

# Mechanical Testing – Cryogenic Material Characterization

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National Research Center of the Helmholtz Association

•www.kit.edu

## Cryogenic material laboratory within ITEP



- Characterize materials at operational temperatures → 400 K – 4.2 K



- Advantage of combination of test methods in one laboratory with expertise of about 30 years

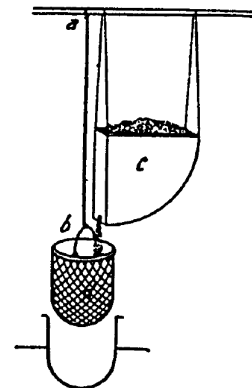
# Content

- Motivation:
  - Structural and Functional Materials
  - Example: ITER Cryogenic Magnet System
- Loads to be considered:
  - Thermal Expansion
  - Thermal Conductivity
  - Lorentz Forces
- Material Tests:
  - Tensile Machines
  - Sensors
- Methods:
  - Tensile Test
  - Fracture Toughness
  - Fatigue Crack Growth Rate
  - Low Cycle Fatigue
  - Impact Test



Leonardo da Vinci  
(1452-1519)

www.vivoscuola.it

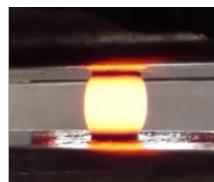


Lund, Byrne; Civil. Eng. and Env. Syst., Vol. 00, pp. 1 ± 8

# Structural and Functional Materials

- Effect of material fabrication and processing  
→ isotropic or anisotropic properties

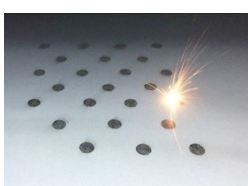
e.g. classical metal casting/forging...



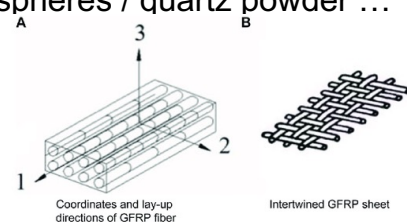
e.g. pure or filled polymers ...

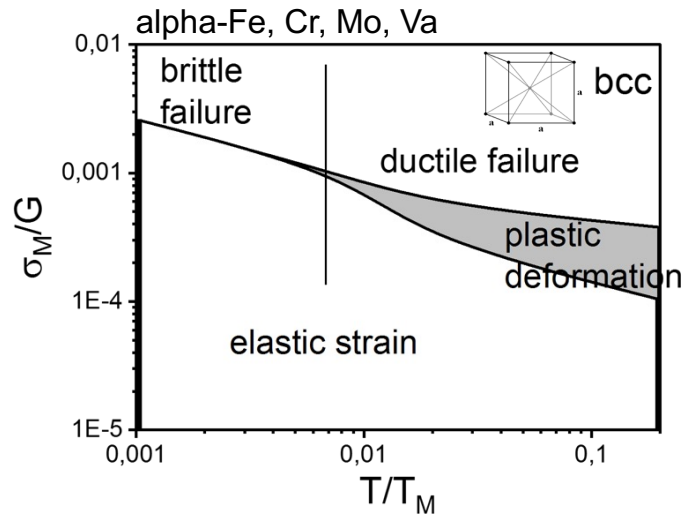
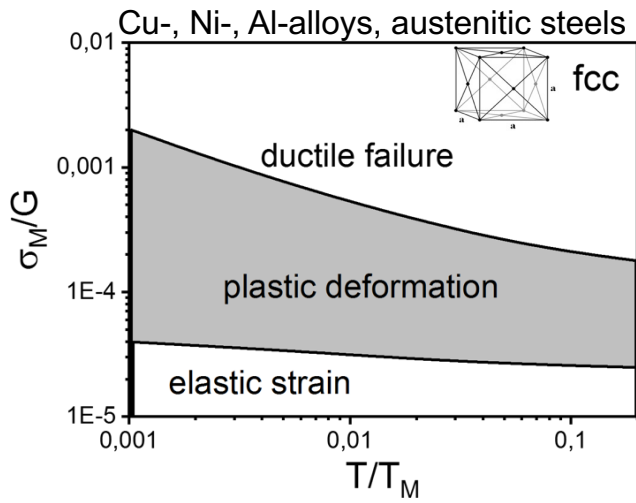


to advanced manufacturing SLM/DED...



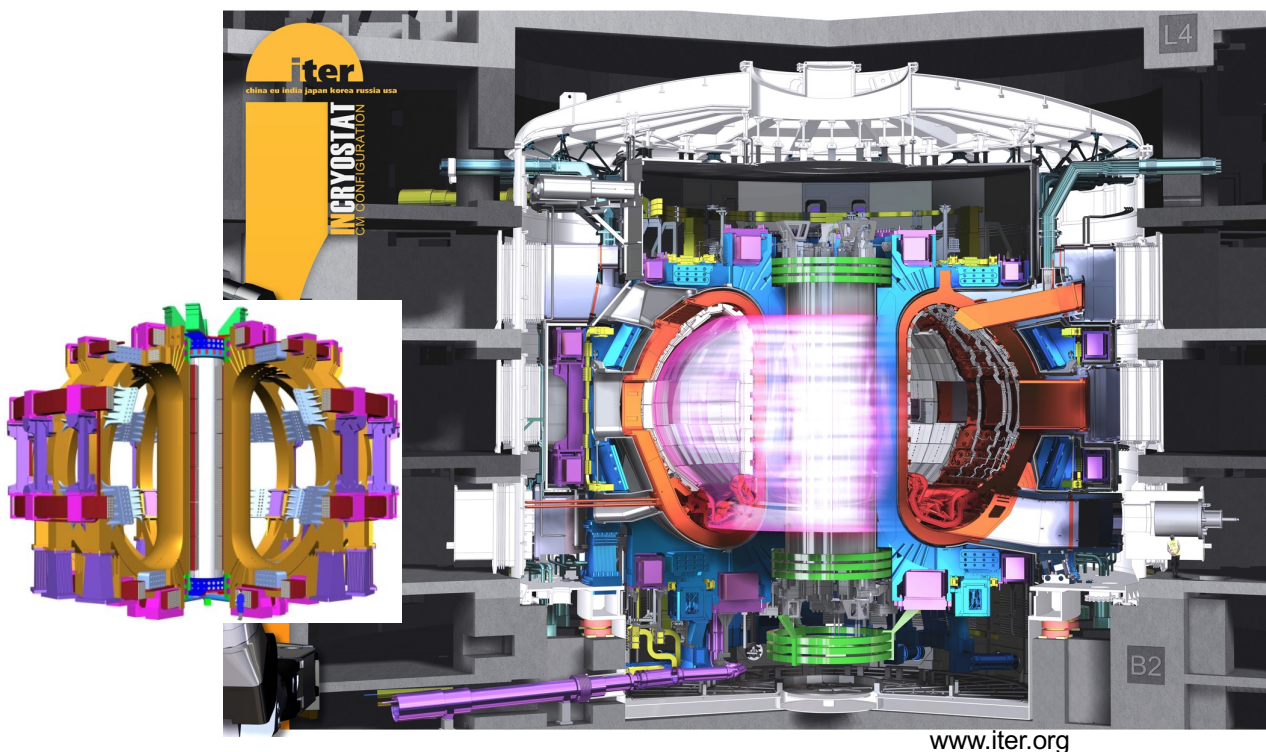
carbon or glass fibers  
glass spheres / quartz powder ...





Adjustment of mechanical properties via grain-size, precipitation, solution hardening  
Alloys can show complex behaviour

## Example: ITER Cryogenic Magnet System

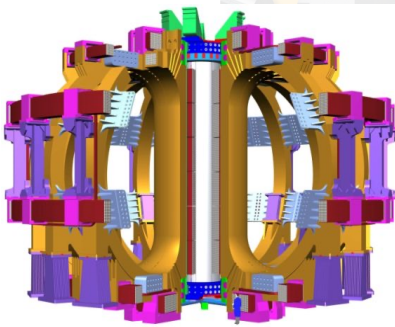




# Example: ITER Cryogenic Magnet System

The cold mass of the magnet system contains:

structural material (mainly austenitic steel) and  
 the superconducting cable (copper, Nb3Sn, electrical insulation, steel)  
 ... and many, many joints



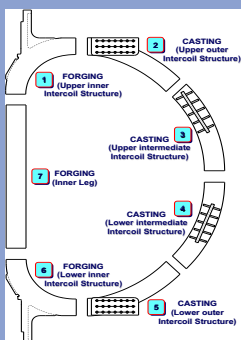
Loads to be considered:

- Gravitational forces due to the mass of the system
- Thermal forces due to cool down of the coils
- Lorentz forces due to the magnetization

www.iter.org

# Example: ITER Cryogenic Magnet System

## Structural material

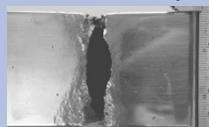
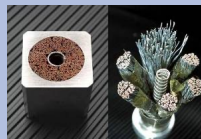


Modified 316LN  
 Cast, forged & rolled plates



## Superconducting cables

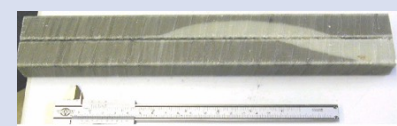
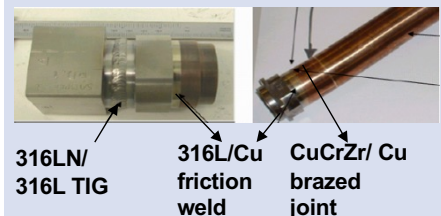
- Conduit  
 Type 316LN  
 Incoloy 908  
 Aluminium
- Stabilizer  
 OFHC copper
- Insulation  
 GFRP / Epoxy
- Superconductor  
 Nb<sub>3</sub>Sn TF/CS  
 NbTi / PF  
 HTSC for current leads  
 CS conduit (51 mm x 51 mm)  
 fatigue tests



## Joints

- Welding
- Solder
- Adhesive bonding

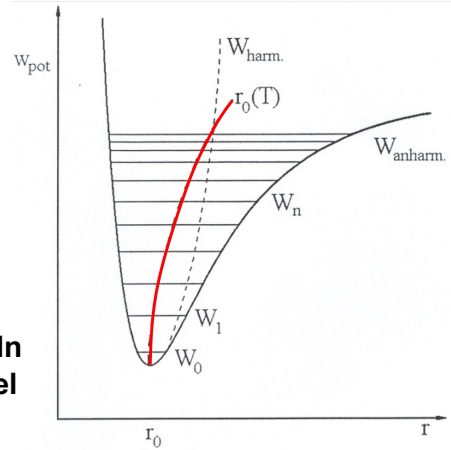
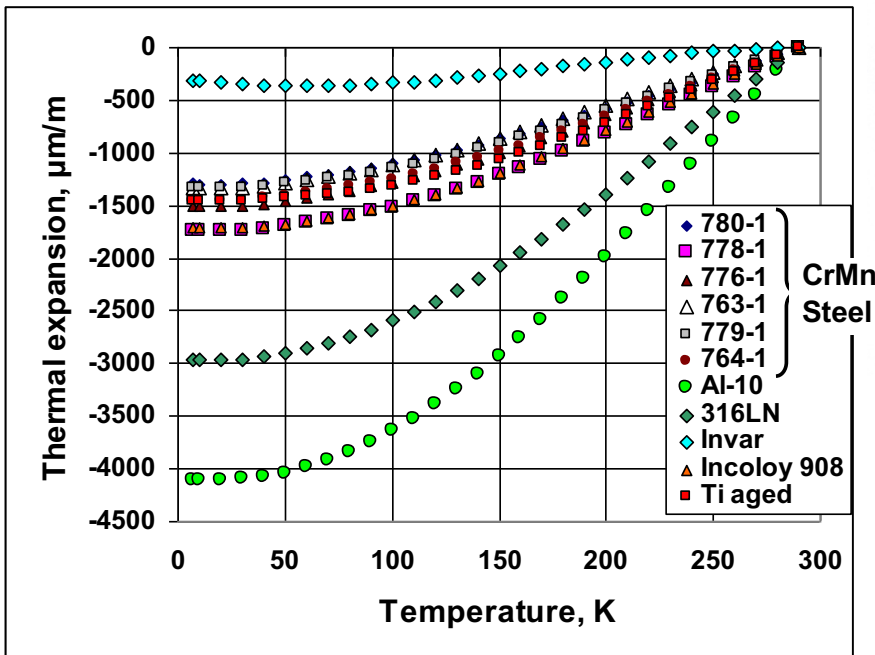
e.g. cable joints



3-point bending of  
 insulation material



# Thermal Expansion



**TE 290 K - 4 K:**  
 PA 13.500 µm/m  
 Polyamide  
 PEEK 10.200 µm/m  
 Polyetheretherketone

# Thermal Conductivity

Typical temperature dependences of thermal conductivity

**High thermal conductivity**

caused by

- electrons
- phonons

**Medium thermal conductivity**

Electrons & phonons exist but are blocked by scattering

**Low conductivity**

No electrons & almost no phononic components

# Lorentz Forces

The Force on a conductor in a magnetic field results from  $F = l \cdot B \times I$   
(Lorentz-Force)

- e.g. for the TF coil:  $I = 68 \text{ kA}$ ;  $B = 11.5 \text{ T}$  at the conductor

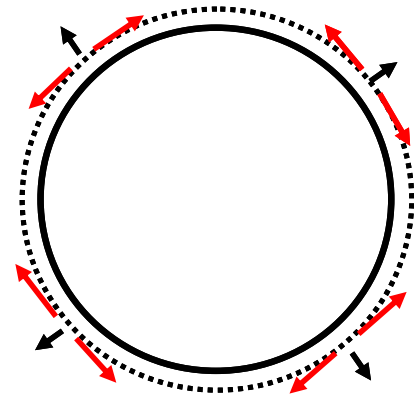
this results in a radial force per length

$$F = I \cdot B = 68 \cdot 11.5 \text{ kA T} = 782 \text{ kA N/(Am)} = 782 \text{ kN/m}$$

- to counteract the **radial force**

the conductor is exposed to a **hoop stress**

that stretches the conductor.



# National & International Standards

DIN 50125	Prüfung metallischer Werkstoffe - Zugproben
DIN EN ISO 6892-1	Metallic materials - Tensile testing - Part 1: Method of test at room temperature
DIN EN ISO 6892-2	Metallic materials - Tensile testing - Part 2: Method of test at elevated temperature
DIN EN ISO 6892-3	Metallic materials - Tensile testing - Part 3: Method of test at low temperature
DIN EN ISO 6892-4	Metallic materials - Tensile testing - Part 4: Method of test in liquid helium
ISO 12135	Metallic materials - Unified method of test for the determination of quasistatic fracture toughness
DIN EN ISO 3506-1	Fasteners - Mechanical properties of corrosion-resistant stainless steel fasteners - Part 1: Bolts, screws and studs
DIN EN ISO 3506-2	Fasteners - Mechanical properties of corrosion-resistant stainless steel fasteners - Part 2: Nuts

# National & International Standards

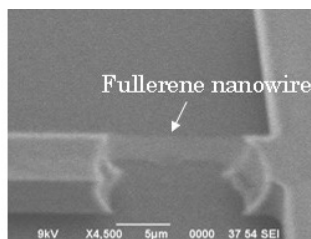
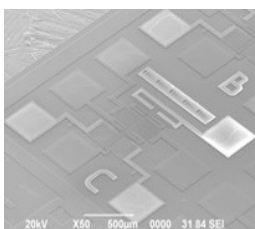
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DIN EN ISO 6892-3	Metallic materials - Tensile testing - Part 3: Method of test at low temperature
DIN EN ISO 6892-4	Metallic materials - Tensile testing - Part 4: Method of test in liquid nitrogen
ISO 12135	Metallic materials - Determination of the yield strength
DIN EN ISO 3506-1	Fasteners - stainless steel - Hex bolts
DIN EN ISO 3506-2	Fasteners - stainless steel - Hex nuts

- 1) Property of interest
- 2) Read the standard
- 3) Follow or adapt the procedure to your needs

- ASTM E 74 for load verification devices. The primary standards are masses of weights accurate to 0.005% of their values.
- ASTM E 83, "Standard Practice for Verification and Classification of Extensometers"
- ASTM E 1823 "Standard Terminology Relating to Fatigue and Fracture Testing"
- ASTM E 399 "Standard Test Method for Linear-Elastic Plane-Strain Fracture Toughness of Metallic Materials"
- ASTM E 1820 "Standard Test Method for Measurement of Fracture Toughness"
- ASTM E 647 "Standard Test Method for Measurement of Fatigue Crack Growth Rates"
- ASTM E 23 "Standard Test Methods for Notched Bar Impact Testing of Metallic Materials"

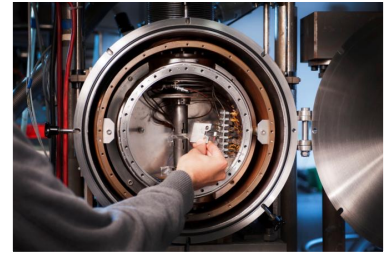
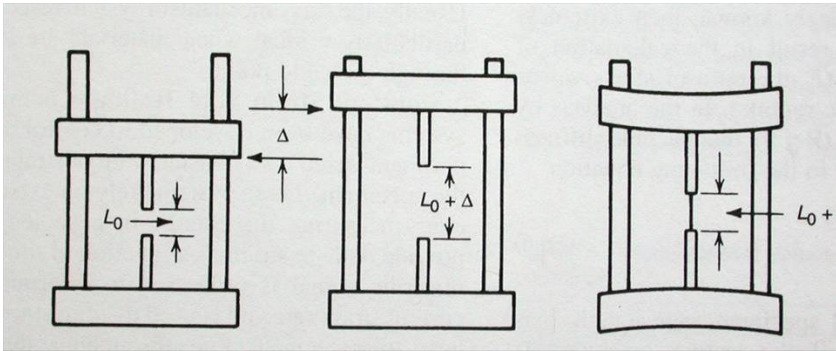
# Tensile Machines

Piezo-electrical,  
 electro-mechanical or  
 servo-hydraulic test facilities  
 for applied loads ranging from Nano- up to Giga-Newton





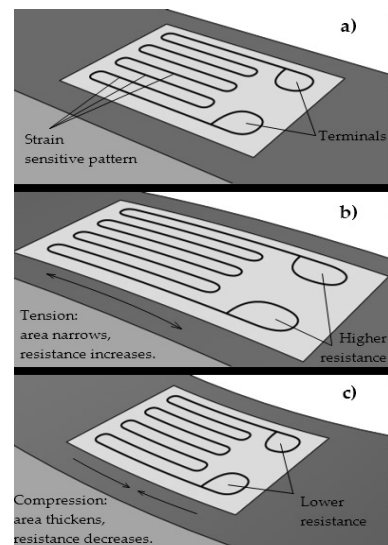
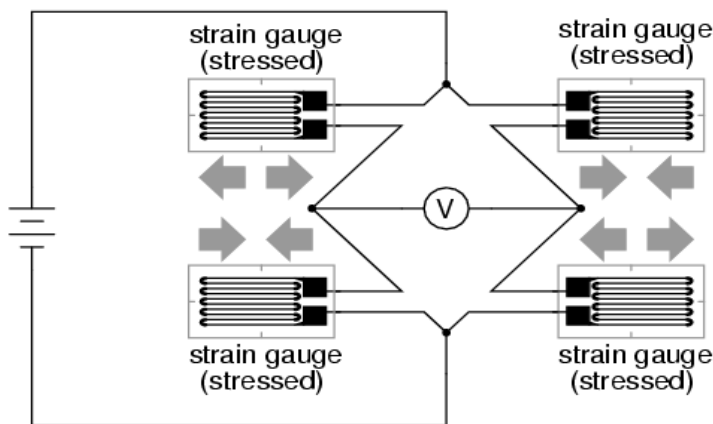
# Tensile Machines



- stiffness of facility
- alignment of load-line and specimen
- mounting of sensors:
  - load cell in series (possible bias by bellows)
  - extensometer in parallel on or close to specimen (possible bias by facility stiffness or thermal exp.)
- force free mounting of specimen (possible bias by cooling/heating)
- maintain stable temperature during test

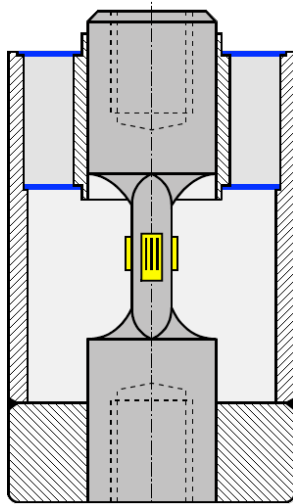
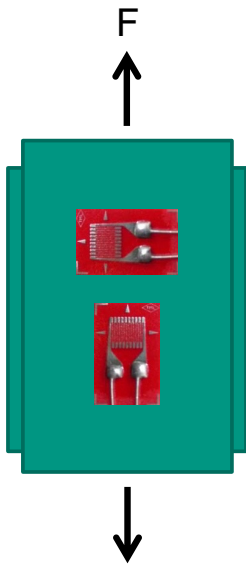
# Sensors: Strain Gauge

Full-bridge strain gauge circuit

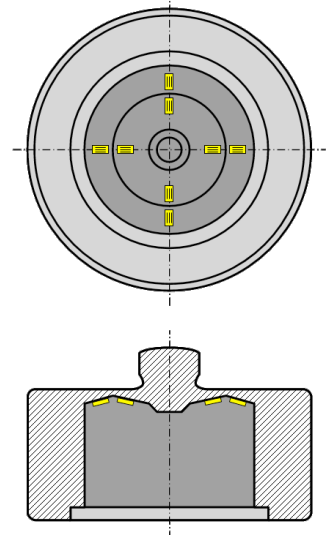


# Sensors: Load Cell

- Using elastic behavior of materials (e.g. CuBe, steel alloys...) strain gauges are used for load cell application



cylindrical rod - elastic body

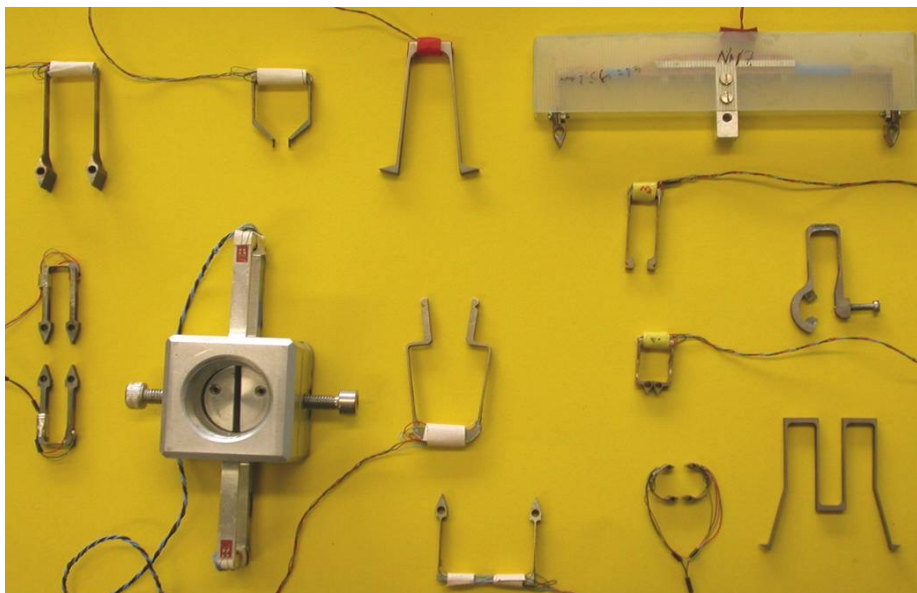


membrane - elastic body

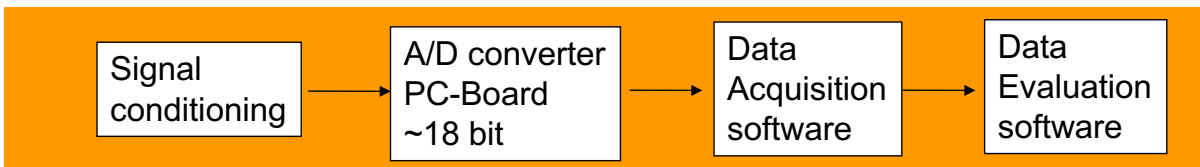
# Sensors: Extensometer and Data Acquisition



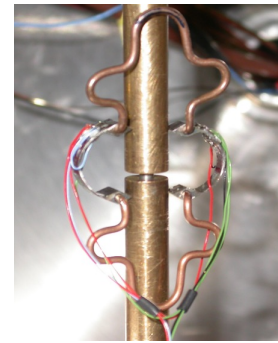
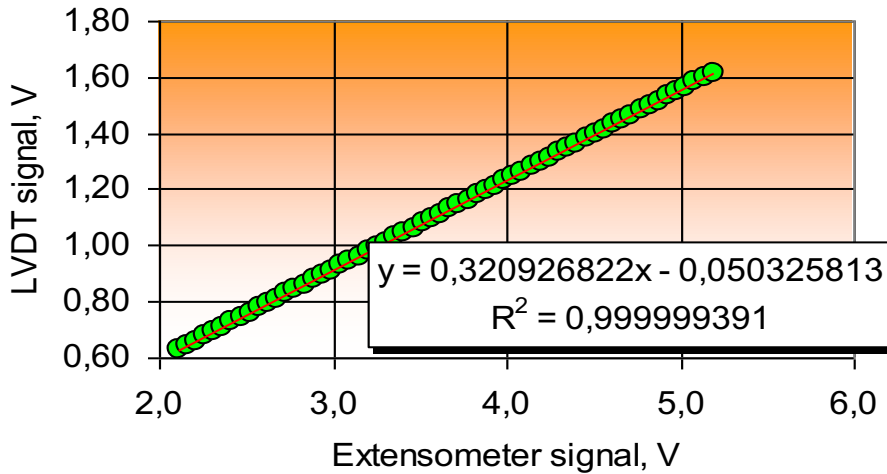
Physical world



Signal conditioning



# Sensors: Extensometer Calibration

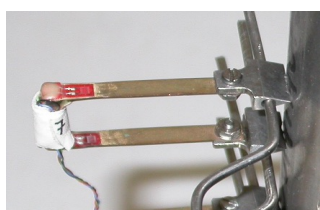
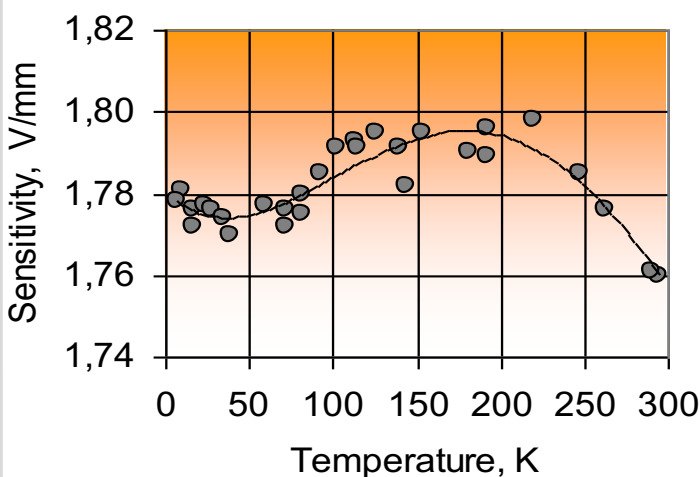


Extensometer under calibration with 12 mm starting length (mass = 0.5 g).

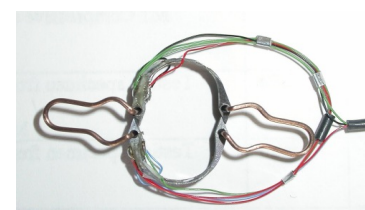
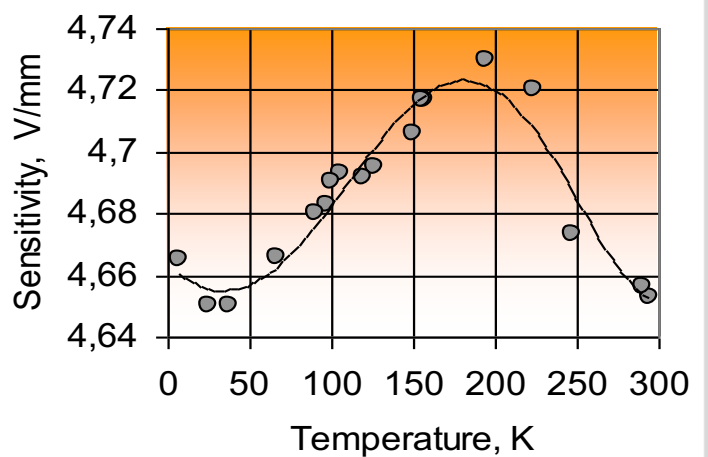
Diagram shows LVDT versus extensometer signal during defined displacement of tensile machine



# Sensors: Temperature Influence on Calibration



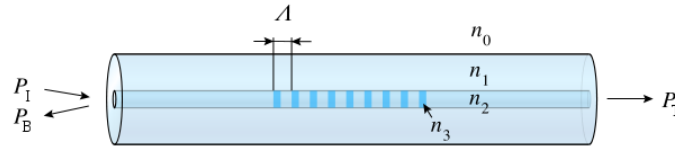
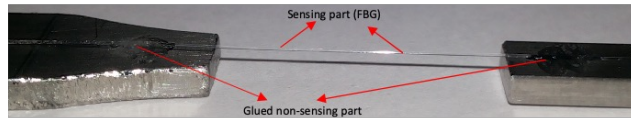
Type A



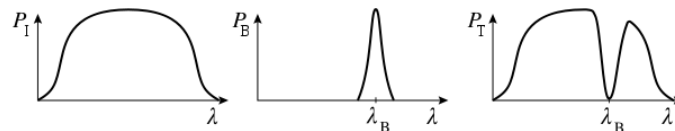
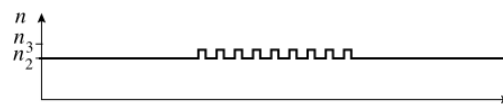
Type I



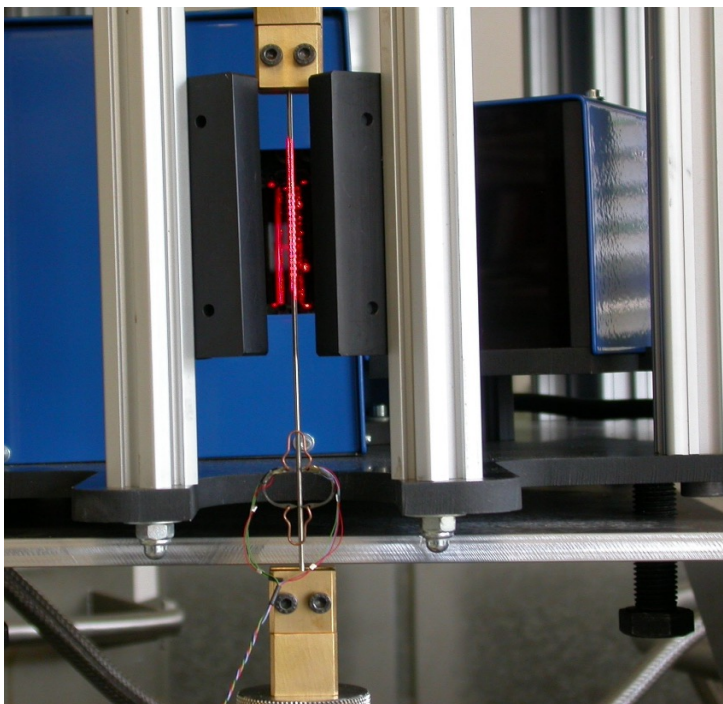
# Sensors: Fiber Bragg Optic for Strain Measurement



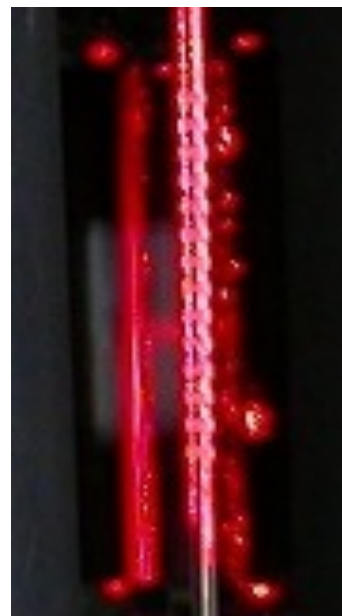
Parameter	Specification
Company	FBGS
FBG type	DTG-LBL-830_1550
FBG diameter (coated)	195 $\mu\text{m}$
Initial Bragg wavelength	$\sim 1550 \text{ nm}$
Braggmeter accuracy	$\pm 2 \text{ pm}$
Glue used	M-Bond 610 Epoxy-phenol adhesive



# Sensors: Double Laser Beam for Strain Measurement



Macroscopic stepwise change of reflectivity (dark/bright)

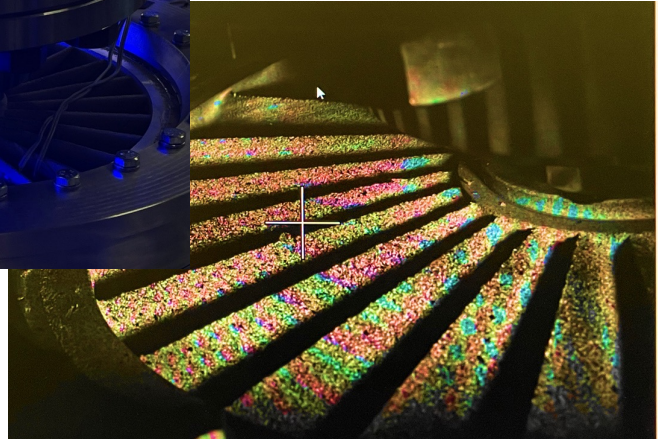
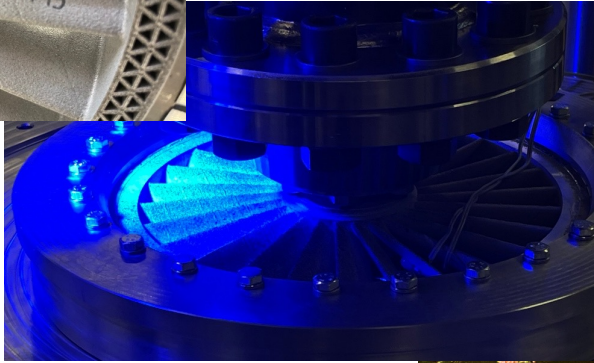


# Sensors: Digital Image Correlation (DIC)

1D Laser Beam → 2D/3D Differential Imaging Correlation



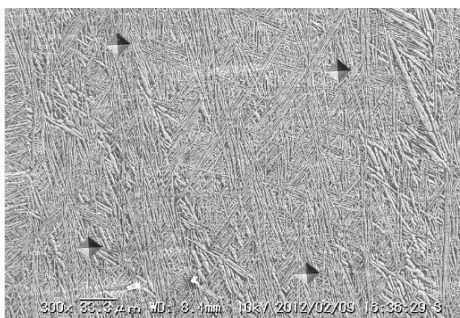
black/white spray paint for surface recognition by optical camera (high fps)



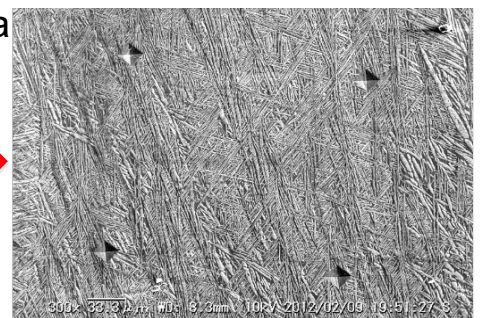
# Sensors: Digital Image Correlation (DIC)

before deformation

after deformation

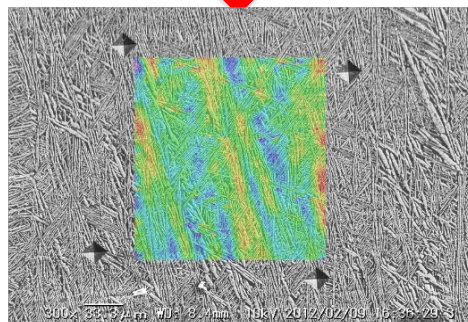
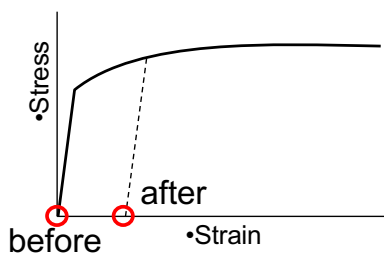


SEM image of same area



comparison

calculate strain

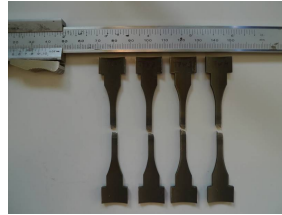


resolved local strain

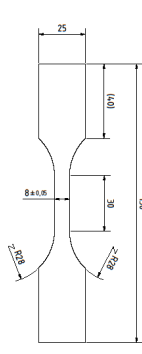
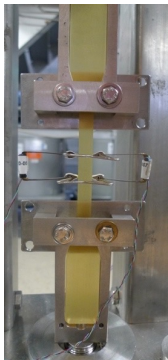
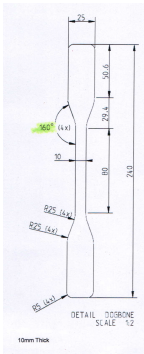


# Tensile Test

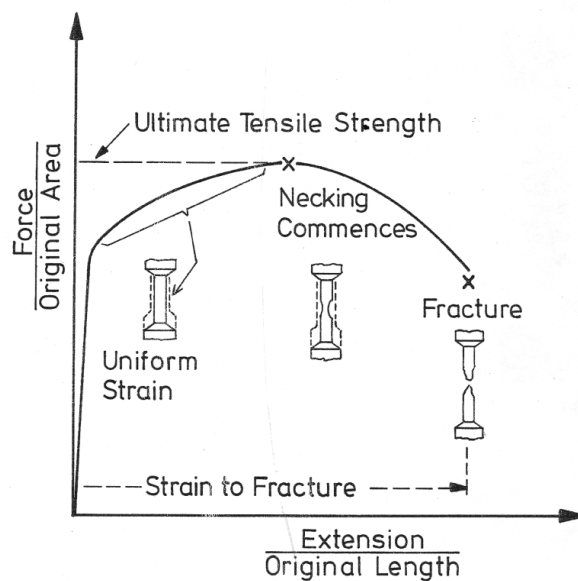
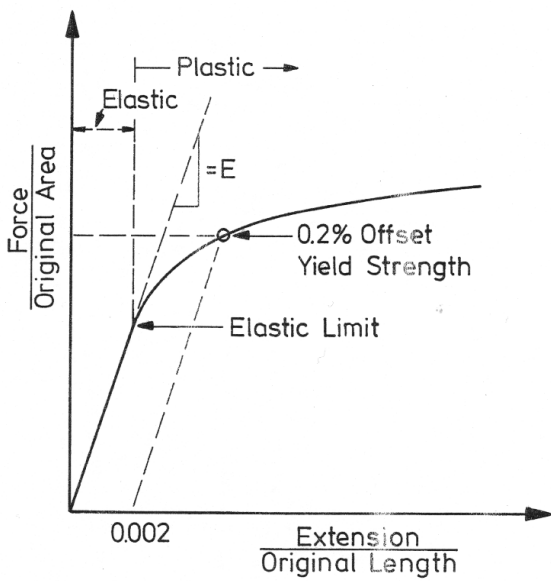
## ISO 6892 / ASTM E8/E8M specimen size metallic materials



## ISO 527 & ASTM D638 plastic & D3039/D3039M fiber reinforced tensile



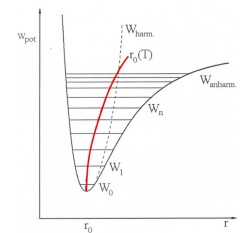
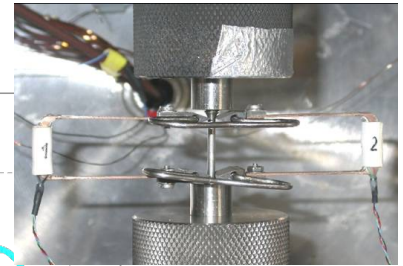
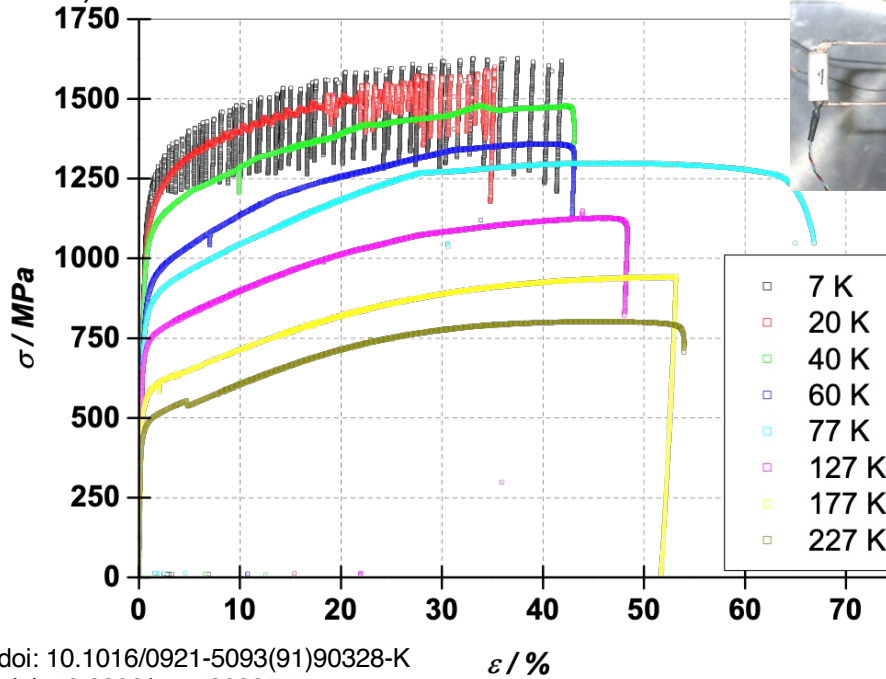
# Tensile Test





# Tensile Test

## 316LN (1.4429) tensile test series



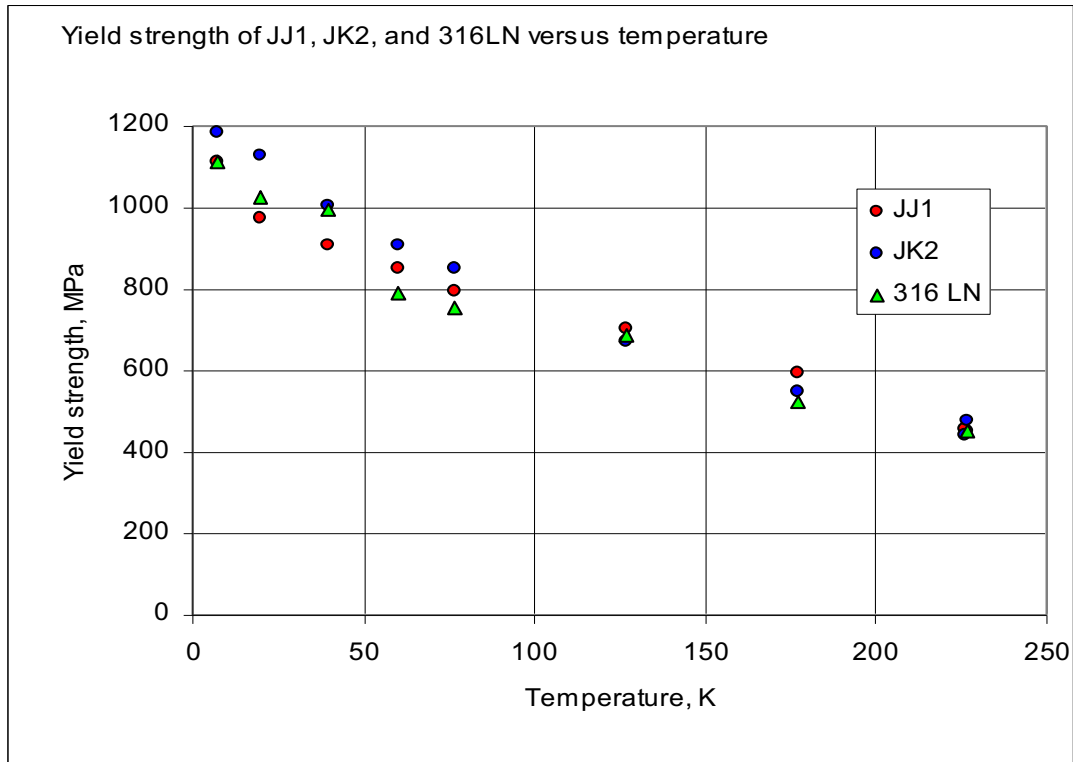
Obst, Nyilas doi: 10.1016/0921-5093(91)90328-K  
 Tirulina et al doi: 10.3390/met12030514

# Tensile Test

Database – Material Information Sheet	
Alloy:	Type 316LN
Designation/heat No.:	Model 1, 66 ton forging
Heat-treatment:	Solution heat treated
Fabrication process:	66 ton Ingot forging
Material use:	ITER TF- case inner leg coil structure
Manufacturer:	Ingot Thyssen, Krefeld, Germany, Forged by KIND, Germany

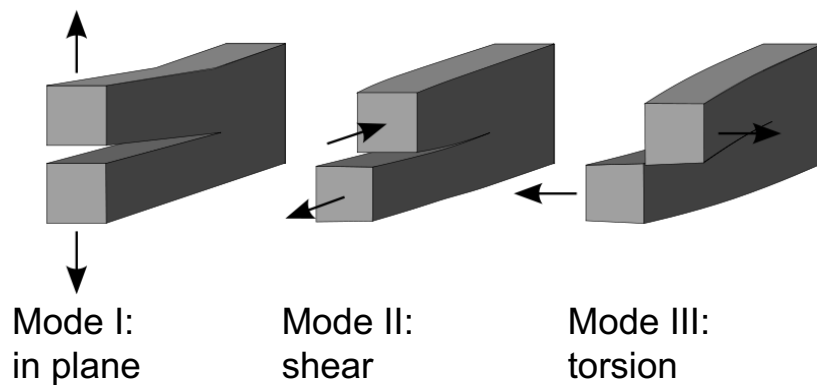
Composition, weight %																
C	Si	Mn	P	S	Cr	Ni	Mo	N	Cu	Ca	Co	Al	V	O	B	Mg
0.018	0.370	2.01	0.028	0.002	17.23	13.46	2.52	0.182	-	-	-	0.009	0.060	-	-	-

File & heat	T K	Young's Modulus GPa	Yield Strength MPa	UTS MPa	Total elongation %	Reduction of area %
KIND-227	227	175	453	824	58,3	
KIND-177	177	186	521	943	53,2	
KIND-127	127	185	686	1125	49,9	73,7
KIND-77	77	185	755	1268	66,5*	65,5
KIND-60	60	187	790	1326	43,4	66,9
KIND-40	40	193	996	1480	42,8	60,9
KIND-20	20	193	1028	1600	35,7	47,4
KIND-7	7	197	1111	1628	41,9	43,8



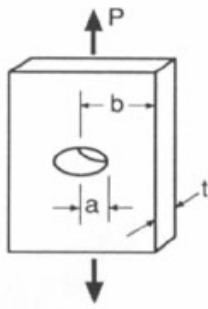
# Fracture Toughness

Concept of fracture mechanics  
Crack shapes and geometry factors

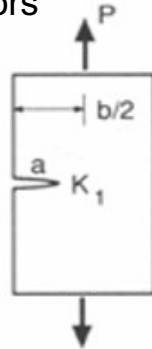


# Fracture Toughness

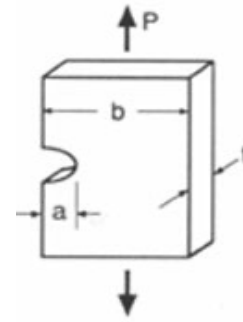
Concept of fracture mechanics  
Crack shapes and geometry factors



Interior crack  
 $Y = 1.00$



through-thickness  
surface crack  
 $Y = 1.12$



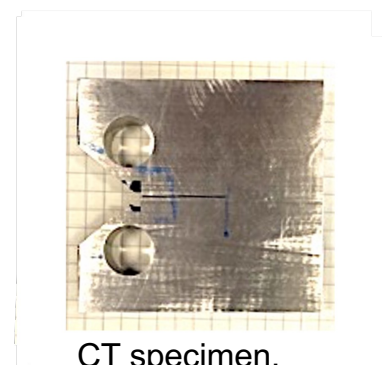
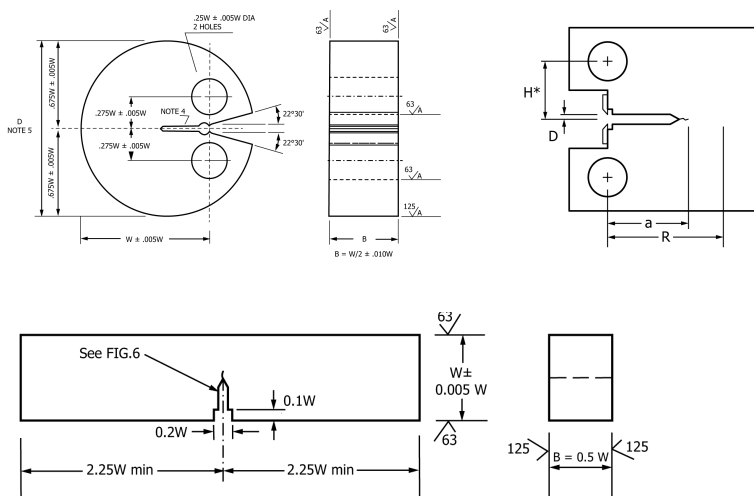
half-circular  
surface crack  
 $Y = 0.73$

Stress intensity

$$K_I = Y \cdot \sigma \sqrt{\pi \cdot a}$$

# Fracture Toughness / Fatigue Crack Growth Rate

■ ISO 12135 / ASTM E1820 fracture toughness

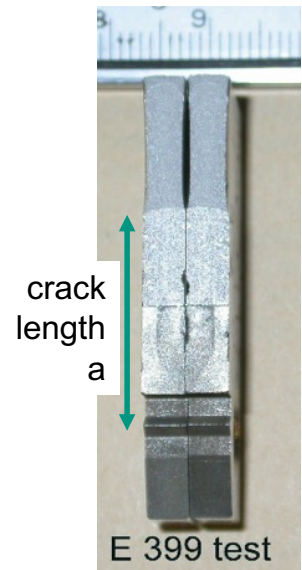
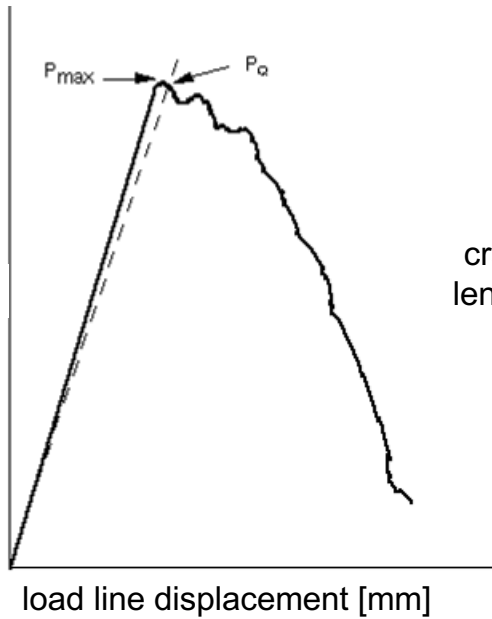
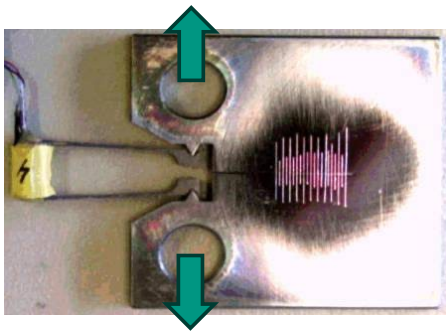


CT specimen,  
43 x 45 x 15 (mm)



# Fracture Toughness

LEFM - linear elastic fracture mechanics [K-concept], ASTM Standard E 399  
 Quasi-static loading until sudden failure

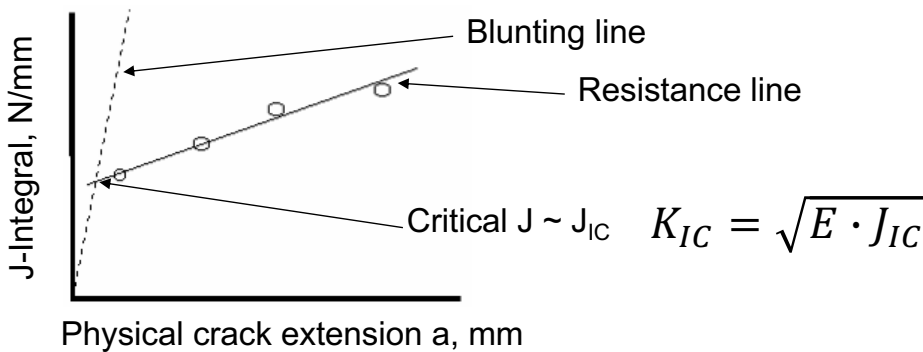
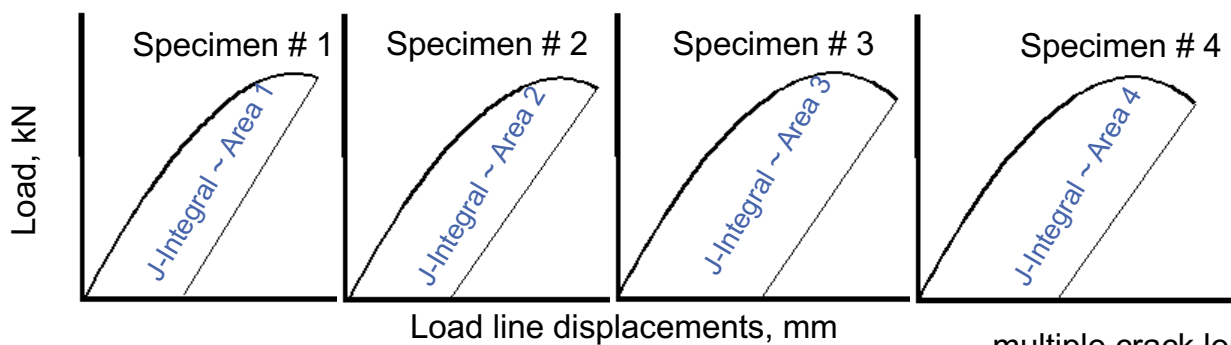


$$K_{IC} = Y \sigma_c \sqrt{\pi a} \quad a_c = \frac{1}{\pi} \left( \frac{K_{IC}}{Y \sigma} \right)^2$$

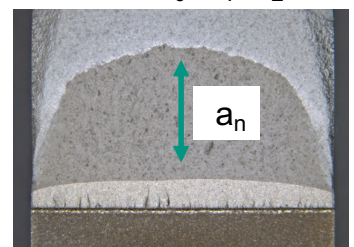
geometry factor  $Y$   
 stress at failure  $\sigma_c = P_Q / A$   
 crack length  $a$

# Fracture Toughness

Elastic plastic fracture mechanics [ J-concept], ASTM E1820 multi specimen method

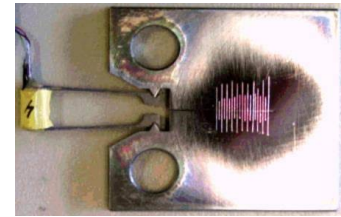
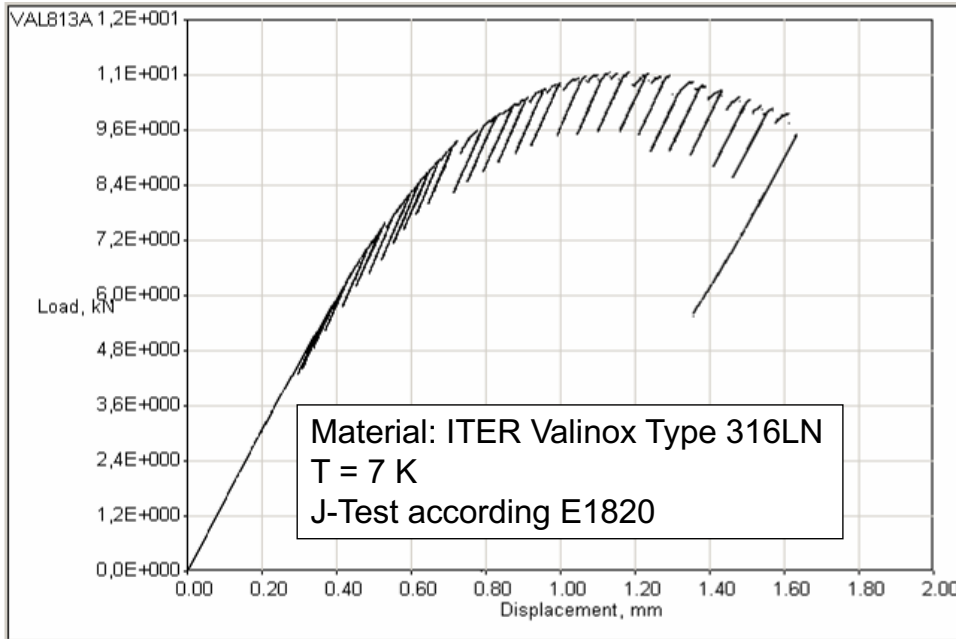


multiple crack lengths  
 $a_0, a_1, a_2, \dots$



# Fracture Toughness

Elastic plastic fracture mechanics [J-concept], ASTM E1820 **single** specimen method



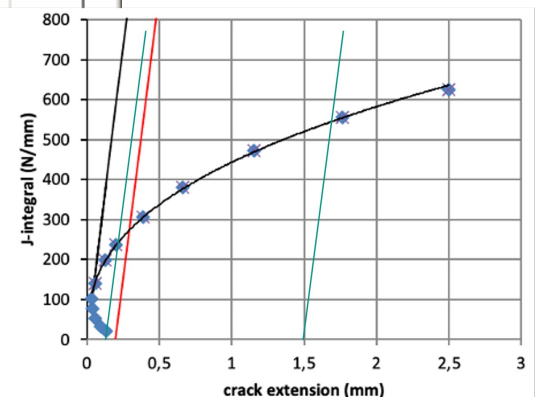
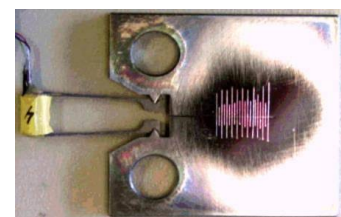
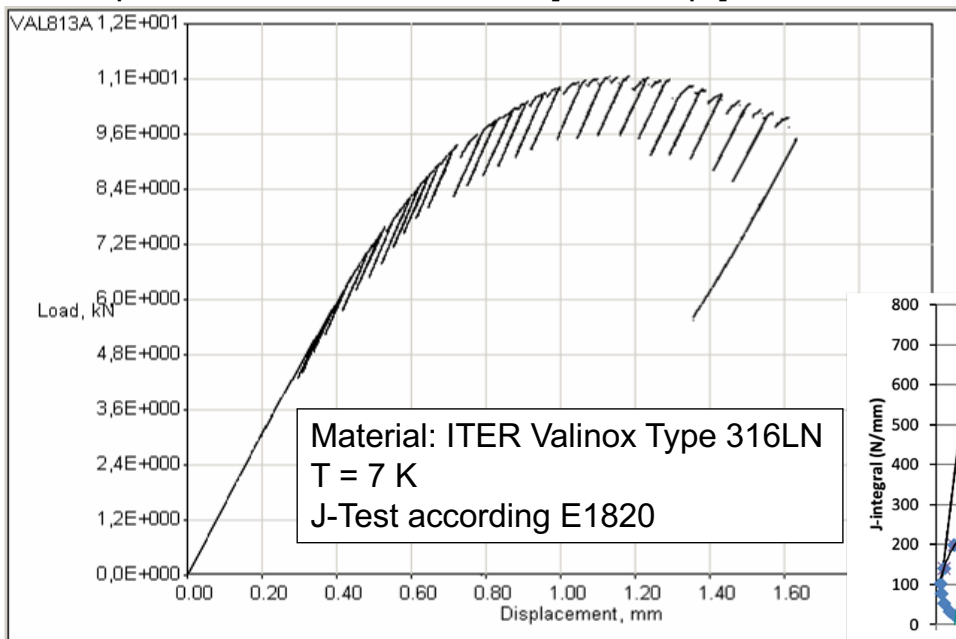
slope kN/mm  
of each unloading  
→ crack length  $a_0, a_1, a_2, \dots$

curve area  
at each unloading  
→ integral  $J_1, J_2, J_3, \dots$

Fernández-Pisón et al doi: 10.1016/j.engfracmech.2021.108042  
Weiss, Nyilas doi: 10.1111/j.1460-2695.2006.00963.x

# Fracture Toughness

Elastic plastic fracture mechanics [J-concept], ASTM E1820 **single** specimen method

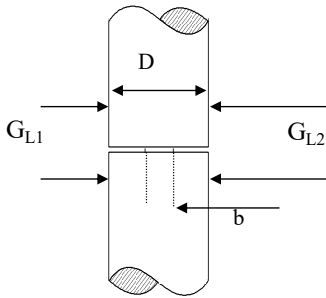


Fernández-Pisón et al doi: 10.1016/j.engfracmech.2021.108042  
Weiss, Nyilas doi: 10.1111/j.1460-2695.2006.00963.x

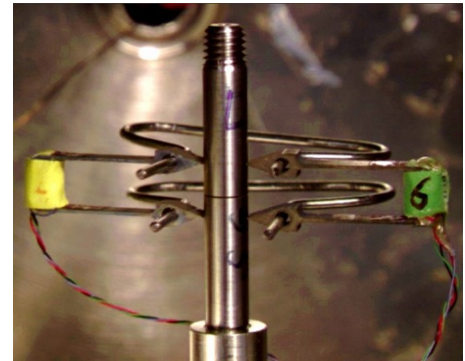
# Fracture Toughness: J Evaluation on Tensile Test (JETT)

Elastic plastic fracture toughness test method developed based on a fundamental solution on J Evaluation on Tensile Test

J. R. Rice et al., Some further results of J Integral analysis and estimates, in: "ASTM Special Technical Publications 536", (1973) p. 231

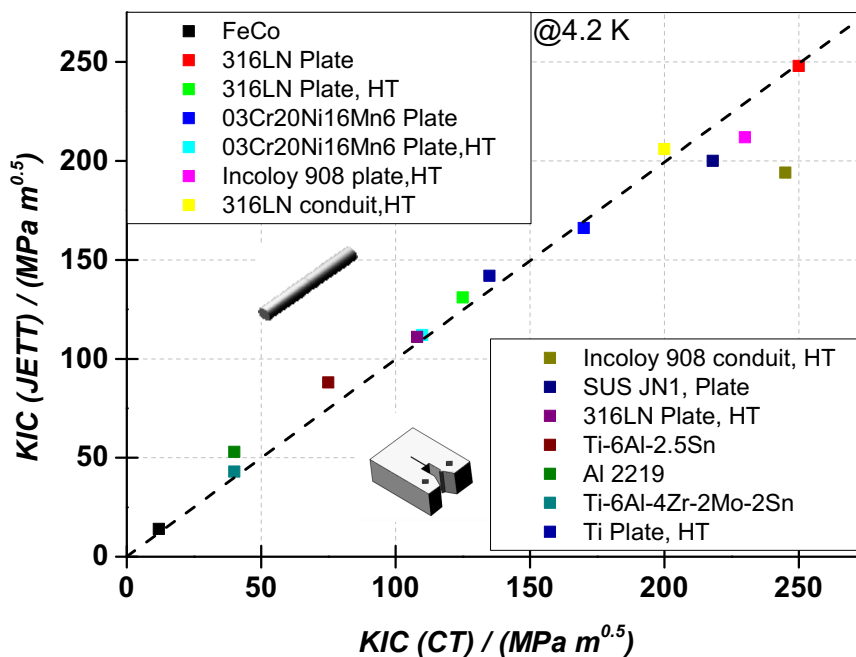


- D = round bar diameter
- b = net section diameter
- d<sub>ex</sub> = extensometer reading
- d<sub>nc</sub> = elastic displacement
- d<sub>c</sub> = d<sub>ex</sub> - d<sub>nc</sub>



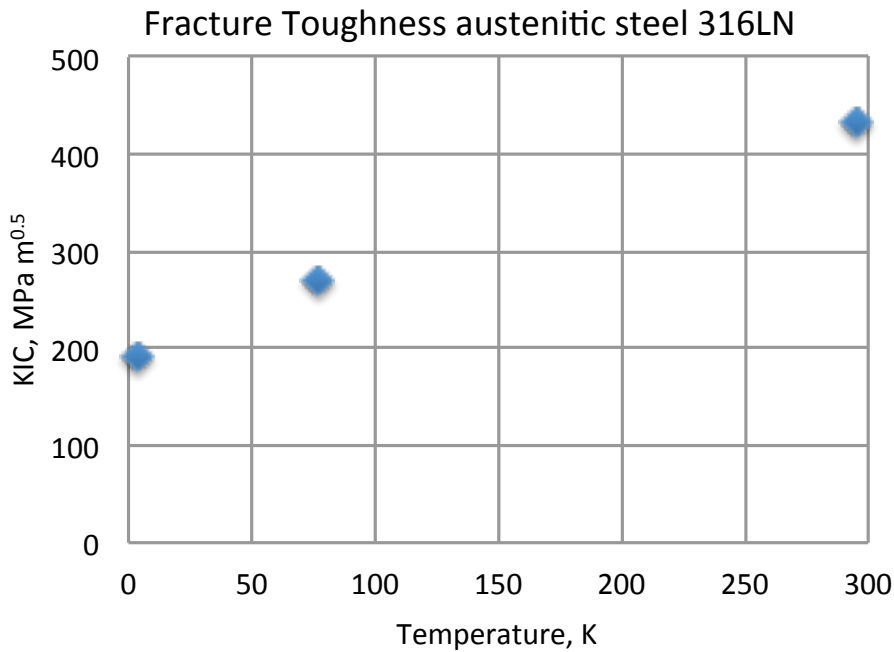
$$J = \frac{1}{2 \cdot \pi \cdot b^2} \left[ 3 \cdot \int_0^{d_c} P \cdot d(d_c) - P \cdot d_c \right]$$

# Fracture Toughness: J Evaluation on Tensile Test (JETT)



Correlation between JETT test results of various materials and valid ASTM tests



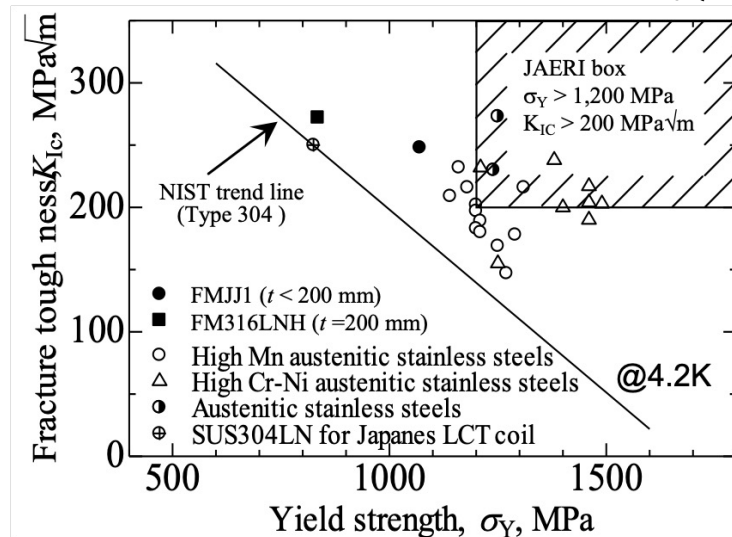
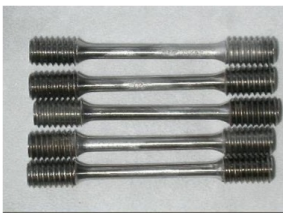


## Tensile & Fracture Toughness: High Strength Materials

■ Combining *quasi-static* mechanical performance of materials

■ Tensile test (strength)  
→  $\sigma_Y$  (MPa)

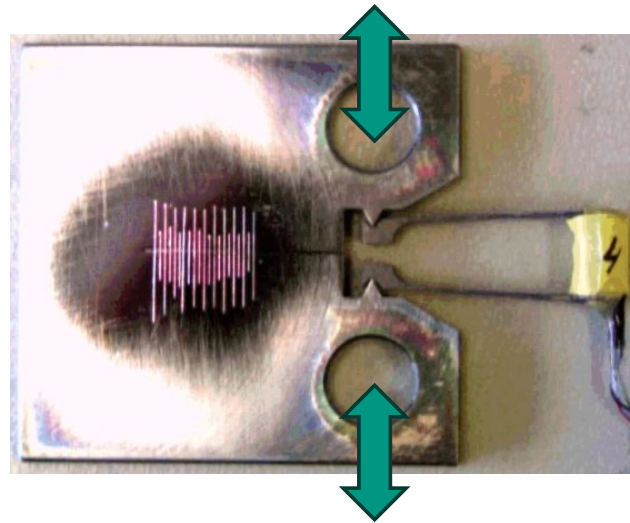
■ Fracture toughness (ductility)  
→  $K_{IC}$  (MPa $\sqrt{m}$ )



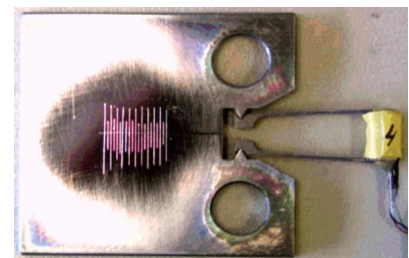
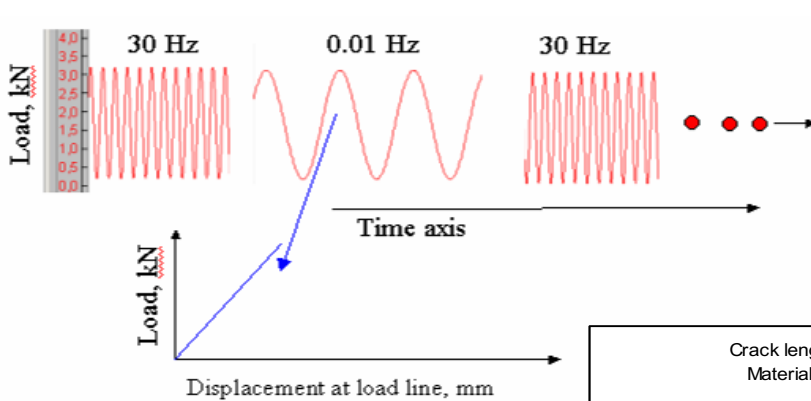
Nishimura, AIP Conference Proceedings 1574, 333 (2014)



# Fatigue Crack Growth Rate (FCGR)



# Fatigue Crack Growth Rate (FCGR)



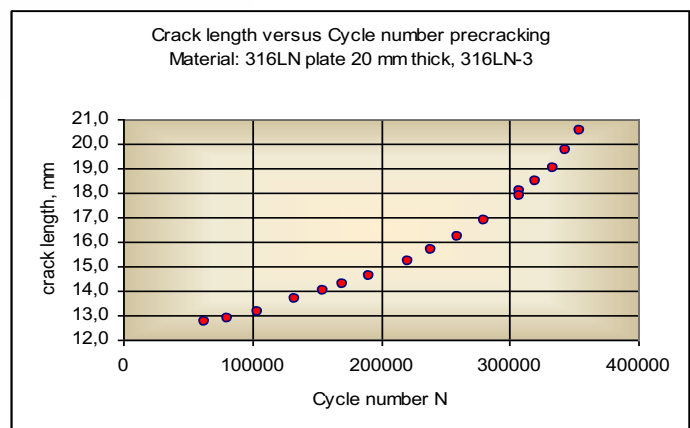
CT specimen, 43 x 45 x 4

Start cycling with  $\Delta\sigma = \text{const}$  and 30 Hz

$$\Delta K = Y\Delta\sigma \sqrt{\pi a(N)}$$

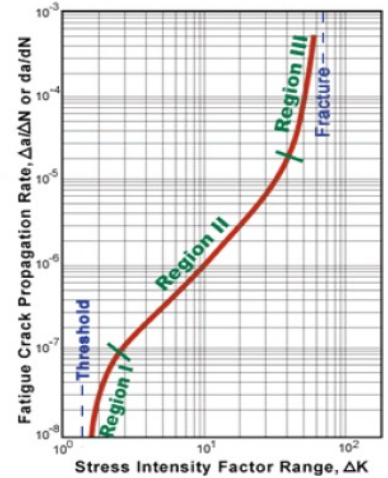
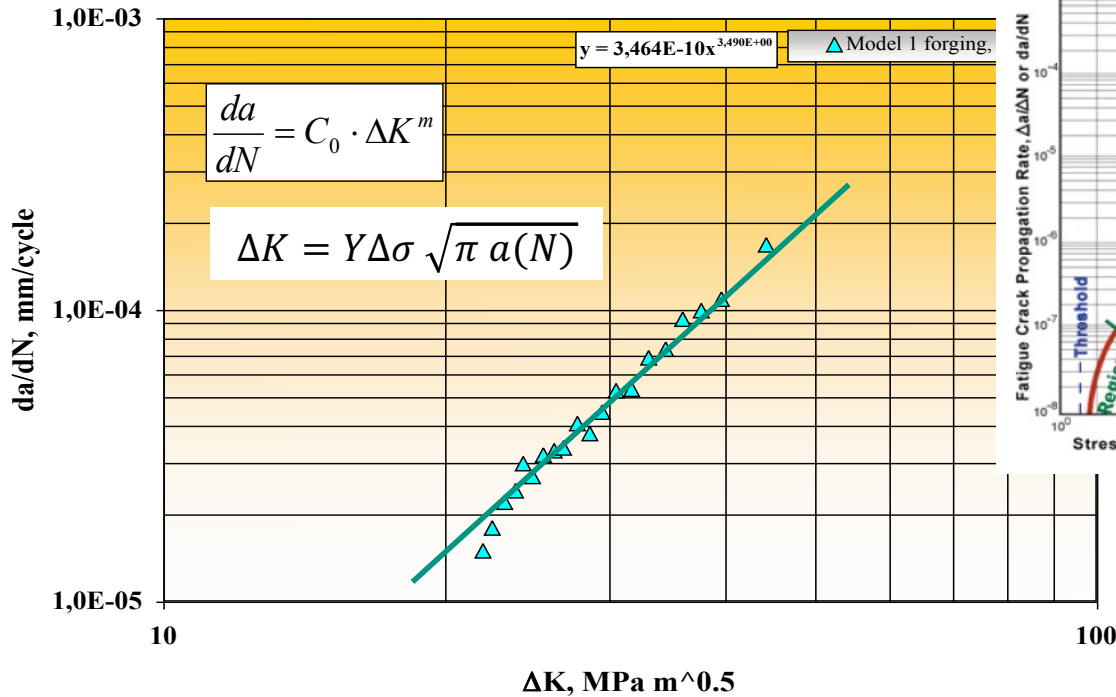
For each 1000 cycles  
acquisition of compliance

→ Function crack length versus  
cycles and  $da/dN$



# Fatigue Crack Growth Rate (FCGR)

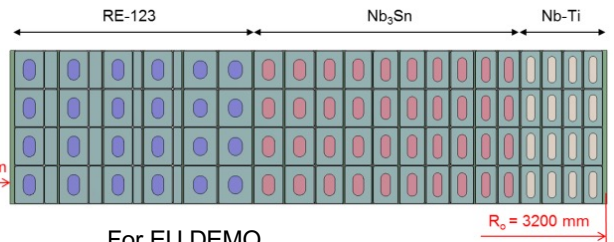
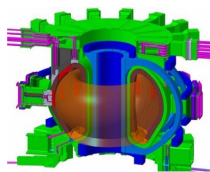
FCGR at 4K, R=0.1 (amplitude min/max ratio)



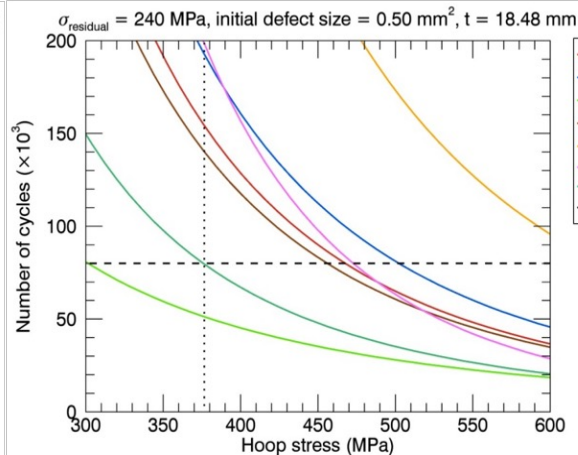
# Fatigue Crack Growth Rate (FCGR)

Example from one of the DEMO Central-Solenoid magnet designs

After how many cycles  $N$  the critical crack length  $a_c$  of jacket thickness  $t$  is reached?



SC+SS graded design



- JK2LB (Iter CS)
- EK1-JJ1 (ITER IL)
- SS 316LN (modified and aged)
- Incoloy 908
- EC1 (ITER OL, Cast)
- Inconel 718
- SS 316LN
- DEMO ( $N \times 2 \text{ peaks} \times \text{SF}$ )

safety factors:  
 2x cycles,  
 2x defect area,  
 1.5x fract.tough.

For EU DEMO  
 20,000 plasma cycles  $\times 2 \times 2 = 80,000$  cycles  
 the hoop stress needs to be reduced to  $\approx 375$  MPa in case of SS 316LN.

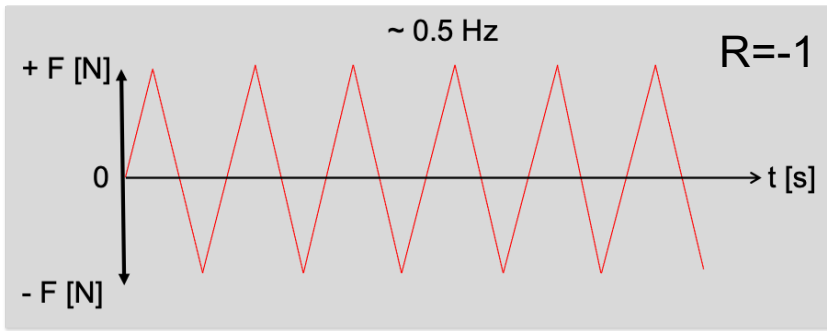
For higher loads or higher cycle numbers, other materials have to be used or even developed.

For a future fusion power plant with planned  
 156,000 cycles  $\times 2 \times 2 = 624,000$  cycles  
 even with EC1 material the hoop stress should be  $< 325$  MPa.

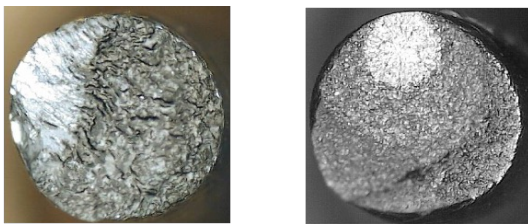
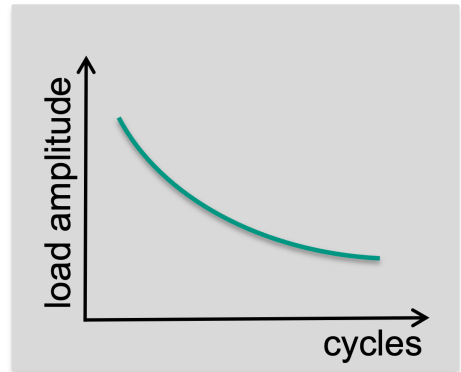
→ Fatigue is the main driver that limits the design



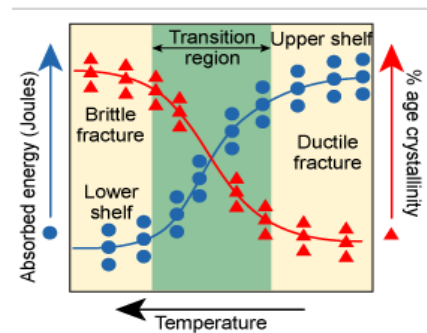
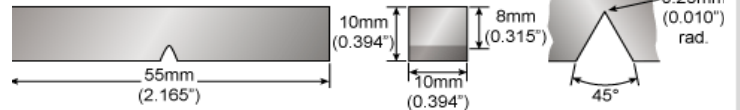
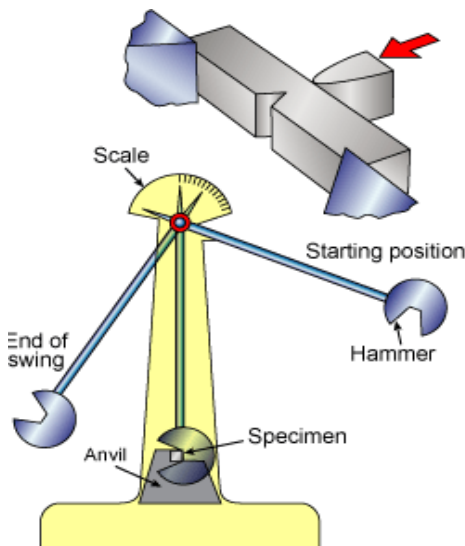
# Low Cycle fatigue (LCF)



Start with +/-F and ~0.5 Hz until failure of specimen  
change load level for each specimen  
→ lifetime curve (Wöhler curve)



# Impact Test



Fast and easy test to examine ductile/brittle transition by absorbed impact energy

Drawback: results at cryogenic temperature <77K  
only comparable under same test condition/geometry,  
but no basic material property!



**Thanks for listening!**



Dr. Klaus-Peter Weiss  
*klaus.weiss@kit.edu*



Cryogenic Materialtests Karlsruhe  
**CryoMaK**

## References

- Standards: DIN / EN / ISO / JIS / ASTM
- Materials at Low Temperatures - ASM International - ISBN 978-0871701466
- Materials for Low Temperature Use – Oxford University Press – ISBN 978-0198591634
- Nonmetallic Materials and Composites at Low Temperatures - Plenum Publishing Corporation - ISBN 978-0306400773
- Experimental Techniques for Low Temperature Measurements: Cryostat Design, Materials, and Critical-Current Testing - Oxford University Press - ISBN 978-0198570547
- Schaum's Outline of Strength of Materials - Mcgraw Hill Book Co - ISBN 978-0070466173
- Atlas of Stress-Strain Curves - ASM International - ISBN 978-0871707390
- Mechanical Properties of Materials at Low Temperatures - Plenum Pub Corp - ISBN 978-0306305146
- Deformation and Fracture Mechanics of Engineering Materials - John Wiley & Sons - ISBN 978-0471012146
- History of Strength of Materials - Dover Pubn Inc - ISBN 978-0486611877
- <http://cryogenics.nist.gov/MPropsMAY/material%20properties.htm>
- <http://www.slac.stanford.edu/cgi-wrap/getdoc/slac-tn-03-023.pdf>
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