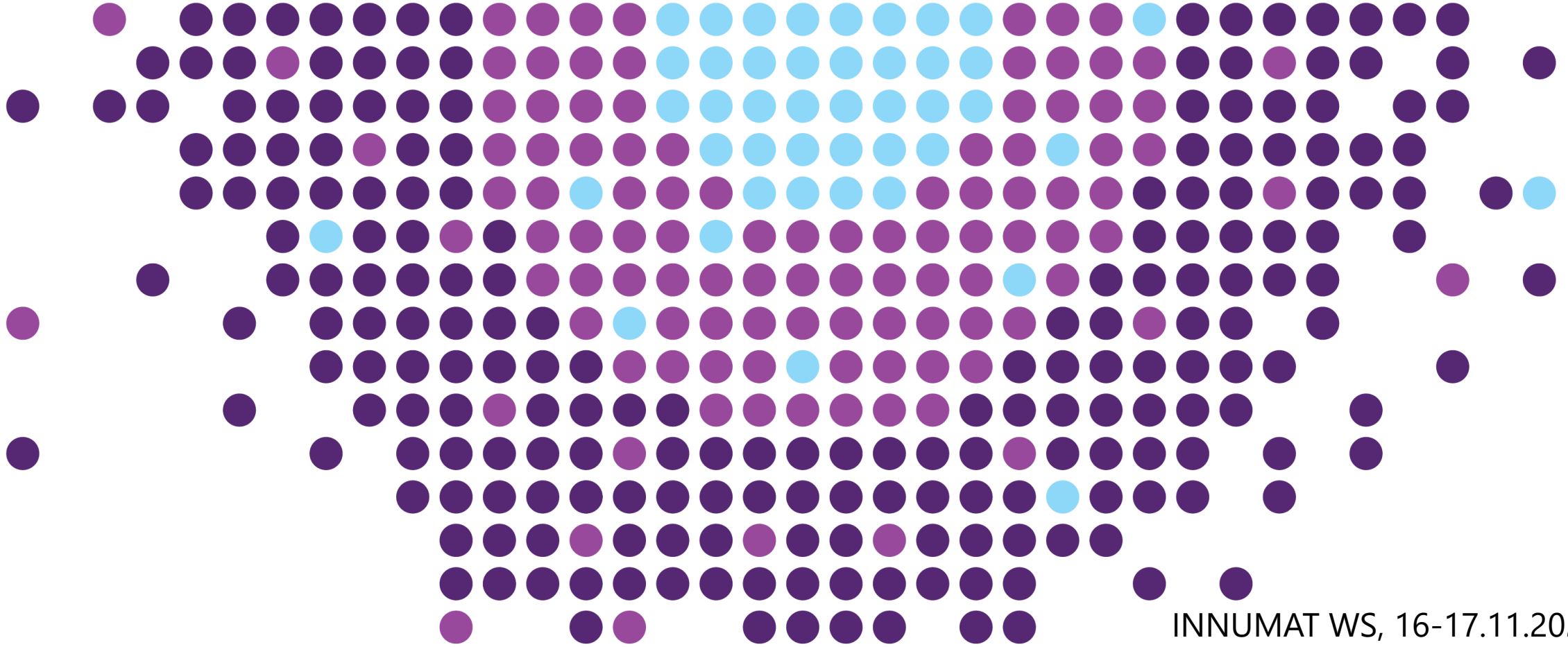


INNUMAT WS, 16-17.11.2023

sck cen
Belgian Nuclear Research Centre

# INTERLABORATORY STUDY on SP and SPC using the Reference Materials BCR-661 (tensile) and BCR-425 (creep)

# ILS: background (ECCC, WS64, INNUMAT, EUROfusion)

- SCK CEN participation in three European projects and support to EuroFusion (ongoing, all incorporating SP or SPC in some form)
- EU-PATRICIA – 1515Ti cladding tubes – creep and corrosion interaction, miniature testing → determination of effective corrosion depth (with miniature tests**, incl. SP**)
- EU-INNUMAT – support for 'codification' of project materials and test types
  - Qualification of miniature tests (SP in particular)
  - **Follow the European Creep Collaborative Committee (ECCC) activities → SPC in particular**
- EU-NUCOBAM – challenging data sets, e.g. SPC on semi-brittle materials (SP and SPC)
- EUROfusion
  - Reduced activation 9%CrWVTa steel EUROFER 97, supporting SP and SPC data is being generated (JRC open access)

→ **INITIATE a multi-project-multi-network-open for external labs- round robin**

→ Increase the available material data base → verification / qualification of the SP and SPC performance in **support of the revision of the SP/SPC standard (EN 10371) and the French nuclear code RCC-MRx**

# What is the ECCC? – a long standing collaboration on creep data

The European Creep Collaborative Committee (ECCC) was founded in 1991 to co-ordinate the generation, collation and analysis of creep data for metals commonly used for high temperature plant within Europe. (~30 member organisations).

i) co-ordinate the generation of creep data throughout Europe (national creep groups)

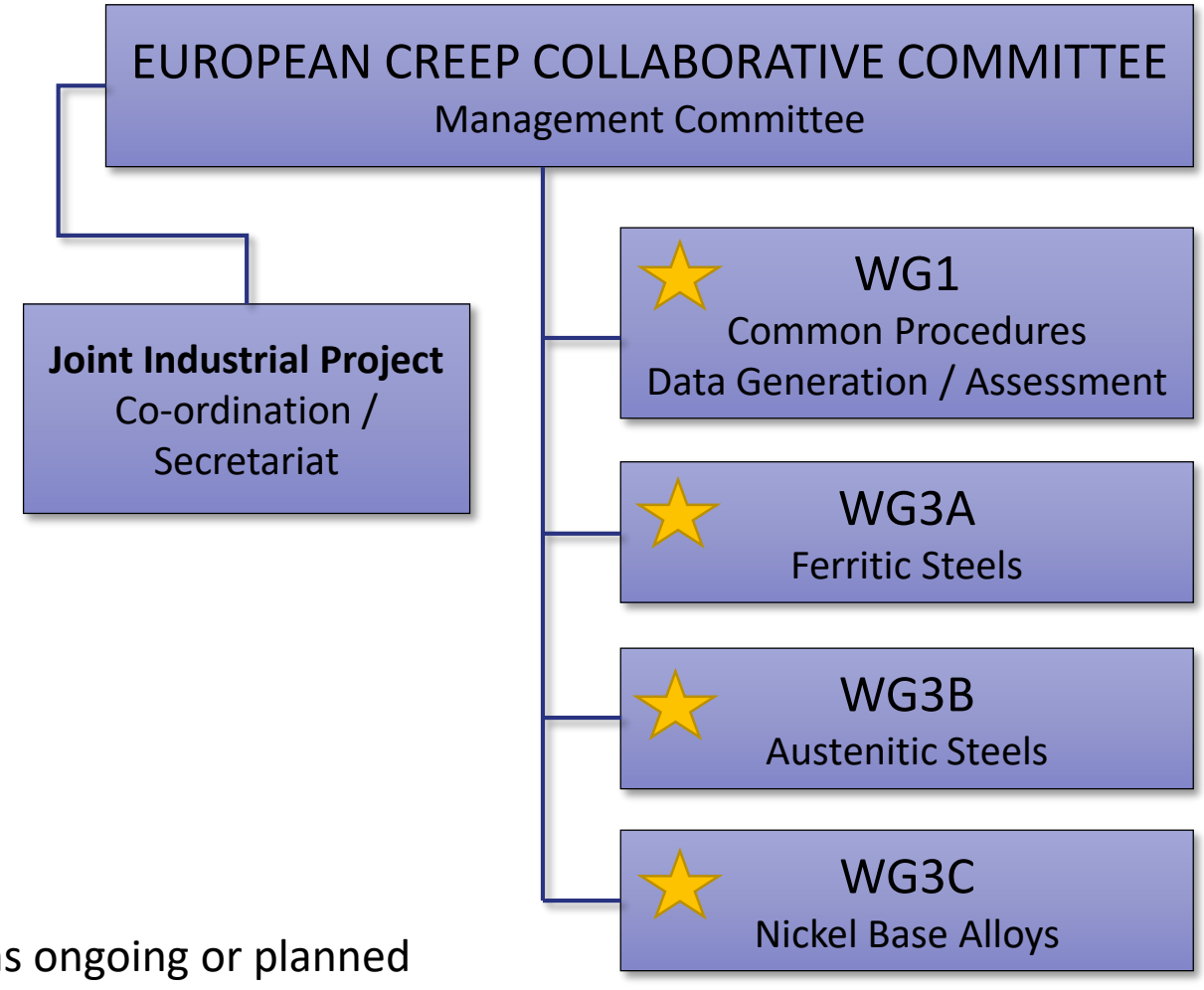
ii) interact with, and supply information to the formal European Standards organisations

iii) mutually exchange technical information on material developments

iv) develop rules for data generation, collation/exchange and assessment.

Close links with ECISS/ CEN. Formal/informal links with e.g. NIMS, JSMS, ASME …

Website : https://www.eccc-creep.com/ : datasheets, guidelines, webinars/conferences etc. mostly free of charge.

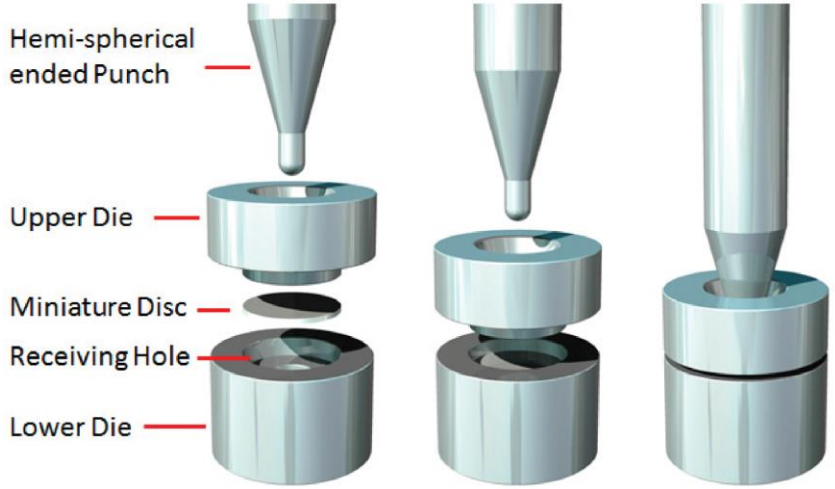


# Objectives of this presentation

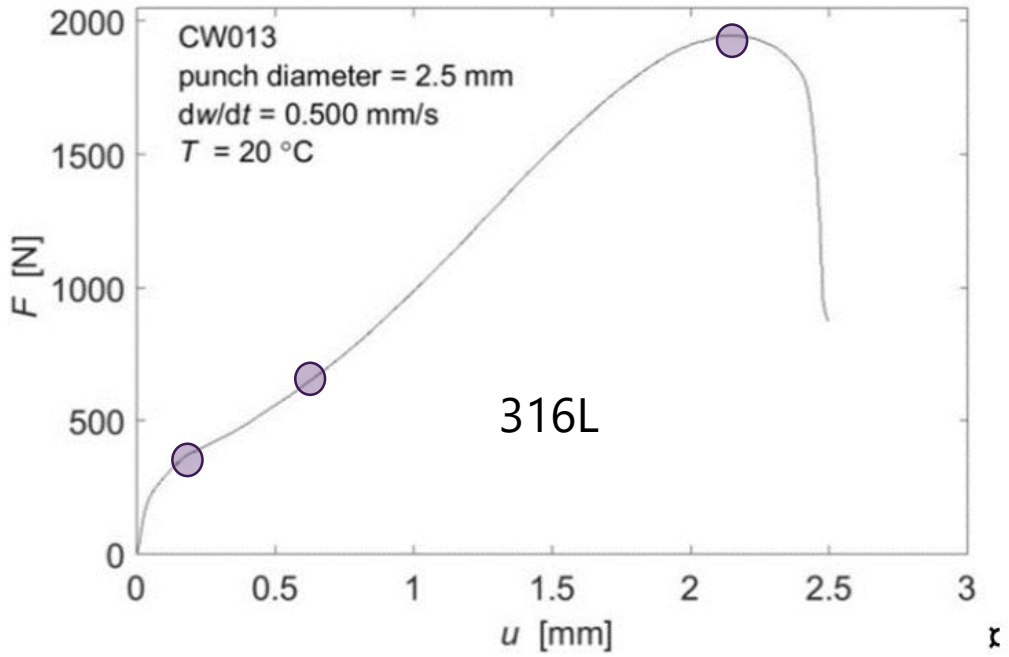
## Incentives

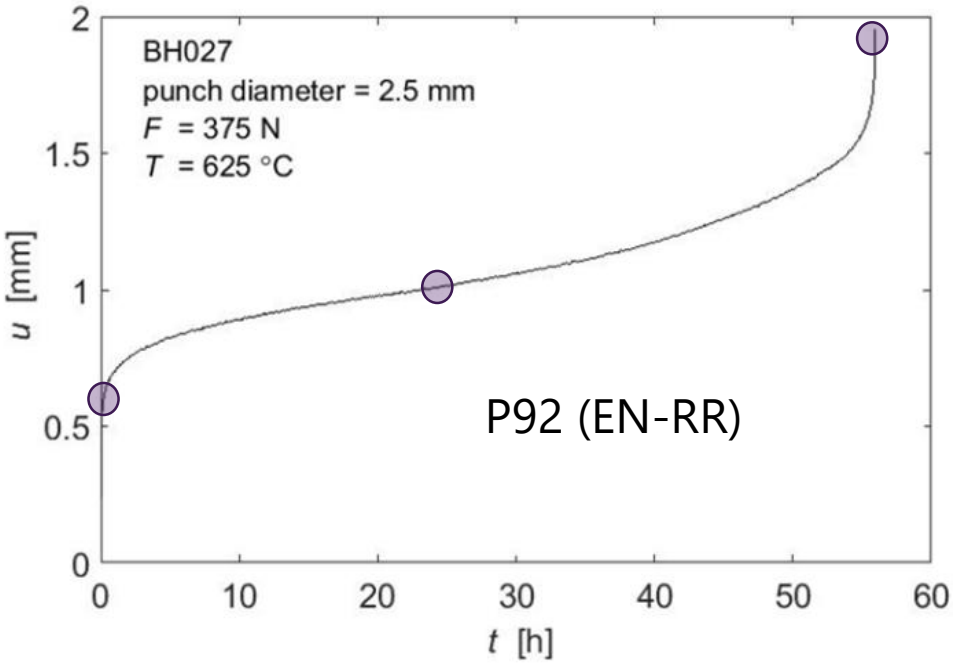
- Present the multi-project-multi-network collaboration to set up for supporting and further qualifying miniature techniques, in particular the small punch test
- Describe the objectives, challenges and sought outcomes.
- Discussion on initially planned actions and further invite laboratories within INNUMAT as well as external organizations with SP & SPC capabilities.

# Small Punch (SP) ‘tensile’ / Small Punch Creep (SPC)




| Material property | Equation |
|---|---|
| Tensile strength estimate $R_m$ | $R_m = \beta_1 \cdot \frac{F_{max}}{h_0 \cdot v_{max}}$ |
| Proof stress estimate $R_p$ | $R_p = \beta_2 \cdot \frac{F_e}{{h_0}^2}$ |
| Creep strength estimate $R_{t/T}$ | $\sigma_{eq}(F, u_{min}) = \frac{F}{\beta_3 \cdot u_{min}^{\beta_4}}$ |



# Techniques to be included in the RR / ILS

- Tensile properties by Small Punch SP (target for the campaign)
  - REFERENCE UNIAXIAL TENSILE testing not needed for BCR reference material
  - Miniature tensile (potential add-on)
  - Others (?)
- Creep properties by Small Punch Creep SPC:
  - UNIAXIAL CREEP (not needed for BCR reference material) – reassessing time to failure may be warranted
  - IC – Impression creep (creep strain rates only, potential add-on)
  - Miniature creep tensile (potential add-on)

# ILS - Conducting an Inter-laboratory Study to Determine the Precision of a Test Method

- Projects / participation from: INNUMAT + ECCC + WS64 + Kagoshima Univ. & CRIEPI (JAPAN)
- Two main Objectives:
  - ➢ Gather a substantial publicly available data base of SP and SPC data for the reference materials
  - ➢ Show that the reference materials used for tensile and creep testing (uniaxial) are also suitable for SP and SPC
  - Use BCR-661 as SP reference material (RT tensile)
    - Also study the miniature SP (∅3 mm, 250 μm)?
  - Use BCR-425 as SPC reference material (UA Creep, 600°C, 160 MPa) → shorten the test time (increase temperature?) and define the sought 'constant' test parameters
- Use ILS standard ASTM E691-21
- Recommendations for future revision(s) of the SPC and SP standards: EN 10371-21 (SP and SPC) and ASTM E3205 - 20 (SP)



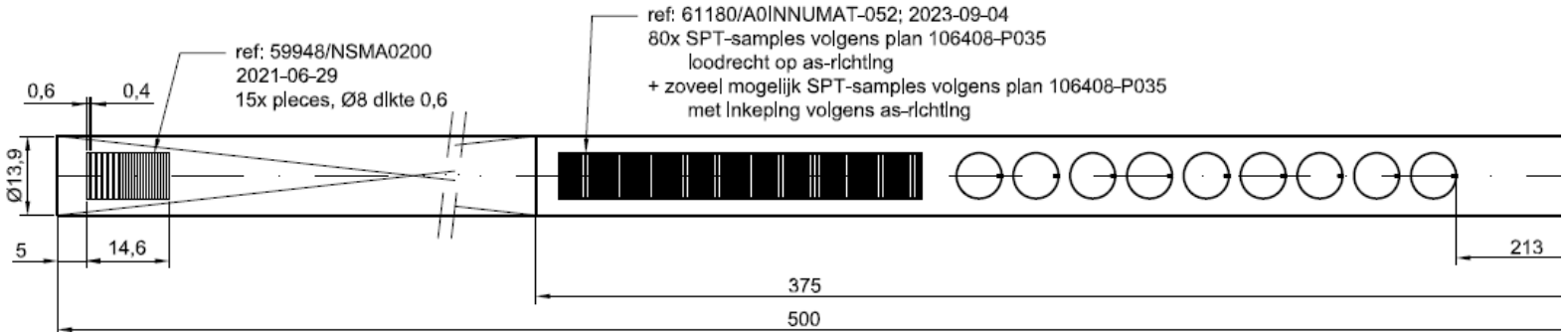
Designation: E691 – 21

# Differences and challenges in comparisons between techniques

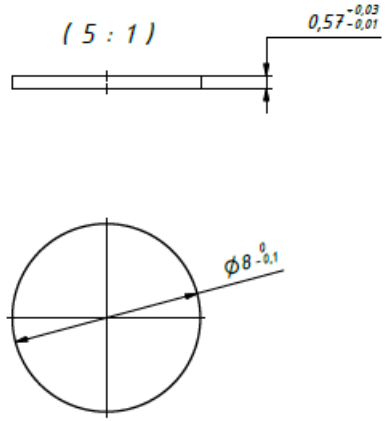
- **Tensile properties:**
  - SP – uncertainty of force to stress conversion (friction, specific force to stress conversion methods) → new force to stress equations? → combine SP and SPC methods (?)
  - Miniature tensile – size effect?

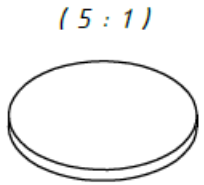
- **Creep properties:**
  - UNIAXIAL CREEP – assess historical data for time to failure, …?
  - SPC – need for different reference criteria, e.g. deflection or deflection rate at specified time test time, time to failure, time to minimum deflection rate, …
  - IC – Impression creep (creep strain rate only?) + time to impression depth?
  - Miniature creep tensile - direct comparison to BCR strains and strain rates

# SP samples BCR-661 @ SCK CEN

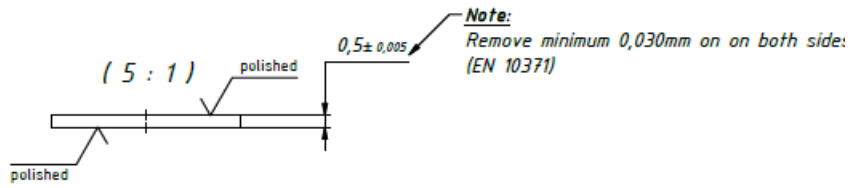


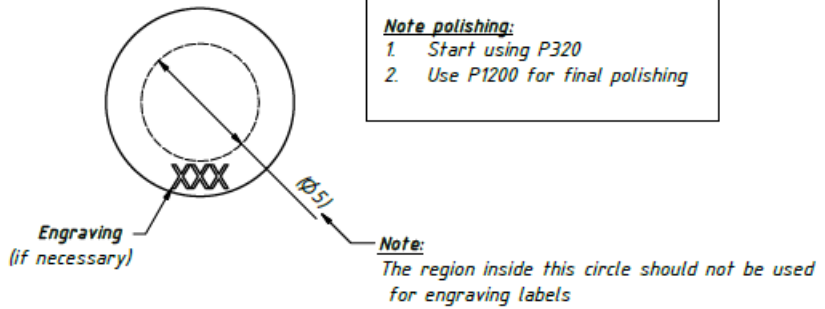

**WORK PREPARATION:** *EDM-cut*

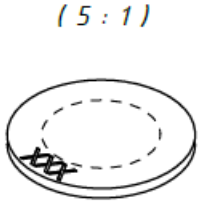



**FINISHING:** *Polished*



# BCR-661 Tensile test (RT) reference material

- The certified reference material BCR – 661 alloy is a Nimonic alloy for qualifying tensile testing according to **EN 10002-1**.
- BCR-661 grain size: GE= 8.5-9 ASTM → approx. grain size → d~ 16 to 19 microns → > 25 grains over the SP thickness, OK
- SCK CEN can currently supply 'blanks', i.e. EDM cut samples with a thickness of approximately 0.57 mm extracted from rod bars ∅ 14 mm, l=500 mm
- JRC , Joint Research Centre– Petten (NL), also has a rod of this material (the fabrication of BCR-425 priority)

**References:**

CERTIFIED REFERENCE MATERIAL BCR® – 661, https://crm.jrc.ec.europa.eu/p/40456/40485/By-analyte-group/Mechanical-properties/BCR-661B-NIMONIC-75-FOR-TENSILE-PROPERTIES/BCR-661B

## CERTIFICATE OF ANALYSIS

**BCR ® - 661**

| NICKEL BASE ALLOY | | | |
|---|---|---|---|
| | Ambient temperature tensile properties | | |
| | Certified value [2] | Uncertainty [3] | Unit |
| 0.2 % Proof stress ($R_{p0.2}$) [1] | 300 | 8 | MPa |
| 0.5 % Proof stress ($R_{p0.5}$) [1] | 318 | 7 | MPa |
| Tensile strength ($R_m$) [1] | 750 | 14 | MPa |
| Elongation at fracture (A) [1] | 40.9 | 0.9 | % |
| Reduction in area at fracture (Z) [1] | 60 | 4 | % |

1) As defined in EN 10002-1.

2) Certified values are values that fulfil the highest standards of accuracy. The given value(s) represent(s) the unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory and/or with a different method of determination. The certified value and its uncertainty are traceable to the International System of units (SI).

3) The uncertainty is the expanded uncertainty of the certified value with a coverage factor k = 2 corresponding to a level of confidence of about 95 % estimated in accordance with ISO/IEC Guide 98-3, Guide to the Expression of Uncertainty in Measurement (GUM:1995), ISO, 2008.

# Example of existing BCR-661 small punch 'tensile' data (SCK CEN, JRC open access project)

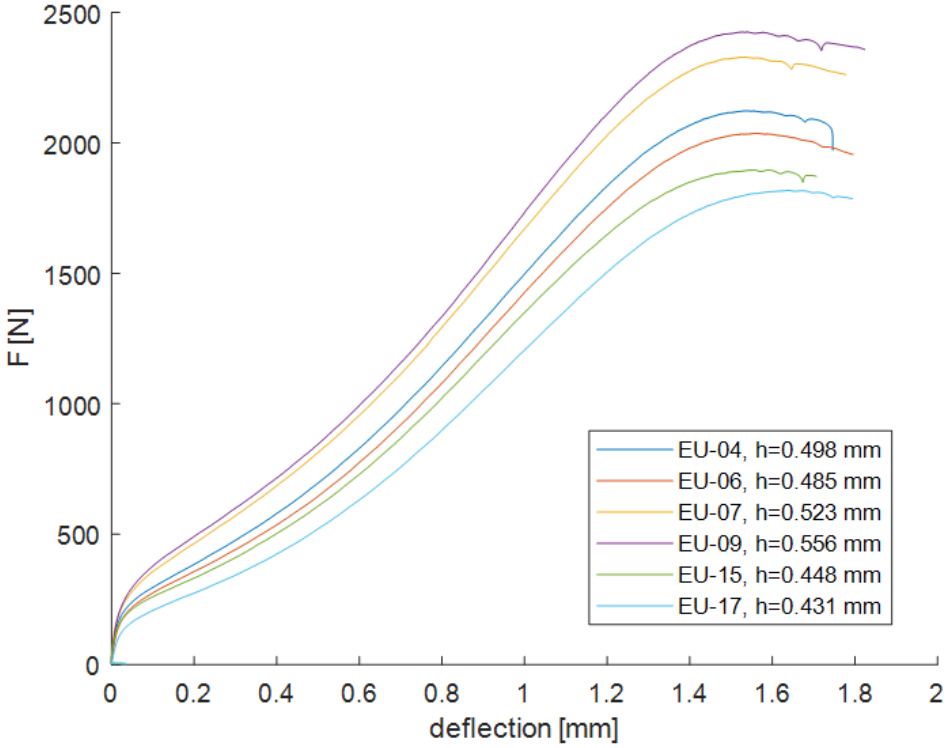
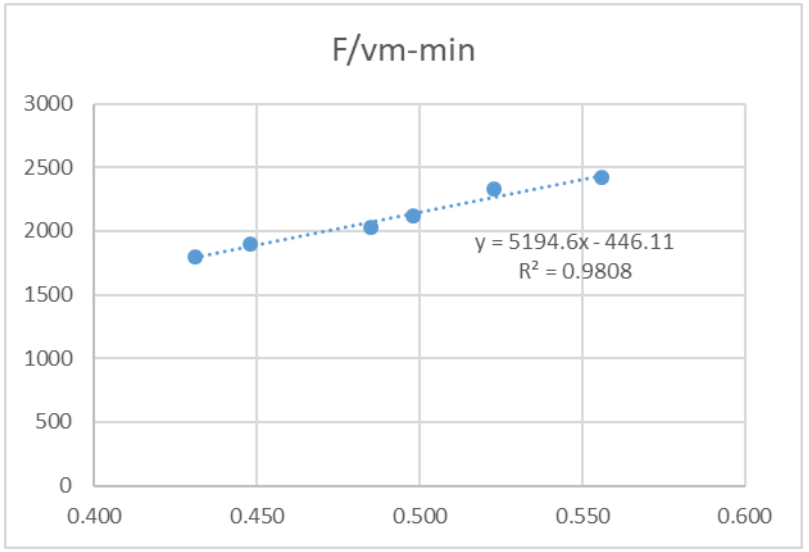
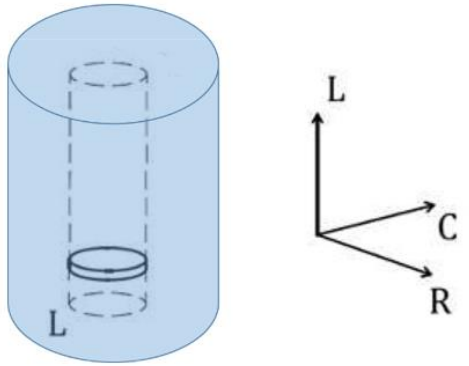



Figure 3. Force-Deflection (F-u) curves at room temperature for the different sample thicknesses




*Testing of reference material BCR-661 (Nimonic 75). Open Access to JRC Research Infrastructures; 2020-1-RD-EMMA-SMPA, SPT-INVEST – to be published Dec. 2023*

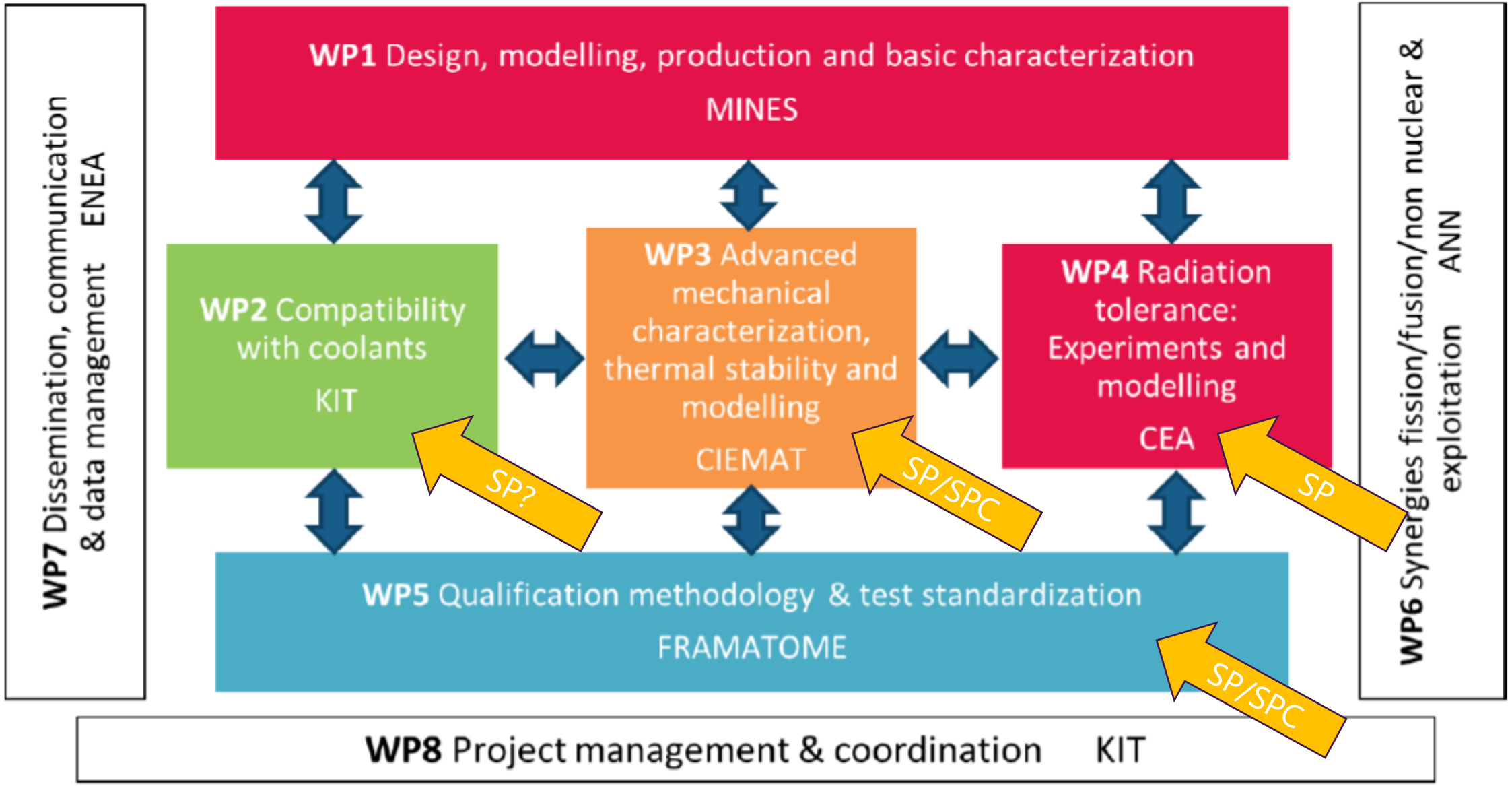
# BCR-425 creep reference material for SPC

- The certified reference material BCR – 425 alloy Nimonic 75 for creep testing has been selected for the round robin. - JRC , Joint Research Centre– Petten (NL), supplies samples extracted approximately 0.57 mm extracted from rod bars ∅ 14 mm, l=500 mm.
- BCR-425 grain size: GE= 8.0-9 ASTM → approx. grain size → d~ 16 to 23 microns → > 20 grains over the SP thickness, OK
- The reference unaixal creep tests using this material are carried out at the temperature $T_{test}$ =600±2°C and stress level $\sigma_{test}$=160 MPa, Prior to testing the sample should soak (age) in the test machine **at the test temperature for 20h**. The stress must be applied after this ageing treatment.
- **Certified properties:**
- **Creep Rate** at 400h: $7.2 \cdot 10^{-5}$ /h, extended uncertainly $5 \cdot 10^{-6}$ / h → need to find a new definition for SPC
- **Time to 2%** strain: 278h, extended uncertainly 16 h → need to find a new definition for SPC
- **Time to 4%** strain: 557h, extended uncertainly 30 h. → need to find a new definition for SPC

**References:**


1. CERTIFIED REFERENCE MATERIAL BCR® – 425, CERTIFICATE OF ANALYSIS: ISBN 92-826-1742-4, Luxembourg; https://crm.jrc.ec.europa.eu/p/40456/40485/By-analyte-group/Mechanical-properties/BCR-425-NIMONIC-75-FOR-CREEP-TESTING/BCR-425
2. D. Gould, M.S. Loveday: The certification of Nimonic 75 alloy as a creep reference material -CRM 425; Directorate General Science, Research and Development; EUR 13076, 1990

# Innovative structural materials for fission and fusion - INNUMAT



## WP1: Material procurement & harvesting (**WS64 R&D proposal**)

- Reference materials (WP2)
- New materials (WP3) + interactions with prior and ongoing material development projects
- In-service materials + damaged materials (WP4-5) + interactions with industry

## WP2: Reference

- Ref-SP
- Ref-SPC
- Round-Robin

## WP3: Ranking and Mat. development

- HEA
- AM (SLM)
- Other?
  e.g. alumina forming steels

## WP4: Damage & Interaction

- Aging
  e.g. aging factors
- Irradiation,
  e.g. negligible limits
- Damage interaction, e.g. Creep-Fatigue

## WP5: In Service & LTO

- Base materials
- Welds
- Claddings & surface treatments
- Materials
  'Health Clinique'

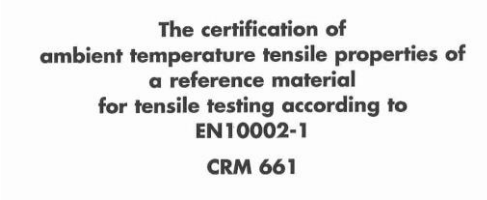
## WP6: Methodology

- FEA
- Low ductility materials
- Uniaxial estimates equivalent properties
- Support to standards & design
- Round-Robin

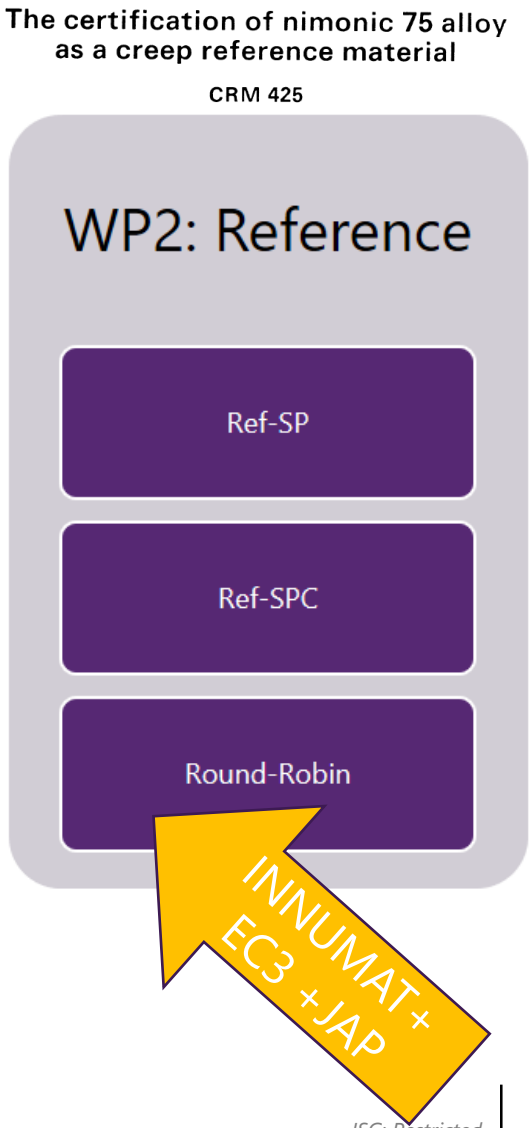
## WP7:Data base, Assessment & Dissemination

- Data Base: Mat DB or other (WP2-5)
- Combining all WP2-6 results to a 'code of practice' (CEN Technical Report through the TC?) on the of SP&SPC
- Open access reports and journal papers

# Reference MATERIALS (WP2)




- Introducing 'tensile' and creep 'primary' reference curves that can be used for test laboratory / equipment validation testing. The initial material choice for this task is the Nimonic 75 nickel based alloy used as a reference both for uniaxial testing CRM 661 in EN-10002-1 [REF], and BCR (CRM) 425 for creep testing in ISO 204 [REF].
- **Introduce 'secondary' reference materials (?)** for the most used classical materials and steels. e.g. copper, aluminum, low alloy steel, carbon-manganese steel, austenitic stainless steel, etc.
- Include an inter-laboratory round robin to verify that the acquired test curves have sufficiently low data scatter and that the ductility of the chosen reference material is not limiting the use as reference material for the SP.
- The public 'data set library', starting with RT SP curves to be extended by existing and new SP data as well as SPC data
- **MATDB !**



# Status of Labs (SP & SPC) – INNUMAT (8)

- CIEMAT (Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas, Spain)- joins SP+SPC.
- HZDR (Helmholtz Zentrum Dresden-Rossendorf, Germany) – joins SP+SPC
- JRC (Joint Research Centre, the Netherlands)- joins SP+SPC
- NCBJ (Narodowe Centrum Badan Jadrowych, Poland) - joins SP **(∅3 mm)**
- SCK CEN (Studiecentrum voor Kernenergie - Centre d'Étude de l'énergie Nucléaire, Belgium) – joins SP
- CCFE (UKAEA - United Kingdom Atomic Energy Authority, UK) - joins SP
- VTT (Teknologian tutkimuskeskus VTT oy, Finland) - joins SPT+SPC
- WUT (Poiltechnika Warszawska, Poland) - open

# ECCC +Japan + WS64 (SP & SPC)

- ECCC – Italian partners: INAIL, Ansaldo Energia, +1
- ECCC – Finland: VTT  (also in INNUMAT)
- Japan: Kagoshima Univ, + CRIEPI?
- WS64, … ?

Others:

- University of Oviedo- MARIA CRISTINA RODRIGUEZ GONZALEZ cristina@uniovi.es - SPT

# Studied Lab specific parameters (SP)- ASTM E691

X labs perform 3 tests, thus producing a set of ≥ 24 tests (min 6 labs!)

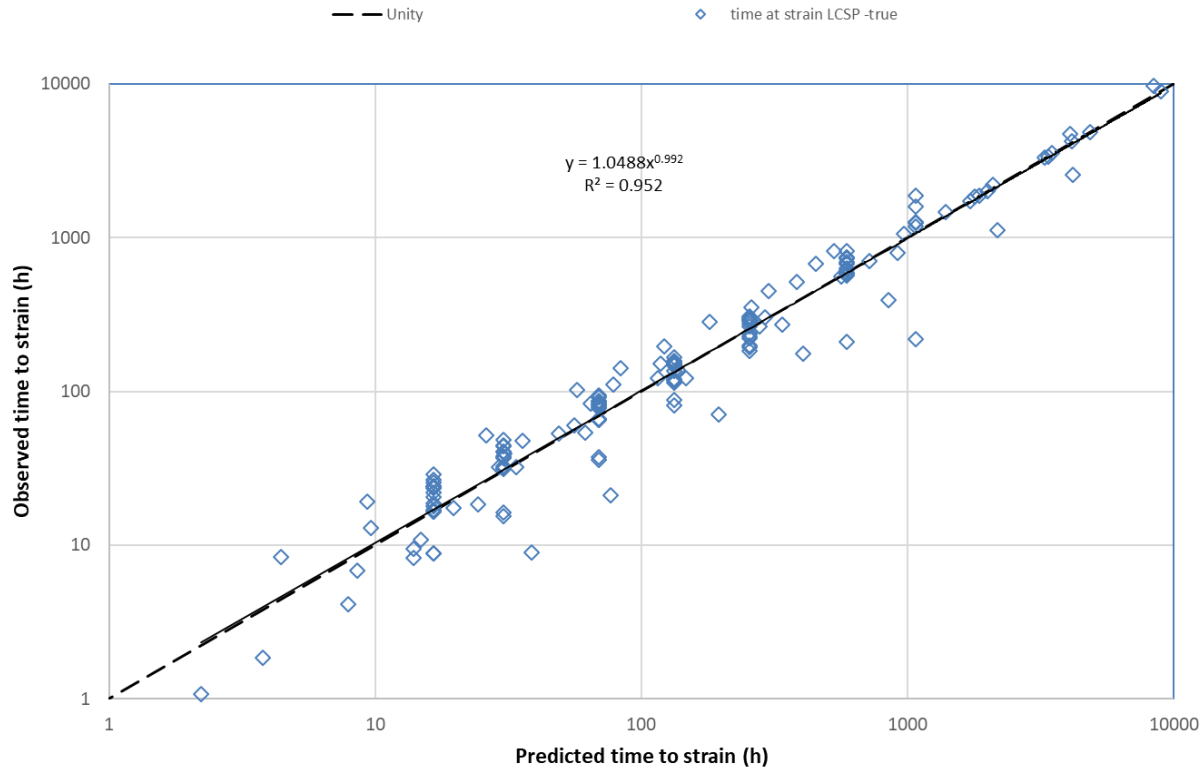
- ***ID Lab***: Laboratory identifier #1-#8
- ***p***: number of the tests min. 3(?)/ lab. The tests coming from labs contributing more or less test, are considered separately, so that the degree of freedom for the statistical assessment becomes: ***pmax*** ≥ 24 tests

(If the test method specifies that only one test determination is to be made, then the test determination value is the test result, ASTM E139)

- ***TM***: Testing Machine type: EM = electro/servo mechanical machine with related control, IH = in-house specialized testing machine
- **LC:** Load cell capacity (kN) and / or calibration range used
- **DE**: type of deformation measurement: *w, v* or *u* (load-line displacement w, displacement v, deflection u)
- **d:** type of punch tip, B=ball, H=hemispherical puncher → **measured ball / punch diameter**
- **D:** Measured diameter of the receiving hole (lower die) (EN 10317 – e.g. H6, ∅4 mm +0.008)
- **Cha:** Chamfer type, C=chamfer, R=radius → **measured diameter D + Chamfer**
- ***Gauge***: type of extensometer transducer for *v* or *u* (see above DE) S=strain-gauges-technology-based transducer, I = inductive transducer, C = Capacitive transducer, O=other
- **Cp**: Compliance used for punch and push rod (mm/N) for *w a*nd *v* only (see Gauge)
- ***h0***: sample thickness, (EN 10317 - ∅8 mm / 0.5±0.005) – report specimen specific deviations

# Initial suggestions on reference material comparison values (SPC)

- Time to failure (?)
- Time to minimum deflection rate
- Deflection at specified time
- Deflection rate at specified time
- Time to specified deflection
- Time to specified deflection rate




- → modifications to 600°C / 160 MPa????

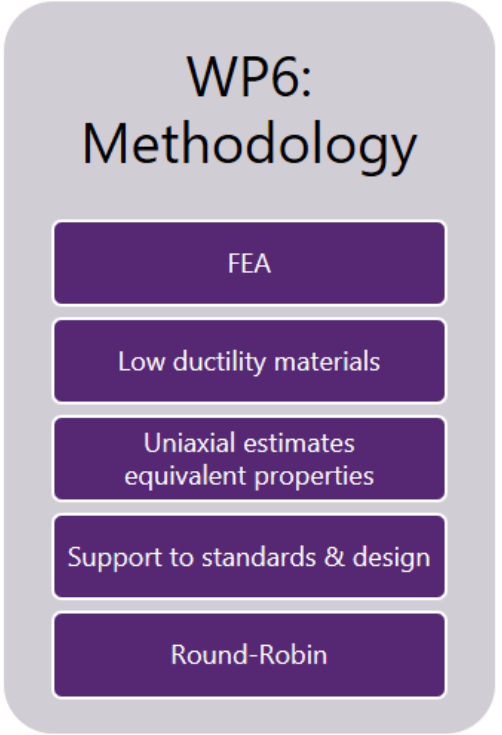
# ECCC + Open access interaction

## WP1 (Methods)

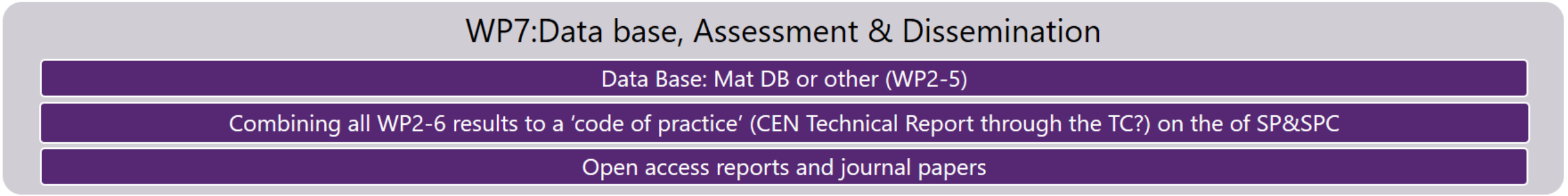
- Miniature testing, AU 3, Miniaturised Specimen Creep Testing, e.g. Small Punch
  - Reference material (**Round robin on tensile SP testing, BCR-661 and also for creep BCR-425**
  - Screening tests / evaluation → initial analysis of **BCR-661 (RT SP tests) – EC open access report soon to be published (SCKCEN-JRC)**
- Creep strength assessments based on SPC (WG1, WG3A, WG3B and C)
- Creep strain modelling, Plastic and Creep Damage (?) – SPC modelling (JRC)
- Supporting data for EUROFER 97 data book + updates on 316L(N)
- Collaboration between ECCC and CEN WS64 (design codes)

# Methodology → INNUMAT WP5

- Testing technique development to support and improve the testing methodologies themselves for future standard revisions and application in design codes.
- Establish methodology for SP and SPC on semi-brittle or embritteled materials, e.g. irradiated materials and the use in hot cell environments (follow up to EERA JPNM project TASTE+, PATRICIA (and INNUMAT)



# Future actions: Data base, reporting and dissemination




- Public dissemination of reference data library, including the primary and secondary (?) reference materials
- **'Drive' the publication of relevant data** and initiate a CEN Technical report as a supporting 'code of practice' for the utilization of the testing standard EN10371.
- The establishing close collaboration between many users of the test method , project consortiums, the national representatives and mirror groups of the small punch standard work group.

# EC3, INNUMAT and WG64 Round robins, Nimonic 75 (BCR-661 or 425)

1. SP 'tensile' with BCR-661 (Initiative INNUMAT → WS64→EC3)
2. SPC creep with BCR-424 (initiative JRC) – test sampling to be discussed with JRC, …

(1) – Samples (blanks) available for BCR-661 (80+80 samples)

- Propose RR test matrix for RT (INNUMAT + others, e.g. CRIEPI)
- Propose matrix for ECCC testing at HT?

(2) - Compare BCR-661 and BCR-425 up to HT?

- Propose matrix for ECCC SPC testing, including (ECCC, incl. CRIEPI, WG64+ INNUMAT?