DUCTILE-TO-BRITTLETRANSITION AND UPPER SHELF IN STEELS: SCATTER ANALYSIS[1]

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Abstract

The characterization of ferritic steels in the ductile-to-brittle transition region by means of fracture mechanics is problematic, there exists a large scatter in the experimental results. There is also some dependence of the results and their scatter on the thickness of the specimen. Originally, this problematic was explained by means of two separate viewpoints: one was the treatment of test results using statistics (mainly Weibull distributions) and the other one was the effect of loss of constraint. Now it is accepted some interaction between statistical and constraint loss effects. Much efforts have been made in the last decades to explain this phenomenon, and as a result of this, a round robin program that involves many laboratories all over the world was carried out. Based on the results obtained in this round robin, the variation of both the scatter and the minimum experimental values, taking into account the test temperature and the specimen thickness, were analyzed in this paper. This paper also introduces an interpretation of the beginning of the upper shelf and its interaction with the ductile-to-brittle region, that explains the difference of scatter and toughness for different specimen sizes, showing that the beginning of the upper shelf zone is dependent on the specimen size.

Key words: Ductile-to-brittle transition; Weibull; Upper shelf; Fracture.

TRANSIÇÃO DÚTIL-FRÁGIL E PATAMAR SUPERIOR EM AÇOS: ANÁLISE DE DISPERSÃO

Resumo

Aços ferríticos na transição dúctil-frágil são difíceis de caracterizar por mecânica da fratura porque apresentam muita dispersão nos resultados experimentais. Também, os resultados e a dispersão apresentam dependência com a espessura do corpo de prova. Originalmente, o problema foi explicado por duas teorias diferentes: tratamento de dados por estatística (principalmente estadística de Weibull) e, a outra, por queda de restrição às deformações. Atualmente, é aceito que existe alguma interação entre estas duas teorias. Muito esforço foi feito durante as últimas décadas para explicar este fenômeno e um programa tipo “round robin”, envolvendo muitos laboratórios no mundo, foi realizado. Baseados nos resultados obtidos no “round robin”, a variação da dispersão e também dos valores mínimos experimentais, levando em conta a temperatura e a espessura, são analisados neste trabalho. O presente trabalho também introduz uma interpretação do começo do “upper shelf” e a sua interação com a região de transição dúctil-frágil, que explica a diferença de dispersão e de tenacidade à fratura para distintos tamanhos de corpos de prova, mostrando que o começo do “upper shelf” é dependente do tamanho de corpo de prova.

Palavras-chave: Transição dúctil-frágil; Weibull; Patamar superior; Fratura.

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[1] *Contribuição técnica ao 63° Congresso Anual da ABM, 28 de julho a 1° de agosto de 2008, Santos, SP, Brasil*

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1 INTRODUCTION

The importance of fracture mechanics characterization of ferritic steels is mainly related to their use as pressure vessel structural material. An incorrect determination of the fracture toughness would lead to an over conservatism with unbalances between safety and economy in design, or even worse in opposite cases, it could cause catastrophic failures of the component.

The determination of a characteristic fracture toughness value for ferritic steels in the ductile to brittle transition region becomes problematic due to the great scatter observed. This is generally attributed to a probabilistic effect, resulting from the distribution of low toughness triggering points for cleavage initiation in the volume surrounding the crack front. Specimen size plays an important role on the measured fracture toughness because it would not only influence the exposed material volume but also different thickness would cause variations in constraint.(1) The interaction between statistical and constraint loss effects is primarily attributed to reductions in experimental toughness values associated with large thickness.(2)

Weibull distribution is the most used statistics function adjust experimental results of fracture toughness in the ductile-to-brittle transition region of ferritic steels. This distribution may have two (2P-W) or three parameters (3P-W), and some authors use some of the parameters as fixed. This is the case of the 3P-W distribution proposed by Wallin(3) (Equation 1) and adopted by ASTM to calibrate the Master Curve (MC) in the E1921 Standard.(4)

This standard describes the procedure to determine a reference temperature, T_0, which characterizes the fracture toughness of ferritic steels that experience onset of cleavage cracking at elastic, or elastic-plastic K_{Jc} instabilities, or both. After testing similar specimens at a given temperature, converting the elastic-plastic J values into equivalent linear-elastic K_J and censoring the data by a size criterion, overmuch slow stable crack growth and for cleavage absence, a three parameter Weibull distribution (with the slope parameter equal to 4 and the threshold parameter equal to 20 MPa $m^{1/2}$) is used to characterize the scatter of the data, Equation (1). Then a mean toughness value is obtained and T_0 can be determined and the Master Curve located in the temperature axis.

$$P = 1 - \left\{ \exp\left[- \left(\frac{K - 20}{K_0 - 20} \right)^4 \right] \right\} \qquad \textbf{Eq. (1)}$$

The MC is valid for data sets for which most of the specimens are not censored by a size criterion.(5) This method requires at least 6 valid specimens. Because this proposal has been adopted by an ASTM Standard, several researches are using it.

Recently ESIS has sponsored a program, as a round robin, where over 700 specimens have been tested from -154°C up to room temperature, with thicknesses ranging from 12 to 100 mm.

From an engineering viewpoint, it would be desirable to be able to determine by means of laboratory tests the beginning of the upper shelf, so that the materials and operating conditions at temperatures above the transition region would be fully established. This upper shelf beginning obtained at the laboratory must be the same than that of the actual structure.

The objetive of this work was to analyze the round robin data, emphasizing the effects of temperature, specimen thickness and size of the dataset on the prediction of the behavior in the transition regime and the beginning of the upper shelf region. This paper also introduces an interpretation of the beginning of the upper shelf and its interaction with the brittle to ductile region, that explains the difference of scatter and toughness for different specimen sizes, showing that the beginning of the upper shelf zone is dependent on the specimen.

2 MATERIAL AND METHOD

The used dataset corresponds to the European round robin published by Heerens and Hellmann.(6) The material used in the Euro dataset was a quenched and tempered pressure vessel steel DIN 22NiMoCr37, tested using four C(T) specimen sizes (12.5 mm, 25 mm, 50 mm and 100 mm, identified as 1/2T, 1T, 2T and 4T respectively), at eight different temperatures in the ductile-to-brittle transition regime. The measured parameter was the J-integral, calculated according to ESIS P2-92 procedure.(7) There were 24 individual data sets as shown in Table 1, most of them having at least 30 specimens.

A general analysis of the experimental data was performed, studying the minimum values and the scatter of results. The size effect on the beginning of the upper shelf was also analyzed. As they were available in terms of J, when necessary, they were converted into K equivalent values by using:

$$K_{J_C} = \sqrt{\frac{J_C.E}{1-\nu^2}} \qquad \textbf{Eq. (2)}$$

Table 1. Description of dataset sizes

Set	T (ºC)	Size	Number of tests
1	-154	1/2 T	31
2		1T	34
3		2T	30
4	-91	1/2 T	31
5		1T	34
6		2T	30
7		4T	15
8	-60	1/2 T	31
9		1T	34
10		2T	30
11	-40	1/2 T	30
12		1T	32
13		2T	30
14	-20	1/2 T	31
15		1T	30
16		2T	30
17		4T	15
18	0	1/2 T	30
19		1T	30
20		2T	30
21		4T	16
22	20	1T	10
23		2T	30
24		4T	15

For the determination of the reference temperature T_0, the censored results were replaced by the "dummies" ones, according to Wallin's recommendations.(5) Thickness conversion was performed in the method corresponding to 3P-W with two fixed parameters, as indicated in the ASTM Standard.(4) In this method, the values were converted to 1T K_{Jc},. In this way, different values of T_0 for the MC construction were obtained.

3 RESULTS

3.1 General Description of Results

Figures 1 to 7 show, for each temperature, the scatter bands of experimental results taking into account whether cleavaje or maximum load occurred in the tests, separated by thickness. The absolute minimum experimental result for each temperature and the corresponding limit value of toughness for each thickness are also shown in the figures.

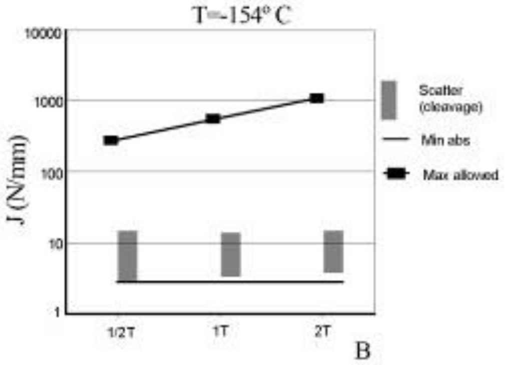

Figure 1. Experimental results for T=-154ºC

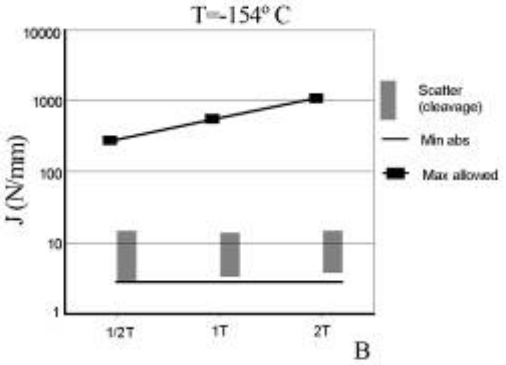

Figure 2. Experimental results for T=-91ºC

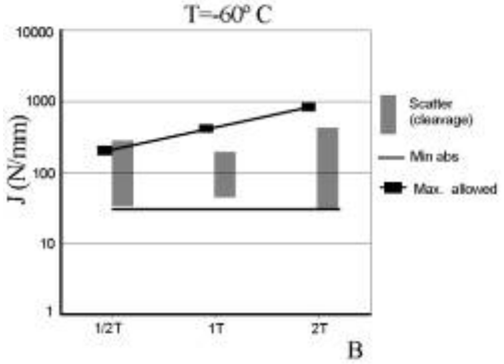

Figure 3. Experimental results for T=-60ºC

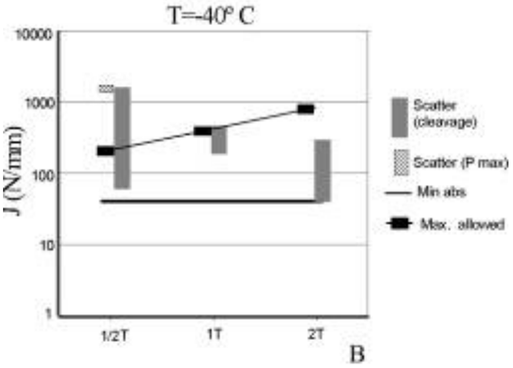

Figure 4. Experimental results for T=-40ºC

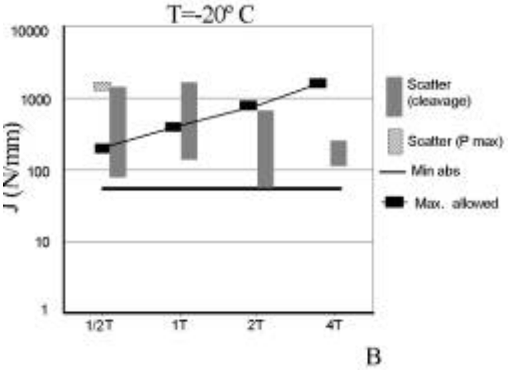

Figure 5. Experimental results for T=-20ºC

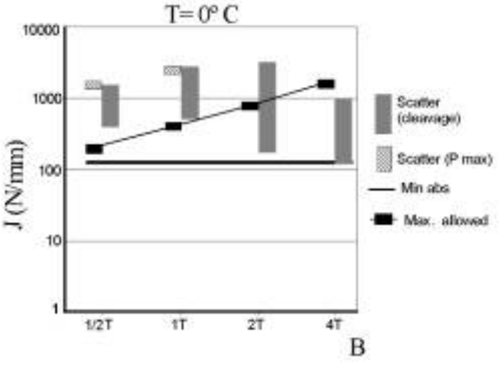

Figure 6. Experimental results for T=0ºC

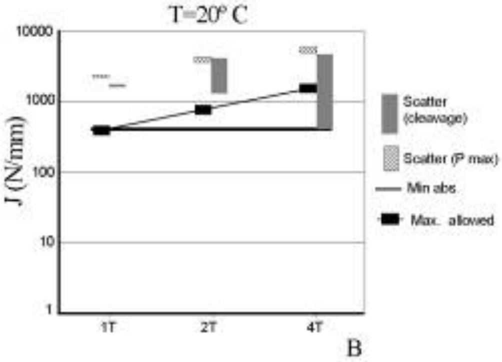

Figure 7. Experimental results for T=20ºC

3.2 Determination of Temperature T_0

Table 2 shows T_0 (ºC) and K_{median} (MPa.m$^{1/2}$) values obtained for each temperature according to ASTM E1921 Standard,$^{(4)}$ taking into account individual dataset (for each thickness) and all the data together.

Table 2. Values of T_0 and K_{median}

Temp	Thickness	T_0	K_{median}
-154	1/2T	-38.43	37.79
	1T	-52.20	40.12
	2T	-59.42	41.61
	All	-51.60	40.00
-91	1/2T	-89.44	97.95
	1T	-95.81	106.70
	2T	-96.37	107.53
	4T	-96.46	107.65
	All	-94.55	104.89
-60	1/2T	-79.51	131.41
	1T	-86.18	145.12
	2T	-109.53	209.39
	All	-97.72	173.33

Temp	Thickness	T_0	K_{median}
-40	1/2T	-92.93	221.35
	1T	-89.72	210.05
	2T	-84.89	194.24
	All	-88.52	205.98
-20	1/2T	-	-
	1T	-92.78	309.02
	2T	-86.30	276.68
	4T	-81.52	255.29
	All	-87.21	281.02
0	1/2T	-	-
	1T	-	-
	2T	-92.81	438.27
	4T	-89.82	415.73
	All	-91.59	428.86

In some cases, identify as "–" in the table, the value of T_0 was not possible to be determined because the minimum number of valid results was inferior to 6.

Table 3 shows the absolute minimum and maximum experimental values obtained for each temperature, and the value of K_{median} calculated using equation (3).

$$K_{J(med)} = 30 + 70\exp\left[0.019(T - T_0)\right] \quad \text{Eq. (3)}$$

As T_0 is not the same for all temperatures, the K_{median} value will depend on the T_0 used in Equation (3). For instance, K_{median} for T= -60º C calculated using T_0 obtained for -154 ºC dataset is 89.67 MPa.m$^{1/2}$, while if we use T_0 for -20º C, the resulting K_{median} (for T=-60 ºC) is 147.39 MPa.m$^{1/2}$.

Table 3. K_{median} obtained from different datasets

Temp (ºC)	K_{Min} (MPa.m$^{1/2}$)	K_{median} -154	K_{median} -91	K_{median} -60	K_{median} -40	K_{median} -20	K_{median} 0	K_{Max} (MPa.m$^{1/2}$)
-154	25,18	40,00	52,62	54,03	50,17	49,68	51,39	56,90
-91	58,08	63,11	104,88	109,53	96,78	95,14	100,79	169,16
-60	82,96	89,67	164,96	173,33	150,35	147,39	157,58	306,62
-40	95,16	117,26	227,34	239,59	205,98	201,65	216,55	622,95
-20	115,18	157,60	318,57	336,49	287,33	281,01	302,79	605,39
0	169,22	216,59	451,97	478,17	406,30	397,05	428,90	828,40
20	302,79	302,84	647,05	685,35	580,25	566,72	613,30	1235,28

Table 3 does not show the column corresponding to K_{median} for 20ºC, because no T_0 is available for such a case. The row identified as 20ºC gives the K_{median} for this temperature but obtained with T_0 values calculated using results tested at other temperatures different from 20ºC.

Figures 8 y 9 show different Master Curves obtained using different T_0 values. Figure 8 is plotted using logarithmic scale, and it also shows the absolute experimental minimum and maximum values for each temperature. Figure 9 is plotted using linear scale, and besides the Master Curves it shows the absolute experimental minimum value for each temperature.

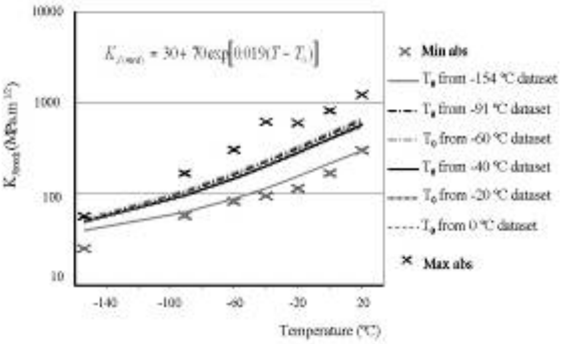

Figure 8. Master Curves obtained from different datasets (logarithmic scale)

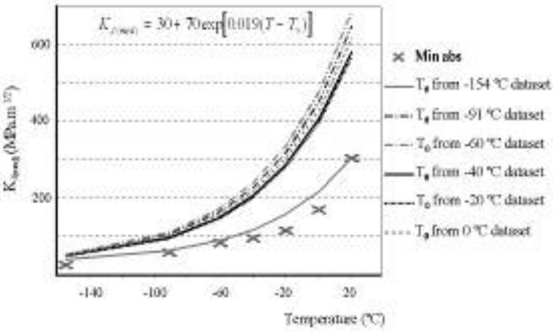

Figure 9. Master Curves obtained from different datasets (linear scale)

4 DISCUSSION

It is generally accepted that the scatter of the results increases as the specimen thickness decreases, and that fracture toughness increases with a temperature increment. Some of the round robin results show this tendency, although there are some sets of data where the first condition does not seem to be verified, especially in the upper third of the transition region near the upper shelf. The occurrence of a variable number of non valid results, mainly in the zone of high temperatures, complicate the analysis, and it could hide some trends. It can be seen in Figures 1 to 7 that the major transgressions to the limits occur for smaller thicknesses and higher temperatures.

Landes and colaborators proposed, for a given temperature, that as specimen size increases, diminishes the scatter,(1) maintaining the lower bound but not the median.(9)

The following is a brief analysis and discussion of the experimental results of the round robin:

- At -154ºC: A great difference in the scatter band for specimens with different thicknesses cannot be observed. The minimum and maximum values are similar, independently of the specimen thickness.
- At -91ºC: it can be observed something similar to -154ºC, except for 4T thickness that has a minimum value consistent to the others but the dataset has lower scatter.
- At -60ºC: there is an anomaly respect to what is predicted by the theory of the weakest link. The minimum values are similar for all thicknesses, but for ½ T specimen the scatter band is smaller than the one corresponding to the largest specimen (2T thickness). For ½ T dataset, there are some results exceeding the maximum permissible for that thickness. All the data correspond to unstable fracture.
- At -40ºC: it is observed what is predicted by the theory of the weakest link, i.e. the mean toughness value and the scatter increase as thickness decresases. For the smaller thickness dataset, there exists a large amount of results exceeding the maximum permissible toughness, being necessary to censor or correct them. For 1/2T dataset, unstable fracture did not occur in 5 tests.
- At -20º: the scatter undergoes a similar variation as that corresponding to -40ºC, but in this case the minimum experimental value has a larger variation as the thickness of the specimen change. Also, there are other thicknesses for which the experimental results exceed the maximum permissible toughness. For ½ T specimens, most of the results must be censored. There are 21 out of 31 tests that show no unstable fracture.
- At 0ºC: all experimental results for ½ T and 1 T specimens exceed the maximum permissible toughness. For ½ T specimens, only 3 out 31 tests show unstable fracture, and for 1T this behavior is observed in 7 tests. Besides this, for these datasets the scatter band is narrower than that corresponding to greater thicknesses.
- At 20 ºC: again, it is observed an anomaly respect to what is predicted by the weakest link theory, the scatter band increases as the thickness increases. The minimum and the maximum experimental results are observed for the bigger specimens. Maximum load is reached in 9 tests (over 10) and 21 tests (over 30) for 1T and 2T specimens respectively. The whole dataset results for 1T and 2T specimens, and some for 4T specimens, are invalid because they exceed the maximum permissible toughness.

It is also interesting to note that for each temperature, the absolute minimum experimental value for all thickness seems to be that corresponding to 1T specimen, except for low temperatures datasets.

At low temperatures there is a better approximation to experimental minimum that are similar for different thicknesses and same temperatures.

In general at higher temperatures, the minimum toughness are observed for the bigger thicknesses. This may be related to the fact that small specimen datasets have a lot of results exceeding the maximum permissible toughness, so for these cases the experimental J_C values were overvalued.

4.1 Analysis of the Limits Between this Transition Curve and the Upper Shelf

Taking into account the results described in the previous section, and the interpretation of the curve for the transition region made by Perez Ipiña et al.,(8) in this section an analysis of the limits between this transition curve and the upper shelf is presented (Figure 10).

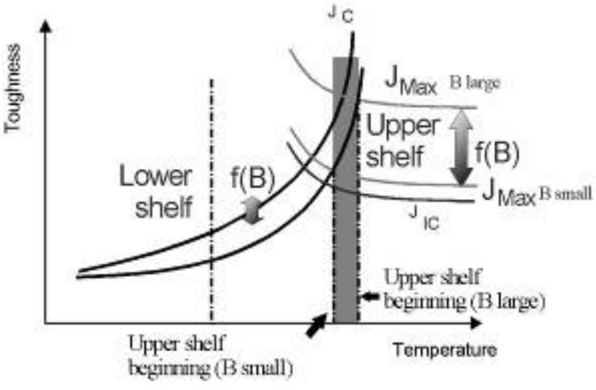

Figure 10. Reinterpretation of the curve for the transition region

Figure 10 shows that, based on the proposal of Landes and McCabe,$^{(9)}$ there is only one curve for the lower bound toughness in the transition zone. The upper limit of toughness in this region is given by a curve that depends on the specimen thickness. Something similar occurs in the upper shelf, where there is an unique curve for the dependency of J_{IC} with temperature, but different curves for the attainment of J_{max} are observed, and these curves are dependent of the specimen size.

Small specimens have the maximum load occurrence close to the onset of stable crack growth (J_{IC}), while large specimens show the plateau after a significant stable crack growth, and consequently J_{max} will be greater.

The intersections between cleavage and J_{max} curves define the change from the transition zone to the upper shelf region, and they occur at well different temperatures depending on the size of the specimens as Figure 10 shows.

It is observed in Figure 7 that for T=20°C and 1/2T a small scatter is present, while greater scatters are observed for larger specimen sizes. As explained before, small specimens reach the maximum load (and then they are unloaded) for J values close to J_{IC}, while the achievement of the J_C value requires greater stable crack growths. Then, near the limit between the transtion region and the upper shelf, small specimens can present lower scatter than larger ones. Small specimens can also show larger minimum values because lack of constraint, and they cannot reach enough driving force to trigger high cleavage toughness, that can do large specimens.

As Figure 10 shows, small specimens present the beginning of the upper shelf at lower temperatures comparing with larger specimens. This means that, while in laboratory tests results may correspond to the upper shelf region, it would be possible that brittle fracture triggers in real structures for the same material and temperature.

4.2 Analysis of Temperature T_0

The values of T_0 obtained using different datasets should be similar. As it is shown in table II, this is the case for the T_0 obtained for temperatures ranging from -91º C up to 0ºC, although not for T= -154ºC. Wallin$^{(5)}$ says that -154ºC corresponds to the lower shelf region, and this author and ASTM standard restrict the use of data obtained only at temperatures in the range T_0 ∀ 50ºC.

Both figures show that the Master Curve obtained using T_0 calculated with dataset

tested at -154ºC is distant from the other ones. For temperatures different to -154ºC, similar Master Curves are obtained, meaning that any temperature except -154ºC may be used for the construction of the Master Curve.
All the calculated Master Curves are located between the minimum and maximum experimental values.

5 CONCLUSIONS

1. A general description of experimental fracture toughness results is presented in this work, taking into account the temperature, the specimen size and the attainment or not of cleavage. Certain anomalies respect to what is predicted by the weakest link theory are observed in this analysis.
2. Similar Master Curves are obtained when different temperatures are used for the calculation of the reference temperature T_0, meaning that any temperature corresponding to the transition region may be used for the construction of the Master Curve.
3. An interpretation of the limit between the transition region and the upper shelf is proposed, showing that this limit is size-dependent.
4. The lack of prediction, using the weakest link theory, of the behavior of experimental results is mainly due to the dependence of the upper limit of toughness, the curve for the attainment of J_{max}, on the specimen thickness and loss of constraint.

Aknowledgements

To CONICET (National Council of Scientific and Technological Research of Argentina), ANPCyT (National Agency for the Promotion os Science and Technology of Argentina) for the economical support of the project.

REFERENCES

1 LANDES,J.D.; SHAFFER,D.H. Statistical characterization of fracture in the transition region. Fracture Mechanics, 12th Conference, ASTM STP 700, p. 368-382, 1980.
2 RATHBUN, H.J.; ODETTE, G.R.; HE, M.Y.; YAMAMOTO, T. Influence of statistical and constraint loss size effects on cleavage fracture toughness in the transition – A model based analysis. Engineering Fracture Mechanics, v. 73, p. 2723–2747, 2006.
3 WALLIN, K. A Simple Theoretical Charpy V-K_{IC} Correlation for Irradiation Embrittlement. ASME Pressure Vessels and Piping Conference, Innovative Approaches to Irradiation Damage and Fracture Analysis, PVP- v.170, ASME, 1989.
4 ASTM E 1921. Standard Test Method for determination of Reference Temperature, T_0, for Ferritic Steels in the Transition Range. ASTM , Annual Book of Standards, v. 03.01, 1997.
5 WALLIN, K. Master curve analysis of the “Euro” fracture toughness dataset. Engineering Fracture Mechanics, v. 69, p. 451-481, 2002.
6 HEERENS, J.; HELLMANN D. Development of the Euro Fracture Toughness Dataset. Engineering Fracture Mechanics, v. 69, p. 421-449, 2002.

7 ESIS P2-92. Procedure for determining the fracture behaviour of materials, January 1992.

8 PEREZ IPIÑA, J.E., CENTURION, S.M.C.; ASTA E. Minimum Number of Specimens to Characterize Fracture Toughness in the Ductile-to-Brittle Transition Region. Engineering Fracture Mechanics, v. 47, n. 3, p. 457-463, 1994.

9 LANDES,J.D., McCABE,D.E. Effect of section size on transition behavior of structural steels. Scientific paper 81-1D7-Metal-P2, Westinghouse R&D Center, 1982.