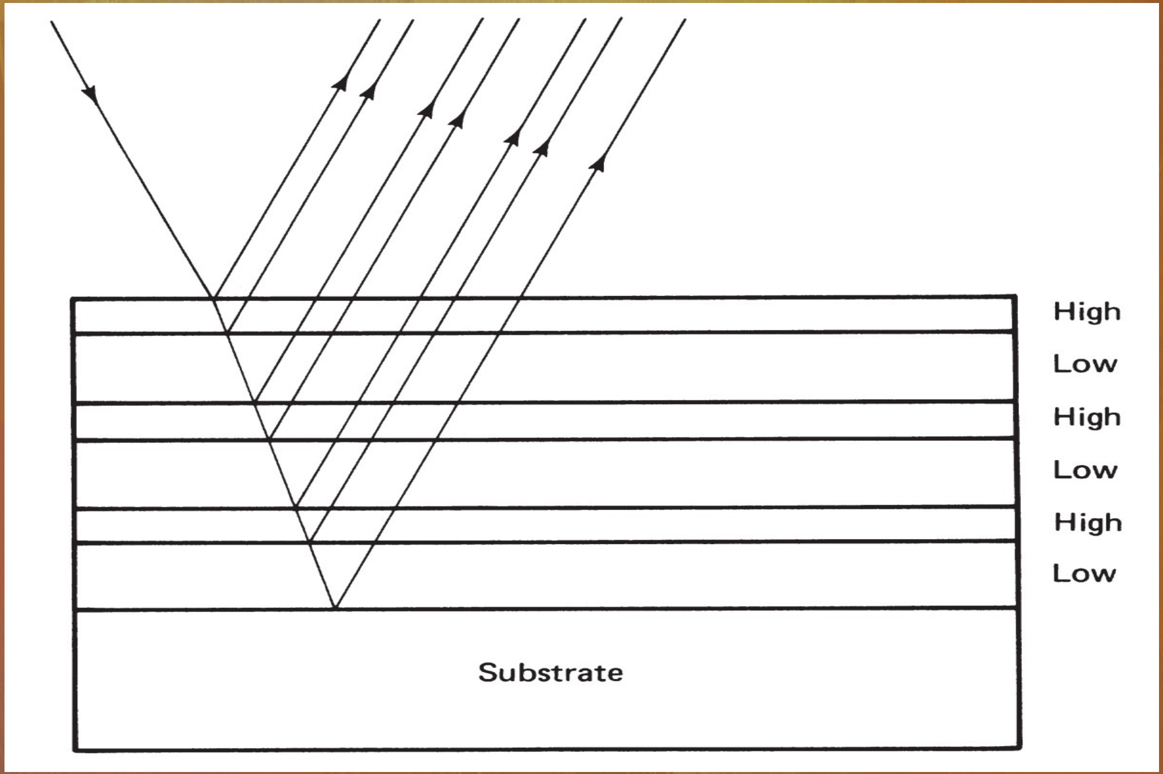
Outline

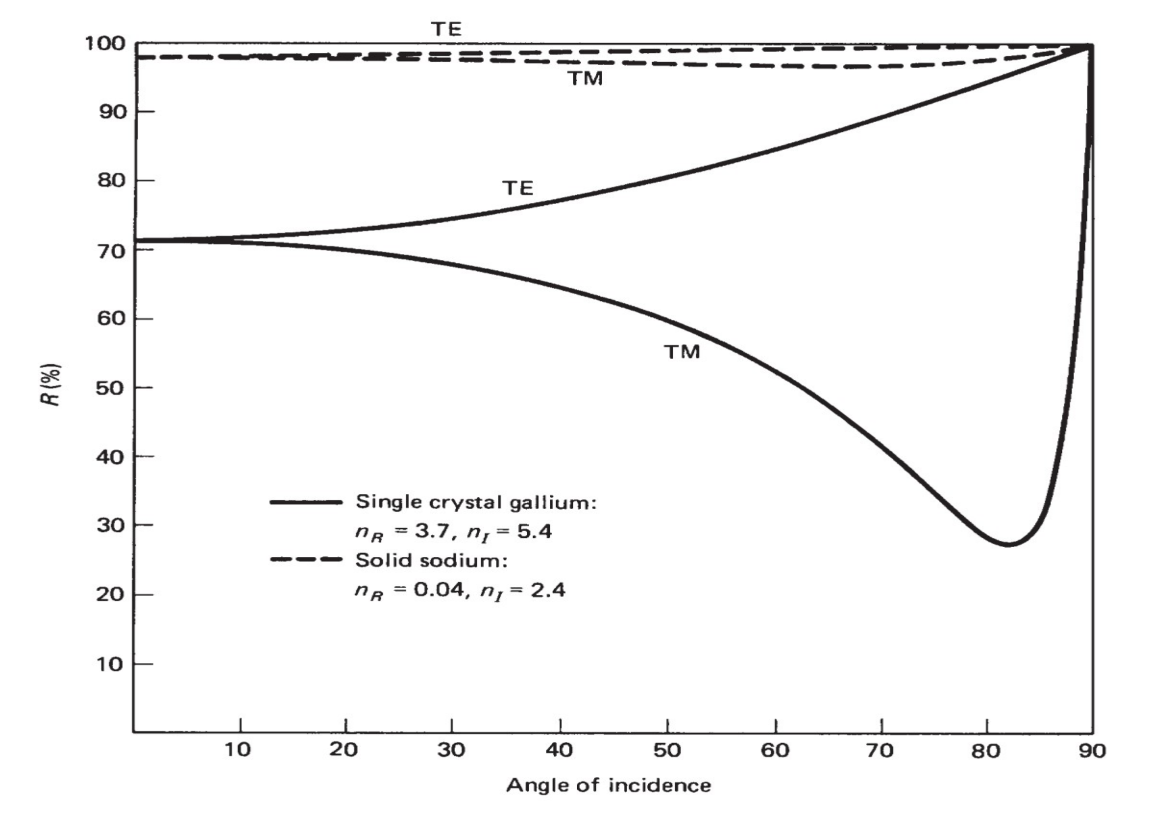
- Plane-polarized reflectance spectroscopy
  - Hydrogen plasma tube design
  - Experimental data on deposition enhancement
  - Temperature calibration
- Conclusion

General Procedure



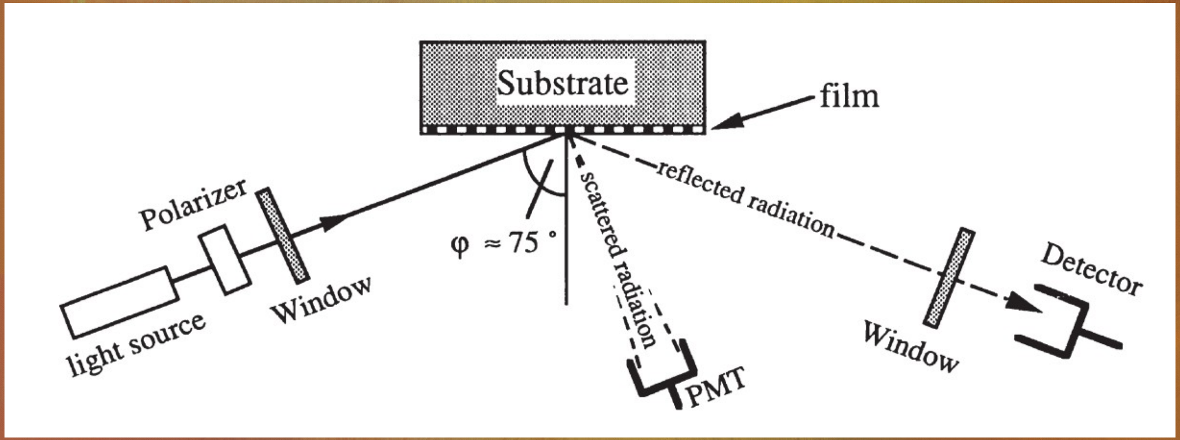
Sinusoidal output due to constructive/destructive interference during growth

Increase Optical Sensitivity



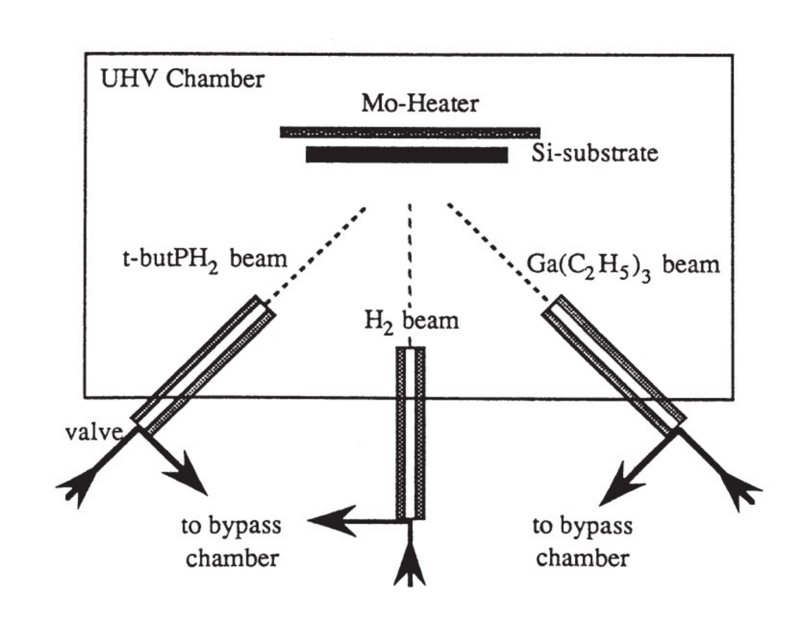
Brewster angle for maximum transmission

Plane–polarized Reflectance Spectroscopy (PRS) with Laser Light Scattering (LLS)



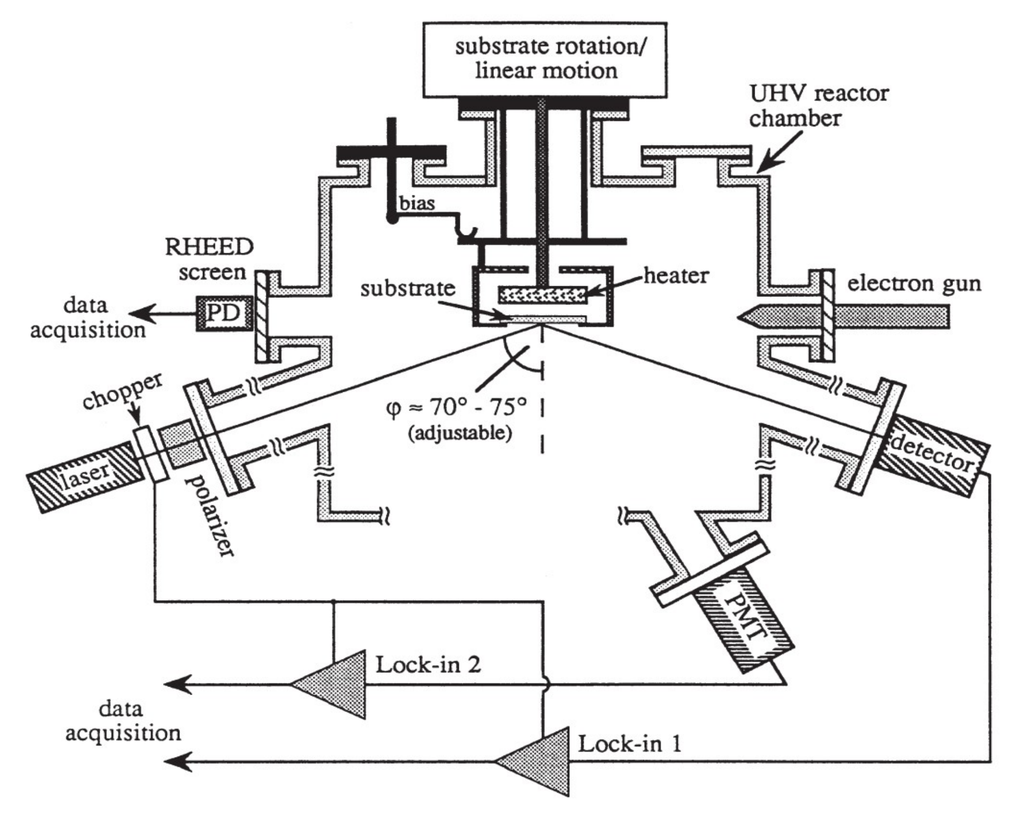
Pseudo-Brewster angle for Si = 75 deg

Mass Flow Diagram for Chemical Beam System



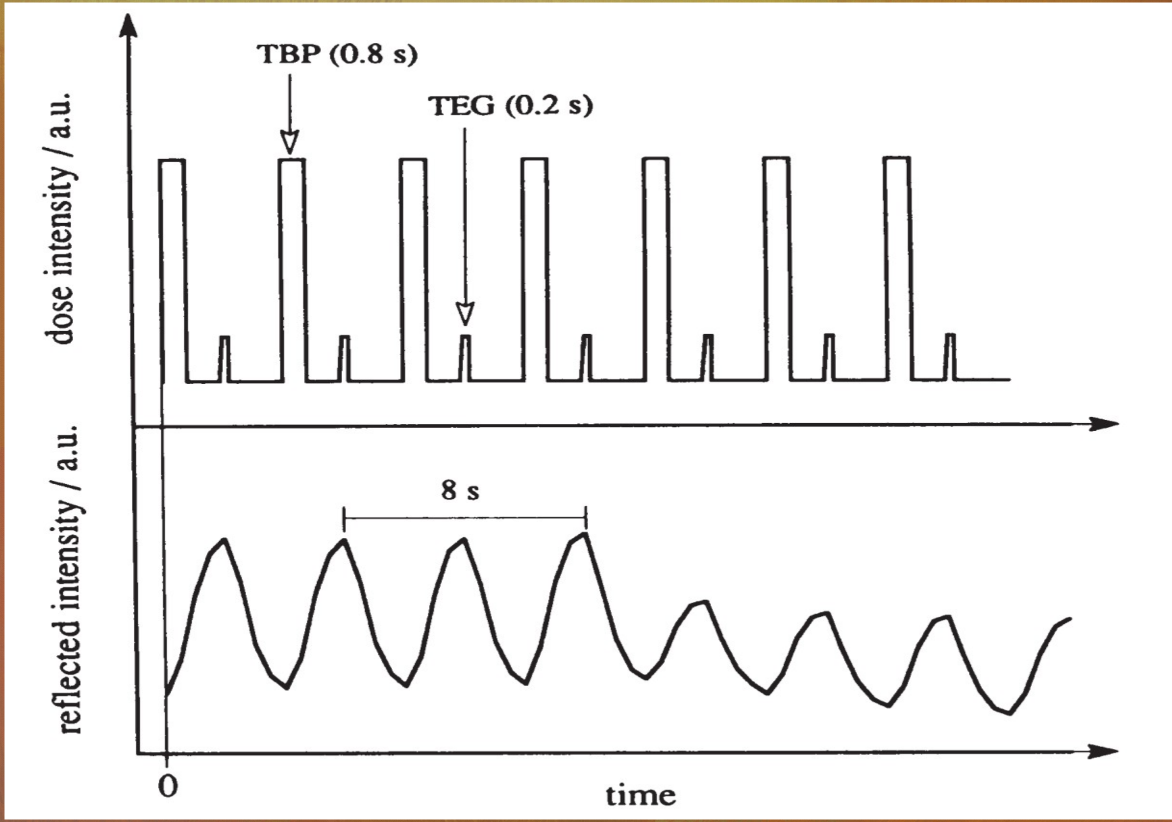
Pulsed metalorganic precursors

Instrumental View of Chemical Beam System



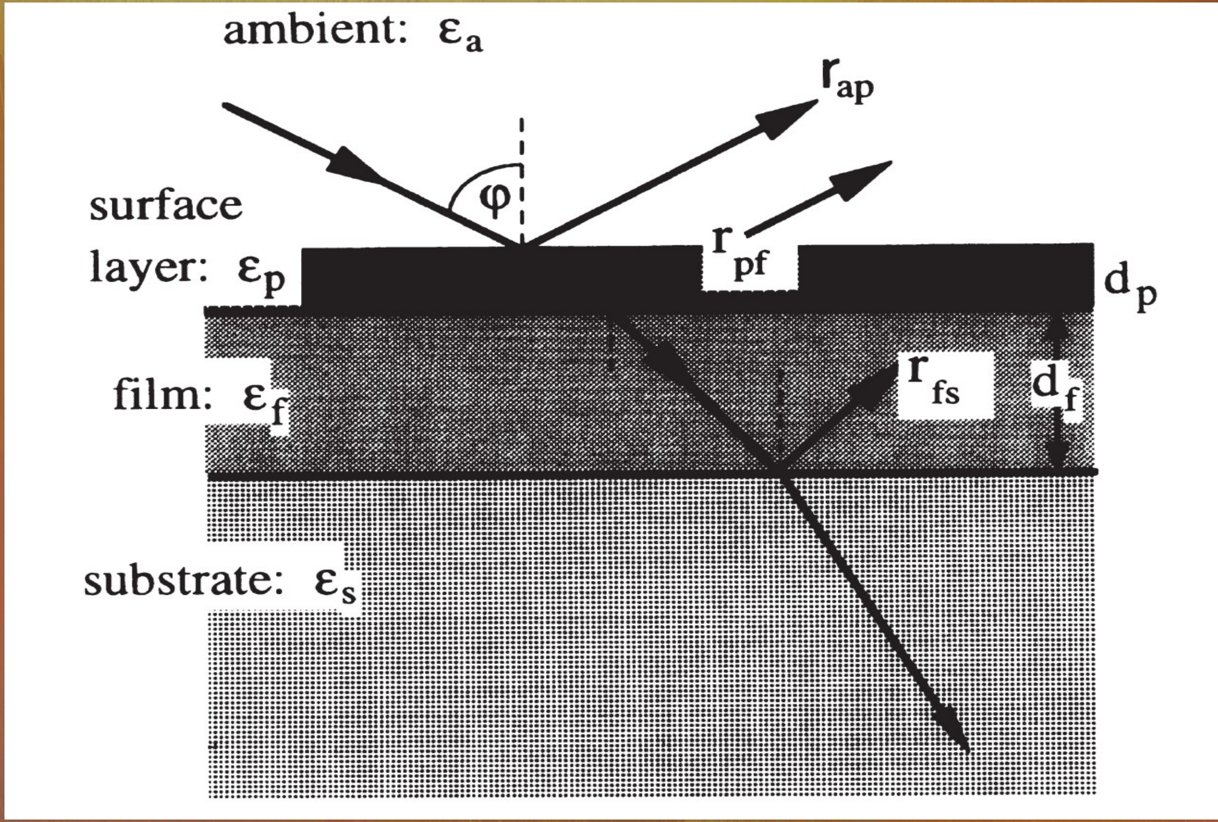
Plane Polarized Reflectance at 70 and 75 deg

Vapor Source Pulse Sequence



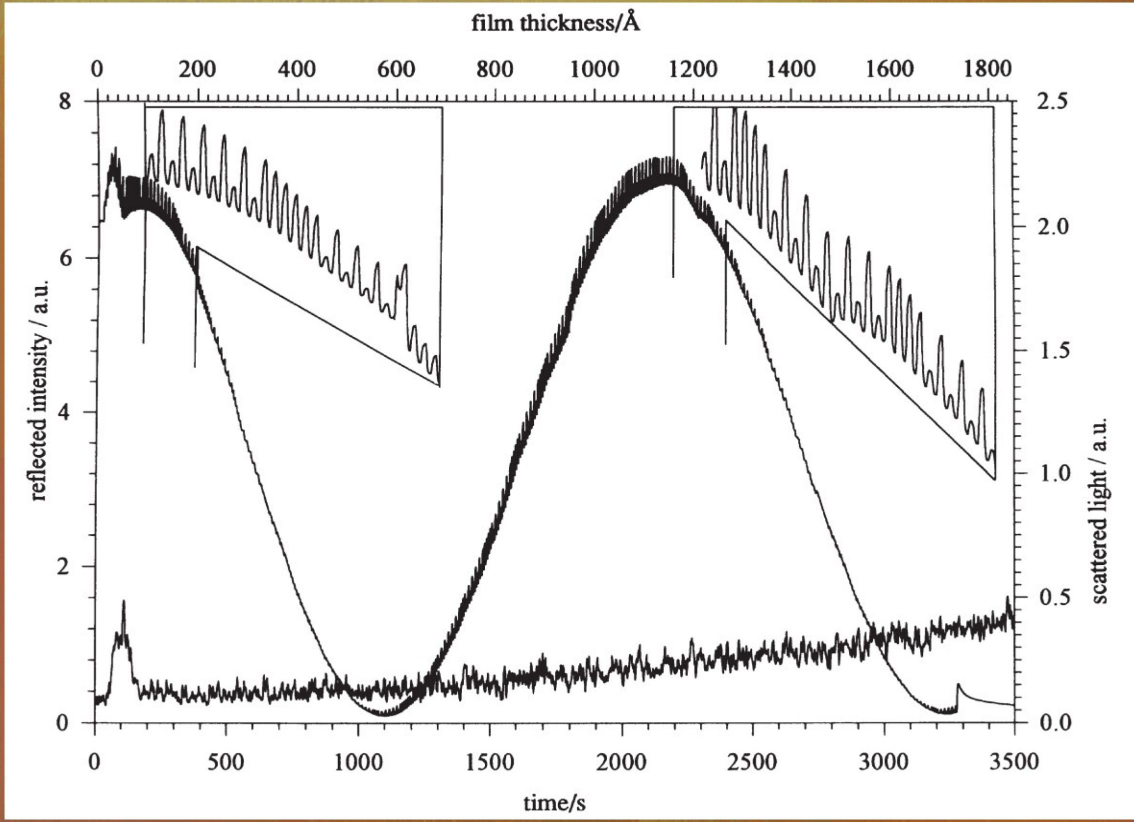
Reaction layer on the order of 2 monolayers thick

Optical Mediums and Interfaces



Chemical beam epitaxy of GaP on Si

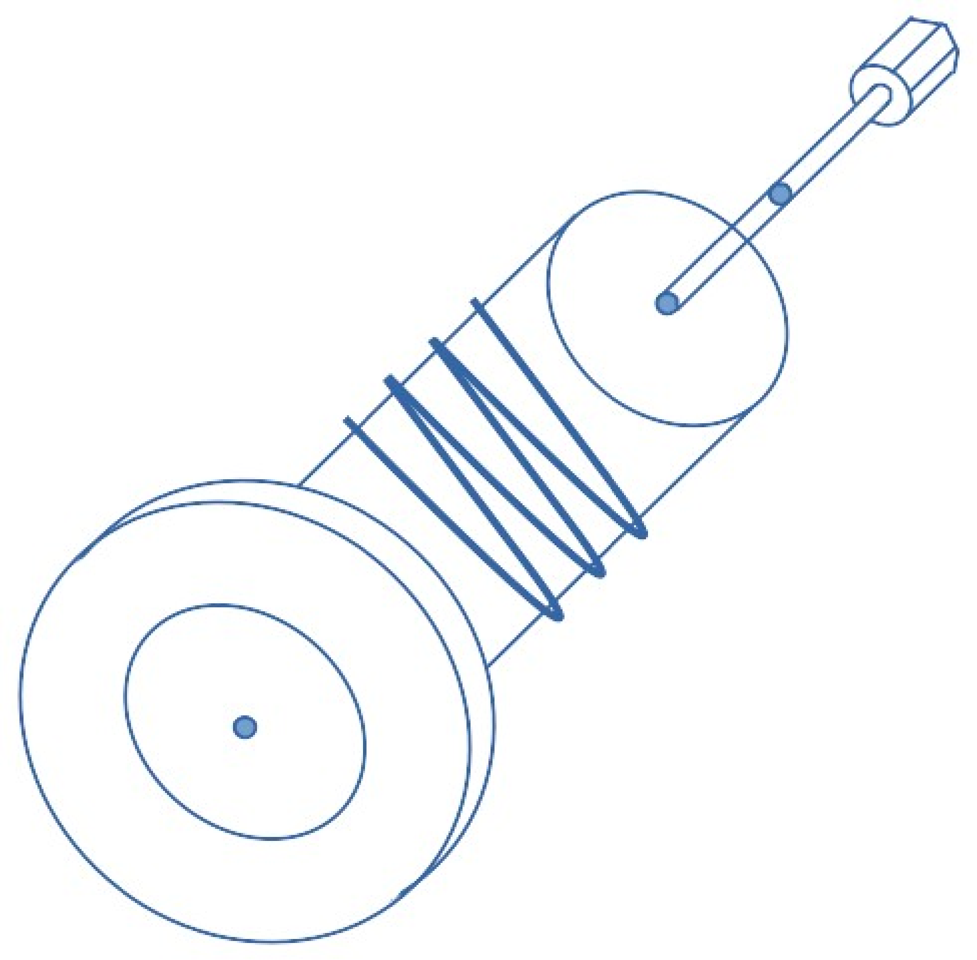
Plane–polarized Reflectance Signal (Upper)



Laser light scatter intensity (Lower)

Plasma Tube

Property	
Tube diameter	25 mm
Tube length	100 mm
Orifice size	1.5 mm
Pathlength: orifice to substrate	150 mm
Tube Pressure @ 5 sccm	100 mTorr
RF copper coil	1/8 in



RF induction: 49 W @ 13.56 Mhz

Mass Flow Parameters

Pressure	=	5×10^{-5}	*Torr*
H_2	=	5	*sccm*
TBP	=	0.8	
TEG	=	0.04	

Pulsing:	Main	Bypass
TBP	$0 \rightarrow 0.8$ s	
TEG	$1.5 \rightarrow 1.8$	$1.8 \rightarrow 2.5$

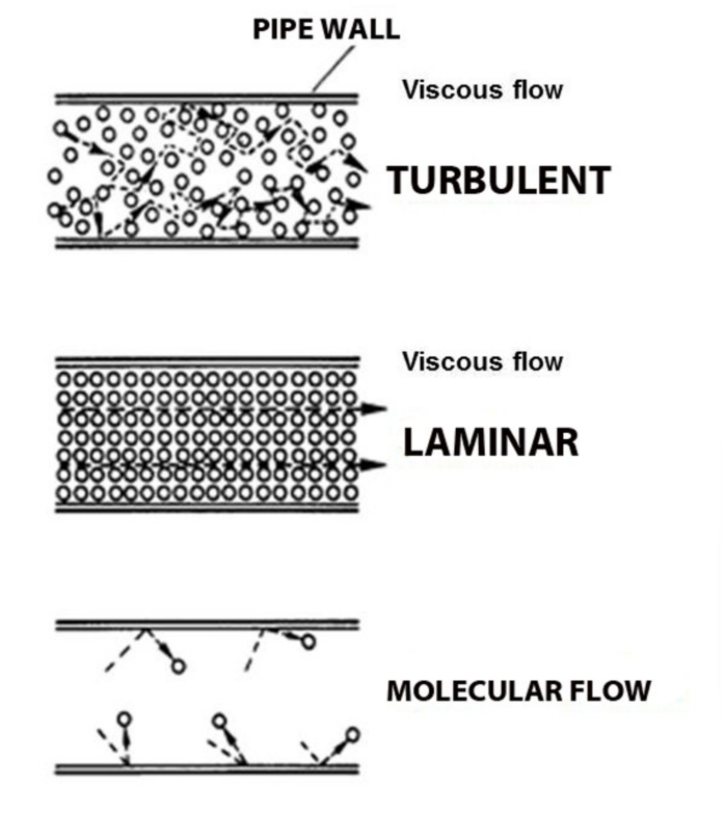
During an overall growth cycle of 3 s, TEG flows into the main growth chamber for 0.3 s, is vented into a separate bypass chamber for 0.7 s, and has an accumulated flux to the surface:

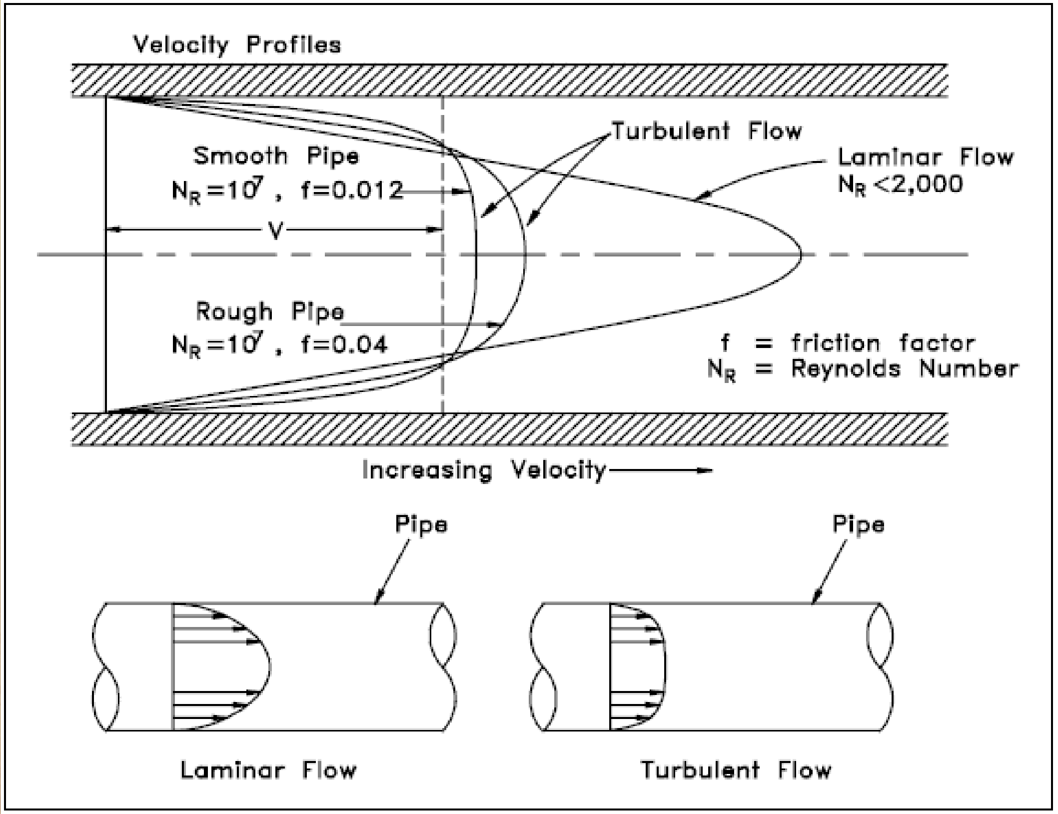
$$NetTEG = 0.04sccm\left(\frac{2.3s}{3s}\right) = 0.031sccm$$

Therefore, the realized TBP/TEG flow ratio becomes 26.

GaP heteroepitaxy

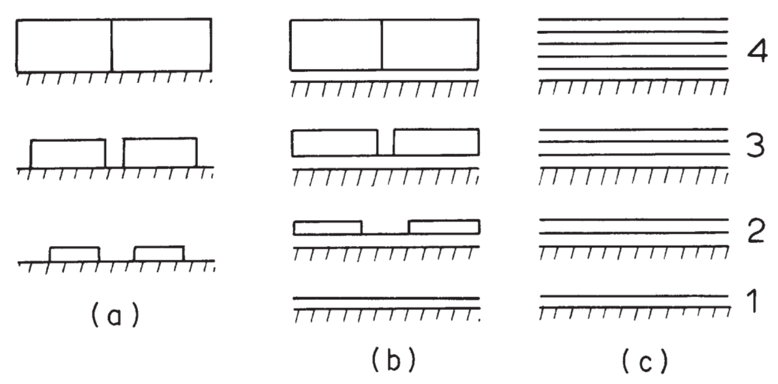
Flow Regimes





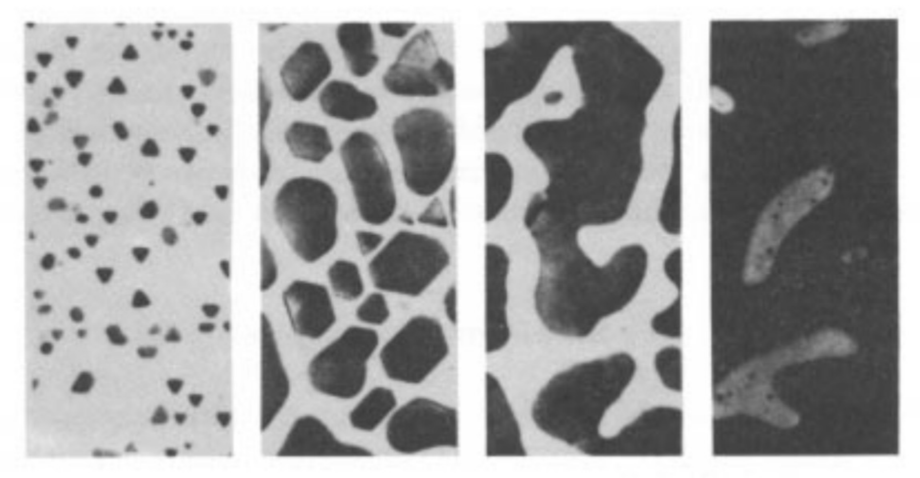
Mean free path > critical dimension

Growth Models



Issues
(1) lattice constant mismatch
(2) wetting angle [polarity]

3D



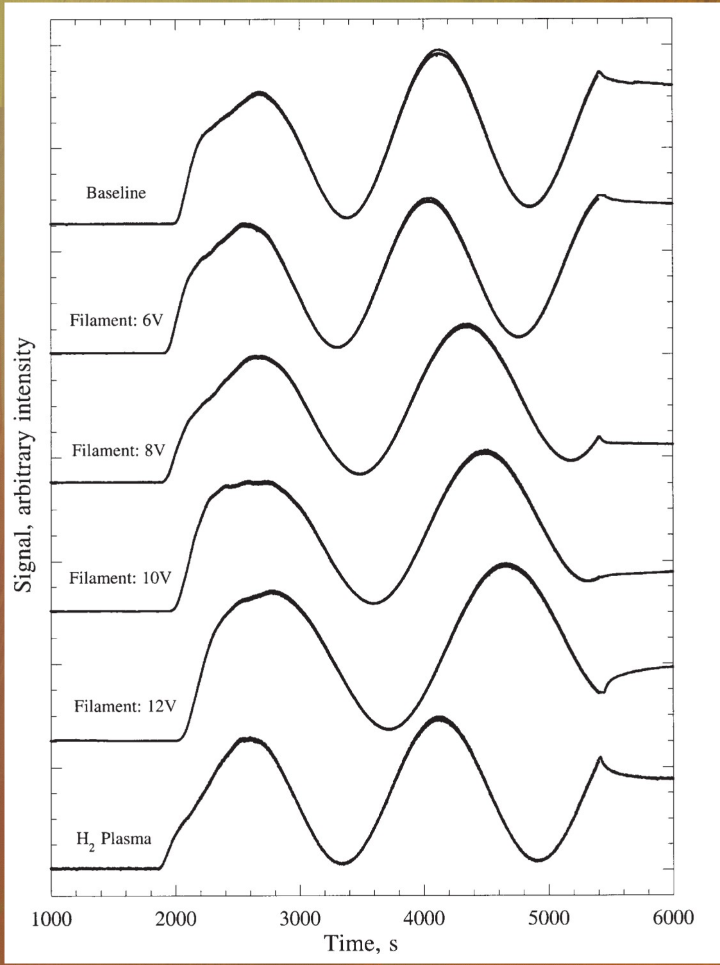
Heteroepitaxy
(a) 3D
(b) layer → 3D @ critical thickness

Homoepitaxy
(c) layer by layer

Reflection @ 75 deg

Filament power:
16 to 49 W

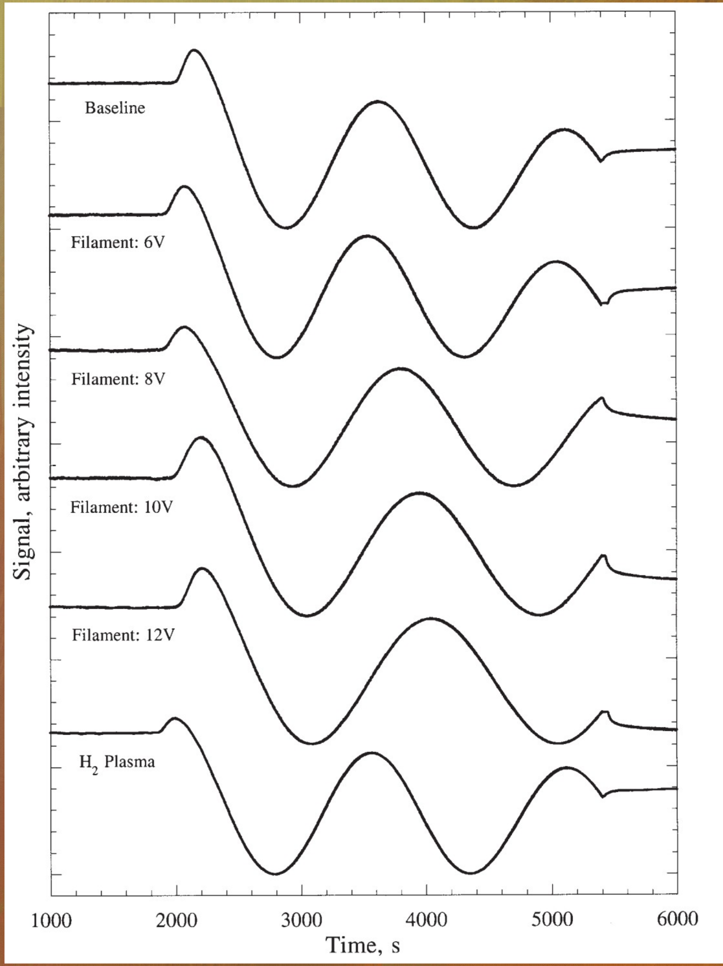
Deposition enhancement



Reflection @ 70 deg

Filament power:
16 to 49 W

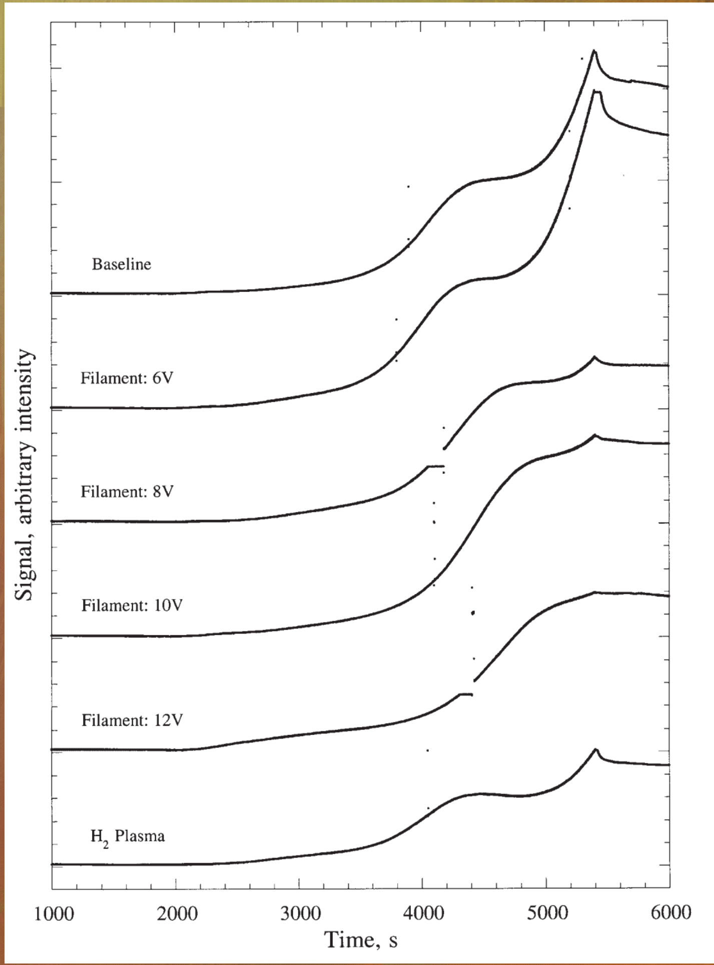
Deposition enhancement



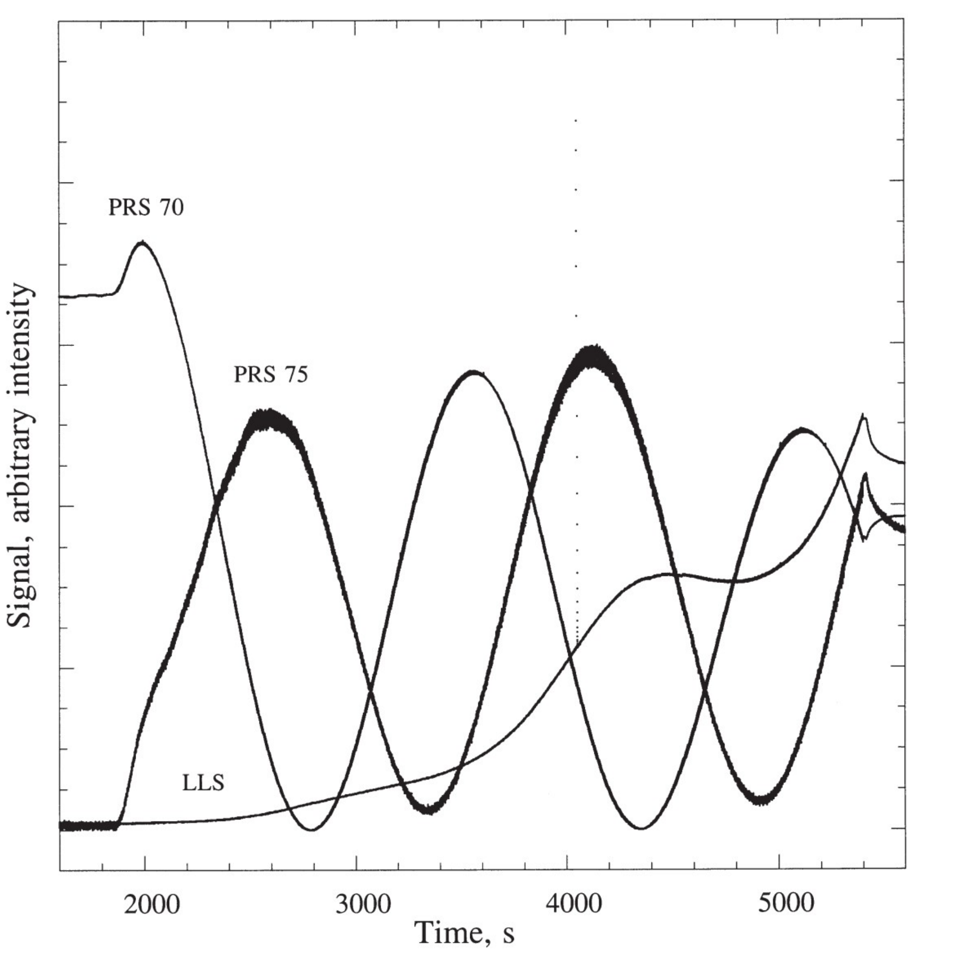
Scatter

Filament power:
16 to 49 W

Deposition enhancement



Hydrogen Plasma



GaP heteroepitaxy

Temperature Measurement

$\varepsilon_1 = 1$
Vacuum

$\phi_1 = 75$ deg
Si Brewster angle

$\lambda = 632.8$ nm
HeNe laser

$$r_{k,k+1} = \frac{\varepsilon_{k+1}\sqrt{\varepsilon_k - \varepsilon_1 \sin^2\phi_1} - \varepsilon_k\sqrt{\varepsilon_{k+1} - \varepsilon_1 \sin^2\phi_1}}{\varepsilon_{k+1}\sqrt{\varepsilon_k - \varepsilon_1 \sin^2\phi_1} + \varepsilon_k\sqrt{\varepsilon_{k+1} - \varepsilon_1 \sin^2\phi_1}}$$

where: r = *complex reflectivity or Fresnel coefficient*
ε = *overall dielectric constant*
ϕ = *incident angle from the normal*

$$r_{1,2} = \frac{\varepsilon_2\sqrt{1 - \sin^2\phi_1} - \sqrt{\varepsilon_2 - \sin^2\phi_1}}{\varepsilon_2\sqrt{1 - \sin^2\phi_1} + \sqrt{\varepsilon_2 - \sin^2\phi_1}} = \frac{a - b}{a + b}$$

define: $a = \varepsilon_2\sqrt{1 - \sin^2\phi_1}$
$b = \sqrt{\varepsilon_2 - \sin^2\phi_1}$

$$\varepsilon_2(\Re) = 15 + 2.1x10^{-3}T + 1.5x10^{-6}T^2$$

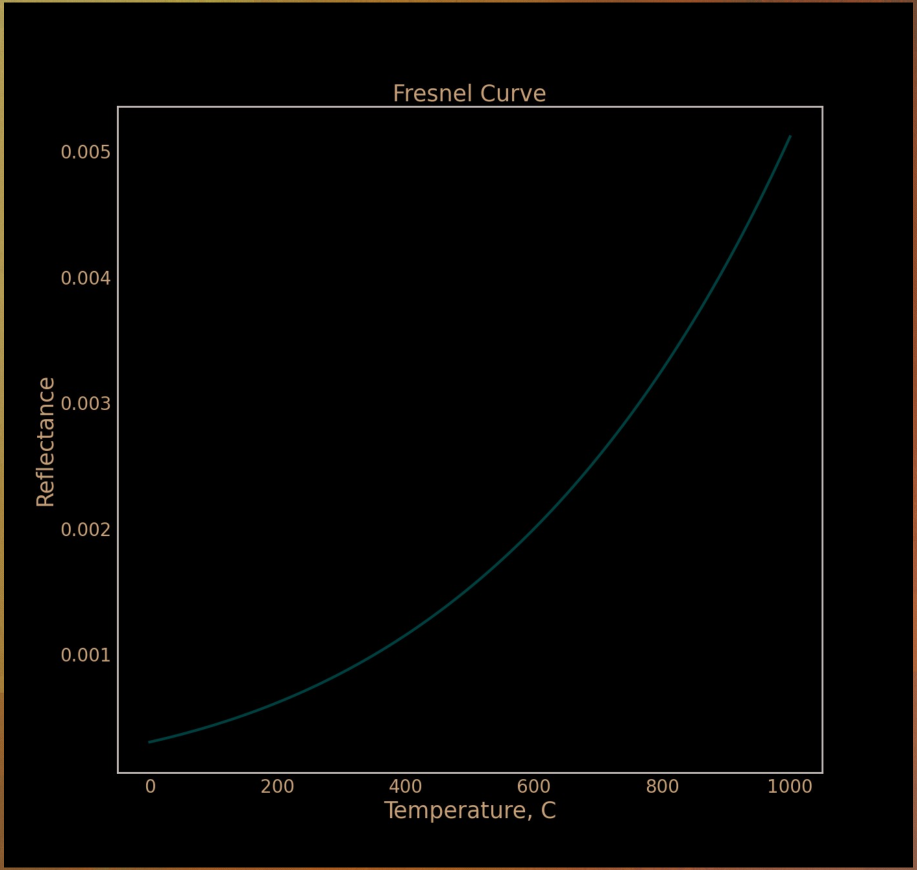
$$\varepsilon_2(\Im) = 0.132e^{0.0025T}$$

where: $\Re$ *denotes real*
$\Im$ *denotes imaginary*
with: T *in* $[C]$

Computation

reflect.py

```python
class reflect:

    def __new__(self):

        lower = 0
        upper = 1010
        step  = 10
        Tarray, Rarray  = self.calc(lower, upper, step)
        self.plot(Tarray, Rarray)

    def calc(lower, upper, step):

        Tarray = []
        Rarray = []
        phi    = 75.*math.pi/180.

        for T in range(lower, upper, step):
            Re=15.+2.1e-3*T+1.5e-6*T*T
            Im=.132*math.exp(2.5e-3*T)
            epsilon=complex(Re,Im)

            a=epsilon*cmath.sqrt(1.-cmath.sin(phi)*cmath.sin(phi))
            b=cmath.sqrt(epsilon-cmath.sin(phi)*cmath.sin(phi))

            r=(a-b)/(a+b)
            R=r*r.conjugate()

            Tarray.append(T)
            Rarray.append(R.real)
        return Tarray, Rarray
```



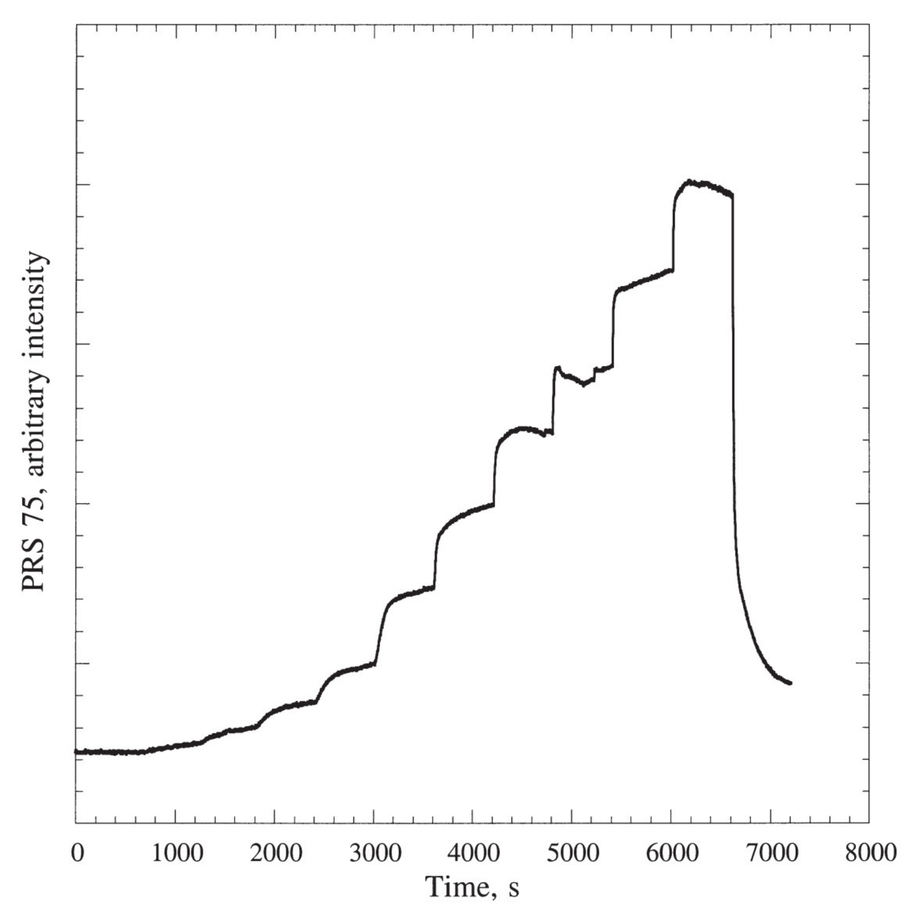
Measurement

$R = rr^*$

$$R = \frac{I}{I_o}$$

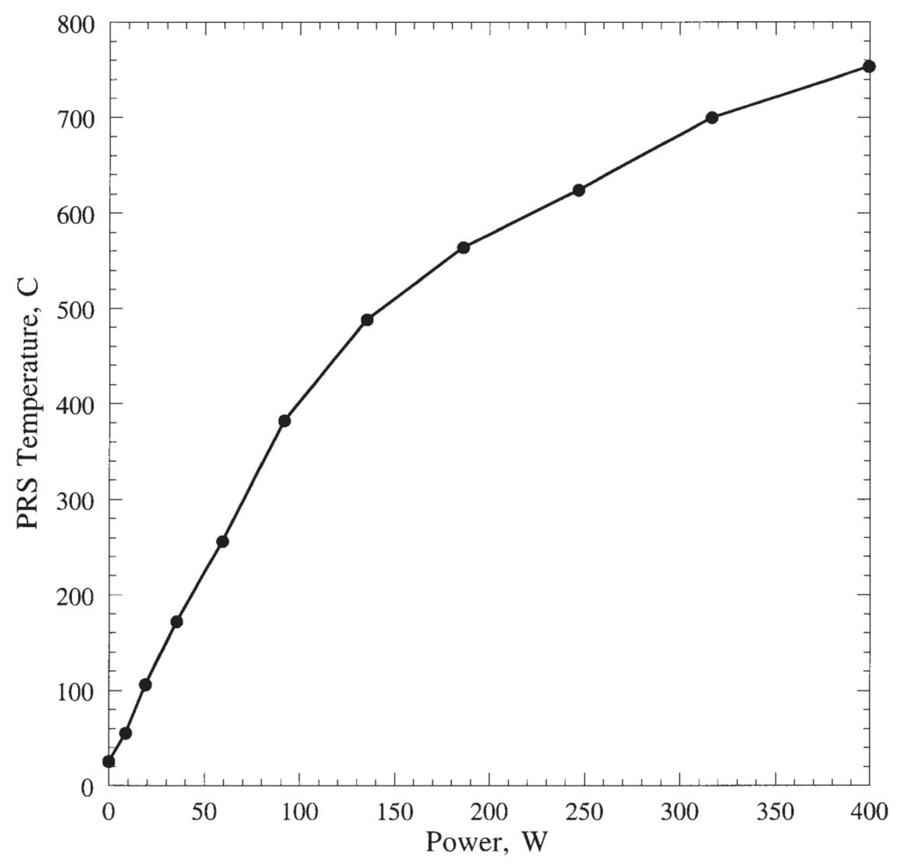
where: R = *true reflectance*
I = *reflectance intensity*
I_o = *beam intensity*

I_o derived from room temperature



Heater Calibration: Si

Current	Voltage	Power	Temperature
0.0 A	0.0 V	0.0 W	25 C
3.0	2.9	8.7	55
4.0	4.8	19.2	106
5.0	7.1	35.5	172
6.0	9.9	59.4	256
7.0	13.1	91.7	382
8.0	16.9	135	488
9.0	20.7	186	564
10.0	24.7	247	624
11.0	28.8	317	700
12.0	33.3	400	753



Reflectance measurements

Conclusion

(1) When polarized light is involved, can get resolution substantially lower than underlying wavelength.

(2) Hydrogen plasma seems to work better than filament methods to stimulate growth surface in molecular flow regime.

(3) A curious plateau appears in the scattered intensity signal at a thickness near 2400 Å, which could be an elastic to plastic stress/strain transformation, leading to 3D growth.

(4) If dielectric constants are known for a particular substance, temperature measurements can readily obtained from reflectance intensity.