# MATERIALS CHARACTERIZATION AT CRYOGENIC TEMPERATURES

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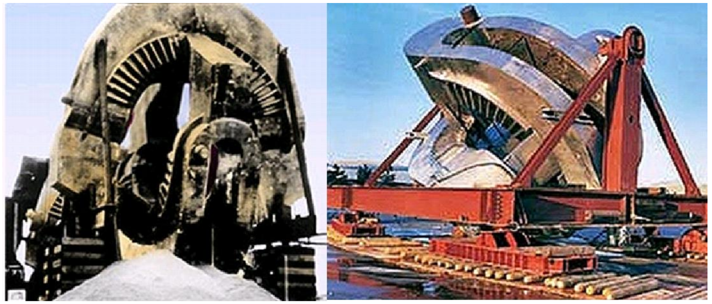
- Overview about materials performance for structural design
- Tensile measurements
- Fracture toughness measurements
- History of FCGR and its concept
- FCGR evaluation between 80s and 90s, and today
- Concept of uncertainty and its basics (true value ?)
- Application of uncertainty in case of FCGR
- Paris law region as model equation
- Evaluation of the best estimate and its uncertainty term

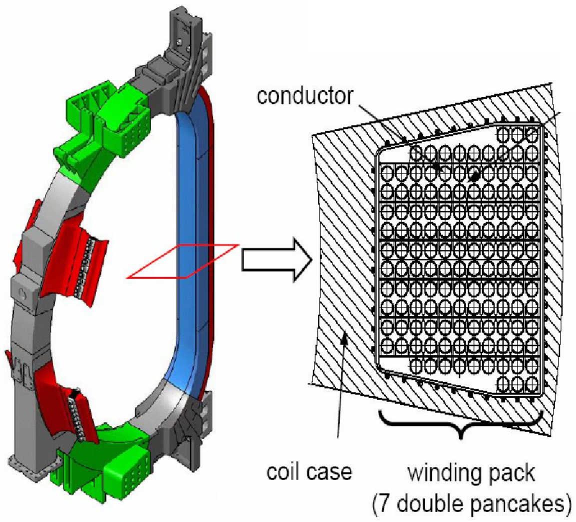
# Structural steels: A common feature is the heavy wall thickness

BEBC, Cern (1975-1985)
3.7 m Ø x 4 m (316LN)

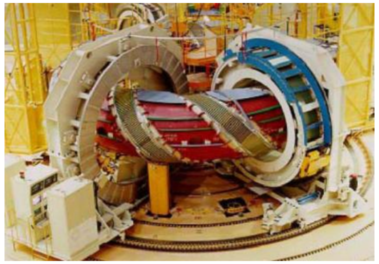


MFTF-B, Livermore (304N): Built in 80s, 1986 shut down without any service

Tore Supra, Cadarach
(1988 – 2003; 6 min Plasma)
1.8 K service (316LN)

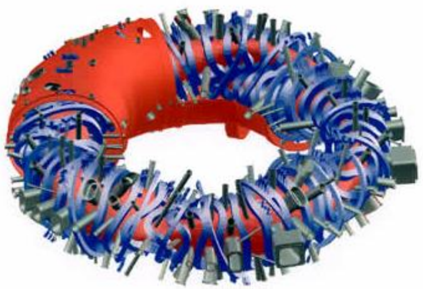



ITER, Cadarach TF coil design
2008 (+~ 10 y)



LHD, NIFS, Japan SUS 316L
1998 March 31: 1st Plasma
Total cold mass 850 t

LCT- EU, Karlsruhe & TFMC
1.8 K service (316LN) (1984-2005)



W7 – X, Greifswald: 50 non-planar/20 planar coils ~ 2013/2014

- **A common feature of all these magnets is the similarity to pressure vessels likened to gas pressure, magnetic field lines try to expand the structure with equivalent pressures up to 1000 bar at temperatures near 4 K**

- **Beside the tension also bending stresses are inevitable due to manufacturing and fault conditions**

- **No code or standard guidance exists for the design of magnet structures for service at 4 K**

- **Designers usually follow parts of various codes like Secs. III or VIII of ASME Boiler and Pressure Vessel Code**

- **However, blind obedience to existing codes may result either excessive heavy or dangerous fracture-prone structures**

## An example from past times about the design rules: MFTF B

### Historical: Recommended design factors for Mirror magnets

**<u>Base metal</u>**

**Design stress <2/3 yield (primary tension and combined)**

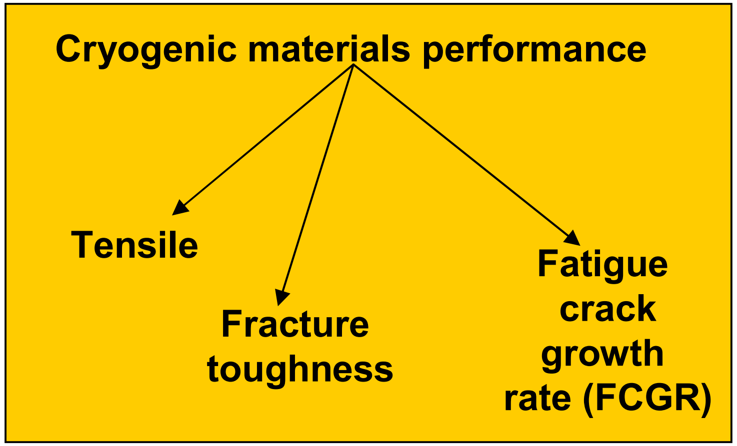
**Design stress < 90% yield (primary bending)**

**Design stress < 1/3 UTS**

**Design stress <** $\frac{1}{2}\cdot\frac{KIC}{\sqrt{\pi\cdot a}}$

**Design stress cycles < 4 lifetimes**

**The lowest stress values should be used**

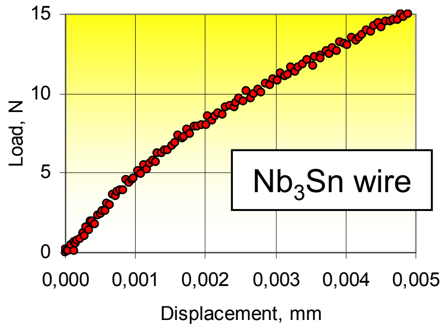


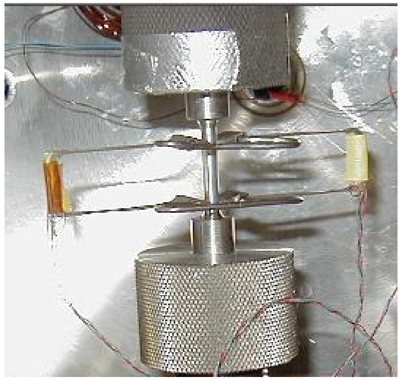

**At this stage according to these past tests the toughness was the major problem with the consumable welds**
**Welds were manufactured by SMA ( practically manual)**

**Yield strengths of 304N base metal values were 840 MPa and $K_{IC}$ values were 60 – 120 MPa√m**

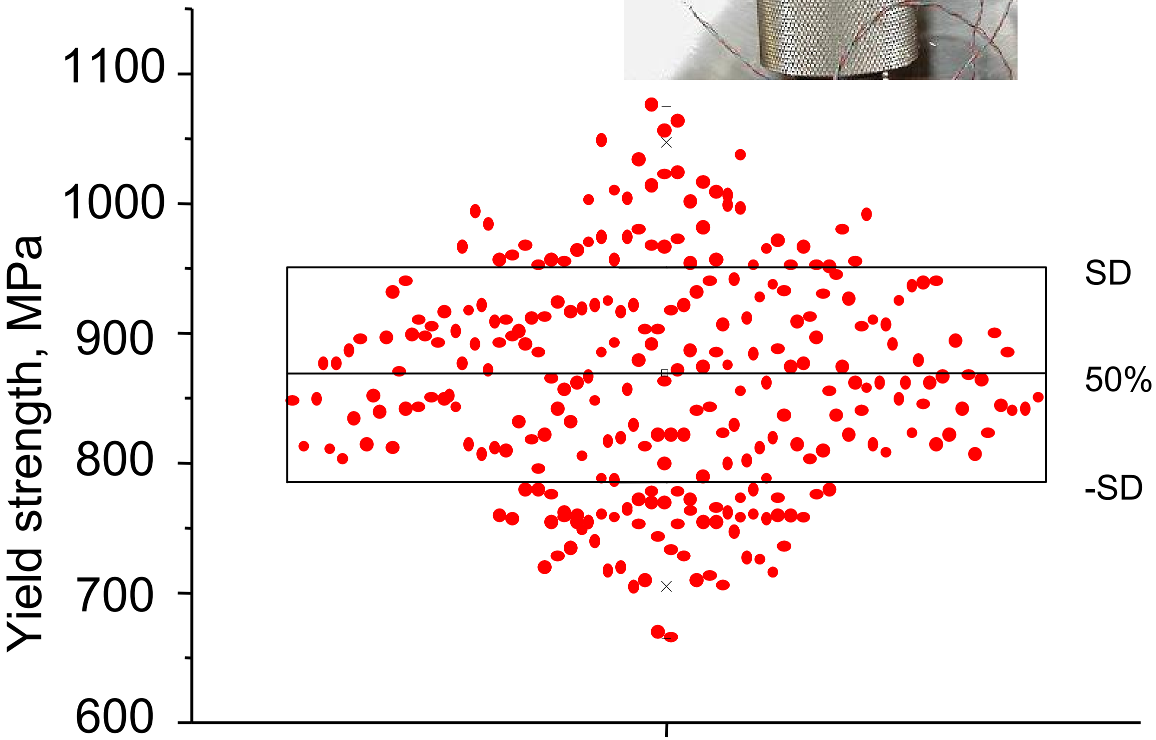
**$\delta$ - Ferrite versus hot cracking was one of the main culprit**

# Yield strength properties of cast nitrogen strengthened materials: W7 - X

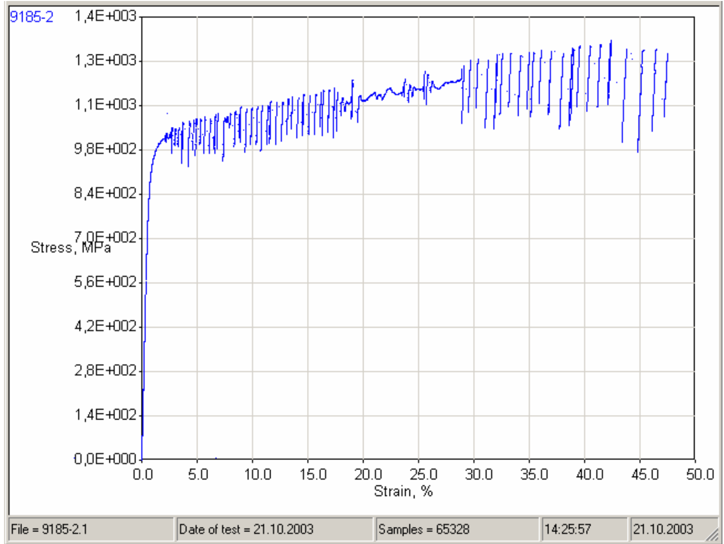


**Yield strength of cast material measurements of 259 specimens with respect to different heats**

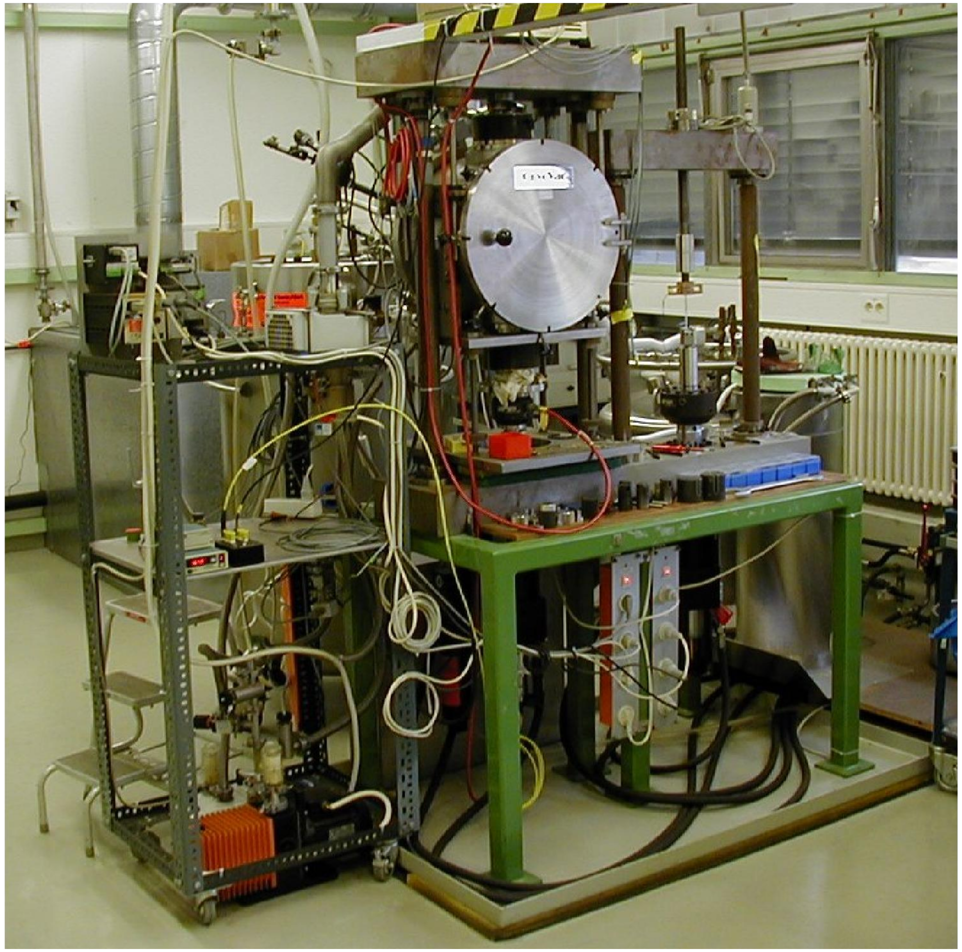



**Collected data of 295 specimens**

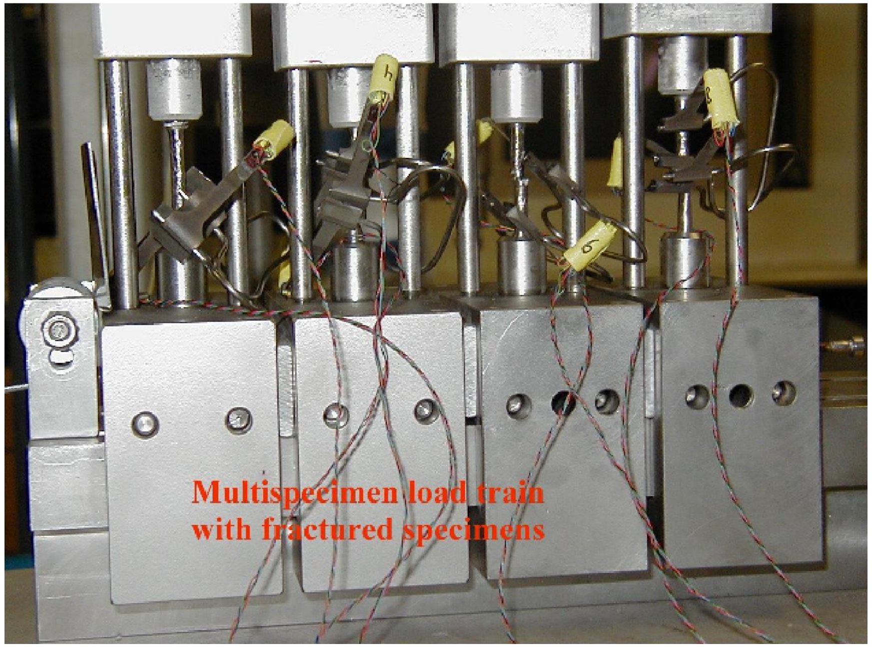



## Used cryogenic test facilities: +/- 25 kN MTS Type & 630 kN Schenck Type

The facilities are located at Karlsruhe Institute of Technology (KIT), former Forschungszentrum Karlsruhe. In addition all following measurements are done at Forschungszentrum Karlsruhe



Variable temperature (295 K -7 K)




LHe, LN2, or RT

## The Struggle for Recognition of Engineering Fracture Mechanics

The post WW II work led to the development of modern fracture mechanics with a modification of the Griffith idea from WWI in which the resistance to crack extension was assumed to be plastic deformation at the leading edge of the crack rather than solid state surface energy (Irwin 1948, Orowan 1949). Linear elasticity theory predicts that the stresses become infinite as a crack tip is approached. In real materials, however, the stresses are limited by the flow stress, and a plastic zone develops at the end of the crack.

**LEFM**

ASTM E399-83

**Limitations are the size of the valid specimens For a structural material with**

$K_Q = 200 \quad MPa\sqrt{m}$

$\sigma_y = 1000 \quad MPa$

$$B \geq 2.5 \cdot \left(\frac{K_Q}{\sigma_y}\right)^2$$

$B = 100 \quad mm$

**EPFM**

E813-81

E813-88

JIS Z 2284 – 98

Small size specimens

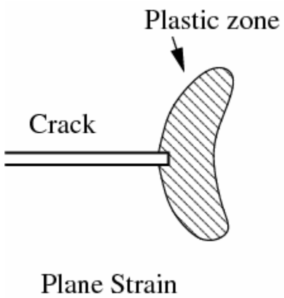
$J_Q = 200 \quad \frac{N}{mm^2}$

$\sigma_y = 1000 \quad MPa$

$$B \geq 25 \cdot \frac{J_Q}{\sigma_y}$$

$B = 5 \quad mm$

**ASTM E1820-99a**


**Specific aspects on crack advance during J-test method for Structural materials at cryogenic temperatures**

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*Received in final form 28 September 2005*
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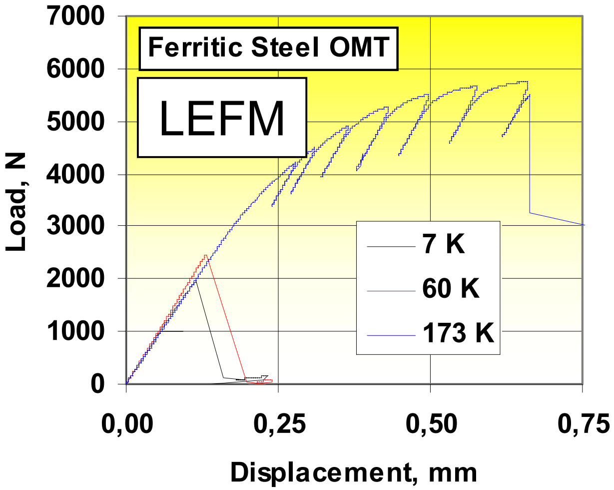
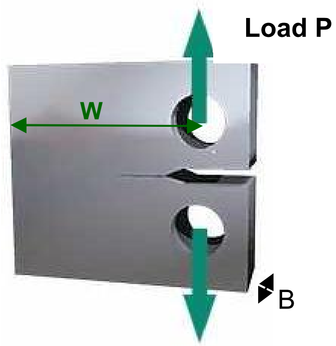
# J-Integral: Small scale specimens, its energy absorption, & fracture toughness



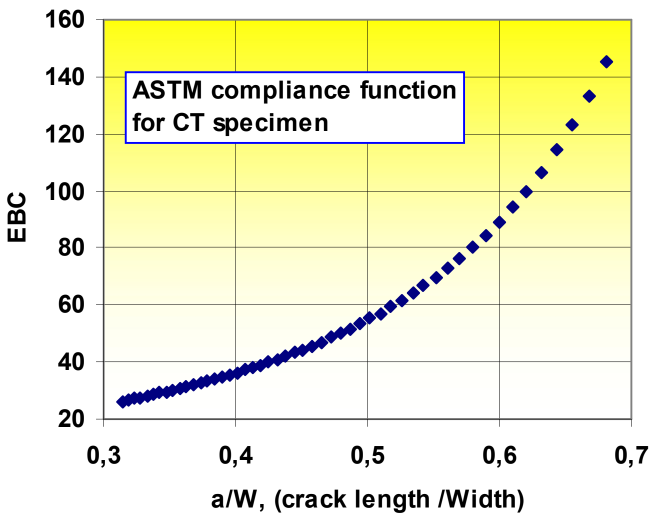



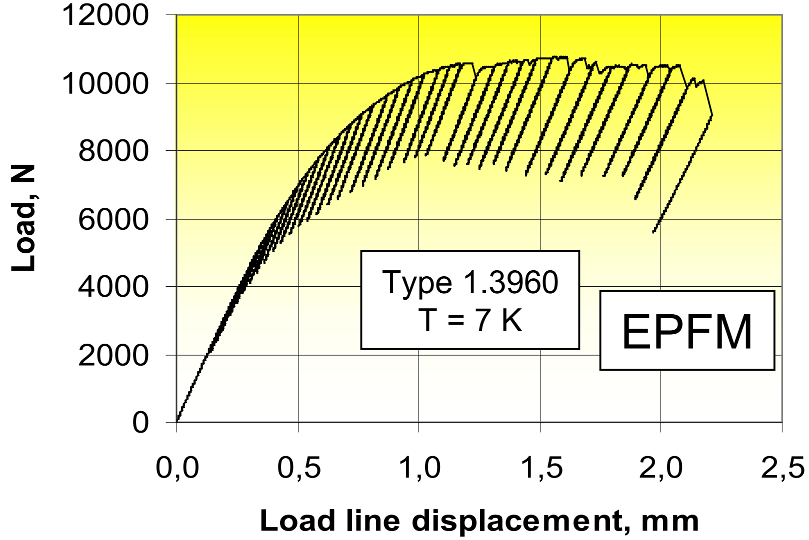

Crack advance (unloading compliance method)

Decrease of stiffness (or increase of compliance)

$$J_{El} = \frac{K^2 \cdot (1-\upsilon^2)}{E} \qquad J_{Pl} = \frac{2 \cdot Area}{B \cdot b}$$

$$J = J_{El} + J_{Pl}$$

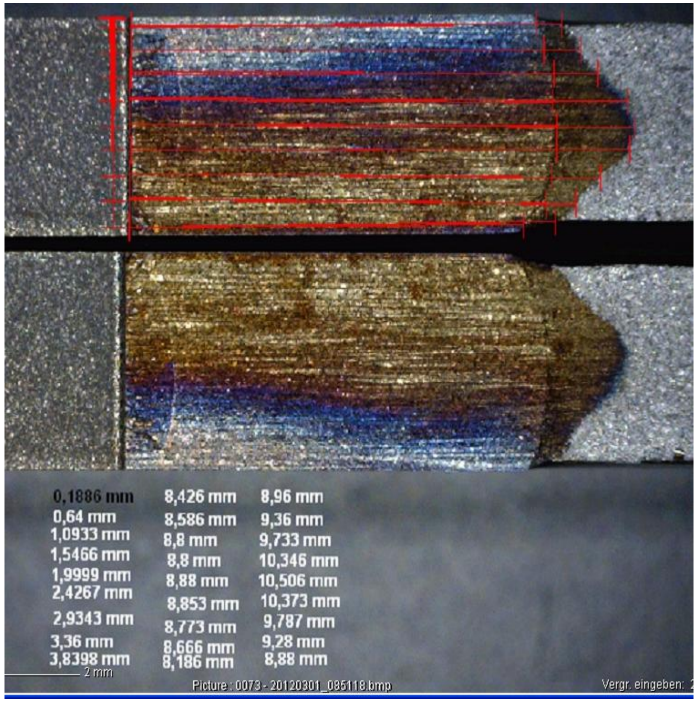


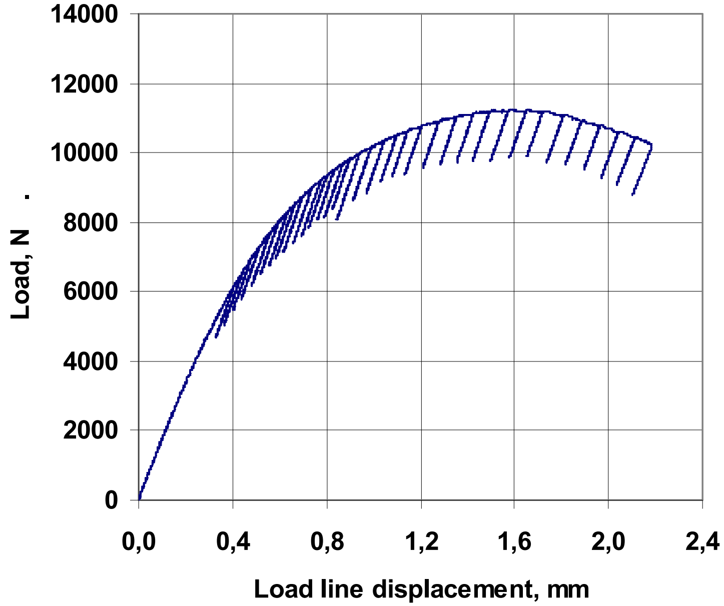



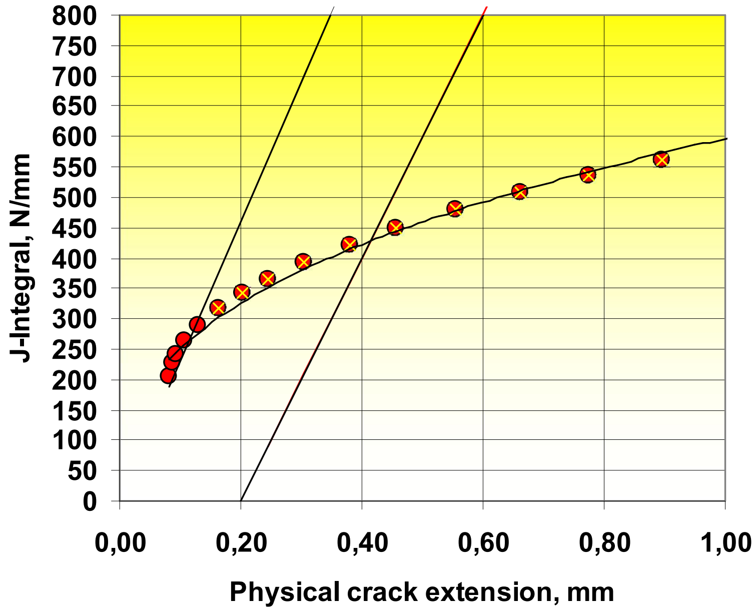

Stress intensity of a compact tension specimen

$$\Delta K = \frac{\Delta P}{B \cdot \sqrt{W}} \cdot \frac{\left(2+\frac{a}{W}\right)}{\left(1-\frac{a}{W}\right)^{1.5}} \cdot \left(0{,}886 + 4{,}64 \cdot \frac{a}{W} - 13{,}32 \cdot \left(\frac{a}{W}\right)^2 + 14{,}72 \cdot \left(\frac{a}{W}\right)^3 - 5{,}6 \cdot \left(\frac{a}{W}\right)^4\right)$$

# J-test of Type 316LN rolled plate material at 7 K

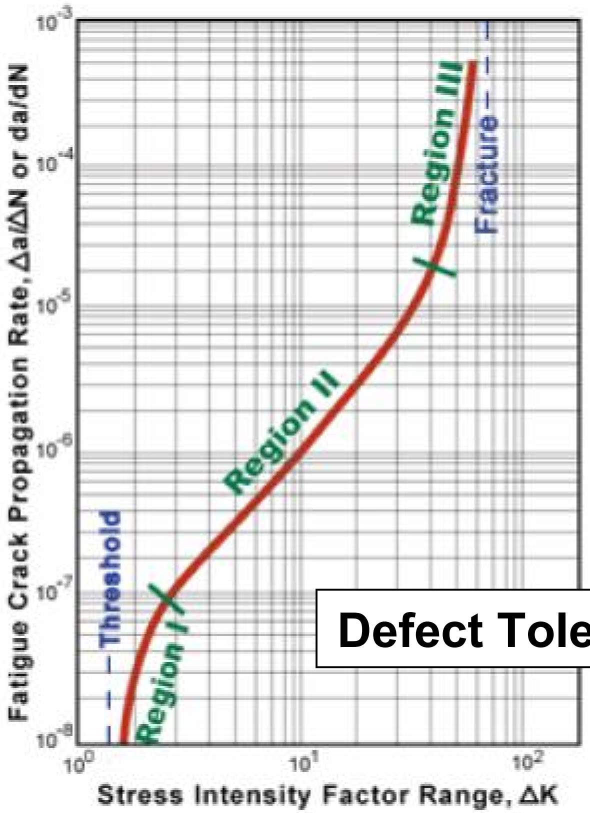


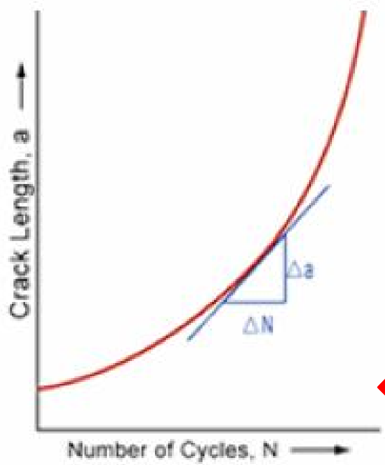



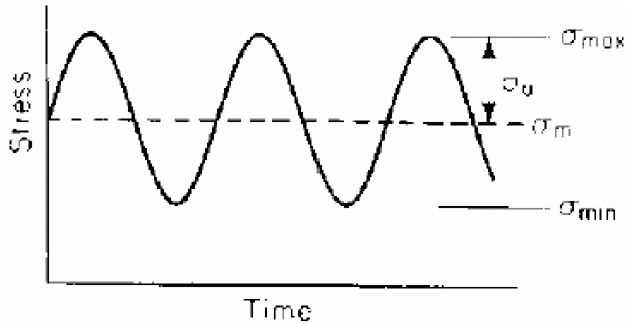

J-test with rolled plate material Specimen: Rol- L-1
Date: February 29, 2012; T = 7 K ;No side grooves
Specimen details:
B =4, W = 36 mm , $a_{notch}$ = 12 mm, $a_f$ = 20,708 mm,
$a_p$ = 21,788 mm; $J_q$ = 425 N/mm; Estimated $K_{IC}$ = 295 MPa√m

# Cyclic loading of components and load bearing members (FATIGUE)



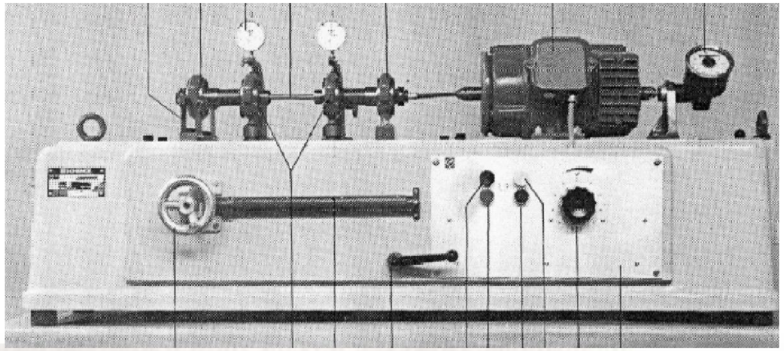



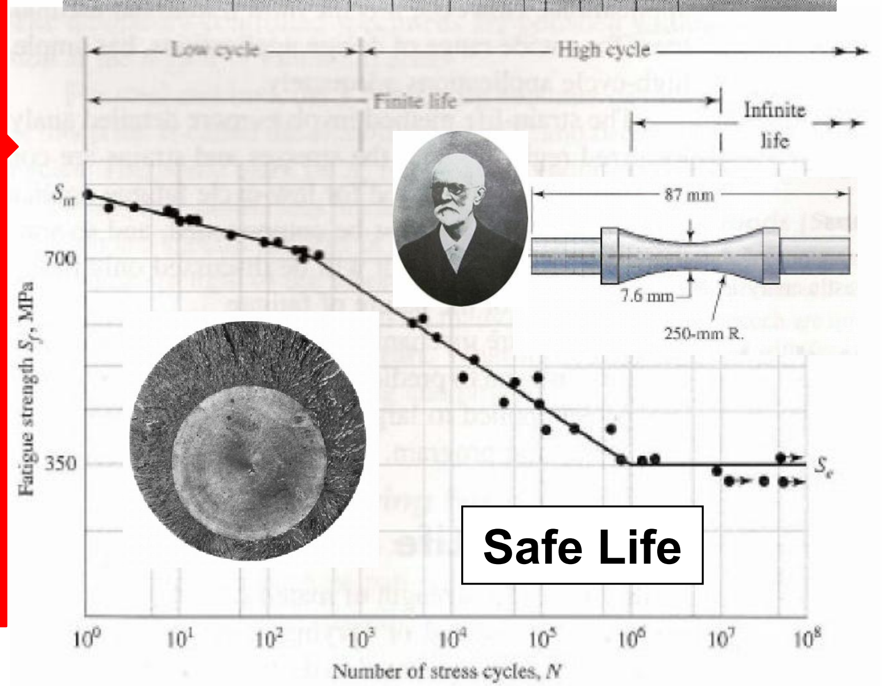

**Defect Tolerance**




*Load Range*

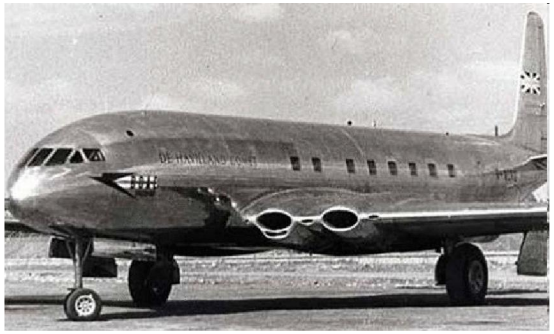
$$R = \frac{\sigma_{\min}}{\sigma_{\max}} \approx \frac{K_{\min}}{K_{\max}}$$






**Safe Life**

# Cyclic loading and airplane problems around 50s; Comet Jetliner



**Comet 1 prototype first flew on 27 July 1949.**

Design flaws, including dangerous stresses at the corners of the square windows and installation methodology, were ultimately identified. As a result, the Comet was extensively redesigned with oval windows, structural reinforcement and other changes.

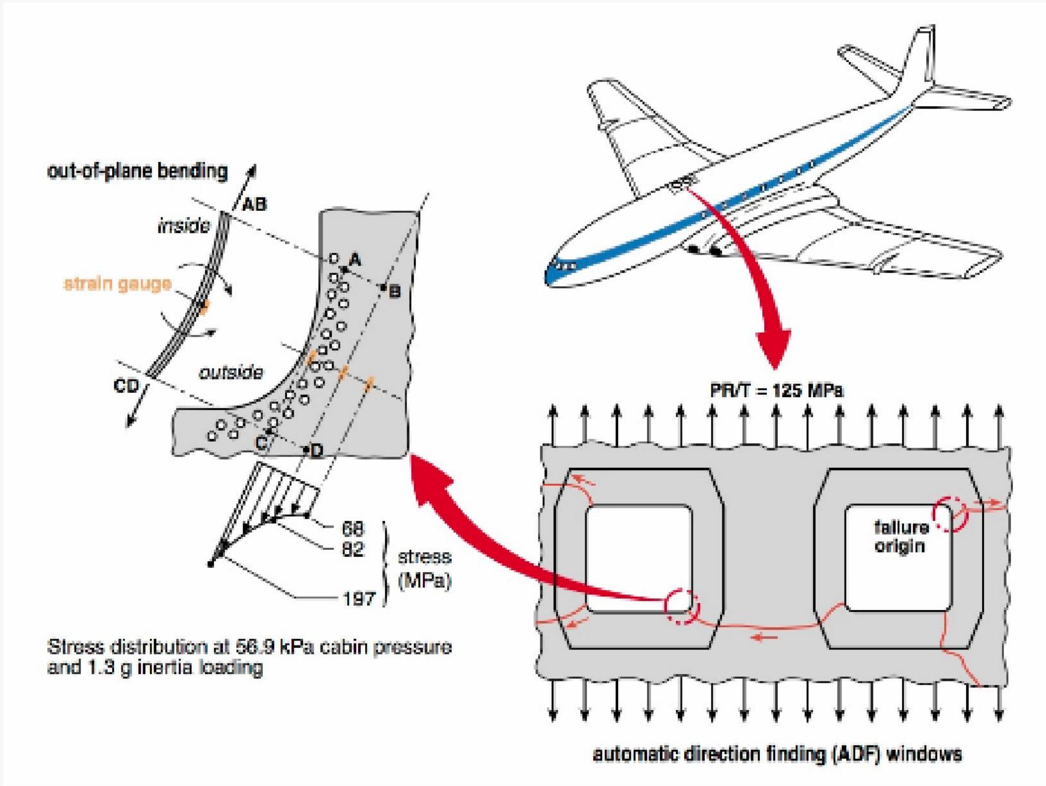
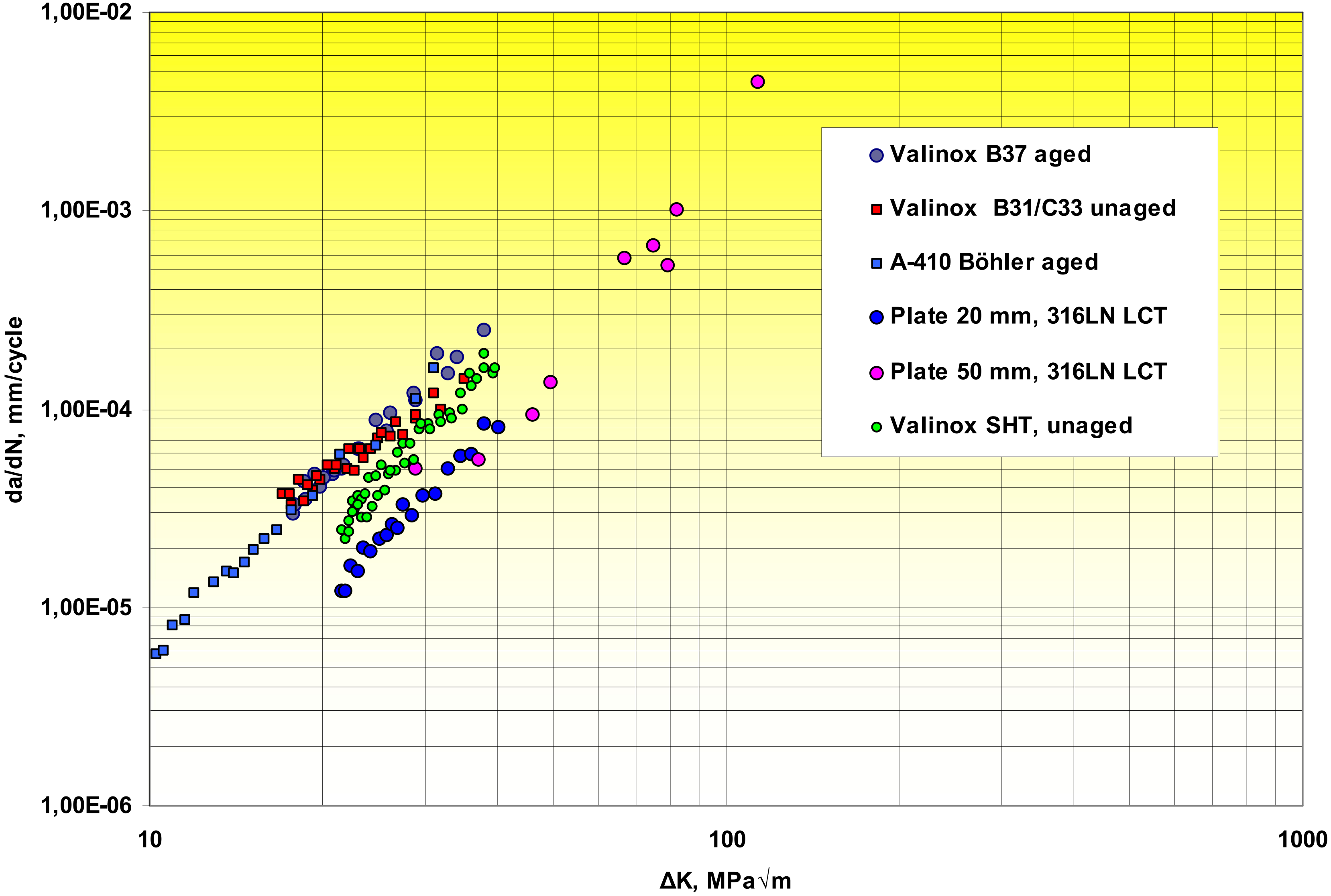


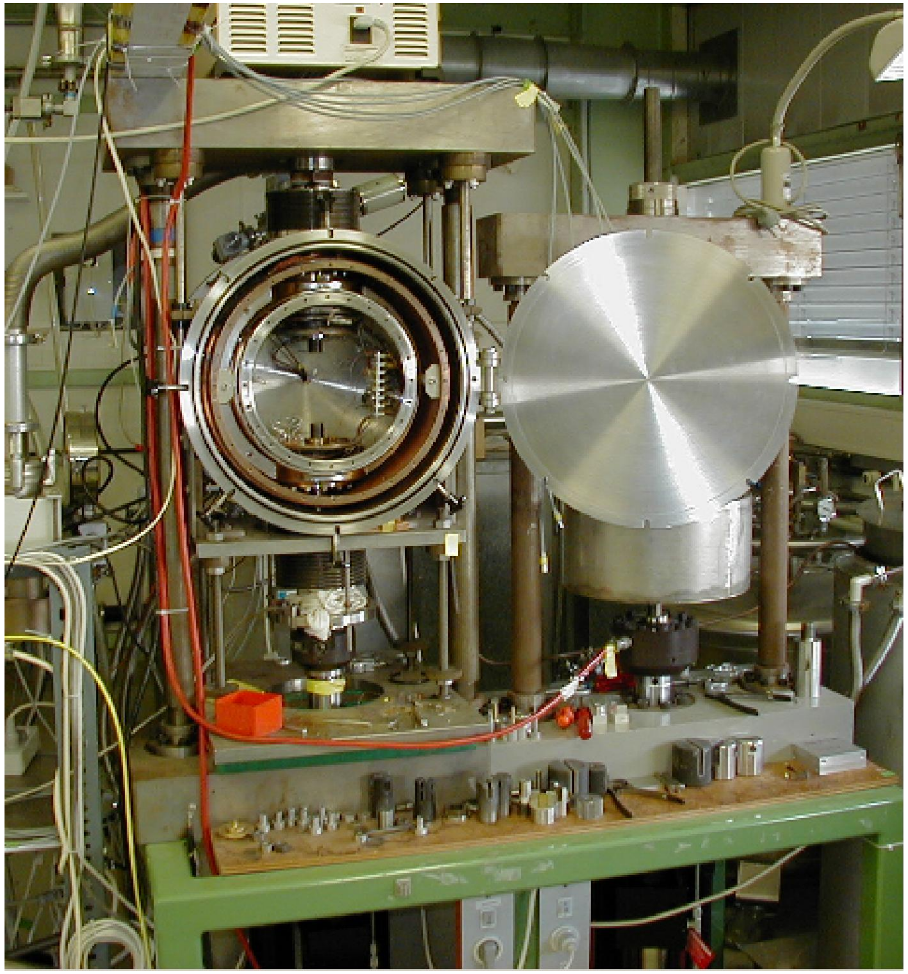

Fig. 2. Failure origin of Comet G-ALYU around square windows (1, 2).

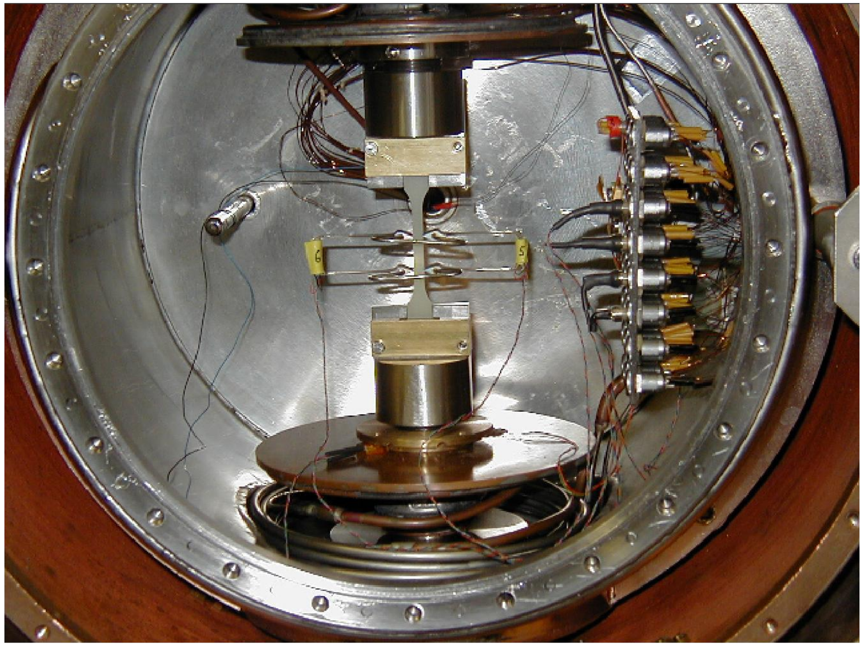
**1954:** 'Metal fatigue' caused Comet Crashes The public inquiry into the Comet airliner disasters has heard that metal fatigue was the most likely cause of two recent crashes.

# Collection of FCGR data for 316LN at early 90s

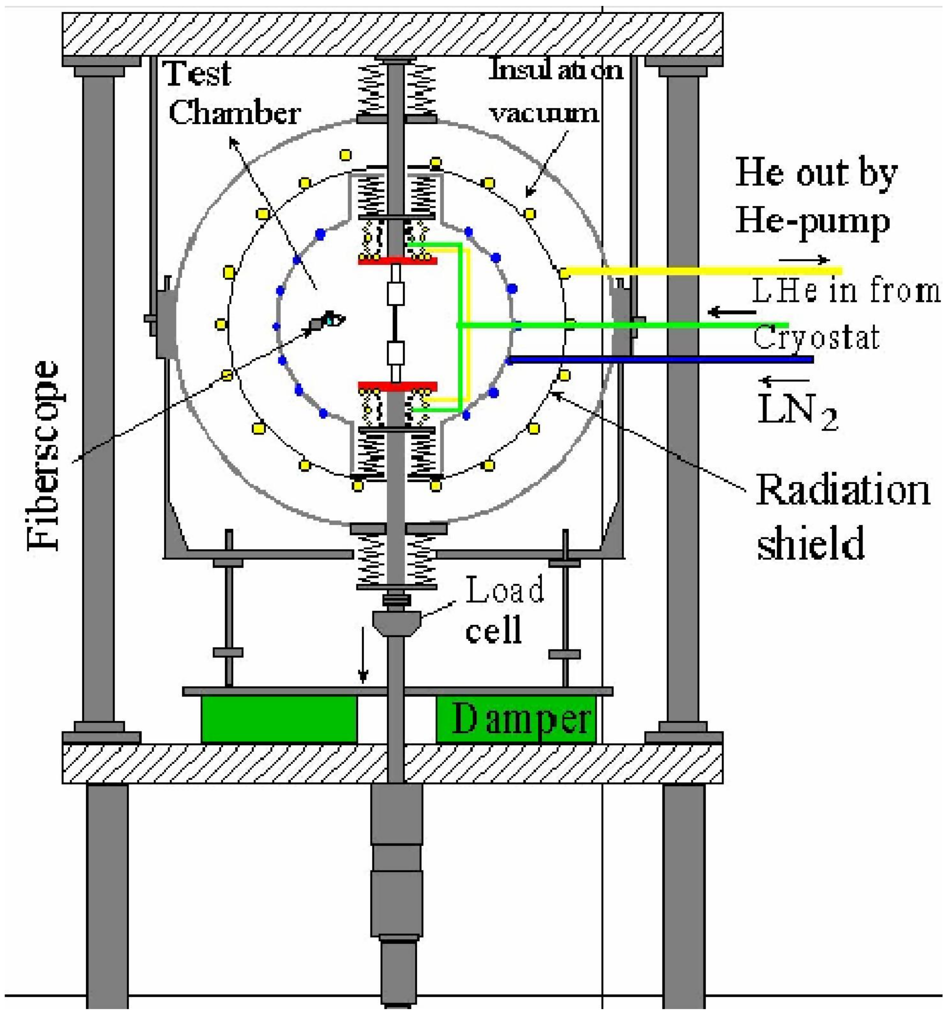


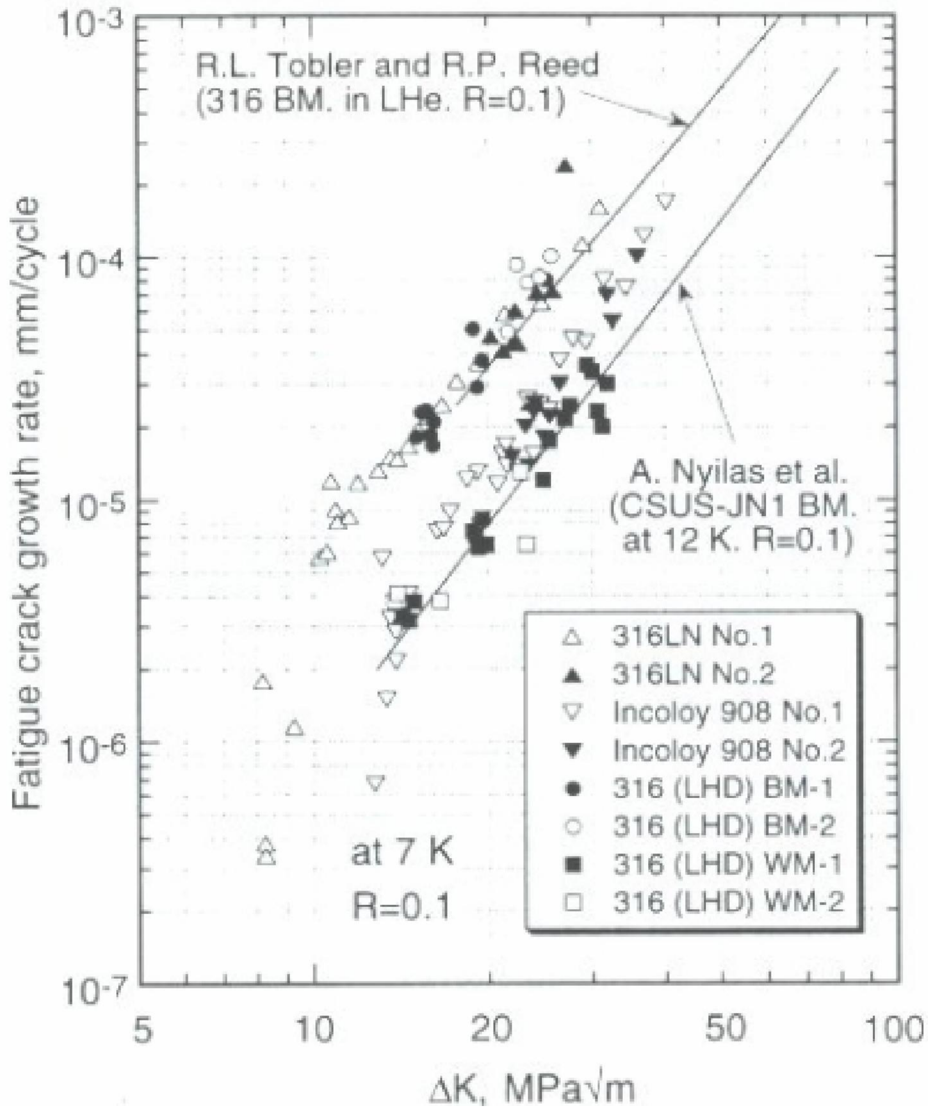
## Existing variable temperature cryogenic test facility working since 1987





## Historical measurements : Microscopic crack detection at cryogenics



# Historical measurements : Traveling microscope

FATIGUE CRACK GROWTH RATE OF SUS 316
AND WELD JOINT WITH NATURAL CRACK AT 7 K

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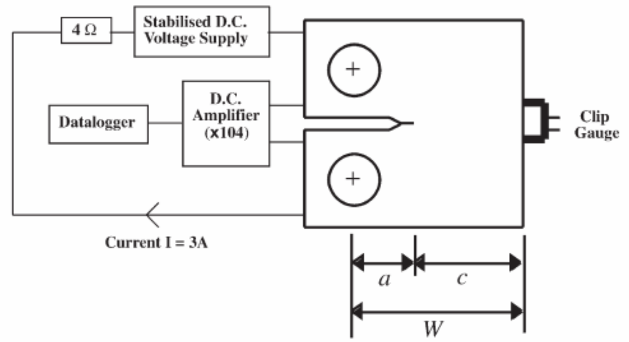
Toki, Gifu 509-52 Japan
[2]Forschungszentrum Karlsruhe, ITP
D-76021 Karlsruhe, Germany

urnal of Fatigue 23 (2001) 375–382



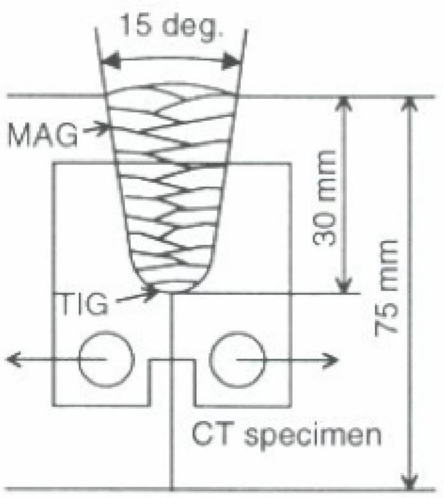




Figure 1. Configuration of welded joint.

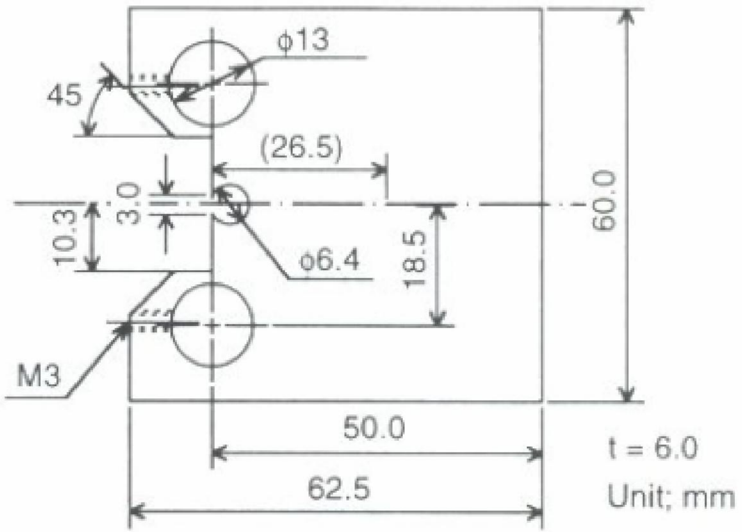



Figure 2. Compact tension specimen.

## Life analysis using the Paris law

$Co := 2.84 \cdot 10^{-13}$ $m := 3.58$ $\Delta K := 114$ $Kic := 200$ $\pi = 3.142$

$Y := 1$ $S := 2$ $\Delta\sigma := 400$ $R := 0.1$ $ao := 0.002$

$$ac := \frac{1}{\pi} \cdot \left( \frac{Kic}{S \cdot Y \cdot \Delta\sigma} \right)^2 \qquad ac = 0.02 \qquad Kc := 195$$

$$\frac{da}{dN} = C_0 \cdot \Delta K^m$$

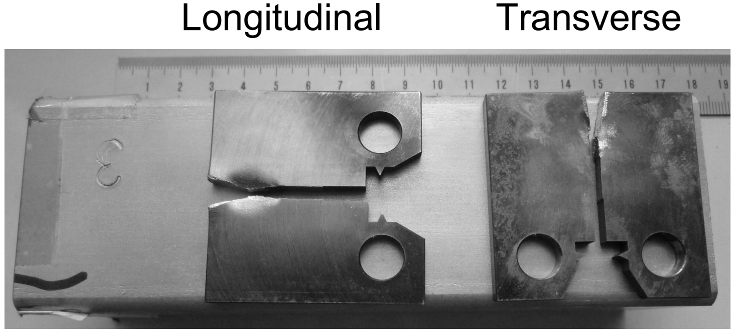
**Paris law**

$$Nf := \left( \frac{1}{Co \cdot Y^m \cdot \Delta\sigma^m} \right) \cdot \int_{ao}^{ac} \frac{1}{\left(\sqrt{\pi \cdot a}\right)^m} \, da \qquad Nf = 31533$$

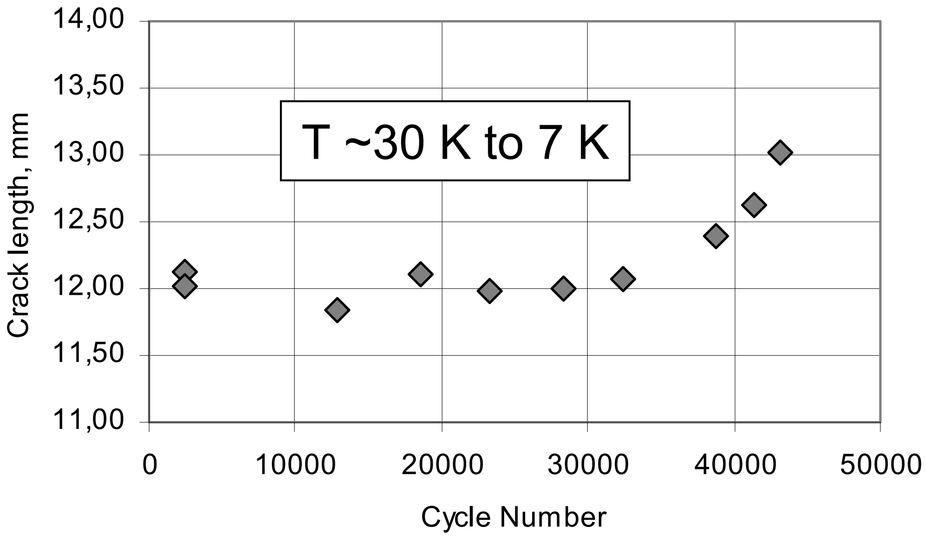
**Forman considers the load range R. Forman could determine that da/dN goes to infinity if $K_{max}$ the critical K-value nears**

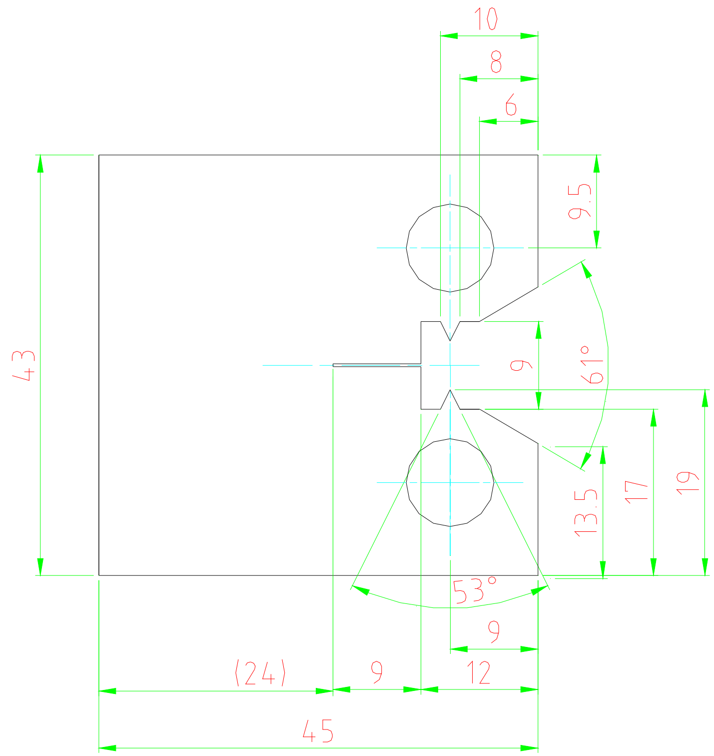
$$Nf := \int_{ao}^{ac} \frac{1 \cdot (1 - R) \cdot Kc - \left(\Delta\sigma \cdot Y \cdot S \cdot \sqrt{\pi \cdot a}\right)}{\left[ Co \cdot \left(\Delta\sigma \cdot Y \cdot S \cdot \sqrt{\pi \cdot a}\right)^m \right]} \, da \qquad Nf = 198130$$

## Sample machining & specimen & test details today




Crack length versus cycle number at 7 K for
46_(3)_CO+ST_2.5%+AG_FCGR+J1C_LD_1 specimen



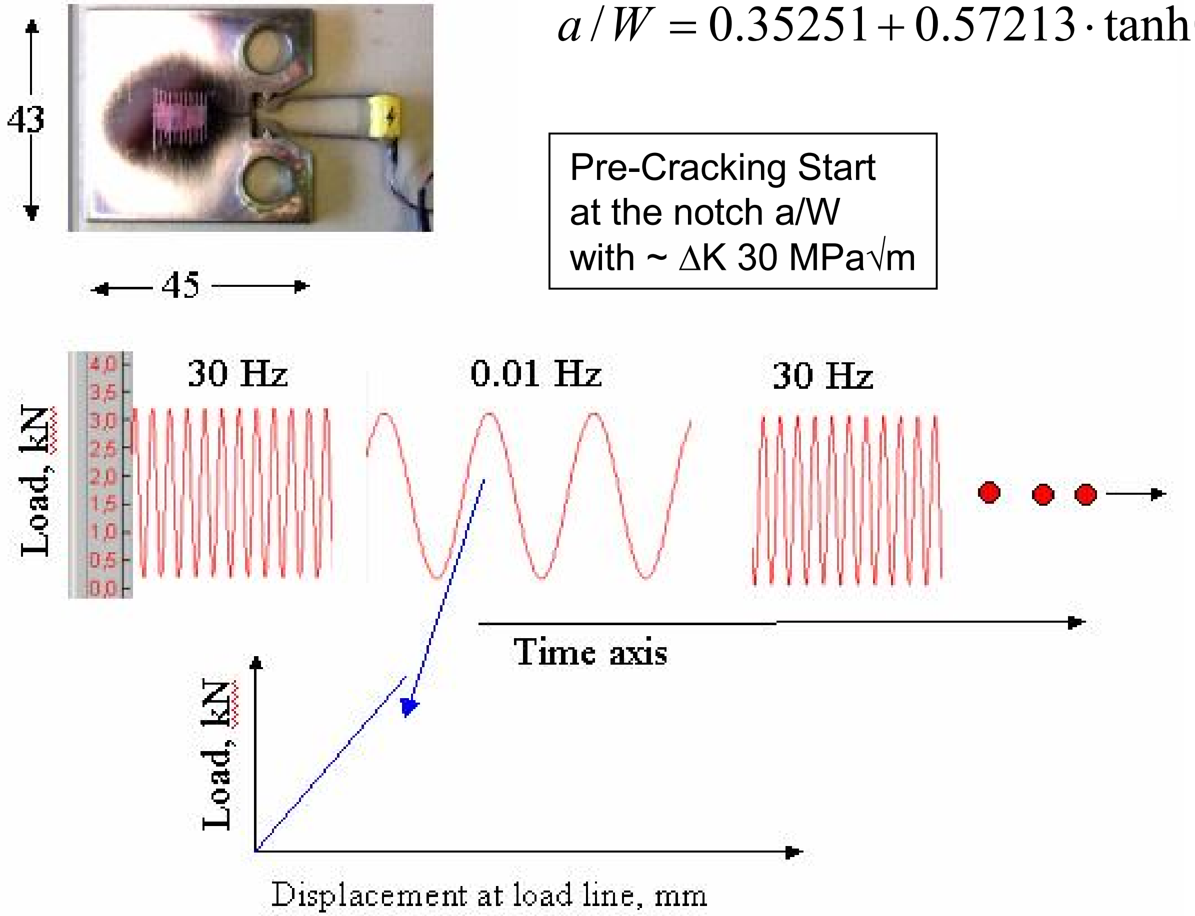

Pre-Cracking Start
at the notch position a/W = 0.33
with ~ ΔK 30 MPa√m

## Fatigue Crack Growth Rate (FCGR) Measurement Procedure

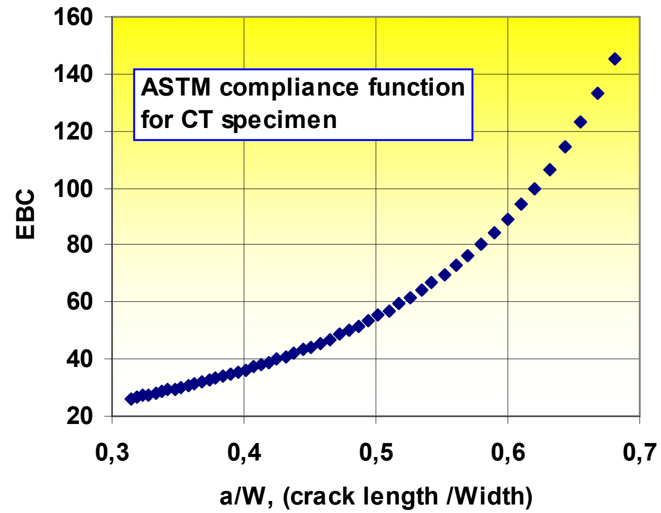
a/W = f( EBC)

$$EBC = Young's\ \ modulus \cdot Thickness \cdot \frac{1}{Stiffness}$$

$$a/W = 0.35251 + 0.57213 \cdot \tanh\left(0.85068 \cdot \left(\log(EBC) - 1.26064\right)\right)$$

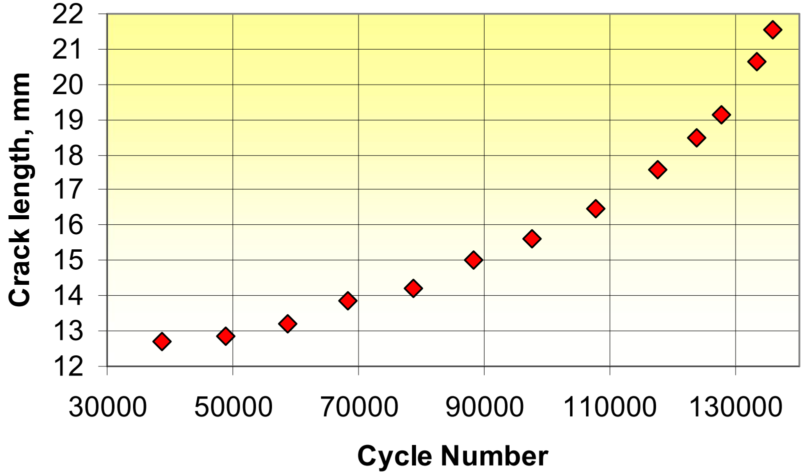



Pre-Cracking Start at the notch a/W with ~ ΔK 30 MPa√m

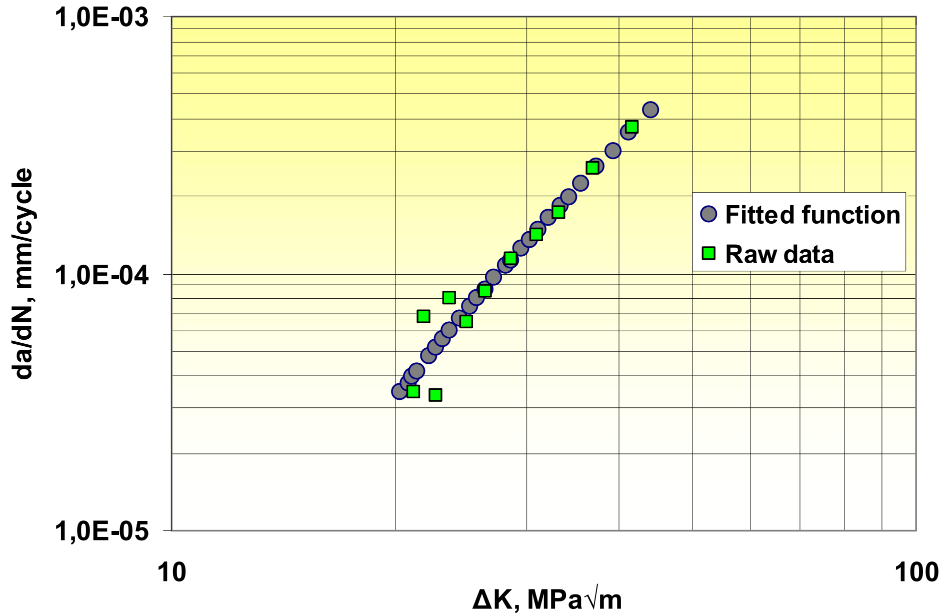


## Obtained FCGR raw data & its analysis by curve fitting

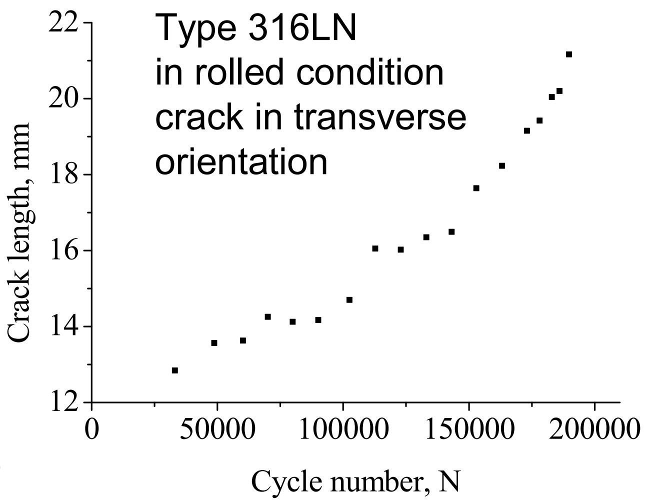
| a/W | crack length, mm | Slope, kN/mm | Regression Coefficient % | Load min, kN | Load max, kN | Cycle number | ΔK, MPa√m |
|---|---|---|---|---|---|---|---|
| 0,35 | 12,7 | 46759,6 | 99,93 | 0,27 | 2,70 | 38700 | 20,6 |
| 0,36 | 12,9 | 45799,0 | 99,93 | 0,27 | 2,70 | 48736 | 20,9 |
| 0,37 | 13,2 | 43828,5 | 99,94 | 0,27 | 2,70 | 58632 | 21,4 |
| 0,39 | 13,9 | 40253,1 | 99,90 | 0,27 | 2,70 | 68397 | 22,4 |
| 0,39 | 14,2 | 38515,7 | 99,93 | 0,27 | 2,70 | 78592 | 23,0 |
| 0,42 | 15,0 | 34783,2 | 99,95 | 0,27 | 2,70 | 88315 | 24,4 |
| 0,43 | 15,6 | 32156,0 | 99,93 | 0,27 | 2,70 | 97575 | 25,5 |
| 0,46 | 16,5 | 28690,1 | 99,96 | 0,27 | 2,70 | 107621 | 27,2 |
| 0,49 | 17,6 | 24631,1 | 99,97 | 0,27 | 2,70 | 117468 | 29,9 |
| 0,51 | 18,5 | 21816,6 | 99,97 | 0,27 | 2,70 | 123611 | 32,2 |
| 0,53 | 19,1 | 19819,6 | 99,98 | 0,27 | 2,70 | 127532 | 34,2 |
| 0,57 | 20,6 | 15868,4 | 99,93 | 0,27 | 2,70 | 133347 | 39,4 |
| 0,60 | 21,6 | 13643,9 | 99,92 | 0,27 | 2,70 | 135916 | 43,6 |



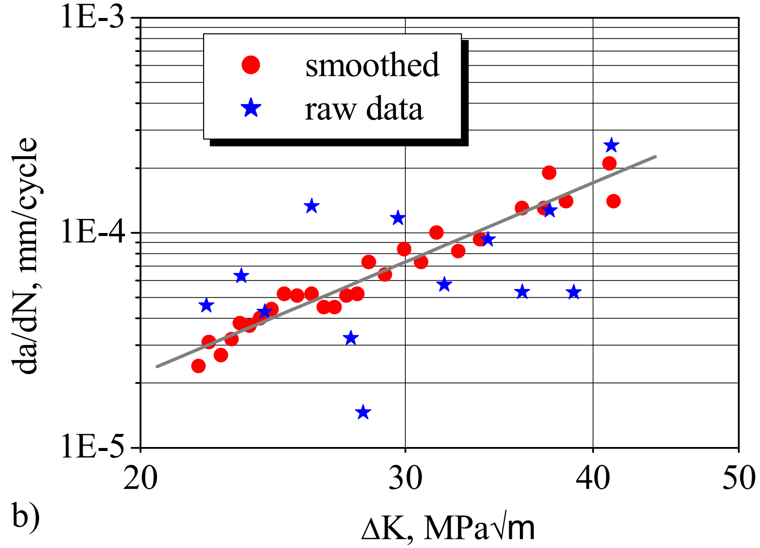

Specimen XX_FCGRTD_1
Raw data and smoothing of
raw data by using the DataFit Software



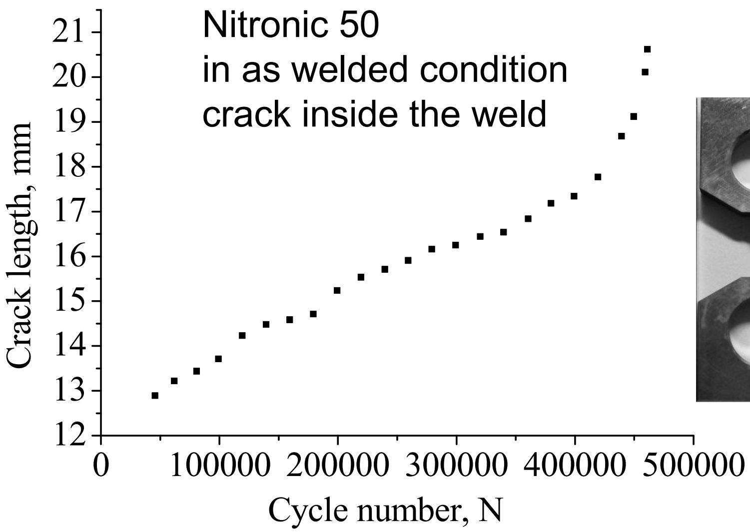
# FCGR investigations at 7 K of two different materials

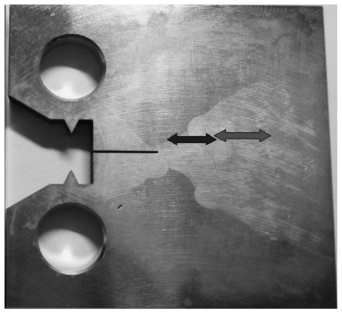



a)

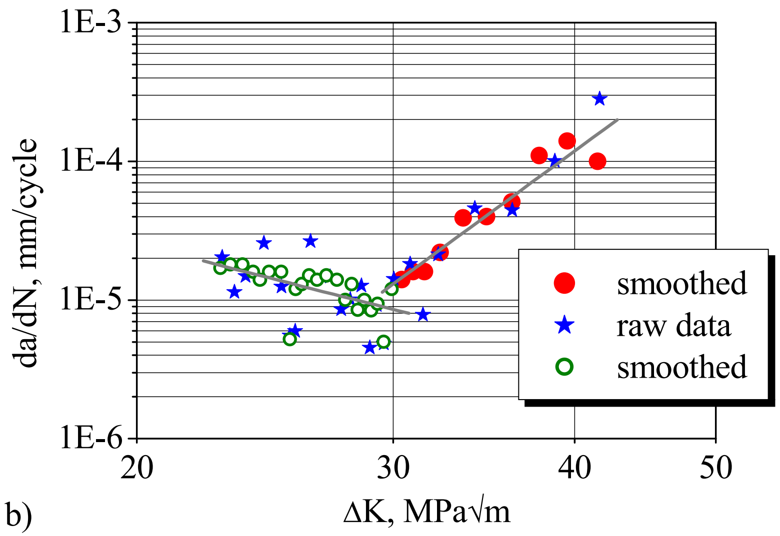



b)



a)




b)

## Uncertainty as "new" tool since around 1990

**Measurement and its reliability**

- For reporting the result of a measurement of a physical quantity, it should be obligatory to indicate the quality of the result quantitatively ***so*** that those who use it, can assess its reliability.
- Without such an indication, measurements cannot be compared. It **is** therefore necessary that there be a readily implemented, easily understood, and generally accepted procedure for characterizing the quality of a result of a measurement, that is, for evaluating and expressing its ***uncertainty.***

**GUM Method : "The Guide to Expression of Uncertainty in Measurement" was originally published jointly by seven international organizations.**
**These organizations are BIPM, IEC, IFCC, ISO, IUPAC, IUPAP and OIML.**

**http://www.gum.dk/e-gumfaq-publishers/GUMpublishers.html**

## Uncertainty concept a brief description

The result of any physical measurement comprises two parts:
an estimate of the true value of the measurand
and the uncertainty of this "best" estimate.

One can attempt to measure the true value by measuring "**the best estimate**" using uncertainty evaluations such as Type A (repeated measurements in the laboratory in general expressed in form of Gaussian distribution) and Type B (previous experiments, literature data, manufacturer's information, etc. in general in form of rectangular distribution) uncertainties.

| Type A uncertainty | Type B uncertainty | Combined uncertainty |
| --- | --- | --- |
| $u_A = \frac{s}{\sqrt{N}}$ | $u_B = \sqrt{\left(\frac{1}{3}\right) \cdot M_1^2 + \left(\frac{1}{3}\right) \cdot M_2^2 + \cdots\cdot}$ | $u_c = \sqrt{u_A^2 + u_B^2}$ |

The GUM, within this context, is a guide for a transparent, standardized documentation of the measurement procedure.

## Model equation and the procedure of uncertainty determination

$$\frac{\partial a}{\partial N} = C_0 \cdot \Delta K^m$$

Paris law (linear regime, “stage II”) in double log graph

$$\Delta K = \frac{\Delta P}{B \cdot \sqrt{W}} \cdot \frac{\left(2 + \frac{a}{W}\right)}{\left(1 - \frac{a}{W}\right)^{1.5}} \cdot \left(0{,}886 + 4{,}64 \cdot \frac{a}{W} - 13{,}32 \cdot \left(\frac{a}{W}\right)^2 + 14{,}72 \cdot \left(\frac{a}{W}\right)^3 - 5{,}6 \cdot \left(\frac{a}{W}\right)^4\right)$$

$$\frac{\partial a}{\partial N} = R = f(C_0, m, \Delta P, W, B, a)$$

## Combined standard uncertainty and its treatment

Model equation $\Rightarrow$ $$\frac{\partial a}{\partial N} = C_0 \cdot \Delta K^m \Rightarrow \frac{\partial a}{\partial N} = R = f(C_0, m, \Delta P, W, B, a)$$

Combined standard uncertainty using the Gaussian distribution law

$$u_c = \sqrt{\left(\frac{\partial R}{\partial C_0}\right)^2 u_1^2 + \left(\frac{\partial R}{\partial m}\right)^2 u_2^2 + \left(\frac{\partial R}{\partial \Delta P}\right)^2 u_3^2 + \left(\frac{\partial R}{\partial W}\right)^2 u_4^2 + \left(\frac{\partial R}{\partial B}\right)^2 u_5^2 + \left(\frac{\partial R}{\partial a}\right)^2 u_6^2}$$

$u_1 \cdots u_6$ are uncertainty coefficients,

whilst the derivatives are the sensitivity coefficients

Partial differentiation & determination of sensitivity coefficients

$$\frac{\delta R}{\delta C_0} = \Delta K^m \quad \& \quad \frac{\delta R}{\delta m} = C_0 \cdot \Delta K^m \cdot \ln \Delta K$$

$$\frac{\partial R}{\partial \Delta P} = C_0 \cdot \Delta K^m \cdot \frac{m}{\Delta P} \quad \& \quad \frac{\partial R}{\partial B} = -C_0 \cdot \Delta K^m \cdot \frac{m}{B}$$

## Partial differentials for width “W” and crack length “a”

$$\frac{\partial R}{\partial W} = C_0 \cdot \Delta K^m \cdot m \cdot \frac{\left[F_A + F_B + F_C + F_D\right]}{\Delta P} \cdot B \cdot F_E$$

$$\frac{\partial R}{\partial a} = C_0 \cdot \Delta K^m \cdot m \cdot \frac{\left[-\frac{2 \cdot F_A}{\left(2 + \frac{a}{W}\right)} + F_F - \frac{F_D \cdot \sqrt[5]{W}}{\sqrt[3]{W}}\right]}{\Delta P} \cdot B \cdot F_E$$

## Functions $F_A$ ….. $F_F$ of the partial derivative equation

$$F_A = -\Delta P \cdot \left(2+\frac{a}{W}\right)\cdot \frac{\left(8.886+4.64\cdot\left(\frac{a}{W}\right)-13.32\cdot\left(\frac{a}{W}\right)^2+14.72\cdot\left(\frac{a}{W}\right)^3-5.6\cdot\left(\frac{a}{W}\right)^4\right)}{\left(1-\frac{a}{W}\right)^{1.5}\cdot 2\cdot B\cdot\sqrt[3]{W}}$$

$$F_B = -\Delta P \cdot a \cdot \frac{\left(8.886+4.64\cdot\left(\frac{a}{W}\right)-13.32\cdot\left(\frac{a}{W}\right)^2+14.72\cdot\left(\frac{a}{W}\right)^3-5.6\cdot\left(\frac{a}{W}\right)^4\right)}{\left(1-\frac{a}{W}\right)^{1.5}\cdot B\cdot\sqrt[5]{W}}$$

$$F_C = \Delta P \cdot \frac{\left(2+\frac{a}{W}\right)\cdot\left(-4.64\cdot\frac{a}{W^2}+26.64\cdot\frac{a^2}{W^3}-44.16\cdot\frac{a^3}{W^4}+22.4\cdot\frac{a^4}{W^5}\right)}{\left(1-\frac{a}{W}\right)^{1.5}\cdot B\cdot\sqrt{W}}$$

$$F_D = -1.5\cdot\Delta P \cdot \frac{\left(2+\frac{a}{W}\right)\cdot\left(8.886+4.64\cdot\left(\frac{a}{W}\right)-13.32\cdot\left(\frac{a}{W}\right)^2+14.72\cdot\left(\frac{a}{W}\right)^3-5.6\cdot\left(\frac{a}{W}\right)^4\right)}{\left(1-\frac{a}{W}\right)^{2.5}\cdot B\cdot\sqrt[5]{W}}$$

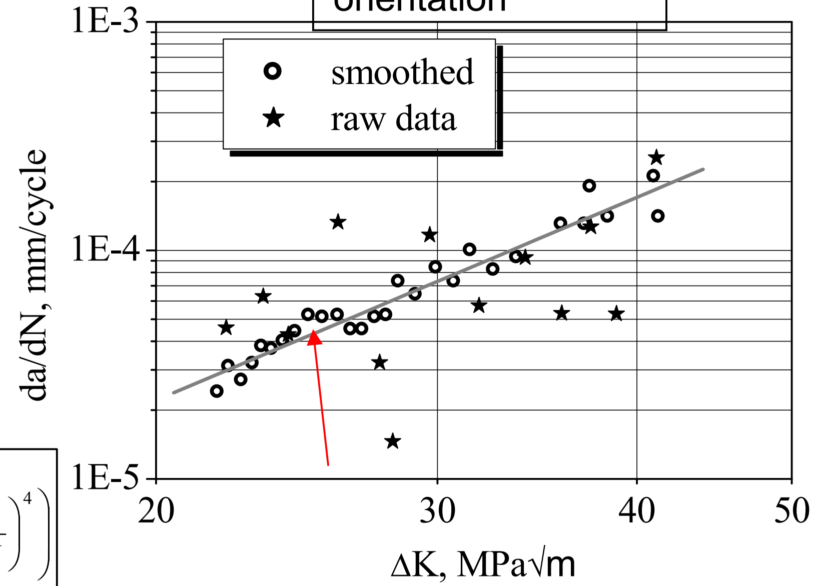
$$F_E = \frac{\sqrt{W}}{\left(8.886+4.64\cdot\frac{a}{W}-13.32\cdot\left(\frac{a}{W}\right)^2+14.72\cdot\left(\frac{a}{W}\right)^3-5.6\cdot\left(\frac{a}{W}\right)^4\right)}\cdot\frac{\left(1-\frac{a}{W}\right)^{1.5}}{\left(2+\frac{a}{W}\right)}$$

$$F_F = \Delta P \cdot \frac{\left(2+\frac{a}{W}\right)\cdot\left(4.64\cdot\frac{1}{W}-26.64\cdot\frac{a}{W^2}+44.16\cdot\frac{a^2}{W^3}-22.4\cdot\frac{a^3}{W^4}\right)}{\left(1-\frac{a}{W}\right)^{1.5}\cdot B\cdot\sqrt{W}}$$

## Computation of sensitivity values

Type 316LN
in rolled condition
crack in transverse
orientation

Computed Paris law constants
$C_0 = 3.226.10^{-9}$ & $m = 2.949$




with
a = 1.5 cm, W = 3.6 cm, B = 0.4 cm, ΔP = 2.5 kN

$$\Delta K = \frac{\Delta P}{B \cdot \sqrt{W}} \cdot \frac{\left(2 + \frac{a}{W}\right)}{\left(1 - \frac{a}{W}\right)^{1.5}} \cdot \left(0{,}886 + 4{,}64 \cdot \frac{a}{W} - 13{,}32 \cdot \left(\frac{a}{W}\right)^2 + 14{,}72 \cdot \left(\frac{a}{W}\right)^3 - 5{,}6 \cdot \left(\frac{a}{W}\right)^4\right)$$

ΔK is ~ 25 MPa√m

Results of the computed partial differentials (sensitivity coefficients)

| $\frac{da}{dN}$ | $\Delta K$ | $\frac{\partial R}{\partial C_0}$ | $\frac{\partial R}{\partial m}$ | $\frac{\partial R}{\partial \Delta P}$ | $\frac{\partial R}{\partial W}$ | $\frac{\partial R}{\partial B}$ | $\frac{\partial R}{\partial a}$ |
|---|---|---|---|---|---|---|---|
| $4.311 \cdot 10^{-5}$ | 25.066 | $1.336 \cdot 10^{4}$ | $1.918 \cdot 10^{-4}$ | $5.085 \cdot 10^{-5}$ | $-5.724 \cdot 10^{-5}$ | $-3.178 \cdot 10^{-4}$ | $9.50 \cdot 10^{-5}$ |

## Uncertainty terms for m and $C_0$ and its treatment

Assumption: experimental errors are identical for all four tests as the uncertainties resulting from the geometrical constraints has been considered elsewhere. Therefore, m and $C_0$ are determined using the experimental results.

| Specimen, condition, & code | $C_0$, specimen # 1 | $C_0$, specimen # 2 | absolute difference | m, specimen # 1 | m, specimen # 2 | absolute difference |
|---|---|---|---|---|---|---|
| 316LN rolled plate, longitudinal | $1.79\cdot10^{-9}$ | $2.65\cdot10^{-9}$ | $8.64\cdot10^{-10}$ | 3.092 | 3.012 | 0.080 |
| 316LN rolled plate, transversal | $3.23\cdot10^{-9}$ | $1.37\cdot10^{-9}$ | $1.86\cdot10^{-9}$ | 2.949 | 3.193 | 0.244 |

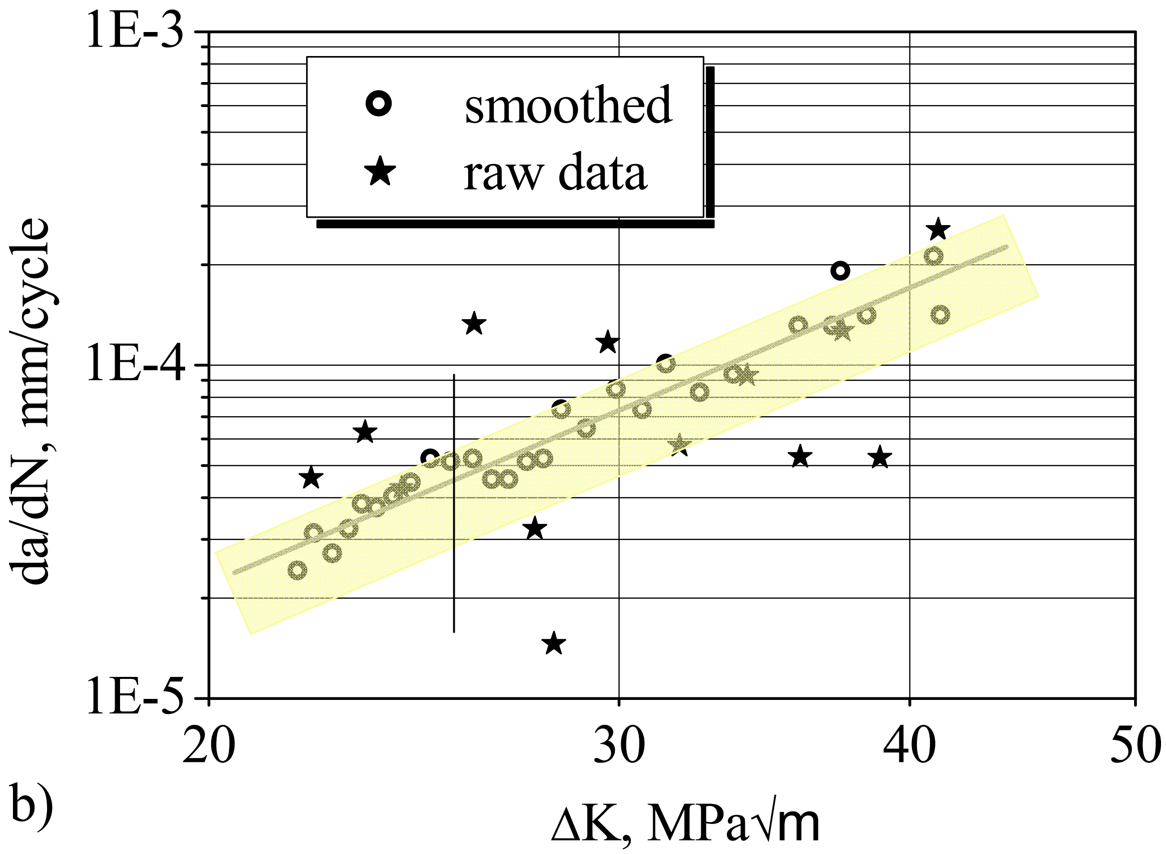
For m, a, $C_0$, and, $\Delta P$ treatment according to Type B. W and B according to Type A

$$u_c = \sqrt{\left(1.336\cdot10^{4}\right)^2\cdot\left(3.93\cdot10^{-10}\right)^2+\left(1.918\cdot10^{-4}\right)^2\cdot(0.047)^2+\left(5.085\cdot10^{-5}\right)^2\cdot(0.0008)^2+\left(-5.724\cdot10^{-5}\right)^2\cdot(0.002)^2+\left(-3.178\cdot10^{-4}\right)^2\cdot(0.002)^2+\left(9.5\cdot10^{-5}\right)^2\cdot(0.115)^2}\quad\frac{mm}{cycle}$$

$$\boxed{u_C = 1.51\cdot10^{-5}\quad\frac{mm}{cycle}}$$

$$\boxed{\frac{da}{dN} = R = 4.311\cdot10^{-5}\quad\pm\quad1.51\cdot10^{-5}\quad\frac{mm}{Cycles}\quad for\quad \Delta K = 25.07\quad MPa\sqrt{m}}$$

## Best estimate and the uncertainty of the measurement




Type 316LN
in rolled condition
crack in transverse
orientation