Development and validation of SSTT towards standardization

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http://rdgroups.ciemat.es/web/materiales/

@MxeCiemat

IFMIF-DONES Users Workshop (on-line), 26–27 de September de 2022

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- Why we talk about **Small Specimens tests Tecniques**?
- Historical use -> Nuclear Fission/Fusion
- Standardization
  - International actions
  - ASTM example

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Why we talk about SSTT

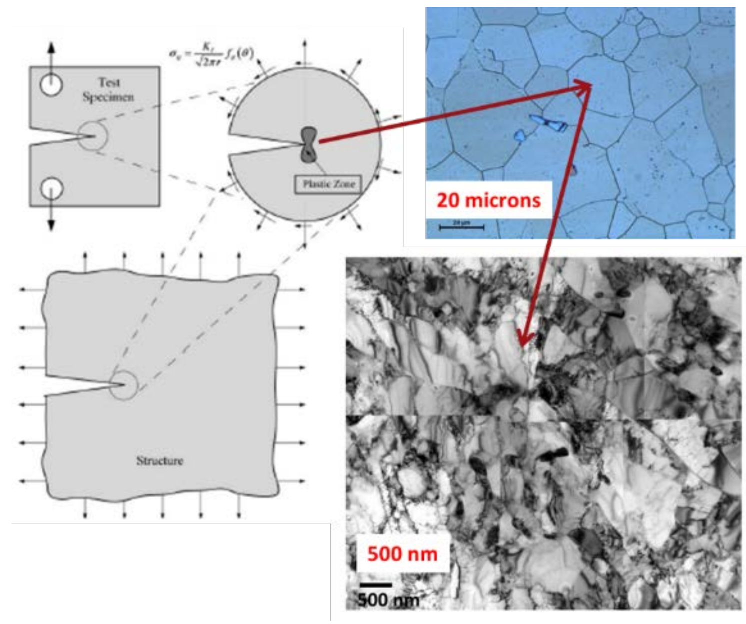
- Materials used in engineering applications as structural components are subject to loads, defined by the application purpose.
- The mechanical properties of materials characterize the response of a material to loading.
- Mechanical properties are determined following **standards**, which define testing machines, **specimen shapes and sizes**, experimental procedures, and data interpretations.
  - Consistent,
  - Uniform, and
  - Reproducible

Mechanical properties are driven by the **microstructure** and **stress state** and thus "relevant volume" should be tested to determine properties that are **independent of the specimen size** to be transferred to engineering structures

Transferability

Test specimen

Structure



Why we talk about SSTT

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- The verification of the structural components of a fusion power reactor requires design criteria developed specifically for those components at the unique conditions at which they are operated
- The RCC-MRx design and construction code constitutes a single document that covers in a consistent manner the design and construction of mechanical components for high temperature reactors, research reactors and fusion reactors
- Need to acquiring and gathering the data which describe the physical and mechanical behaviour of the material, necessary to apply the component design rules available in the RCC-MRx code
- Materials and Design rules have been extended in order to be applicable for nuclear components likely to operate:
  - in creep conditions (significant creep)
  - in irradiated conditions (significant irradiation)

	Fission (Gen. I)	Fission (Gen. IV)	Fusion (DEMO/PROTO)	Spallation (MYRRHA)
Structural alloy T_{max}	<300˚C	500-1000˚C	550-1000˚C	400-600˚C
Max dose for core internal structures	~1 dpa	~30-150 dpa	~150 dpa	≤60 dpa/fpy
Max transmutation helium concentration	~0.1 appm	~3-10 appm	~1500 appm (~10000 appm for SiC)	~2000 appm/fpy
Particle Energy E_{max}	<1-2 MeV	<1-3 MeV	<14 MeV	several hundred MeV

Mechanical data at RELEVANT irradiation conditions

Key challenge for fusion reactor --> main reason why IFMIF/DONES is required

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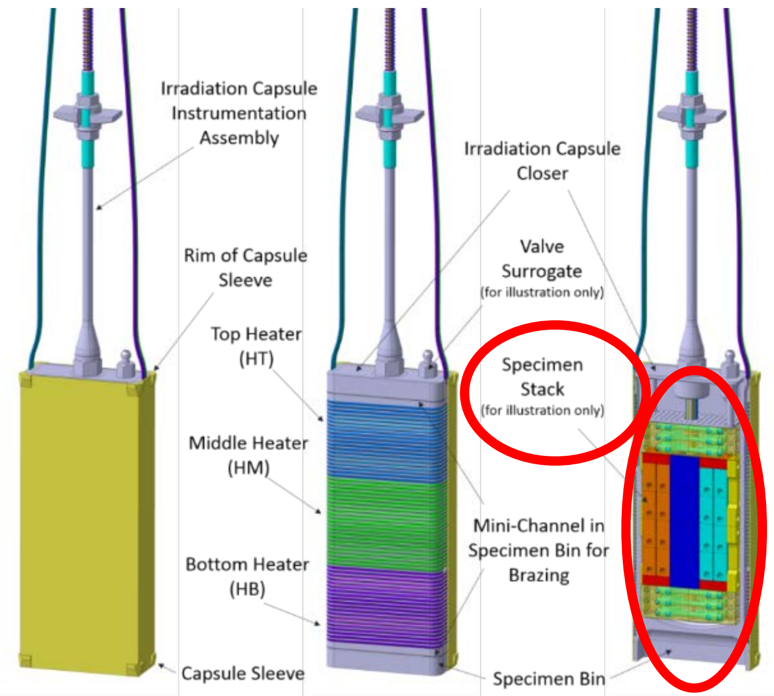
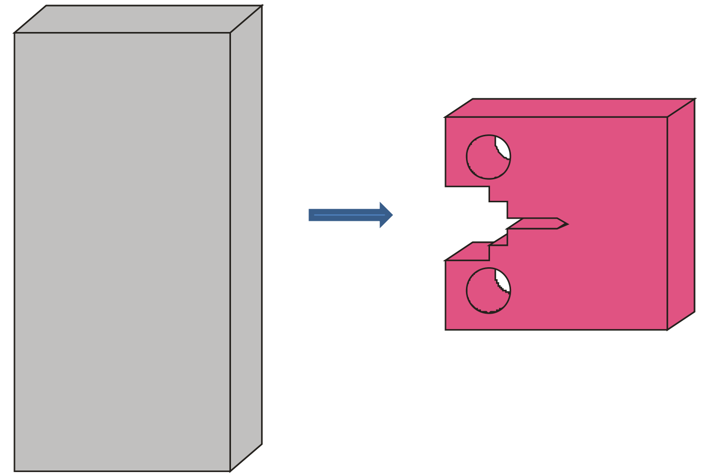
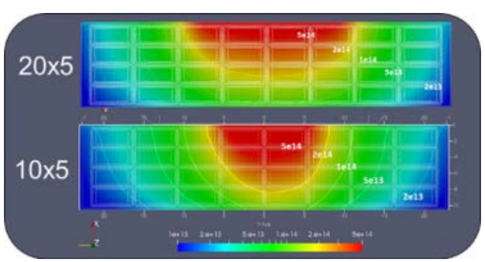
Why we talk about SSTT





- Each capsule of IFMIF/DONES offers a cuboid volume 79 mm x 39 mm x 16.5 mm (54 cm³) for specimens

- A standard fracture toughness specimen, with a thickness of 16 mm to be accommodated within the capsule, has a dimensions of 40 mm x 38.4 mm x 16 mm



Neutron flux (n/cm2/s) distribution on the horizontal cut-plane
Qiu, Y. "IFMIF-DONES HFTM neutronics modeling and nuclear response analyses" Nuclear Materials and Energy Volume 15, May 2018, Pages 185-189

hardly two standard specimens should be irradiated within the same capsule

Reduce the dimensions of the testing specimens

Why we talk about miniature specimens

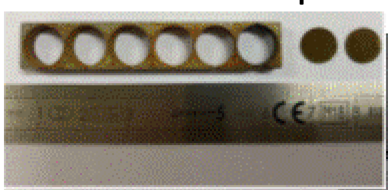
- The miniature (or small) specimen mechanical test technology refers to methods of characterizing the mechanical behavior of materials using specimen sizes **smaller** than the **standard** specimen sizes
  - Sub-size specimens: Same shape but smaller dimensions than the ones on standards
  - New designs: Small punch

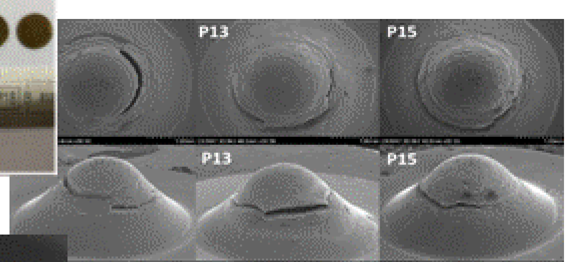
Small Specimens Test Techniques

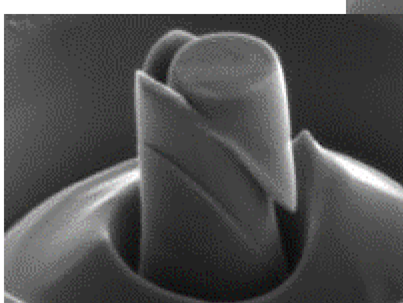
Miniaturized Test Techniques

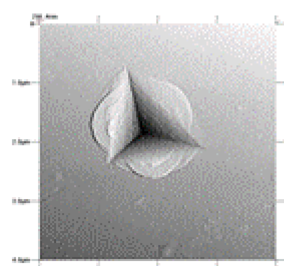
based on novel, unconventional technique

In-Situ techniques







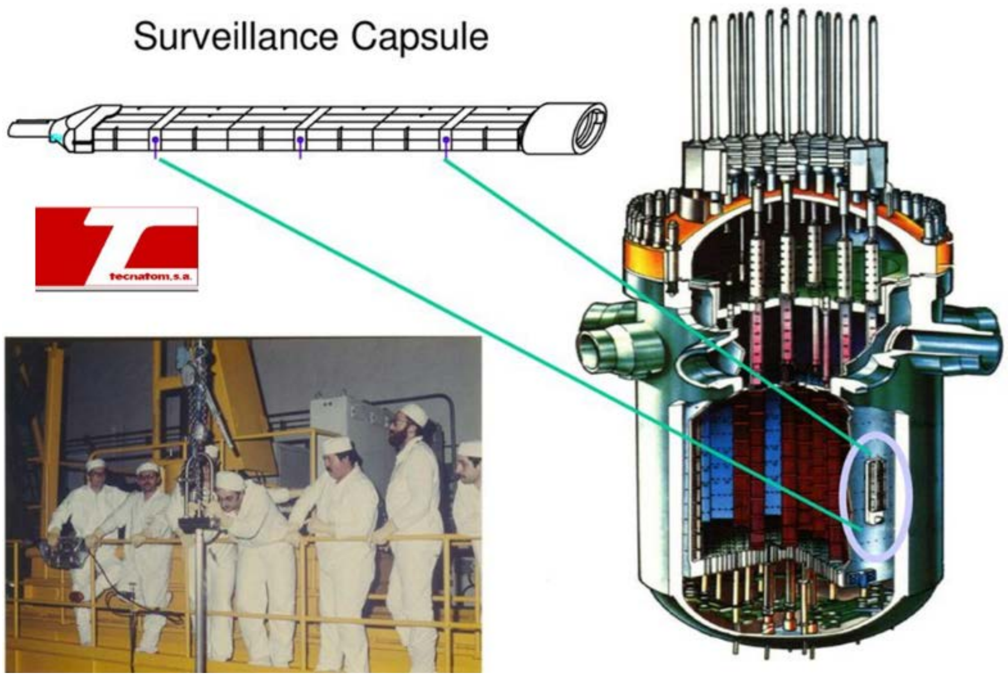


Sub-size specimens

based on a miniaturization or scaling down of what might be termed "conventional" test specimens

Historical use -> Nuclear Fission/Fusion

- The first attempts to miniaturize mechanical test specimen arise in the 70´s for testing irradiated materials in fission reactors due to
  - the ***limited space*** in material tests reactors,
  - concerns about gamma heating or ***fluence gradients*** in large specimens
  - ***dose to personnel*** in post-irradiation testing



Historical use -> Nuclear Fission/Fusion

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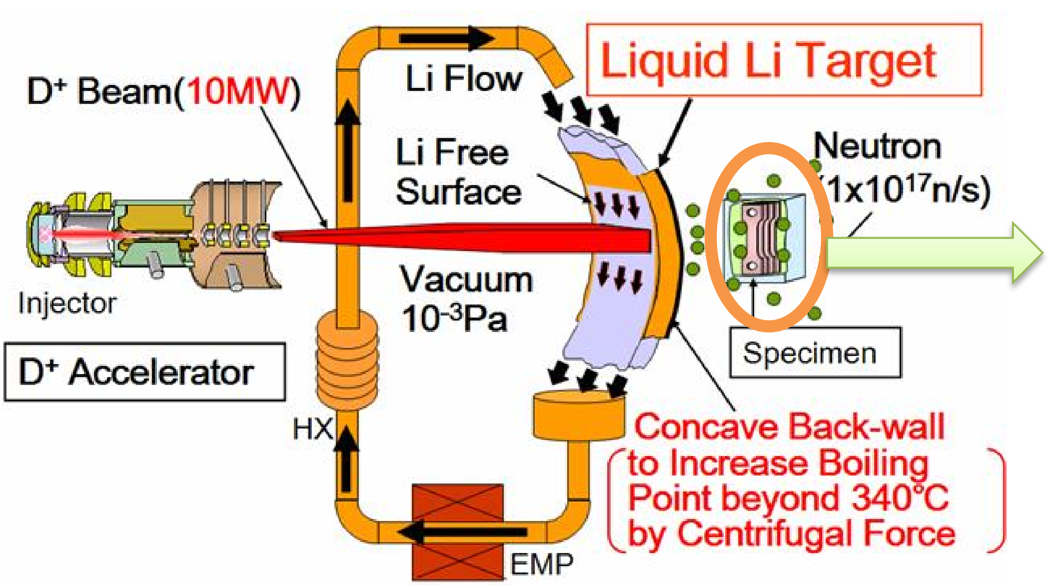
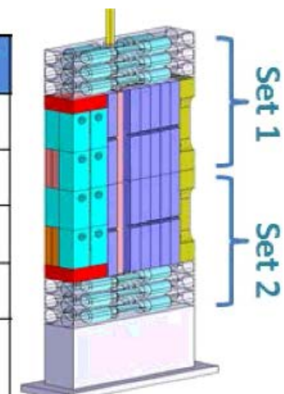
- Blue Ribbon (Cottrell) Panel in 1983
- Fusion materials irradiation test facility (FMIT) project (1978-85)
- International fusion materials irradiation facility (IFMIF) 1980s
- IFMIF/DONES

Neutron radiation source of energy and fluence similar to those that will exist in a Fusion Reactor, to study and select the materials that will be needed for that project and in future fusion power plants



Specimen type	Geometry
Tensile	
Fatigue	
Bend/Charpy DFT	
Creep	
Crack growth	
Fracture toughness	



A. Moslang

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Historical use -> Nuclear Fission/Fusion

- It should be noted that apart from the use of SSTT to characterize the mechanical behaviour of irradiated materials, the application of SSTT is also quite useful in areas as:
  - **Insufficient material** to make full size specimens when a new material is being developed,
  - **In-use component tested**, such as when a scoop sample is taken from an operating component, e.g. reactor pressure vessel, steam pipes, gas turbine blades
  - **Environmental tests**, e.g. liquid metal embrittlement, stress corrosion cracking, environmental assisted fatigue.
  - Determination of properties of **small components**, e.g. coatings, spot welds.
  - **High-throughput screening techniques** to support quality control and certification tasks, e.g. for the rapid screening of large materials design spaces.

SSTT Standarization

- The lack of standardization, not only for the specimen geometry but also for the testing protocol and data uncertainty analysis, is highlight in all the SSTT activities since decades
- ***Early effort for standardization*** on the use of SSTT for fission and fusion applications was launched by ***ASTM*** in the 80's and a task group was thus formed under the auspices of ASTM Subcommittee E10.02 on "*Behavior and Use of Metallic Materials in Nuclear Systems*" to consider whether a set of recommended practices for individual small-specimen techniques might be useful and, if so, to write such practices.
- They agree to organise a serie of ***symposium on small-specimen testing***, being the first held in 1983 in Albuquerque, New Mexico, and focused on test techniques for fusion reactors and the last one was held in 2014 in Houston.

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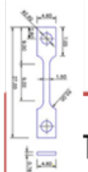
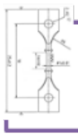
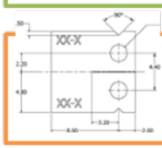
SSTT Standarization

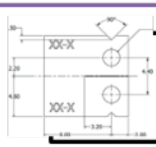
- **IAEA** Coordinated Research Project (CRP) Towards the Standardization of Small Specimen Test Techniques for Fusion Applications (F13017) 2017-2021


Tensile


Creep


Low Cycle Fatigue


Fracture toughness


Fatigue crack growth

IAEA
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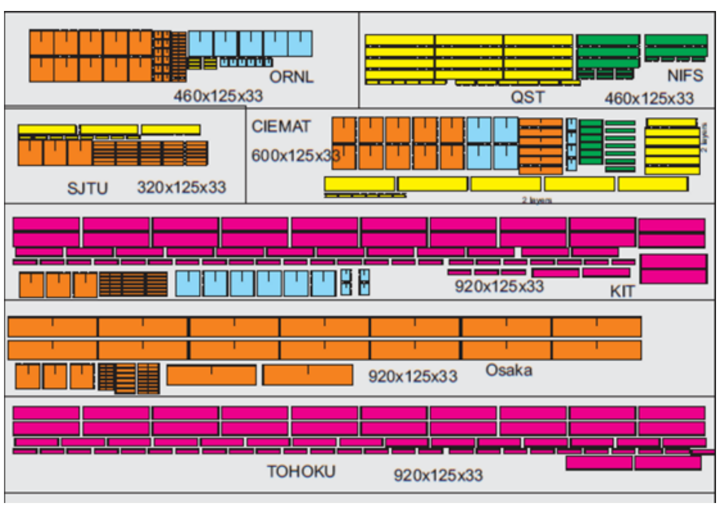


IAEA CRP 'Towards the Standardization of Small Specimen Test Techniques For Fusion Applications - phase II

To focus the experimental campaign on High temperature (300ºC-550ºC) tensile (1st priority) and Fracture toughness (2nd priority).

Same heats as in the 1st CRP (eurofer 97-3 plate, CLAM and F82H).

SSTT Standarization

EUROfusion

H2020 EUROFUSION 2015-2020

HE EUROFUSION 2021-2024

2015 | 2016 | 2017 | 2018 | 2019 | 2020

2021 | 2022

A review of existing data and procedures to tests and analyse small specimen tests
- tensile,
- impact,
- low cycle fatigue
- fracture toughness,
- fatigue crack growth

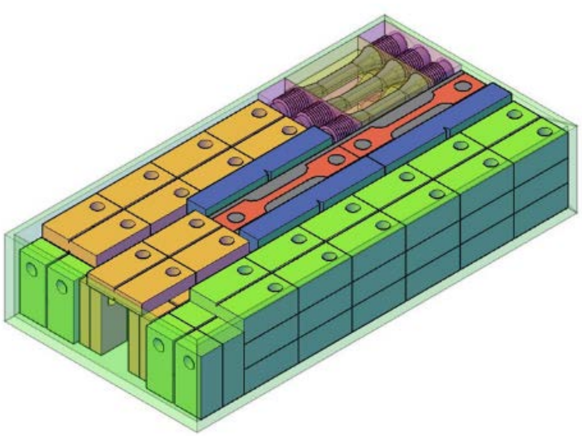
- Recommended number of specimens/test
- Procedure to obtain specimens from irradiated coupons

Statistical analysis of the effect of specimen size
- Tensile, creep
- Impact
- Fracture toughness
- Fatigue crack growth

Critical evaluation of specimen size already included in the testing standards
-> Geometry recommendations
Specific requirements for small specimens tests techniques

Effect of specimen size
- Low cycle fatigue
- Fatigue crack growth
- Ductile fracture toughness

- Tensile tests; 3PB tests; Fracture toughness tests
- Non irradiated and irradiated W and EU97



transversal activity between ENS / DONES and MAT (DEMO: EDDI / SDQ / HHFM / IRRAD; TBM),

RAFM, CuCrZ, W

Tensile, LCF, FT

Roadmap for standarization

SSTT Standarization

- Some mechanical properties by the use of small specimens has already reached the sufficient maturity to ***be included in testing standards***.
  - ***Miniature V-notch impact*** specimens that are already included on the ISO 14556 and ASTM 2248 testing methods.
  - ASTM E3205 and EN 10371 ***small punch testing***
  - ***Mini-CT*** international Round Robin for T_0 determination of RPV steels
- New actions
  - Small tensile ISO 6892 Metallic materials -- Tensile testing -- Part 5: Guideline for testing miniaturised test pieces (NEW CEN WORK ITEM LAUNCHED by aluminium industry)
  - CEN Workshop (CEN/WS) 'Fracture toughness evaluation methodologies applied to advanced high strength steel sheets'. ->fracture toughness evaluation in metal sheets.

SSTT Standarization

Challenges for a new standard

Effect of specimen size

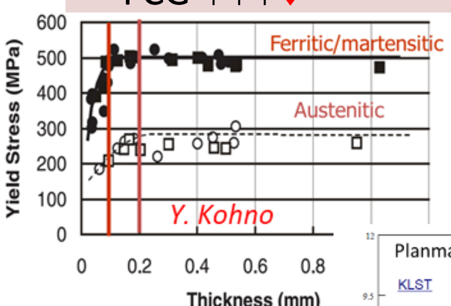
- Tensile – ↑↑↓
- Impact – ↑↑↑↓
- Fracture toughness
  - DBTT - ↑↑↑↓ Ductile - ↓
- Creep ↑↑↑↓
- LCF ↓
- FCG ↑↑↑↓

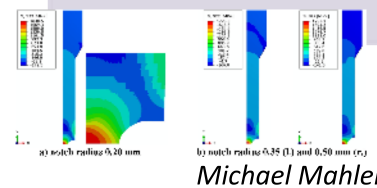
Testing procedure

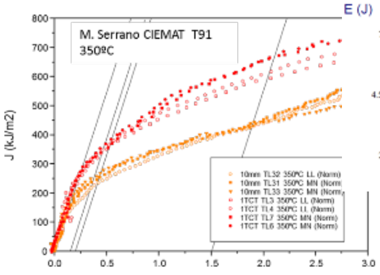
- Specimen preparation
- Dimensional control
- Testing parameters
- Monitoring
- ..

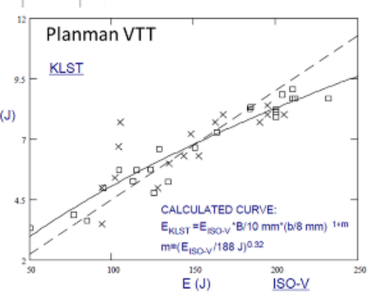
Data analysis

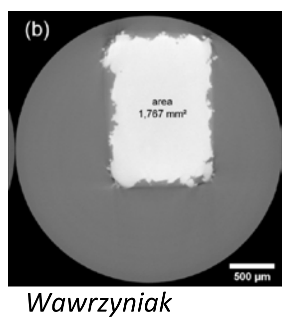
- FEM analysis
- Uncertainty analysis
- Precision and bias
- Interlaboratory exercices

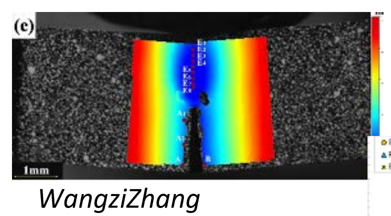


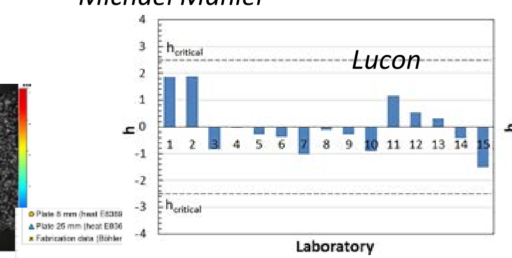


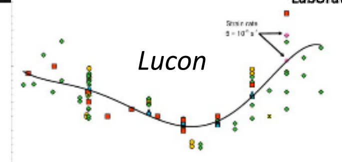












SSTT Standarization

- Standardization needs depends on the type of mechanical tests
  - Complete new testing standard?
    - Small punch tests
  - Modification of existing one ?
    - Non mandatory annex for small impact test
    - Modification of testing analysis for T_0 determination
  - One single standard for testing technique or one that include all the SSTT ?
  - ASTM or ISO ?

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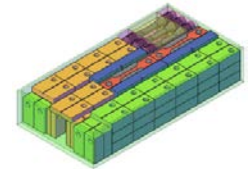
Standarization route

- ASTM example New Standard Activity

www.astm.org

Determine if new standard is needed →

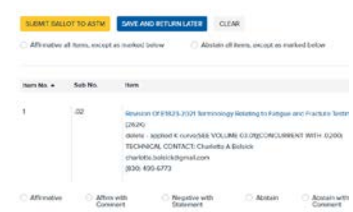


Identify key stakeholders → EUROfusion, FUSION FOR ENERGY, RCC-MRx

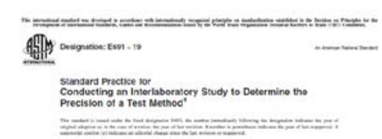
Identify Committee and Subcommittee → Committee E10 – Nuclear Applications

Register a Work Item → ASTM Stimulates **participation from outside of task group**

Balloting →



Final publication →


Designation: E691 – 19

Standard Practice for
Conducting an Interlaboratory Study to Determine the Precision of a Test Method

Standarization route

- **ASTM Work Item**
  - 1. Scope
  - 2. Referenced Documents
  - 3. Terminology
    - 3.1 Definitions
  - 4. Significance and Use
  - 5. Apparatus
  - 6. Test Specimens
  - 7. Procedure
  - 8. Post-Test Examination
  - 9. Report
  - 10. Precision and Bias (MANDATORY)
  - 11. Keywords

An **interlaboratory study (ILS)** is a multi lab study done for the specific purpose of producing data that will be used to develop a Precision & Bias statement and Research Report in order to demonstrate the expected variability of a test method.

Where numerical data have been generated to establish the precision and bias of a test method, a **research report** is required

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Standarization route

- **Interlaboratory study** – ASTM E691 – 19 An American National Standard Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
  - Minimum laboratories: 6
  - Minimum materials representing different test levels: 3
  - Minimum nº of tests per laboratory: 3
  - *"Before investing laboratory time in the full scale ILS, it is usually wise to conduct a pilot run with only one, or perhaps two, material(s)"*
  - ***Main statistics***
    - *h* = the between-laboratory consistency statistic
    - *k* = the within-laboratory consistency statistic
    - r= Repeatability ranges
    - R= Reproducibility ranges

This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

ASTM INTERNATIONAL **Designation: E691 – 19** An American National Standard

Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[1]

This standard is issued under the fixed designation E691; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This practice describes the techniques for planning, conducting, analyzing, and treating the results of an interlaboratory study (ILS) of a test method. The statistical techniques described in this practice provide adequate information for formulating the precision statement of a test method.

1.2 This practice does not concern itself with the development of test methods but rather with gathering the information needed for a test method precision statement after the development stage has been successfully completed. The data obtained in the interlaboratory study may indicate, however, that further effort is needed to improve the test method.

1.3 Since the primary purpose of this practice is the development of the information needed for a precision statement, the experimental design in this practice may not be optimum for evaluating materials, apparatus, or individual laboratories.

1.4 *Field of Application*—This practice is concerned exclusively with test methods which yield a single numerical figure as the test result, although the single figure may be the outcome of a calculation from a set of measurements.

1.4.1 This practice does not cover methods in which the measurement is a categorization; however, for many practical purposes categorical outcomes can be scored, such as zero-one scoring for binary measurements or as integers, ranks for example, for well-ordered categories and then the test result can be defined as an average, or other summary statistic, of several individual scores.

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E456 Terminology Relating to Quality and Statistics
E1169 Practice for Conducting Ruggedness Tests
E1402 Guide for Sampling Design
E2282 Guide for Defining the Test Result of a Test Method

3. Terminology

3.1 *Definitions*—Terminology E456 provides a more extensive list of terms in E11 standards.

3.1.1 *accuracy, n*—the closeness of agreement between a test result and an accepted reference value. **E177**

3.1.2 *bias, n*—the difference between the expectation of the test results and an accepted reference value. **E177**

3.1.3 *interlaboratory study, (ILS) in ASTM, n*—a designed procedure for obtaining a precision statement for a test method, involving multiple laboratories, each generating replicate test results on one or more materials.

3.1.4 *observation, n*—the process of obtaining information regarding the presence or absence of an attribute of a test specimen, or of making a reading on a characteristic or dimension of a test specimen. **E2282**

3.1.5 *precision, n*—the closeness of agreements between independent test results obtained under stipulated conditions. **E177**

[1] This practice is under the jurisdiction of ASTM Committee E11 on Quality and Statistics and is the direct responsibility of Subcommittee E11.20 on Test Method Evaluation and Quality Control.

Current edition approved Sept. 1, 2019. Published October 2019. Originally approved in 1979. Last previous edition approved in 2018 as E691 – 18. DOI: 10.1520/E0691-19.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

1

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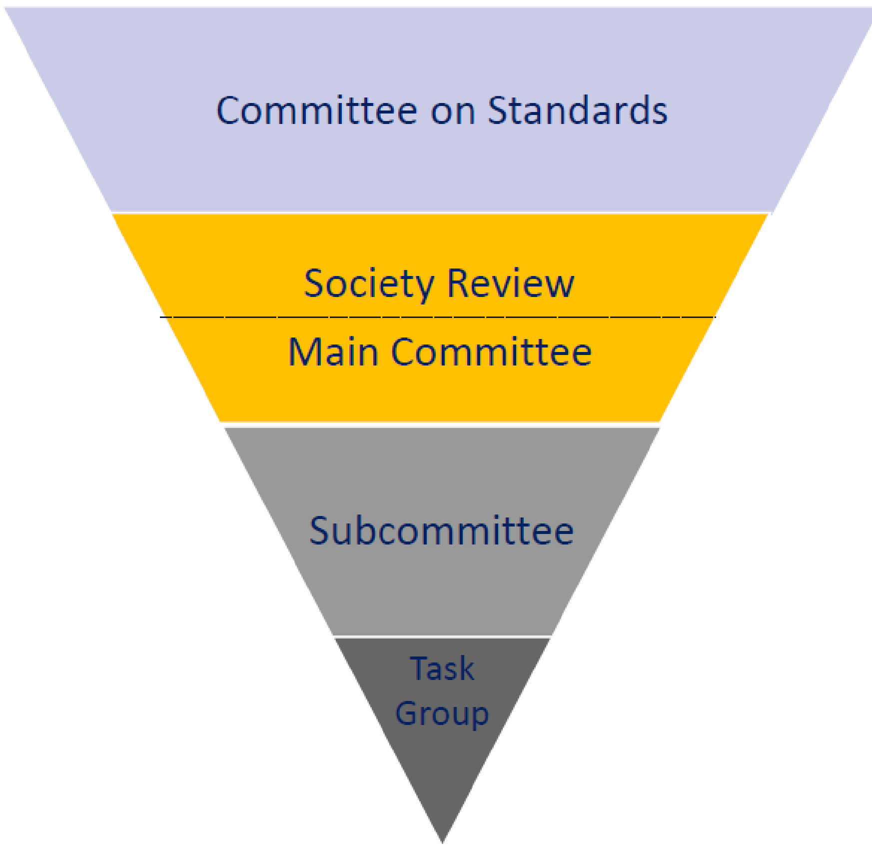
Standarization route

Balloting



Final Review and Approval of Procedures
(9 Member Group)

Society Review
NO % Return Required
NO % Affirmative Required

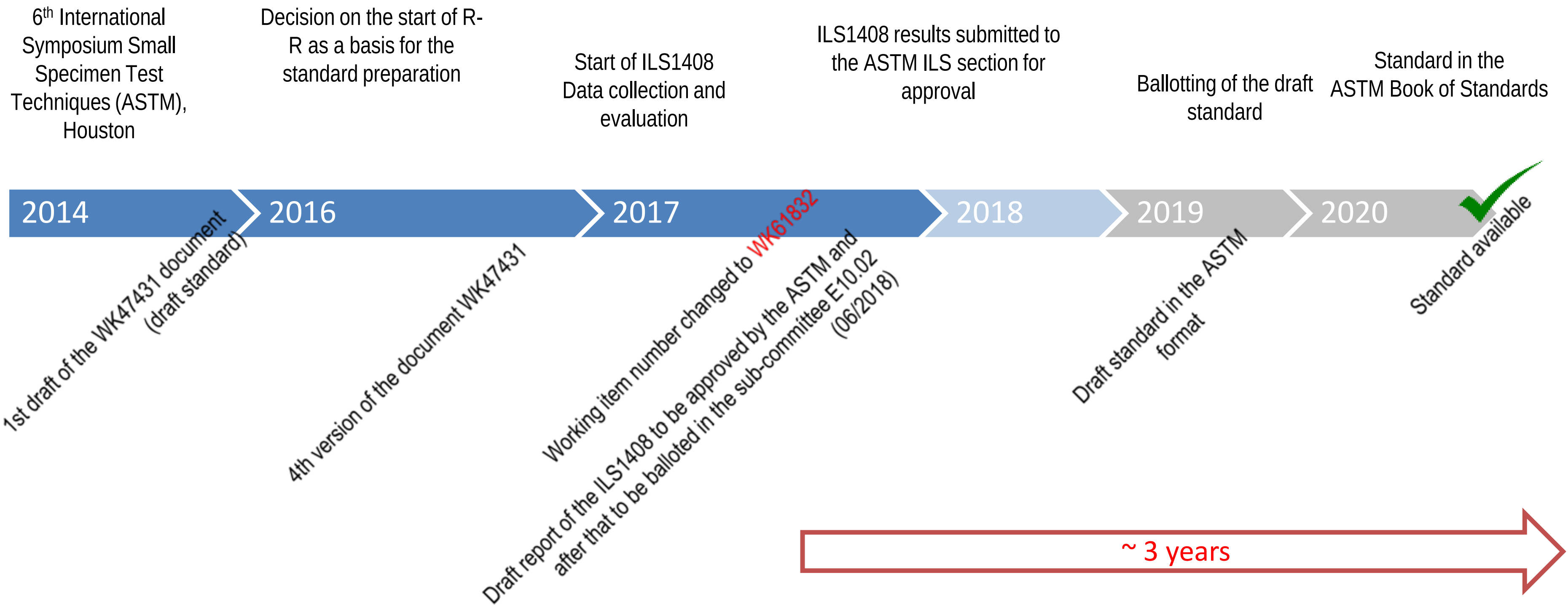
Main Committee
60% Return Required
90% Affirmative Required

Subcommittee
60% Return Required
2/3 Affirmative Required

Task Group
Membership Not Required

ASTM standard

- Standardization Schedule (small punch)



Summary

- The need to validate SSTT is common for different material qualification process (Fusion, Fission, In-service, High througthput..)
- Some mechanical properties by the use of small specimens has already reached the sufficient maturity to be included in testing standards (small impact, small punch)
- International efforts are on going for the standardization of SSTT needed to obtain engineering relevant mechanical properties (IAEA, EUROfusion, other H2020 projects)