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FRACTURE TOUGHNESS DETERMINATION OF EUROFER97 BY TESTING SMALL CT SPECIMENS

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ABSTRACT

The use of small specimen test techniques (SSTT) to determine mechanical properties of irradiated materials has been studied over the past decades both in fission and fusion programs, but also to characterise and optimise new materials by nuclear and non-nuclear communities. Currently a number of activities are running that focus on the standardisation of SSTT to determine fracture toughness properties for fusion reactor materials (IAEA [1], EUROfusion [2], F4E [3]), and to support the long-term operation of light-water reactors (CRIEPI [3]). The determination of the T_0 *reference temperature (ASTM E1921 [4]) has been successfully determined by testing small specimens (W=8mm, B=4mm) of non-irradiated and irradiated pressure vessel materials. However, some concerns exist regarding to the use of the Master Curve on ferritic-martensitic steels, not only with SSTT but also with 1T CT specimens. The main concern is the slope of the MC [7, 8], that seems to be steeper than the standard one. In this paper, the fracture toughness of Eurofer97 has been obtained by testing small CT specimens with the geometry selected in IFMIF-DONES (W=9.2mm, B=4.6mm) in the transition region.* T_0 *has been determined and compared to the one obtained from 0.5T CT specimens. The scatter of the results has also been assessed to validate the scatter description of the MC.*

Keywords: Eurofer97, Fracture Toughness, Master Curve, Small Specimen Testing Technique, Fusion

1. INTRODUCTION

Nuclear materials can be exposed to extreme working conditions, including large temperature changes and high neutron irradiation fluence, which can lead to the embrittlement of the material, among other effects. One of the main parameters to evaluate this effect is the reference temperature, T_0, of the Master Curve (MC) approach described in the standard ASTM E1921 [1]. This approach is a combination of mechanistic modelling and a statistical approach that allows the characterization of the ductile to brittle transition region with a reduced number of tests, taking into account the effect of thickness of the test sample and the scatter of the results in this range of temperatures [2].

The MC approach is usually applied by testing conventional fracture toughness specimens, which require relatively large volumes of material. Given the current needs of the nuclear programs to reduce the size of the specimens, such as for the test modules of IFMIF-DONES (International Fusion Materials Irradiation Facility – DEMO-Oriented NEutron Source) [3] or for the life extension of nuclear plants, several programs have focused their efforts on the application of small specimen test techniques. For instance, CRIEPI organized a round-robin on reactor pressure vessel (RPV) steels that successfully estimated T_0 with miniature compact tension specimens (miniCT) (0.16T CT) [4].

In this work, the applicability of miniCT´s in combination with the MC approach has been evaluated on Eurofer97, the European reference reduced activation ferritic-martensitic (FM) steel for the first wall and blanket applications of the future fusion reactor DEMO (DEMOnstration power plant) [5]. T_0 has been determined with miniCT specimens and compared to the value obtained with conventional 0.5T CT specimens [6]. The scatter of the results and the slope of the MC have also been assessed to validate the application of the MC to Eurofer97, given the concerns of previous works on the fit of the shape of the MC for FM steels, mainly for its slope [7, 8].

2. MATERIALS AND METHODS

The tested Eurofer97 was provided by the Karlsruhe Institute for Technology (KIT) in the form of a 5 mm thick plate, produced by Bohler Bleche GMBH. After normalizing at 980º C, the plate was tempered at 760º C for 90 minutes. The composition is detailed in TABLE 1. The Young's modulus of the material, E (GPa), has been represented by Equation (1), proposed in the ASTM E1921 standard [1]. The yield stress, σ_y

(MPa), is represented as a function of temperature by Equation (2), as proposed in [9].

$$E = 204 - \frac{T}{16} \quad (1)$$

$$\sigma_y = 503.89 + 38.98e^{(-0.0149\,T)} \quad (2)$$

where T represents the temperature in Celsius degrees.

The employed geometry of the miniCTs is the proposed geometry for the test modules of IFMIF-DONES, as shown in FIGURE 1 [3]. The specimen width, W, is 9.2 mm and the thickness, B, is 4.6 mm. The length of the wire-cut slit is 6.7 mm.

A servo-hydraulic testing rig of 100 kN of capacity with a chamber cooled with liquid nitrogen down to -150° C has been employed. The pre-cracking of the specimens has been performed according to the recommendations of ASTM E1921 [1] with a target value of allowed fatigue crack growth of 0.60 mm. A total number of 13 tests have been performed to obtain the Master Curve reference temperature.

Initially, some samples were tested without side grooves on the specimens. A high number of tests with excessive ductile crack growth in the temperature testing range were obtained, which had to be discarded. Given the difficulties in testing these specimens, side grooves were employed for the rest of the specimens, trying to avoid excessive ductile crack growth. The side grooves have been machined on the specimens after precracking, following the ASTM E1921 [1] recommendations and applying the geometry shown in FIGURE 1.

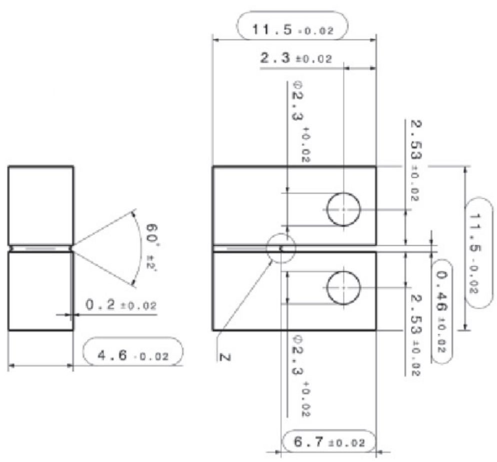

FIGURE 1: GEOMETRY OF THE MINICT SPECIMENS [3].

TABLE 1: CHEMICAL COMPOSITION OF THE EUROFER97 STEEL, UNITS IN WT%

Cr	W	Si	Mn	Ti	O	C	Fe
8.95	1.06	0.031	0.55	0.001	0.0007	0.11	Bal.

3. RESULTS AND DISCUSSION

To assess the influence of the side grooves and of the testing temperature, the results have been presented separately according to the following criteria: i) Application of the MC approach on results obtained from specimens with side grooves or specimens with and without side grooves, ii) Application of the MC approach on valid data or valid and censored data.

3.1. Specimens with side grooves

3.1.1. Valid and censored data

The 1T-adjusted fracture toughness results obtained from the tested specimens with side grooves are shown in FIGURE 2. The obtained reference temperature, -134° C, is in good agreement with the value obtained from 0.5T CT specimens, -129° C [6]. It can also be seen that the 2% and 98% tolerance bounds fit the scatter of the results. However, the 5% tolerance bound does not fit the scatter, nor does the mean distribution, being most of the results below the mean value in the proximity of the lower tolerance bounds.

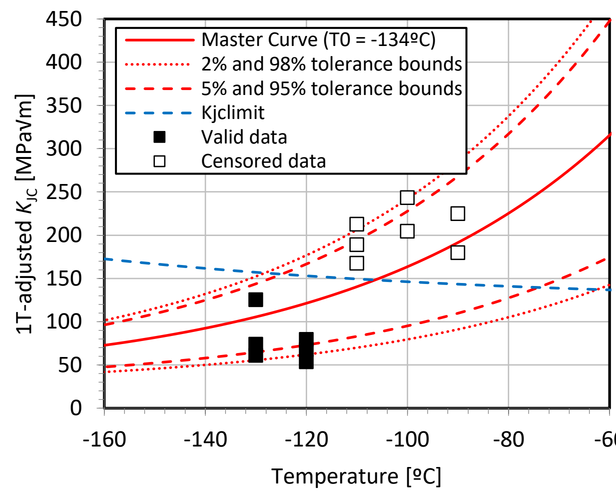

FIGURE 2: MASTER CURVE FRACTURE TOUGHNESS RESULTS FOR SIDE GROOVED SPECIMENS FROM VALID AND CENSORED DATA.

3.1.2. Valid data

The 1T-adjusted fracture toughness results that meet the $K_{JClimit}$ and $K_{Jc\Delta a}$ requirements obtained from specimens with side grooves are shown in FIGURE 3. These tests provided a more conservative T_0 value (-116° C) compared to the value obtained from valid and censored data (-134° C) and to the value obtained from 0.5T CT specimens (-129° C) [6]. In this case, the 2% and 98% tolerance bounds, as well as the 5% and 98% ones, fit accurately the scatter of the data. The distribution with regards to the mean value is not satisfied, being most of the data below the mean value, although closer to it than when valid and censored data are considered.

In addition, it is important to note that in this case all the performed tests are below the obtained T_0 value. As a result, the use of valid data in this case could be considered equivalent to estimating the reference temperature by testing below T_0.

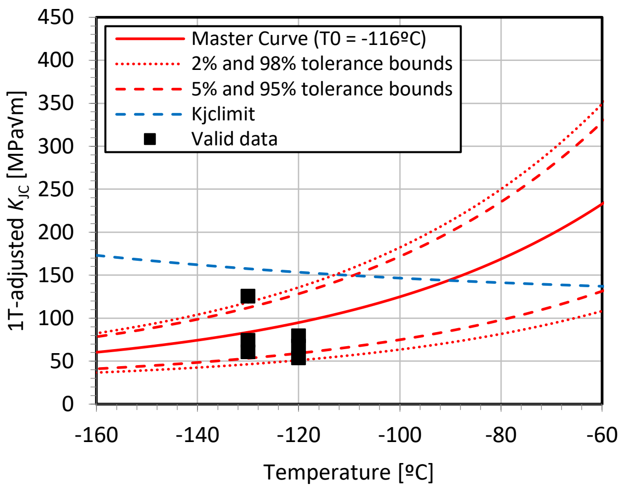

FIGURE 3: MASTER CURVE FRACTURE TOUGHNESS RESULTS FOR SIDE GROOVED SPECIMENS FROM VALID DATA.

3.2. Specimens with and without side grooves

3.2.1. Valid and censored data

The 1T-adjusted fracture toughness results obtained from all of the tests performed, with and without side grooves, is shown in FIGURE 4. The addition of the specimens without side grooves to the data set does not influence the obtained T_0 (-134º C) or the fit of the tolerance bounds to the scatter of the results.

3.2.2.Valid data

The 1T-adjusted fracture toughness results obtained from the tests performed, with and without side grooves, that meet the $K_{JClimit}$ and $K_{Jc\,\Delta a}$ requirements is shown in FIGURE 5. The addition of the specimens without side grooves to the data set does not seem to have a significant influence on the obtained T_0, leading to slightly more conservative results (-112º C). It does not have any effect on the fit of the tolerance bounds to the scatter of the results.

In this case again, all the performed tests are below the obtained T_0 value, with one exception. As a result, the use of valid data in this case could be considered equivalent to estimating the reference temperature by testing below T_0 too.

3.3. Discussion

From the results obtained in this case of study, the following results can be derived:

- Statistically, the use of only valid data is equivalent to testing below T_0 and the use of valid and censored data is equivalent to testing above and below T_0. This effect is related to the size of the specimens employed and its influence on $K_{JClimit}$. Due to this effect, the ASTM E1921 standard [1] recommends testing below T_0 when testing small specimens.
- Evaluations with valid and censored data are closer to the T_0 obtained with 0.5T CT specimens. Evaluations with only valid data lead to conservative results (with a difference of ~20ºC). This suggests that testing below T_0 might lead to conservative results when using miniCT´s on FM steels. Further research is required to better understand this phenomenon.
- All evaluations seem to have a good distribution with 2% and 98%. Evaluations with only valid data have a good distribution with the 5% and 95% tolerance bounds too. None of the evaluations have a good distribution with regards to the mean value. This effect is in good agreement with previous works on the fit of the shape of the MC for FM steels, mainly for its slope [7, 8]. Its influence will be further analyzed in the next section.
- The use of side grooves seems to reduce the scatter of the results. It does not seem to have any effects on evaluations with valid and censored data, but it might lead to relatively more conservative results when testing below T_0. Further testing is required to analyze this effect.

4. INFLUENCE OF THE SHAPE OF THE MASTER CURVE

Given that the MC does not seem to satisfactorily describe the obtained results and given the concerns of previous authors on the shape of the MC for FM steels [7, 8], the shape of the MC has been further analyzed. In this case, the influence of the parameter, C, shown in Equation (3) and which represents the slope or the steepness of the transition of the MC, has been evaluated.

$$K_{\mathrm{Jc(med)}} = 30 + 70\, e^{C(T-T_0)} \qquad (3)$$

where $K_{Jc(med)}$ is the mean value defined by the MC in MPa√m.

T_0 has been determined in all cases for values of C in the range 0.01 to 0.05, assessing its influence on the reference temperature obtained and on the fit of the valid data with regards to the mean value and to the tolerance bounds. To be able to represent the results, shown in FIGURE 6 and FIGURE 7, three different criteria have been established: i) Fit of the results to the inferior tolerance bound, ii) Fit of the results to the superior tolerance bound, iii) Fit of the results to the mean value. The evaluations with different C values have been classified depending on the number of criteria that are satisfied. The tolerance bounds of 5% and 95% and 2% and 98% have been evaluated, as it can be seen in FIGURE 6 and in FIGURE 7, respectively.

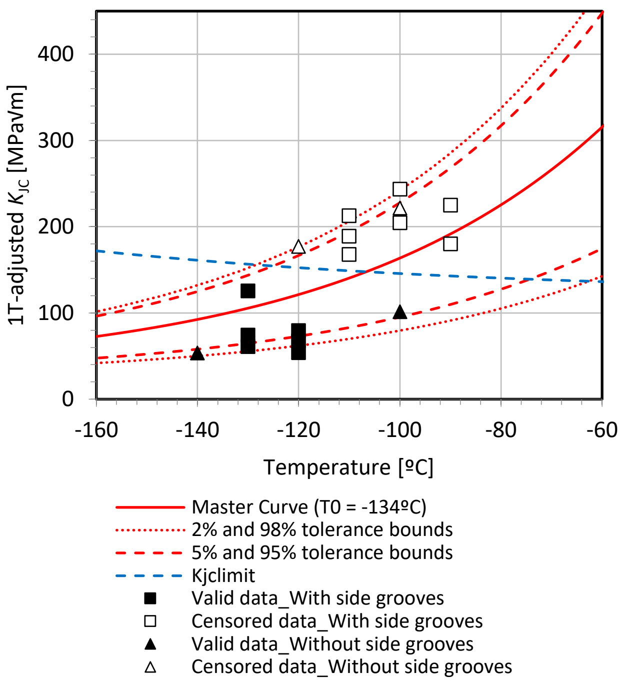

FIGURE 4: MASTER CURVE FRACTURE TOUGHNESS RESULTS FOR SPECIMENS WITH AND WITHOUT SIDE GROOVES FROM VALID AND CENSORED DATA.

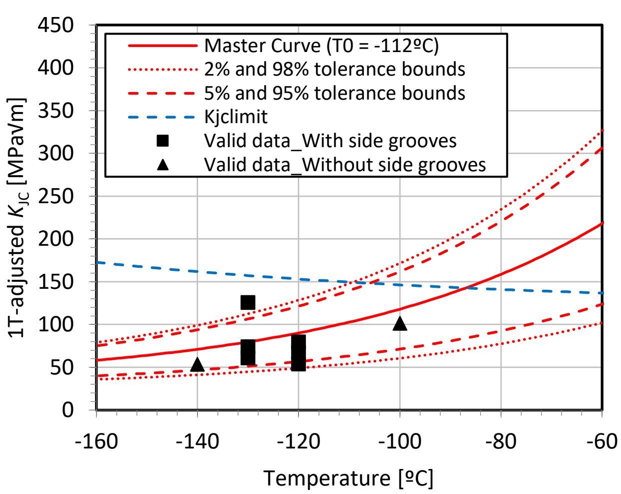

FIGURE 5: MASTER CURVE FRACTURE TOUGHNESS RESULTS FOR SPECIMENS WITH AND WITHOUT SIDE GROOVES FROM VALID DATA.

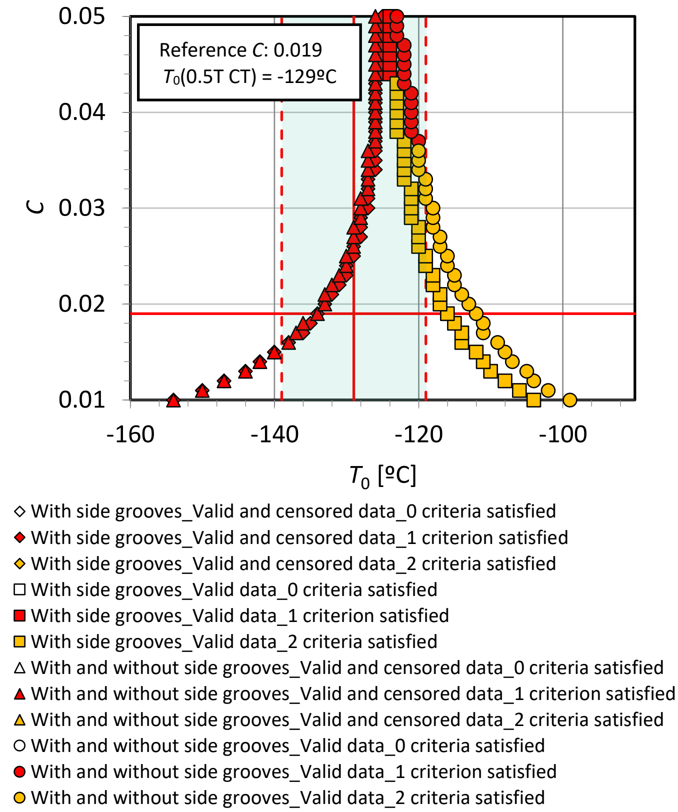

FIGURE 6: ANALYSIS OF THE INFLUENCE OF THE SLOPE ON THE MASTER CURVE FOR SIDE GROOVED SPECIMENS. TOLERANCE BOUNDS APPLIED: 5%, 95%.

From the performed analysis, the following observations can be made:

- For higher values of C, the obtained T_0 values seem to converge to a unique value around -125º C, regardless of the use of valid or censored data or the use or lack of side grooves. The obtained value is within ±4º C of the value obtained with 0.5T CT´s.
- Regardless of the value of C, side grooves do not seem to influence the estimations obtained with valid and censored data. Evaluations with only valid data are influenced by side grooves, leading to more conservative results when specimens without side grooves are considered.
- Regardless of the value of C, none of the data sets are satisfactorily described by the mean value.
- 2%, 5%, 95% and 98% tolerance bounds fit the scatter of the results of the valid tests up to values of C of 0.036. If valid and censored data are considered, 95% and 98% tolerance bounds fit the scatter of the results independently from the C values, but the 2% tolerance bound only exhibits good fits for values of C in between 0.015 and 0.037.

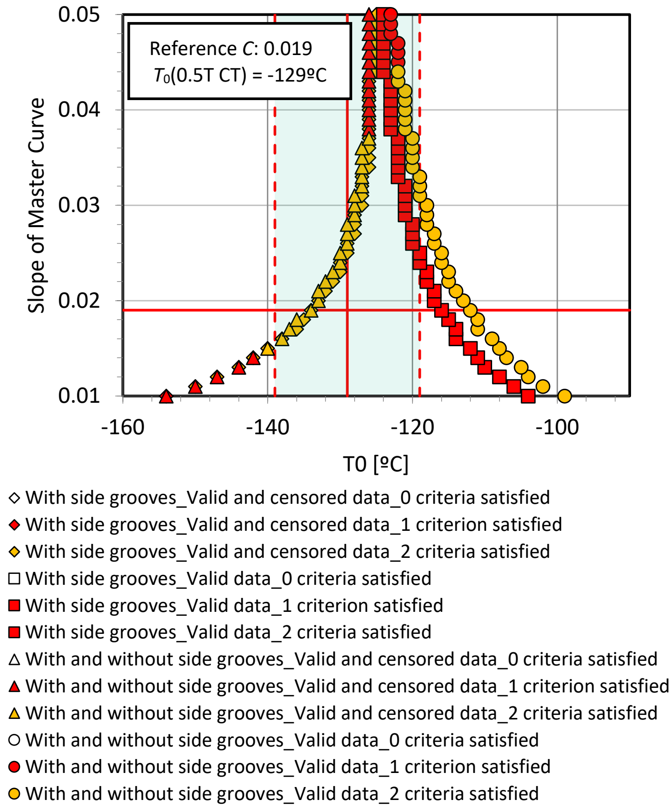

FIGURE 7: ANALYSIS OF THE INFLUENCE OF THE SLOPE ON THE MASTER CURVE FOR SPECIMENS WITH AND WITHOUT SIDE GROOVES. TOLERANCE BOUNDS APPLIED: 2%, 98%.

- The determined T_0 values are within ±10º C from the value obtained from 0.5T CT's for values of C greater than 0.016. and 0.031 for valid data and valid and uncensored data, respectively.

Briefly, the use of C values greater than 0.031 seem to provide results within ±10º C from the value obtained from 0.5T CT's. The use of values in between 0.031 and 0.036 seem to provide the best fit to the tolerance bounds. As a result, it seems that the C values that exhibit the most satisfactory description of the obtained results are in the range of 0.031-0.036. Consequently, for the data set analysed, the use of Equation (4) is advised for Eurofer97 with miniCT´s, applying a value of C = 0.034 instead of 0.019 as proposed by ASTM E1921 for RPV steels [1]. The use of 0.019 would lead to conservative results for tests performed below T_0, although exhibiting a good fit to the 2%, 95% and 98% tolerance bounds.

$$K_{\mathrm{Jc(med)}} = 30 + 70\, e^{0.034(T-T_0)} \tag{4}$$

Further research is required to better analyse the shape of MC for FM steels. The influence of the athermal part of the curve (30 and 70 in Equation (4)) needs to be assessed to try to achieve a better distribution of the results with $K_{\mathrm{Jc(med)}}$.

5. CONCLUSION

The Master Curve approach has been successfully applied to evaluate the reference temperature of Eurofer97 using the miniature CT specimens proposed for the test modules of IFMIF-DONES. The influence of the testing temperature, the use of side grooves and the shape of the MC have been analyzed. The following conclusions can be drawn from the analysis:

- Influence of side grooves:
  - MiniCT specimens without side grooves seem to have a greater scatter.
  - Their use does not seem to influence the results obtained from valid and censored data, although they seem to lead to more conservative reference temperatures if only valid data are evaluated.
  - Further research is required to better understand their effect.
- Influence of testing temperature:
  - Statistically, for the data set analyzed, testing below T_0 is equivalent to only using valid data.
  - Estimations only using valid data lead to more conservative results, with a difference of ~20º C for a C value of 0.019.
- Influence of the shape of the MC:
  - Higher values of C seem to converge to a unique T_0 value, within ±5º C of the T_0 obtained with 0.5T CT´s.
  - For the analyzed data set, the use of C = 0.034 is advised to achieve results within ±10º C of the T_0 obtained with 0.5T and a good fit to 2%, 95% and 98% tolerance bounds.
  - If C = 0.019 is applied, the 2% tolerance bound exhibits a good statistical fit.

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