Basic considerations for fracture toughness measurements of MPA CVD diamond to be used in nuclear fusion

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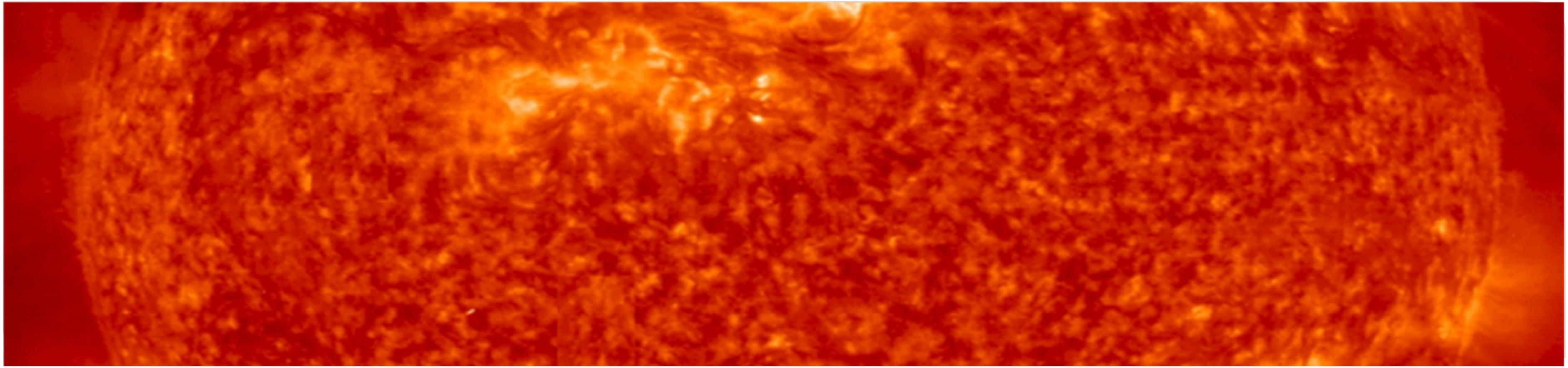
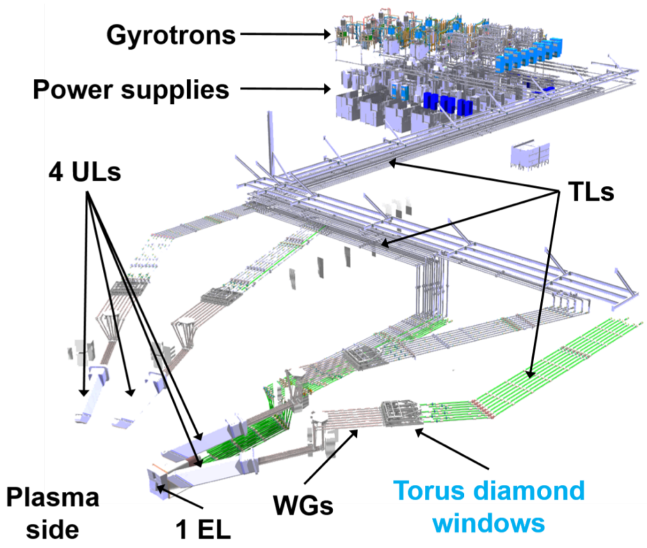
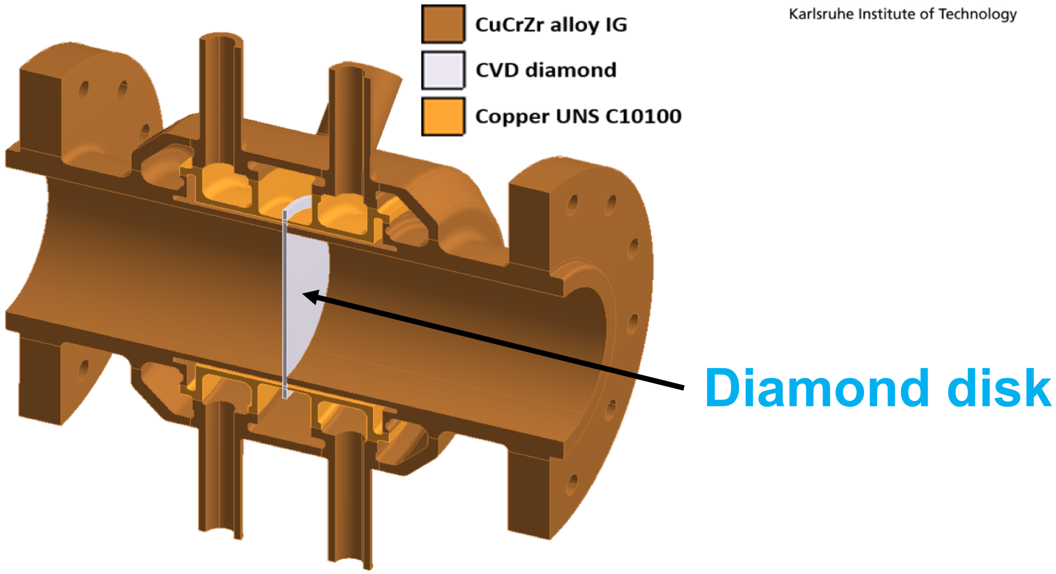


Outline

- Context and basic idea
- MPA CVD diamond and properties
- How to measure the diamond fracture toughness
- Experimental setup and samples
- Characterization techniques
- Numerical analyses
- Summary and outlook

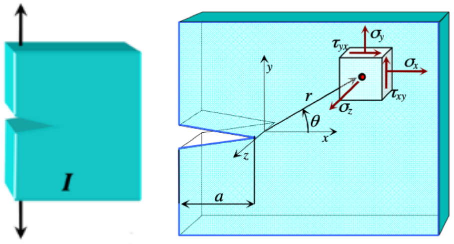
Context





- Fundamental <u>safety role</u> of diamond disks in fusion reactors
- Qualification process of disks based on loss tangent only
- However, <u>failure to fracture</u> is the main failure mode for the disks

Basic idea



$$\sigma_i = \frac{K_I}{\sqrt{2\pi r}} f_i(\theta)$$

- Diamond, LEFM applies
- Stress intensity factor: $K_I = \beta\, \sigma_0\, (\pi\, a)^{1/2}$
- At the critical value, K_{IC} is the fracture toughness
- No crack propagation occurs if $\mathbf{K_I < K_{IC}}$

1. Measure the fracture toughness of MPA CVD diamond
2. Investigate microstructure / defects role on fracture toughness & loss tangent

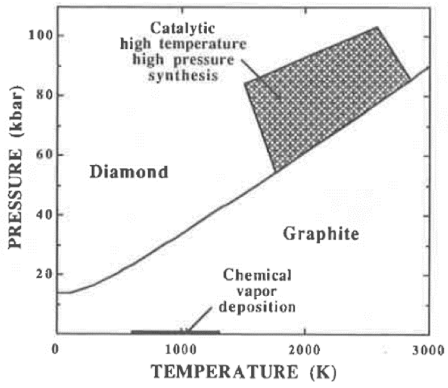
K_{IC}

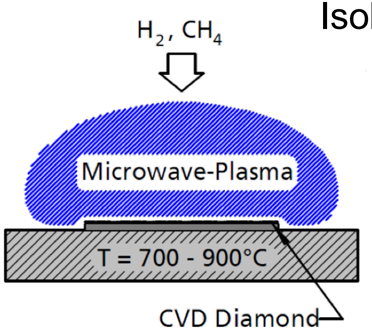
3. Examine series of ITER disks by 3D digital light microscopy
4. Perform numerical analyses for ITER disks worst load scenarios

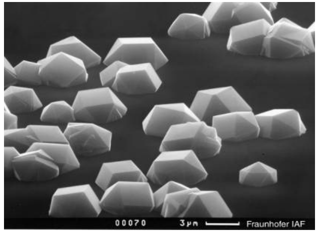
a, σ_0, β

5. Assess the structural integrity of diamond disks

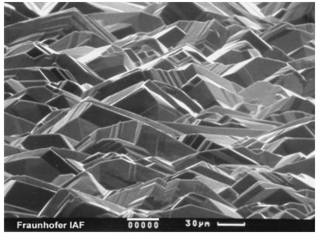
MPA CVD diamond



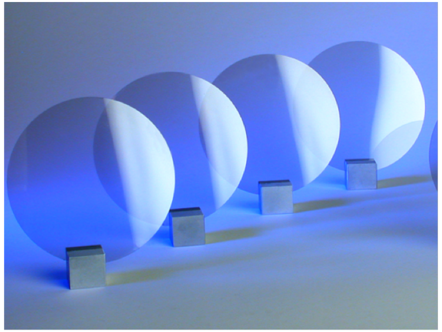


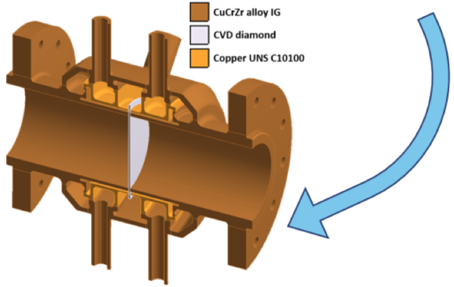


Isolated crystallites (nucleation)



Polycrystalline plate





- Diamond growth by **Microwave Plasma Assisted (MPA) Chemical Vapour Deposition (CVD)**
- **Unique** solution for MW-class, CW operation
- Growth rate of 0.1 to 10 per hour
- Disk **resonant** thickness t = 1.11 mm (ITER)

$$t = n\frac{\lambda_m}{2}$$

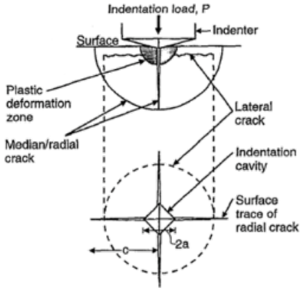
Fracture toughness (K_{IC}) of diamond - literature

Karlsruhe Institute of Technology

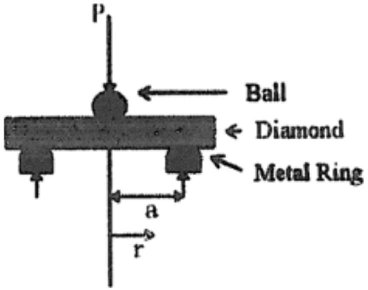
Fracture toughness (MPa m$^{1/2}$)	Error (MPa m$^{1/2}$)	Type of diamond	Thickness (µm)	Shape	Dimensions (mm)	# of samples	Test method	Code	Papers no.	Year
6.3	**-**	**MPA CVD diamond**	**150 to 200**	**Disk**	**⌀25**	**2**	**Tensile test**	**E-399**	**10, 1**	**1995**
5.6	**0.4**	**MPA CVD diamond**	**150 to 200**	**-**	**-**	**8**	**Indentation**		**10**	**1995**
5.3	**1.3**	**MPA CVD diamond**	**400**	**-**	**-**	**11**	**Indentation**		**6**	**1991**
8.7	**0.3**	**MPA CVD diamond**	**880**	**Rectangular**	**13 x 18**	**-**	**Double torsion**		**8**	**1998**
8.3	**0.4**	**MPA CVD optical diamond**	**1000**	**Rectangular**	**13 x 18**	**5**	**Double torsion**		**3**	**2004**
8.5	**1**	**MPA CVD mechanical diamond**	**1000**	**Rectangular**	**13 x 18**	**2**	**Double torsion**		**3**	**2004**
6.5	1.2	Arc-discharge CVD diamond	244 (aver.)	Disk	⌀7 to ⌀16	5	Ball on ring		5	1992
7.6	1.8	Arc-discharge CVD diamond	244 (aver.)	Disk	-	4	Indentation		5	1992
8	-	Arc-discharge CVD diamond	485	Rectangular	2 x 10	9	Three-point	E-399	7	2000
9.2	-	Arc-discharge CVD diamond	485	Rectangular	2 x 10	8	Three-point	E-399	7	2000
4.6	-	Arc plasma jet CVD	300 to 700	Disk	⌀8	-	Ball on ring		13	1998
6	-	CVD diamond	300	-	-	-	Indentation		2	1994
6.8	1.1	Arc plasma jet/hot filament CVD	450	Rectangular	2,5 x 12	3	Three-point	E-399	12	2001
3.4	-	Natural diamond type Ia and IIa	-	-	-	9	Indentation		4	1981
13	-	PDC - cobalt phase	700 (aver.)	Rectangular	~15 x 30	5	Double torsion		11	1994

Methods for K_{IC} measurements - literature

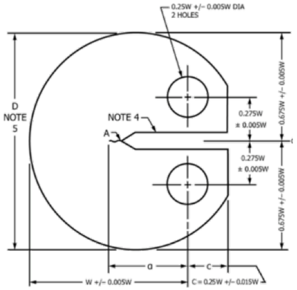
Indentation



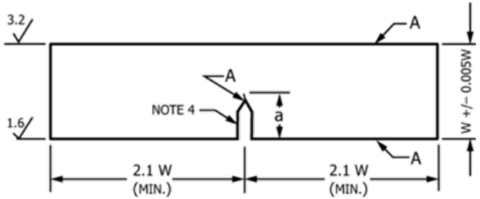
Ball on ring



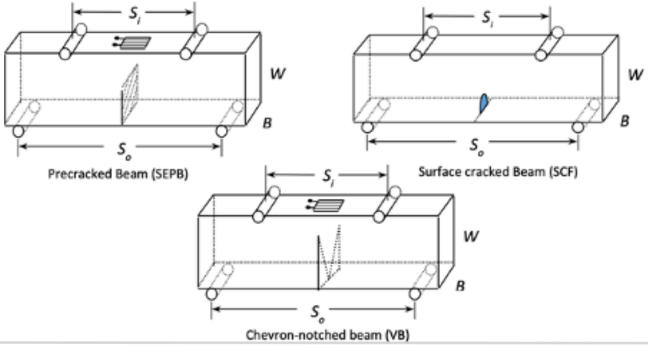
Tensile (ASTM E399)



3 PB (ASTM E399)

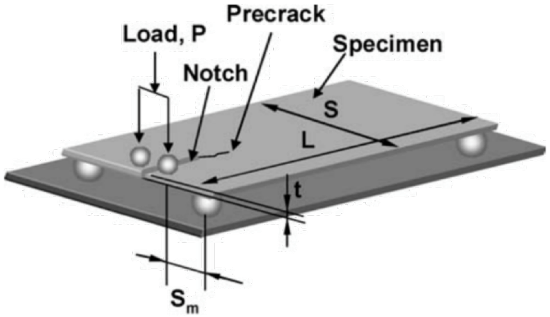


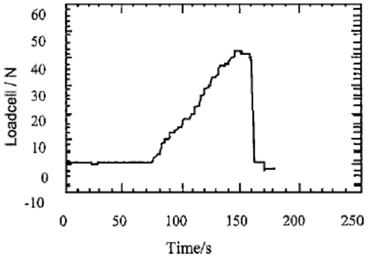
3 PB or 4 PB (ASTM C1421)



- Some methods are only approximate
- Some methods covered by Standard Codes require specifications that cannot be fulfilled for diamond
- The only suited method in our case is the Double Torsion

Double torsion (DT) method: the choice

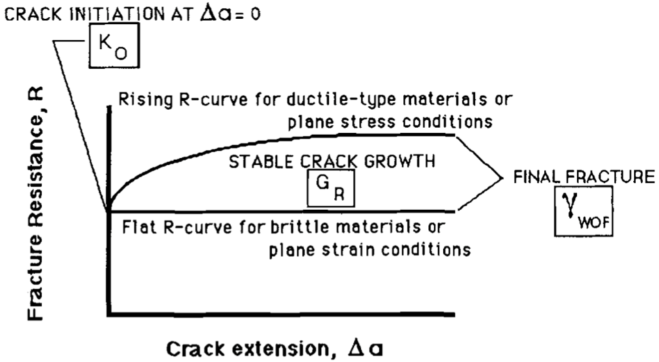




Plane strain

$$K = P S_m \left(\frac{3}{S t^4 (1-\nu) \psi} \right)^{1/2}$$

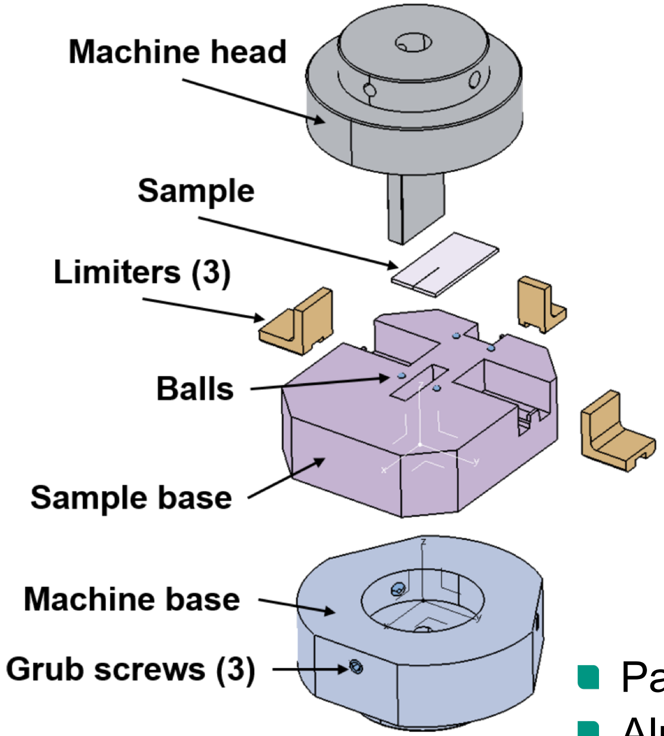
$$\psi = 1 - 0.6302\tau + 1.20\tau \exp(-\pi/\tau)$$



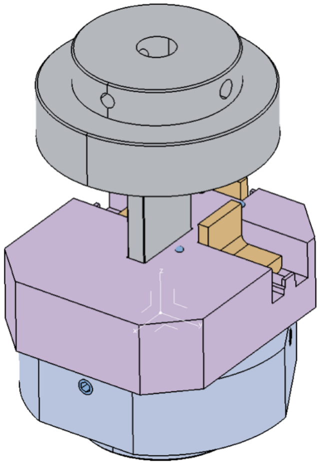
- Method applied to a very extensive range of materials
- However, it has not been standardized yet
- Key features:
  - A relatively simple method
  - K_I independent of crack lenght for a certain range
  - Ideal method for opaque materials

A. Shyam et al., J. Mater Sci 41, 2006

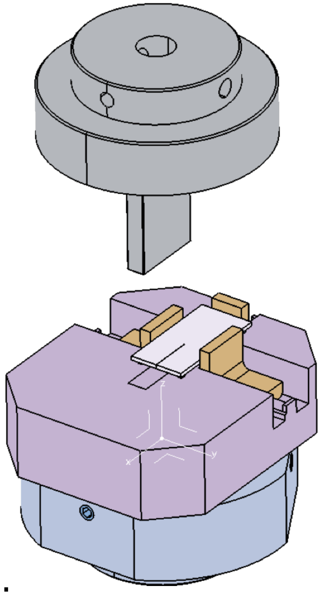
DT method: design of the experimental setup



1. Alignment of parts



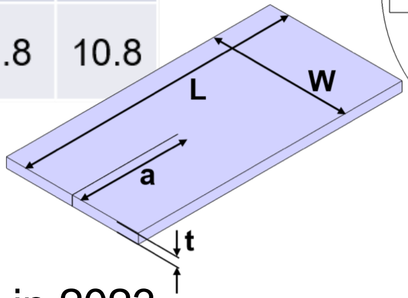
2. Sample positioning

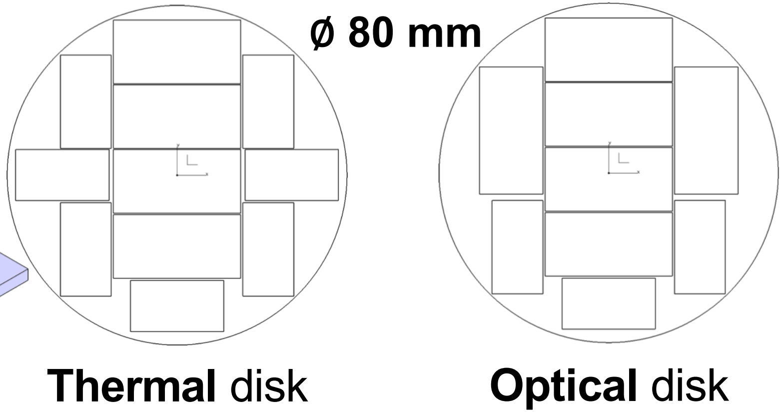


- Parts to integrate in a classical testing machine
- Almost ready to generate drawings for workshop

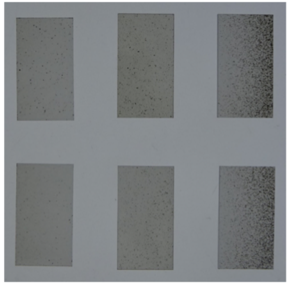
Rectangular-shape diamond samples

	t (mm)	W (mm)	L (mm)	L/W	W/t
Big samples	1.11	15	30	2.0	13.5
Small samples	1.11	12	22	1.8	10.8



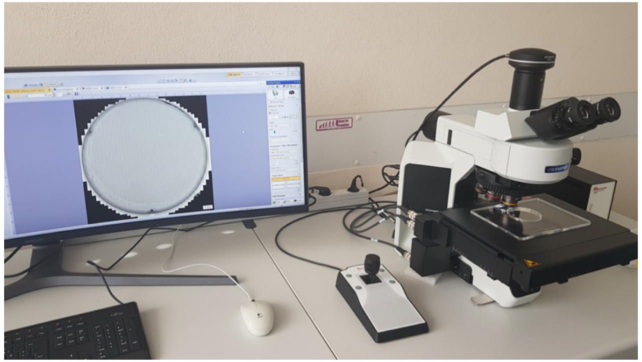


- **Two diamond disks** to fabricate in 2023
- **Best compromise** for number and dimensions of diamond samples
- **High cost** of diamond is the limiting factor for a good statistics in the experimental measurements



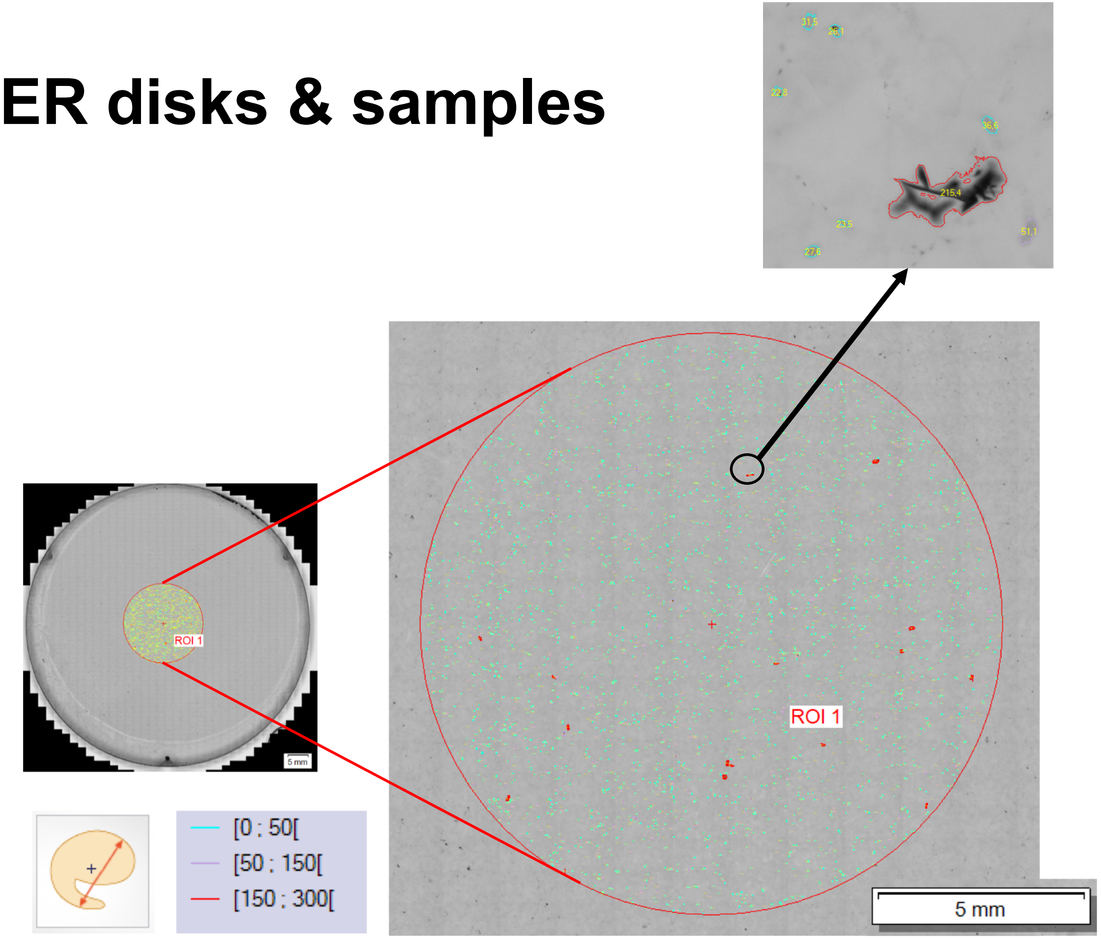
Preliminary samples: 4 optical and 2 thermal small samples

Microscopy activity – ITER disks & samples



Olympus BX53M digital light microscope

- Automatic scans across the thickness (25 layers, 10x lens, 200 GB, ~8 hours)
- Global view on cracks distribution
- Local 3D analysis for detailed cracks configurations (3D cellSens software)



Other characterization techniques

- **Loss tangent measurements**

  Acceptance criteria for ITER disks:
  $3.5x10^{-5}$ for the D50
  $6.0x10^{-5}$ for the D90

- **Raman**

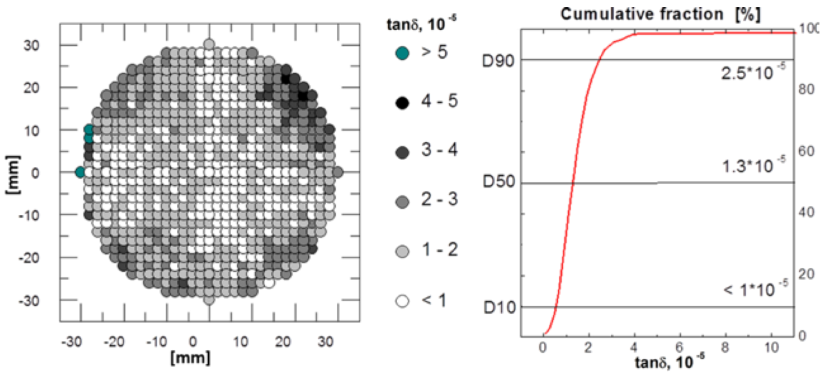
  Test measurements at ISSP, Riga (LV)

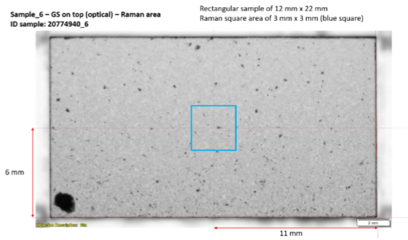
- **EBSD**

  Test measurements on going

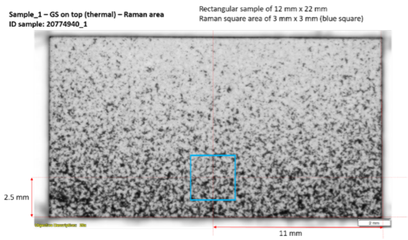
- **XRD, Neutron diffraction**

  To plan yet



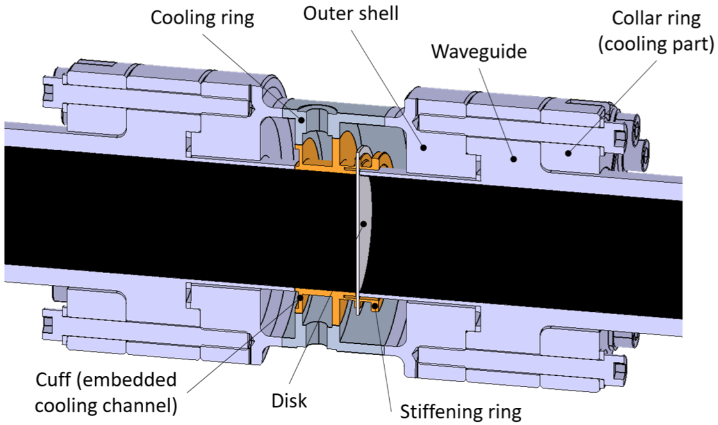


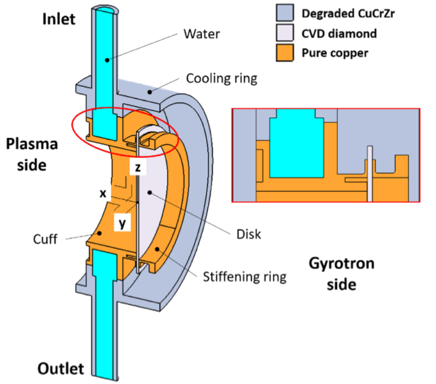
Optical quality
preliminary sample #6

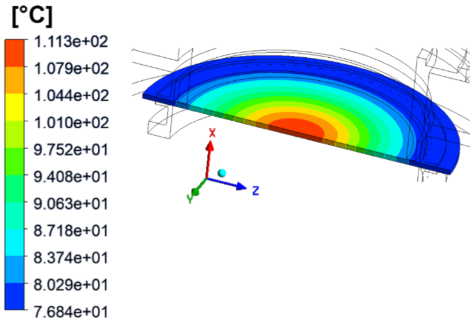


Thermal quality
preliminary sample #1

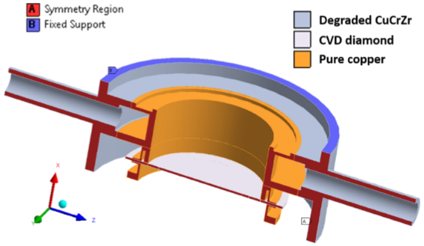
Numerical analyses

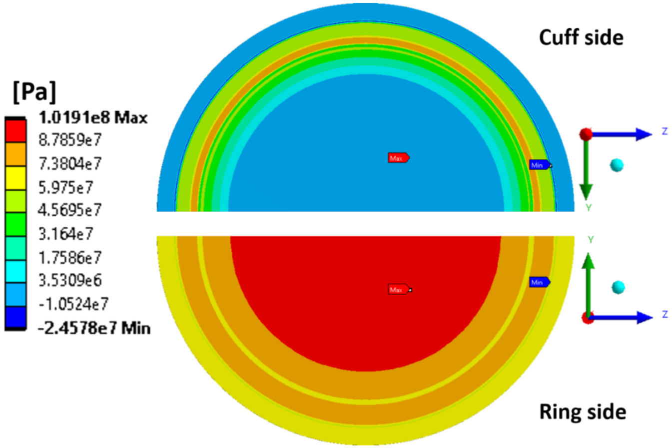






Worst scenario for disk:
NO + 2 bar event





Summary & outlook

- A deeper mechanical characterization of MPA CVD diamond regarding its main failure mode is required
- The method for fracture toughness measurement was selected and the design of the experimental setup carried out
- Numerical analyses for worst load scenario of the diamond disks were performed

- Generate drawings of the setup and carry out the experiments
- Carry on the microstructural investigations on diamond samples and ITER disks

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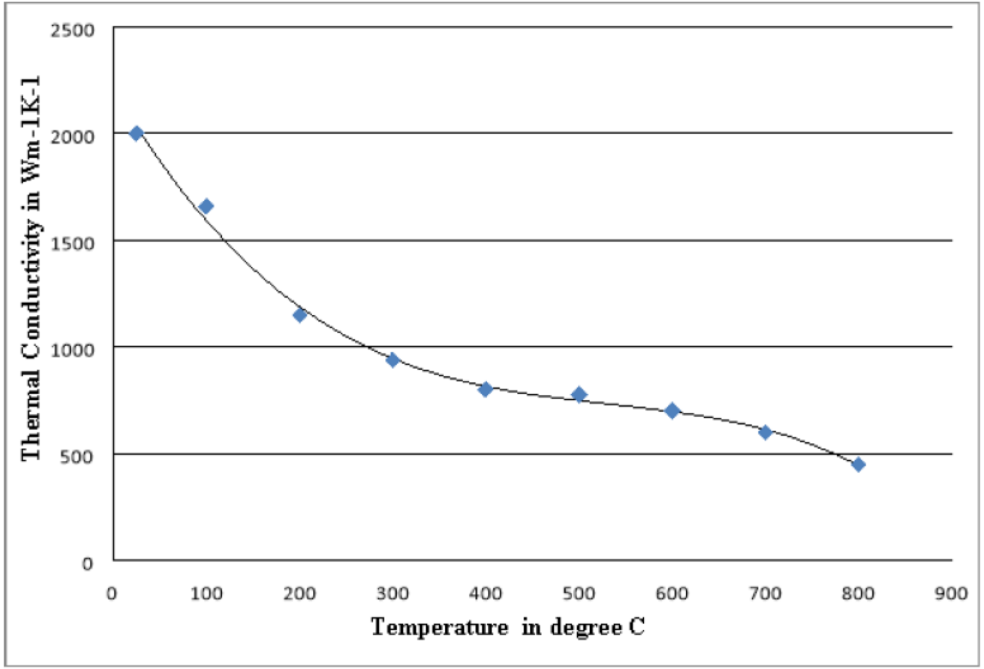
Temperature dependent properties in analyses



- **Design safe limit of 250 °C**
  - Decreasing of thermal conductivity
  - Increasing of loss tangent

A. Pai, ITER_IDM_TMT6EY, 2016