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DUCTILE-TO-BRITTLE TRANSITION TEMPERATURE CORRELATION OF CONVENTIONAL AND MINIATURIZED SPECIMENS FOR CHARPY IMPACT TESTING ON X65, L80 AND P110

Teófilo Jacob Freitas e Souza
Fernando Buiatti Rodrigues
Dênis Soares Freitas
Sinésio Domingues Franco
Universidade Federal de Uberlândia, Av. João Naves de Ávila, 2121, Santa Mônica, 38400-902, Uberlândia – MG
teofilojacob@gmail.com, fernandobuiatti@yahoo.com.br, denissfreitas@hotmail.com, sdfranco@ufu.br

Marcelo Torres Piza Paes
Ricardo Reppold Marinho
Petrobras, Centro de Pesquisas e Desenvolvimento Leopoldo Américo M. de Mello, Cidade Universitária, 21941-915, Rio de Janeiro - RJ
mtpp@petrobras.com.br, reppold@petrobras.com;

Abstract. *The Charpy impact test is used to determine the impact strength of the material and its transition temperature from a ductile fracture behavior to the brittle. In certain situations, the manufacturing of conventional test bodies is not possible since the material may have a complex geometry or the component in which the analysis is desired to be in full operation. In order to avoid stopping an equipment or analyzing complex geometry components, miniaturized specimens are manufactured. This study aimed to correlate the ductile-to-brittle transition temperature (DBTT) results for Charpy testing between conventional and miniaturized specimens. The materials analyzed were API 5L X65, API 5CT L80 and API 5CT P110 steels, usually used in the oil and gas industry. Initially, testing and transition temperature calculation procedures were established. Concluding the work, it was possible to observe a reduction of the ductile-to-brittle transition temperature between conventional and miniaturized test specimens, in the order of -42.3 °C.*

Keywords: *Charpy impact test, miniCharpy, ductile-to-brittle transition temperature, correlation.*

1. INTRODUCTION

The determination of material mechanic properties is, on engineering, essential to guide the professionals on the project of a mechanical component. In this way, different mechanical tests are performed to obtain the various properties of the material. The purpose of the Charpy impact test is to simulate fractures that occurred in the field. The main result of this test is the energy expended to break the test body. Therefore, by evaluating the spent energy at different test conditions, it is possible to determine ductile-to-brittle transition temperature (DBTT) of the tested material. The result is an embrittlement curve that indicates the temperature range at which fracture resulting from test exhibits a change from ductile behavior to brittle behavior.

Normally, conventional samples standardized by international and national standards have unfavorable dimensions for certain situations. Some inconvenient scenarios are when the component, from which a test piece have to be removed, is small or with complex geometry. On the other hand, at times when the test piece should be removed from a working component. In such cases, smaller scale test bodies are machined and tested.

Authors such as Louden et al (1988) and Lucon et al (2015) carried out correlation studies in Charpy impact tests between specimens of different dimensions. Lucon et al (2015) obtained correlation results between conventional (10 × 10 × 55 mm³) and miniaturized (3 × 4 × 27 mm³) test specimens for API 5L X52, X65, X70 and X100 steels, observing a mean reduction of the ductile-to-brittle transition temperature in order of -43.8 °C. According to Louden et al (1988), this reduction is expected, since decreasing the size of the specimen implies a change in the state of notch stresses. The change in tenacity to the notch causes the material to appear to be more ductile at a given temperature and, therefore, decreases its ductile-to-brittle transition temperature.

The evaluation of ductile-to-brittle transition in materials of the area of oil and gas undergoes extensive experimentation. Therefore, this work aims to identify the effect of the miniaturization of the test specimen on the

transition temperature values for API 5L X65, API 5CT L80 and API 5CT P110 steels by means of Charpy impact tests, with standardization of ISO 148-1: 2010 standards and ISO 14556: 2015, respectively.

2. EXPERIMENTAL PROCEDURE

The specimens used in this study were machined from tubes made from API 5L X65, L80 and API 5CT P110 materials. The chemical compositions for the steels in this work are indicated in Tab. 1. In turn, Tab. 2 shows the mechanical properties of these steels. Nicolosi (2015), who evaluated the same samples of material from which the test bodies of this study were machined, obtained the results of the mechanical properties.

Table 1. Chemical compositions measured in API 5L X65, L80 and API 5CT P110 steels (% by weight).

Material	C	Si	Mn	P	S	Al	Cr	Mo	V	Ti	Nb	Co
X65	0.070	0.278	1.438	0.010	0.014	0.031	0.047	0.059	0.036	0.002	0.025	0.010
L80	0.266	0.231	1.077	0.010	0.015	0.022	0.307	0.055	0.003	0.022	0.001	0.006
P110	0.248	0.225	1.053	0.010	0.007	0.023	0.326	0.052	0.003	0.027	-	0.003

Table 2. Mechanical properties obtained by tensile and hardness tests for API 5L X65, L80 and API 5CT P110 steels (NICOLOSI, 2015).

Material	Yield strength (MPa)	Tensile strength (MPa)	HV10
X65	473	561	184
L80	514	621	204
P110	857	929	290

Conventional and miniaturized test specimens were manufactured and tested in accordance with ISO 148-1: 2010 and ISO 14556: 2015. Table 3 shows the dimensions and tolerances that must be adopted during the machining and dimensional verification of the specimens, according to the norms cited. A calibrated pachymeter and micrometer were used, with scales of 0.01 mm and 0.001 mm respectively, to measure the section of the specimens. The dimensional control of the notch was performed, through an optical microscope equipped with a software that allows the dimensional evaluation of the obtained images.

Table 3. Dimensions of conventional and miniaturized Charpy test specimens (ISO 148-1: 2010 and ISO 14556: 2015).

Dimensions (mm)	Conventional specimens	Miniaturized specimens
Specimen length	55.0 ± 0.6	27.0 ± 0.6
Specimen height	10.000 ± 0.075	4.00 ± 0.05
Specimen width	10.000 ± 0.075	3.00 ± 0.05
Notch depth	2.000 ± 0.075	1.00 ± 0.05
Angle of notch	45° ± 2°	60° ± 1°
Notch-root radius	0.250 ± 0.025	0.100 ± 0.025
Surface roughness	< 5 µm	< 2 µm

A wire electrical discharge machine (EDM) was used to fabricate the conventional and miniaturized test bodies, which were machined from samples of the tubes of each steel. The notch of conventional specimens was machined using the broaching process, while the same wire EDM process machined the notch of the miniaturized specimens. During machining, the main parameter observed was the pipe rolling direction. Samples should always be manufactured from the same lamination direction to avoid variations in microstructure of different specimens. The anisotropy of the material can generate variations of result if the specimens are removed from different directions. Figure 1 shows the comparison of conventional and miniaturized specimens after fabrication.
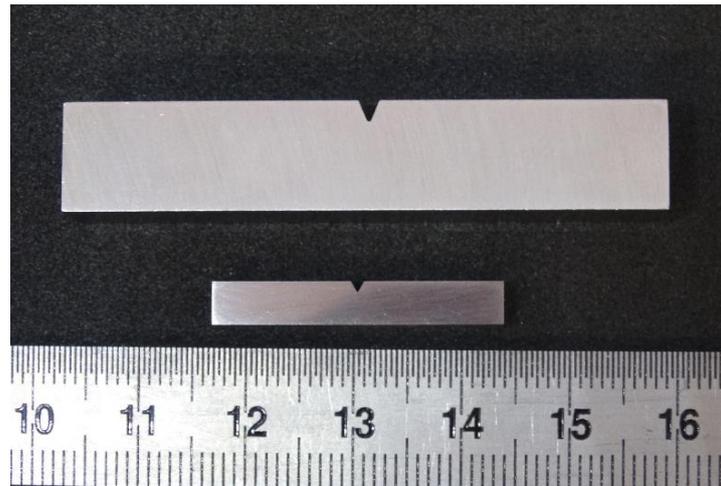
Figure 1. Comparison between miniaturized and conventional specimens after fabrication.

The tests of the conventional specimens were performed in two different moments and equipment, following the procedures of ISO 148-1: 2010. For specimens of X65 steel the test temperature range was -60 °C to 20 °C, which the equipment used was an impact pendulum with a maximum capacity of 300 J and an impact velocity of 5.3 m /s. For L80 and P110 steels, conventional test bodies were tested on an equipment with a capacity of 406 J and an impact velocity of 5.5 m / s. The tests were performed in a temperature range of -80 °C to 20 °C. The test methodology chosen was to test five specimens for each temperature. Thus, 20 specimens were manufactured for API 5L X65 material and 25 specimens for L80 and P110.

The tests on the miniaturized specimens of the three steels covered temperatures between -196 °C and 20 °C and were performed following ISO 14556: 2015. Unlike the conventional test specimens, each miniaturized specimen was tested at a different temperature within the given range.

The Laboratory of Technology in Friction and Wear (LTAD), projected the impact test machine used, called MiniCharpy, shown in Fig. 2. The maximum initial potential energy was 14 J, and the impact velocity was 3.2 m/s. 20 specimens were manufactured for L80 and P110 materials, and 24 specimens for API 5L X65, totaling 64 samples. This is an instrumented equipment, so in addition to the energy absorbed by the specimen, it is possible to evaluate other parameters such as the variation of the speed and angle of the pendulum during the test and the force as a function of time or deformation.
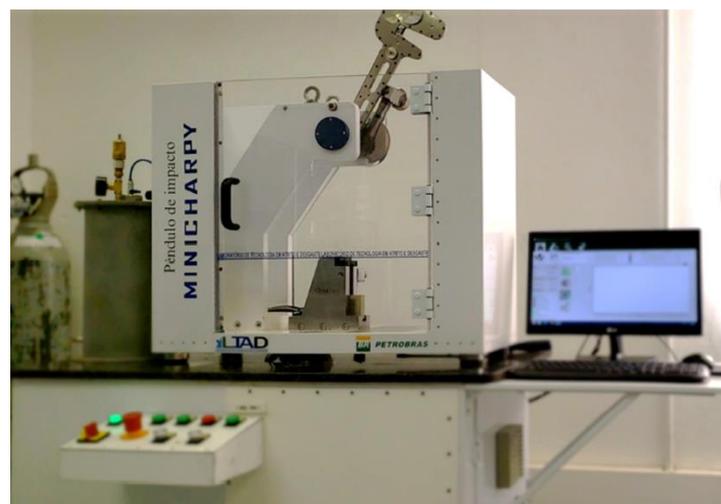

Figure 2. Miniaturized specimen impact tester MiniCharpy.

For cooling and control of the test temperature, the conventional specimens were immersed in ethyl alcohol baths, conditioned to the required temperature. With temperature stabilization for five minutes, the specimens were transferred to the anvils. The time between transfer samples and completion of the test did not exceed five seconds as required by ISO 148-1: 2010.

Temperature control in miniaturized samples was performed using a thermal conditioner, shown in Fig. 3, designed by the LTAD. This conditioner utilizes nitrogen gas and liquid nitrogen, creating an adjustable nitrogen vapor flow over the specimen to achieve the desired temperature. Temperature measurement of the specimen was done locally by welding AWG-30 Type K thermocouples in the center of each specimen near the notch region.
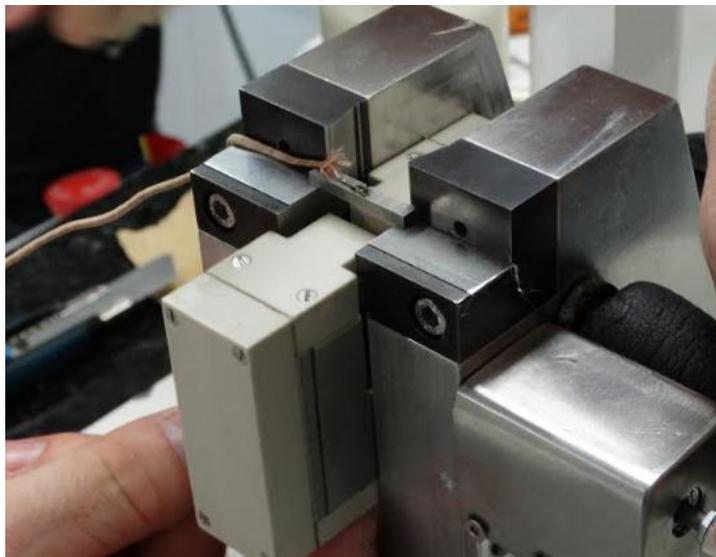

Figure 3. Placement of specimen and thermal conditioner before testing.

3. RESULTS AND DISCUSSION

Figures 4 and 5 show the results of the Charpy impact tests on conventional and miniaturized test bodies for the three steels studied. The ductile-to-brittle transition curves shown were obtained from the application of an exponential regression function at the points. The transition temperature of each material was calculated by the inflection point of the curve. The results of the tests on conventional (Fig. 4) and miniaturized specimens (Fig. 5) showed that the X65 steel has the highest impact strength, while the L80 has an intermediate impact strength and the P110 has the lowest impact strength. This is an expected result, since P110 steel has a higher mechanical strength than L80 and X65 (Tab. 2). Table 4 shows a summary of ductile-to-brittle temperatures transition results and the reduction of this temperature between conventional and miniaturized tests.

In the tests of conventional test bodies (Fig. 4) it was not possible to calculate the transition temperature of the X65 steel because the energy results remained in the region of ductile fracture. Therefore, the results in X65 steel are only qualitative. For the tests on the L80 and P110 steels, the calculated ductile-to-brittle transition temperatures were -73.8 °C and -65.2 °C, respectively. The average transition temperature between the two steels is -69.5 °C.

For the tests on miniaturized specimens (Fig. 5) it was possible to calculate the ductile-to-brittle transition temperature for the three materials, since results were obtained in the regions of ductile and brittle fracture. The transition temperatures calculated for steels X65, L80 and P110 were respectively -116.3 °C, -104.7 °C and -118.9 °C. The average transition temperature was -113.3 °C, considering all three steels. There was, therefore, a reduction of the ductile-to-brittle transition temperature between tests on conventional and miniaturized test specimens. Correlating the transition temperatures between the different tests, a mean reduction of -42.3 °C of tests in conventional test specimens for the miniaturized ones was observed.

Table 4. Transition temperature results in conventional and miniaturized samples, as well as reduction of the ductile-to-brittle transition temperature between tests.

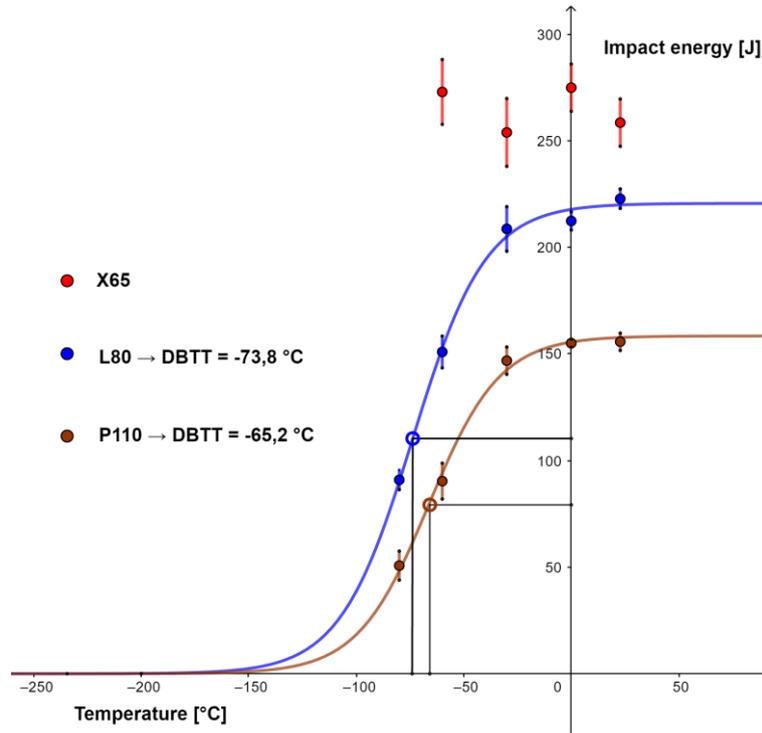| Material	API 5L X65	API 5CT P110	API 5CT L80	Mean
DBTT on conventional samples (°C)	-	-73.8	-65.2	**-69.5**
DBTT on miniaturized samples (°C)	-116. 3	-104.7	-118.9	**-113.3**
Reduction of DBTT (°C)	-	-30.9	-53.7	**-42.3**

Figure 4. Charpy impact test in conventional samples results as function of the test temperature for the X65, L80 and P110 materials, showing the regression curves, calculated transition temperatures and the deviation ranges of the tests at each temperature.
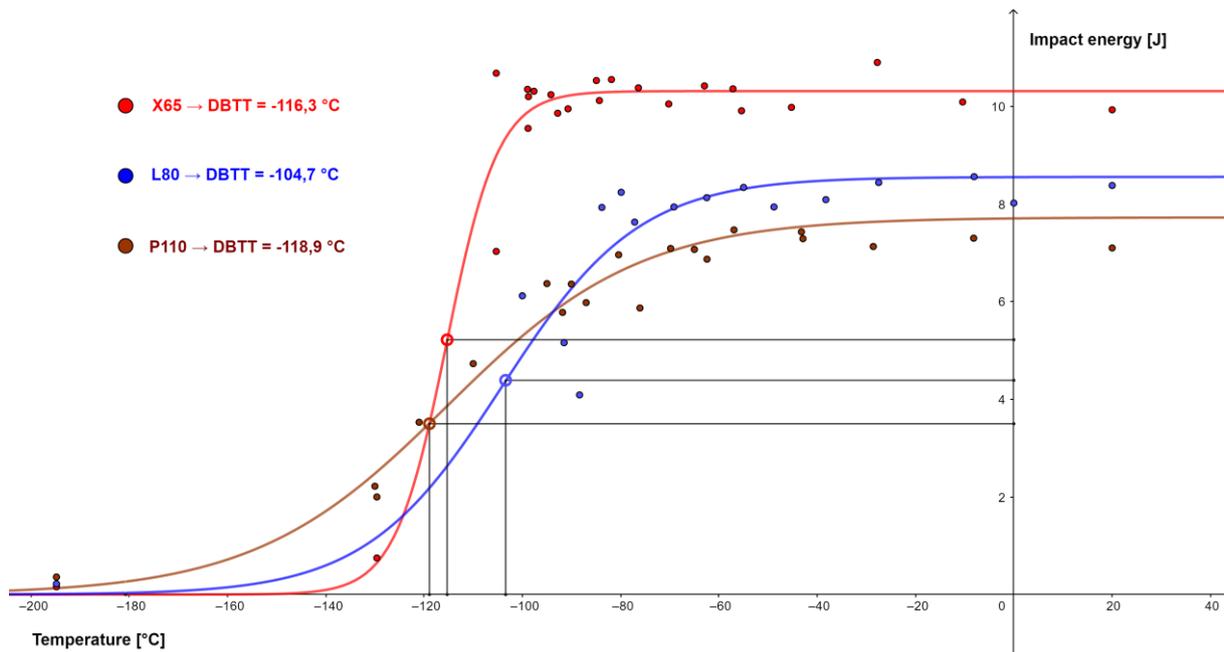

Figure 5. Results of the Charpy impact test on miniaturized samples as a function of the test temperature for materials X65, L80 and P110, showing the regression curves and calculated transition temperatures.

Still by the Fig. 5 it was observed that the X65 material, compared to L80 and P110, presented a smaller ductile-brittle transition region, where only a difference of -30 °C differentiated results from ductile to brittle fracture. This behavior of the ductile-to-brittle transition range is mainly due to the carbon content of the material. The lower the carbon content in the chemical composition of the steel, the lower its ductile-brittle transition range.

The instrumentation of the equipment allowed the construction of force graphs due to the deformation of the specimens during the impact. Figure 6 shows the curves of three of the X65 steel tests, with fracture results in the

ductile, ductile-to-brittle and brittle transition regions. It can be observed that the weakening of the specimen leads to the increase of the maximum test force. In addition, since the energy absorbed during impact is the area under the strain force curve, the energy reduction is evident with the weakening of the specimen.
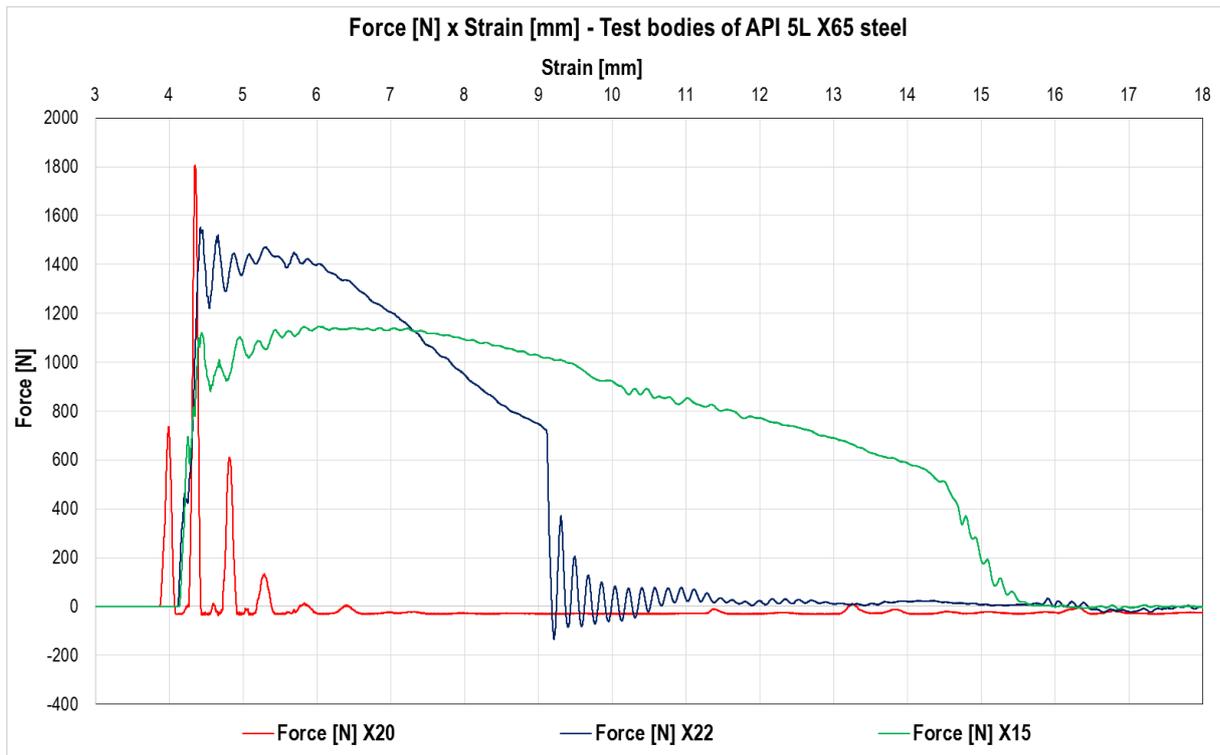

Figure 6. Force [N] x Deformation [mm] graphs of tests in specimens with fracture results in the ductile (X15), ductile-brittle transition (X22) and brittle (X20) regions.

After the tests on the miniaturized specimens, the fracture surface was investigated in the three materials using a scanning electron microscopy. Figures 7 and 8 shows the results of analysis on X65 steel specimens, representative for the other materials. The specimens analyzed were tested at -194 °C (Fig. 8) and 20 °C (Fig. 7). The observed fractures are typical of Charpy impact tests, where the predominant failure mechanisms are almost cleavage (Fig. 8), characteristic of beneficiated steels, and plastic deformation with formation of dimples (Fig. 7).
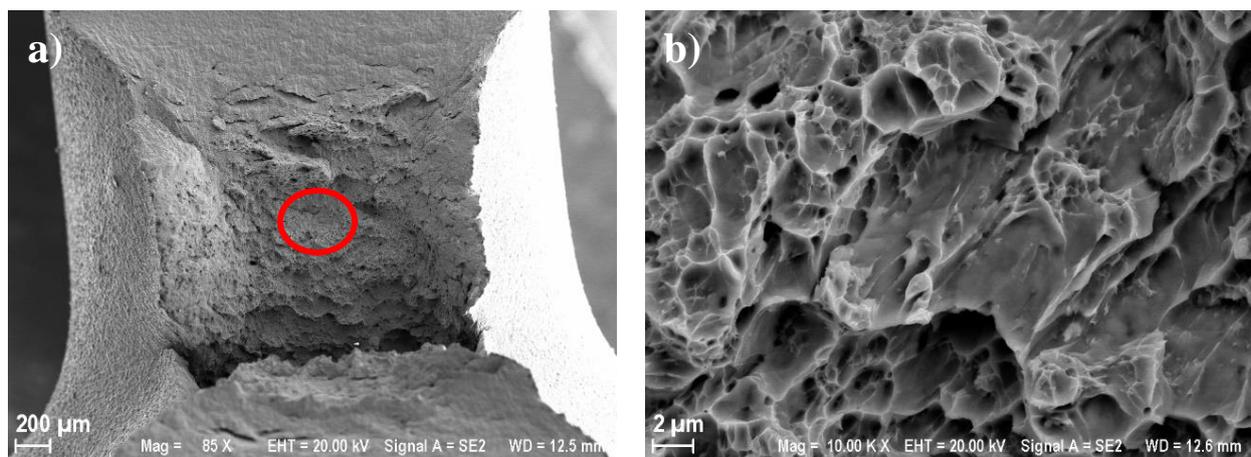

Figure 7. Macro (a) and micro (b) ductile fracture images resulted from miniaturized specimen test at 20 ° C for API 5L X65 steel.
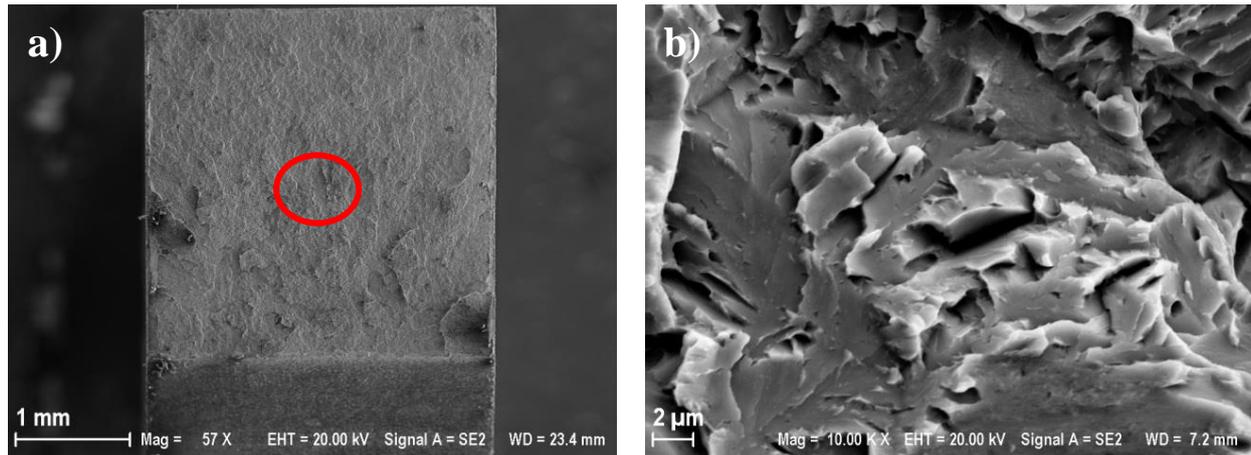
Figure 8. Macro (a) and micro (b) fragile fracture images resulted from miniaturized specimen testing at -194 ° C for API 5L X65 steel.

4. CONCLUSIONS

a) In tests on conventional test bodies, it was not possible to calculate the transition temperature for X65 steel. New tests are required on lower temperatures and with a higher energy capacity equipment. However, for the L80 and P110 steels, the ductile-to-brittle transition temperatures were calculated and were equal to -73.8 °C and -65.2 °C, respectively;

b) In tests on miniaturized specimens, fracture results were observed in the three characteristic regions (ductile, transition and fragile). Therefore, it was possible to calculate the transition temperature of the three materials, equal to -116.3 °C for API 5L X65, -104.7 °C for L80 and -118.9 °C for P110;

c) There was a reduction of the ductile-to-brittle transition temperature between the tests in conventional specimens for the miniaturized ones. The mean reduction value was -42.3 °C. This result reaffirms the variation behavior on state of notch stresses with the decrease of the test body, as shown by several authors.

d) The instrumentation of the tests on miniaturized specimens provided results that allow a better understanding of the mechanical behavior during impact. Highlighting the maximum impact force value and the force application behavior as a function of specimen deformation;

e) The observed fractures are typical of Charpy impact tests in the three regions (ductile, transition and fragile). The predominant failure mechanisms are plastic deformation with dimple formation for ductile fractures and almost cleavage for fragile fractures.

5. ACKNOWLEDGEMENTS

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