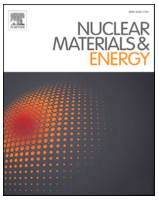
Nuclear Materials and Energy 39 (2024) 101679

Contents lists available at ScienceDirect

Nuclear Materials and Energy

journal homepage: www.elsevier.com/locate/nme



Study of small tensile specimen thickness effects in copper oriented for IFMIF-DONES irradiation

N. García-Rodríguez [*], F. Canillas, F. Arranz, M. Roldán, F. Sánchez, E. Leon-Gutierrez

CIEMAT, Avda, Complutense, 40,28040 Madrid, Spain

ABSTRACT

Small specimen testing technology (SSTT) refers to the examination of tiny samples of materials to evaluate their suitability for use in fusion reactors. This includes assessing their thermal, mechanical, and radiation properties, as well as their ability to withstand extreme conditions. SSTT plays a critical role in the development of nuclear fusion technology, which holds the potential to provide a virtually limitless and clean energy source. SSTT is imperative in cases where not much material is available or must be subjected to special conditions that limit it.

One of the main challenges in nuclear fusion is the development of materials that can withstand the harsh environment inside a fusion reactor. These materials must be able to withstand high temperatures, intense radiation, and mechanical stress, and be able to maintain their structural integrity for long periods. Small specimen testing allows characterizing and evaluating these properties in a controlled and efficient manner.

Small specimen samples, under conditions similar to those in a fusion reactor, will provide crucial information for the design and development of fusion components when irradiated at IFMIF- DONES. The results of SSTT are used to refine materials, optimize production processes, and develop new materials with improved properties.

In this work, numerous tensile tests (SSTT) have been carried out on pure copper, a material of interest for fusion with different thicknesses, analyzing the effect of the variation of this variable in the determination of mechanical properties.

Introduction

The process of electroforming allows for the creation of small, intricate components that are challenging to fabricate using other methods. This aspect is crucial for the IFMIF-DONES project, where space for irradiation is limited, and the demand for miniature samples is high.

Particularly, electroformed pure copper, with its capacity to be shaped into complex geometries and its inherent low impurity levels, emerges as an ideal candidate for components in fusion and particle accelerator devices, such as those in the IFMIF-DONES project. The technology's ability to produce fine-grained copper with excellent mechanical properties and high thermal conductivity is particularly beneficial for applications that require both structural integrity and efficient heat dissipation.

In the realm of materials research, the study of small specimens is gaining increasing importance across various fields. Some examples are listed here: (1) samples obtained from in-service equipment [1,2], (2) specimens suitable for scanning and transmission electron microscopy [3], (3) nuclear fusion research with small volume available for irradiation [4,5] and (4) geometries leading to stress distributions more representative of operating conditions [6]. In this work, special attention is given to the type of specimens that will be used for irradiation in IFMIF-DONES. However, such geometries and sizes can find diverse applications across various fields.

Comparison of tensile test results obtained with small specimens and standarized size specimens help to assess how accurate the extrapolation of the measured values is; some authors have done such comparison for standard cylindrical specimens [1] as well as for planar specimens [3].

The material properties that are useful from the mechanical engineering point of view include both strengths parameters such as the ultimate tensile strength (UTS) and yield strength (YS), and ductility parameters, such as elongation and area reduction. Previous research indicated that the ultimate tensile strength (UTS) and yield strength (YS) of thin steel specimens were observed to be consistent with those of bulk materials. However, it was noted that the thickness of the material had a significant impact on the total elongation [7,8].

The value of thickness seems to have a threshold below which it has some influence on UTS and YS values, such threshold is estimated in 0.2 mm [2] or 0.18 mm [3] for small specimens. The explanation for such influence is the small long-range back stress field existing in very thin specimens. Some authors point out the influence of thickness in the

---

[*] Corresponding author.
E-mail address: Nerea.garcia@ciemat.es (N. García-Rodríguez).

https://doi.org/10.1016/j.nme.2024.101679
Received 27 December 2023; Received in revised form 6 May 2024; Accepted 16 May 2024

value of total elongation for both standard size specimens [9] and small specimens [10], having larger total elongation values for bigger thicknesses, explained for the necking geometry that presents notable deformation only in thickness direction for very thin specimens. Little values of thickness are also reported to lead to bigger variability in measured total elongation values [1,11] justified by relatively big impact of differences in machining and lack of uniformity in dimensions.

The width of the specimens seems to have very little influence if any, in the results of the tensile tests [12], although usually is not of interest the use of extremely little values of width and therefore not many experiments have been carried out in this direction. A study conducted by Sergueeva et al. [13], determined that the outcomes of tensile tests are influenced by not only the inherent properties of the material and the testing parameters (such as strain rate and temperature) but also by the size and shape of the test specimens. The study reiterated that there was no discernible impact of the gauge length on the ultimate tensile strength (UTS) and yield strength (YS). However, a substantial reduction in both uniform and total elongation was observed in thin sheets as the gauge length-to-width ratio increased.

The present work aims to investigate the effect of thickness on the tensile properties of materials of interest for fusion, with a focus on future studies of irradiated material at the IFMIF-DONES facility. In particular, the study has focused on measuring specimens of electroformed pure copper with small geometries.

Materials and experimental procedure.

The material used in this research work was manufactured using the novel method of electroforming, obtaining a piece with a total thickness of 48 mm. Chemical analysis was conducted by performing elemental combustion analysis, plasma spectroscopy and flame emission. It was determined that all the impurities examined were below detected the threshold considered in Table 1 [14]. The specimens were then precision-machined using wire electrodischarge machining, aligning with the planes parallel to the electroforming surface. Notably, the specimen surfaces were left in their original state without grinding only cleaned removing the machining fluids. The measured average roughness was found to be Ra = 2.6 μm. This is nearly three orders of magnitude greater than the smallest thickness among the tested specimens.

The fine grain structure of electroformed pure copper contributes to its mechanical robustness, making the material's properties less dependent on the specimen size. This feature is particularly advantageous in the fabrication of miniature samples for irradiation, as it ensures consistent material behavior regardless of the sample's dimensions.

The microstructure of the specimens, both in their as-received state and after testing, was analyzed using a Field Emission Scanning Electron Microscope (FESEM), specifically a Zeiss Auriga with an in-lens detector, located at CIEMAT in Madrid. Additionally, for a more comprehensive preliminary investigation, a Scanning Transmission Electron Microscope (STEM) was employed. This microscope, a JEOL 3000F equipped with an ADF detector, is housed at CNME, Madrid. The sample preparation followed conventional metallographic techniques, culminating in electropolishing using a Struers Lectropol device with D2 electrolyte at a temperature below room temperature. For STEM analysis, the samples were sectioned and thinned to a thickness of approximately 100 μm, with the final thinning carried out using a Struers Tenupol device employing the same electrolyte.

Table 1
Results of chemical analyses. All analysed impurities are below the detection level.

Elements	Detection level
Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Fe, Mg, Mn, Mo, Ni, Pb, Sb, Se, Si, Sr, Ta, Te, Ti, V, Zn, Zr	6 ppm
Bi, K, Na	20 ppm
C_total, S_total	0.01 %
P	200 ppm

The specimen's geometry can be observed in Fig. 1. Table 2 shows the different labelled of each specimen in relation with the width (W) and the thickness (T).

Samples were mechanically tested on a screw-driven machine Kammrath & Weiss Type MZ0-1 with displacement controlled by LVDT and a 10 kN Load Cell. Five samples for each dimension were tested for the evaluation of the test reproducibility.

The following mechanical parameters were obtained by using the engineering stress-engineering strain curve: (a) Yield Strength (YS) obtained drawing a parallel line to the straight part of the curve at a distance of 0.2 % in elongation axis; (b) Ultimate Tensile Stress (UTS), as the maximum stress value in the curve, and c) the strain to failure (ε_f) as elongation reach when specimen fails.

Results

The material underwent electropolishing and etching using D2 Struers electrolyte at a reduced voltage, aimed at selectively targeting the grain boundaries, as depicted in Fig. 2 (left image). Subsequent analysis using Scanning Transmission Electron Microscopy (STEM) revealed a distinct face-centered cubic (FCC) morphology characterized by twin boundaries. However, the presence of dislocations was not prominently observed, as can be seen in Fig. 2 (right image). The microstructural examination of the pre-tested samples, as shown in Fig. 2, indicated an average grain size of 0.93 ± 0.53 μm. This grain size is considered optimal for replicating bulk material behavior in the tested specimens. Furthermore, the specimens in this study met the minimum volume criteria for the gauge section, ensuring that the data obtained are comparable with those from standard test specimens.

In Fig. 3 one curve was represented for each geometry. Each represented curve correspond to the average of the five tests under the same conditions.

In Fig. 4, pictures of the after tested W3T3 and W3T0.2 are shown. SEM images of the same specimens in the failure area are represented in Fig. 5.

Discussion

The obtained results indicate that YS and UTS values seem not dependent of the specimen thickness. It is necessary to note that, although not significantly, the point W3T0.2 does experience a noticeable decrease in UTS value. This is probably due to reaching a threshold thickness.

However, the elongation to fracture increases with increasing thickness due to availability of sufficient volume of material in the gauge section, enabling the specimen to resist fracture until reaching the neck's material volume.

Comparison of specimens with identical width (W3) and different thickness (T3, T1.5, T0.8, T0.4 and T0.2) shows that thinner specimens exhibit less total elongation (Fig. 6), as opposite to UTS values, which are just slightly lower when the thickness is smallest (Fig. 7). Thus, thickness significantly influences specimen ductility.

The effect of total elongation with size specimen can be adjusted by the Bertella-Oliver formula [15]. In particular, the elongation of different specimen dimensions (1,2) can be compared using:

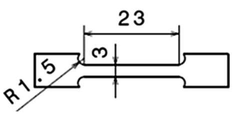


Fig. 1. Geometry of specimens tested (in mm).

Table 2

Nomenclature of specimens tested.

Nomenclature	Width (mm)	Thickness (mm)
W3T3	3	3
W3T1.5	3	1.5
W3T0.8	3	0.8
W3T0.4	3	0.4
W3T0.2	3	0.2
W1.5 T1.5	1.5	1.5
W0.8 T0.8	0.8	0.8

$$e_{f,(1)} = e_{f,(2)} \left(\frac{K_{(2)}}{K_{(1)}}\right)^{n}$$

Where $_{(1,2)}$ is the slimness value, defined as:

$$K_{(1,2)} = \frac{L_{0(1,2)}}{\sqrt{a_{0(1,2)} \cdot b_{0(1,2)}}}$$

$L_{0(1,2)}$ is the initial gauge length, $a_{0(1,2)}$ the thickness, and $b_{0(1,2)}$ the width of the gauge section.

Fig. 8 represents the total elongation as a function of the slimness ratio ($L_0/\sqrt{a_0}\ b_0$). The relationship provided by the Bertella-Oliver formula is only considered valid under the conditions.

$\frac{b_0}{a_0} < 20$ and $K < 25$. In Fig. 7 have been represented the specimens W3T3, W3T1.5, W3T0.8, W3T0.4, where $\frac{b_0}{a_0} = 1$ is all samples and the maximum $K = 21$. A fitting has also been made, providing an exponent of $n = 0.30$. The reduction in total elongation with specimen size, as well as the constancy UTS, is expected [16], even in the case of pure copper [17].

The previous analysis has considered the influence of thickness with a constant width. It is interesting to take into account the potential effect of the width-to-thickness ratio.

Considering the pairs W3T1.5 and W1.5 T1.5, with equal thickness and a factor of 2 difference in width, it can be observed that the values of UTS and YS do not undergo significant changes. The variation in elongation is relatively small, and the point falls within the error bar. This point of maximum elongation has also been represented in Fig. 8.

For the pair W3T0.8 and W0.8 T0.8, the behavior is similar; there are no significant changes observed in the values of UTS and YS. However, there is a considerable decrease in maximum elongation. This point is also represented in Fig. 8. It can be observed that the trend reasonably.

fits the Bertella-Oliver fit. However, in this case, $K > 25$ (28.8), which means that the Bertella-Oliver relationship cannot be directly applied. Nonetheless, the observed behavior does not deviate significantly from the ideal case.

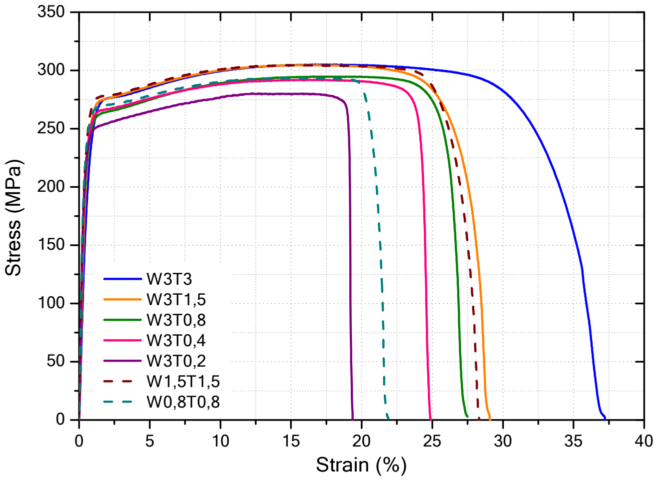


Fig. 3. Average stress–strain curves for specimen tested.

The different ductility is evident in their failure modes. Fig. 4 shows the more ductile specimen (W3T3) exhibiting a typical cup-cone fracture, unlike the specimen with the least elongation at fracture (W3T0.2), which shows limited necking. SEM images (Fig. 5) confirm that both specimens have many grains in the fracture area, validating the test results comparability with standard specimens.

Electroformed pure copper stands out as a highly suitable material for the IFMIF-DONES project, offering the dual benefits of complex shape fabrication and excellent mechanical properties. Its potential for producing small, consistent samples for irradiation, coupled with its high thermal conductivity, makes it an asset in nuclear fusion research. While the question of anisotropy in its mechanical behavior warrants further investigation, the current understanding underscores the material's adaptability and effectiveness in high-demand applications.

Conclusions

Tensile tests have been conducted on electroformed pure copper specimens with small geometries. This type of study is crucial, in some cases, due to the limited availability of material or specific irradiation requirements, particularly in applications related to nuclear fusion. The results obtained for flat specimens ranging from 3 to 0.2 mm in thickness reveal negligible variations in the measured values of Ultimate Tensile Strength (UTS) and Yield Strength (YS).

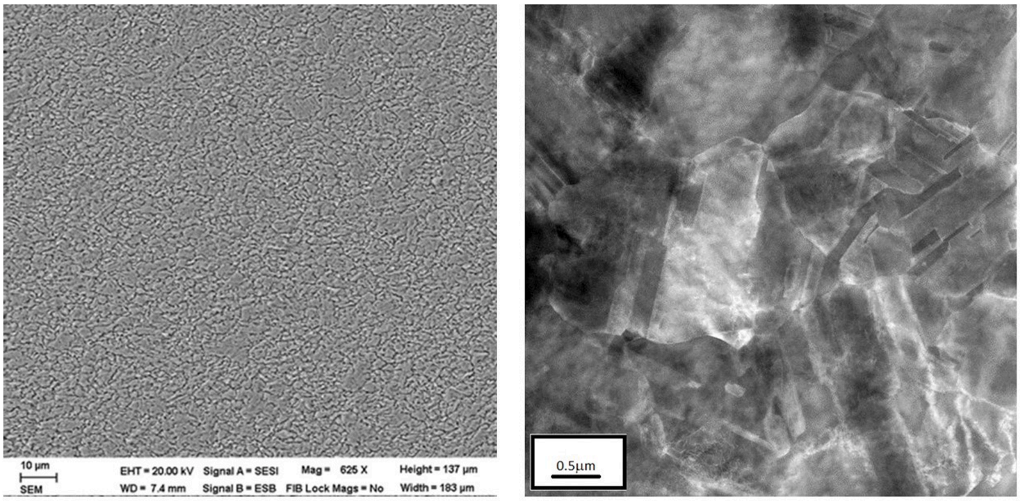


Fig. 2. FESEM micrograph of etcehd surface and (left) STEM ADF image of an as received Cu ELF sample (right).

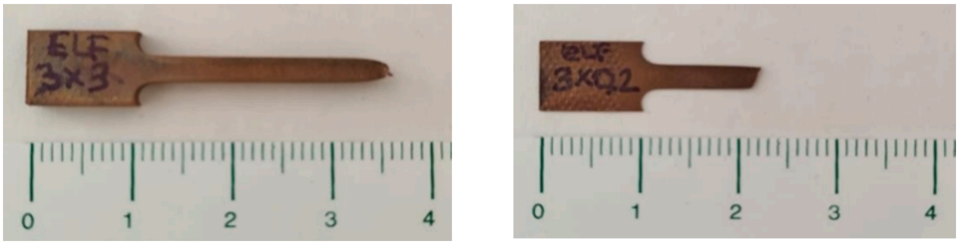


Fig. 4. Pictures of specimens tested, W3T3 (left) and W3T0.2 (right).

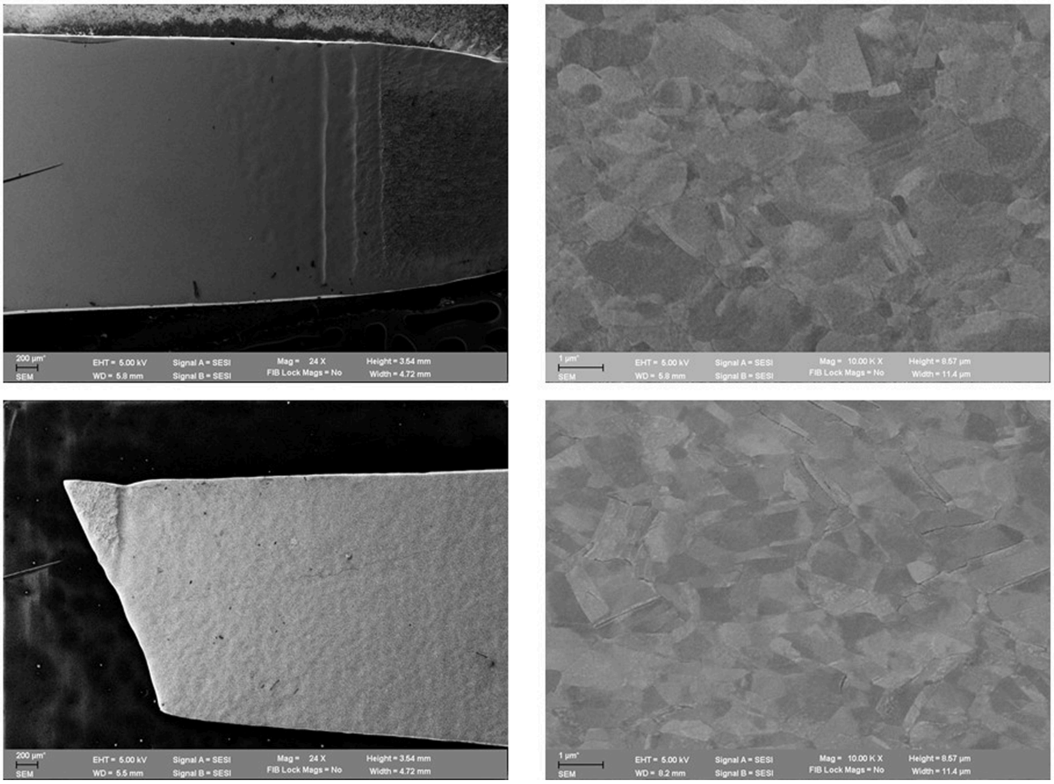


Fig. 5. Images of SEM of specimens tested, W3T3 (above) and W3T0.2 (bottom). The images were taken from the failure area.

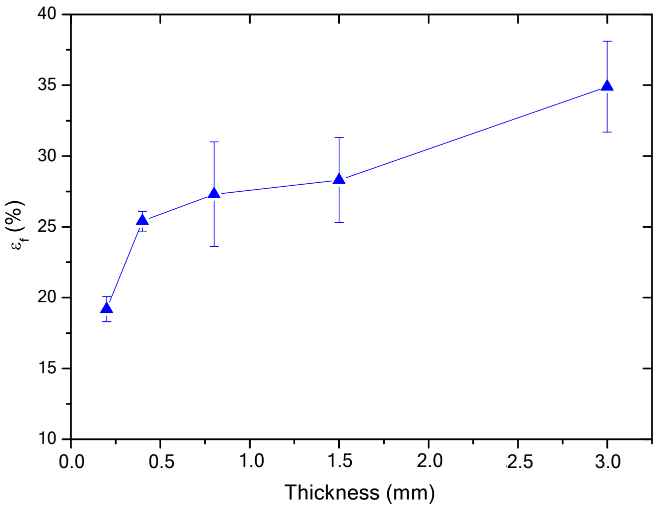


Fig. 6. Comparison of total elongation of specimens W3 type tested with same width but different thickness.

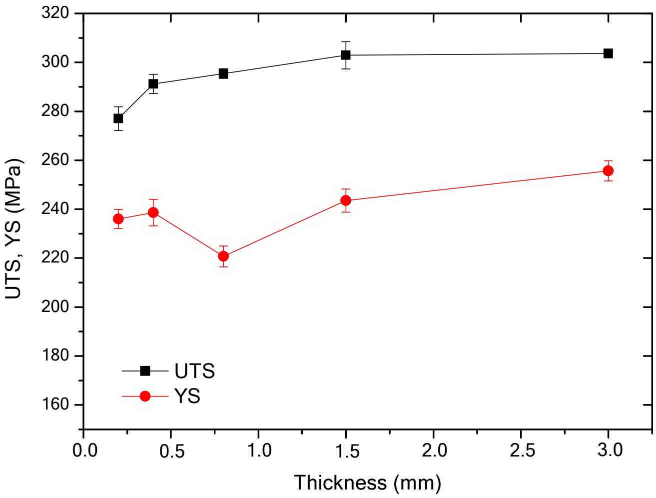


Fig. 7. Comparison of total UTS and YS of specimens tested W3 type with same width but different thickness.

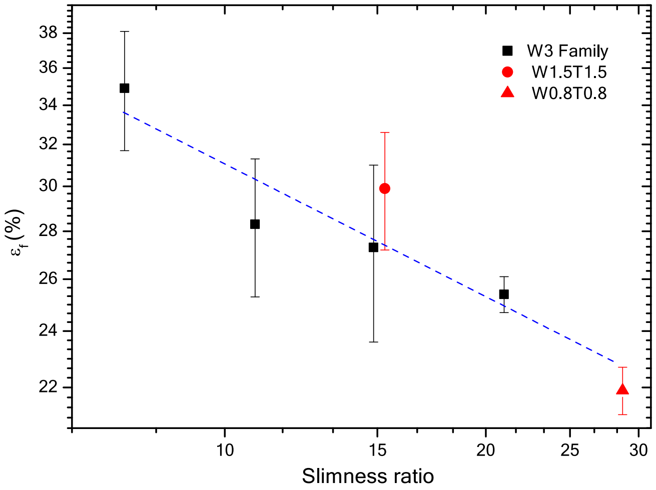


Fig. 8. Total elongation versus the slimness ratio ($L_0/\sqrt{a_0} \cdot b_0$) for the selected samples W3T3, W3T1.5, W3T0.8, W3T0.4 (black) and W1.5 T1.5, W0.8 T0.8 (in red). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

However, notable changes are observed in total elongation, which decreases with sample size. Within the specific validity range, this dependency can be predicted by the Bertella-Oliver formula. In this study, the exponent obtained is $n = 0.30$.

In the regions near the fracture, greater ductility is observed in thicker specimens. Likewise, electron microscopy has confirmed that the fracture zone contains a large number of grains, enabling these studies to be comparable to what would be obtained with bulk measurements.

In conclusion, this study serves as a foundational starting point, producing results that align with findings reported in the existing literature. These outcomes provide a basis for the systematic investigation of such materials, particularly in applications related to fusion or other scenarios that necessitate the use of small-sized specimens. The findings from this research contribute valuable insights that can inform and guide further exploration and development in the utilization of materials for specific applications, laying the groundwork for more in-depth studies and potential advancements in the field. It will be particularly relevant in the test case for the validation of materials in IFMIF-DONES.

CRediT authorship contribution statement

N. García-Rodríguez: Writing – original draft, Investigation, Conceptualization. **F. Canillas:** Writing – review & editing. **F. Arranz:** Writing – review & editing. **M. Roldán:** Conceptualization, Investigation, Writing – original draft. **F. Sánchez:** Writing – review & editing. **E. Leon-Gutierrez:** Conceptualization, Investigation, Project administration, Supervision, Writing – original draft.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The authors are unable or have chosen not to specify which data has been used.

Acknowledgements

This work has been partially funded by the MINECO Ministry under project FIS2013-40860R and the agreement as published in the Spanish BOE (BOE n14, p. 1988) and Fusion National Laboratory (LNF) internal budget.

The authors want to acknowledge Montserrat Martin for her support in the necessary sample preparation.

References

[1] Kundan Kumar, Arun Pooleery, K. Madhusoodanan, R. N. Singh, J. K. Chakravartty, B. K. Dutta and R.K. Sinha. Use of Miniature Tensile Specimen for Measurement of Mechanical Properties, Procedia Engineering 86 (2014) 899–909, Doi: 10.1016/j.proeng.2014.11.112.

[2] Kundan Kumar, K. Madhusoodanan, R.N. Singh, Miniature test techniques for life management of operating equipment", Nuclear Engineering and Design Volume 323 (2017) 345–358, https://doi.org/10.1016/j.nucengdes.2017.03.00.

[3] M. N. Gussev, J. T. Busby, K. G. Field et al, Role of Scale Factor During Tensile Testing of Small Specimens, Small Specimen Test Techniques: 6th Volume. ASTM. 2015. STP 1576, Doi: 10.1520/STP157620140013.

[4] G.E. Lucas, G.R. Odette, H. Matsui, A. Möslang, P. Spätig, J. Rensman, T. Yamamoto, The role of small specimen test technology in fusion materials development, Journal of Nuclear Materials Volumes 367–370, Part B 1 (2007) 1549–1556, https://doi.org/10.1016/j.jnucmat.2007.04.034.

[5] E. Wakai, S. Nogami, R. Kasada, A. Kimura, H. Kurishita, M. Saito, Y. Ito, F. Takada, K. Nakamura, J. Molla, P. Garin, Small specimen test technology and methodology of IFMIF/EVEDA and the further subjects, J. Nucl. Mater. 417 (6) (2011) 1325–1330, https://doi.org/10.1016/j.jnucmat.2011.02.011.

[6] O.N. Pierron, D.A. Koss, A.T. Motta, Tensile specimen geometry and the constitutive behavior of Zircaloy-4, J. Nucl. Mater. 312 (2–3) (2003) 257–261, https://doi.org/10.1016/S0022-3115(02)01554-4.

[7] K. Miyahara, C. Tada, T. Uda, N. Igata, The effects of grain and specimen sizes on mechanical properties of type 316 austenitic stainless steel, J. Nucl. Mater. 133–134 (1985) 506–510.

[8] A. Kohyama, K. Hamada, H. Matsui, Specimen size effects on tensile properties of neutron-irradiated steels, J. Nucl. Mater. 179–181 (1991) 417–420, https://doi.org/10.1016/0022-3115(91)90113-L.

[9] D.N. Hanlon, S.M.C. van Bohemen, S. Celotto, Critical Assessment 10: Tensile elongation of strong automotive steels as function of test piece geometry, Mater. Sci. Technol. 31 (4) (2015) 385–388, https://doi.org/10.1179/1743284714Y.0000000707.

[10] Y.H. Zhao, Y.Z. Guo, Q. Wei, A.M. Dangelewicz, C. Xu, Y.T. Zhu, T.G. Langdon, Y. Z. Zhou, E.J. Lavernia, Influence of specimen dimensions on the tensile behavior of ultrafine-grained Cu, Scr. Mater. 59 (2008) 627–630, https://doi.org/10.1016/j.scriptamat.2008.05.031.

[11] Malpally, Deepthi Rao, Uncertainty Analysis of Mechanical Properties from Miniature Tensile Testing of High Strength Steels, (2014), All Graduate Theses and Dissertations, Paper 4029. http://digitalcommons.usu.edu/etd.

[12] L. Han, N. Reynolds, I. Dargue, G. Williams, The Effect of Specimen Dimensions on Obtained Tensile Properties of Sheet Metals, Key Eng. Mater. 410–411 (2009) 481–491, https://doi.org/10.4028/www.scientific.net/KEM.410-411.481.

[13] A.V. Sergueeva, J. Zhou, B.E. Meacham, D.J. Branagan, Gage length and sample size effect on measured properties during tensile testing, Materials Science and Enginnering: A 526 (1–2) (2009) 79–83, https://doi.org/10.1016/j.msea.2009.07.046.

[14] F. Arranz, M. Serrano, A. Muñoz a, B. Szcepaniak, B. Brañas, O. Nomen, J. Castellanos, Stress strain curves for thick electroformed Cu pieces, Fus. Eng Des. 127 (2018) 17–22, https://doi.org/10.1016/j.fusengdes.2017.12.014.

[15] D.A. Oliver Proposed New Criteria of Ductility from a New Law Connecting the Percentage Elongation with Size of Test-Piece. Proc. Inst. Mech. Eng. 115(1) 827-864 (1928) Doi: 10.1243/PIME_PROC_1928_115_019_02.

[16] P. Zheng, R. Chen, H. Liu, J. Chen, Z. Zhang, X. Liu, Y. Shen, On the standards and practices for miniaturized tensile test – A review, Fus. Eng. Design 161 (2020) 112006, https://doi.org/10.1016/j.fusengdes.2020.112006.

[17] W.J. Yuan, F. Zhou, Z.L. Zhang, Y.J. Su, L.J. Qiao, W.Y. Chu, An analysis on necking effect and stress distribution in round cross-section specimens of pure copper with different diameters, Mat. Science Eng. a. 561 (2013) 183–190.