Fatigue & Fracture of
Engineering Materials & Structures

doi: 10.1111/j.1460-2695.2009.01432.x

Size effects in the transition region and the beginning of the upper shelf for ferritic steels

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Received in final form 13 December 2009

ABSTRACT The determination of a characteristic fracture toughness value for ferritic steels in the ductile-to-brittle transition regime becomes difficult due to the scatter observed in the results. As the temperature increases, ductile mechanisms become more active and sometimes no brittle fracture occurs. Close to the upper shelf, and contrary to what happens when only cleavage occurs, the scatter diminishes as the temperature increases, and it is also size-dependent: more scatter is found for larger sizes than for smaller specimens. An interpretation of the phenomena that takes place from the transition region up to the beginning of the upper shelf is presented in this work. This interpretation explains the difference in scatter and toughness among different sample sizes, and it also validates that the beginning of the upper shelf is dependent on the size of the sample or structure. Results from the Euro Dataset Round Robin were used to validate this interpretation.

Keywords ductile-brittle transition; fracture toughness testing; size effect.

NOMENCLATURE

2P-W = two-parameter Weibull statistics distribution
3P-W = three-parameter Weibull statistics distribution
½T, 1T, 2T, 4T = specimens of 12.5 mm, 25 mm, 50 mm and 100 mm thicknesses, respectively
C(T) = compact specimen
da = ductile crack growth
J = the J-integral
J_c = critical value of J leading to cleavage fracture
J_{IC} = fracture toughness at the initiation of slow stable crack growth
J_{max} = value of J at maximum load
K_{Jmed} = median fracture toughness in experiments
K_{JC} = an elastic-plastic equivalent stress intensity factor derived from J_C value
P_{max} = maximum load reached in a test
s.g. = side grooved specimen
T = test temperature
T_0 = reference temperature in the Master Curve

INTRODUCTION

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

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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 statistics is generally employed, sometimes two-parameter Weibull (2P-W) and also three-parameter Weibull (3P-W). A particular case is its use in the Master Curve, proposed by Wallin[3] and adopted by ASTM in the E1921 standard.[4] This standard makes use of a 3P-W with two fixed parameters. It determines a reference temperature, $\mathbf{T_0}$, that is employed to calibrate the Master Curve, for a reference thickness of 1.

At the upper shelf, where there are no cleavage values, the scatter is much lower and the behaviour of a cracked body is evaluated in terms of a resistance curve (J-R) and initiation values. The instability is due to a ductile crack growth mechanism and characterized by instability analysis based on the tearing modulus.[5]

Landes and Shaffer[1] and later Landes and McCabe[6] proposed that the scatter in the brittle-to-ductile region is size-dependent and also that a size-independent lower bound threshold exists. Perez Ipiña *et al.*[7] proposed subregions in this zone, according to the coexistence of different mechanisms at the same temperature.

From an engineering viewpoint, it would be desirable to be able to determine the beginning of the upper shelf by means of laboratory tests, so that the materials and operating conditions at temperatures above the transition region would be fully established. It is imperative that this upper shelf beginning obtained in laboratory be the same as that of the actual structure. Nevertheless, as Wallin[8] stated,

> *'Fracture initiation is possible at very high K_{JC}-values and at high temperatures. No absolute "upper shelf" transition temperature was found. . . . The brittle to ductile transition is not a true material property. It is always related to the structural size. A large structure, allowing for much ductile crack growth will have a higher transition temperature than a smaller structure of the same material and this is true even if the constraint of the structures is the same.'*

As there are still some aspects not fully understood, recently ESIS sponsored a round robin in which over 700 specimens have been tested from −154 °C up to room temperature, with thickness ranging from 12 to 100 mm.

The objectives of this work were to analyse the variation of scatter in the high transition region and also the effect of size on the determination of the beginning of the upper shelf. Data from the ESIS round robin were employed in this analysis. An interpretation of the size effect at the superior third of the transition and at the beginning of the upper shelf is presented.

Table 1 Description of data sets and their behaviour

Data set	T (°C)	Size	Number of tests	Number of tests without cleavage	Number of tests with *da*
1	−154	½T	31 + 1	0 + 0	0 + 0
2		1T	34 + 5	0 + 0	0 + 0
3		2T	30 + 2	0 + 0	0 + 0
4	−110	½T	55	0	0
5	−91	½T	31	0	6
6		1T	34	0	4
7		2T	30	0	0
8		4T	15	0	0
9	−60	½T	31 + 31	0 + 1	1+ 8
10		1T	34	0	17
11		2T	30	0	0
12	−40	½T	30 + 2	5	27
13		1T	32	0	26
14		2T	30	0	6
15	−20	½T	31	21	28
16		1T	30	0	26
17		1T s.g.	20	0	18
18		2T	30	0	15
19		4T	15	0	10
20	−10	1T	5	1	5
21	0	½T	30	27	30
22		1T	30 + 11	23 + 9	30 + 11
23		2T	30	0	26
24		4T	16	0	14
25	20	1T	10	9	10
26		2T	30	21	30
27		4T	15	3	15

In the last three columns, when an extra data set has been tested, the '+' symbol is used to differentiate it from the original data set.

MATERIAL AND METHOD

The data sets used correspond to the European round robin published by Heerens and Hellmann.[9] The material used was a quenched and tempered pressure vessel steel DIN 22NiMoCr37, and it was tested using four C(T) specimen sizes (thicknesses of 12.5 mm, 25 mm, 50 mm and 100 mm, identified as ½T, 1T, 2T and 4T respectively), at eight different temperatures, mostly in the ductile-to-brittle transition regime. The measured parameter was the J-integral, calculated according to ESIS P2–92 procedure.[10] Table 1 shows the description of the 27 individual data sets. Note that most of the data sets have at least 30 specimens.

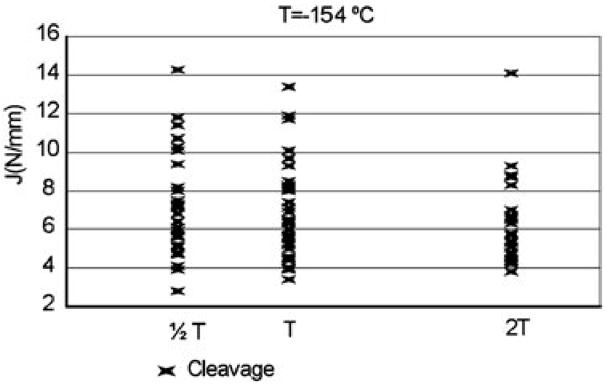

Fig. 1 Experimental results for T = −154 °C.

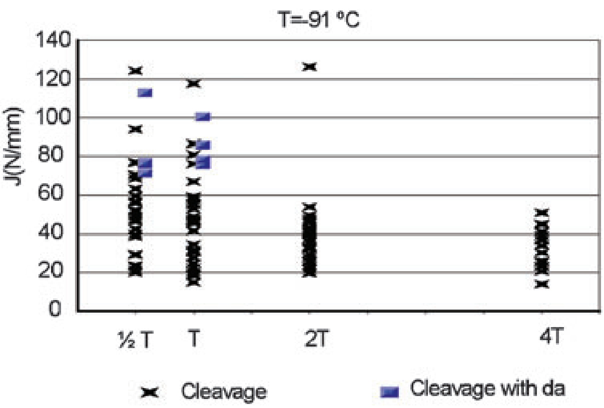

Fig. 2 Experimental results for T = −91 °C.

A general analysis of the experimental data was performed, studying minimum values, the scatter and the effects of stable crack growth and non-cleavage tests on the set.

RESULTS, ANALYSIS AND DISCUSSION

Figures 1–7 show the scatter bands of the round robin results for each temperature and size, taking into account whether stable crack growth occurred or not before cleavage, or maximum load was achieved in the tests. Table 1 indicates the number of tests that failed by different mechanisms in each sub-data set.

Landes and collaborators proposed that, for a given temperature in the brittle-to-ductile region, the scatter diminishes as size increases,[1] maintaining the lower bound but not the median.[6] Results obtained in the round robin show this tendency, although not always, especially in the superior third of the transition, near the beginning of the upper shelf. The occurrence of many non-valid results, especially at higher temperatures, complicates the analysis and can mask tendencies. As it can be appreciated,

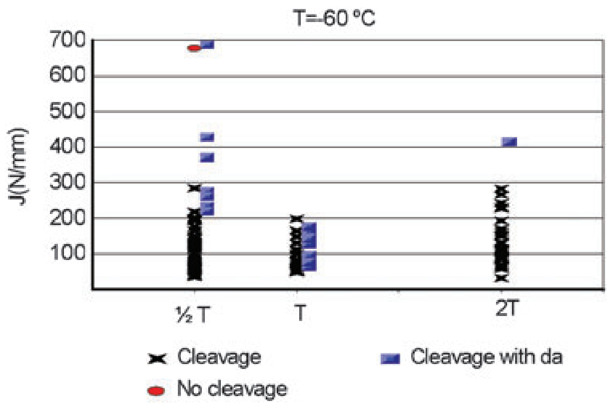

Fig. 3 Experimental results for T = −60 °C.

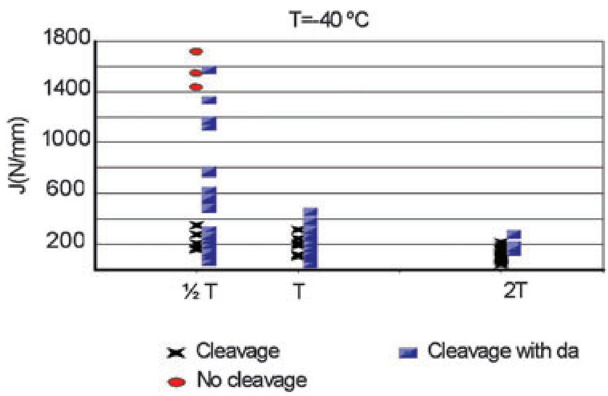

Fig. 4 Experimental results for T = −40 °C.

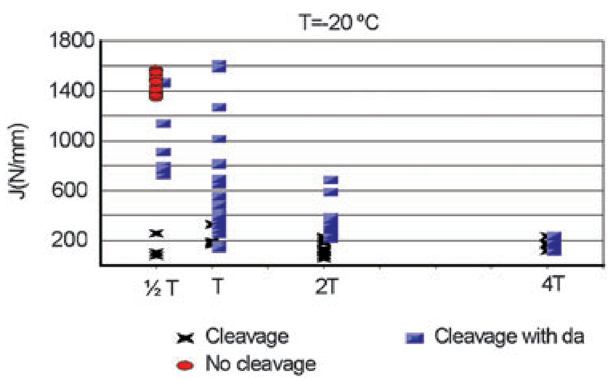

Fig. 5 Experimental results for T = −20 °C.

most important transgressions occur for smaller thicknesses and higher temperatures (Figs 4–7).

The following is a brief description of the experimental results of the round robin:

– At −154 °C, a great difference in the scatter bands for specimens with different thicknesses cannot be observed. The minimum and maximum values are similar, independently of the specimen thickness. Wallin[8] considers that this

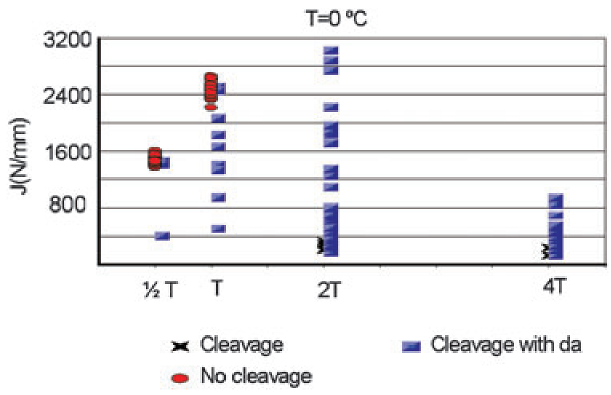

Fig. 6 Experimental results for T = 0 °C.

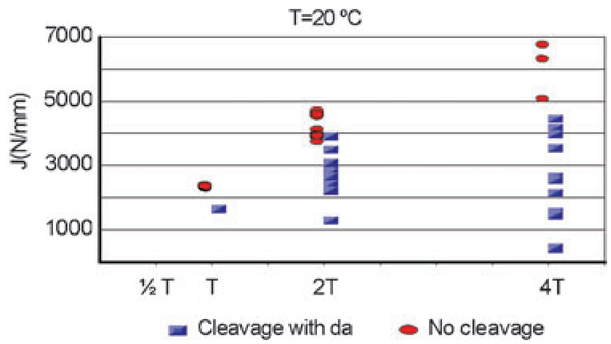

Fig. 7 Experimental results for T = 20 °C.

temperature corresponds to the lower shelf. All tests, but one of ½T, presented no unique initiation site for cleavage.

- At −110 °C, only ½T specimens were tested, showing all cleavage without stable crack growth. From a total of 55 tests, 40 of them showed one initiation site for cleavage; and for the rest of the samples, the existence of a unique site for cleavage initiation was doubtful.
- At −91 °C, the minimum toughness value was consistent for all thicknesses. All 4T specimens presented no initiation sites for cleavage (one specimen doubtful) and the scatter of this set was lower than in the other thicknesses. The scatter is larger for smaller specimens than for larger ones (one single test in 2T is out of this tendency).
- At −60° C there is an anomaly in the predictions by the weakest link theory based on the model proposed by Landes *et al*. The minimum values are similar for all thicknesses, although for 1 T specimens the scatter band is smaller than the one corresponding to the biggest specimens (2T thickness). In order to investigate this discrepancy, an extra set of 31 ½T C(T) specimens were machined from broken pieces of 2T C(T) specimens.[9] The newly machined ½T C(T) specimens exhibited much larger toughness scatter compared to the originally tested ½T C(T) specimens. For ½T data set, there are some results exceeding the maximum permissible toughness for that thickness. All data correspond to unstable fracture, although ½T and 1T sets presented some tests with cleavage after some amount of stable crack growth. At this temperature, specimens with a unique site of cleavage initiation were present in the data sets of all thicknesses.
- At −40 ° C, predictions of the model proposed by Landes *et al.*, based on the weakest link theory can be observed: mean toughness value and scatter increase as thickness decreases. There exists a large amount of results exceeding the maximum permissible toughness for the smallest thickness data set. For ½T data set, unstable fracture did not occur in 5 tests, while cleavage occurred in all specimens for larger sizes. For ½T, 27 out of 32 specimens failed after some extent of stable crack growth, while 26 out of 32 for 1T and 6 out of 30 for 2T.
- At −20° C, 21 out of 31 ½T tests showed no unstable fracture and only 3 specimens cleaved with no stable crack growth, 4 out of 30 for 1T specimens, 2 out of 20 for side grooved 1T specimens, 15 out of 30 for 2T specimens and 5 out of 15 for 4T specimens cleaved with no stable crack growth.
- At −10 °C, only 5 tests for 1T specimens were performed. One of them did not present cleavage, while the other 4 failed after some amount of stable crack growth.
- At 0 °C, all experimental results for ½T and 1T specimens exceeded the maximum permissible toughness. For ½T specimens, only 3 out of 31 tests showed unstable fracture although after some stable crack growth. This behaviour was observed in 9 tests for 1T specimens. For larger specimens, all the specimens failed by cleavage, although 26 out of 30 in 2T and 14 out of 16 in 4T with some extent of stable crack growth. The scatter bands for ½T and 1T are narrower than those corresponding to larger thicknesses.
- At 20 °C, no ½T tests were performed. All tests for all sizes presented stable crack growth before the end of the test, although 9 out of 10 for 1T, 21 out of 30 in 2T and 3 out of 15 for 4T were tests where no cleavage occurred. Again an anomaly is observed related to what the weakest link theory according to Landes *et al*. predicts: the scatter band increases as the thickness increases. Both, the minimum and the maximum of the experimental results are observed for the biggest specimens. The whole data set results for 1T and 2T specimens, and some for 4T specimens, resulted invalid because they exceeded the maximum permissible toughness.

According to the results described above, some remarks have to be made:

- The occurrence of defined initiation sites of cleavage appears as size-dependent, occurring at lower temperatures for smaller sizes. In case of considering, following some authors,[8,11] that when cleavage does not present a unique

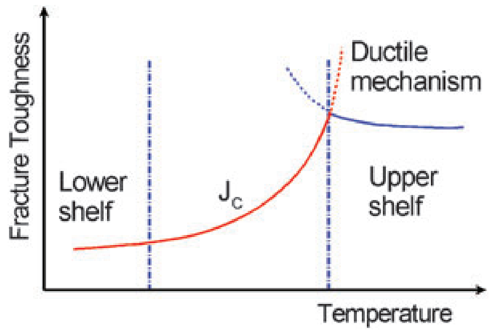

Fig. 8 Ductile-to-brittle transition curve including ductile mechanism.

site for initiation, the material is working in the lower shelf, it seems that the beginning of the transition region could be also size-dependent.

- There is a better approximation to similar experimental minimum values for different thicknesses and equal temperatures at lower temperatures than at higher ones.

At higher temperatures, the minimum toughness results were generally observed for the larger thicknesses. This may be related to the fact that small specimen data sets have a lot of results exceeding the maximum permissible toughness, so for these cases there is no enough stress triaxiality at low loads to trigger the cleavage.

Traditionally the ductile-to-brittle transition curve was obtained by means of impact tests, typically Charpy-V test, and the absorbed energy was plotted against temperature without separating the involved mechanisms. A single curve of absorbed energy versus temperature is obtained in this way, including not only the transition region but also the lower and upper shelves. When it is analysed in terms of fracture mechanics parameters, two different mechanisms have to be considered. The intersection of a cleavage curve with a 'ductile mechanism' toughness curve has to be considered, at least in a first approximation, as the limit between the transition region and the upper shelf (Fig. 8). As there is scatter in the transition region, Ericksonkirk and Ericksonkirk[12] proposed the intersection between the K_{Jmed} given by ASTM (Master Curve) with the curve of variation of J_{IC} as the limit between the transition region and the upper shelf.

When the size effect on the scatter of cleavage is considered, and following the model proposed by Landes and McCabe[6] that a size-independent threshold exists, there will be a region, between vertical lines in Figs 9a and b, where this scatter will be limited by the occurrence of ductile mechanism, i.e. lack of cleavage. Note that in the region between the intersection of the ductile mechanism curve with the curves of cleavage, the scatter diminishes as temperature increases as a consequence of the ductile mechanisms impeding attainment of enough driving force to trigger the largest values of cleavage scatter. The figures show that the region of temperatures where both mechanisms coexist widens as size diminishes because the intersection of the ductile mechanism curve with the upper bound of cleavage occurs at lower temperatures.

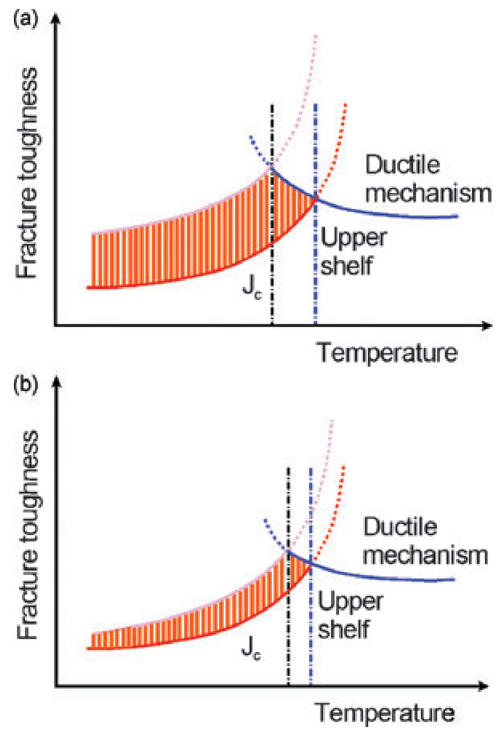

Fig. 9 Ductile-to-brittle transition curve including scatter: (a) small specimens, (b) large specimens.

As it is well known, there is no single-parameter characterization of the upper shelf as a unique upper shelf curve would require. One cannot use just the initiation curve to define the fracture behaviour in this region. R-curves and tearing instability must also be considered. Nevertheless, from the viewpoint of laboratory practice, tests with no cleavage are ended after load begins to diminish, and commonly the upper shelf is considered to have begun when all the tests correspond to maximum load values.

In order to analyse the size effect in the transition region and the beginning of the upper shelf, the sub-regions proposed by Perez Ipiña *et al.*[7] will be used (Fig. 10a & b). As scatter in ductile mechanisms is much lower than in cleavage, only median values of J_{IC} and J_{max} were considered. Several sub-regions can be defined in the transition:

I: All specimens fracture by cleavage without any stable crack growth.

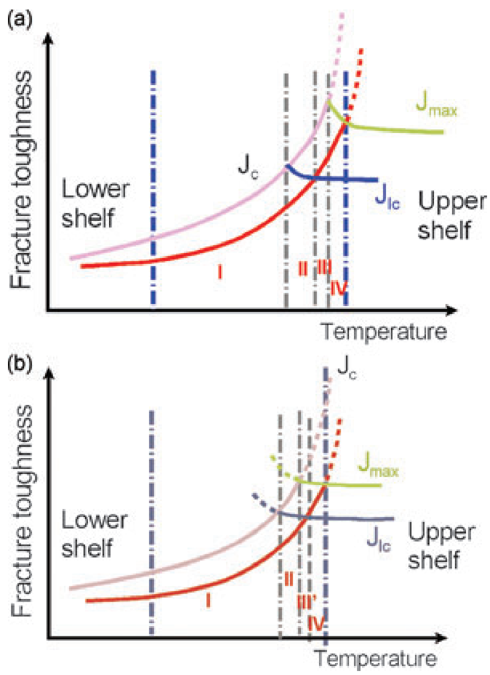

Fig. 10 (a) Sub-region III and (b) Sub-region III' in the ductile-to-brittle transition curve.

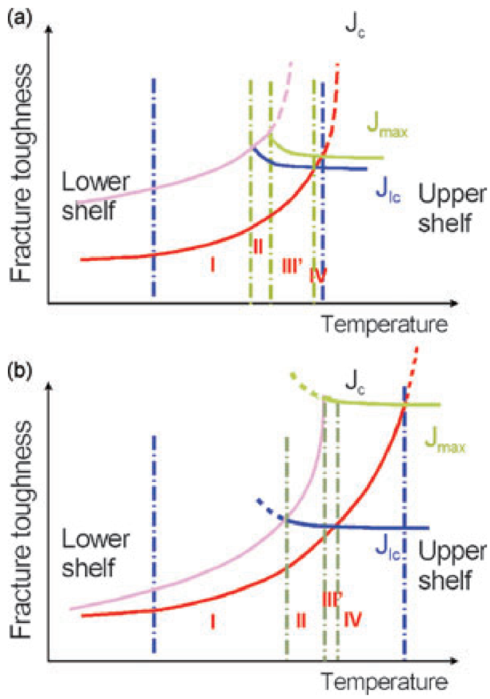

Fig. 11 Sub-regions in the ductile-to-brittle transition curve: (a) small specimens, (b) large specimens.

II: Some specimens fracture by cleavage without any stable crack growth, while others fracture by cleavage after some amount of stable crack growth.

III: No cleavage without stable crack growth occurs. All the specimens fracture by cleavage after some amount of stable crack growth, or:

III′: some specimens fracture without stable crack growth, others with stable crack growth, and others reach the maximum load condition and do not present instability. III or III′ will be present depending on the crossing of curves J_{IC} with lower bound cleavage and the crossing of J_{max} with upper bound cleavage curves. When the intersection of J_{IC} with the cleavage lower bound curves and the intersection of J_{max} with the cleavage upper bound curves occur at the same temperature, there will be no region III nor III′.

IV: IV: Some specimens fracture after some amount of stable crack growth, while others reach the maximum load condition and do not present instability.

For higher temperatures, no cleavage occurs and this behaviour corresponds to the upper shelf.

Maximum load toughness is size-dependent: small specimens present the P_{max} plateau close past the stable crack growth initiation, while large specimens require more stable crack growth to reach this plateau, giving then larger J_{max} than small specimens. Maximum load curves intersect the cleavage curves – the upper cleavage curve is also size-dependent – at different temperatures for different sizes (Fig. 11). Region IV widens and displaces towards higher temperatures as size increases, making the beginning of the upper shelf also size-dependent, as Wallin[8] has already stated.

Although the experimental verification of the proposed interpretation can be masked by the problem of size limits, there are clear tendencies that corroborate this explanation:

1 Effect of size on the number of initiation sites (unique or multiple): for −154 °C no stable crack growth was present before cleavage in any test of any size. As there was no a unique trigger point in the specimens of all sizes but ½T, then 1T and 2T can be situated in the lower shelf and ½T at sub-region I. For T = −110 °C (only ½T specimens tested), 40 out of 55 specimens presented 1 initiation site, while the other 15 specimens were doubtful, meaning that this is sub-region I for this specimen size. For −90°C, all specimens of all sizes, except one of the 4T data, presented well-defined unique trigger points: this would mean that ½T, 1T and 2T results were at the transition region, while it is not sure the location of the 4T specimens (lower shelf or sub-region I).

2 Effect of size on stable crack growth and cleavage: at temperatures lower than −91 °C there were no tests presenting stable crack growth. However, at −91 °C, ½T specimens presented 6 out of 31 tests with stable crack growth before cleavage, while in 1T specimens 4

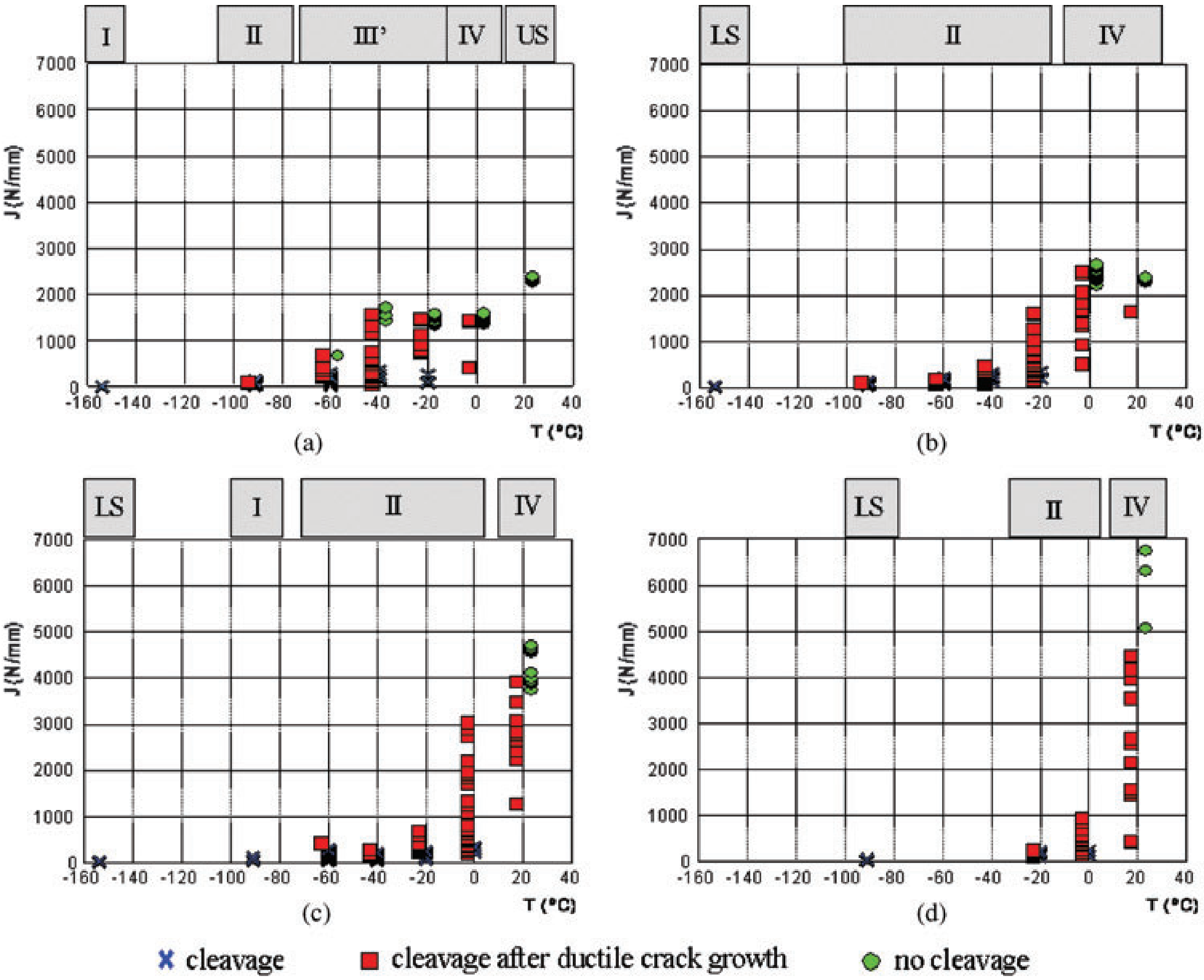

Fig. 12 Location of lower shelf (LS), sub-regions and upper shelf (US) for different specimen sizes: (a) 1/2T, (b) 1T, (c) 2T and (d) 4T.

out of 34 tests presented stable crack growth. No stable crack growth before cleavage was present for 2 and 4T specimens. Specimens identified as ½T and 1T at this temperature can be situated in sub-region II, while 2T and 4T specimens in sub-region I. For −40 °C, all 1T and 2T specimens presented cleavage, although some specimens presented stable crack growth (sub-region II). At −20 °C, all 1T (with and without side grooving), 2T and 4T specimens presented cleavage with some specimens with cleavage after stable crack growth. At 0 °C, only 2T and 4T sizes can be situated in sub-region II, while no size meet these requirements at 20 °C.

3 The coexistence between cleavage and maximum load results began at T = −40 °C for B = ½T, while it began at −10 °C for B = 1T; and at 20 °C for specimens 2T and 4T. In all cases, results of cleavage with and without stable crack growth and maximum load are present in each set, that is, sub-region III′. Any set presenting results corresponding to all specimens with cleavage following with stable crack growth, were defined as sub-region III.

4 Low scatter for maximum load results: although the mean value of J_{max} results increases as size increases, the scatter in J_{max} values that each size presents is low compared to the scatter shown by the specimens that broke by cleavage, as figures at temperatures 0 °C and 20 °C clearly shown.

5 Reduction in scatter as temperature increases and size diminishes: as consequence of the above mentioned in (4), the total scatter in sub-regions III′ and IV for a set diminishes as temperature increases and size diminishes.

6 Size effect for the beginning of the upper shelf: Although there are no sets in the round robin that completely verify the beginning of the upper shelf according to the proposed interpretation, the experimental results seem to verify that the beginning of the upper shelf is size-dependent.

Table 2 Identification of sub-regions of the data sets used

Data set	T (°C)	Size	Sub-region*
1	−154	½T	I
2		1T	Lower shelf
3		2T	Lower shelf
4	−110	½T	I
5	−91	½T	II
6		1T	II
7		2T	I
8		4T	Lower shelf or I**
9	−60	½T	III′
10		1T	II
11		2T	II
12	−40	½T	III′
13		1T	II
14		2T	II
15	−20	½T	III′
16		1T	II
17		1T s.g.	II
18		2T	II
19		4T	II
20	−10	1T	IV
21	0	½T	IV
22		1T	IV
23		2T	II
24		4T	II
25	20	1T	IV
26		2T	IV
27		4T	IV

*Considering the beginning of the transition when at least one test presented one initiation site.
**Data show one doubtful specimen.

Table 2 and Fig. 12 summarize the position of each subset in terms of sub-regions for all sizes and temperatures.

CONCLUSIONS

1. An analysis of the fracture toughness results of the European round robin is presented in this work, taking into account temperature, specimen size and the attainment or absence of cleavage. Anomalies regarding to what is predicted by the weakest link based model proposed by Landes *et al*. were observed in this analysis, especially in the upper third of the transition.
2. Four different sub-regions already identified were verified with the experimental results
3. According to this interpretation, scatter diminishes as temperature increases in sub-regions III and IV.
4. It is also postulated, and verified, that the sub-regions widens as the size diminishes.
5. For a given temperature in sub-regions III and IV, the scatter also reduces as specimen size diminishes. This was verified with the experimental data.
6. This interpretation also explains that the limit between the transition region and the upper shelf is size-dependent.
7. The occurrence of defined initiation sites of cleavage appears as size-dependent, occurring at lower temperatures for smaller sizes.

Acknowledgements

The authors would like to acknowledge to CONICET (National Council of Scientific and Technological Research of Argentina) for the economic support to the project.

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