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Abstract

The evaluation of material fracture mechanic properties has had an increasing need, especially in the oil and gas industries. This scenario requires quality assurance of fracture toughness tests. This article describes the activities carried out when implementing the Quality Management System (QMS) for the fracture toughness tests at the Physical Metallurgy Laboratory (LAMEF) in the Federal University of Rio Grande do Sul (UFRGS), Brazil, in order to achieve the management and technical requirements of ISO/IEC 17025. Since LAMEF was previously accredited in performing other tests, most of the adequacy was related to the technical requirements of the ISO/IEC 17025 standard. After performing the required adjustments and after an external audit, the Brazilian Institute of Metrology, Quality and Technology (Inmetro) accredited LAMEF to the fracture toughness tests. The accreditation of these tests is unprecedented in Brazil, and we expect this work to encourage other Brazilian and world laboratories to seek the implementation and accreditation of QMS for fracture mechanics tests.

keywords: fracture mechanics, quality management system, ISO/IEC 17025, accreditation.

1. Introduction

The current demand for reliable test results reflects on the implementation of Quality Management Systems (QMS) according to ISO/IEC 17025 requirements in testing and calibration laboratories (Chung *et al.*, 2006; Resnizky *et al.*, 2006; Zapata-García *et al.*, 2007; Lopes *et al.*, 2014). Thus, seeking laboratory accreditation is, nowadays, a “survival requirement” (Cortez, 1999). Implementing QMS in university and research environments is challenging, but important in order to meet customer needs (Grochau *et al.*, 2010). Research institutes may see the implementation of a QMS as a chance to improve their performance (Biasini, 2012).

The Brazilian accreditation body is the General Coordination for Accreditation (Cgcre) from the National Institute

of Metrology, Quality and Technology (Inmetro). Cgcre is signatory to the mutual recognition arrangements of the International Laboratory Accreditation Cooperation (ILAC) (Brazil, 2013; Silva *et al.*, 2013). Accreditation of testing and calibration laboratories is based on NBR ISO/IEC 17025 standard, which is identical to ISO/IEC 17025 (ABNT, 2005; ABNT, 2015).

In metallurgical testing, it is important to obtain fracture toughness properties because increasingly the oil & gas industries require high performance materials. Therefore, for this application, it is indispensable to know the K_{IC} and the critical value of CTOD materials.

Fracture toughness K_{IC} of material is defined as the ability of material to resist the load in the presence of a sharp crack

before failure. The Crack Tip Opening Displacement or CTOD test measures the resistance of a material to the propagation of a crack in an elasto-plastic material (ASM, 1996).

According to the Brazilian Network of Testing Laboratories (RBLE), by 2013, no Brazilian laboratories were accredited to carry out fracture toughness tests (Inmetro, 2015). Thus, this paper intends to present the process of QMS implementation for K_{IC} and CTOD fracture toughness tests on the Physical Metallurgy Laboratory (LAMEF) from the Federal University of Rio Grande do Sul (UFRGS) until the achievement of the accreditation, and present the difficulties and strengths in this process. We also expect this work to encourage other Brazilian and world laboratories to seek the implementation

and accreditation of QMS to fracture mechanics tests.

After the implementation of the

2. Material and method

The first step of the work relates the necessary adjustment to attend the managerial requirements of ISO/IEC 17025 that affect the fracture toughness tests. As a QMS has been implemented in the Laboratory since 2008, few additional adjustments were required.

The present work describes the modifications made in order to attend

QMS to the fracture toughness tests, it was possible to notice a considerable improvement in the execution of these tests

ISO/IEC 17025 technical requirements. Grochau and Caten (2012) propose a process approach based on ten steps in the implementation of QMS in testing laboratories. Step 7, which refers to the technical requirements, is subdivided into the following steps: “Personnel requirements; Accommodation and environmental conditions; General test methods and method

in the Laboratory. Inmetro confirmed the extension of scope of the Laboratory in early 2014.

validation; Equipment requirements and Assuring the quality of test results”.

Finally, we present the adjustment to the measurement uncertainty requirements related to the measurement of K_{IC} and CTOD parameters.

Therefore, the results of this work are presented through seven stages as shown on Fig. 1.

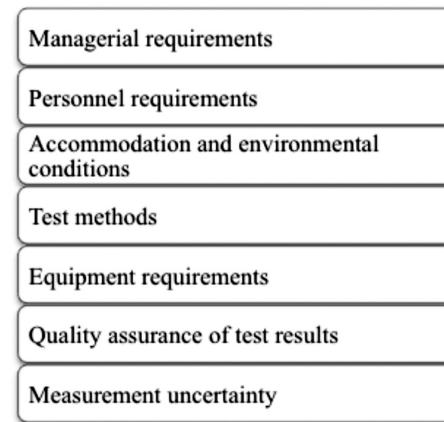


Figure 1
Implementation steps.

3. Results and Discussion

3.1 Managerial requirements

LAMEF has been accredited by Cgcre/Inmetro for performing mechanical tests since 2010. Therefore, there have been few managerial adjustments to the

fracture mechanics tests (Fabricio *et al.*, 2014). ‘Mechanical Testing’ has already been included in the LAMEF Quality Manual, eliminating any need to review

it. The test procedure and specific spreadsheets have been included in the existing master document list and inserted in the internal software for document control.

3.2 Personnel requirements

A staff of engineers carries out mechanical tests with the support of

graduate students. The list of people qualified to perform the tests has been

registered on a specific form and on a technical manager declaration.

3.3 Accommodation and environmental conditions

The room where the fracture toughness tests are carried out is the same where other tests are executed,

such that no great modifications have been necessary. The laboratory monitors and records environmental tem-

perature. The access to the testing area is controlled.

3.4 Test methods

The personnel who are qualified to perform the tests have elaborated a test procedure based on standards BS 7448-1, BS EN ISO 15653 and ASTM E1820 for the fracture toughness CTOD test, and another procedure based on BS 7448-1, BS EN ISO 15653, ASTM E399 and ASTM E1820 for the K_{IC} test. Since the standards are similar, it was possible to create a single procedure for each kind of test. Slight differences between the standards that affect the test method have been registered for

the procedures.

ASTM E399 standard is used in the plane strain fracture toughness K_{IC} testing, ASTM E1290 for critical CTOD and combined test standard ASTM E1820 for three fracture parameter (K , J , CTOD) testing (Zhu and Joyce, 2012). Meanwhile, BS 7448-1 and BS EN ISO 15653 are used for both CTOD and KIC parameters.

In the critical CTOD calculation, ASTM E1820 standard uses a conversion from critical J (J -Integral), while

BS 7448-1 and BS EN ISO 15653 standards use plastic hinge model, estimating CTOD from the crack mouth opening displacement. ASTM standards tends to give a smaller value of the critical CTOD than the evaluated in BS standards (Thagawa *et al.*, 2010). Thus, ASTM model can be considered more conservative for this parameter.

For K_{IC} calculation, measurement model is practically the same between all standards. BS 7448-1 considers as a ‘B’ value the specimen thickness, while

BS EN ISO 15653, ASTM E399 and ASTM E1820 consider $\sqrt{B_N}$ as specimen thickness, where B_N represents net thickness between side grooves in the

case of sidegrooved alternative specimens. Test conditions are basically the same between standards.

Test conditions and requisites are

basically the same between standards. The main difference between test standards refers to measurement model, as previously described.

3.5 Equipment requirements

The equipment used in the measurement of parameters that directly affect the test results is identified, controlled and calibrated annually, according to standard recommendations. The standards for fracture toughness testing indicate the following requirements for measurement

equipment (BS, 1991; ISO, 2002; BS, 2010; ASTM, 2011):

- Accuracy of $\pm 1\%$ (or better) on force measurement;
- Accuracy of $\pm 1\%$ (or better) on displacement measurement;
- Maximum error of ± 0.02 mm or $\pm 0.2\%$ on dimensional measure-

ment of test specimen.

Every time any of this apparatus is calibrated, a critical analysis of the calibration results is executed in order to verify its compliance to the test standard requirements. If any deviation is found, the equipment is taken out of service.

3.6 Quality assurance of test results

It was not possible to participate on Proficiency Tests (PT) programs, since no PT were available for CTOD/ K_{IC} tests. Neither was possible to carry out bilateral comparisons with other laboratories, since no Brazilian laboratories were accredited

on fracture toughness tests. In these cases, alternative methods can be used, such as regular use of certified reference materials, test replication, internal quality control, etc. (ABNT, 2005).

Thus, an Intra-laboratorial com-

parison between test executors has been carried out. Ten test specimens from the same material were tested (five for each operator), and the parameter K_{IC} was obtained. These values are shown in Table 1, in MPa \sqrt{m} .

| Repetition | Operator A | Operator B |
|-----------------------------------|------------|------------|
| 1 | 28.9 | 29.9 |
| 2 | 28.1 | 29.2 |
| 3 | 27.8 | 33.6 |
| 4 | 29.6 | 28.9 |
| 5 | 30.5 | 30.4 |
| Average | 29.0 | 30.4 |
| Standard deviation of the average | 0.49 | 0.84 |

Table 1
 K_{IC} values varying the test executor.

The analysis from the differences among test executors was through Analysis of Variance (Anova). Since the variance between operators (F_{calc}) was

lower than the residual variance (F_{tab}) at a confidence interval of 95%, it is possible to conclude that the differences between test executors were not significant

against the random (residual) error. This result is considered satisfactory.

This quality assurance procedure is carried out annually.

3.7 Measurement uncertainty

The standards BS 7448, ISO 12135 and BS EN ISO 15653 present the same measurement model of fracture toughness CTOD. However, there are some differences on the specimen geometry. Therefore, we have designed a test spreadsheet (which also includes uncertainty calculation) for two types of specimen geometry: compact tension

(CT) and three-point bend specimens (SEB). All spreadsheets have followed the steps of the Guide to the Expression of Uncertainty in Measurement (ISO/GUM) (JCGM, 2008). Two forms have been designed to K_{IC} test, based on the measurement model of ISO 12135 for CT and SEB specimens.

Briefly, four uncertainty spread-

sheets have been prepared. As sources of uncertainty for each of them, the uncertainty value from the calibration certificate and the equipment resolution have been considered for dimensional and force variables. The standard deviation between the results has also been considered (Fabricio and Strohaecker, 2014).

For example, the measurement model of K_{IC} for three-point bend specimens (SEB) is shown in Equation 1, and the uncertainty sources considered in

$$K_{ic} = \frac{S}{W} \frac{F_Q}{((B B_N W)^{0.5})} g_1\left(\frac{a_0}{W}\right)$$

Equation 1

| Variable | Uncertainty Source |
|--------------|---|
| F_Q | Uncertainty statement from calibration certificate and equipment resolution |
| S | Uncertainty statement from calibration certificate and equipment resolution |
| B | Uncertainty statement from calibration certificate and equipment resolution |
| B_N | Uncertainty statement from calibration certificate and equipment resolution |
| W | Uncertainty statement from calibration certificate and equipment resolution |
| $g_1(a_0/W)$ | It was not considered, since this value is considered a constant according to ISO 12135 |
| K_{IC} | Standard deviation between test specimens |

the measurement of this parameter are presented in Table 2.

Table 2
Uncertainty sources in the measurement of K_{IC} (Extracted with modification from Fabricio and Strohaecker, 2014).

3.8 Consolidation of the scope

After the adequacy and an external audit, the scope of accreditation to fracture toughness tests was consolidated as shown on Table 3.

| Field of activity / Product | Test description | Standard / Procedure |
|---|-------------------------------------|-----------------------|
| Metallurgy / Metallic materials and welds | CTOD fracture toughness testing | BS 7448-1:1991 |
| | | BS EN ISO 15653:2010 |
| | | ASTM E1290:2008e1 |
| | | ASTM E1820:2011 |
| | | Petrobras N-1678:2009 |
| | | Petrobras N-1859:2005 |
| | K_{IC} fracture toughness testing | BS 7448-1:1991 |
| | | BS EN ISO 15653:2010 |
| | | ASTM E399:2009e2 |
| | | ASTM E1820:2011 |

Table 3
Fracture toughness testing accreditation scope.

4. Conclusions

After the implementation of the required adjustments, it was possible to extend the QMS based on ISO/IEC 17025 to the fracture toughness tests. By meeting the standard requirements, it was possible to obtain the accreditation by Cgcre/Inmetro for K_{IC} and

CTOD tests in early 2014. LAMEF was the first (and, so far, the only) Brazilian laboratory to obtain accreditation by Cgcre/Inmetro for fracture toughness tests.

We expect to encourage other testing laboratories to seek accreditation by

conformity assessment organisms, especially those laboratories that perform mechanical fracture tests. Thus, it will be possible to stimulate and improve the measurement of mechanical properties of metallic materials, especially for the oil & gas industries.

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tory's management for the encouragement in this process.

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