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Reference material development process for tensile test method

Çekme test metodu için referans malzeme geliştirme yöntemi

Alper İNCESU[1]* iD *, Betül ERCAN[1]* iD *, Engin ÇEVİK[1]* iD *, Yasin AKGÜL[1]* iD

[1]MARGEM Laboratories, Iron and Steel Institute, Karabük University, Karabük, Turkey.
alperincesu@karabuk.edu.tr, betulusta@karabuk.edu.tr, engincevik@karabuk.edu.tr, yasinakgul@karabuk.edu.tr

Abstract

Reference materials (RMs) are one of the basic materials used by the laboratories to prove the reliability of their test results. In this research, preliminary studies have been carried out to develop RM to use in tensile tests according to ISO 6892-1. For this purpose, bone shape flat specimens were prepared from EN 10130:2006 quality 1 mm thick sheet steel related to the given standard. Homogeneity tests and inter-laboratory comparison tests were performed for calculation of measurement uncertainty and value assignment of proposed RM. Results were compared with commercial certified reference materials (CRMs). It was found that relative uncertainty values of the proposed RM were coherent with commercial CRMs. Therefore, these produced RM can be used in quality control studies for laboratories, also by the production route proposed in this study, every laboratory can be produced RM related to their needs.

Keywords: Reference material, ISO 6892-1, Tensile test, Steel.

Öz

Referans malzemeler (RM'ler), laboratuvarların test sonuçlarının güvenilirliğini kanıtlamak için kullandıkları temel malzemelerden biridir. Bu araştırmada, ISO 6892-1'e göre çekme testlerinde kullanılmak üzere RM'nin geliştirilmesi için ön çalışmalar yapılmıştır. Bu amaçla, verilen standarda uygun EN 10130: 2006 kalite 1 mm kalınlığında çelik sacdan kemik şeklinde yassı numuneler hazırlanmıştır. Önerilen RM'nin ölçüm belirsizliği ve değer atamasının hesaplanması için homojenlik testleri ve laboratuvarlar arası karşılaştırma testleri yapılmıştır. Sonuçlar ticari sertifikalı referans malzemelerle (CRM'ler) karşılaştırıldı. Önerilen RM'nin göreceli belirsizlik değerlerinin ticari CRM'ler ile uyumlu olduğu bulundu. Bu nedenle üretilen bu RM'ler laboratuvarlar için kalite kontrol çalışmalarında kullanılabilir, ayrıca bu çalışmada önerilen üretim yolu ile her laboratuvar kendi ihtiyaçlarına göre RM üretebilir.

Anahtar kelimeler: Referans malzeme, ISO 6892-1, Çekme testi, çelik.

1 Introduction

Testing and analysis services provided by independent organizations are of great importance for international sales of commercial products to be possible. The market for global testing and analysis services stood at US$ 17.9 bn in 2014 and is anticipated to rise at a compound annual growth rate (CAGR) of 5.6 % from 2015 to 2023. The opportunities in the market are projected to reach US$ 29.1 bn by the end of the forecast period [1]. Accreditation is very crucial in terms of the international reliability and competitiveness of laboratories. The national and international validity of the reports prepared by the laboratories is only provided by accreditation [2]. Accuracy of the operator within the scope of accreditation is provided by the reference materials (RMs) whose properties are determined on the calibrated test devices. RMs are employed in laboratory accreditation and all stages of the measurement process, including method validation, in-house calibration, quality control as well as in inter-laboratory comparisons [3],[4]. Besides, RMs can be used to estimate measurement uncertainty and calibration test devices [5]. Annual sales growth rates of 10-20 % in RMs have been observed for the last few years and it is expected to increase further as accredited laboratory awareness increases [6].

The reference materials by ISO are divided into two classes as certified reference materials (CRMs) and reference materials (RMs) [2]. Both RMs and CRMs are homogeneous and stable materials that provide certain property values with traceable deviation values according to the related test method [7]. RMs are divided into 5 main categories [8]; chemical composition, biological and clinical properties, physical properties, engineering properties, and miscellaneous properties. Some engineering properties of materials can be determined by a tensile test that is a crucial method to characterize the mechanical properties of metallic materials.

Assigned values and measurement uncertainties are given in the certificates of CRMs that are sold by global manufacturers like the National Institute of Standards and Technology (USA), Joint Research Center (Belgium), HRT Labortechnik GmbH (Germany), etc. However, fewer publications are explaining how these values are obtained by statistical assessments, especially in the field of mechanical testing under the engineering properties category of CRMs [5],[9]. In this study, studies will be discussed related to independent analytical methods in different laboratories, and obtained data were statistically tested for material homogeneity, equality of means, normality, and outliers to produce the RMs from flat type specimen from EN 10130:2006 [10] quality 1 mm thick sheet steel for tensile test method according to ISO 6892-1 metallic materials [11].

2 Methodology & Results

2.1 Project design of reference material production

Reference material production is mainly composed of several steps as defined in ISO Guide 35:2017 [7]. As explained in ISO 10989, reference materials have several categories and

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*Corresponding author/Yazışılan Yazar

subcategories. So, first of all, the selection of true related categories and subcategories of reference material about the related sector is very important. After selecting the right area, the second important issue is the material from which properties will be obtained depending on the desired parameters. In the next stage, it is another important argument to prepare samples correctly from the material in a way that does not impair the actual properties of the material and to ensure that these prepared samples are correctly packaged and stored. Homogeneity is a very important matter to ensure that every unit of reference material has the same value for a property that specified. Sufficient stability for their intended use should be guaranteed by RMs. Customers as end-users should be able to rely on the assigned value at any time within the validity period of the certificate. After all these studies, CRM production is completed with certificate preparation according to the data obtained and stability monitoring. The ISO 17043 accreditation is required for the processes taken into the box with dashed lines in Figure 1.

In this study, RM produced with its assigned values and their uncertainties without certification. The main steps for the production and maintenance of reference material were given as schematically outlined in Figure 1.
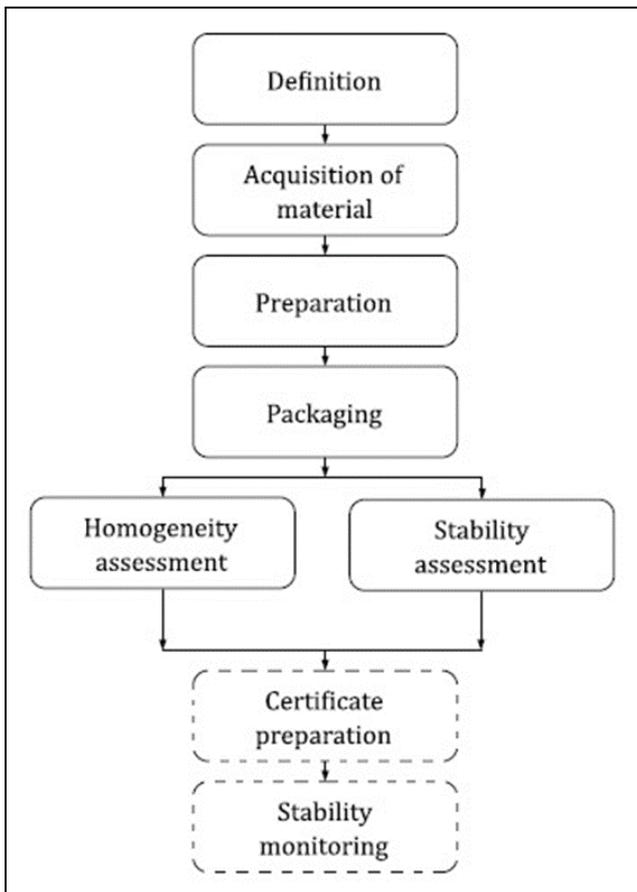
Figure 1. Main steps for production and maintenance of reference material [7].

Since the tensile test is one of the preferred test methods to determine the mechanical properties both in scientific studies [12],[13] and commercial activities, the production of reference material for tensile testing has been taken into consideration in this study. EN 10130:2006 quality 1 mm thick sheet steel was selected as a material for determination of mechanical

properties of yield strength (Rp0.2), tensile strength (Rm), and elongation (A) as the production of reference material under the category of engineering properties. To obtain these properties from the selected material, the tensile test was performed related to ISO 6892-1 standard method B at room temperature of about 23±5 °C [8]. Sheet steel with 1000 mm x 1000 mm dimensions and 1 mm thickness was supplied from Erdemir, Turkey. Tensile test specimen sampling was made from on this sheet steel material. The steel sheet supplied for making a tensile sample is a commercial product, means that it has to show the same mechanical and chemical properties in all its regions. Therefore, the entire surface of the sheet was used in the direction of the rolling mill to extract the tensile samples. Then, random test batches were prepared from the samples. 100 tensile test specimens that had standard dimensions (Figure 2) were prepared related to ISO 6892-1 standard. The wire cutting method was performed to tolerate uncertainties because of sample preparation.
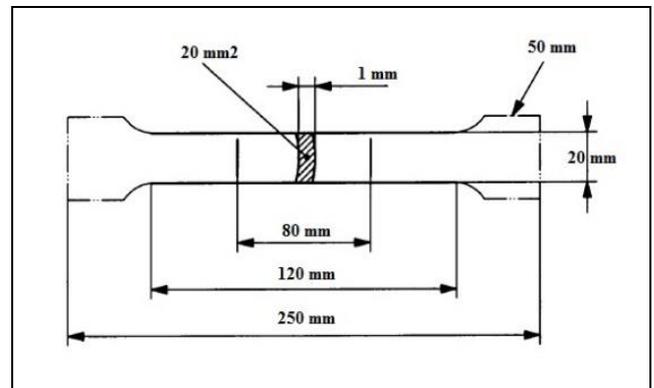
Figure 2. Dimensions of tensile test specimen [8].

2.2 Stability

EN 10130:2006 quality 1 mm thick sheet steel is used for preparing all the samples. Chemical composition of steel sheet was given in Table 1.

Table 1. Chemical composition of EN 10130:2006 quality steel sheet (wt.%).

C	Mn	P	S	Si	Al	Fe
0.041	0.253	0.008	0.008	0.023	0.043	Bal.

This is commercial steel that has certified values as min. 224.6 MPa yield strength (Rp0.2), min. 325.6 MPa tensile strength (Rm) and min. 32% elongation (A). There is no uncertainty or standard deviations were given in the certificate of the material related to duration. [14] states that reference material should have an expiration date where instability has been demonstrated or is considered possible, after which reference value is no longer guaranteed. Roebben and Lamberty reported that the uncertainty contribution from instability is considered to be insignificant for certification of Charpy V-notch (mechanical test) reference test pieces [9]. According to Brammer Standard Company, Inc., there is no expiration date is specified CRM for tensile testing due to fact that it is solid and stable [15]. To sum up, it is not appropriate to mention an exact expiration date for reference materials produced from metals.

2.3 Homogeneity studies & Assessments

Homogeneity and stability are two crucial characteristics of RMs and CRMs [9]. 100 test pieces were prepared, and 28 test specimens were randomly chosen within these pieces. Two methods followed to determine assigned values and uncertainty of homogeneity studies;

- – Randomly selected 10 samples tested by the same operator and same testing machine,
- – Sample groups of 3 randomly selected test samples were tested in different laboratories by an organization of inter-laboratory comparison tests.

All laboratories where homogeneity and interlaboratory comparison tests have been carried out have TS EN ISO 17025 accreditation for the TS EN ISO 6892-1 tensile test method on metallic materials. The calibrations of the test equipment, the competence of the test operators and the suitability of the environmental conditions (temperature and humidity) were guaranteed by the accreditation of the TS EN ISO 17025: General Requirements for the Competence of the Test and Calibration Laboratories provided by the requirements of the TS EN ISO 6892-1 standard.

Route for stability and homogeneity studies were given in Figure 3.
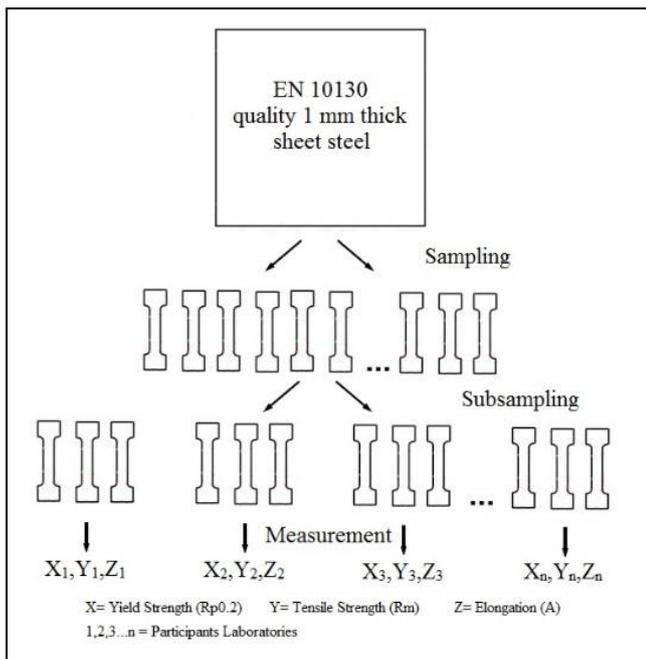
Figure 3 Route for stability and homogeneity studies.

Table 2 shows the homogeneity test results of specimens. Tests were performed by the same operator using the same testing machine. It can be observed that the tensile properties of the prepared samples provide the product standard values (EN 10130:2006 quality sheet steel).

Table 3 shows the inter-laboratory comparison test results. Only the elongation values of lab.1 and lab.2 were below the EN 10130:2006 quality sheet steel values of the material given in Table 3. However, these values (32.73±1.64 and 30.85±1.64) exceed the minimum value with the addition of the standard deviation values to average values. Therefore, the results of these two laboratories are included in the calculation of value assignment and measurement uncertainty.

Table 2. Homogeneity test results of specimens.

	$R_{p0.2}$ (MPa)	R_m (MPa)	A (%)
Specimen1	276	375	34.2
Specimen2	258	371	34.3
Specimen3	284	374	34.1
Specimen4	279	371	33.3
Specimen5	266	376	33.5
Specimen6	284	374	34.9
Specimen7	268	371	33.2
Specimen8	262	370	33.5
Specimen9	278	374	34.8
Specimen10	285	376	33.8
Average Value	274.0	373.2	34.0
EN 10130:2006 quality sheet steel	224.6 (min.)	325.6 (min.)	32 (min.)
Standart Deviation (δ)	9.31	2.14	0.57
Standard Uncertainty (u_{hom})	2.94	0.68	0.18

Table 3. Inter-laboratory comparison test results.

	Average Value of $R_{p0.2}$	Average Value of R_m	Average Value of A
Lab.1	291	384.5	32.73
Lab.2	279	376.7	30.85
Lab.3	271	372.3	32.50
Lab.4	275	386.0	35.40
Lab.5	272	396.0	31.90
Lab.6	278	373.3	34.23
Average Value	278.0	381.5	32.94
Standard Deviation (δ)	7.4	9.1	1.64
Standard Uncertainty ($u_{int.lab}$)	3.02	3.72	0.67

The certified value was obtained from an inter-laboratory comparison with 6 laboratories. Calculation of uncertainties of homogeneity, inter-laboratory comparison, and reproducibility were given in equations of Eq. 1, Eq. 2, respectively.

$$u_{hom} = \frac{\delta}{\sqrt{n}} \tag{1}$$

$$u_{int.lab} = \frac{\delta}{\sqrt{n}} \tag{2}$$

Standard uncertainty (u_{hom}) was calculated according to Eq.1 while standard uncertainty ($u_{int.lab}$) was calculated according to Eq. 2. The measurement uncertainty values obtained in the homogeneity tests were lower than the measurement

uncertainty values obtained in the inter-laboratory comparison experiments. The reason for this is that homogeneity tests were carried out by a single operator on the same device, and comparisons between laboratories were carried out by different operators on different devices. Operator and different test devices are included in the system as a variable. As a matter of fact, Shamsuri and Darus [16] detected differences between the uncertainties even in their study with the same device and 3 different operators. The aim of this study is to determine the maximum measurement uncertainty range that can be obtained with the uncertainty values assigned, no matter which laboratory, in which test device and by which operator the samples are tested.

Standard uncertainty values were compared with reproducibility uncertainty of ZStE 180 sheet steel in Table 4. Uncertainty values are coherent with ZStE 180 sheet steel. For $Rp_{0.2}$ and elongation values, u_{hom} and $u_{int.lab}$ were lower than ZStE 180 sheet steel.

In ISO 6892-1 standard, values are given as 95% level of confidence with coverage factor k=2. Thus, reproducibility uncertainty values of ZStE 180 sheet steel were divided by two to express standard uncertainty. Uncertainty values of prepared samples and ZStE 180 sheet steel were given in Table 4.

Table 4. Uncertainty values of prepared samples and ZStE 180 sheet steel.

Uncertainty Values	Average Value of $R_{p0.2}$	Average Value of R_m	Average Value of A
Standard Uncertainty (u_{hom})	2.94	0.68	0.18
Standard Uncertainty ($u_{int.lab}$)	3.02	3.72	0.67
ZStE 180 sheet steel Reproducibility Uncertainty [11]	4.95	2.10	4.95

2.4 Values assignment & Uncertainty calculation

The process of combining the results obtained from the stability assessment and homogeneity with the results obtained from the characterization studies to determine the assigned values and uncertainties is called value assignment. The product information sheet or certificate contains these assigned values and uncertainties [7]. The mean value of individual results may also be adopted as the assigned value where differences between data set means are insignificant compared with the effects of variation within each data set.

A weighted mean is usually calculated using the general form of equation (Eq. 3):

$$y_{char} = \sum_{i=1}^{p} w_i x_i / \sum_{i=1}^{p} w_i \tag{3}$$

y_{char}= is assigned value.

w_i = is the weight applied to each data set mean xi.

x_i = value from data set.

Total uncertainty values calculations were given in equations of Eq. 4, Eq. 5, Eq. 6.

$$u_{Rp0.2} = \sqrt{u_{hom-Rp0.2}^2 + u_{int.lab-Rp0.2}^2} \tag{4}$$

$$u_{Rm} = \sqrt{u_{hom-Rm}^2 + u_{int.lab-Rm}^2} \tag{5}$$

$$u_A = \sqrt{u_{hom-A}^2 + u_{int.lab-A}^2} \tag{6}$$

The extended measurement uncertainties are given at k=2 and 95% confidence interval. Calculation of extended measurement uncertainty formulas were given as equations of Eq. 7, Eq. 8, Eq. 9.

$$U_{Rp0.2} = u_{Rp0.2} * k \tag{7}$$

$$U_{Rm} = u_{Rm} * k \tag{8}$$

$$U_A = u_A * k \tag{9}$$

Table 5 shows the reference values of produced reference material by EN 10130:2006 quality 1 mm thick sheet steel for tensile test method related to standard ISO 6892-1. Relative measurement uncertainty ($U_{rel.}$) was calculated according to Eq 10. $U_{rel.}$ values of our produced RM and commercial CRMs were compared in Table 6.

Table 5. Reference tensile properties of produced RM.

Property	Assigned value (y_{char})	Measurement uncertainty (U)
Yield strength ($Rp_{0.2}$)	275.5 MPa	8.42 MPa
Tensile strength (Rm)	376.3 MPa	7.56 MPa
Elongation (A)	33.60 %	1.39 %

Table 6. Tensile properties of commercial CRMs.

Property	Relative measurement uncertainty ($U_{rel.}$)		
	CRM-1	CRM-2	RM (Produced)
Yield strength ($Rp_{0.2}$)	0.0155	0.0303	0.0306
Tensile strength (Rm)	0.0062	0.0147	0.0201
Elongation (A)	0.0204	0.1553	0.0414

CRM-1 and CRM-2 were fabricated by European Commission Directorate General Joint Research Centre (Belgium) and Institut fur Eignungsprüfung GmBH (Germany), respectively [17],[18]. The proposed RM can be used as RM for in-house quality control studies. Furthermore, it can be commercialized as CRM by certification procedure.

$$U_{rel.} = \frac{U}{y_{char}} \tag{3}$$

3 Conclusion

In this study, RM for tensile test related to standard ISO 6892-1 was produced, and steps for assigning reference values and calculation of measurement uncertainties were explained in detail. The assigned values with measurement uncertainties are 275.5 MPa ±8.42 MPa, 376.3 MPa ±7.56 MPa, and 33.60% ±1.39% for yield strength, tensile strength, and elongation, respectively. It can be said that relative uncertainty values of

the proposed RM were coherent with commercial CRMs. Thus, results of the study show that properties of produced RM can be alternative to commercial CRMs for in-house quality control studies of laboratories. It is envisaged to set an example for reference materials that can be produced under the heading of engineering properties for the production of reference materials.

4 Author contribution statements

In the scope of this study, the Alper İNCESU and Betül ERCAN in the formation of the idea, the design and the literature review; Engin ÇEVİK in the assessment of obtained results, supplying the materials used and examining the results; the Yasin AKGÜL the spelling and checking the article in terms of content were contributed.

5 Ethics committee approval and conflict of interest statement

There is no need to obtain permission from the ethics committee for the article prepared. There is no conflict of interest with any person/institution in the article prepared.

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