Mechanical Testing Issues of Advanced Materials for GenIV, ADS and Fusion Systems

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Materials issues associated with current reactor design

- Tensile strength
- Toughness
- Aqueous corrosion resistance
- Radiation damage tolerance

Materials issues with GenIV

- Strength
- Toughness
- Fatigue: thermal & mechanical
- Creep & creep-fatigue interaction
- New aspects of coolant compatibility (SCWR, VHTR & GFR, SFR & LFR, MSR)
- Radiation damage @ high dose levels
- Fabrication routes and joining techniques

Damage interaction, transients associated with DBA and NDBA become more critical issues, as materials are taken closer to their limits.

=> Importance of (non-standard) thermo-mechanical, environmental testing

Outline

- **Small specimen geometries** for mechanical testing
- Application of the CEN **Small Punch Creep Testing** Code of Practice to a representative repair welded P91 pipe
- **Thermal Fatigue** Studies for Nuclear Piping Components
- Strain-Controlled **Thermo-Mechanical Fatigue**: Research into Best Practices

Small specimen geometries for mechanical testing

Introduction

Variety of mechanical tests types foreseen in GETMAT project

Tensile, Impact, Fracture toughness, Fatigue, Creep, Creep-Fatigue

Limited material availability due to the fabrication process

14Cr-ODS (PM): 45kg bars of 12mm, or plates of 3mm in thickness

9Cr-ODS (PM): 45kg plates of 6mm thickness

9Cr-ODS (EMS): 200kg plates of 10mm thickness

Material quantities requested from participants exceed the available quantities

Optimised use of test material required

⇓

Small specimen testing

(M. Serrano, CIEMAT)

International symposia

- ASTM SSTT symposia (initiated in 1986, last in 2007)
- IEA Symposium on SSTT for fusion materials (1995 and 1996)

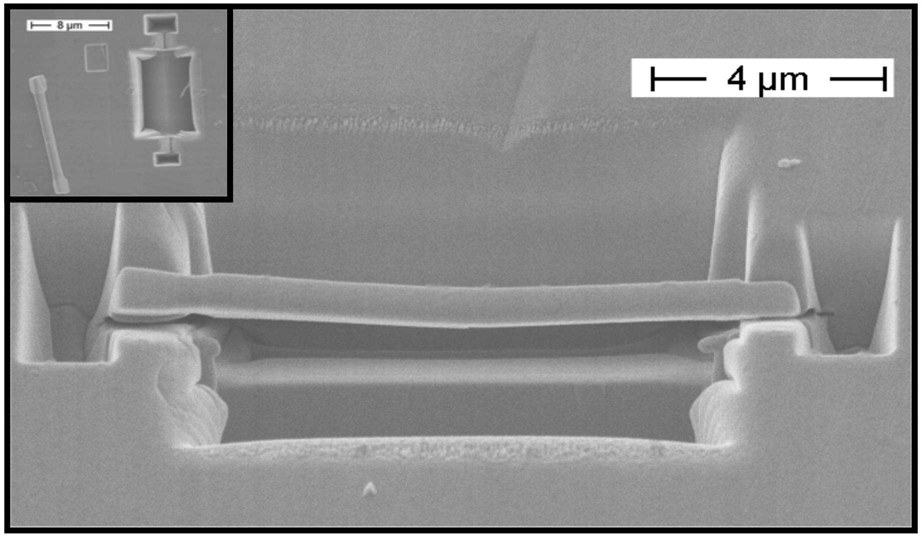
Review papers

- Lucas, G.E., Odette, G.R., Matsui, H., Moslang, A., Spatig, P., Rensman, J., Yamamoto, T. "The role of small specimen test technology in fusion materials development" (2007) Journal of Nuclear Materials, 367-370 B (SPEC. ISS.), pp. 1549-1556

Reactor pressure vessel surveillance – Fracture toughness from pre-cracked charpy specimens

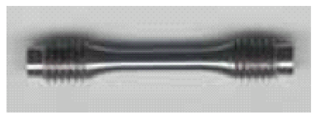
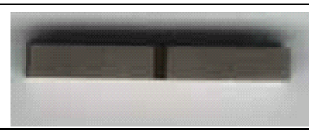
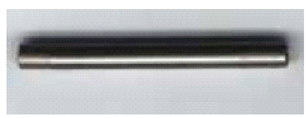
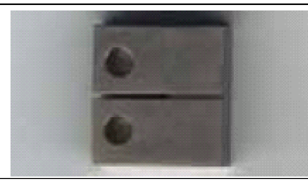
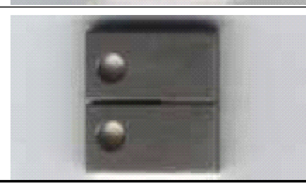
Fusion community – IFMIF irradiation

⬄ ***Micro-mechanical testing***



Focused ion beam (FIB) milled three-point bend specimen in Fe (S. Roberts, U Oxford)

Small Specimens Test Techniques (SSTT)

Specimen type	Present geometry	Comments
Tensile		developed
Fatigue		developed
Bend/Charpy DFT		Standard achieved; R&D ongoing
Creep		Miniaturization needs verification
Crack growth		International R&D ongoing
Fracture toughness		International R&D ongoing

Significant volume reduction (20-125 times) compared to conventional standards

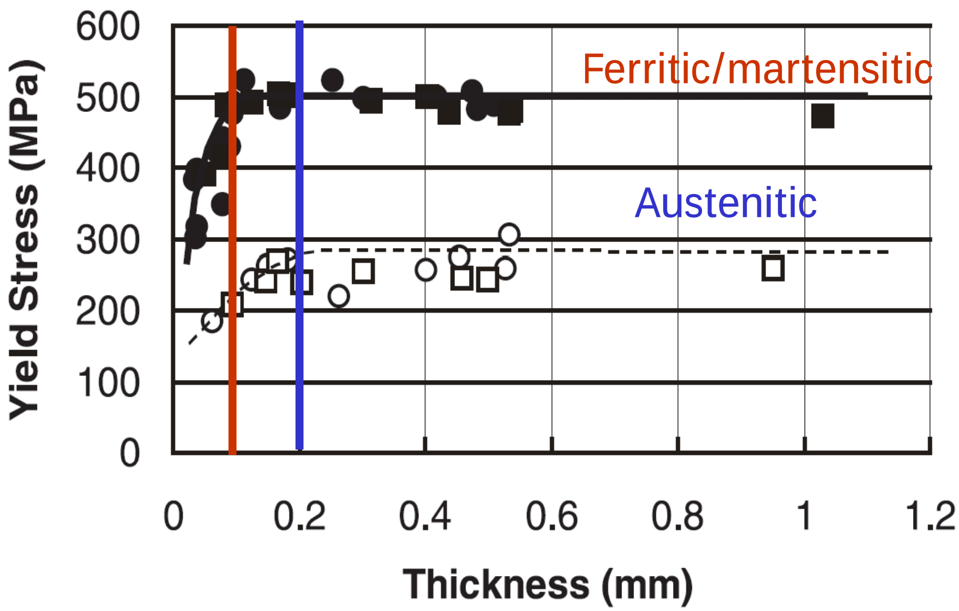
There are "**Developed**" specimen shapes, but are yet to be "**Standardized**" to be used for designing and licensing purposes

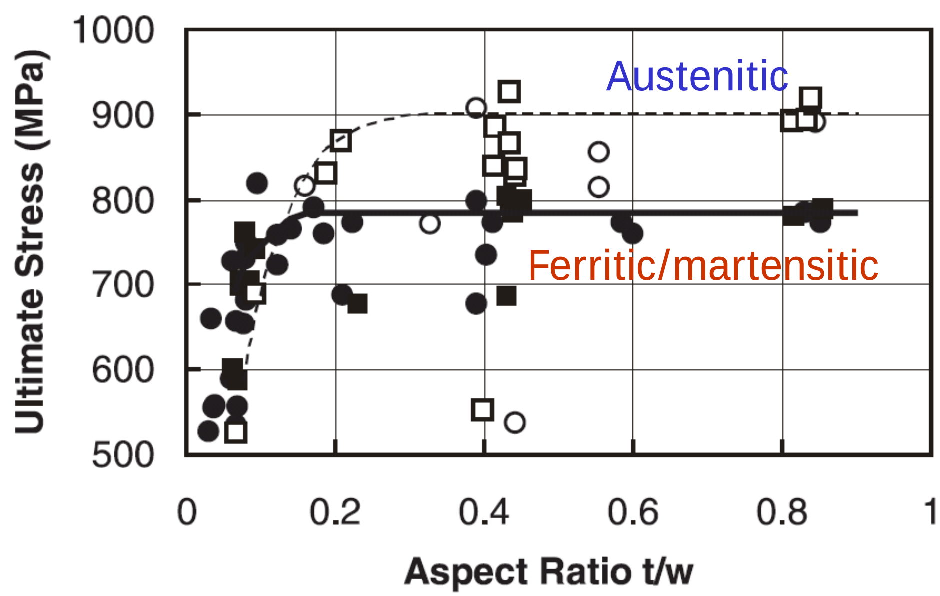
H. Matsui, "Development of Small Specimen Test Technique (SSTT) Standardization in the IMR for its Eventual IFMIF/EVEDA Activity", 24TH SYMPOSIUM ON EFFECTS OF RADIATION ON NUCLEAR MATERIALS AND THE NUCLEAR FUEL CYCLE June 24-26, 2008

SSTT Tensile

Size effect on tensile properties *Y. Kohno et al. JNM 283-287 (2000)*

– Yield strength is thickness independent for thicknesses larger than a critical value.
– Ultimate tensile stress depends on specimen aspect ratio for aspect ratios smaller than a critical value (affected by irradiation).





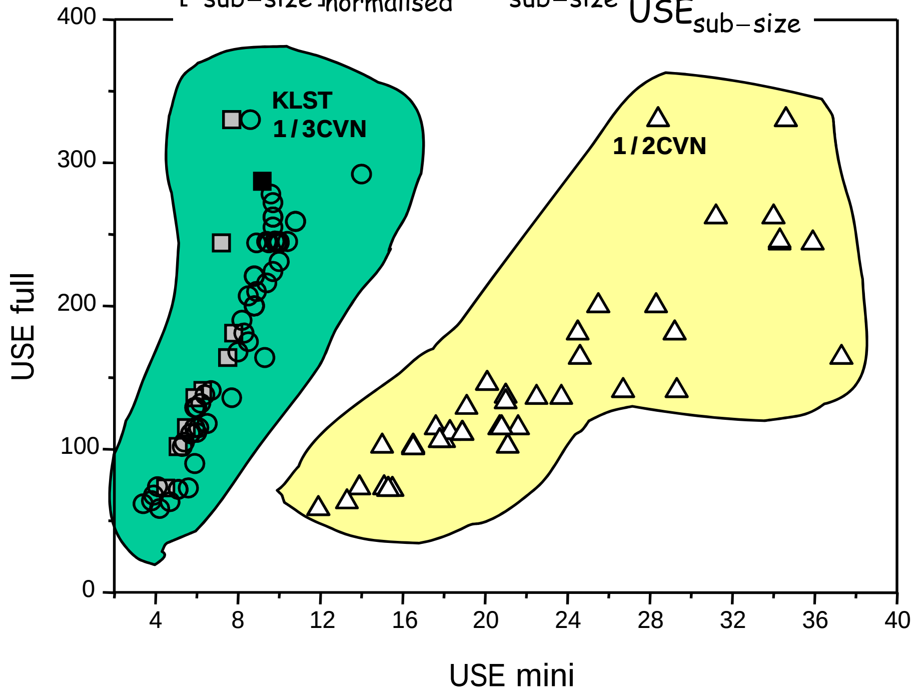
SSTT Impact

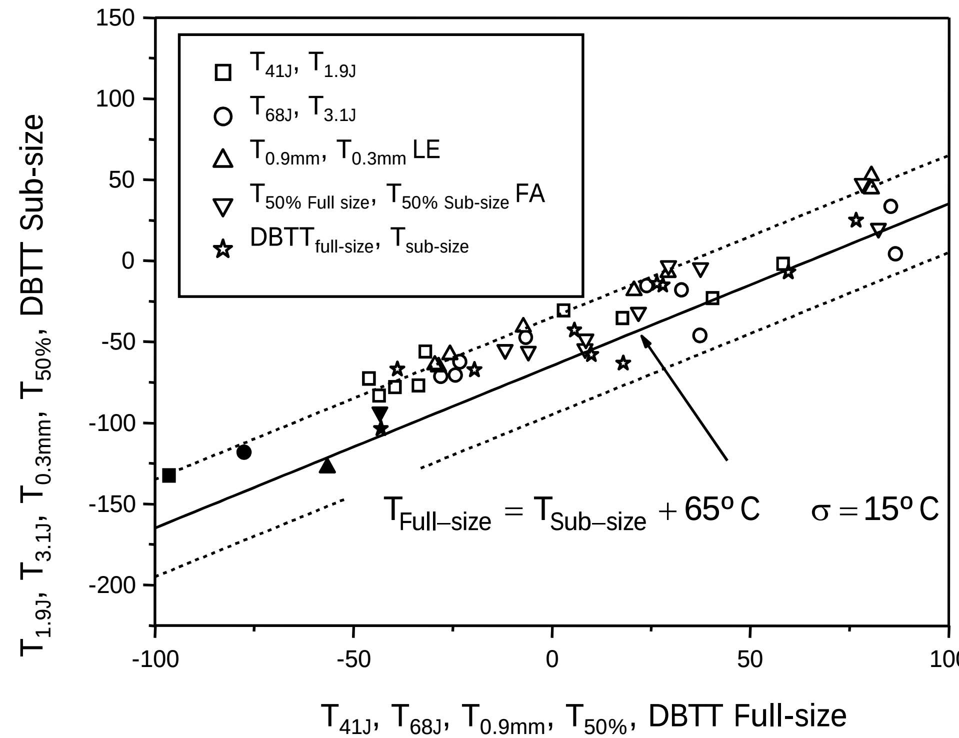
Reduction on specimen size reduces both Upper Self-Energy (USE) and Ductile to Brittle Transition Temperature (DBTT), due to change in stress state.

KLST (DIN) and ½ size Charpy V geometries (ESIS) are widely used.

Empirical correlations exist for 9Cr steels, e.g.

$$\left[E_{sub-size}\right]_{normalised} = E_{sub-size}\frac{USE_{full-size}}{USE_{sub-size}}$$





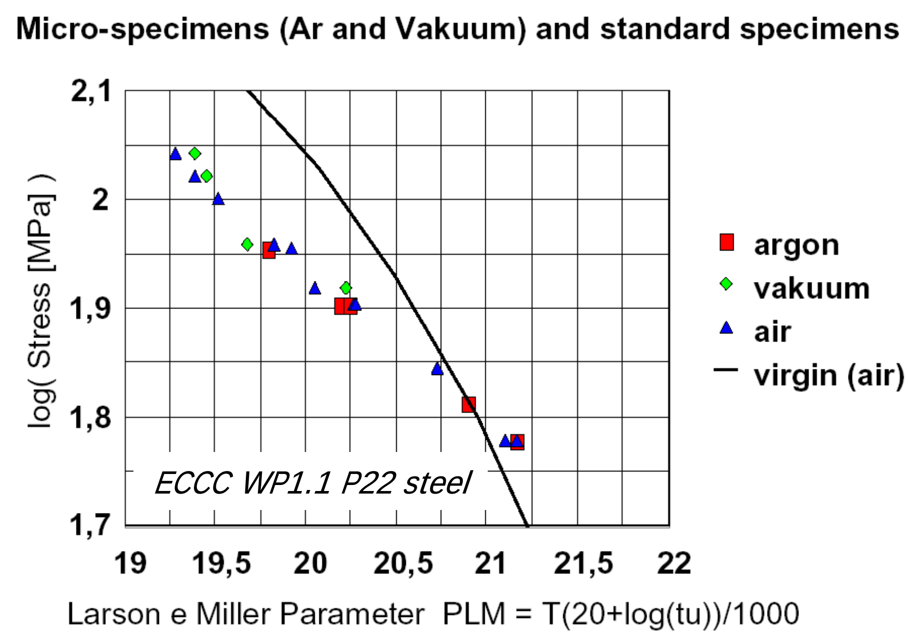
ECCC RECOMMENDATIONS - VOLUME 3 Part III [Issue 4] for gauge section length L_0 and diameter d_0 :

Full-size specimens: $L_0/d_0 > 3$

Sub-size specimens : 3 mm < d_0 < 5 mm

Miniature specimens d_0 < 3 mm

- Generally good agreement between the creep rupture results of miniature specimens and those of conventional sized ones
- However, diffusion processes may induce a size effect, which tends to be more pronounced at lower stress levels [K. Krompholz, JNM 305 (2002) 112–123]
- Size sensitivity may also be associated with oxidation, bending misalignment.



SSTT Fatigue

Size effect for fatigue life is twofold:

- **Statistical** for fatigue crack **initiation** (probability of fatigue crack initiators in the gauge section increases with size).
- **Deterministic** for fatigue crack **growth** until certain load drop level (ligament size)

Recommended standard specimens, e.g. ASTM E606

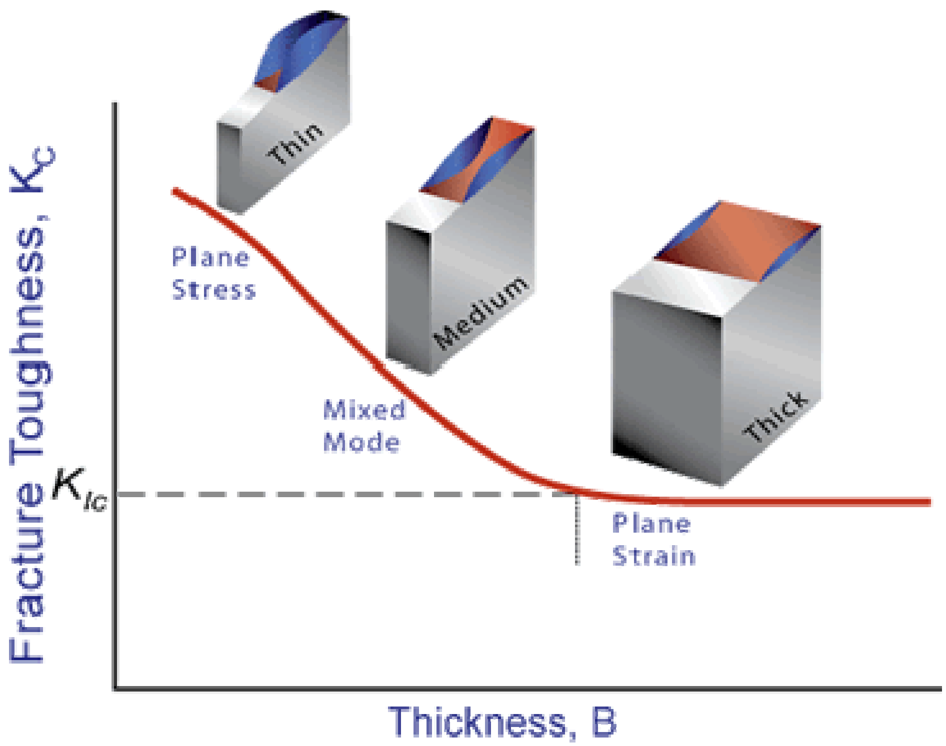
SSTT Fracture toughness

Fracture toughness is usually defined for **small scale yielding conditions** (plastic zone size << specimen dimensions).

Otherwise, fracture toughness is size dependent due to loss of constraint.

⇒ **Size limitations** of fracture toughness specimens included in the standards.

⇒ Relevance for fracture mechanics testing of thick components (e.g. RPV)



Application of the CEN Small Punch Creep Testing Code of Practice to a representative repair welded P91 pipe

SSTT Small Punch (SP)

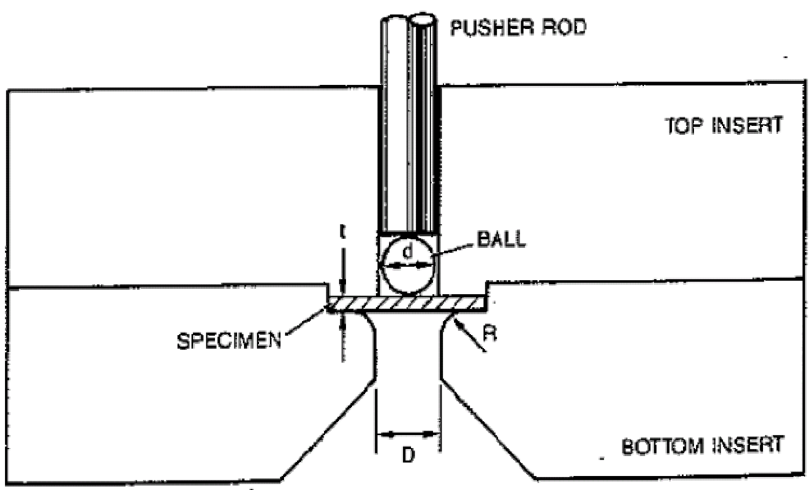
Developed by MTI in 1981, explored in US & Japan in the '80s, SP was introduced in Europe in 1992, in testing of steels.

CEN Workshop Agreement, CWA 15627, “Small Punch Test Method for Metallic Materials” developed on

Part A: A Code of Practice for Small Punch **Creep** Testing

- Time dependence of the deflection in the centre of the specimen is recorded
- Relationship between the SP load and uniaxial creep stress from the stretching membrane theory

Part B: A Code of Practice for Small Punch Testing for **Tensile** and **Fracture** Behaviour.



Lucas, JNM 141-143 (1986) 532-535

Small punch testing – why ?

Advantages

- Capable of generating tensile, toughness, and creep strength data from very small specimens
- Ability to sample from large component, relatively non-destructive (no repair is needed afterwards)
- Test itself is rather simple to perform and inexpensive
- Useful tool in assessing structural component integrity, for instance in weldments, clads and other anisotropic or critically weak regions

Open Questions

- Representativeness of such small specimens
- Reliability of correlation with uniaxial creep data

JRC

CEN WORKSHOP AGREEMENT

CWA 15627

December 2006

ICS 77.040.10

English version

Small Punch Test Method for Metallic Materials

This CEN Workshop Agreement has been drafted and approved by a Workshop of representatives of interested parties, the constitution of which is indicated in the foreword of this Workshop Agreement.

The formal process followed by the Workshop in the development of this Workshop Agreement has been endorsed by the National Members of CEN but neither the National Members of CEN nor the CEN Management Centre can be held accountable for the technical content of this CEN Workshop Agreement or possible conflicts with standards or legislation.

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Institute for Energy

The application of the SP test for creep has gained significant interest in the last decade, also as a result of research coordinated by the European Pressure Equipment Research Council (EPERC) to develop a CEN Code of Practice for the application and use of the small punch test for both creep rupture and tensile and toughness properties.

P91 Material investigated

1. <u>INTEGRITY Project of repair welds</u>

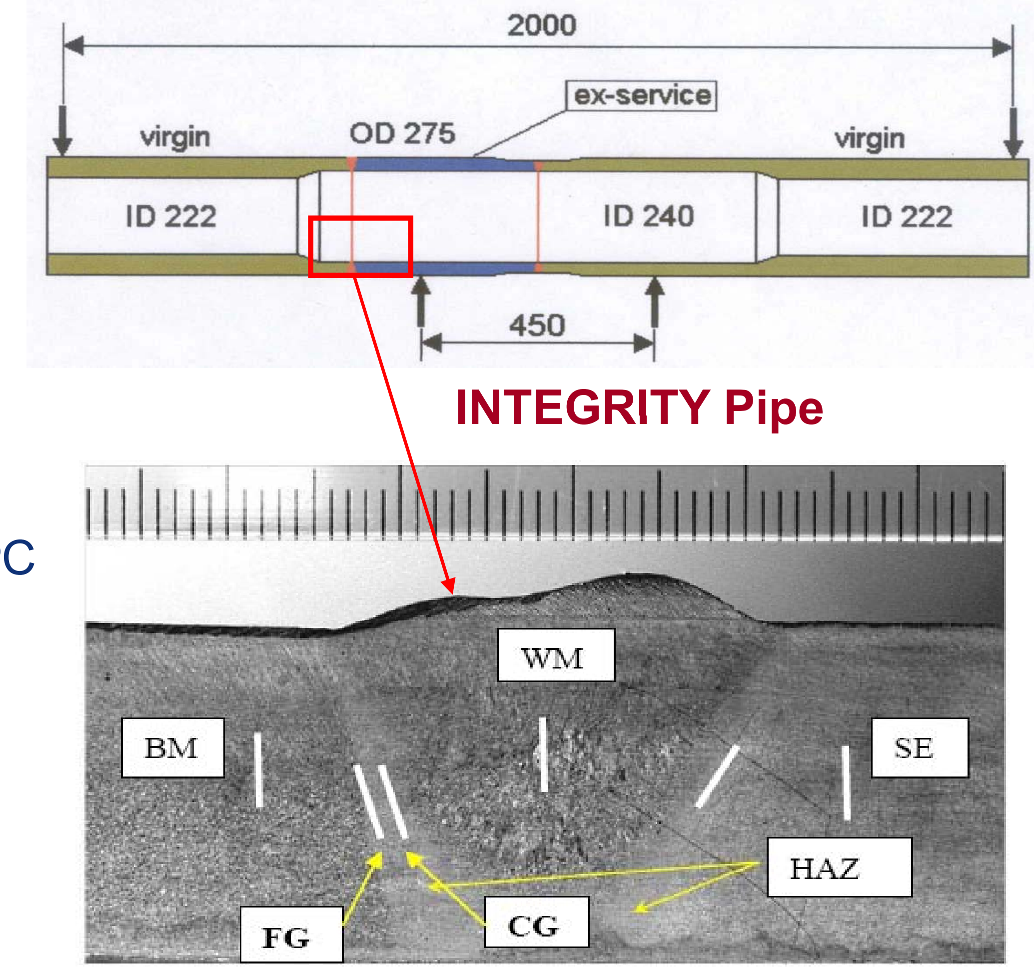
BM (P91 Base material)

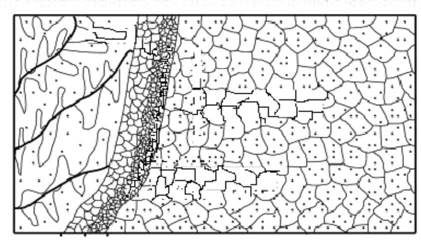
WM (Weld material)

HAZ material

SE (Service exposed*)

*Service exposed conditions: 60kh at 565^{o}C under pressure of 250 bar.



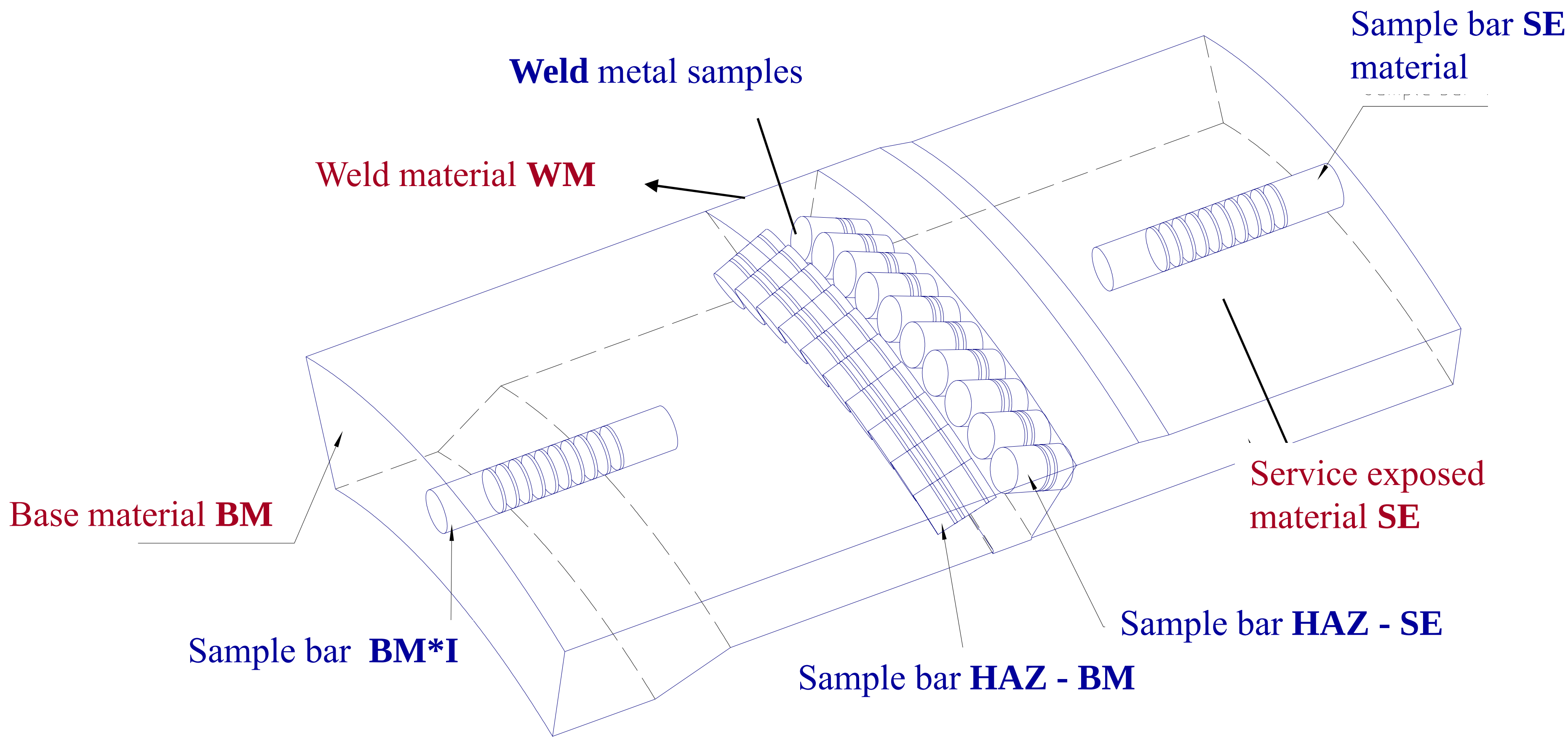


2. <u>BRITE-EURAM LICON Project</u>

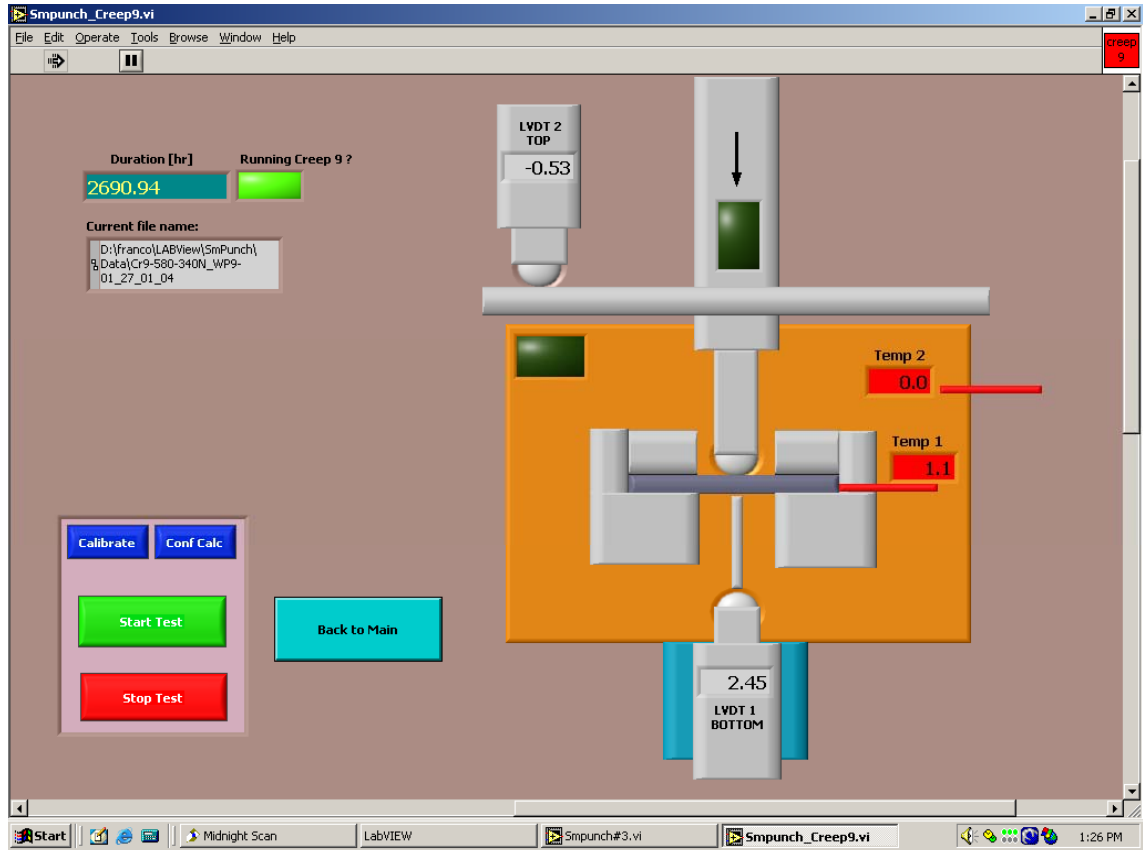
Virgin P91 material

(D. Blagoeva & R. Hurst)

SP sampling (8mm dia x 0.5mm thick)



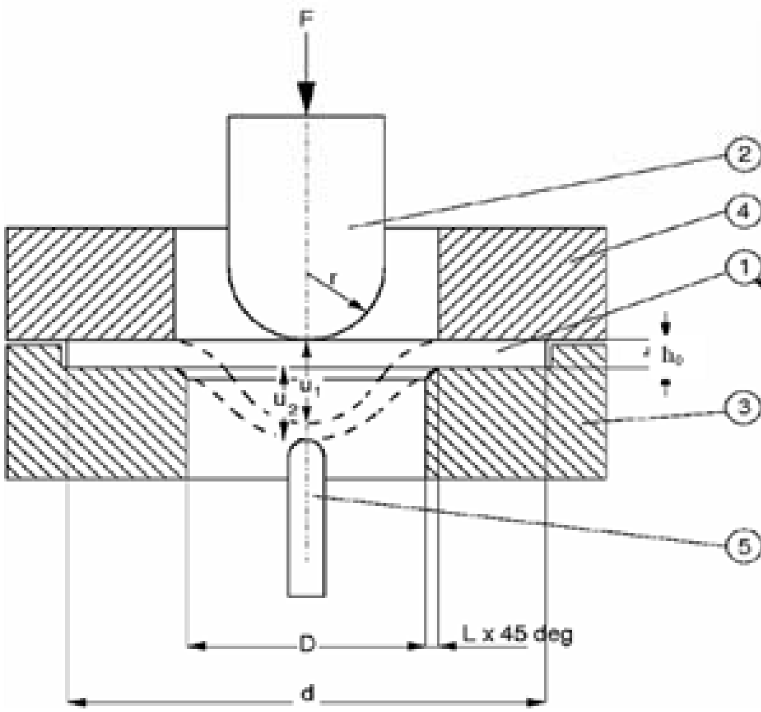
SP Testing Equipment

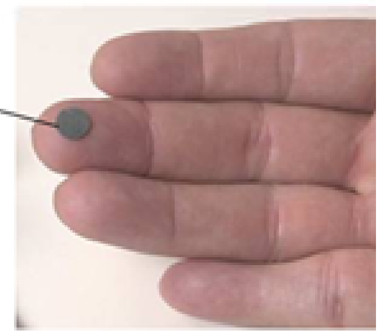


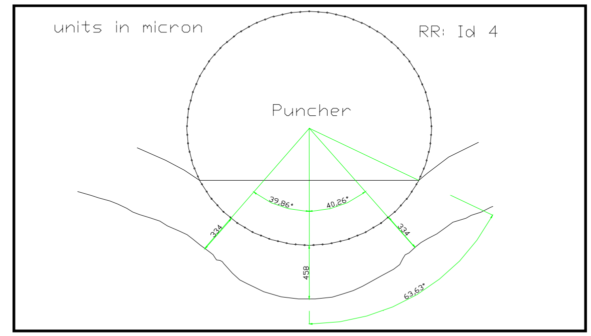
SP control interface

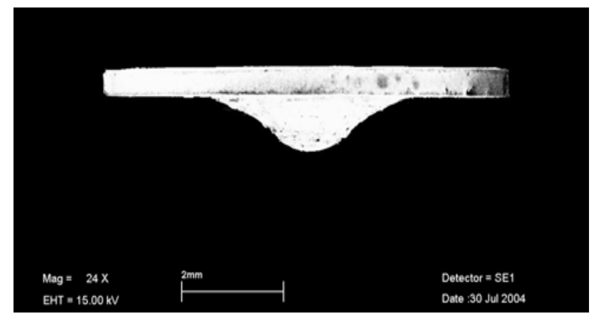
SP creep test rigs at JRC-IE

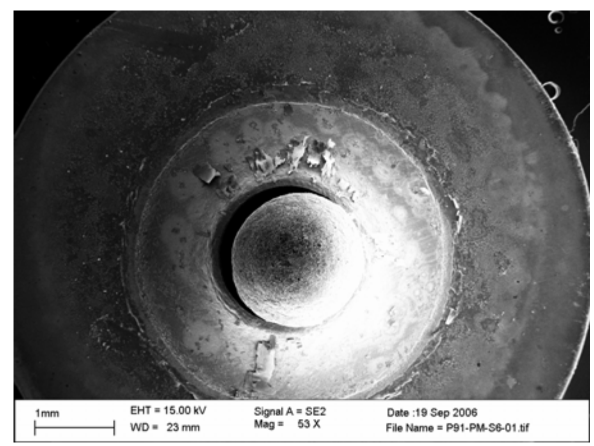
SP schematic









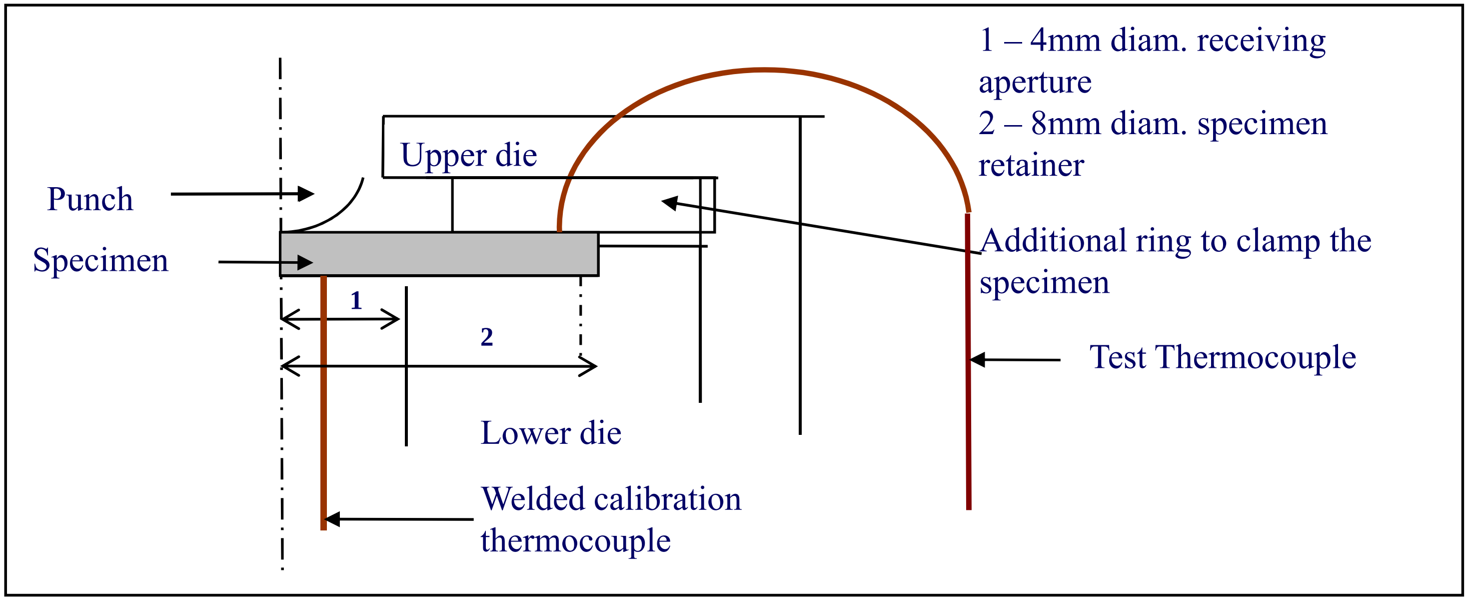


Key
1. SP disc test-piece
2. Hemispherical ended punch
3. Lower die
4. Upper die
5. Dilatometer push rod

- Sample: Disc of Ø d = 8 mm, and thickness h = 0.5 mm
- Hemispherical Puncher: radius r = 1.0 mm
- Receiving hole: R = 4 mm
- Protective atmosphere of Argon

Setting-up SP tests - Code of Practice

1. Accurate temperature control



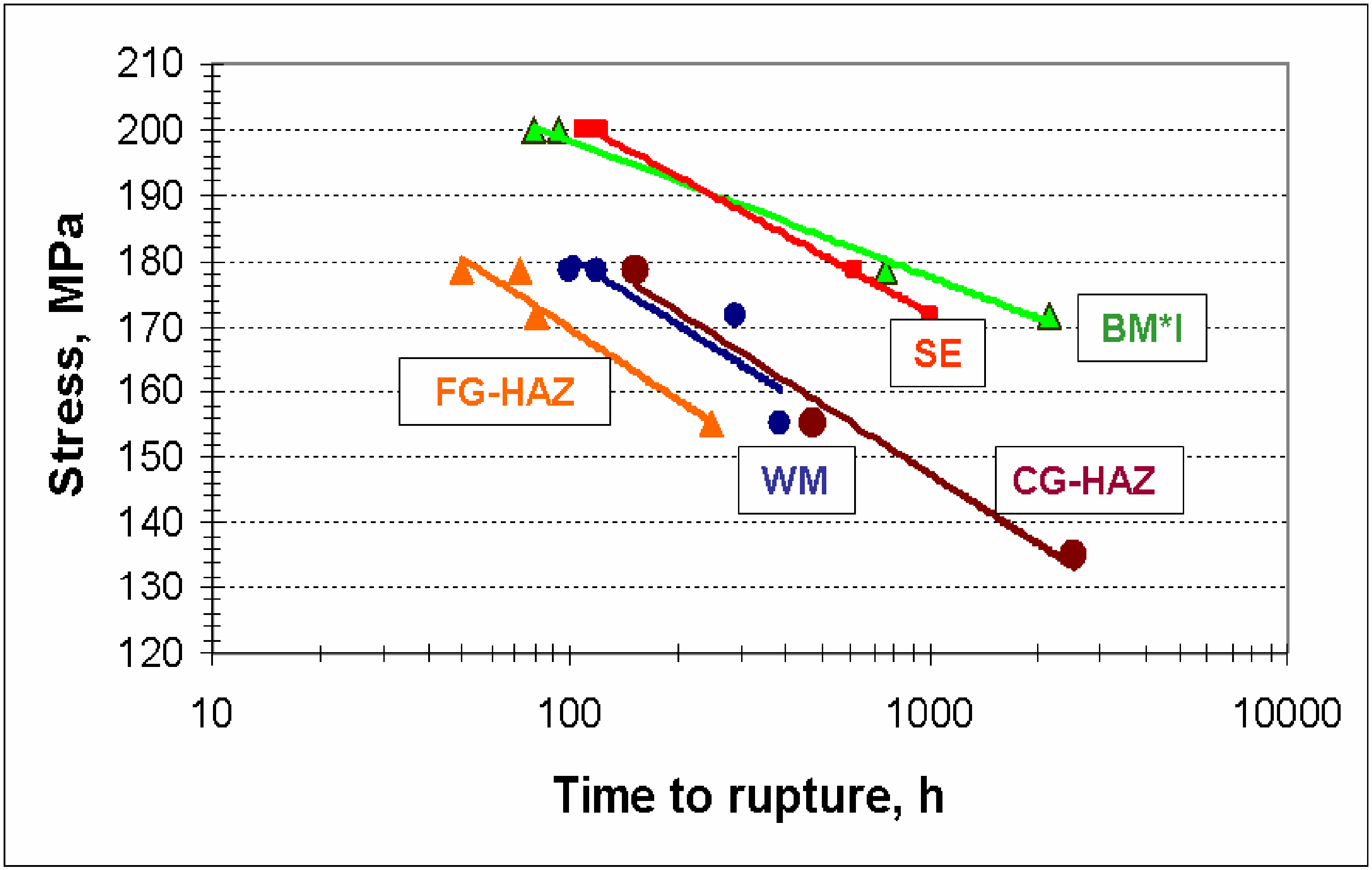
2. Correlating forces and stresses

$$F/\sigma = 3.33\ K_{SP}\ R^{-0.2}\ r^{1.2}\ h$$

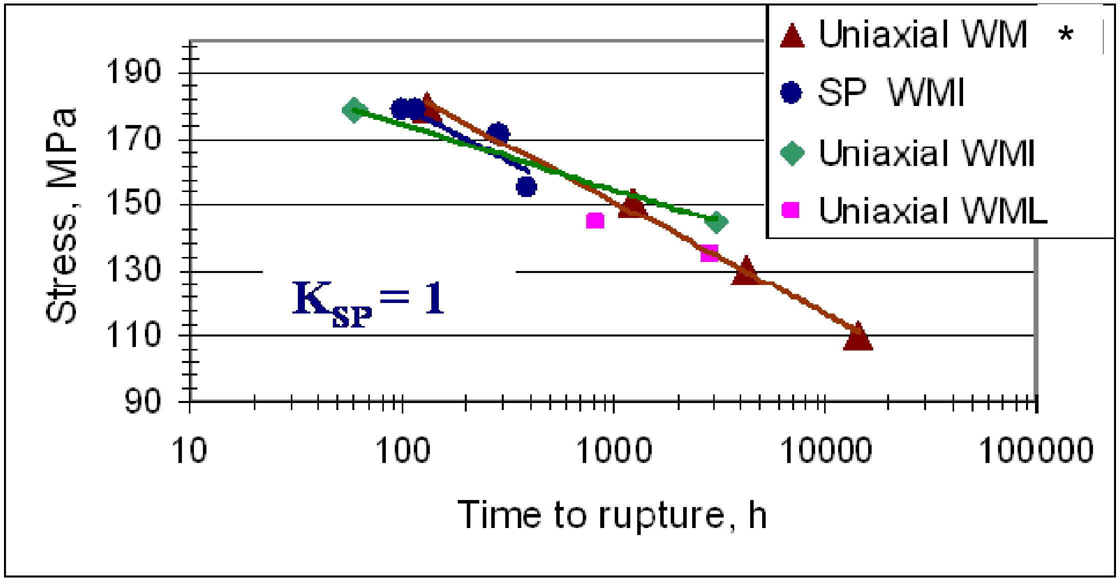
r	radius of the punch indenter
h	specimen thickness
R	radius of the receiving hole
K_{SP}	ductility constant for the material under test

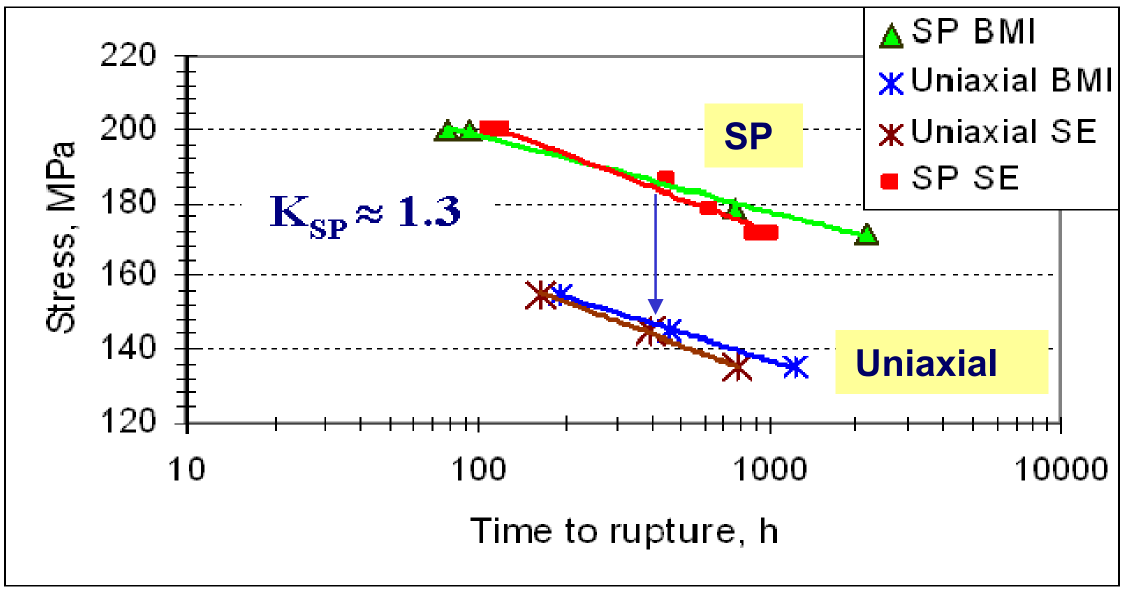
Stress-Rupture Results

Comparison of SP creep rupture results for different zones from the P91 weldment



Stress-Rupture Results



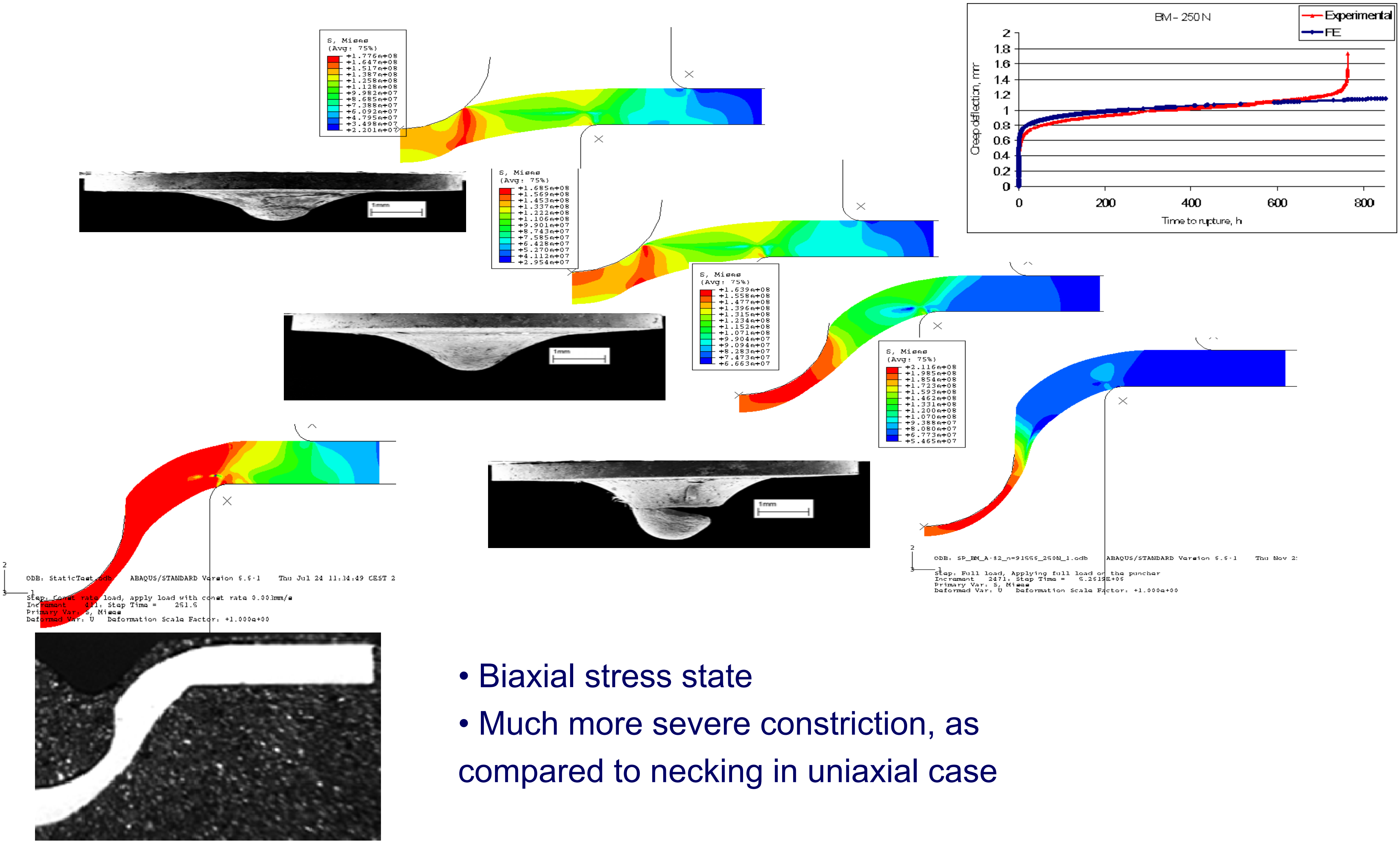


Comparison between uniaxial & SP stress rupture data

$$F_{SP}/\sigma = 3.33\ K_{SP}\ R^{-0.2}\ r^{1.2}\ h$$

* R.Sandström et al. uniaxial creep data for P91 at 600°C

FE Modelling



- Biaxial stress state
- Much more severe constriction, as compared to necking in uniaxial case

Conclusions on SP testing

- The SP test is a reliable method to depict creep behaviour of alloys. SP creep curves are similar to conventional uniaxial creep curves. A correlation between the SP load and creep stress has been demonstrated.

- The SP test leads to differentiation of creep curves and stress rupture properties for BM, weld and HAZ's.

- The SP results clearly indicate that the weld metal is weaker than both the BM and SE and the CG HAZ zone behaves similarly to the WM. The FG HAZ is shown to be the weakest component of the weldment.

- In terms of stress rupture behaviour there appears to be straightforward correlation between uniaxial and SP creep as regards WM tests (default ductility factor from the CoP, $K_{SP} = 1$). However, $K_{SP} \neq 1$ for BM & SE creep results due to larger ductility.

Thermal Fatigue Studies for Nuclear Piping Components

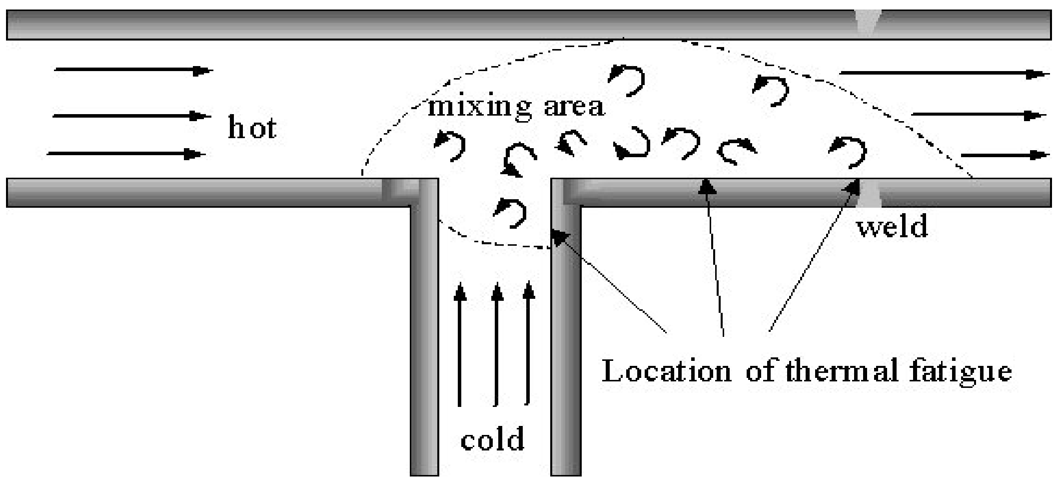
Problem Description

Thermal fatigue due to stratification and mixing of hot and cold water continues to be of importance for the life assessment of the current NPP piping systems.

A specific TF phenomenon, known as ‘mixing tee scenario”, results from turbulent mixing of two fluids at different temperatures.

Here TF damage is influenced by:

- Flow characteristics
- Material properties
- Pipe geometry



Issue: Development of improved practical methods for predicting thermal fatigue damage

(E. Paffumi)

Problem studies

- Thermal striping fatigue damage due to incomplete mixing of fluid streams at different temperature has the potential to occur in a number of areas if sufficiently good heat transfer exists between fluid and component.
- In GEN IV: potential degradation mechanisms for components in liquid-metal cooled fast breeder reactor (LMFBR) and gas cooled fast reactors (GFR).
- Due to large temperature differences between liquid metal emerging from the core and breeder sub-assemblies, thermal striping can arise in LMFBR, in the structures located above the core.
- Other areas of potential occurrence include piping systems in pressurized (PWR) and boiling water (BWR) reactors:

  - at the Residual Heat Removal System (RHRS) of the Civaux NPP Unit-1 1998;

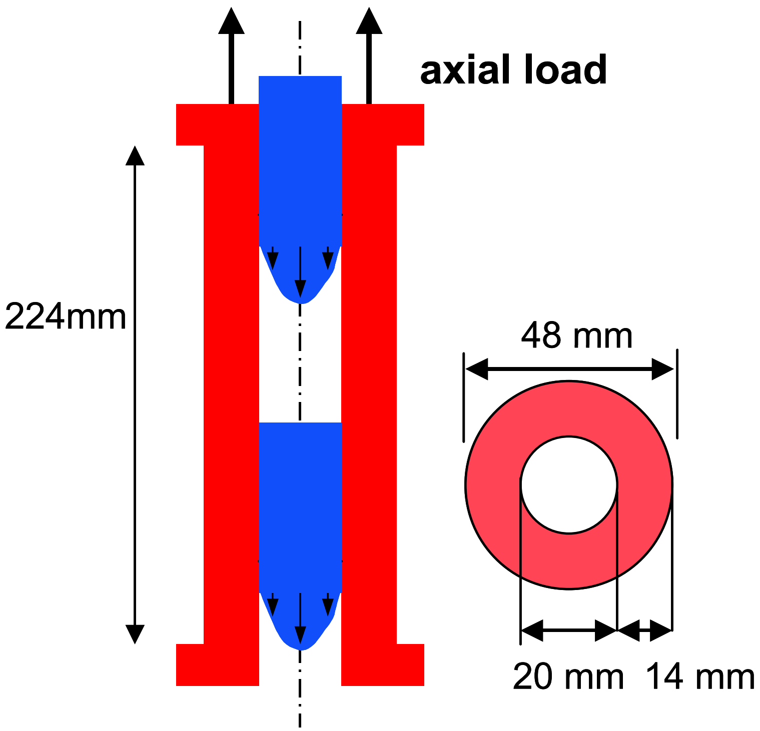
  - at the excessive extraction piping at the Genkai Nuclear Power Station Unit-2 2007

This work is being performed within the framework of NULIFE Thermal Fatigue Network of Excellence

NULIFE

Test Material and Specimen Geometry

Thermal shock experiments on cylindrical specimens from 316L stainless steel



External diameter: 48 mm
Internal diameter: 20 mm

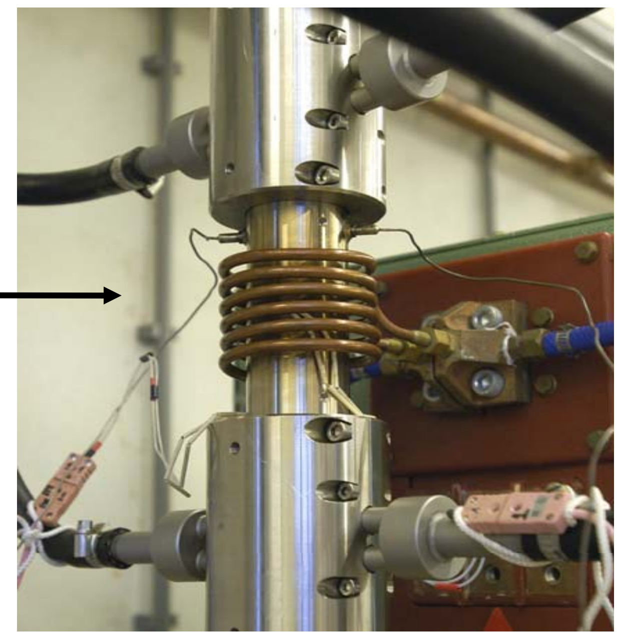
Length: 224 mm
Wall thickness: 14 mm

Experimental set-up

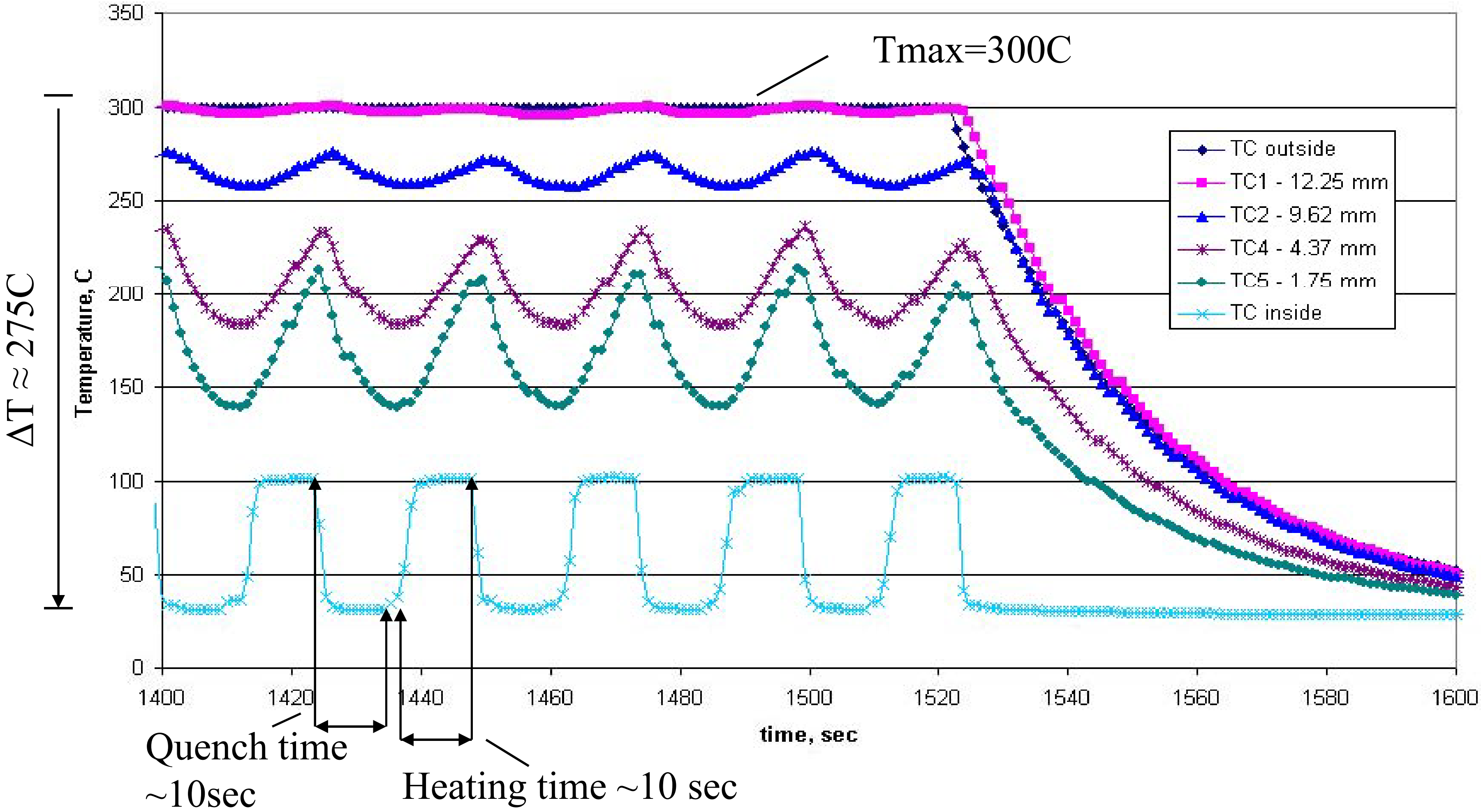
The specimen is heated by electromagnetic induction from outside and quenched internally with room temperature water.

Water quenching lines

Axial loading train



Experimentally imposed thermal cycle



Experimental techniques for fatigue damage detection by test interruption at pre-selected numbers of cycles

- **Crack initiation:**

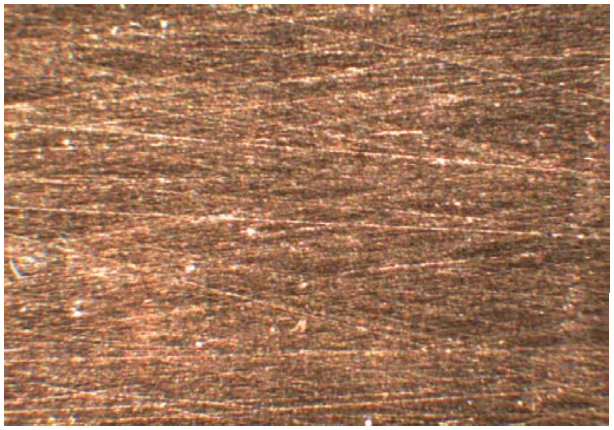
- Cellulose acetate replica technique for the detection of surface cracking (crazing cracks)
- Radiography with panoramic view to provide complementary information about crack distribution revealed by replica technique

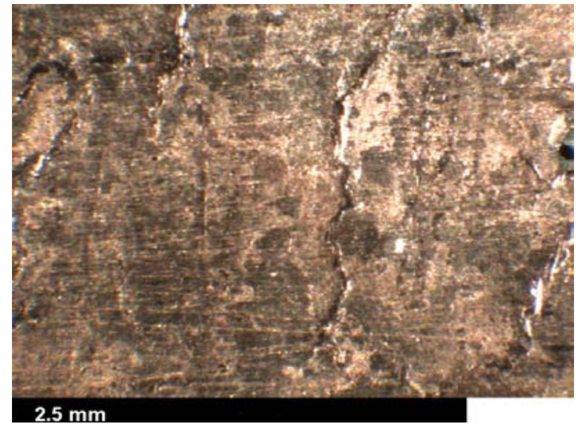
- **Crack growth:**

- Ultrasound time of flight diffraction technique (TOFD) for the through-wall crack characterization - location, length and depth of cracks

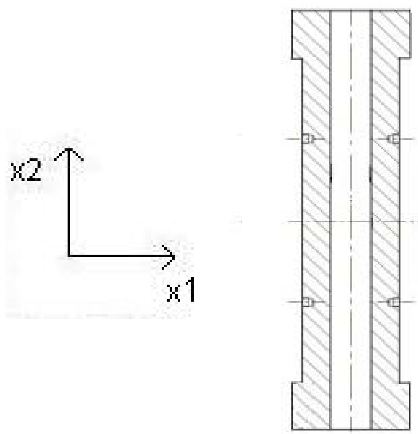
Replica technique

Examples of cracks detected by replica – Test TF1 –T_{max}=300ºC





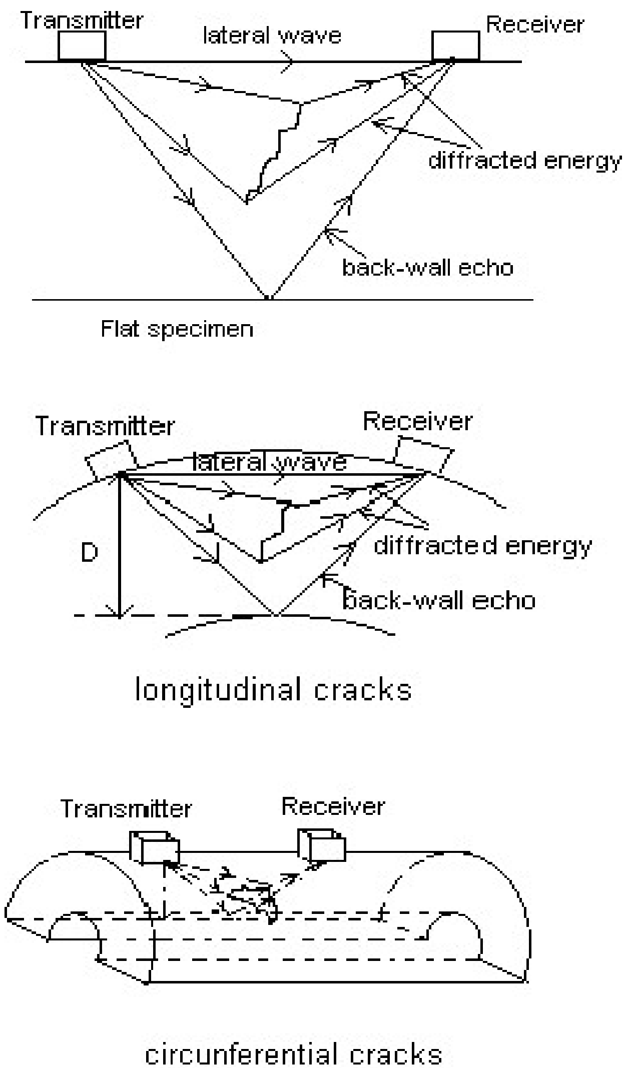
Inner surface before testing

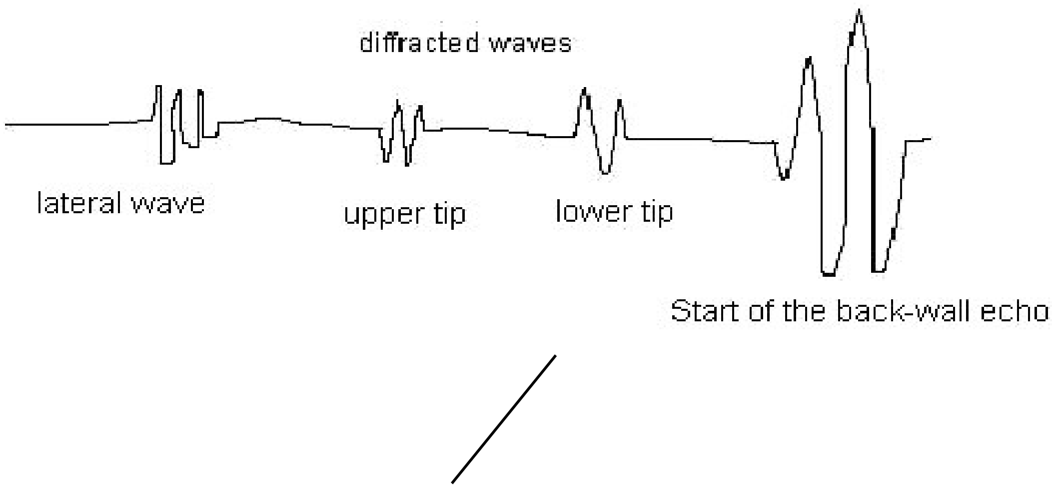


Inner surface after 55600 quenching cycles 25-300ºC

Time quenching: 5sec Time heating: 40 sec

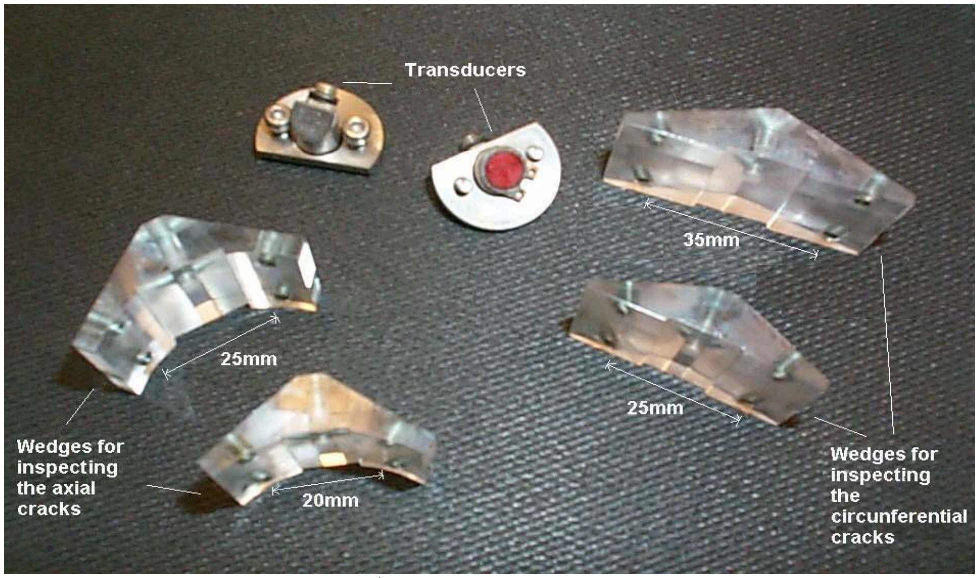
Time of flight diffraction technique (TOFD)





Typical pattern of pulses from an internal flaw

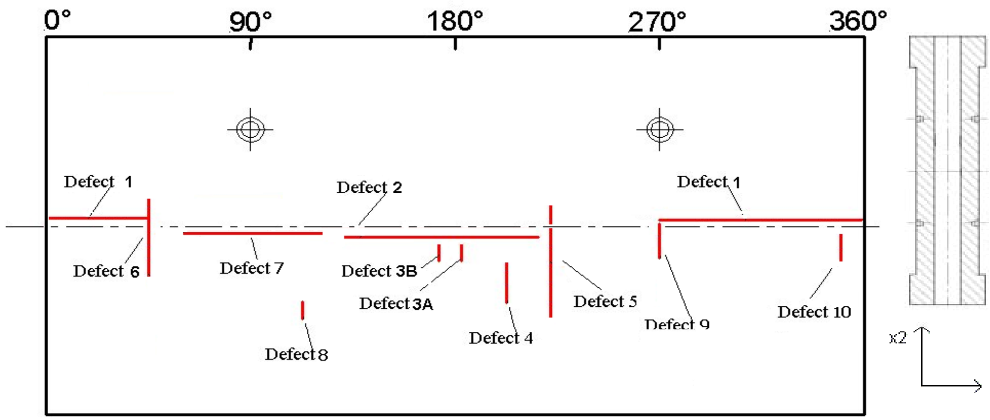
Time of flight diffraction technique (TOFD)



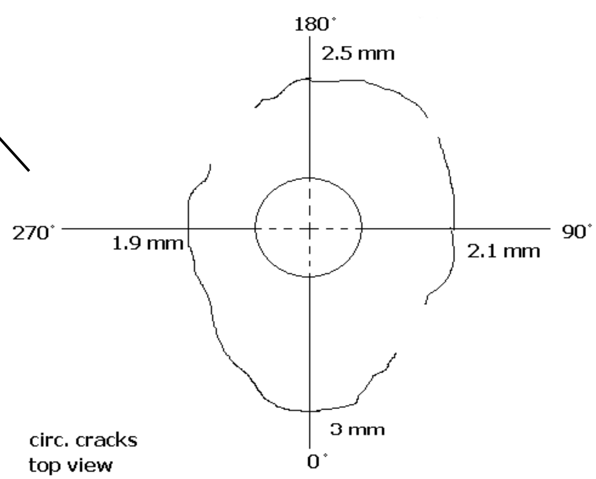
Transducers and wedges used during the inspections

Transducers on a wedge used for inspecting axial cracks

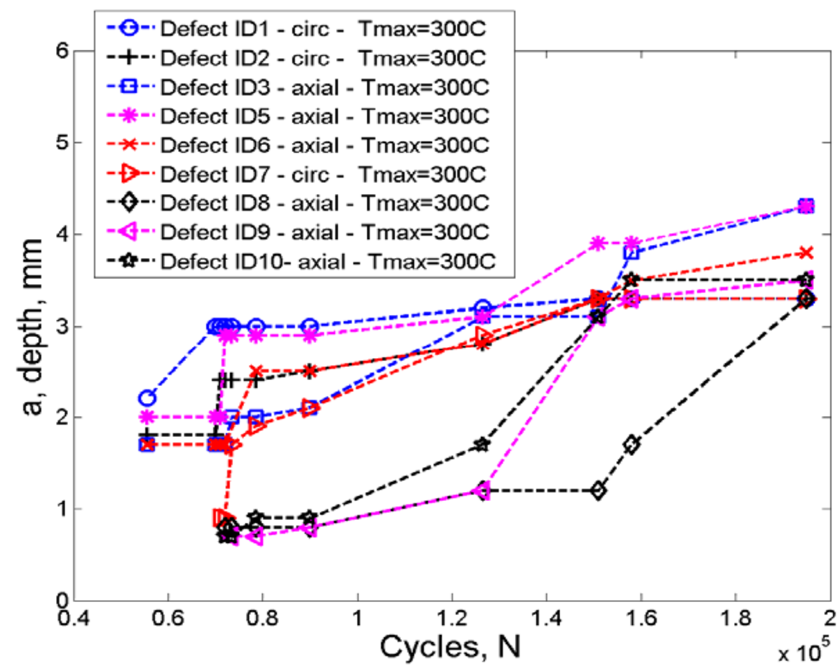
Example TOFD results (Tmax=300ºC)



90000 N



Crack depth versus number of quenching cycles for the defects of the specimen TF1

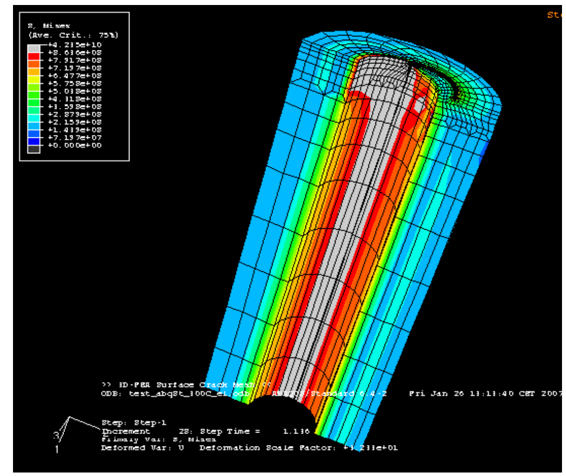


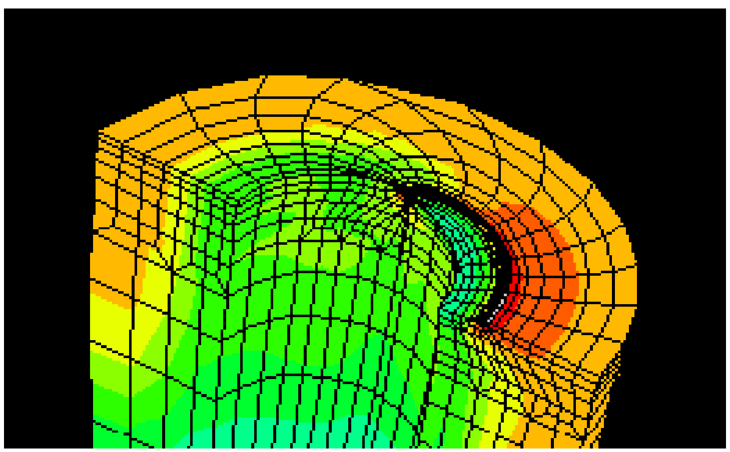
FE simulation of semi-elliptical cracks under thermal fatigue loading

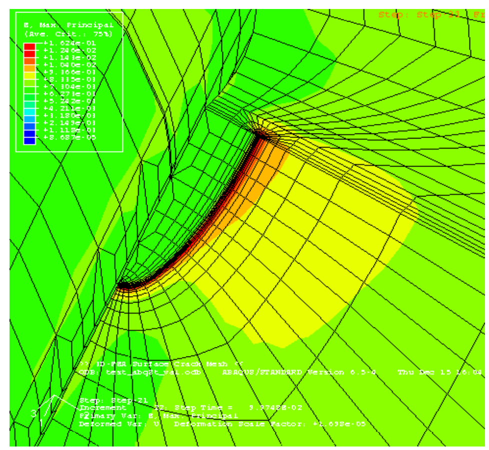
Combination of detailed 2-D and 3-D simulations and experimental data to assess experimental findings

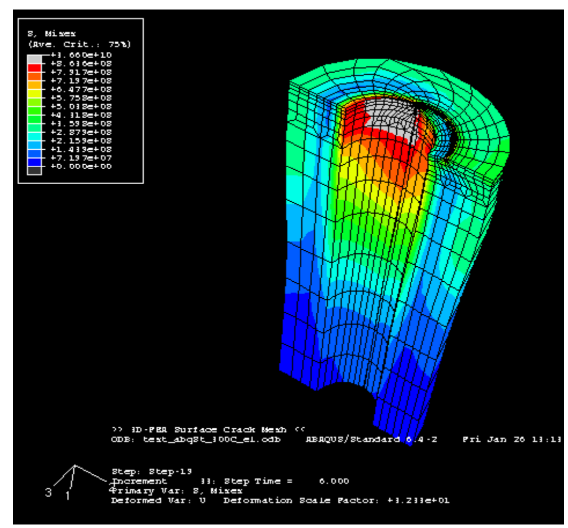
Cracked body analysis based on

- Short (< 1 mm) crack model
- Elastic-plastic long crack fracture mechanics model









Conclusions and further work

- A series of thermal fatigue tests has been carried out on tubular specimens in 316L(N) steel, with test durations up to 200000 cycles. A customized TOFD measurement system allows assessment of the distribution and depth of thermal fatigue cracks.

- Combining FE-generated thermal stress fields with simplified models for semi-elliptical cracks provides a conservative description of the crack growth behaviour.

- The results highlight the importance of well-documented experimental benchmarks for thermal fatigue assessment methods, such as the proposed European procedure being developed in the NULIFE network. This can be used to demonstrate the damage tolerance of existing and future reactor designs.

Strain-Controlled Thermo-Mechanical Fatigue:

Research into Best Practices

Thermo-Mechanical Fatigue (TMF):

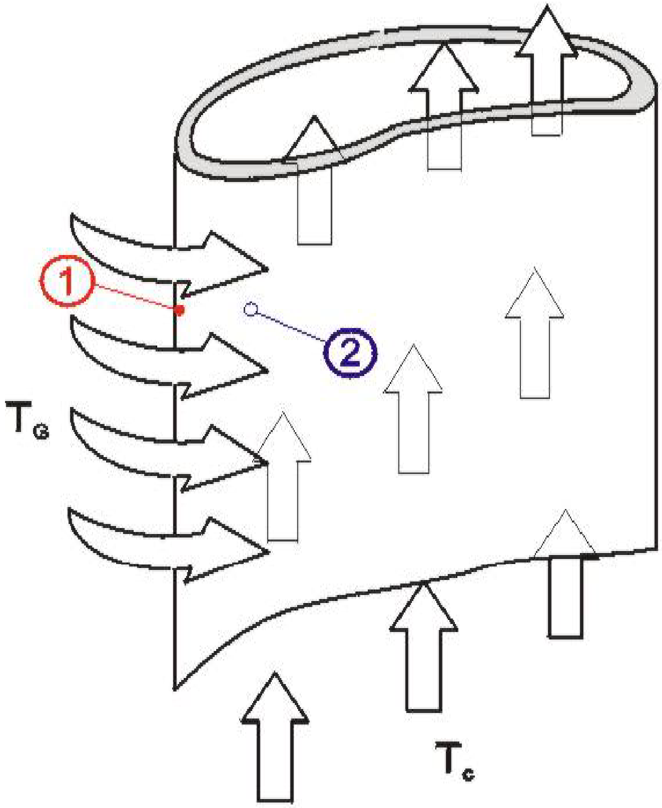
Thermal fatigue of components results from **internal thermal constraints** associated with cyclic temperature gradients.

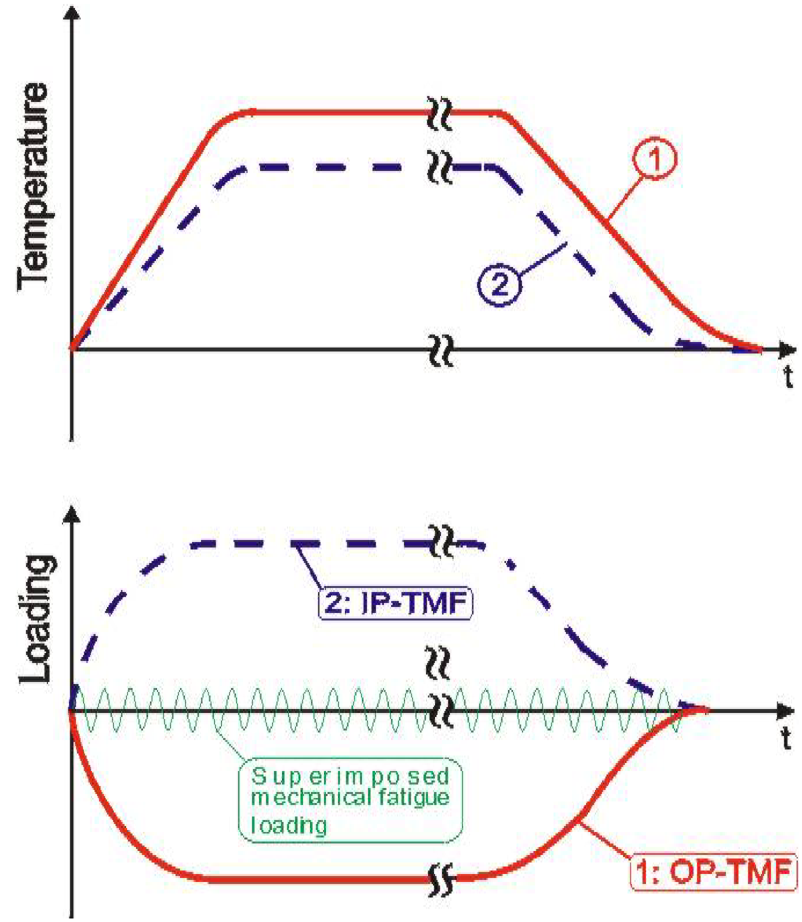
Alternatively, thermal constraints of a representative volume element can be simulated by *uniform temperature and mechanical strain* fields cyclically imposed on a specimen with uniform gauge section.

⇒ strain-controlled **thermo-mechanical fatigue** test

Design & residual life analysis of safety critical components exposed simultaneously to thermal & mechanical loads (gas turbines, aero engines, automotive & process industries)

Mechanical loads due to thermal constraints



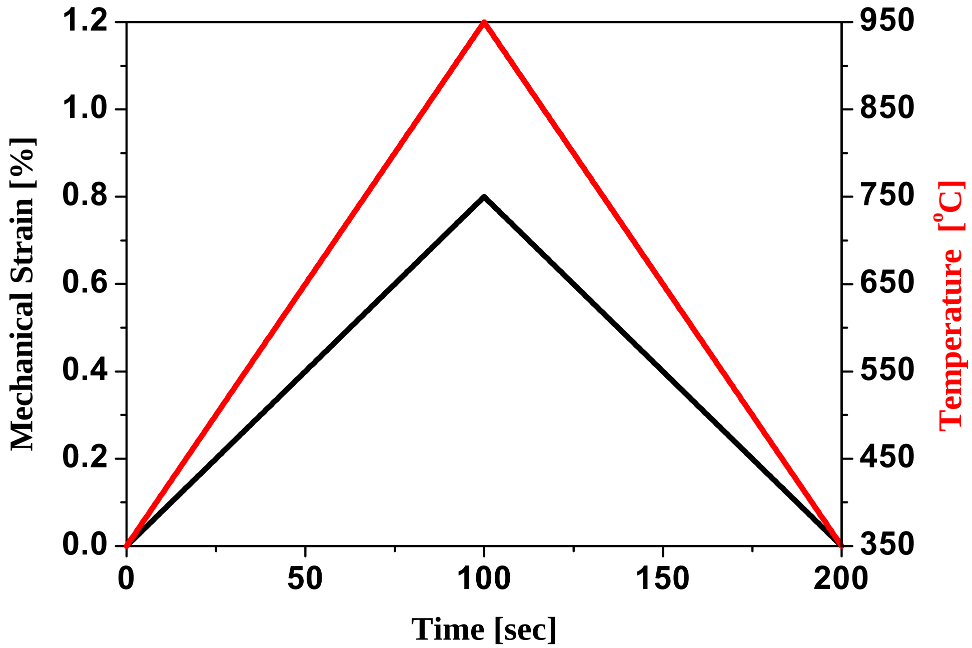


temperature: $T = T_0 + \delta T\, \mathrm{F}(\omega t)$, *mech. strain:* $\varepsilon_m = \varepsilon_{m,0} + \delta\varepsilon\, \mathrm{F}(\omega t - \varphi)$

phase angle φ , $\mathrm{F}(x) = \mathrm{F}(x + 2\pi)$, $\forall\, x$

Triangular TMF waveforms (in strain control without dwell):

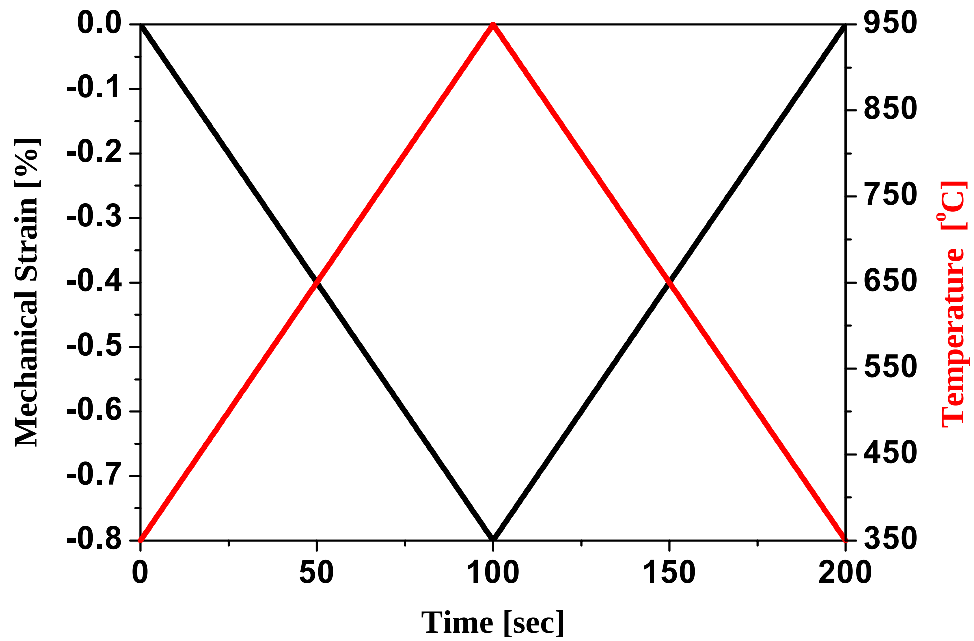
in phase $\varphi = 0$, $R_\varepsilon = 0$



mech. strain ratio:

$R_\varepsilon = \varepsilon_{m,min}/\varepsilon_{m,max}$

out-of-phase $\varphi = 180°$, $R_\varepsilon = -\infty$

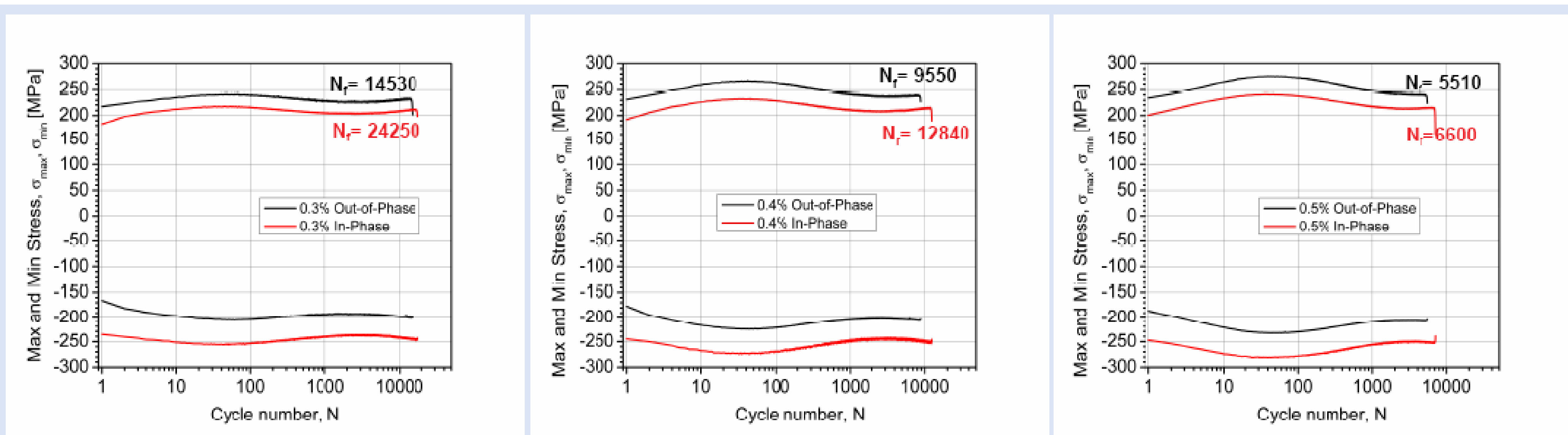


M. Ramesh, PSI: TMF of TP 347

TP (Code)	C	Si	Mn	P	S	Cr	Mo	Ni	Co	Cu	N	Nb	Ti
304L (H)	0.024	0.35	1.49	0.026	0.005	17.9	0.247	10.00	0.088	0.305	0.059	0.001	0.001
316L (B)	0.021	0.26	1.69	0.033	0.003	17.5	2.15	11.14	0.093	0.273	0.0601	0.012	0.003
321 (R)	0.073	0.20	1.87	0.021	0.004	18.0	0.334	10.20	0.055	0.182	0.0098	0.001	0.517
347 (K)	0.058	0.20	1.72	0.025	0.006	17.4	0.441	10.40	0.077	0.268	0.045	0.571	0.001

Out-of-Phase vs. In-Phase TMF

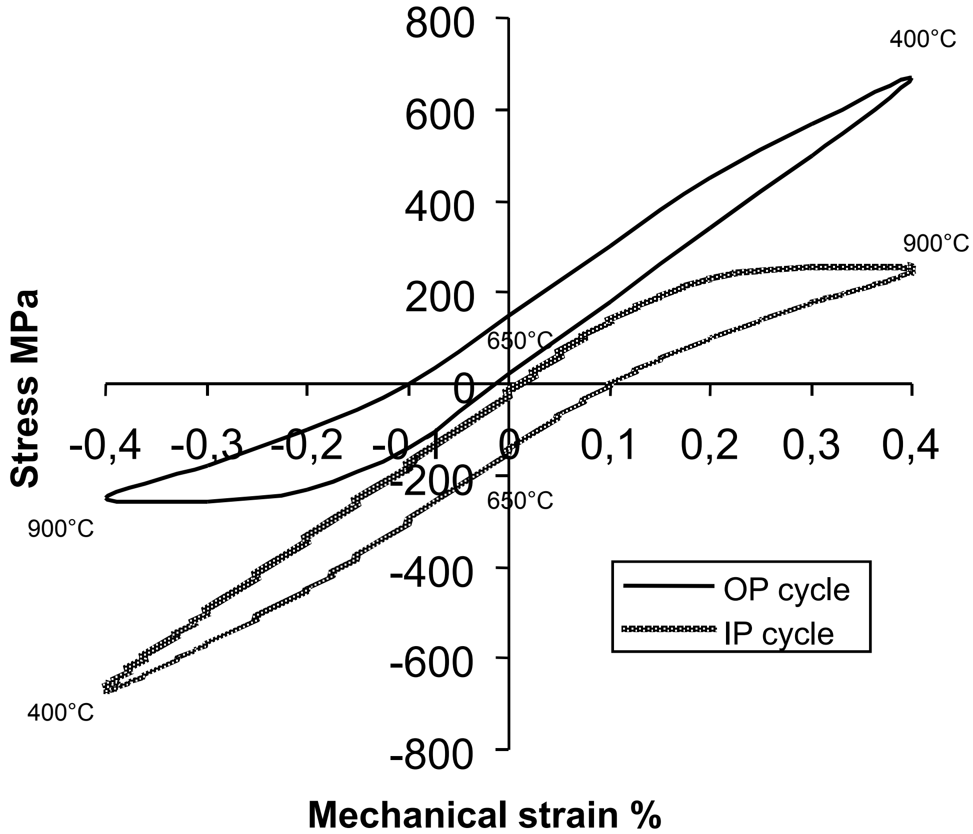
Material TP 347, T = 100 – 340°C, ν = 0.03Hz, ε_m = 0.3, 0.4, 0.5%



OP cycles result in shorter TMF lives

Mechanical Response

Hysteresis loops:



Out-of-phase

In-phase

$$\varepsilon_m = \varepsilon_{el} + \varepsilon_{in}$$

Effect on Number of TMF Cycles to Failure

TMF testing issues

- Load train alignment
- Precision extensometry

LCF | Thermal fatigue | Load-contr. TMF | Strain-contr. TMF

Dynamic temperature measurement & control

Therm. – mech. load phasing

- Pre-cycling, start-up procedures
- Thermal strain compensation

Time based: $\varepsilon_{tot}(t) = \varepsilon_m(t) + \varepsilon_{th}(t)$

Temperature based: $\varepsilon_{tot}(t, T) = \varepsilon_m(t) + \varepsilon_{th}(T)$

TMF-Standard Project GRD2-2000-30014
Thermo-mechanical fatigue – the route to standardisation
(funded within the GROWTH Programme of the EC)

Objectives: to establish a TMF testing platform in Europe
to issue a *validated* code-of-practice
to disseminate and exploit the results

Duration: 48 months (2001 – 2005)
Budget: ~ 1.800.000 Euro (~ 1.080.000 EC contr.)
Consortium: 10 principal contractors
9 assistant contractors, 1 external participant
No. of tests: ~ 400 incl. 120 validation tests

WP 3: Pre-normative research on TMF procedures & tolerances

Task 1: Pre-cycling, start, interrupt, restart procedures

Task 2: Dynamic *T* measurement and control

Task 3: Thermal strain comp., deviations from nominal *T*

Task 4: *T* gradient effects in 3 different sample geometries (solid cylindrical, hollow cyl., solid rectangular)

Task 5: Deviations from nominal phase angle

Challenges in TMF temperature measurement and control:

- Temperature rates (heating & cooling) up to 50°C/s to be controlled with sufficient accuracy (± 5°C or 1% of ΔT)
  -> specimen design, heating system, fast temperature control system
- *T* measurement must not affect crack initiation and TMF life
  -> Avoid any microstructural damage, e.g. by thermocouple attachment
- Heating method (induction, radiation, ...) must not influence temperature readout in the absence of thermal equilibrium
  -> no cold spot at thermocouple, nor direct over-heating of thermocouple
- Long-term stability of dynamic temperature fields must be ensured
  -> no drift of *T* profile

Issues affecting dynamic *T* measurement and control:

- heating method
- specimen geometry
- type of *T* measurement device: thermocouple (TC), pyrometer
- method of TC attachment

These issues are interrelated and cannot be optimized independently !

Heating methods

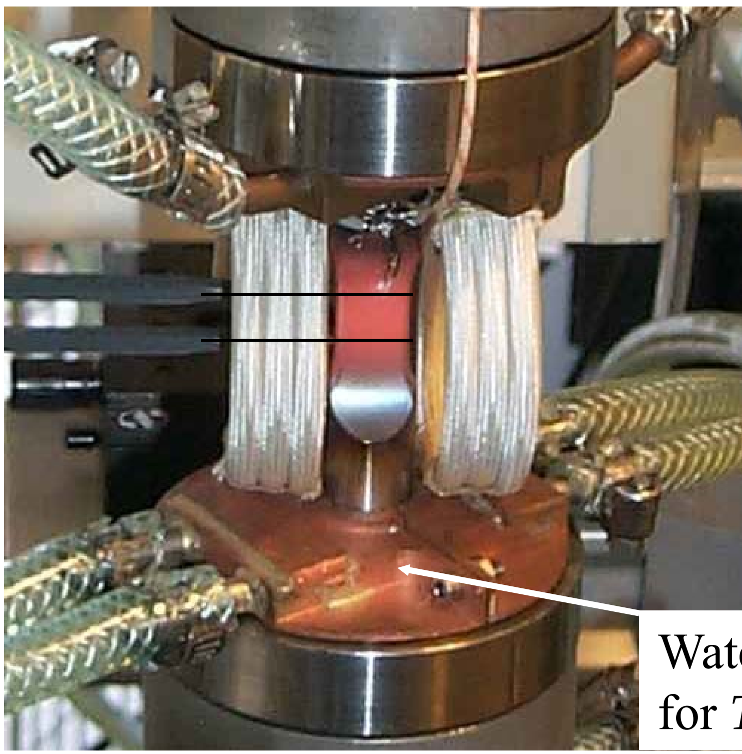
Resistance furnace: too slow

- *Direct ohmic heating:* practicable for miniaturized, e.g. thin-walled tubular, specimens only
- *radiation (bulb) furnace:* appropriate, if
  - excessive radial temperature gradients are avoided
  - issue of reflectivity differences: specimen – TC is addressed
- *Induction heating:* most commonly used in TMF
  - skin effect to be kept sufficiently low
  - "cold spot" to be avoided by proper TC type and attachment method

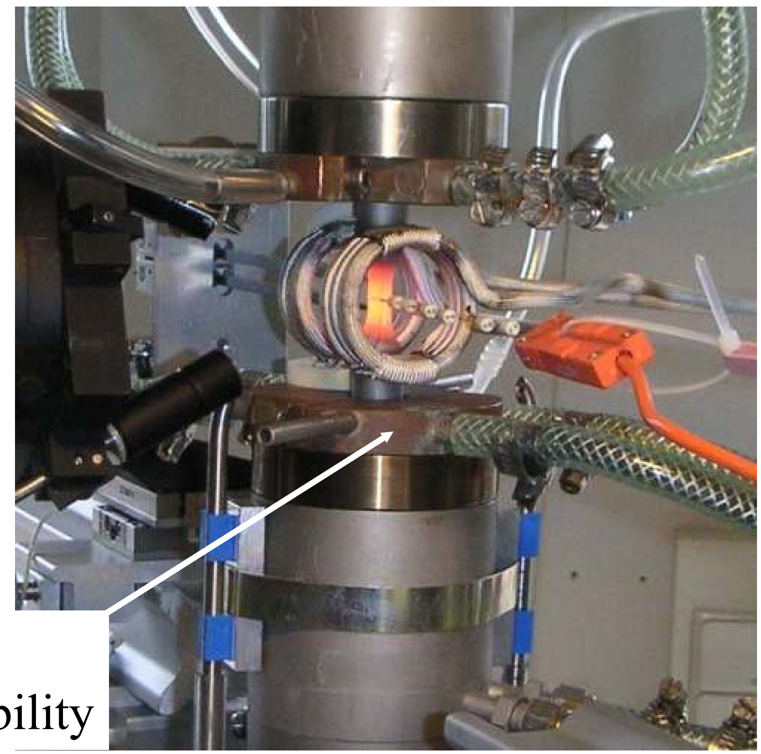
Induction heating

- heating of subsurface layer: Skin effect $d = (4\pi\sigma\mu\nu)^{-1/2}$
- ensure very good thermal contact of TC (not heated directly)

TC spot-welded outside GL



Ribbon TC wrapped around centre

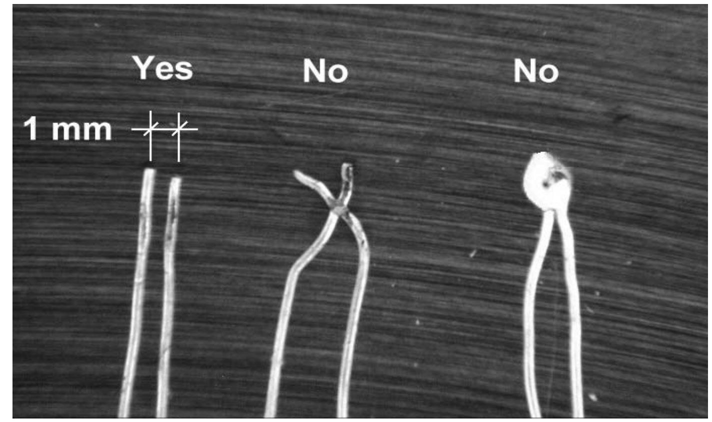


Water cooling
for *T* profile stability

Thermocouples (TC)

T control by **spot-welded** TC **outside** the gauge length:

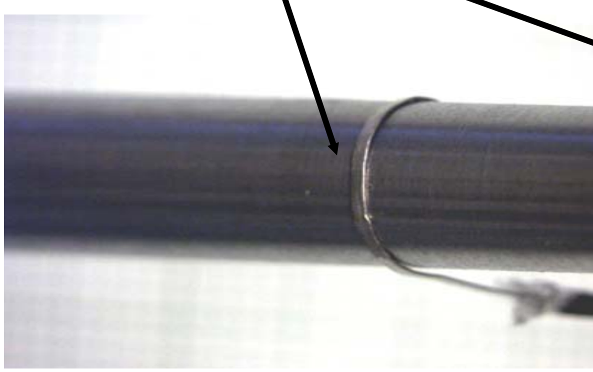
- \+ Stable and good thermal contact
- – Reference temperature in the centre of the GL
- – “Cold spot” can be an issue



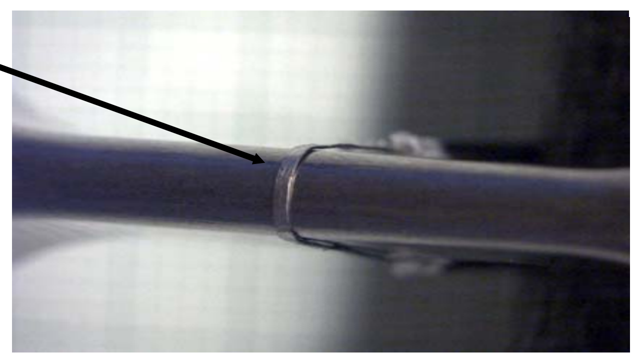
T control by **ribbon** TC **in** the gauge length:

- \+ Direct measurement in GL w/o damage
- \+ No “cold spot“, as contacting length sufficiently long
- – Thermal contact to specimen surface may be insufficient and prone to variation by oxidation, surface rippling, micro-cracking…

T measurement point



Cylindrical specimen



Flat specimen

TMF-Standard summary

TMF-Standard CoP on strain-controlled TMF

- contains the "essence" of 4 years work of 20 European laboratories
- has been validated by ~ 120 TMF tests (OOP and IP)
- comprises a lot of informative material and practical recommendations
- has contributed to improving & harmonizing TMF practices
- provides **underpinning base to the new ISO 12111 standard**:
  - ✓ sign convention of phase angle
  - ✓ recommended specimen geometries
  - ✓ max. allowable temperature deviation
  - ✓ max. allowable temperature gradients (axial, radial, circumferential)
  - ✓ max. thermal hysteresis
  - ✓ Young's modulus verification

Remark

While consensus has been reached as to the max. allowable temperature deviations and temperature gradients, there is still no generally accepted, optimized method of dynamic temperature measurement & control:

- awareness of critical issues remains important;
- use of complementary methods is necessary to ensure accuracy of technique

However, what does „accuracy“ mean in the absence of appropriate methods of

Dynamic Temperature Calibration

? ? ?

In situ calibration of *dynamic* temperature measurement and control using a phase transformation:

Issues to be addressed:

- Temperature ramping at ± 1 … 10º C/s
- No thermal equilibrium between TC & specimen: means of TC attachment
- Little direct heating of TC, mainly indirectly via specimen surface

Will the TC read the right temperature ?

Possibilities for establishing reference temperatures:

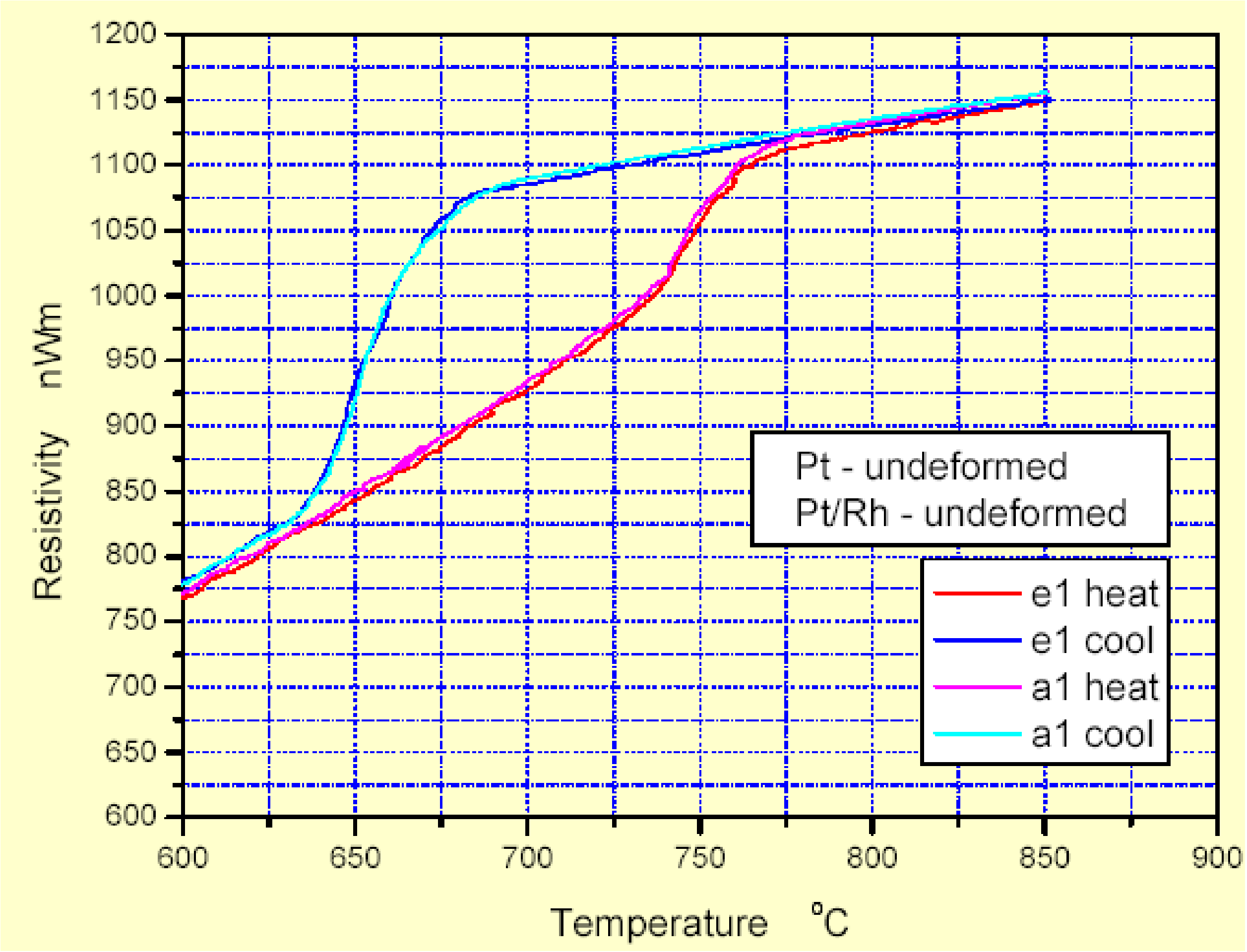
1. NPL: displacive phase transformation of eutectoid steel
2. JRC-IE: solid-liquid phase transition of Al-Cu

NPL method for dynamic T calibration

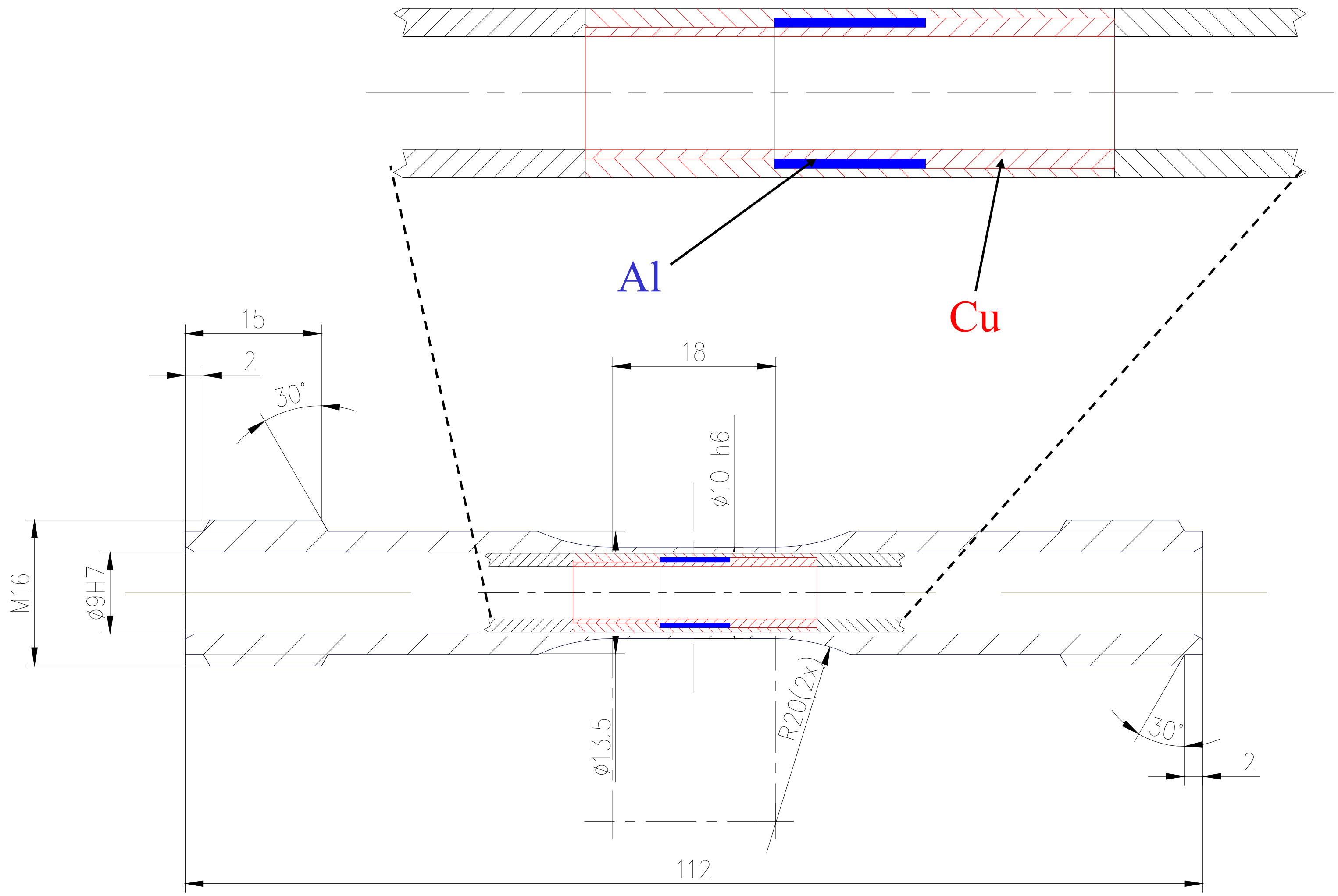
Eutectoid steel (0.8% C) – phase change on heating at ~740°C

Pearlitic (bcc Fe-C / Fe3C) -> austenitic (fcc Fe-C)

Phase change on cooling ~ 680°C (under-cooling > 50°C)

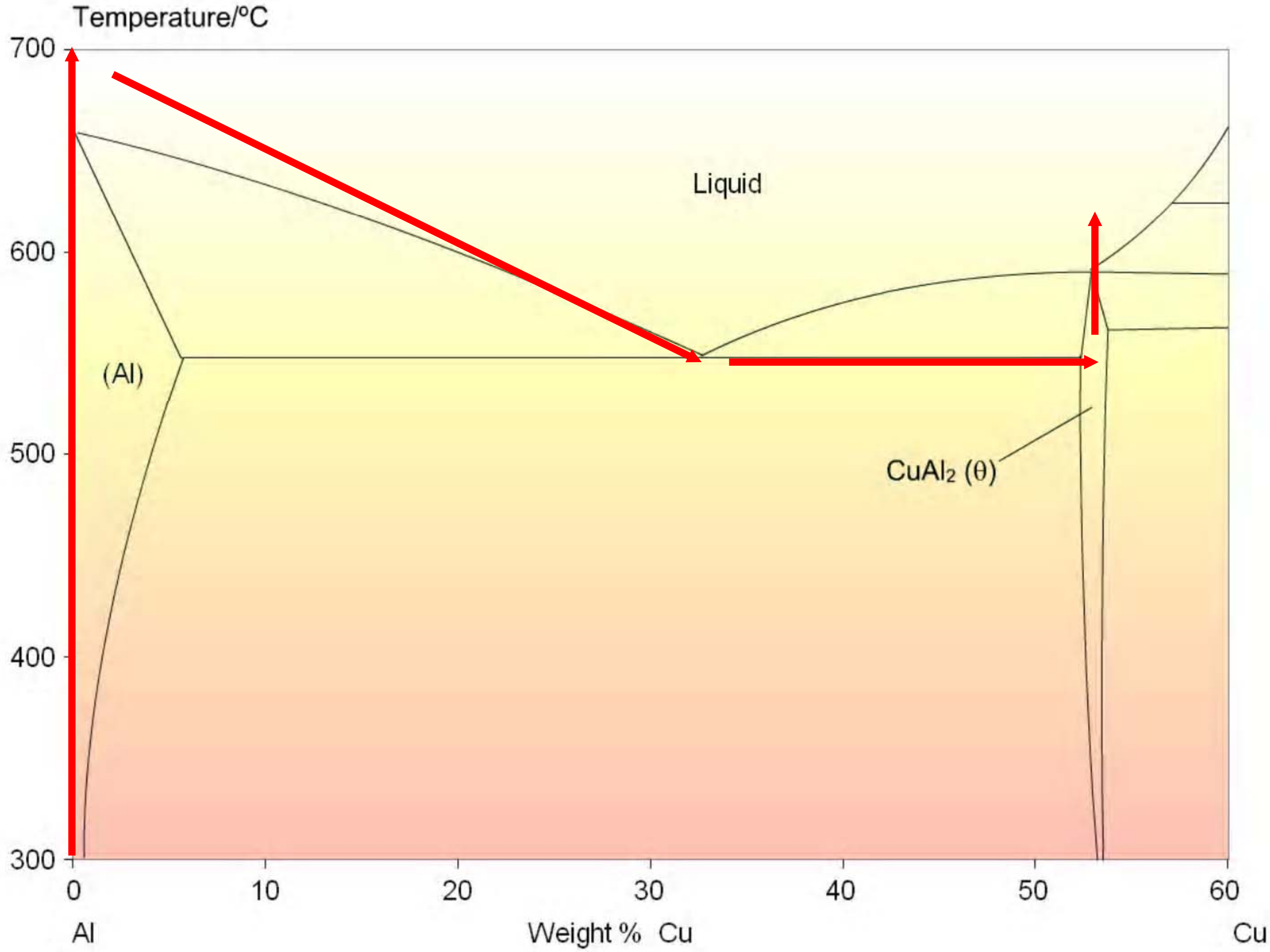


Al ring soldered into Cu case inserted into tubular specimen



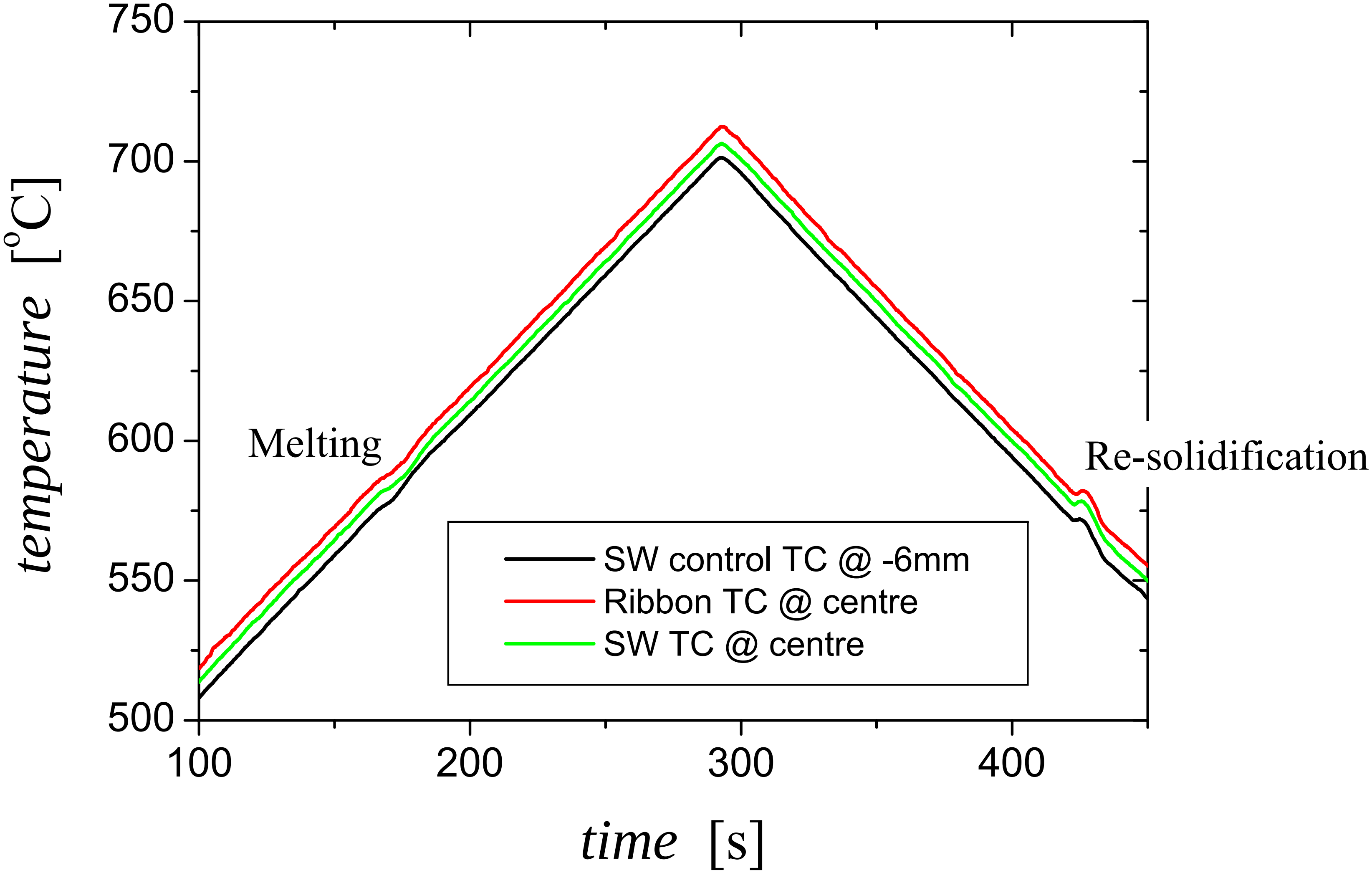
Al-Cu system

eutectic point	@	548°C, 33 wt% Cu
θ phase ($CuAl_2$)	@	591°C, 53 wt% Cu



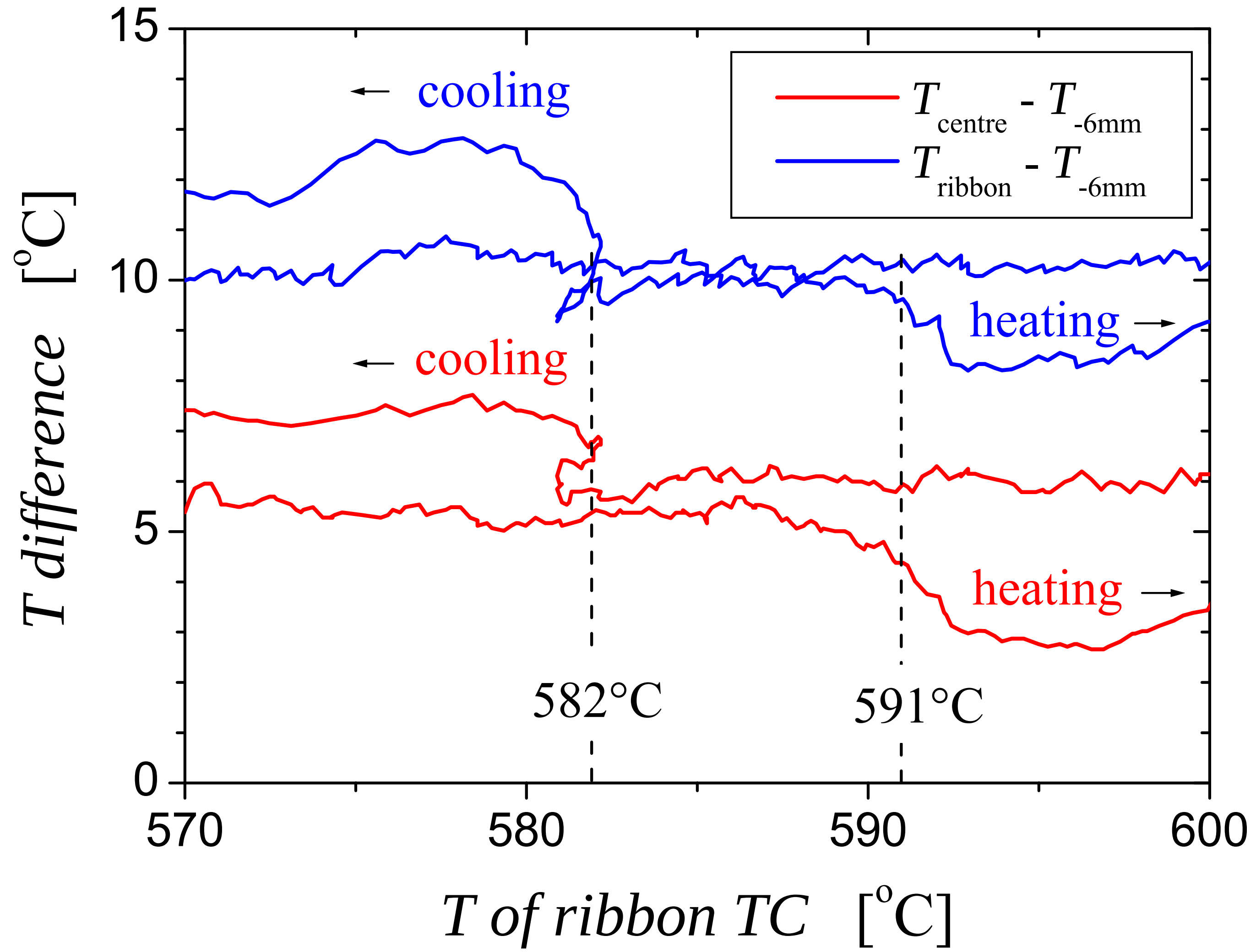
Temperature cycling over θ phase transition

Ribbon TC firmly attached to pre-oxidised surface



Temperature differences

Ribbon TC firmly attached to pre-oxidised surface



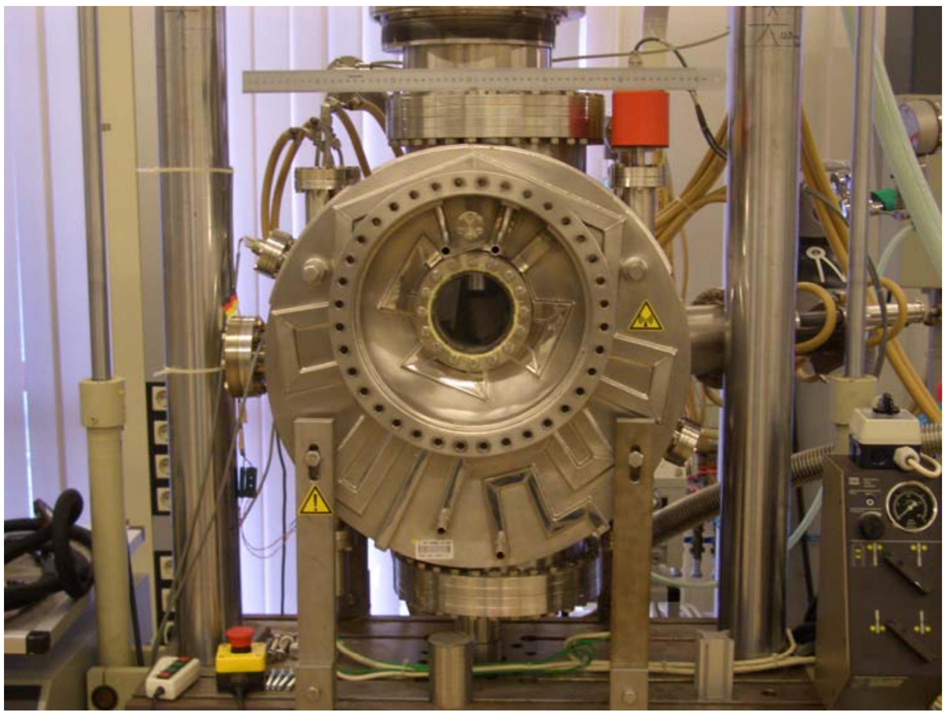
Advantages of Al-Cu system

- No need to alloy intermetallic θ phase with 53 wt% Cu
- Detectable θ phase develops already during 1st cycle
- Sharp solid-liquid phase transition upon heating
- Less hysteresis (under-cooling < 10ºC)
- > 50 cycles possible
- Not ferromagnetic

  (cf. induction heating circuit affected by permeability)

Outlook: Environmental TMF

TMF performance in
- vacuum/inert atmosphere
- corrosive environments
- hydrogen



Setups for low pressure (left) and high pressure (right) environmental LCF/TMF tests