

Types of Diamond-Anvil Cells and how to work with them



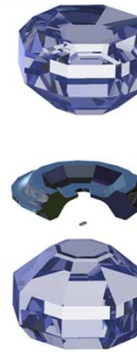
Ronald MILETICH

ronald.miletich-pawliczek@univie.ac.at

Institut für Mineralogie und Kristallographie
Universität Wien



universität
wien

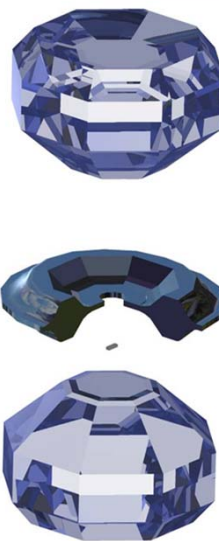
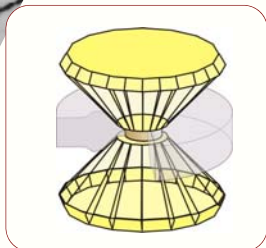


ECM-27 high-P workshop „methods of high-P single crystal x-ray diffraction“

03.08.2012

Introduction

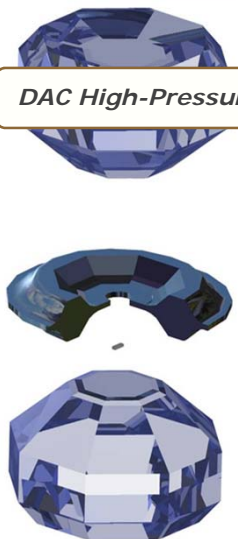
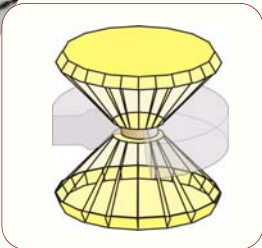
Outline



Introduction

Outline

DAC High-Pressure Technique





Introduction

Outline

DAC High-Pressure Technique

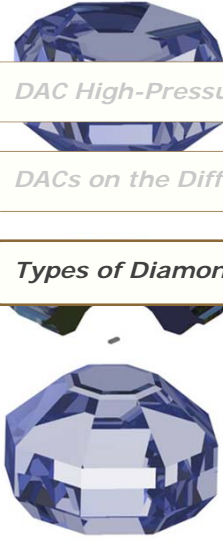
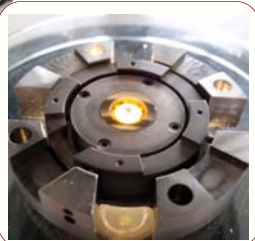
DACs on the Diffractometer



Introduction

Outline

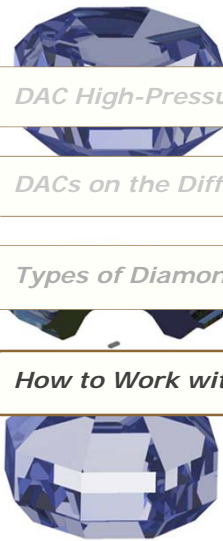
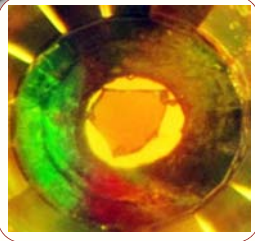
- DAC High-Pressure Technique
- DACs on the Diffractometer
- Types of Diamond-Anvil Cells



Introduction

Outline

- DAC High-Pressure Technique
- DACs on the Diffractometer
- Types of Diamond-Anvil Cells
- How to Work with Them






Introduction

Outline

- DAC High-Pressure Technique**
- DACs on the Diffractometer**
- Types of Diamond-Anvil Cells**
- How to Work with them

Now!


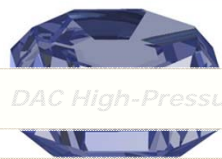





Introduction

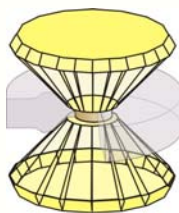
Outline

- DAC High-Pressure Technique
- DACs on the Diffractometer
- Types of Diamond-Anvil Cells
- How to Work with Them**

this afternoon

DAC Principles



part 1

DAC Principles

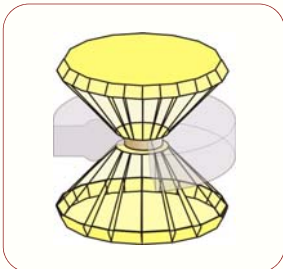
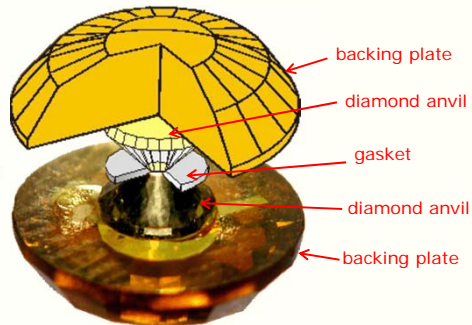
High-Pressure Techniques

1912, Percy Bridgman - Nobel Prize 1946

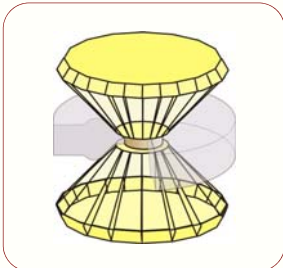
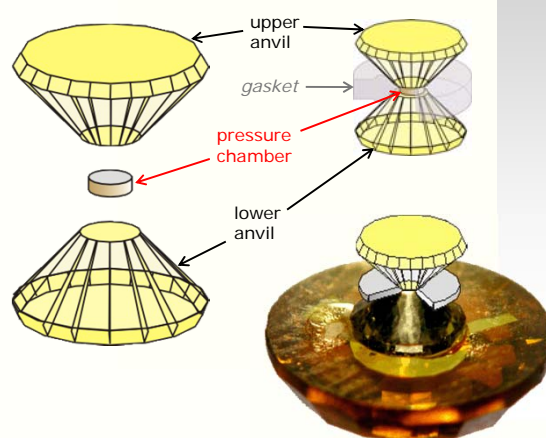


... the pioneer era in high-pressure research

DAC Principles

**DAC High-Pressure Technique****Opposed Anvil Assembly (1/3)**

DAC Principles

**DAC High-Pressure Technique****Opposed Anvil Assembly (2/3)**

DAC Principles

DAC High-Pressure Technique

Opposed Anvil Assembly (3/3)

polycrystalline sample

oriented single crystal sample

hydrostatic pressure transmitting medium

DAC Principles

DAC High-Pressure Technique

Opposed Anvil Assembly (3/3)

polycrystalline sample

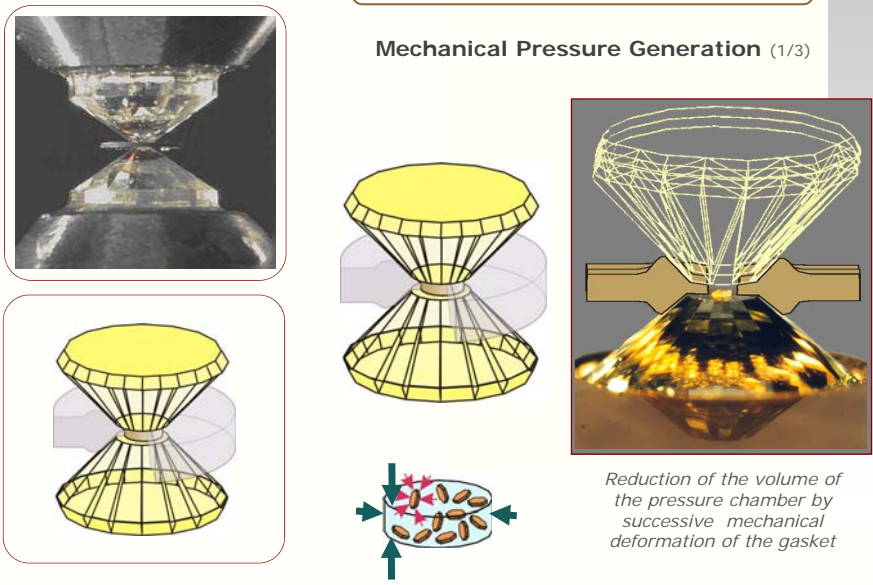
oriented single crystal sample

hydrostatic pressure transmitting medium

DAC Principles

DAC High-Pressure Technique

Mechanical Pressure Generation (1/3)

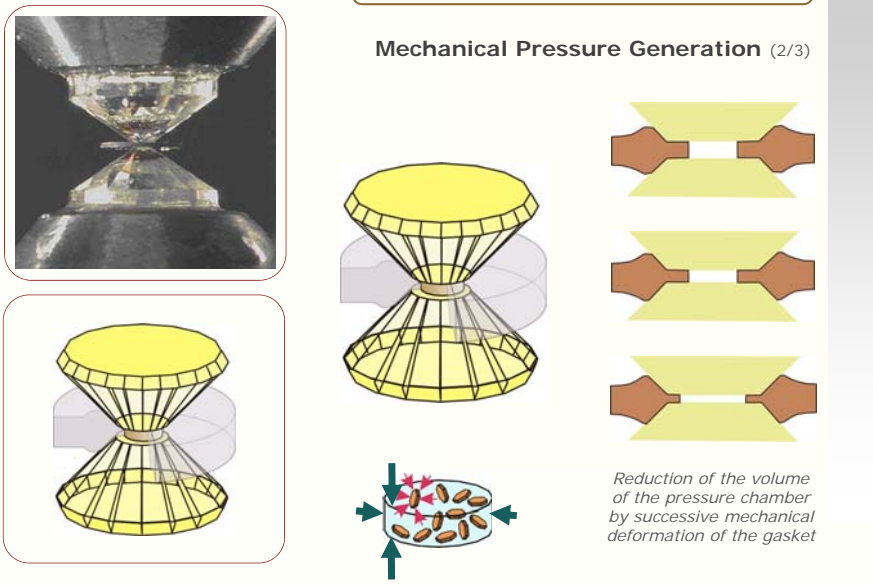


Reduction of the volume of the pressure chamber by successive mechanical deformation of the gasket

DAC Principles

DAC High-Pressure Technique

Mechanical Pressure Generation (2/3)

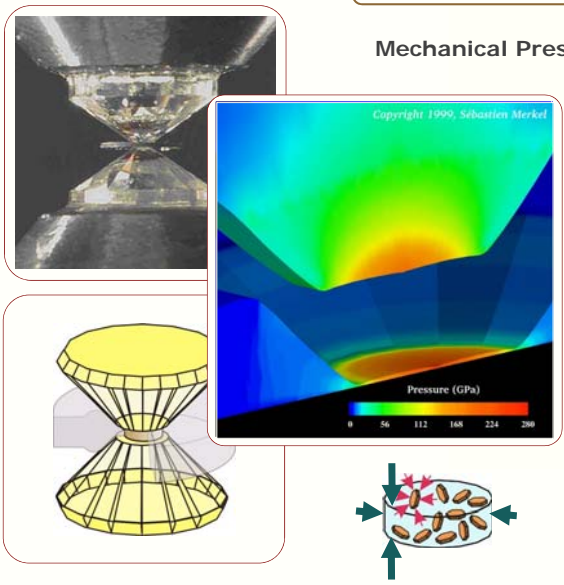


Reduction of the volume of the pressure chamber by successive mechanical deformation of the gasket

DAC Principles

DAC High-Pressure Technique

Mechanical Pressure Generation (3/3)



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Pressure (GPa)

0 56 112 168 224 288

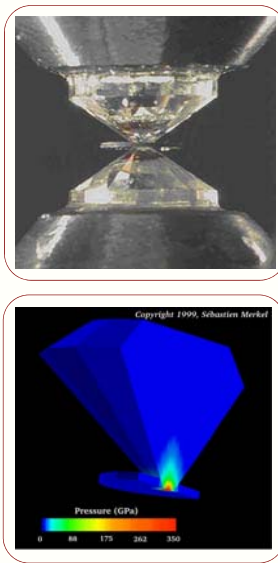
Reduction of the volume of the pressure chamber by successive mechanical deformation of the gasket

DAC Principles

DAC High-Pressure Technique

Specifications of Anvils

Cut, Geometry, and Dimensions

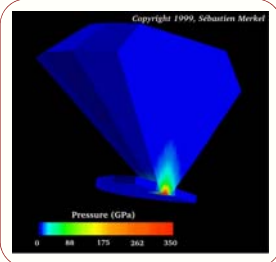
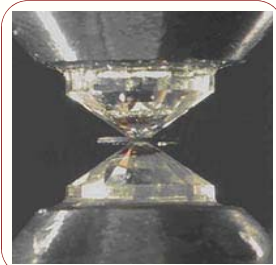
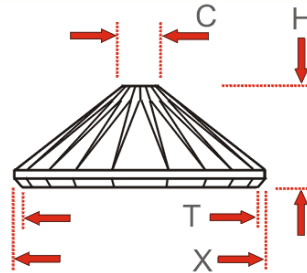


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Pressure (GPa)

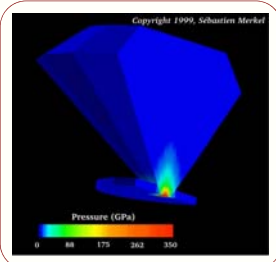
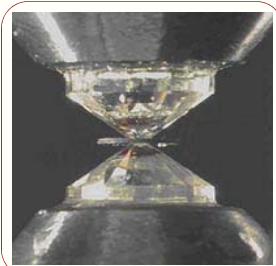
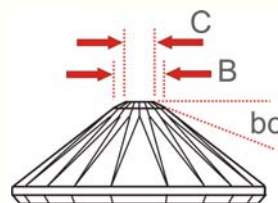
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DAC Principles

**DAC High-Pressure Technique****Specifications of Anvils** (1/6)*Cut, Geometry, and Dimensions*

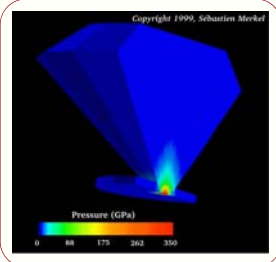
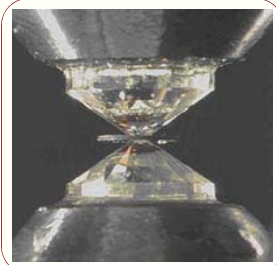
C ... culet-face diameter
 T ... table-face diameter
 X ... girdle diameter
 H ... height between C face and T face

DAC Principles

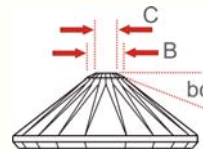
**DAC High-Pressure Technique****Specifications of Anvils** (2/6)*Cut, Geometry, and Dimensions*

C ... culet-face diameter
 B ... bevel-face diameter
 bc ... bevel angle

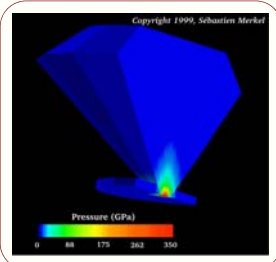
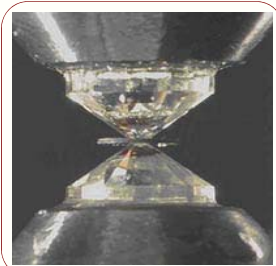
DAC Principles

DAC High-Pressure Technique**Specifications of Anvils** (3/6)

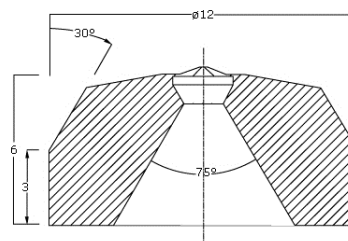
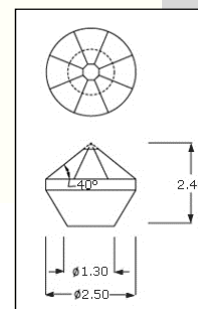
Cut, Geometry, and Dimensions

triple bevel
(Scimed GmbH)

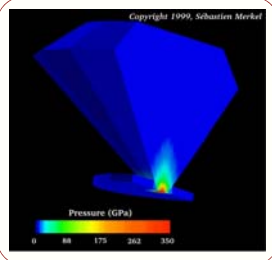
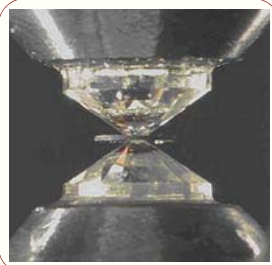
DAC Principles

DAC High-Pressure Technique**Specifications of Anvils** (4/6)

Cut, Geometry, and Dimensions

Almax-Boehler
design

DAC Principles

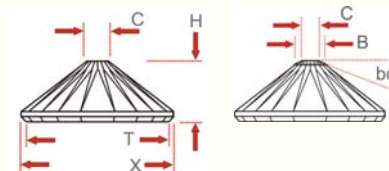
DAC High-Pressure Technique**Specifications of Anvils** (6/6)

Cut, Geometry, and Dimensions

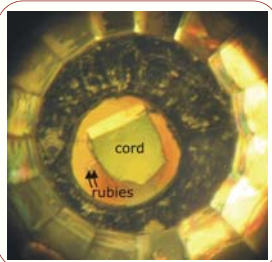
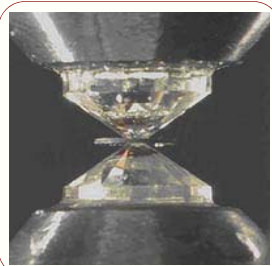
Typical weight: 0.10-0.25 ct
 Material: type Ia, Ib (IIa, IIb)

C	maximum P *
0.60 mm	15 (25) GPa
0.45 mm	40 (50) GPa
0.30 mm	80 (100) GPa
0.20 mm	150 (200) GPa

* X=3.0mm, H=1.4mm




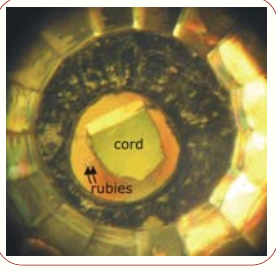
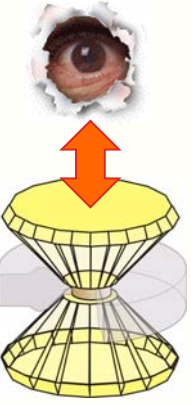
DAC Principles

DAC High-Pressure Technique**Sample Environment and Pressure Calibration**

DAC Principles

DAC High-Pressure Technique

Sample Environment and Pressure Calibration (1/7)


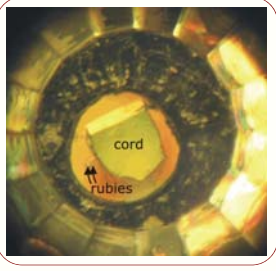
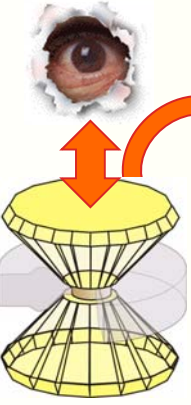
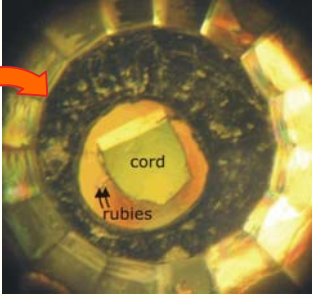




... diamonds are windows to the pressure chamber

DAC Principles

DAC High-Pressure Technique



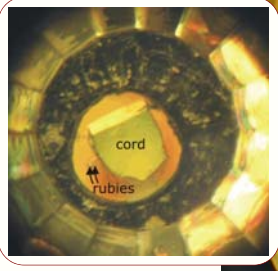
Sample Environment and Pressure Calibration (1/7)

DAC Principles

DAC High-Pressure Technique

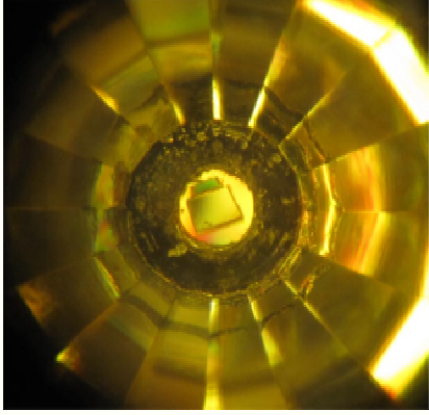
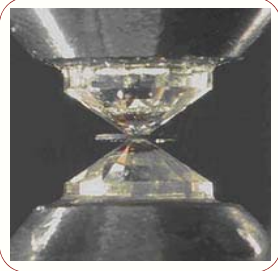
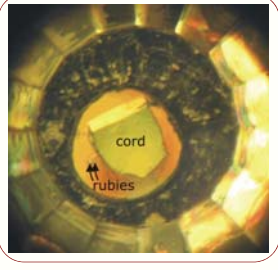
Sample Environment and Pressure Calibration (2/7)

DAC Principles

DAC High-Pressure Technique

Sample Environment and Pressure Calibration (3/7)

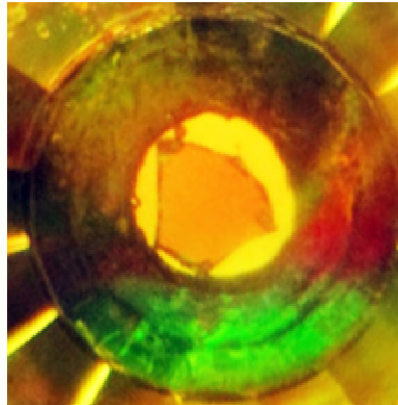
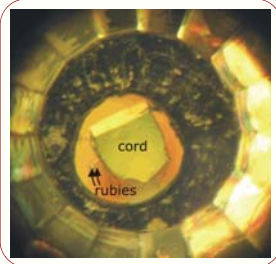




LiAlSi₂O₆ and SiO₂ (quartz) crystals

DAC Principles

DAC High-Pressure Technique**Sample Environment and Pressure Calibration**

(4/7)

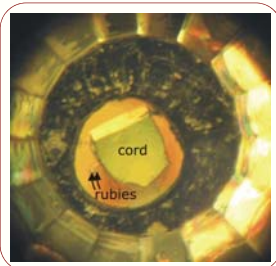
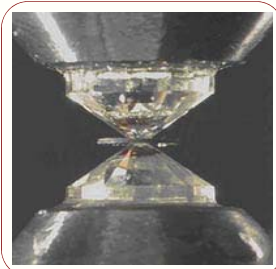
*BaTiSi₃O₉ crystal and ruby spheres*

DAC Principles

DAC High-Pressure Technique**Sample Environment and Pressure Calibration**

(5/7)

Optical pressure sensor: Laser-induced luminescence, e.g. R_1, R_2 spectral lines in ruby



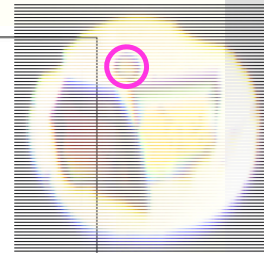
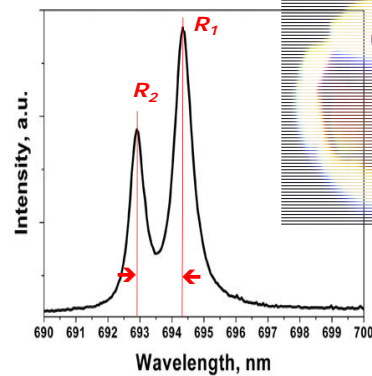
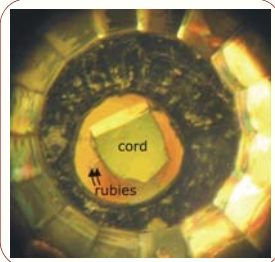
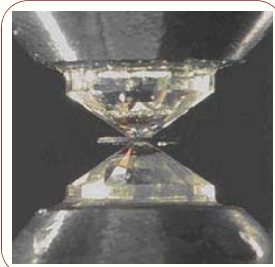
DAC Principles

DAC High-Pressure Technique

Sample Environment and Pressure Calibration

(5/7)

Optical pressure sensor: Laser-induced luminescence, e.g. R_1, R_2 spectral lines in ruby



$$\Delta\lambda/\lambda_0 = +0.365 \text{ nm/GPa}$$

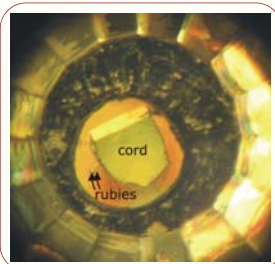
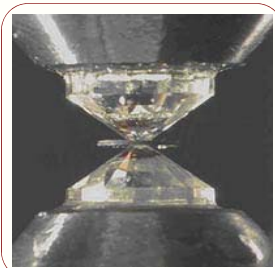
DAC Principles

DAC High-Pressure Technique

Sample Environment and Pressure Calibration

(6/7)

Unit-cell dimensions of internal diffraction standards (Quartz, NaCl, Au, Pt, W, Ne,...)



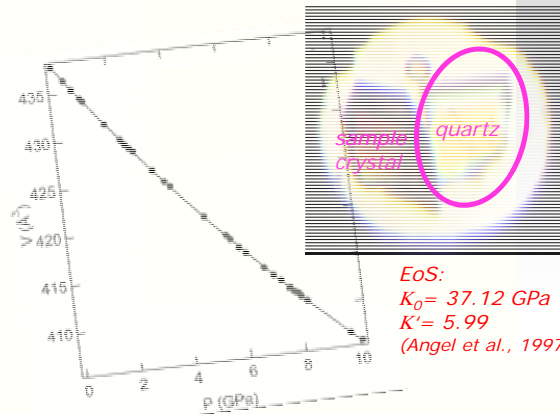
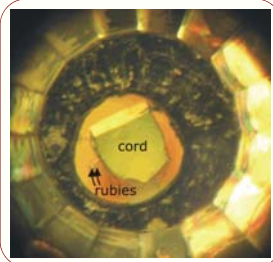
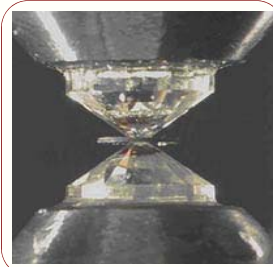
DAC Principles

DAC High-Pressure Technique

Sample Environment and Pressure Calibration

(6/7)

Unit-cell dimensions of internal diffraction standards (Quartz, NaCl, Au, Pt, W, Ne,...)



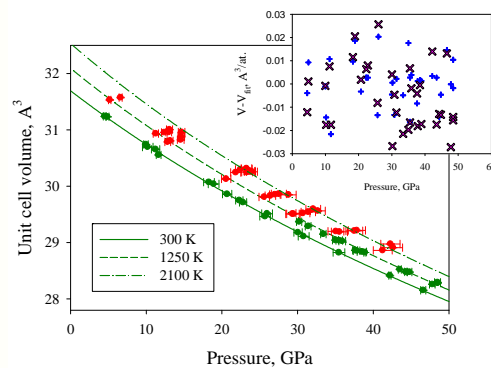
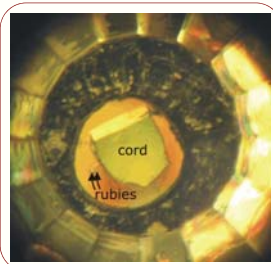
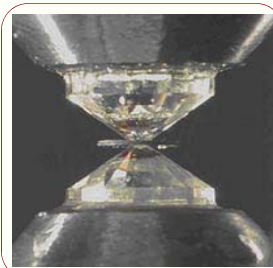
DAC Principles

DAC High-Pressure Technique

Sample Environment and Pressure Calibration

(7/7)

Unit-cell dimensions of internal diffraction standards (Quartz, NaCl, Au, Pt, W, Ne,...)



DAC & Single Xtal XRD

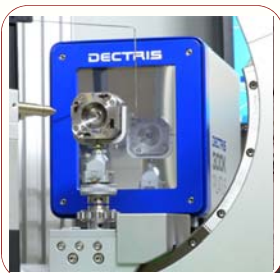


Part 2

DAC & Single Xtal XRD

DACs on the Diffractometer

Various Types of Goniometers

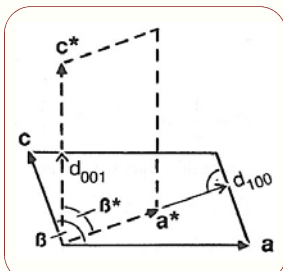


DAC & Single Xtal XRD

DACs on the Diffractometer

Access in reciprocal space (1/8)

Geometric interpretation of the Bragg equation

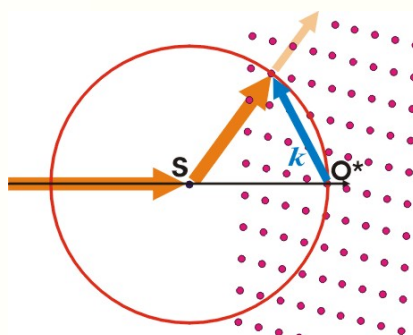
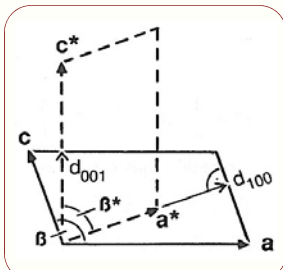


DAC & Single Xtal XRD

DACs on the Diffractometer

Access in reciprocal space (1/6)

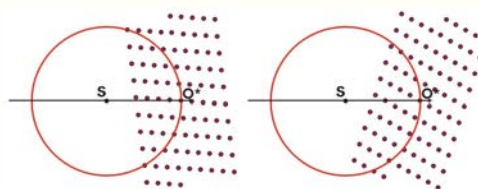
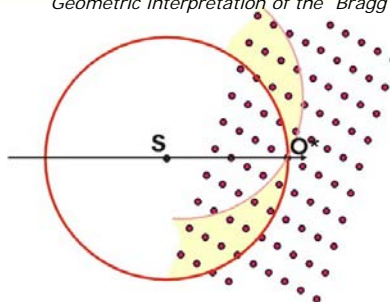
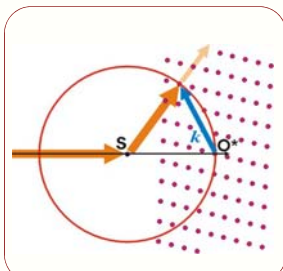
Geometric interpretation of the Bragg equation



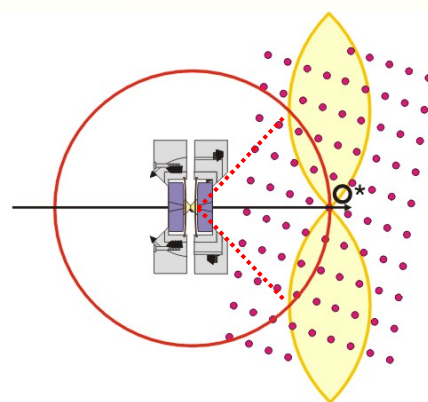
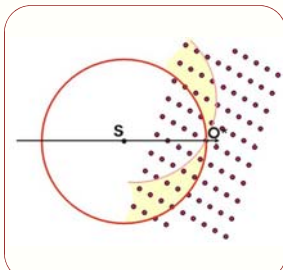
Simple trigonometry:

$$\sin\theta = \frac{\lambda}{2d} = \frac{\lambda/d_{hkl}}{2} = \frac{\lambda d_{hkl}^*}{2}$$

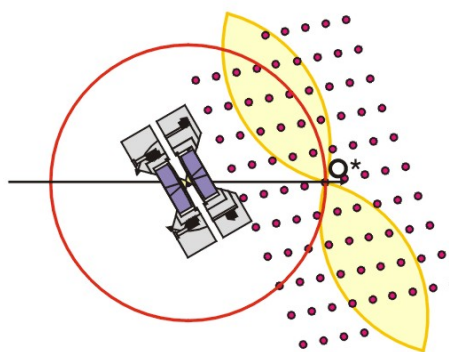
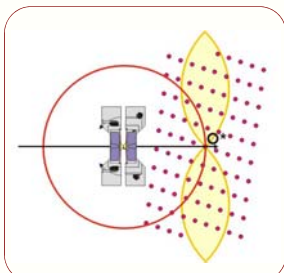
DAC & Single Xtal XRD

DACs on the Diffractometer**Access in reciprocal space** (2/6)*Geometric interpretation of the Bragg equation*

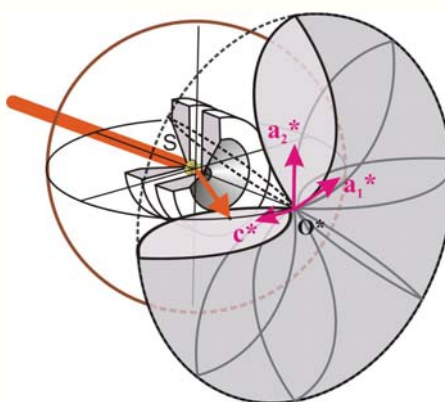
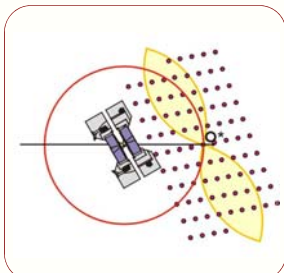
DAC & Single Xtal XRD

DACs on the Diffractometer**Access in reciprocal space** (3/6)*Restrictions determined by DAC opening angle*

DAC & Single Xtal XRD

DACs on the Diffractometer**Access in reciprocal space** (4/6)*Restrictions determined by DAC opening angle*

DAC & Single Xtal XRD

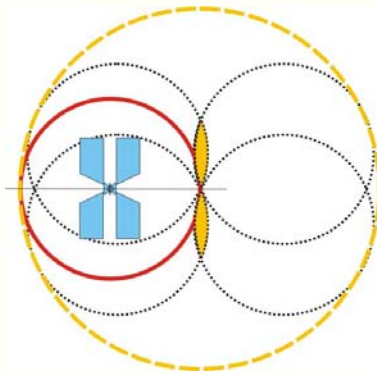
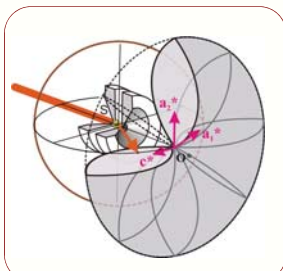
DACs on the Diffractometer**Access in reciprocal space** (5/6)*Restrictions determined by DAC opening angle**toroidal shape of accessible part of the reciprocal space*

DAC & Single Xtal XRD

DACs on the Diffractometer

Access in reciprocal space (6/6)

Restrictions determined by DAC opening angle



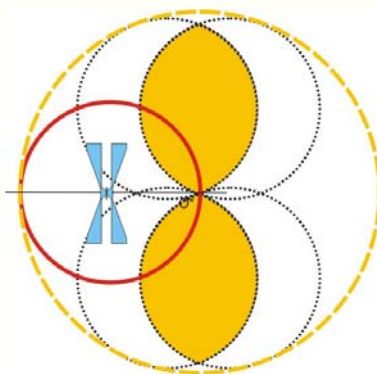
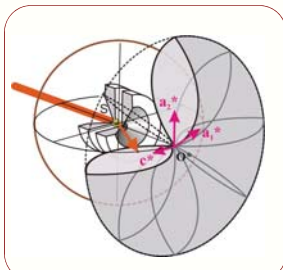
accessible reciprocal space depends on opening angle

DAC & Single Xtal XRD

DACs on the Diffractometer

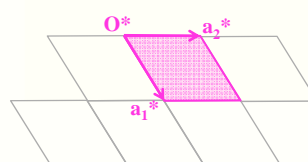
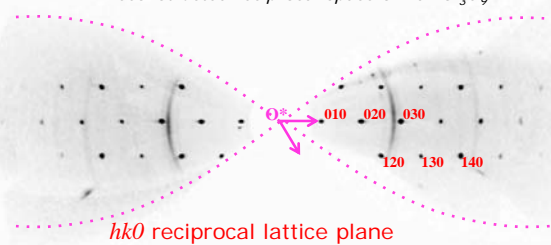
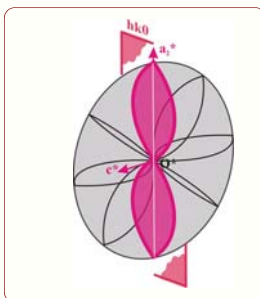
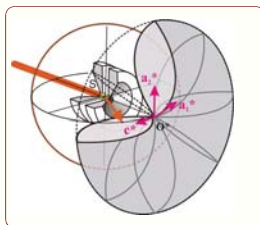
Access in reciprocal space (6/6)

Restrictions determined by DAC opening angle

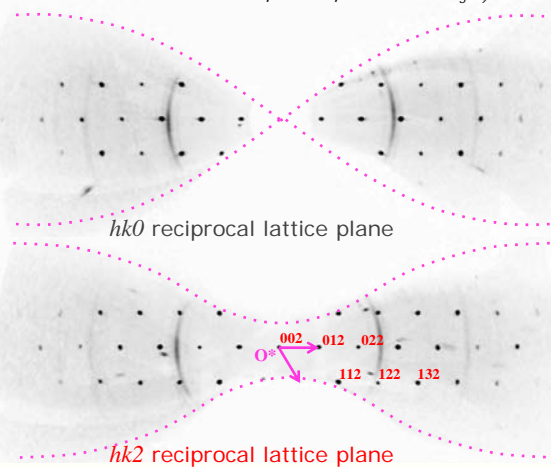
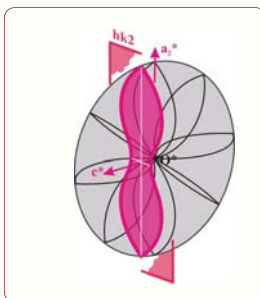
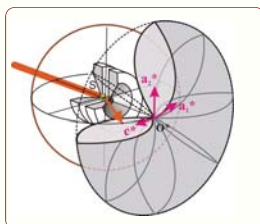


accessible reciprocal space depends on opening angle

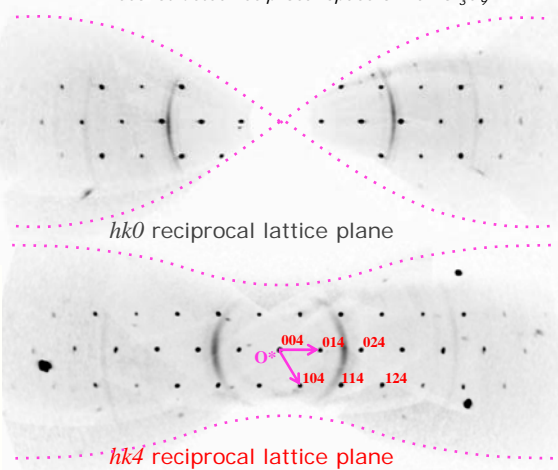
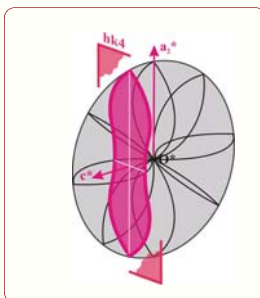
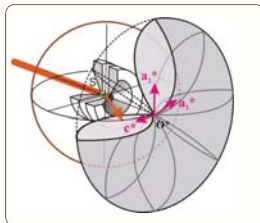
DAC & Single Xtal XRD

DACs on the Diffractometer**Limitations in reciprocal space (1/5)**reconstructed reciprocal space of $\text{BaTiSi}_3\text{O}_9$ 

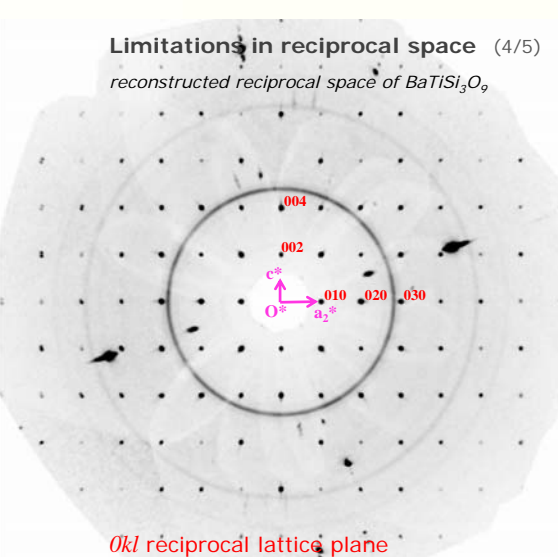
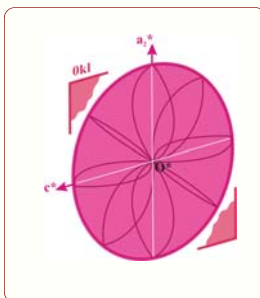
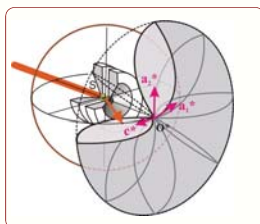
DAC & Single Xtal XRD

DACs on the Diffractometer**Limitations in reciprocal space (2/5)**reconstructed reciprocal space of $\text{BaTiSi}_3\text{O}_9$ 

DAC & Single Xtal XRD

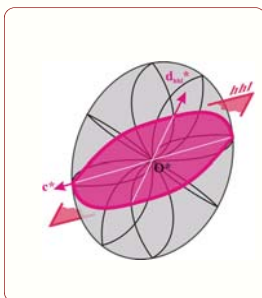
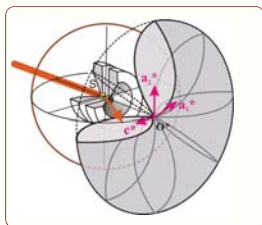
DACs on the Diffractometer**Limitations in reciprocal space (3/5)**reconstructed reciprocal space of $\text{BaTiSi}_3\text{O}_9$ 

DAC & Single Xtal XRD

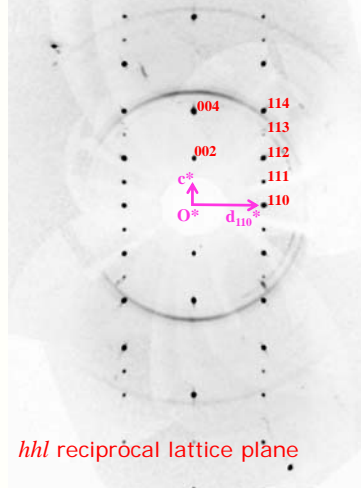
DACs on the Diffractometer**Limitations in reciprocal space (4/5)**reconstructed reciprocal space of $\text{BaTiSi}_3\text{O}_9$ 

DAC & Single Xtal XRD

DACs on the Diffractometer

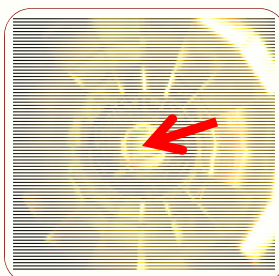


Limitations in reciprocal space (5/5)

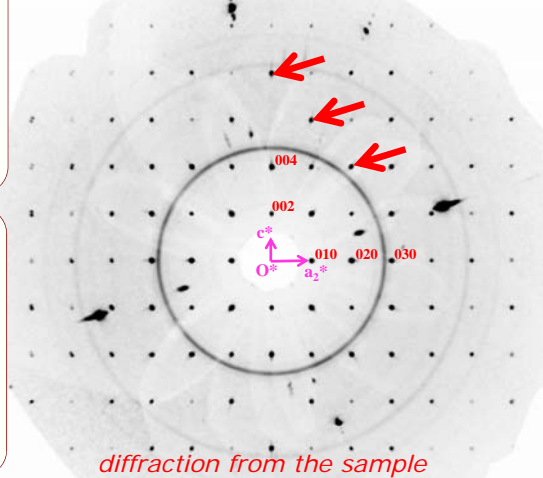
reconstructed reciprocal space of $\text{BaTiSi}_3\text{O}_9$ 

DAC & Single Xtal XRD

DACs on the Diffractometer



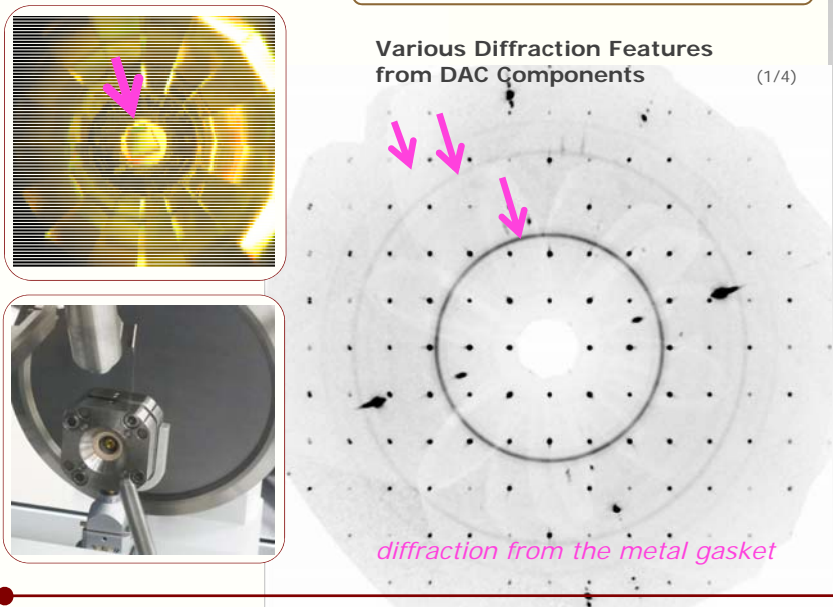
Various Diffraction Features from DAC Components (1/4)



DAC & Single Xtal XRD

DACs on the Diffractometer

Various Diffraction Features from DAC Components (1/4)

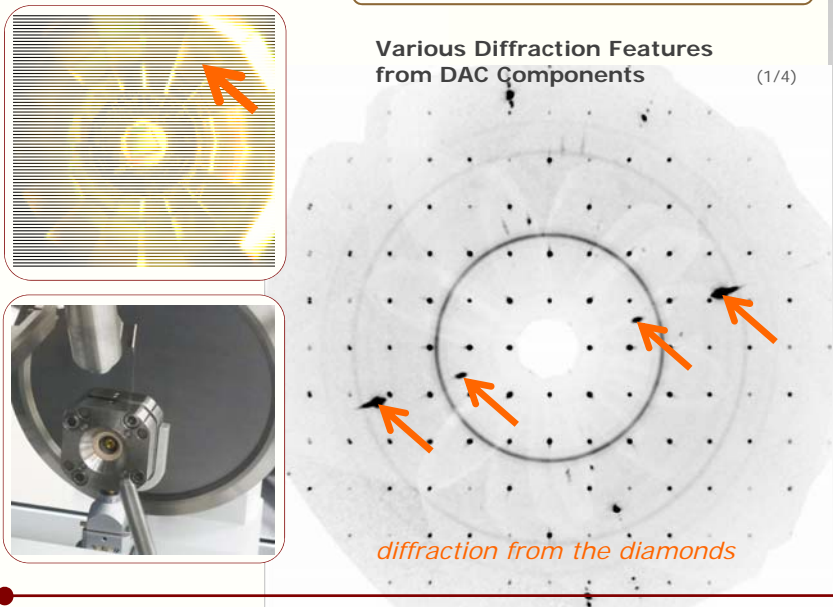


diffraction from the metal gasket

DAC & Single Xtal XRD

DACs on the Diffractometer

Various Diffraction Features from DAC Components (1/4)

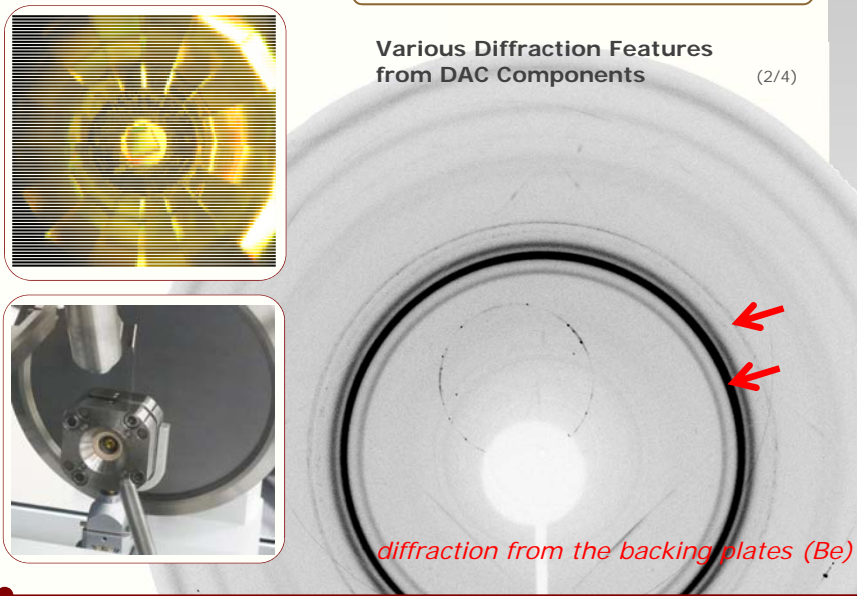


diffraction from the diamonds

DAC & Single Xtal XRD

DACs on the Diffractometer

Various Diffraction Features from DAC Components (2/4)

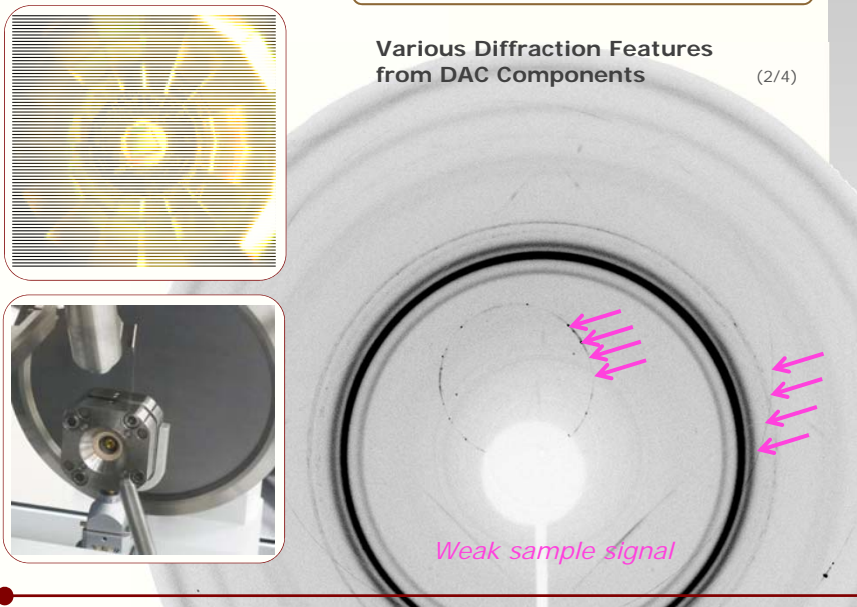


diffraction from the backing plates (Be)

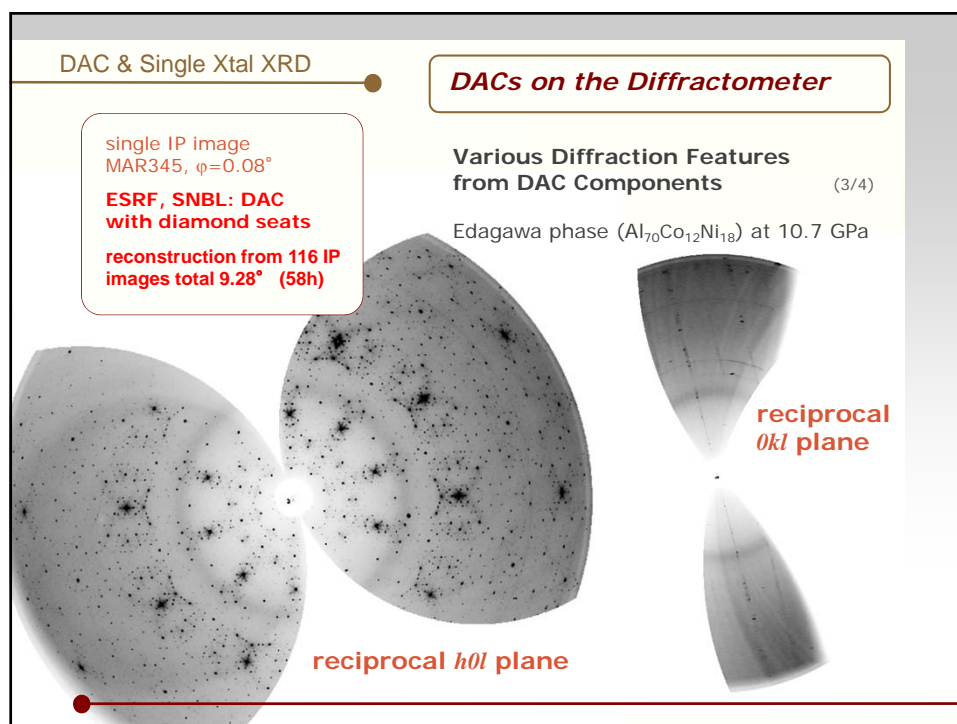
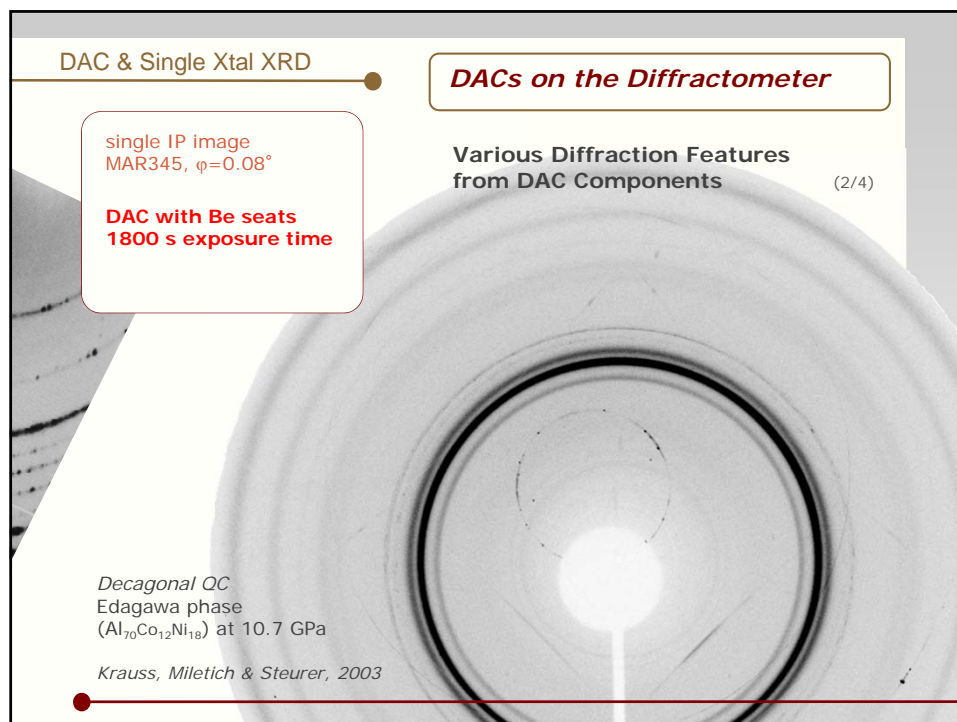
DAC & Single Xtal XRD

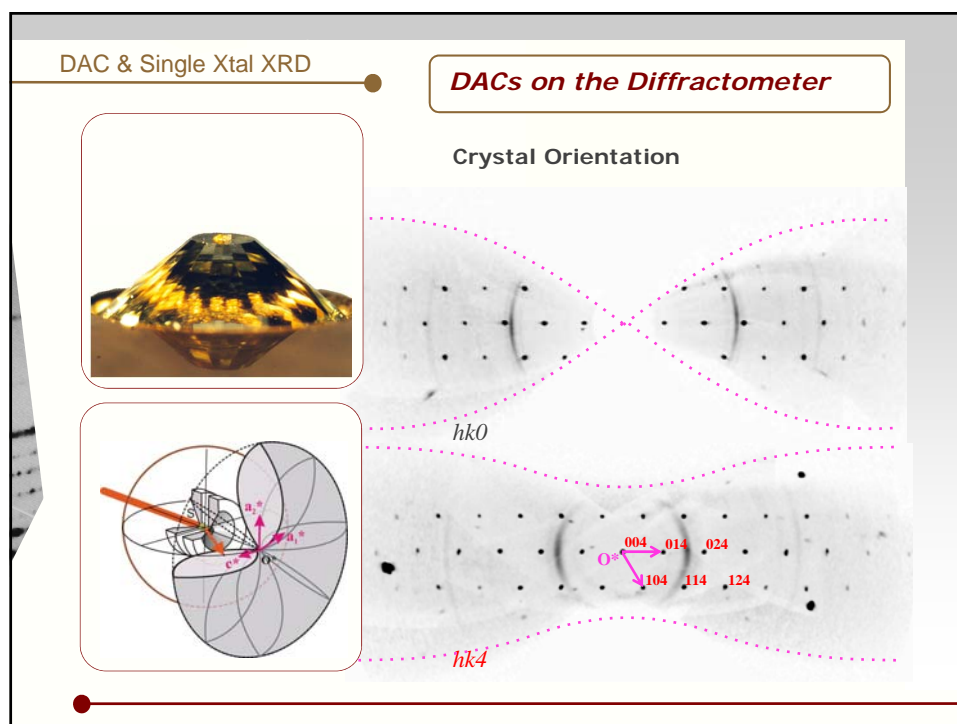
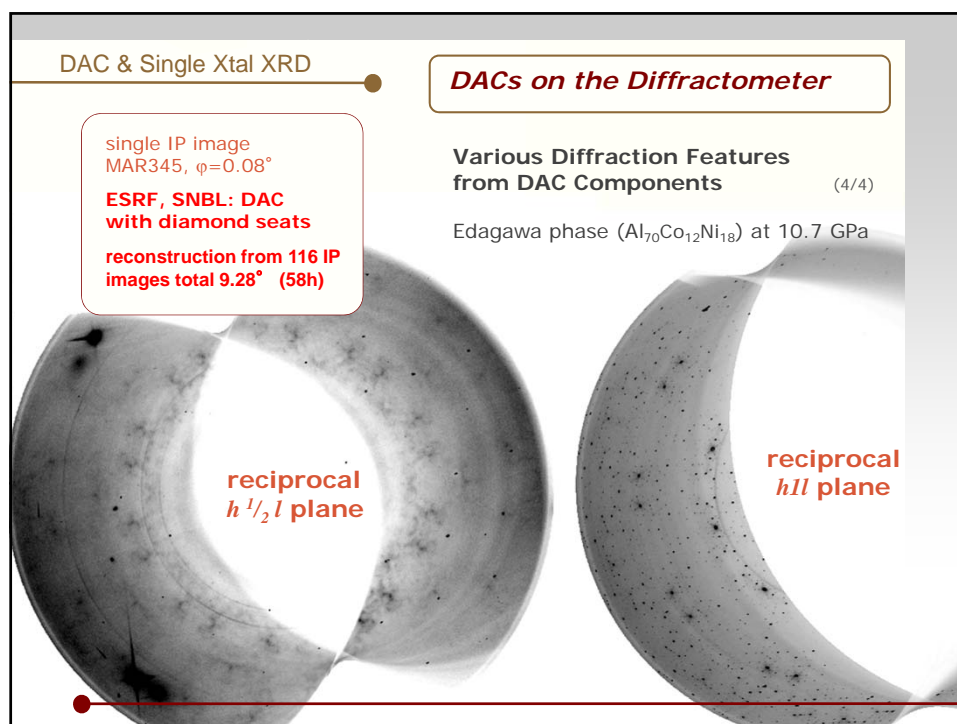
DACs on the Diffractometer

Various Diffraction Features from DAC Components (2/4)



Weak sample signal

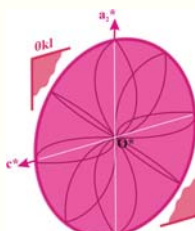
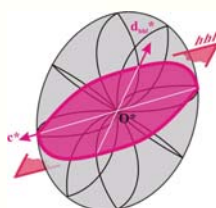
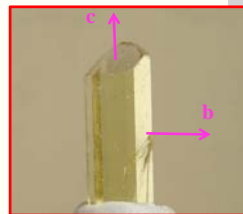
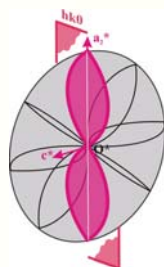
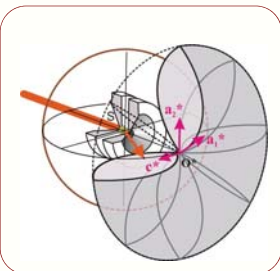
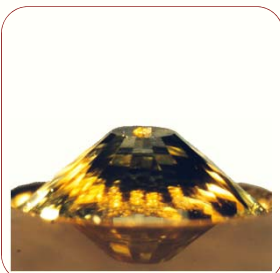




DAC & Single Xtal XRD

DACs on the Diffractometer**Crystal Orientation**

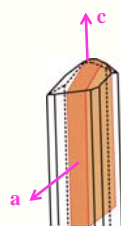
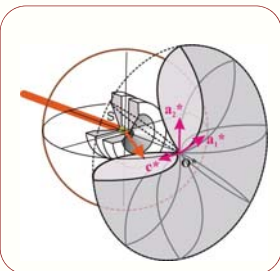
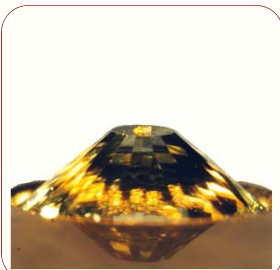
(1/3)



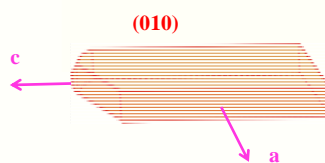
DAC & Single Xtal XRD

DACs on the Diffractometer**Crystal Orientation**

(2/3)



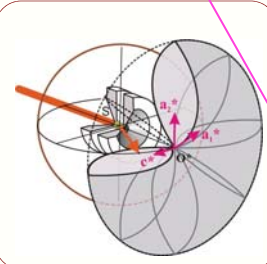
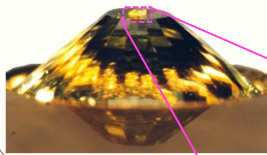
Preparation of oriented sections



DAC & Single Xtal XRD

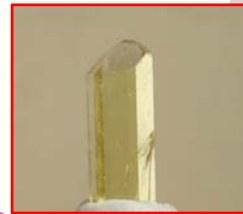
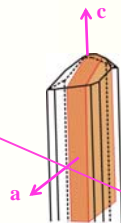
DACs on the Diffractometer

„...directions of highest resolution occur parallel to the culet-face plane...”



Crystal Orientation

(2/3)



Preparation of oriented sections

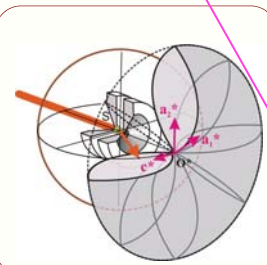
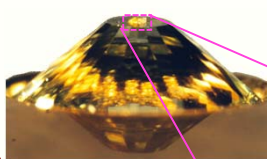
(010)



DAC & Single Xtal XRD

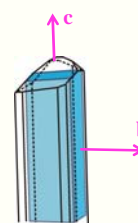
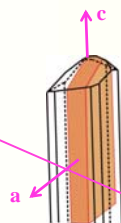
DACs on the Diffractometer

achieving equivalent resolution in all three dimensions

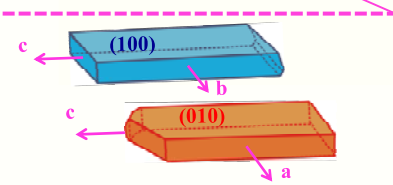


Crystal Orientation

(3/3)



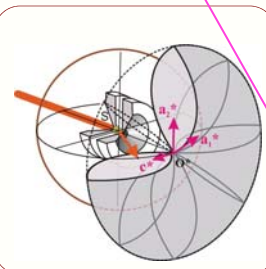
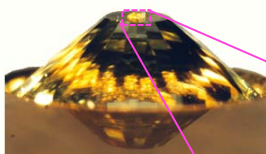
2-crystals mount



DAC & Single Xtal XRD

DACs on the Diffractometer

achieving equivalent
resolution in all three
dimensions

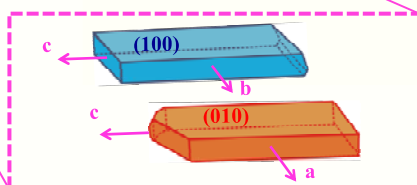


Crystal Orientation

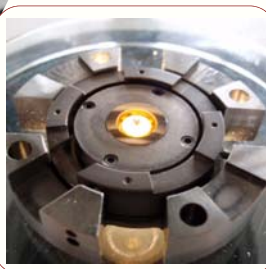
(3/3)



2-crystals mount



DAC Designs

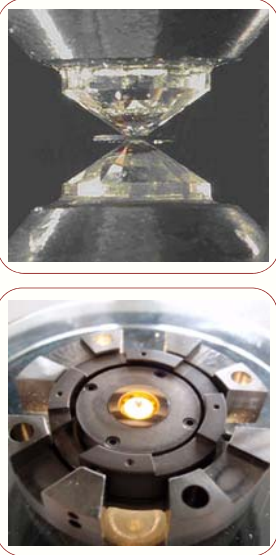


Part 3

DAC Designs

Types of Diamond-Anvil Cells

Demands on DACs for sXRD



DAC Designs

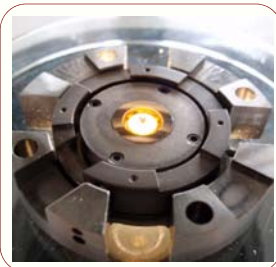
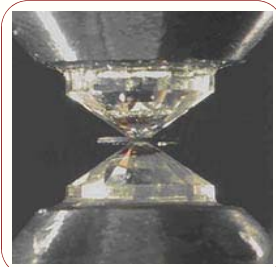
Types of Diamond-Anvil Cells

Demands on DACs for sXRD

- large opening angle
- low background contribution from DAC components (diamonds, Be, gasket)
- small dimensions for low absorption

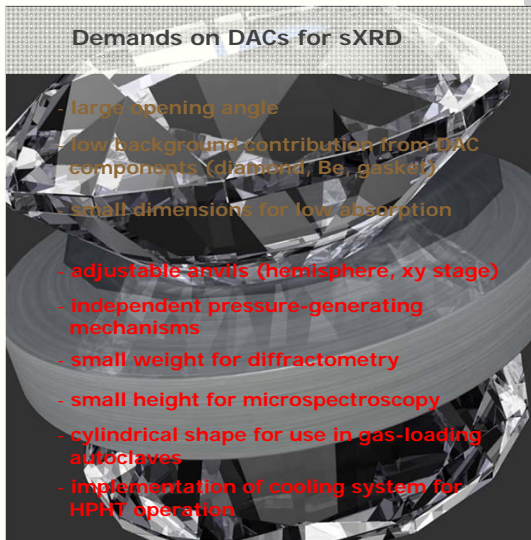


DAC Designs

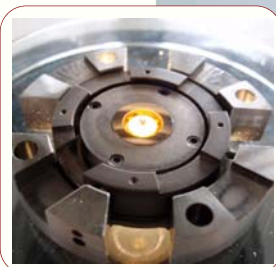
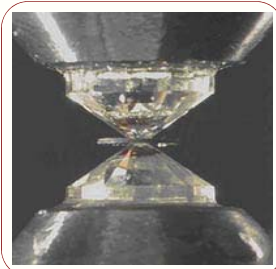
Types of Diamond-Anvil Cells

Demands on DACs for sXRD

- large opening angle
- low background contribution from DAC components (diamond, Be, gasket)
- small dimensions for low absorption
- adjustable anvils (hemisphere, xy stage)
- independent pressure-generating mechanisms
- small weight for diffractometry
- small height for microspectroscopy
- cylindrical shape for use in gas-loading autoclaves
- implementation of cooling system for HPHT operation



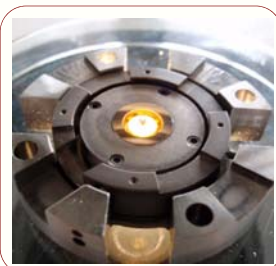
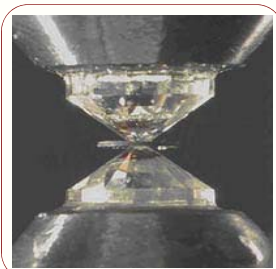
DAC Designs

Types of Diamond-Anvil Cells

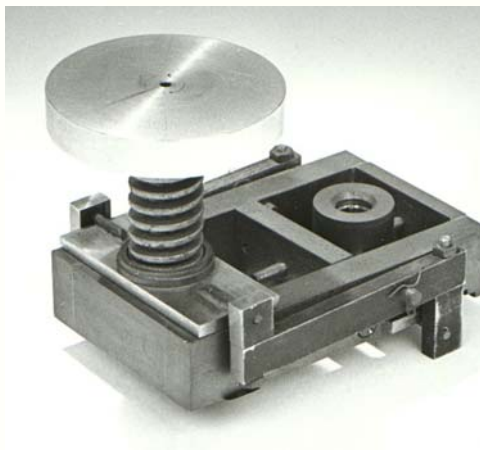
50 years history



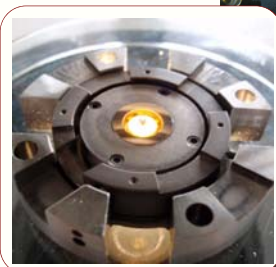
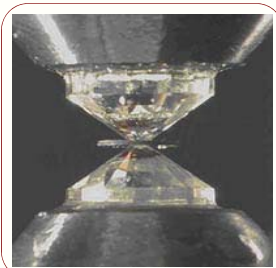
DAC Designs

***Types of Diamond-Anvil Cells*****50 years history**

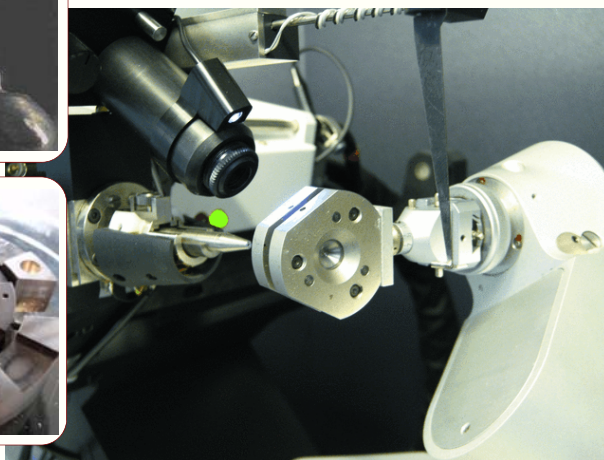
The first DAC (1958, Nat.Bur. Standards)



DAC Designs

***Types of Diamond-Anvil Cells*****50 years history**


triangular Merrill-Bassett DAC



DAC Designs

Types of Diamond-Anvil Cells


50 years history
triangular Merill-Bassett DAC



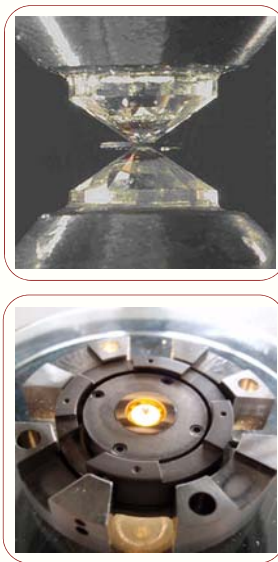
DAC Designs

Types of Diamond-Anvil Cells

50 years history
triangular Merill-Bassett DAC




DAC Designs

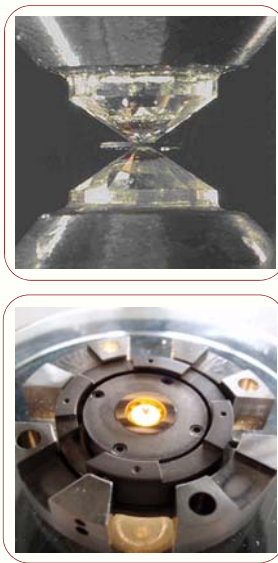


Types of Diamond-Anvil Cells

50 years history
DXR-6 DAC (Diacell Ltd.)


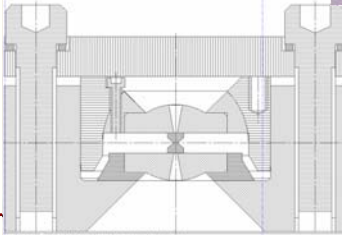
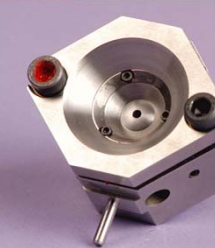



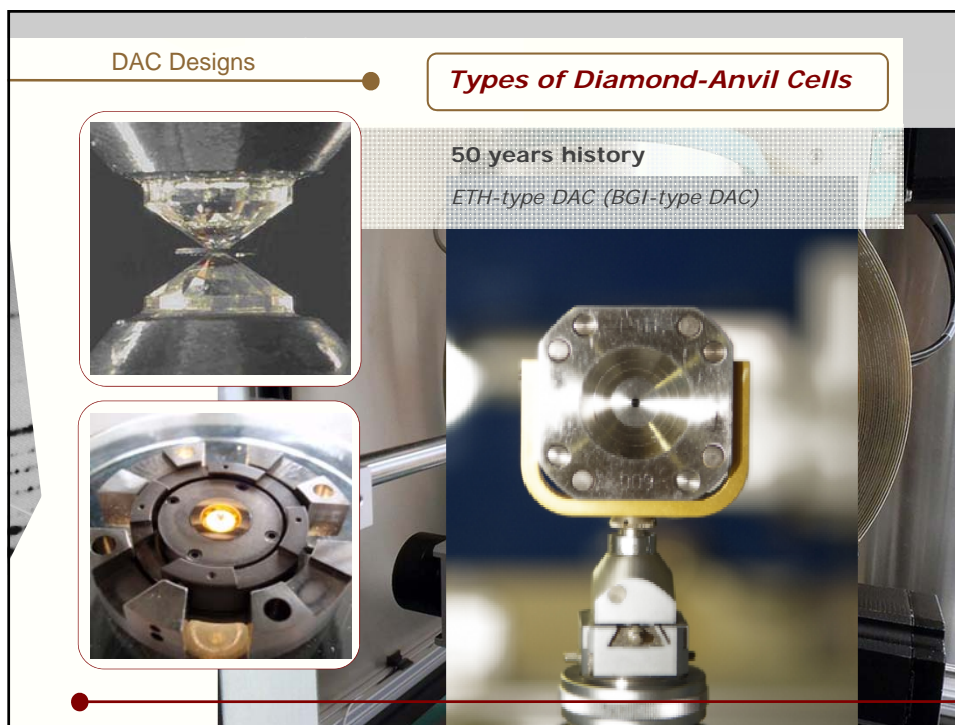
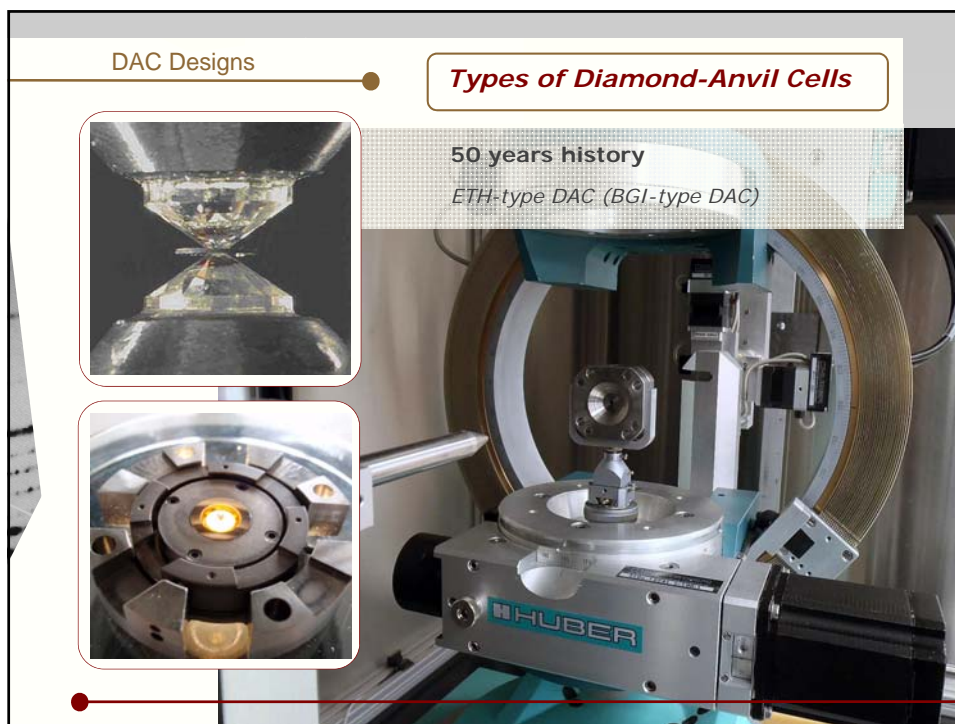
DAC Designs

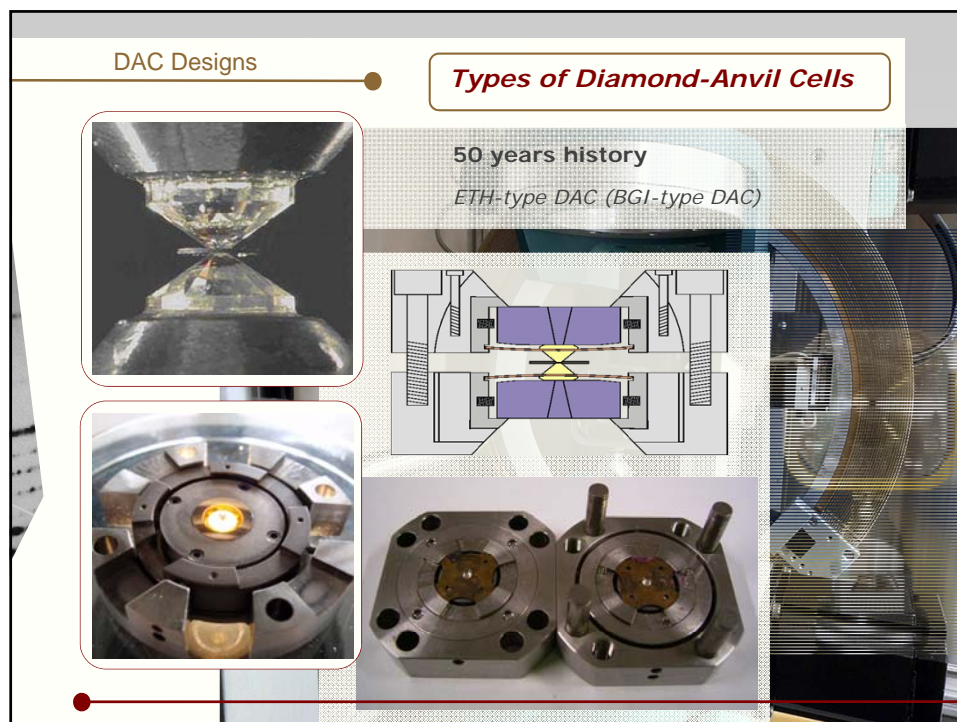


Types of Diamond-Anvil Cells

50 years history
DXR-6 DAC (Diacell Ltd.)

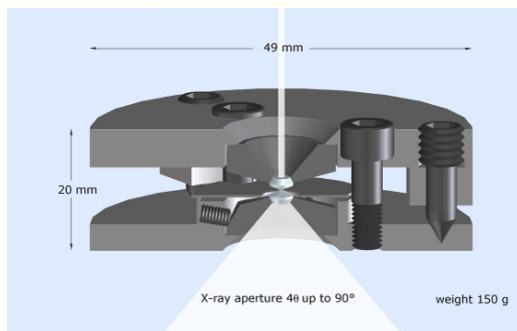
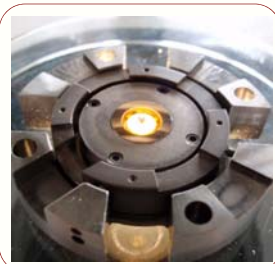
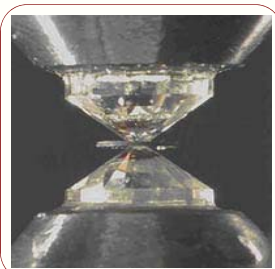




DAC Designs

Types of Diamond-Anvil Cells

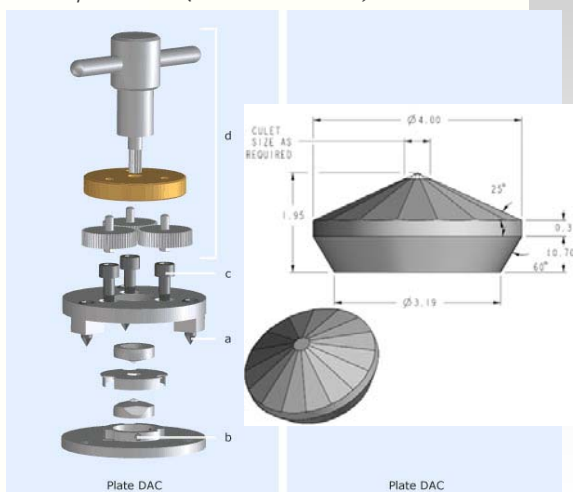
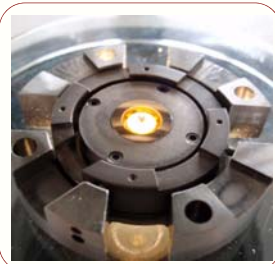
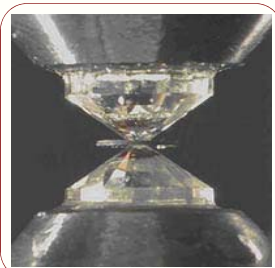
50 years history

plate DAC (Almax Industries)

DAC Designs

Types of Diamond-Anvil Cells

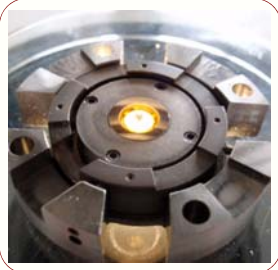
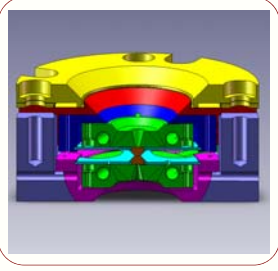
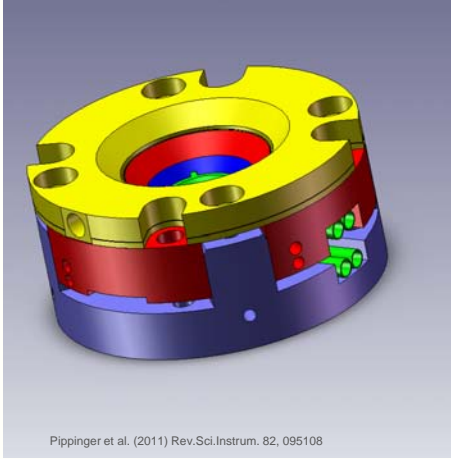
50 years history

plate DAC (Almax Industries)

DAC Designs

Types of Diamond-Anvil Cells

The new HDHPHT-DAC
Heidelberg High-Pressure High-Temperature


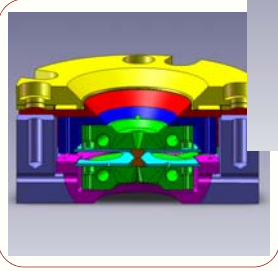
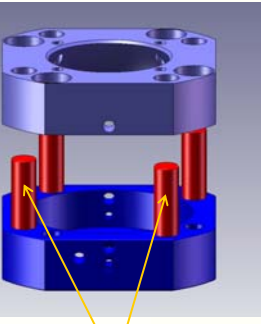
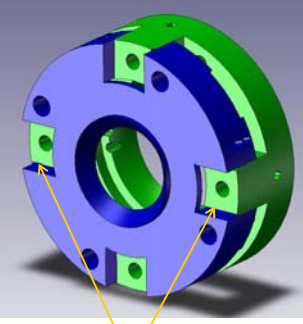
Pippinger et al. (2011) Rev.Sci.Instrum. 82, 095108

DAC Designs

Types of Diamond-Anvil Cells

The new HDHPHT-DAC

new solution

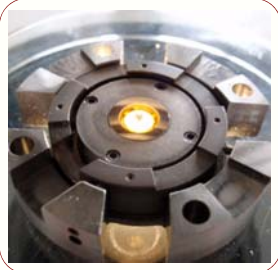
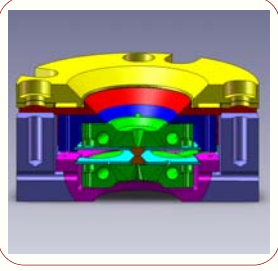
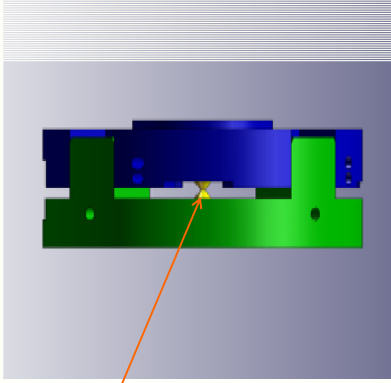
guiding pins

dog clutch guidance

DAC Designs

Types of Diamond-Anvil Cells

The new HDHPHT-DAC (1/13)


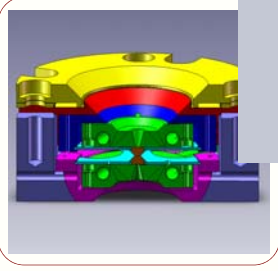
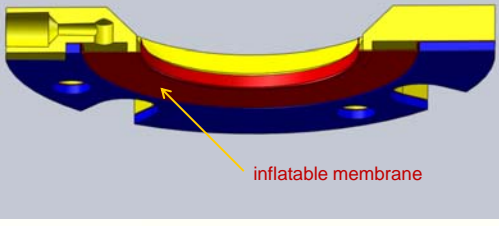
radial optical access

DAC Designs

Types of Diamond-Anvil Cells

The new HDHPHT-DAC

mechanism I: gas-pressure inflated membrane
mechanism II: conventional mechanical bolts

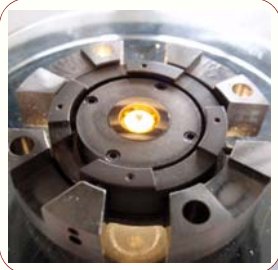
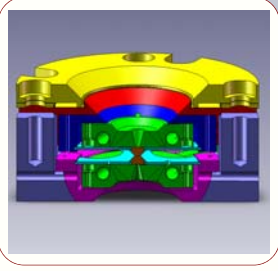
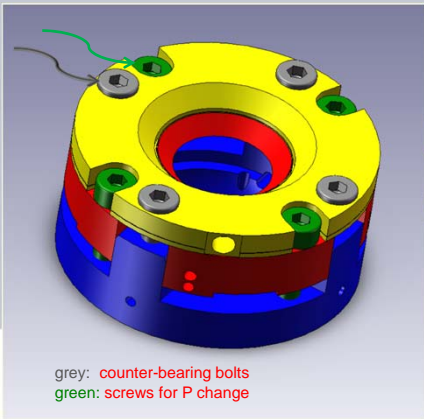
inflatable membrane

dual-mode device for independent pressure generation

DAC Designs

Types of Diamond-Anvil Cells

The new HDHPHT-DAC

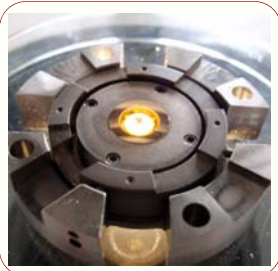
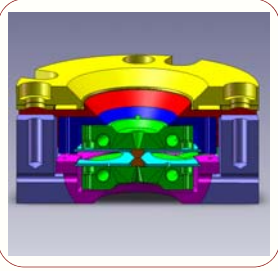
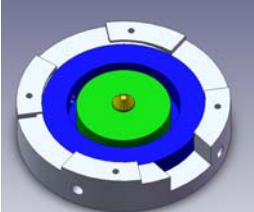
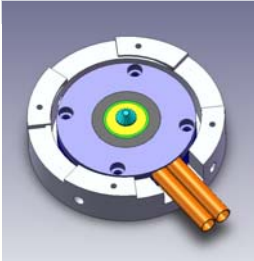




grey: counter-bearing bolts
green: screws for P change

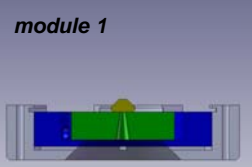
DAC Designs

Types of Diamond-Anvil Cells

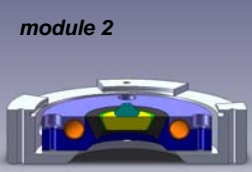
The new HDHPHT-DAC

module 1



module 2

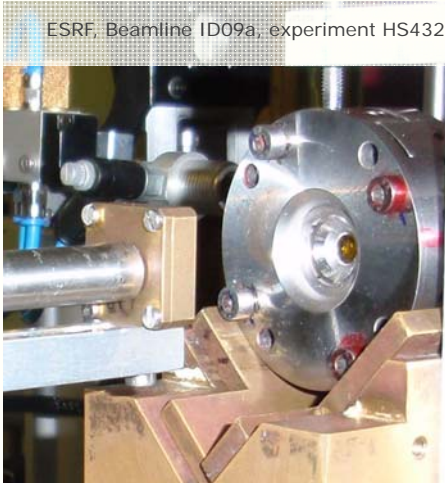
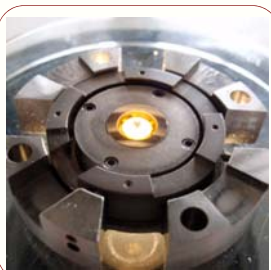
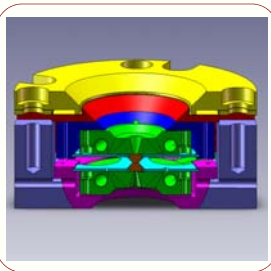


DAC Designs

Types of Diamond-Anvil Cells

The new HDHPHT-DAC

ESRF, Beamline ID09a, experiment HS4323

DAC Designs

Types of Diamond-Anvil Cells

The new HDHPHT-DAC

How to Work with DACs?

see you this afternoon

