

# Extreme conditions



# Extreme?

- P

- T

- E/B

- Light

- Chemistry

- Mechanical

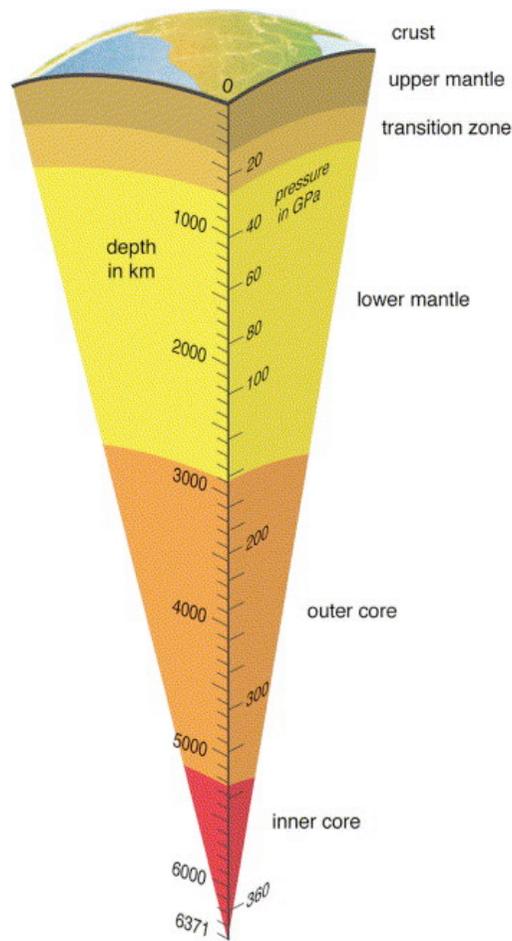
- .....

# Outline of the concepts

- Why Pressure? What is it good for?  
Do we really care?

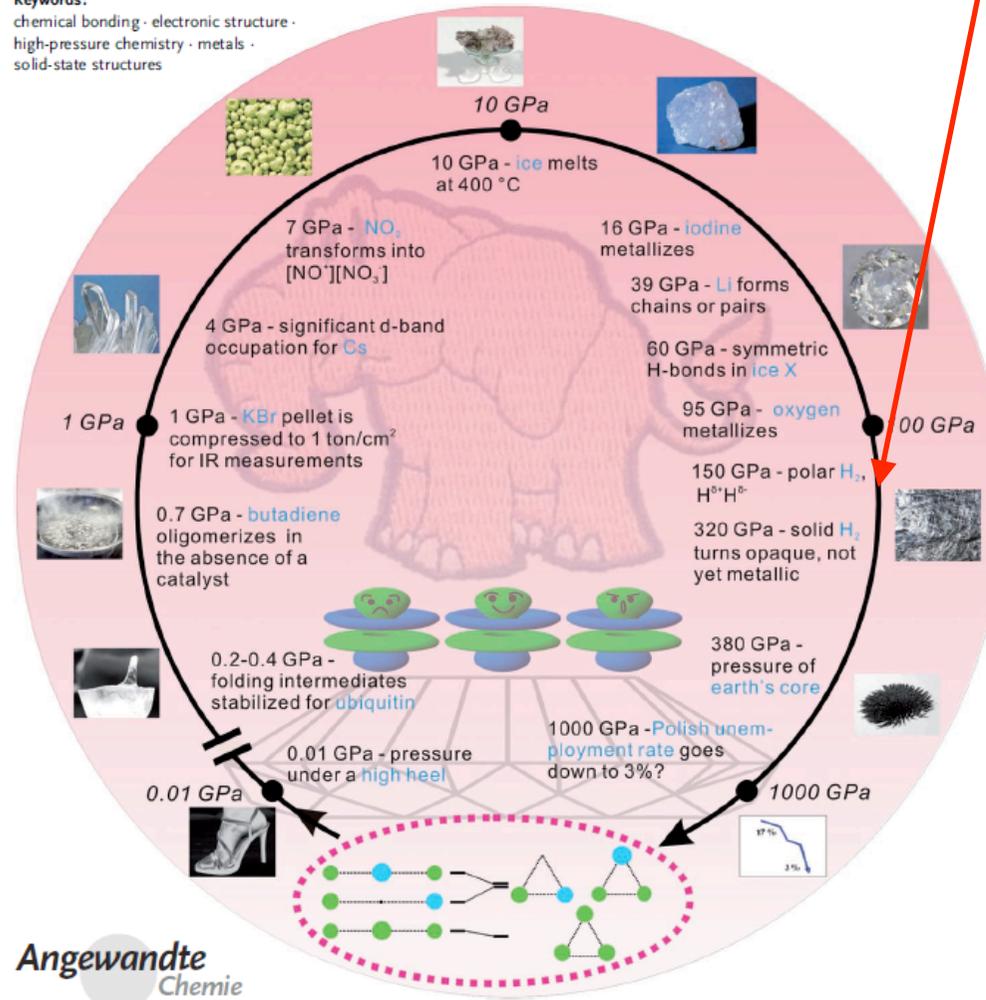
- How can you do it? what are the tricks of the trade? What can I expect out of it?

@ 2000k ad 10GPa nitrogen diamond



**Keywords:**

chemical bonding · electronic structure ·  
high-pressure chemistry · metals ·  
solid-state structures



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Chemie

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- WdV compression
- Relative orientations
- Torsion angles (conformation)
- Bond angles deformation
- Bonds
- Collapse of the molecules



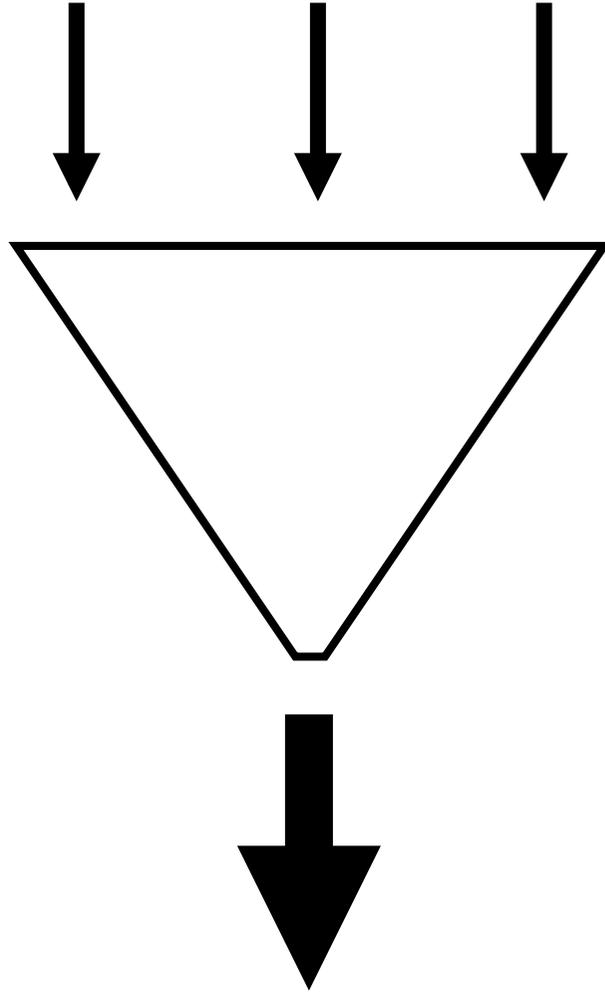
Polymorphism  
↓  
Chemistry

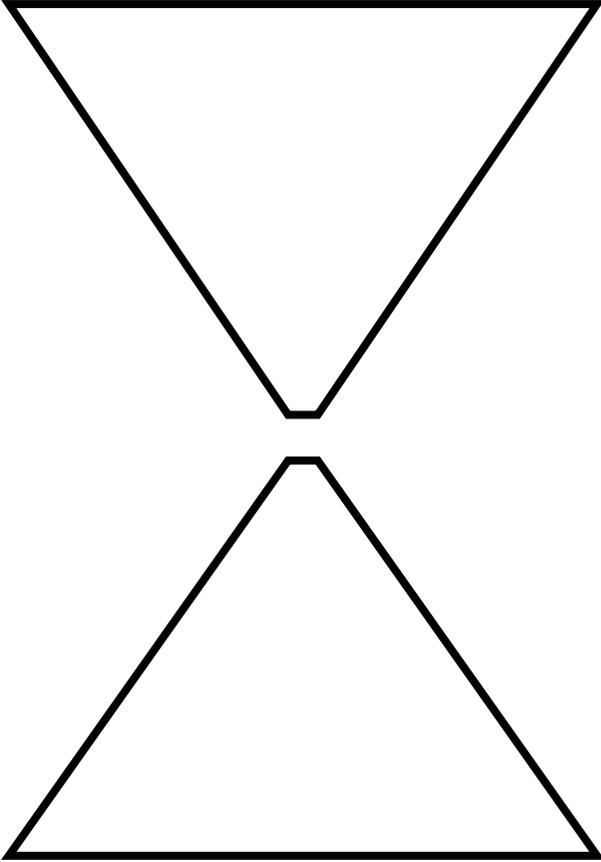
P. Macchi: “there are only few good books of thermodynamics”

Atkins (Physical Chemistry): we can neglect the compressibility of solids

$$P = F/A$$

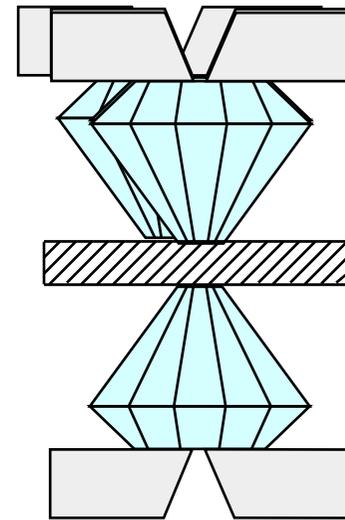






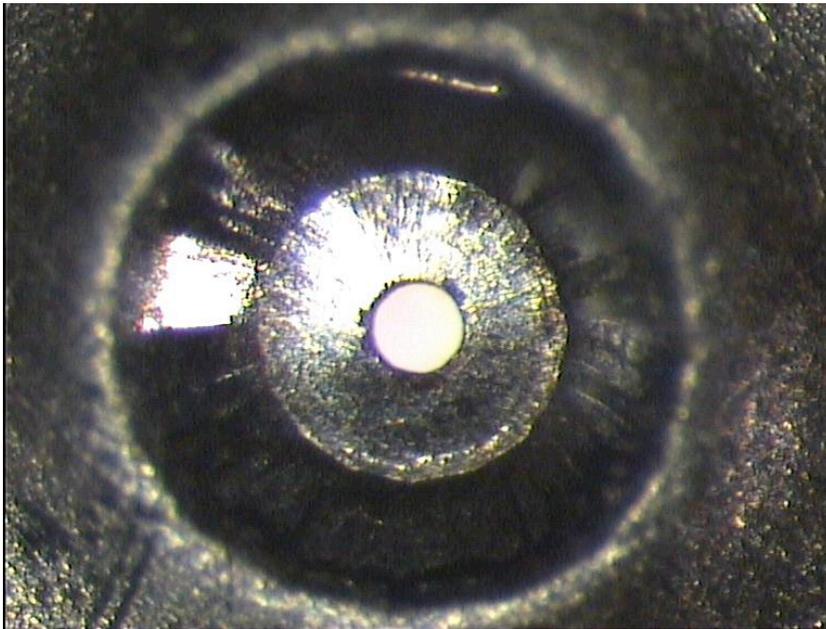
# How is it done?

- Align
- Indent

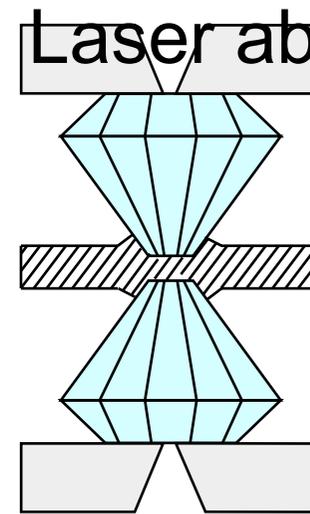


# How is it done?

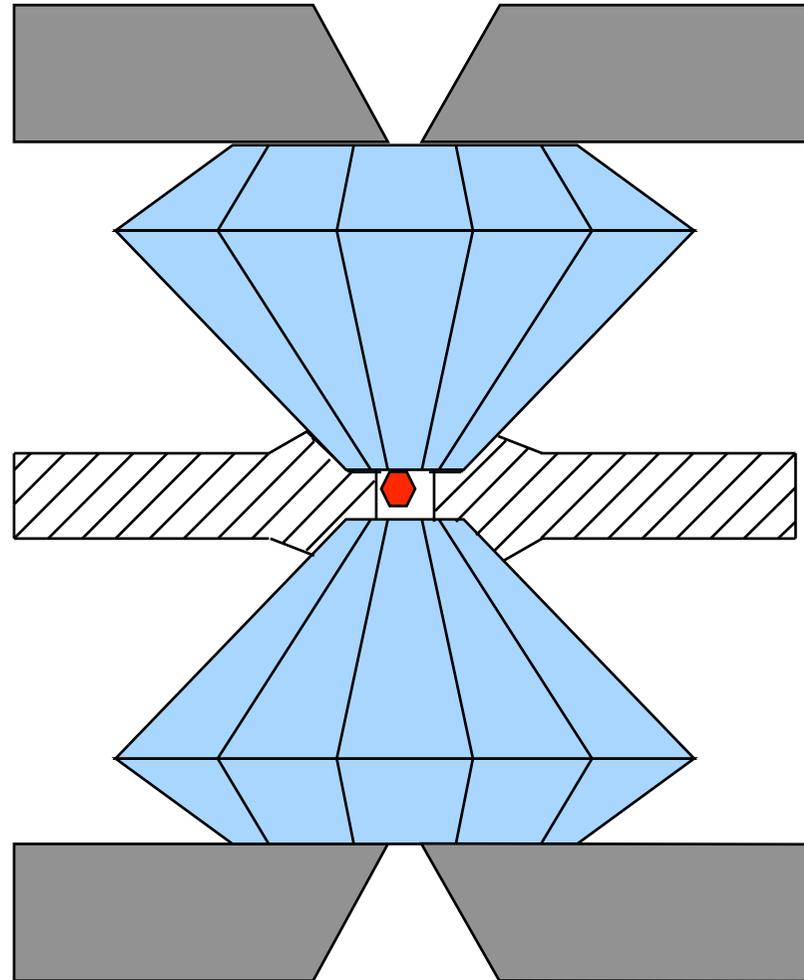
- Align
- Indent
- Drill



- Mechanic
- Spark erosion
- Laser ablation



- Align
- Indent
- Drill
- Fill and close



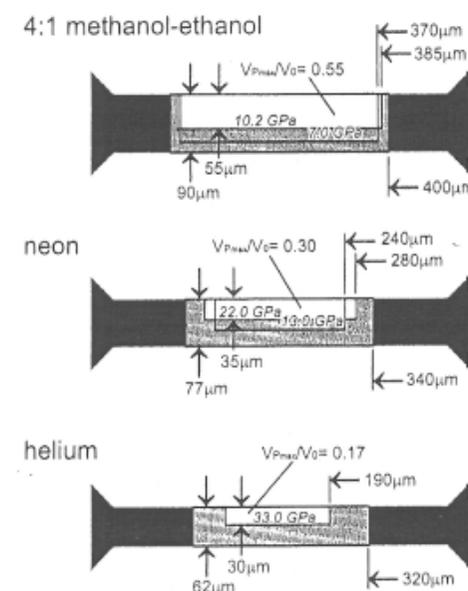
# PTM

P. Macchi: “.. at 7.3 GPa the diffraction becomes broad”

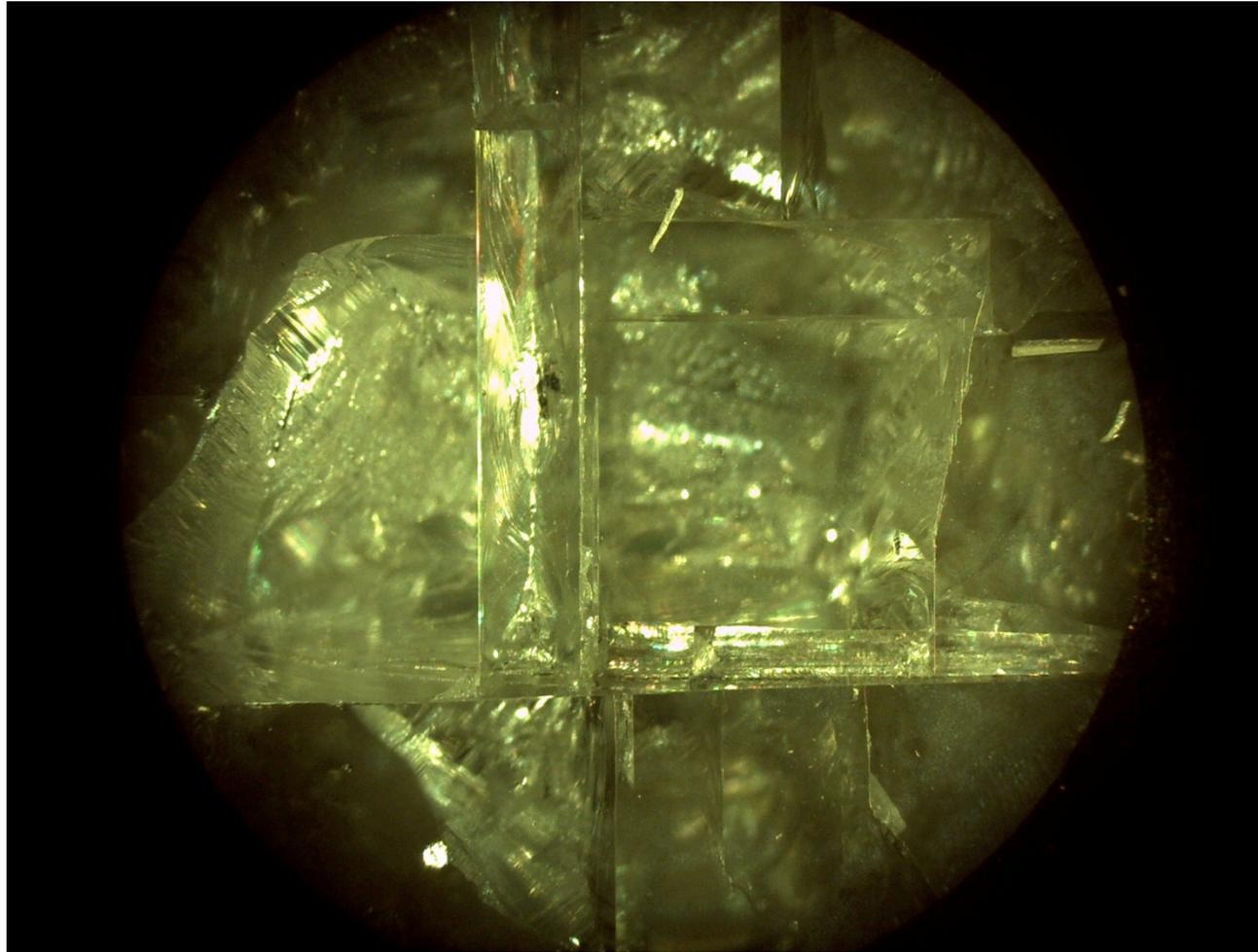
Table 1. Pressure-transmitting media

| Medium                          | freezing $P$ | maximum GPa of (quasi)hydrostaticity | Ref.   |
|---------------------------------|--------------|--------------------------------------|--------|
| Silicon oil                     |              | < 2.0                                | [1]    |
| Water                           |              | 2.5                                  | [1]    |
| Isopropyl alcohol               |              | 4.3                                  | [2]    |
| Glycerine:water (3:2)           |              | 5.3                                  | [3]    |
| Petroleum ether                 |              | 6                                    | [4]    |
| Pentane-isopentane (1:1)        |              | 7.4                                  | [2]    |
| Methanol                        |              | 8.6                                  | [2]    |
| Methanol:ethanol (4:1)          |              | 10.4                                 | [2]    |
| Methanol:ethanol:water (16:3:1) |              | 14.5                                 | [5]    |
| Hydrogen                        | 5.7          | 177                                  | [6]    |
| Helium                          | 11.8         | 60-70                                | [7,8]  |
| Neon                            | 4.7          | 16                                   | [7]    |
| Argon                           | 1.2          | 9                                    | [7]    |
| Xenon                           | ?            | 55                                   | [9,10] |
| Nitrogen                        | 2.4          | 13.0                                 | [11]   |

References: [1] Angel 2000; [2] Piermarini et al 1973; [3] Sidorov and Tsioik 1991; [4] Barnett and Bosco 1969; [5] Fujishiro et al. 1981; [6] Mao and Bell 1979; [7] Bell and Mao 1981; [8] Eremets 1996; [9] Liebenberg 1979; [10] Asaumi and Ruoff 1986; [11] Le Sar et al. 1979.

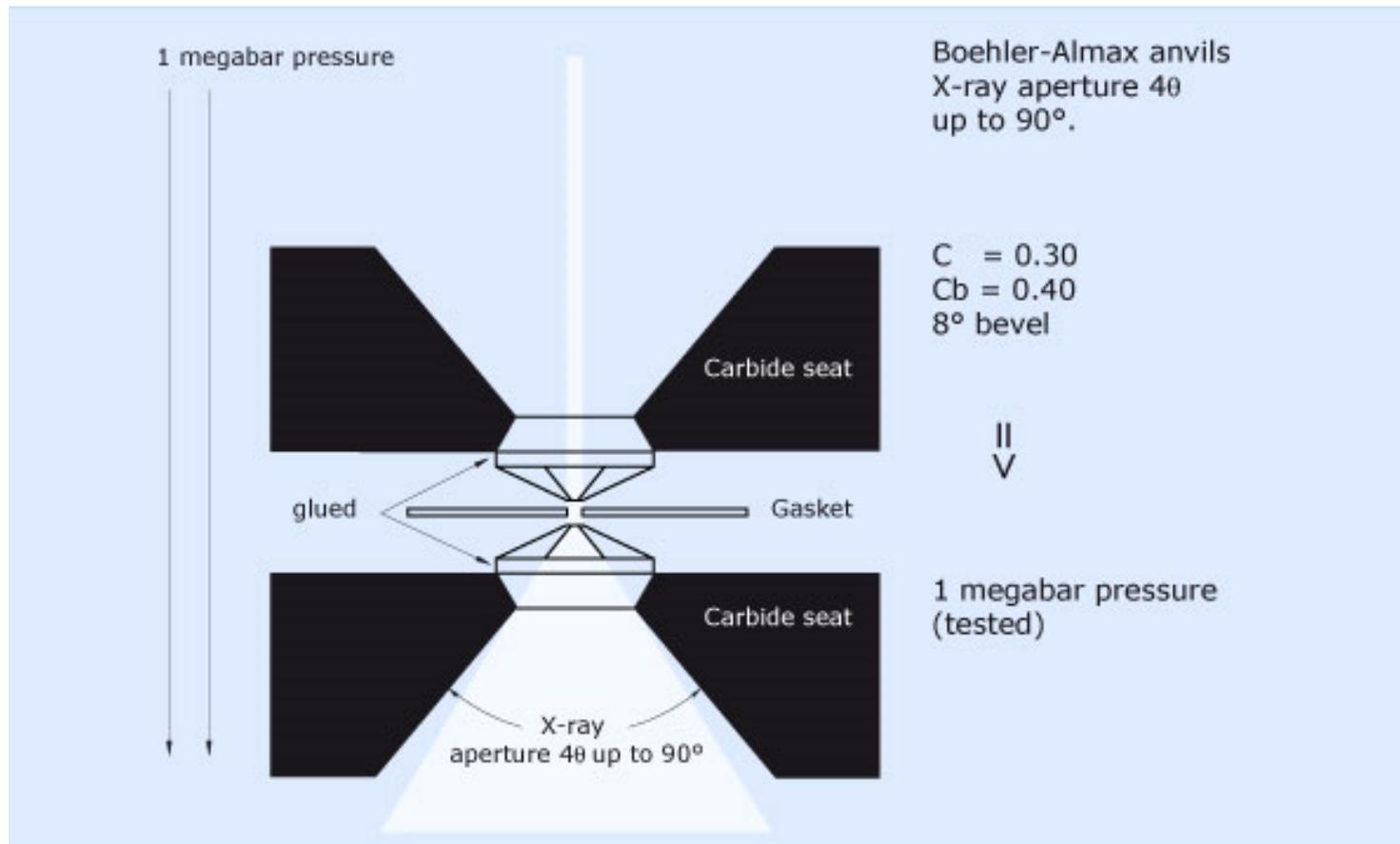


Negative Pressure Compressibility ... (is it because of the quality of thermodynamics books?)



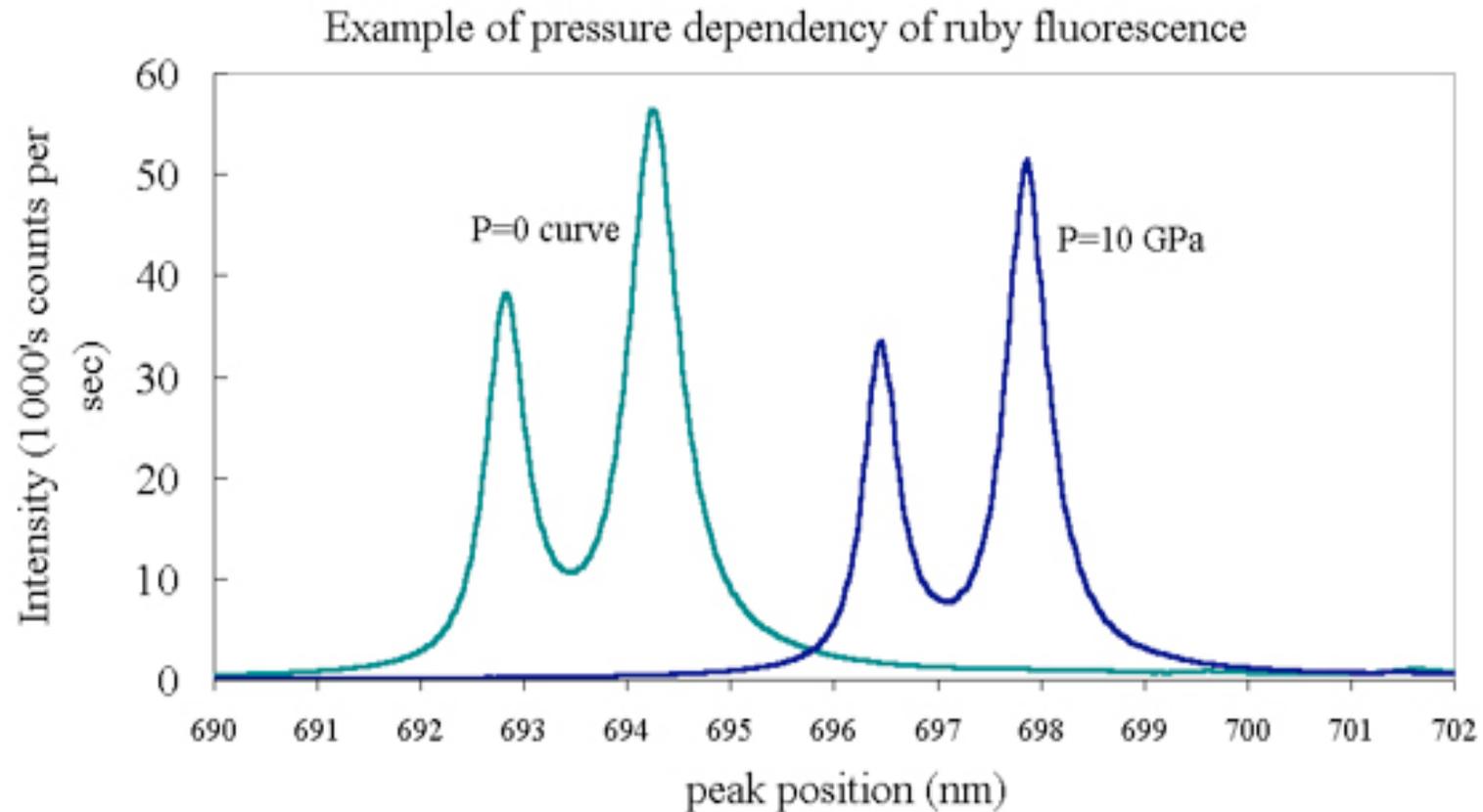
Diamonds are not forever ...

# Bohler Almax



# P measure

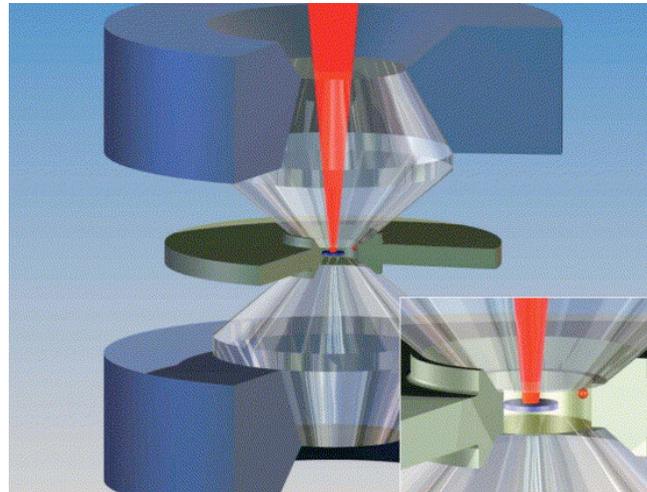
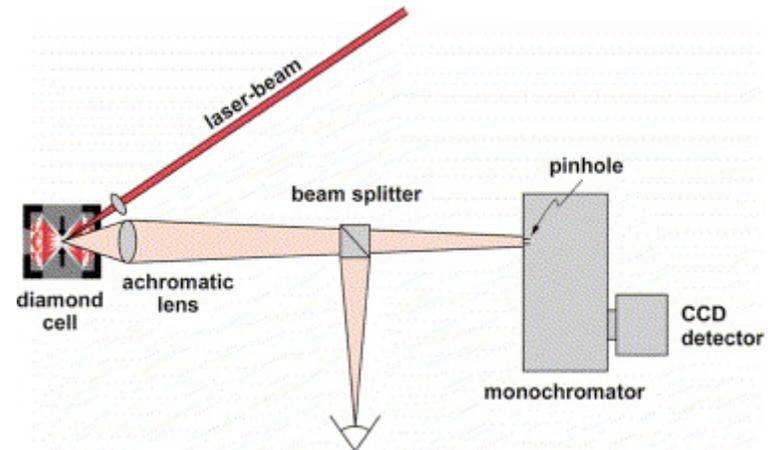
- Spectroscopy: ruby fluorescence
- Diffraction: quartz, gold



# P/T

UP

- resistive heating (don't burn your diamonds)
- laser heating



# P/T

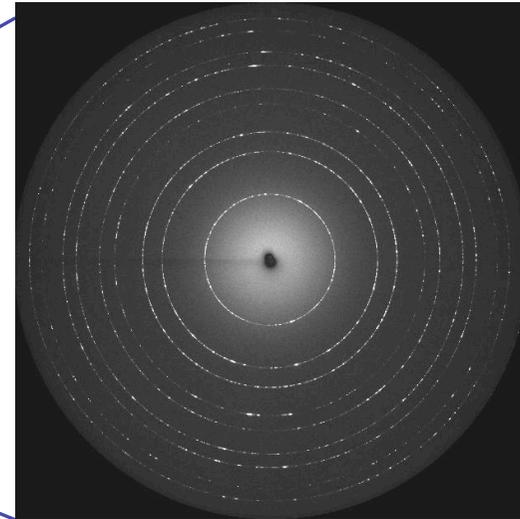
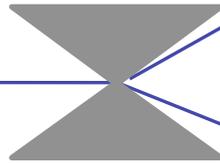
DOWN

- More or less complex cryostats



# setup

L.McCusker: “Area detector gives you broader peaks and restricted range”



- Limited angular access (wavelength choice)
- Very small diffraction volume... (Synchrotron)
- Rocking for statistics
- Very small diffraction volume!
- Diamond (& Compton), PTM, calibrant
- Microstrip (MythenII) vs Area

L.McCusker: “We employ a trick, we spin the sample to gain a bit of statistics”

L.McCusker: “if you look left and right intensities should be the same but they are not, due to texture”

# Processing

Light Graphics Window

EXPERIMENTAL GEOMETRY

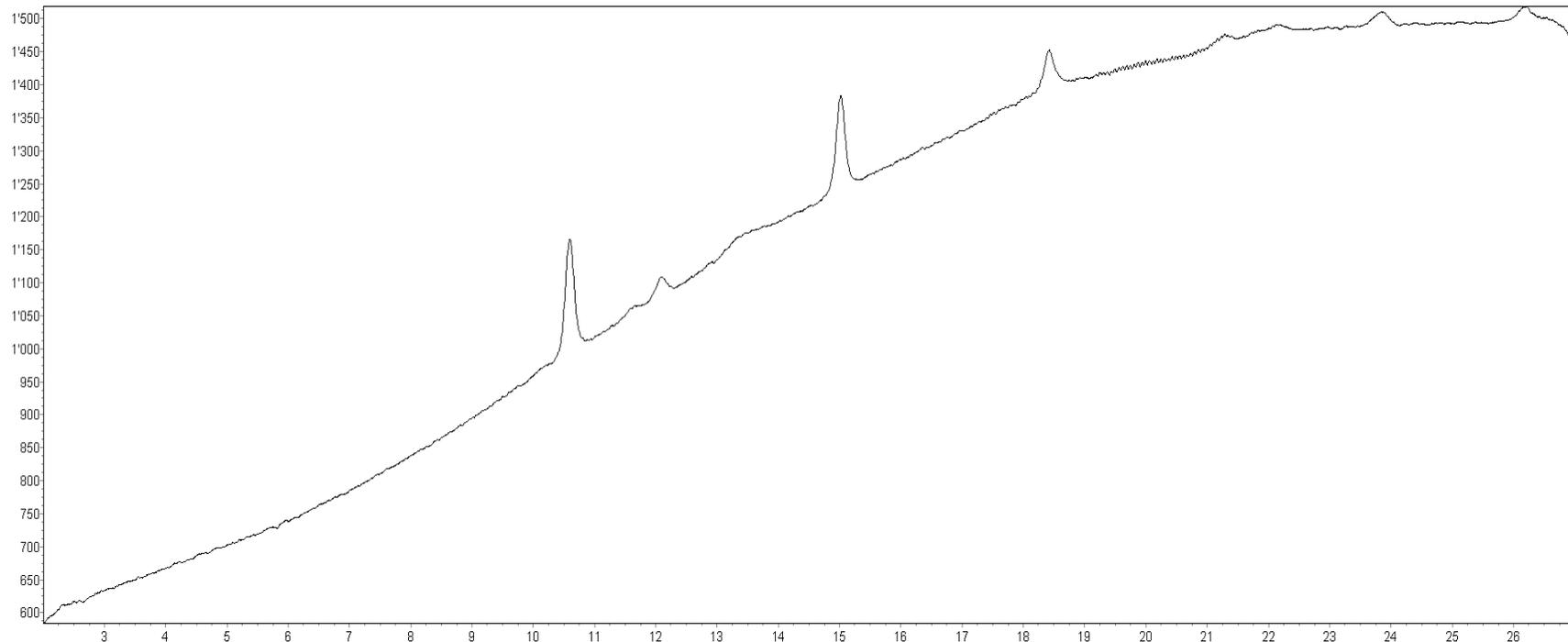
CONTROL FORM

O.K. CANCEL ? HELP INFO

| DESCRIPTIONS                              | VALUES    | CHANGE        |
|---|-----------|---------------|
| SIZE OF HORIZONTAL PIXELS (MICRONS)       | 100.0000  | X-PIXEL SIZE  |
| SIZE OF VERTICAL PIXELS (MICRONS)         | 100.0000  | Y-PIXEL SIZE  |
| SAMPLE TO DETECTOR DISTANCE (MM)          | 329.3629  | DISTANCE      |
| WAVELENGTH (ANGSTROMS)                    | 0.413687  | WAVELENGTH    |
| X-PIXEL COORDINATE OF DIRECT BEAM         | 1713.642  | X-BEAM CENTRE |
| Y-PIXEL COORDINATE OF DIRECT BEAM         | 1732.152  | Y-BEAM CENTRE |
| ROTATION ANGLE OF TILTING PLANE (DEGREES) | -169.9628 | TILT ROTATION |
| ANGLE OF DETECTOR TILT IN PLANE (DEGREES) | 0.227785  | ANGLE OF TILT |

Click on variable to change, or 'O.K.'

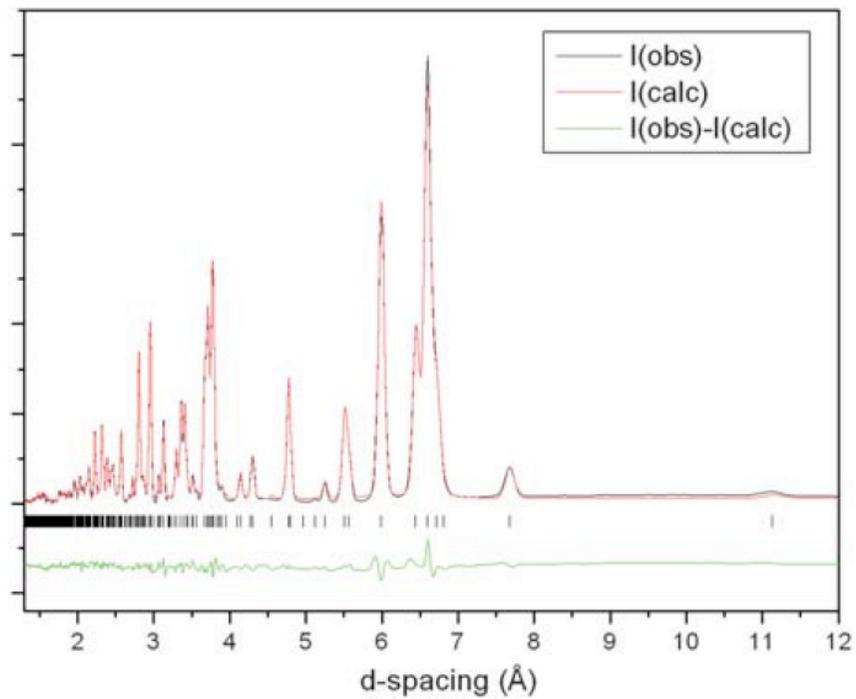
# All for .... This?!?



Even in the worst case enough for EOS and sometimes indexing

N. Masciocchi: “It is the previous knowledge of the sample that allowed us to understand the pattern”

## Energetic materials: CL-20



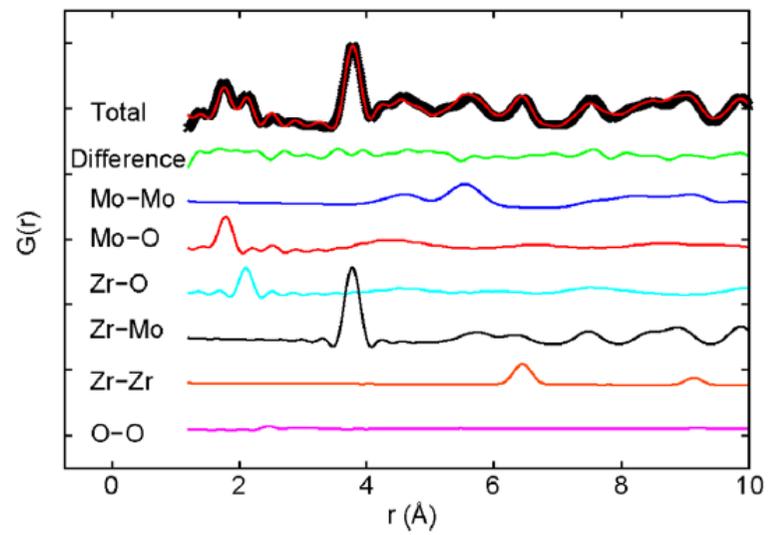
D.I.A. Millar et al., *CrystEngComm* (2010), 12, 2524

## Poly-CO

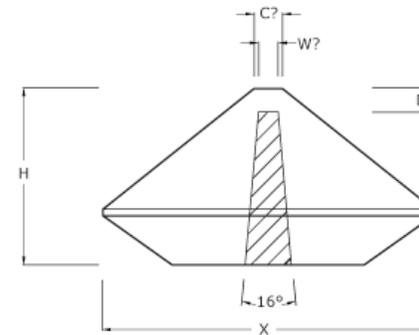


W.J. Evans et al., *Chem. Mater.* (2006), 18, 2520

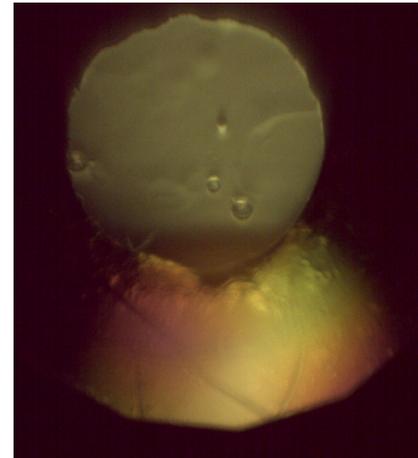
## PDF on $\text{ZrMo}_2\text{O}_8$



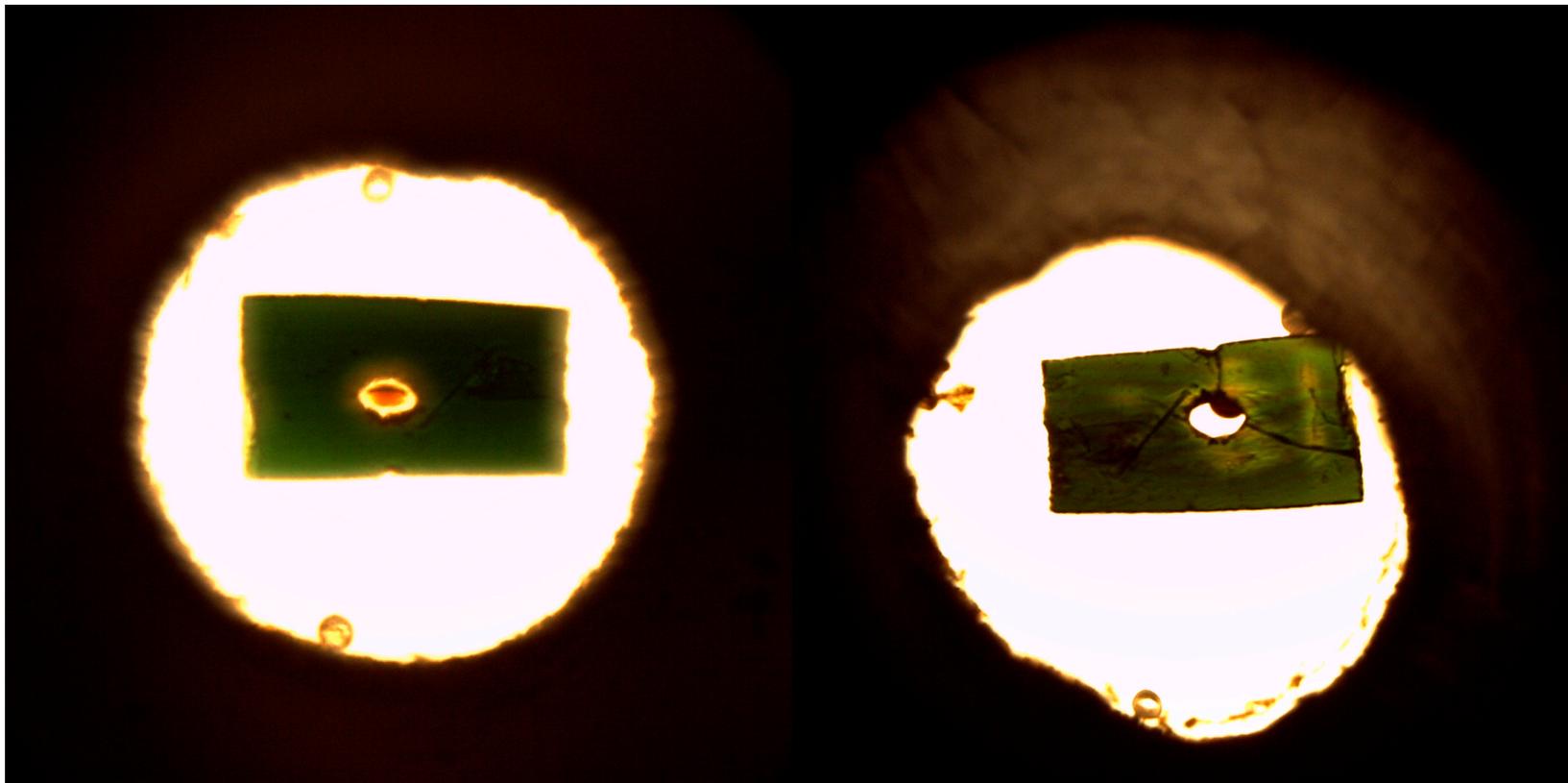
## Partially perforated diamonds



- Polymorphism
- Liquid/gases
- Mechanical properties
- New chemistry
- New physics
- Geophysics



# Questions!



Is your beam a too extreme condition?