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Guidelines for IAEA Small Specimen Test Techniques Master Curve Fracture Toughness Testing

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Materials Science and Technology Division

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1. Introduction

Under the auspices of International Atomic Energy Agency (IAEA), a coordinated research project (CRP) entitled "Towards the Standardization of Small Specimen Test Techniques for Fusion Applications" has started since 2017. The overall objective of the project is to provide a set of guidelines for small specimen test techniques (SSTT) based on commonly agreed best practices on main test techniques including tensile, creep, low cycle fatigue, fracture toughness, and fatigue crack growth rate. This will act as the first step of a full standardization of the SSTT. Fusion structural materials, i.e., reduced activation ferritic/martensitic (RAFM) steels, are used for testing. In addition, the project will create a comprehensive mechanical property database of RAFM steels tested by SSTT.

For the fracture toughness task of the CRP, three testing methods including Master Curve, local approach for ductile crack growth and cleavage fracture, and ductile approach at room temperature will be evaluated. This report focuses on developing guidelines for the round-robin Master Curve testing based on the ASTM standard E1921-19b "Standard Test Method for Determination of Reference Temperature, T_0, for Ferritic Steels in the Transition Range" [1] and commonly agreed best practice from researchers at Oak Ridge National Laboratory (ORNL), Centre for Energy, Environment and Technology (CIEMAT), and UK Atomic Energy Authority (UKAEA).

2. Materials and Test Matrix

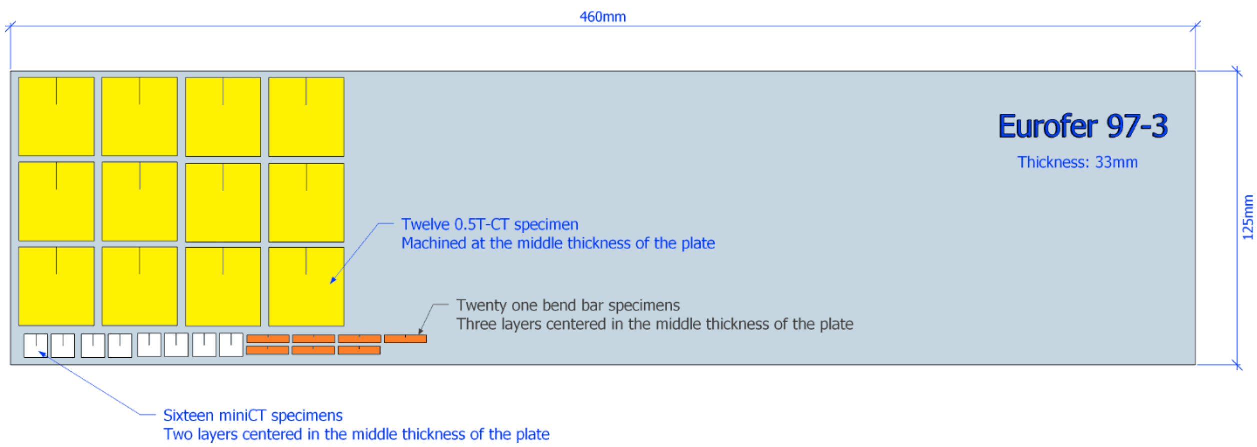
The materials used in the round-robin testing are Eurofer 97 batch-3 and F82H-BA12. Table 1 summarizes the test matrix adopted in the round-robin testing. The specimen machining plan is highlighted in Fig. 1 with individual specimen drawings given in Figs. 2-8. Each participating laboratory will perform the machining independently. The standard specimen configuration and dimensions shall be in line with requirements in E1921-19 sections 7.1 to 7.4 where applicable.

Table 1 Master Curve round-robin testing matrix

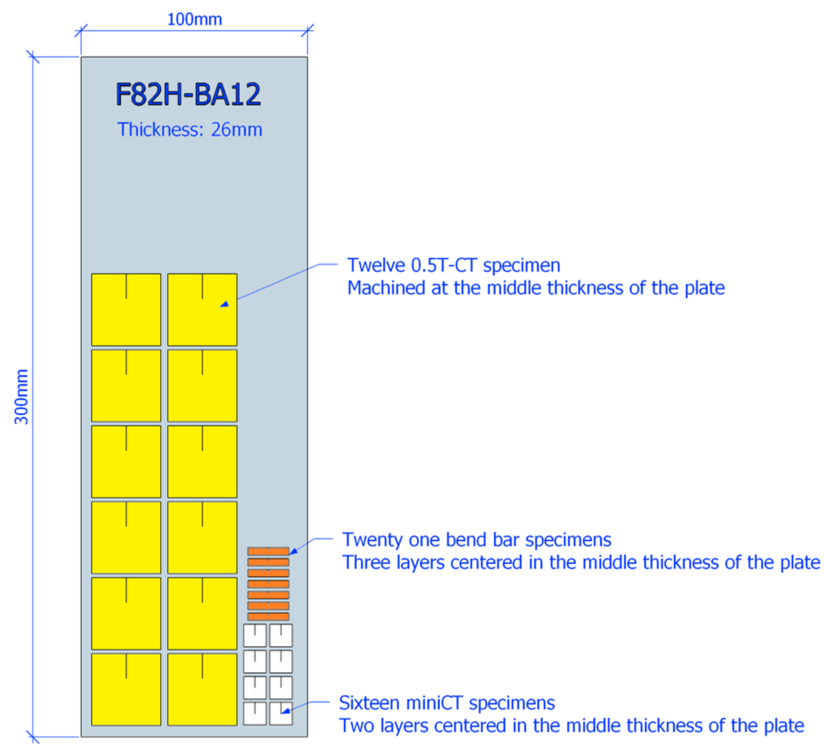
Participants	Materials	Specimen configuration	# of specimens	Orientation	Size of raw material*
X. Chen/M. Sokolov (ORNL)	Eurofer97 batch-3	miniature bend bar (Fig.2)	16	LT	460mm(L) x 125mm(T) x 33mm (S)
		miniCT (Fig.3)	10-12		
		0.5TCT (Fig.4)	8-10		
	F82H-BA12	miniature bend bar (Fig.2)	16	LT	100mm(L) x 300mm(T) x 26mm (S)
		miniCT (Fig.3)	10-12		
		0.5TCT (Fig.4)	8-10		
R. Hernandez Pascual/ M. Serrano (CIEMAT)	Eurofer97 batch-3	PCCVN (Fig.5)	8-10	LT	600mm(L) x 125mm(T) x 33mm (S)
		0.5TCT (Fig.6)			
	F82H-BA12	PCCVN (Fig.5)	8-10	LT	200mm(L) x 300mm(T) x 25mm (S)
		0.5TCT (Fig.6)			
D. Andres (UK	Eurofer97	miniCT (Fig. 7)	10-12	LT	50mm(L) x

AEA)	batch-3	miniCT-DONES (Fig. 8)	10-12		120mm(T) x 33mm (S)
	F82H-BA12	miniCT (Fig. 7)	10-12	LT	40mm(L) x 60mm(T) x 25mm (S) (TBD)
		miniCT-DONES (Fig. 8)	10-12		

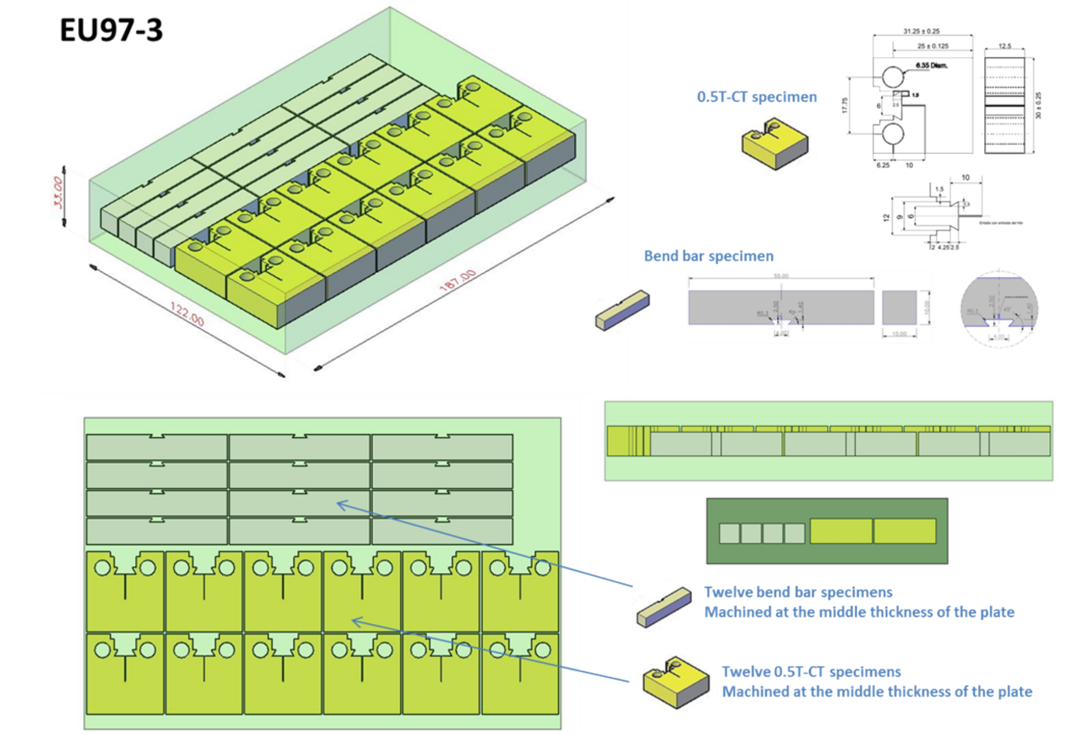
* L: longitudinal, T: transverse, and S: short transverse based on the plate orientation



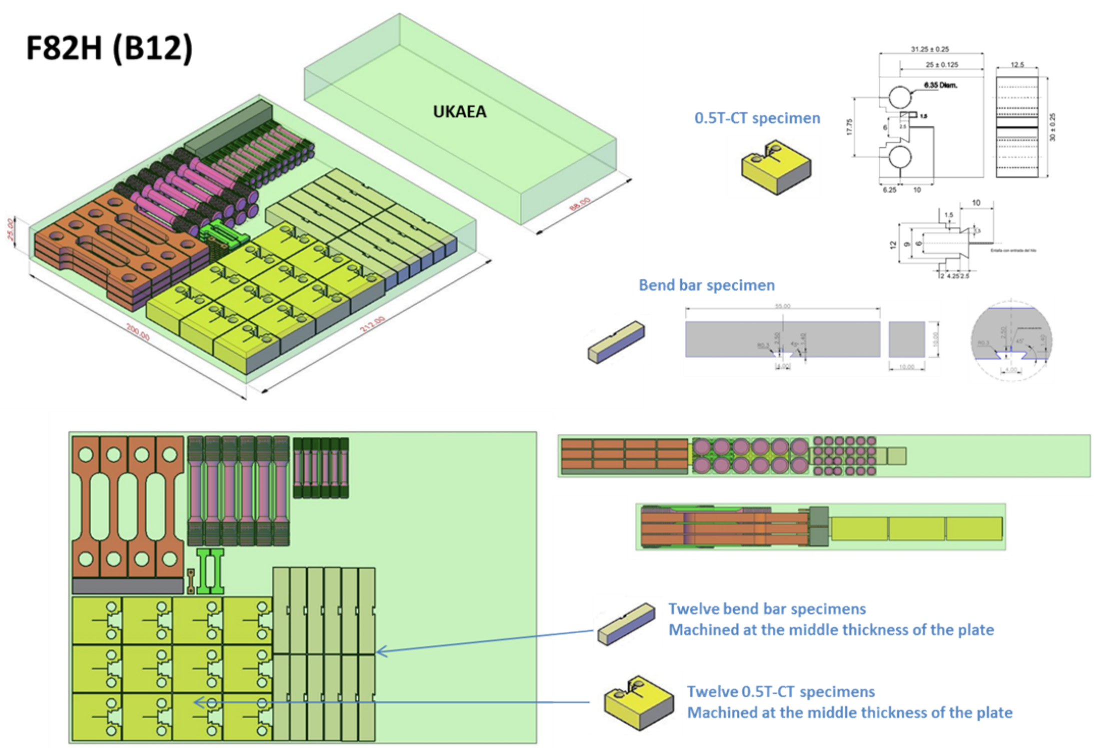
(a)



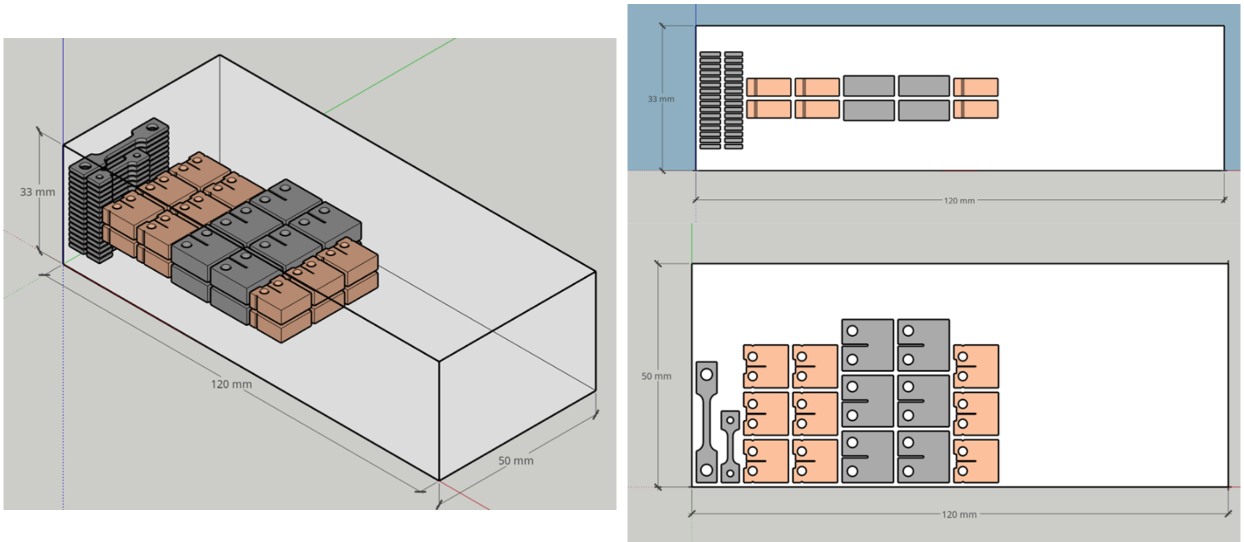
(b)


EU97-3
0.5T-CT specimen
Bend bar specimen
Twelve bend bar specimens
Machined at the middle thickness of the plate
Twelve 0.5T-CT specimens
Machined at the middle thickness of the plate

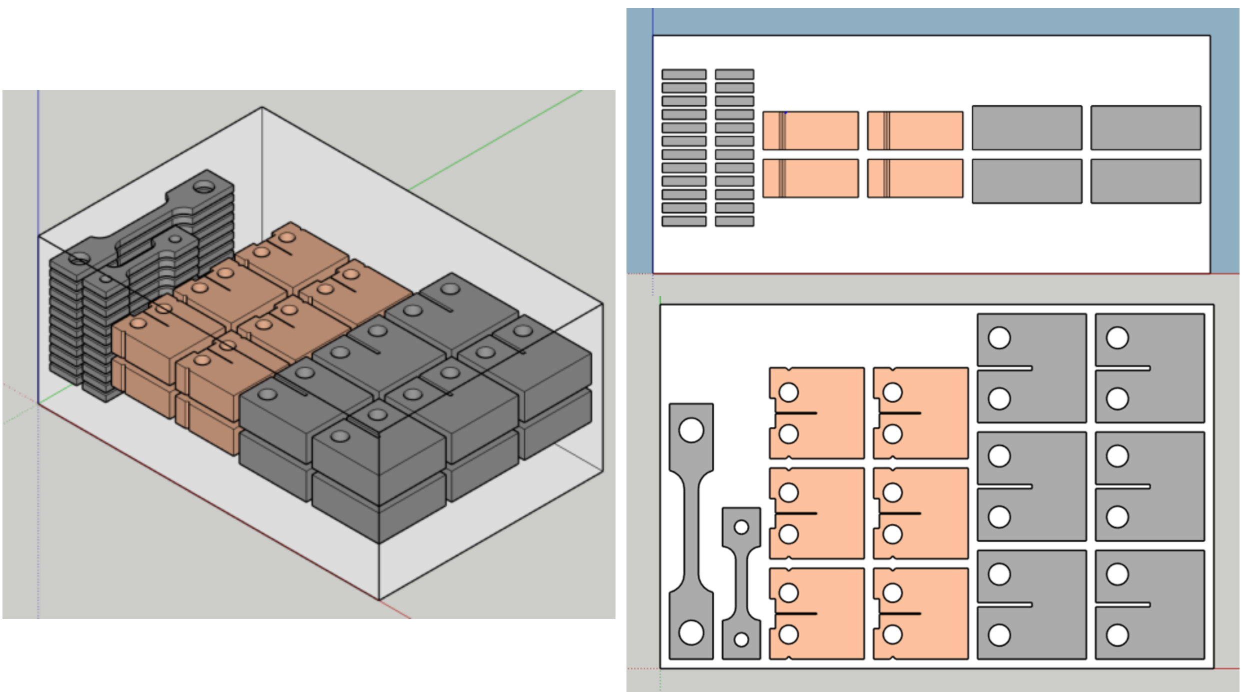
(c)


F82H (B12)
UKAEA
0.5T-CT specimen
Bend bar specimen
Twelve bend bar specimens
Machined at the middle thickness of the plate
Twelve 0.5T-CT specimens
Machined at the middle thickness of the plate

(d)



Eurofer97-3 machining plan (Specimens from left to right: DONES tensile, SSJ3 tensile, mCT-MRF, mCT-DONES, mCT-MRF for FCG) (TBD)
(e)



UKAEA F82H-BA12 machining plan (Specimens from left to right: DONES tensile, SSJ3 tensile, mCT-MRF, mCT-DONES.) (TBD)
(f)

Fig. 1 Specimen machining plans for ORNL in (a) and (b), CIEMAT in (c) and (d), and UKAEA in (e) and (f).

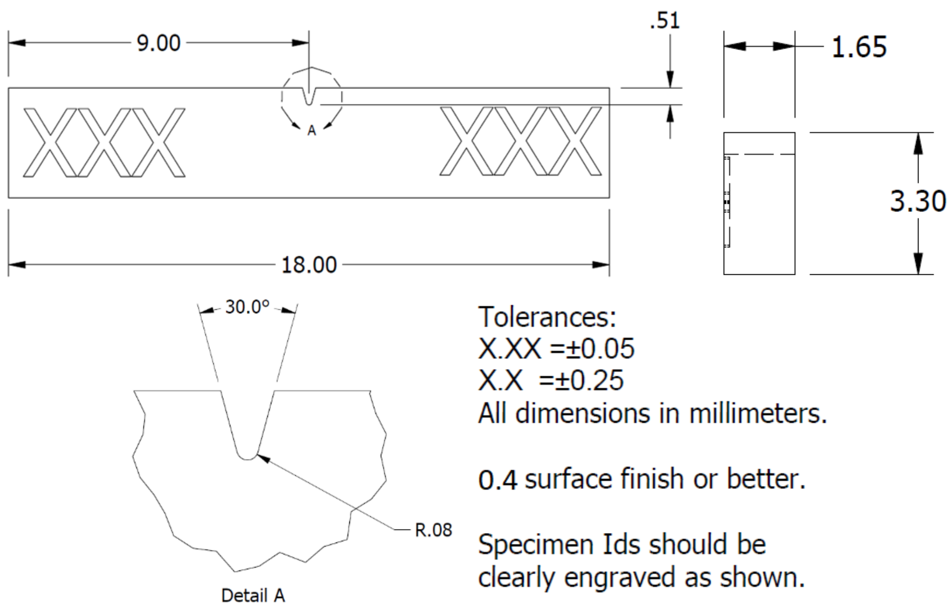


Fig. 2 ORNL miniature bend bar specimen drawing.

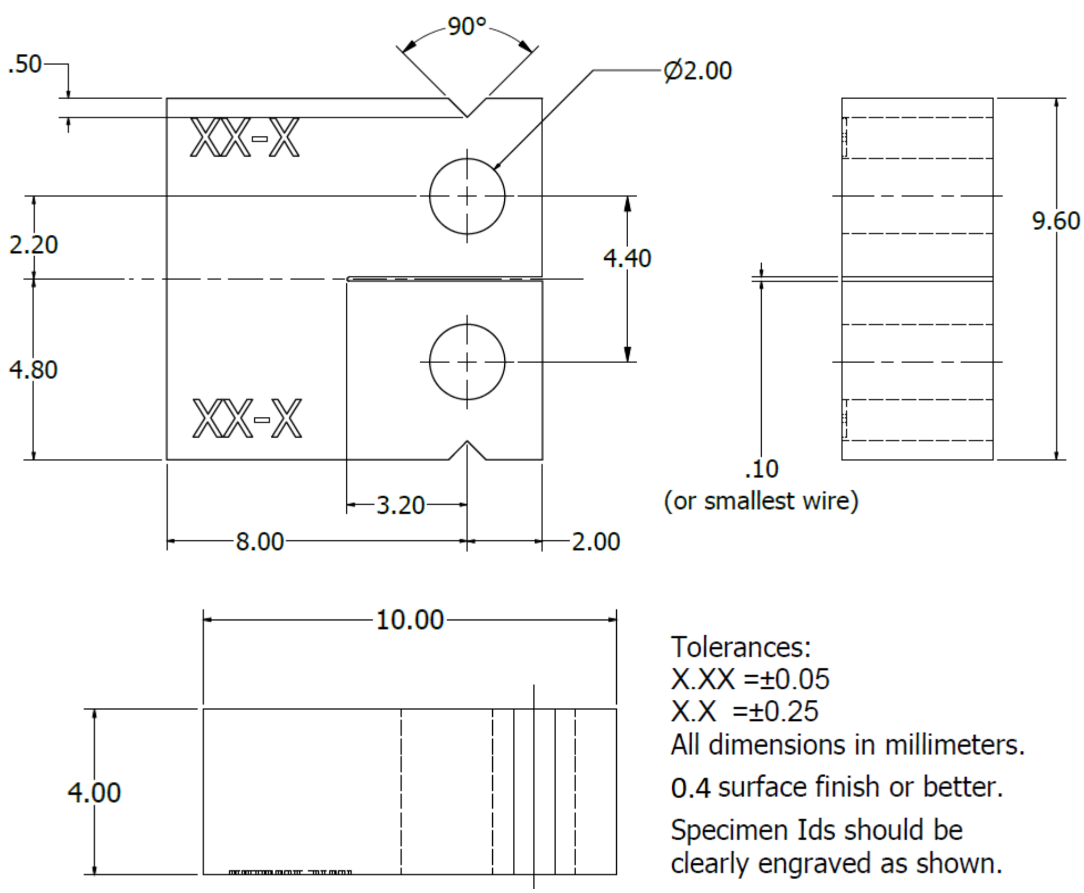


Fig. 3 ORNL miniCT specimen drawing.

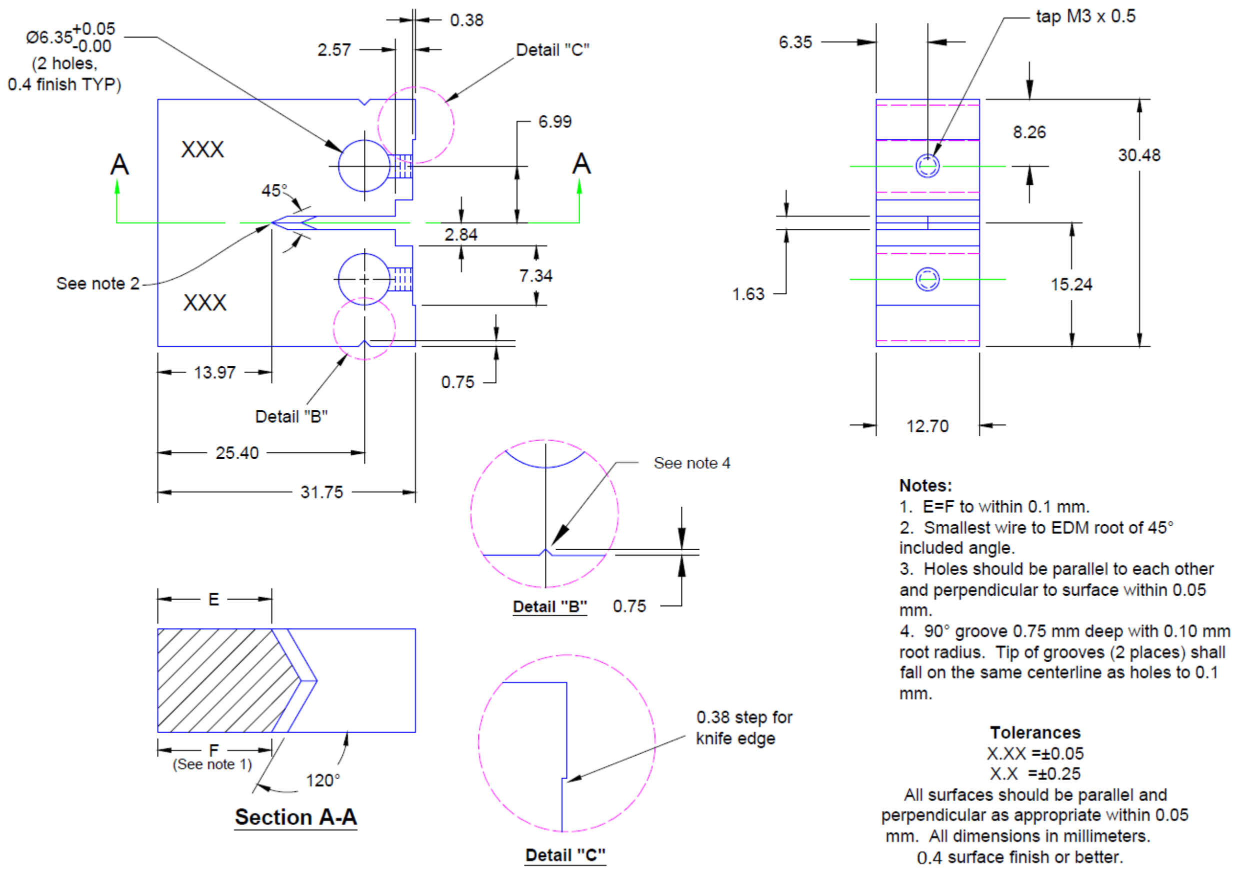


Fig. 4 ORNL 0.5TCT specimen drawing.

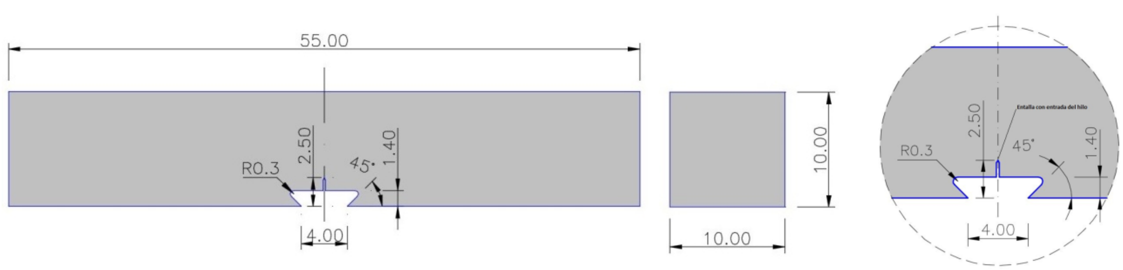


Fig. 5 CIEMAT PCCVN specimen drawing

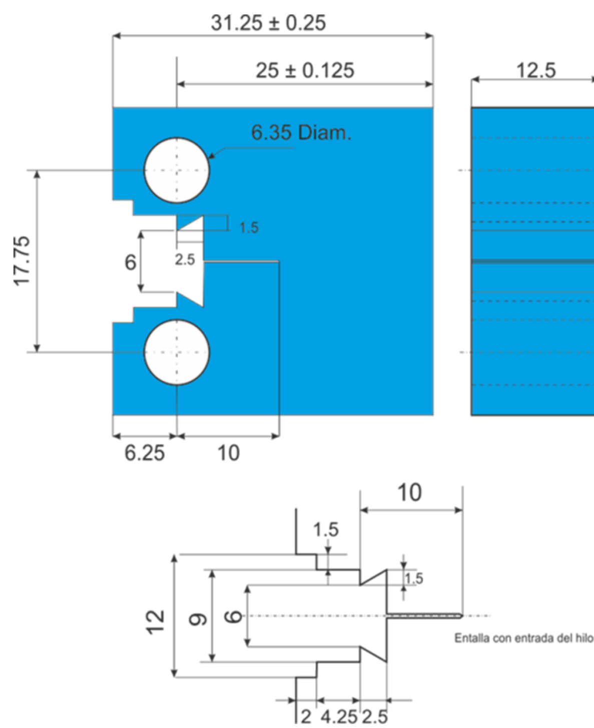
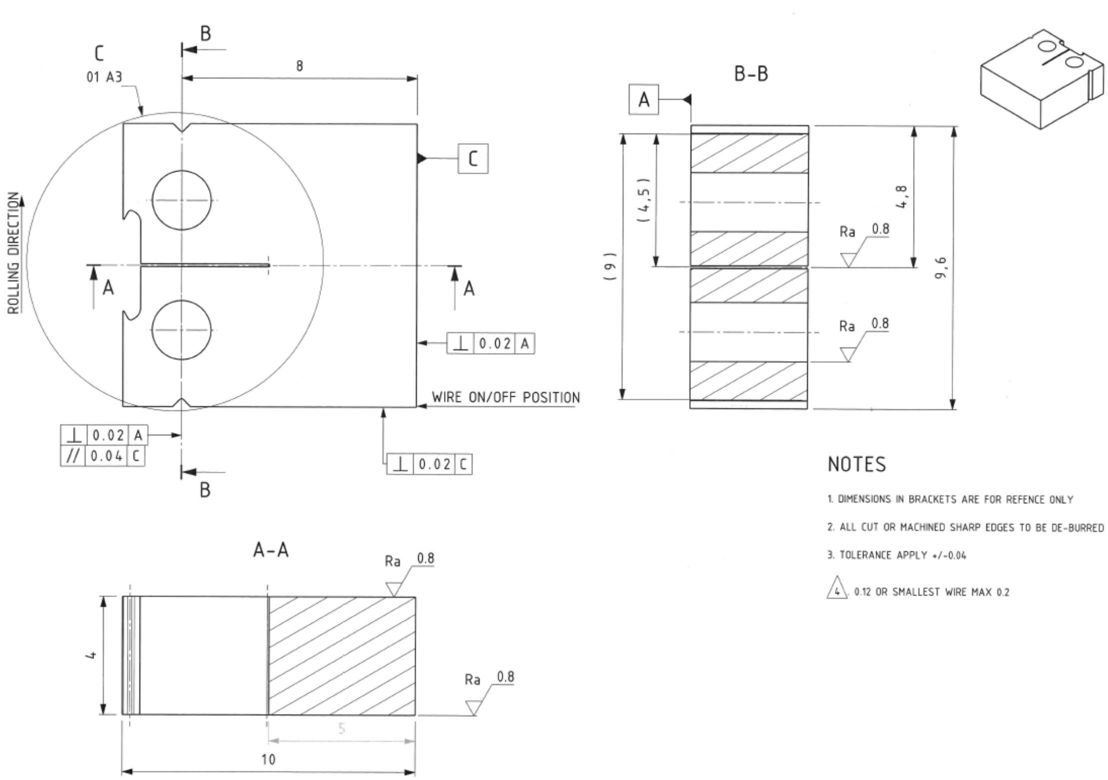
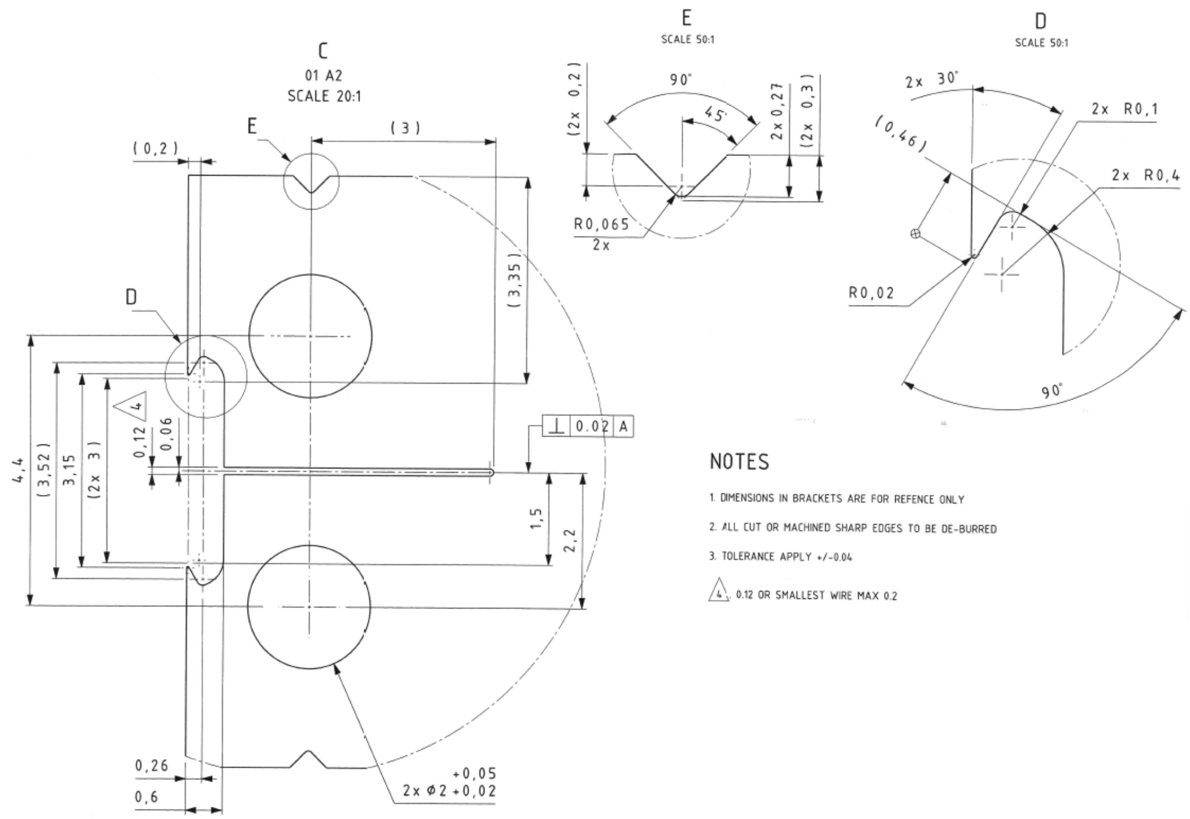


Fig. 6 CIEMAT 0.5TCT specimen drawing

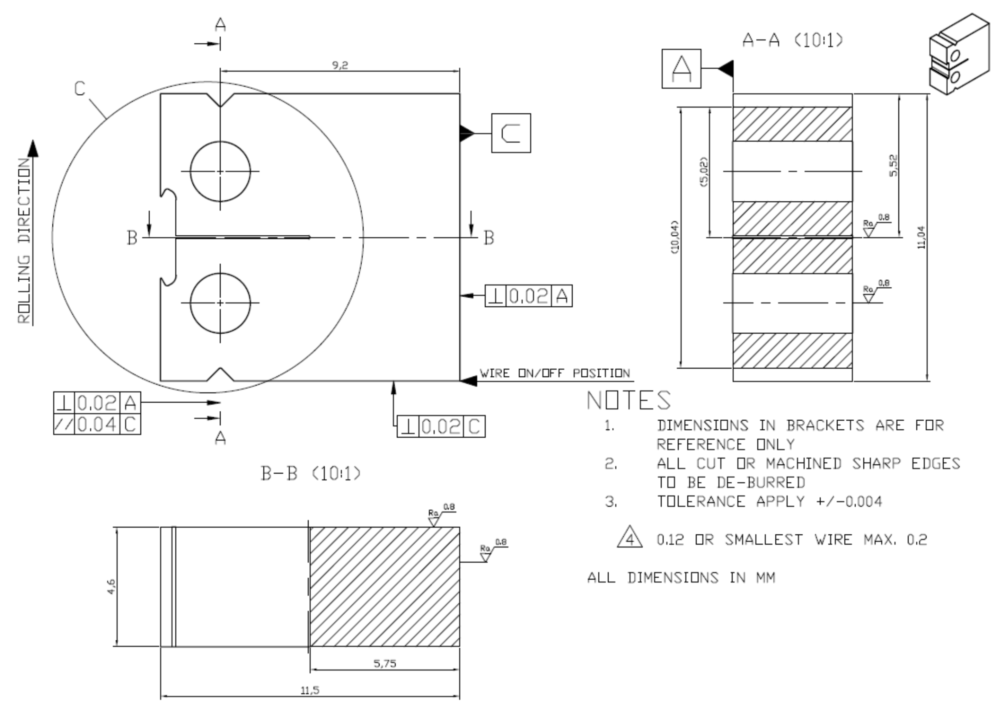


(a)

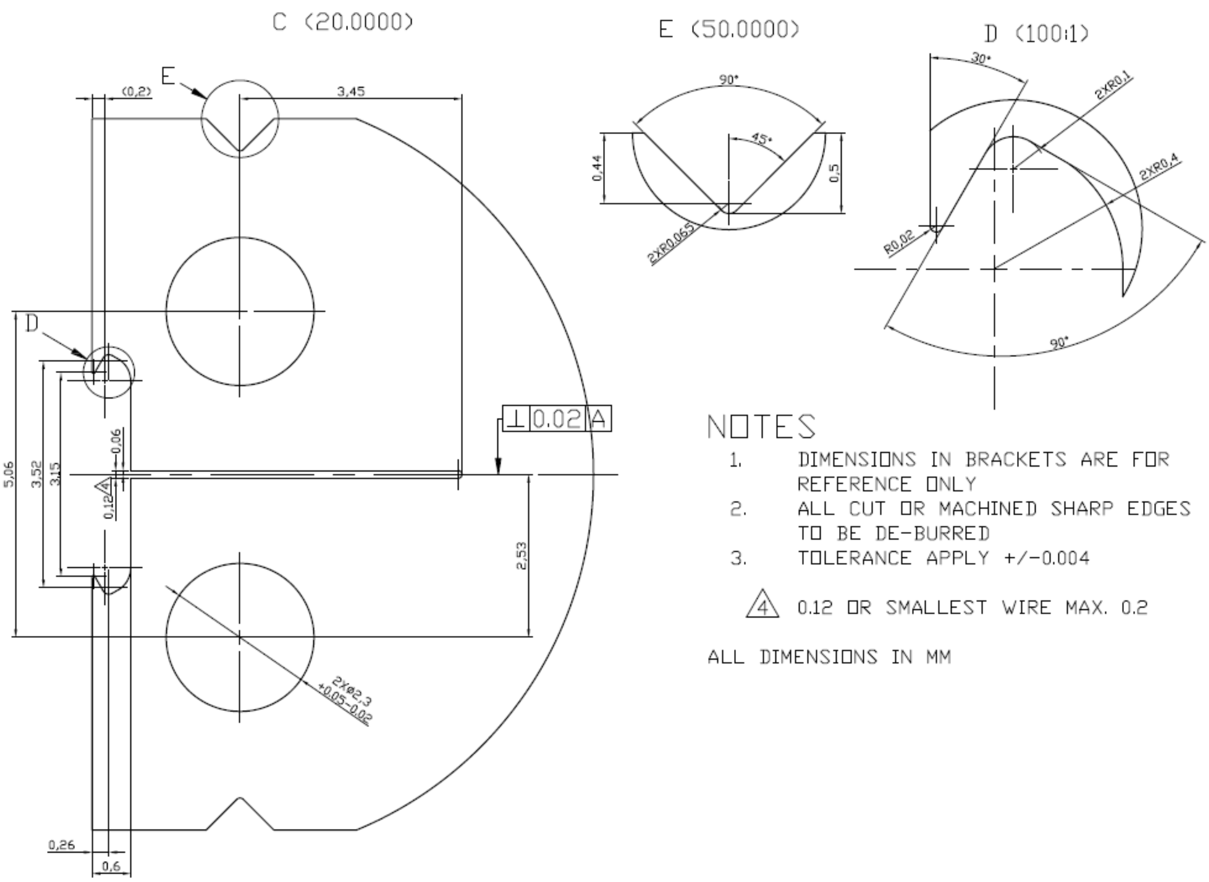


(b)

Fig. 7 UKAEA miniCT specimen drawing: (a) general layout, (b) details of the drawing



(a)



(b)

Fig. 8 UKAEA miniCT-DONES specimen drawing based on the proposed DONES mCT with W=9.2mm: (a) general layout, (b) details of the drawing

3. Testing Procedures

3.1 Fatigue precracking

Main specimen dimensions, such as B (specimen thickness) and W (specimen width), should be measured from each specimen before fatigue precracking. The precracking can be conducted under either force control, displacement control, or K (stress intensity) control. The load cell and displacement gage for specimens shall be calibrated to related ASTM standards or other recognized national or international standards. Fatigue cycling is conducted using a sinusoidal waveform and a frequency close to the highest practical value of the test frame. Precracking is generally most effectively conducted using $R = P_{min}/P_{max} = 0.1$. There are four main requirements need to be fulfilled for fatigue precracking:

1) The maximum fatigue force is less than P_m (control force) as defined in the equations below:

For SE(B) specimens, $$P_m = \frac{0.5 B b_0^2 \sigma_Y}{S} \tag{1}$$

For C(T) and DC(T) specimens, $P_m = \frac{0.4Bb_0^2\sigma_Y}{2W + a_0}$ (2)

where:
b_0 is the initial uncracked ligament and equals W-a_0 (initial crack size),
σ_Y is the effective yield strength and is calculated as the average of the 0.2% offset yield strength σ_{YS} and the ultimate tensile strength σ_{TS},
S is the distance between specimen supports for SE(B) specimens.

2) The maximum stress intensity during fatigue precracking shall fulfill the requirements in Fig. 6 and Table 1 in the E1921-19. This requirement defines the allowable window for the applied stress intensity during fatigue precracking.

3) The initial crack size after fatigue precracking, a_0, shall be 0.5W ± 0.05W for compact, disk-shaped compact, and single-edge notched bend specimens.

4) The crack extension requirements in Fig. 5 and Table 2 in the E1921-19 shall be fulfilled at each of the nine measurement locations defined in section 8.8.1 of the E1921-19.

Side grooves after fatigue precracking are optional however side-grooving will decrease the curvature of the initial crack front. The total side-grooved depth shall not exceed 0.25B. Side grooves with an included angle of 45° and a root radius of 0.5 ± 0.2 mm are recommended per E1921-19 section 7.7.

3.2 Fracture toughness testing

3.2.1 Apparatus

Qualified grips and fixtures described in E1921-19 sections 6.2 and 6.3 shall be used in fracture toughness testing. The displacement gauge, load cell, and temperature measurement devices shall be calibrated to related ASTM standards or other recognized national or international standards. One recommended temperature monitoring method is to weld or spot weld each thermocouple wire separately to the specimen.

3.2.2 Loading rate

Quasi-static loading rate such that dK/dt during the initial elastic portion is between 0.1 and 2 MPa√m/s shall be used. Table 4 in E1921-19 provides a proper load-line loading rate which should be applied in testing as a function of dK/dt and a/W. Prior to testing, it is necessary to verify that the specimen is properly seated into the loading device and that the clip gage is properly seated. This can be achieved by estimating the specimen crack size while working-in the test setup at the test temperature. Working-in is accomplished via repeated preloading and unloading in the linear elastic range at least three times. For each unloading/reloading sequence, estimate the precrack size using the compliance equations in the ASTM E1820 standard. The elastic modulus, E, used in the crack size estimation should be the nominal value for the material at the test temperature, which shall come from either handbook values or dedicated modulus testing per ASTM test method E111 or equivalent. Alternatively, the following equation can be used to determine the nominal value of E at the test temperature:

$$E = 204 - T/16 \text{ GPa} \quad (3)$$

where T = test temperature in °C. The set setup is considered acceptable when the last three consecutive estimated crack sizes are all within 10% of the final precrack size and no individual estimated crack size differs from the mean by more than ±0.002W. To minimize the difference between the precrack size and the working-in estimated crack size, the nominal E value may be adjusted up to 10%.

3.2.3 Test temperature selection

Test temperature T should be chosen such that the medium stress intensity factor $K_{JCc(med)}$ at the test temperature will be about 100 MPa√m for the specimen size selected. For small size specimens, this may not be achievable due to the maximum K_{Jc} capacity limit ($K_{Jclimit}$) defined in Eq. (4). Hence, lower testing temperatures are necessary. For both cases, Table 5 of the E1921-19 shall be consulted to assist the test temperature selection such that $\sum_{i=1}^{3} r_i n_i \geq 1$ where r_i is the number of uncensored data within the i-th temperature range of $(T-T_0)$ and n_i is the specimen weighting factor for the same temperature range in Table 5. In the extreme case (e.g. miniature bend bar testing), the test temperature may be more than 50°C lower than T_0 and the testing temperature is selected by balancing between obtaining as high fracture toughness results as possible and still within $K_{Jclimit}$.

$$K_{Jclimit} = \sqrt{\frac{E b_0 \sigma_{YS}}{30(1-\nu^2)}} \tag{4}$$

where:
σ_{YS} is the 0.2% offset yield strength at the test temperature and can be determined based on E1921-19 section 7.5,
ν is the Poisson's ratio.

3.2.4 Pop-in evaluation

During testing, if pop-in is experienced, the pop-in evaluation according to section 9.2 in the E1921-19 shall be performed to determine if the pop-in event is considered as significant. If so, the specimen shall be considered at the significant pop-in.

3.3 Post-test crack length measurements

Upon completion of fracture toughness testing, measurements on specimen fracture surface shall be performed to obtain the initial crack size, a_0, and the ductile crack growth, Δa, where applicable. If the specimen failed in cleavage, the two halves of the specimen can be easily separated by the test frame and the crack length measurement follows. If the test finished without cleavage fracture from the specimen, either fatigue loading to create beach marks shall be performed before break the specimen or loading the specimen at liquid nitrogen temperature can be used to separate two halves of the specimen. This will make sure the fracture surface is preserved for the crack size measurement. The crack size should be measured at nine equally spaced points centered about the specimen centerline and extending to 0.01B from the free surfaces of plane sided specimens or near the side groove roots on side grooved specimens. Average the two near-surface measurements and combine the average of these two readings with the remaining seven crack measurements. Determine the average of those eight values.

3.4 Fracture toughness calculation, validity check, and censoring

Force-displacement data shall be used to calculate K_{Jc} (elastic-plastic equivalent stress intensity factor derived from the J-integral at the point of onset of cleavage fracture) based on procedures outlined in section 9.1 of E1921-19. The K_{Jc} datum shall be considered invalid if any of the nine physical measurements of the starting crack size differ by more than $0.1(b_0B_N)^{1/2}$ (B_N is the net thickness) from the average starting crack size. The datum is also invalid if the calculated crack length for the test determined in E1921-19 section 8.8.2 differs from the optical average value determined in section 8.8.1 by more than 5%. For valid K_{Jc} datum, if it exceeds maximum K_{Jc} capacity limit $K_{Jclimit}$ and/or slow stable crack growth K_{Jc} limit $K_{Jc\Delta a}$, the datum will be censored based on procedures in E1921-19 section 8.9.2. If both $K_{Jclimit}$ and $K_{Jc\Delta a}$ are violated, the lower value of the two shall be used to replace the K_{Jc} datum for data censoring purposes in the analysis. Once the provision reference temperature T_{0Q} is calculated, follow procedures in E1921-19 section 10.5 to check if T_{0Q} can be validated as the Master Curve reference temperature T_0.

4. Recording

All the testing parameters and results should be input into the excel spreadsheet template entitled "IAEA SSTT Master Curve record sheet."

5. Acknowledgements

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6. References

[1] ASTM E1921-19b^{e1}, Standard Test Method for Determination of Reference Temperature, To, for Ferritic Steels in the Transition Range, ASTM International, West Conshohocken, PA, 2019, www.astm.org