Institute for Reference Materials and Measurements

European Reference Materials

CERTIFICATION REPORT

Certification of Charpy V-notch reference test pieces of 60 J nominal absorbed energy

Certified Reference Material ERM®-FA014q

EUR 24407 EN – 2010

EUROPEAN COMMISSION

The mission of the JRC-IRMM is to promote a common and reliable European measurement system in support of EU policies.

European Commission
Joint Research Centre
Institute for Reference Materials and Measurements

Contact information
Reference materials sales
Retieseweg 111
B-2440 Geel, Belgium
E-mail: jrc-irmm-rm-sales@ec.europa.eu
Tel.: +32 (0)14 571 705
Fax: +32 (0)14 590 406

http://irmm.jrc.ec.europa.eu/
http://www.jrc.ec.europa.eu/

JRC 58889

EUR 24407 EN
ISBN 978-92-79-07561-2
ISSN 1018-5593
DOI 10.2787/26083

Luxembourg: Publications Office of the European Union

Printed in Belgium

CERTIFICATION REPORT

Certification of Charpy V-notch reference test pieces of 60 J nominal absorbed energy

Certified Reference Material ERM®-FA014q

G. Roebben, A. Dean, A. Lamberty

European Commission, Joint Research Centre
Institute for Reference Materials and Measurements (IRMM), Geel (BE)

Disclaimer

Abstract

This certification report describes the processing and characterisation of ERM®-FA014q, a batch of Charpy V-notch certified reference test pieces. Sets of five of these test pieces are used for the verification of pendulum impact test machines according to EN 10045-2 (Charpy impact test on metallic materials, Part 2. Method for the verification of impact testing machines [1]) or according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of test machines [2]). The certified value for *KV* (= energy required to break a V-notched test piece using a pendulum impact test machine) is 61.9 J. The associated expanded uncertainty (1.2 J, $k = 2$ corresponding to a confidence level of about 95 %) is calculated for the mean of a set of five test pieces.

The certified property is defined by the Charpy impact test procedure as described in EN 10045-1 [3] and ISO 148-1 [4]. The certified values are made traceable to the International System of Units (SI) through the corresponding Master Batch ERM®-FA014c of the same nominal absorbed energy (60 J), by testing samples of ERM®-FA014c and ERM®-FA014q in repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at (20 ± 2) °C.

Contents

Glossary

AISI	American Iron and Steel Institute
BCR	Community Bureau of Reference
CRM	Certified reference material
EC	European Commission
EN	European Standard
ERM®	European Reference Material
g	Gravitation acceleration
IMB	International Master Batch
IRMM	Institute for Reference Materials and Measurements
ISO	International Organisation for Standardization
JRC	Joint Research Centre
k	Coverage factor
KV	Absorbed energy = energy required to break a V-notched test piece of defined shape and dimensions when tested with a pendulum impact testing machine
KV_{CRM}	Certified *KV* value of a set of 5 reference test pieces from the Secondary Batch
KV_{MB}	Certified *KV* value of the Master Batch test pieces
LNE	Laboratoire National de Métrologie et d'Essais
MB	Master Batch
min	Minute
n_{MB}	Number of samples of the Master Batch tested during certification of the Secondary Batch
n_{SB}	Number of samples of the Secondary Batch tested for certification
RSD	Relative standard deviation
s	Standard deviation
SB	Secondary Batch

s_h	Standard deviation of the results of the samples of the SB tested to assess its homogeneity
SI	International System of Units
s_{MB}	Standard deviation of n_{MB} results obtained on samples of the MB tested for the certification of the SB
s_{SB}	Standard deviation of n_{SB} results obtained on samples of the SB tested for its characterisation
u_{CRM}	Combined standard uncertainty of KV_{CRM}
U_{CRM}	Expanded uncertainty (k = 2, confidence level 95 %) of KV_{CRM}
u_{char}	Standard uncertainty of the result of the characterisation measurements
$u_{char,rel}$	Relative standard uncertainty of the result of the characterisation measurements
u_h	Homogeneity contribution to standard uncertainty
u_{MB}	Standard uncertainty of KV_{MB}
$u_{MB,rel}$	Relative standard uncertainty of KV_{MB}
$\overline{x}_{MB}$	Mean KV value of the n_{MB} measurements on samples of the MB tested when characterising the SB
$\overline{x}_{SB}$	Mean KV value of the n_{SB} results of the samples of the SB tested for its characterisation
Δh	Difference between the height of the centre of gravity of the pendulum prior to release and at end of first half-swing, after breaking the test sample
ν_{RM}	Effective number of degrees of freedom associated with the uncertainty of the certified value

1 Introduction: the Charpy pendulum impact test

The Charpy pendulum impact test is designed to assess the resistance of a material to shock loading. The test, which consists of breaking a notched bar of the test material using a hammer rotating around a fixed horizontal axis, is schematically presented in Figure 1.

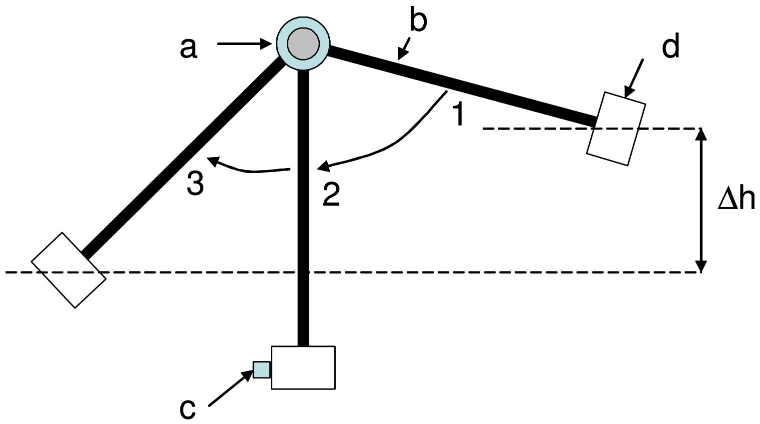

Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft onto which d is fixed: the hammer. The hammer is released from a well-defined height (position 1). When the hammer has reached maximum kinetic energy (shaft in vertical position 2), the hammer strikes c: the test sample, which is positioned on a support and against the pendulum anvils (not shown). The height reached by the hammer after having broken the sample (position 3) is recorded. The difference in height between position 1 and 3 (Δh) corresponds with a difference in potential energy, and is a measure of the energy required to break the test sample.

The energy absorbed by the test sample is very dependent on the impact pendulum construction and its dynamic behaviour. Methods to verify the performance of an impact pendulum require the use of reference test pieces as described in European, American and ISO standards [1, 2, 5]. The reference test pieces dealt with in this report comply with a V-notched test piece shape of well-defined geometry [1, 2], schematically shown in Figure 2.

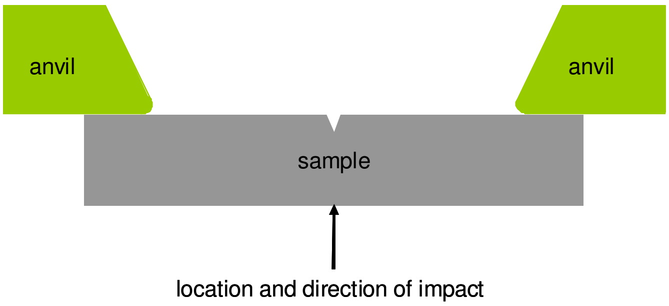

Figure 2: Schematic drawing of a V-notched Charpy sample (top-view), indicating the place and direction of impact.

2 The certification concept of Master Batch and Secondary Batch

2.1 Master and Secondary Batches

The BCR reports by Marchandise et al. [6] and Varma [7] provide details of the certification of the BCR "Master Batches" (MB) of Charpy V-notch certified reference test pieces. The certified value of a Master Batch is obtained using an international laboratory intercomparison.

This report describes the production of a "Secondary Batch" (SB) of Charpy V-notch certified reference test pieces at the Institute for Reference Materials and Measurements (IRMM) of the European Commission's (EC) Joint Research Centre (JRC). The work was performed in accordance with procedures described in the BCR reports [6] and [7]. The certification of a SB is based on the comparison of a set of SB test pieces with a set of test pieces from the corresponding MB under repeatability conditions on a single pendulum.

Since the uncertainty of the certified value of the MB contributes to the uncertainty of the certified value of the SB, the latter is necessarily larger than the former. Nevertheless, as will be shown also in this report, the uncertainty can be kept sufficiently small to meet the requirements of the intended use of the certified reference material (CRM). The MB-SB approach allows cost-efficient production of certified reference test pieces as it successfully transfers the results of the costly international interlaboratory comparison on the MB, to each produced SB.

The BCR reports [6] and [7] were published in 1991 and 1999, respectively. Since 2000, the calculation of the certified value and the estimation of its uncertainty have been updated to an approach compliant with the ISO/IEC Guide 98-3:2008 on the expression of uncertainty in measurement [8]. This revised approach was developed and presented by Ingelbrecht et al. [9, 10], and is summarised below.

2.2 Certification of a Secondary Batch of Charpy V-notch test pieces

The certified absorbed energy of a SB of Charpy V-notch reference test pieces (KV_{CRM}) is calculated from the mean *KV*-value of a set of SB-samples ($\overline{X}_{\text{SB}}$) tested on a single pendulum. This value $\overline{X}_{\text{SB}}$ has to be corrected for the bias of this particular pendulum. The bias of the pendulum at the moment of testing the samples of the SB, is estimated by comparing the mean *KV*-value of a number of samples of the MB ($\overline{X}_{\text{MB}}$), tested together with the SB samples under repeatability conditions, with the certified value of the MB (KV_{MB}). KV_{CRM} is then calculated as follows [10]:

$$KV_{\text{CRM}} = \left[\frac{KV_{\text{MB}}}{\overline{X}_{\text{MB}}} \cdot \overline{X}_{\text{SB}} \right] \qquad \text{Eq. 1}$$

For this approach to be reliable, the pendulum used for the tests on MB and SB in repeatability conditions, must be well performing. In other words, the ratio $\frac{KV_{MB}}{\overline{X}_{MB}}$ must be close to 1. IRMM allows a difference of 5 % (KV_{MB} > 40 J) or 2 J (KV_{MB} < 40 J) between KV_{MB} and $\overline{X}_{MB}$, corresponding with the level of bias allowed for reference pendulums specified in EN 10045-2 [1] and ISO 148-3 [11].

Also, for reasons of commutability, a comparable response of the pendulum to the MB and SB samples is required. This is the reason why MB and SB samples are made from nominally the same steel and why it is checked that the ratio $\frac{KV_{CRM}}{KV_{MB}}$ is close to 1. IRMM allows a difference of 20 % (KV_{MB} > 40 J) or 8 J (KV_{MB} < 40 J) between KV_{CRM} and KV_{MB} to ensure that the MB and SB samples have a comparable interaction with the pendulum.

2.3 Uncertainty of the certified value of a Secondary Batch of Charpy V-notch test pieces

The uncertainty of the certified value of the SB is a combination of the uncertainties of the right-hand side factors in Eq. 1. It is clear that the MB-SB approach necessarily results in a larger uncertainty of the certified value of SB in comparison with the MB. The additional uncertainty depends on the uncertainty of the ratio $\overline{X}_{SB} / \overline{X}_{MB}$. The full measurement uncertainty of the values $\overline{X}_{MB}$ and $\overline{X}_{SB}$ is relatively large. However, when all conditions mentioned above (repeatability conditions, pendulum performance, and commutability between Secondary and Master Batch) are fulfilled, then the uncertainties of the values $\overline{X}_{MB}$ and $\overline{X}_{SB}$ have several contributions in common, in particular the uncertainty due to the bias of the pendulum. These shared uncertainty components do not contribute to the uncertainty of the ratio $\overline{X}_{SB} / \overline{X}_{MB}$, and only the standard deviations of the SB and MB results in the MB-SB comparison test need to be taken into account (see also section 5.3).

3 Participants

The processing of the SB (ERM®-FA014q) test pieces was carried out by the Laboratoire National de Métrologie et d'Essais (LNE), Trappes (FR), using steel bars produced at Cogne Acciai Speciali, Aosta (IT). The MB (ERM®-FA014c) samples used in the characterisation of the SB were provided by IRMM, Geel (BE). The homogeneity of the SB was evaluated based on data obtained at LNE using a pendulum verified according to the criteria imposed by EN 10045-2 [1] and ISO 148-2 [2]. Characterisation of the SB was carried out at IRMM using a pendulum verified according to the criteria imposed by EN 10045-2 [1] and ISO 148-2 [2]. Data evaluation was performed at IRMM.

4 Processing

The ERM®-FA014q test pieces were prepared from AISI 4340 steel. The steel was cast and rolled into bars at Cogne Acciai Speciali (see section 4.1). Production of the test pieces from these bars was performed under the supervision of LNE (see sections 4.2, 4.3, 4.4, and 4.5).

4.1 Processing of hot-rolled bars

The base material consisted of AISI 4340 steel, produced at Cogne Acciai Speciali. To limit the amount of impurities potentially affecting the homogeneity of the fracture resistance, the following compositional tolerances were imposed on the selected steel batch: Mn 0.7 – 0.8, Mo 0.23 - 0.28, Ni 1.7 – 1.85, P < 0.01, Si 0.2 – 0.35, S < 0.008 (mass fractions, in %), which is stricter than generally allowed for AISI 4340. The ingot was hot-rolled, resulting in bars that were 4 m long and with a squared cross-section of 11.5 mm. For the ERM®-FA014q batch, steel was used from ingot number 960133, billet E. A billet is a semi-finished hot-rolled product, in this case of cross-section 108.5 mm, which is between the ingot (560 mm cross-section) stage and the final required bars (11.5 mm cross-section). A full description of the processing and quality check of the steel bars is available in [12].

4.2 Heat treatment of hot-rolled bars

The heat treatment of the hot-rolled bars was performed at Aubert&Duval, Gennevilliers (FR). 22 bars were heat-treated together. Bars were placed onto rollers which slowly move the bars back and forth inside the furnace during the heat treatment to increase the homogeneity of the resulting microstructure. The first heat treatment was an austenisation treatment performed in a furnace of 'class 10 °C' [1] at 850 °C for 30 min. From this furnace, the bars were quenched into oil at 40 °C. After the oil-quench, the samples were annealed in a furnace of 'class 5 °C' at 350 °C for 120 min. After this annealing treatment, the samples were cooled down in air. A second annealing treatment was carried out in a furnace of 'class 5 °C' at 530 °C for 120 min, after which the samples were cooled down in air.

After heat treatment, a limited number of samples (5) were machined for a preliminary check of the obtained energy level. Results indicated an average *KV*-level (64 J), sufficiently close to the target energy (60 J).

4.3 Machining of Charpy test pieces

After the heat treatment the samples were machined to dimensional tolerances imposed in EN 10045-2 [1]. The batch code (Q60) and the individual sample code were engraved twice on one long face of each sample, on both sides of the notch, for fully traceable sample coding also after fracture ('60' indicates the previously used nominal absorbed energy level (60 J); 'Q' is

[1] In a furnace of 'class x °C', the variation of the temperature is smaller than x °C. The furnaces used have 10 heating zones. Each zone has 3 controlling thermocouples and 3 measurement thermocouples. These are regularly calibrated. When one faulty thermocouple is detected, it is replaced by a thermocouple produced with wire from the same roll. When a roll is exhausted, all thermocouples are replaced with new ones.

the letter code as assigned consecutively to batches of the same nominal absorbed energy). The V-notch was introduced using an electro-erosion tool.

4.4 Quality control

When all samples from the batch were fully machined, a selection of 25 samples was made. The dimensions of the 25 samples were checked on December 10, 2007 against the criteria specified in EN 10045-2 [1] (length $55.0^{0.0}_{0.25}$ mm, height 10.00 ± 0.06 mm, width 10.00 ± 0.075 mm, notch angle 45 ± 1 °, height remaining at notch root 8.00 ± 0.06 mm, radius at notch root 0.25 ± 0.025 mm, distance between the plane of symmetry of the notch and the longitudinal axis of the test piece 27.50 ± 0.10 mm). None of the samples were outside the ranges specified in EN 10045-2 [1].

The 25 samples checked for geometrical compliance were impact tested on December 11, 2007 on the Tinius Olsen 358 Joules pendulum - which is one of the French reference pendulums - at LNE. The results are reported in certificate LNE n° F120699/CQPE/2 [13]. The average *KV* of the 25 samples was 62.2 J, which is within the desired energy range (57 J to 63 J). The standard deviation of the test results (*s* = 0.8 J, *RSD* = 1.3 %) was smaller than the maximum allowed 3 %. The variation was checked again during the certification tests at IRMM (see section 5).

4.5 Packaging and storage

Finally, the samples were cleaned and packed in sets of 5, in oil-filled and closed plastic bags. These oil-filled bags, together with a label, again were packed in a sealed plastic bag, and shipped to IRMM. After arrival (January 2008), the 1300 samples (or 260 sets) were registered and stored at room temperature, pending distribution.

5 Characterisation

5.1 Characterisation tests

30 samples from ERM®-FA014q (sets 2, 6, 12, 20, 23 and 29) were tested under repeatability conditions with 25 samples from MB ERM®-FA014c (sets 8, 14, 26, 37 and 41), using the Instron Wolpert PW 30 (serial number 7300 H1527, Instron, High Wycombe, UK) machine of IRMM, an impact pendulum yearly verified according to procedures described in EN 10045-2 [1] and ISO 148-2 [2]. Tests were performed on October 6, 2009 (laboratory temperature 20 ± 1 °C), in accordance with EN 10045-1 [3] and ISO 148 [4]. The measured absorbed energy values were corrected for friction and windage losses as described in ISO 148-2 [2].

An analysis of the normality of the distributions of the measured *KV* values did not indicate a skewed distribution towards higher or lower absorbed energy values. Three stragglers were detected (Grubbs' test, 95 % confidence level, 1 of the 25 MB samples, 2 of the 30 SB samples), but no technical reason was identified that could justify their elimination from the analysis. All data were accepted.

The accepted data obtained on individual test pieces are shown in Figure 3 and in Annex 1. In Figure 3 linear trend-lines are added to illustrate the absence of a significant trend in the results with the analysis sequence (not for the SB and not for the MB; both checked with ANOVA; confidence level > 95 %). The results of the measurements are summarised in Table 1.

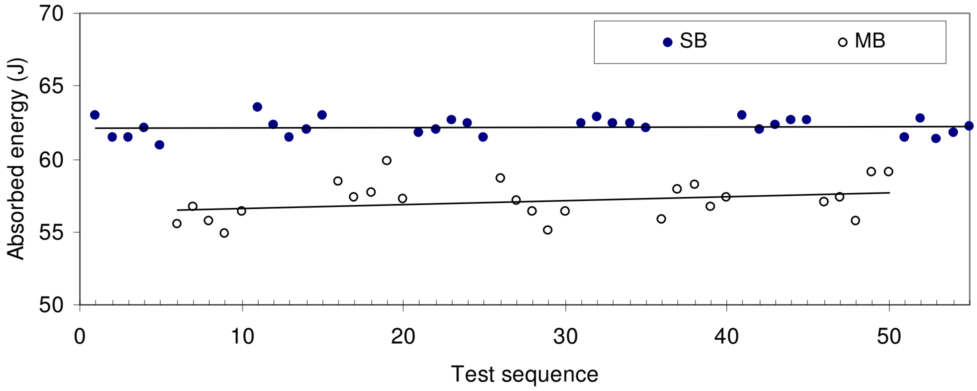

Figure 3: Absorbed energy values of the 30 test pieces of SB (ERM®-FA014q) and 25 test pieces of MB (ERM®-FA014c) displayed in the actual test sequence.

Table 1: Results of characterisation of Batch ERM®-FA014q by comparison with Master Batch ERM®-FA014c.

	Number of test pieces	**Mean value**	**Standard deviation**	**Relative standard deviation**
	n	$\overline{X}$ (J)	*s* (J)	*RSD* (%)
ERM®-FA014c (MB)	25	57.11	1.30	2.28
ERM®-FA014q (SB)	30	62.19	0.61	0.98

The SB-results meet the EN 10045-2 and ISO 148-3 acceptance criteria for a batch of reference materials (RSD < 5 %), as well as the more stringent acceptance criterion (RSD < 3 %) contractually fixed between IRMM and its sample supplier for batches of this nominal absorbed energy level (60 J).

5.2 Data from Master Batch ERM®-FA014c

To calculate KV_{CRM} for ERM®-FA014q one needs KV_{MB} of the MB used, i.e. ERM®-FA014c. Table 2 shows the main MB-data, taken from the Certificate of Analysis of ERM®-FA014c (Annex 2).

Table 2: Certified value and uncertainties from the certification of Master Batch ERM®-FA014c.

	Certified absorbed energy of Master Batch KV_{MB} (J)	**Standard uncertainty of KV_{MB}** u_{MB} (J)	**Standard uncertainty of KV_{MB}** $u_{MB,rel}$ (%)
ERM®-FA014c	56.8	0.4	0.7

5.3 Calculation of KV_{CRM} and of u_{char}

From the data in Table 1 and Table 2, and using Eq. 1, one readily obtains that KV_{CRM} = 61.9 J. The uncertainty associated with the characterisation of the SB, u_{char}, is assessed as in Eq. 2 [10], which sums the relative uncertainties of the three factors in Eq. 1:

$$u_{char} = KV_{CRM}\sqrt{\frac{u_{MB}^{\;2}}{KV_{MB}^2} + \frac{s_{SB}^2}{n_{SB}\cdot \overline{X}_{SB}^2} + \frac{s_{MB}^2}{n_{MB}\cdot \overline{X}_{MB}^2}} \quad \text{Eq. 2}$$

$\overline{X}_{SB}$ and $\overline{X}_{MB}$ were obtained under repeatability conditions. Therefore, the uncertainty of the ratio $\overline{X}_{SB} / \overline{X}_{MB}$ is not affected by the contributions from reproducibility and bias of the pendulum used to compare MB and SB. Table 3 summarises the input quantities of the u_{char} uncertainty budget, their respective statistical properties, and shows how they were combined. The effective number of degrees of freedom for u_{char} is obtained using the Welch-Satterthwaite equation [8].

Table 3: Uncertainty budget for u_{char}

Symbol	Source of uncertainty	Measured value (J)	Standard uncertainty (J)	Probability distribution	Divisor	Sensitivity coefficient	Relative uncertainty (%)	Degrees of freedom
KV_{MB}	certification of MB	56.8	0.4	normal	1	1	0.7	11
$\overline{X}_{SB}$	comparison of SB and MB under repeatability conditions	62.19	0.11	normal	1	1	0.18	29
$\overline{X}_{MB}$		57.11	0.26	normal	1	1	0.46	24
						$u_{char,rel}$ (%)	0.86	22
						u_{char} (J) (calculated from $u_{char,rel}$ and for KV_{CRM} = 61.9 J)	0.53	ν_{char}

6 Homogeneity

The test pieces are sampled from the SB, which is sufficiently, but not perfectly, homogeneous. Therefore, an appropriate homogeneity contribution u_h to the uncertainty of the certified value is required. u_h is related to s_h, the standard deviation between the samples in the SB (*sample-to-sample heterogeneity*), but also depends on the number of samples over which the *KV*-value is averaged. EN 10045-2 [1] and ISO 148-2 [2] specify that the pendulum verification must be performed using 5 test pieces, which is why a CRM-unit consists of a set of 5 test pieces. The appropriate uncertainty contribution must be an estimate of the *set-to-set heterogeneity*, which in the case of a set of 5 test pieces can be calculated as $u_h = \frac{s_h}{\sqrt{5}}$.

Here, u_h is estimated from the results obtained at IRMM on October 6, 2009 (s_h = 0.61 J), which leads to $u_h = \frac{s_h}{\sqrt{5}} = 0.27\ \text{J}$. As is required for a homogeneity test, the samples were randomly selected from the whole batch. The number of samples tested (30) is sufficiently large to reflect the homogeneity of the full SB (1300 samples). It can be noted that u_h is probably a slight overestimation, since it contains also the repeatability of the instrument. However, the latter cannot be separated or separately measured.

7 Stability

The stability of the absorbed energy of Charpy V-notch certified reference test pieces was first systematically investigated for samples of nominally 120 J by Pauwels et al., who did not observe measurable changes of absorbed energy [14]. New evidence for the stability of the reference test pieces produced from AISI 4340 steel of lower energy levels (nominally 15 J, 30 J and 100 J) has been obtained recently, during the International Master Batch (IMB) project [15]. In the IMB-project, the stability of the certified test pieces is judged from the change of the mean of means of the absorbed energy obtained on 7 reference pendulums over a three year period. None of the three regression slopes for the tested energy levels was statistically significant at the 95 % confidence level. Given the large sample-to-sample heterogeneity and the limited number of samples (5) in a CRM unit, the uncertainty contribution from instability is considered to be insignificant in comparison to that of homogeneity.

The main reason for the microstructural stability of the certified reference test pieces is the annealing treatment to which the samples were subjected after the austenisation treatment. Annealing is performed at temperatures where the equilibrium phases are the same as the (meta-)stable phases at ambient temperature (α-Fe and Fe_3C). The only driving force for instability stems from the difference in solubility of interstitial elements in the α-Fe matrix, between annealing and ambient temperature. Relaxation of residual (micro-)stress by short-range diffusion or the additional formation or growth of precipitates during the shelf-life of the certified reference test pieces is expected to proceed, but slowly.

Rather than neglecting the stability issue, efforts are spent to better establish the stability of the certified values of batches of Charpy CRMs. Until such further notice, it is decided to specify a limited shelf-life. A period of 10 years is chosen, counting from the date of the characterisation tests on the SB. Since batch ERM®-FA014q was characterised in October 2009, the validity of the certificate stretches until October 2019.

8 Calculation of combined and expanded uncertainty

As shown in section 5.3, KV_{CRM} = 61.9 J. The uncertainty of the certified value is obtained by combining the contributions from the characterisation study, u_{char}, and from the homogeneity assessment, u_h, as is summarised in the following uncertainty budget (Table 4).

The relevant number of degrees of freedom was calculated using the Welch-Satterthwaite equation, which is a standard equation to calculate the degrees of freedom of a combined uncertainty [8]. The result is sufficiently large (ν_{RM} = 33) to justify the use of a coverage factor k = 2 to expand the confidence level to about 95 %. The obtained expanded uncertainty provides justification for the SB-MB approach followed: U_{CRM} is sufficiently smaller than the verification criterion of 10 % or even 5 % for reference pendulums [11].

Table 4: Uncertainty budget for KV_{CRM}

Uncertainty symbol	Source	Value (J)	Degrees of freedom
u_{char}	characterisation of SB	0.53	22
u_h	homogeneity of SB	0.27	29
Combined standard uncertainty, u_{CRM}		0.60 J	33
Expanded Uncertainty, k = 2, U_{CRM}		1.2 J	ν_{RM}

9 Metrological traceability

The certified *property* is defined by the Charpy pendulum impact test procedure as described in EN 10045-1 [3] and ISO 148-1 [4].

The certified *value* of the MB ERM®-FA014c is traceable to the SI as it was obtained using an interlaboratory comparison, involving a representative selection of qualified laboratories performing the tests in accordance with the standard procedures, on pendulums verified with SI-traceably calibrated tools [16].

The certified value of ERM®-FA014q is made traceable to the certified value of the MB using tests on SB and MB samples under repeatability conditions on a pendulum verified with SI traceably calibrated tools. Therefore, the certified *value* of ERM®-FA014q is traceable to the International System of Units (SI) via the corresponding Master Batch ERM®-FA014c of the same nominal absorbed energy (60 J). Absorbed energy *KV* is a method-specific value, and can only be obtained by following the procedures specified in EN 10045-1 [3] and ISO 148 [4].

10 Commutability

The intended use of the certified reference test pieces is the verification of Charpy impact pendulums. During the certification of the MB, different pendulums were used, each equipped with an ISO-type striker of 2 mm tip radius. Until further notice, the certified values are not to be used when the test pieces are broken with an ASTM-type striker of 8 mm tip radius, although the expected differences can be small for materials with relatively low absorbed energy such as the ERM®-FA014 batches of 60 J nominal absorbed energy [14].

11 Summary: certified value and uncertainty

The certified value and associated uncertainty are summarised in Table 5.

Table 5: Certified value and associated uncertainty for ERM®-FA014q.

Steel Charpy V-notch test pieces		
	Certified value 2) [J]	Uncertainty 3) [J]
Absorbed energy (*KV*) 1)	61.9	1.2

1) The absorbed energy (*KV*) is a method defined measurand. *KV* is the impact energy required to break a V-notched bar of standardised dimensions as defined in EN 10045-1 and ISO 148-1. The certified value is valid only for strikers with a 2 mm tip radius, and in the temperature range of 20 ± 2 °C.

2) The certified *KV* value of batch ERM®-FA014q, and its uncertainty, are traceable to the International System of Units (SI), via the Master Batch ERM®-FA014c of the same nominal absorbed energy (60 J).

3) Estimated expanded uncertainty of the mean *KV* of the 5 specimens (delivered as 1 set), with a coverage factor $k = 2$, corresponding to a level of confidence of about 95 %, as defined in the ISO/IEC Guide 98-3:2008; Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM:1995).

12 Instructions for use

12.1 Intended use

Samples of ERM®-FA014q correspond to the '(certified) BCR test pieces' as referred to in EN 10045-2 [1], as well as to the 'certified reference test pieces' as defined in ISO 148-3 [11]. Sets of five of these certified reference test pieces are intended for the indirect verification of impact testing machines with a striker of 2 mm tip radius according to procedures described in detail in EN 10045-2 [1] and ISO 148-2 [2].

The indirect verification provides an assessment of the bias of the user's Charpy pendulum impact machine. The bias value can be used in the calculation of the measurement uncertainty of Charpy tests on the pendulum after indirect verification [2]. Such uncertainty calculation requires the certified value, the associated uncertainty, and in some cases also the degrees of freedom of the uncertainty, all given on page 2 of the certificate.

12.2 Sample preparation

Special attention is drawn to the cleaning of the specimens prior to the tests. It is mandatory to remove the oil from the sample surface prior to testing, without damaging the edges of the sample. Between the moment of removing the protective oil layer and the actual test, corrosion can occur. This must be avoided by limiting this period of time, while keeping the sample clean.

The following procedure is considered a good practice.

1. First use absorbent cleaning-tissue to remove the excess oil. Pay particular attention to the notch of the sample, but do not use hard (e.g. steel) brushes to remove the oil from the notch.
2. Submerge the samples in ethanol for about 5 min. Use of ultrasonication is encouraged, but only if the edges of the samples are prevented from rubbing against each other. To reduce the consumption of solvent, it is allowed to make a first cleaning step with detergent, immediately prior to the solvent step.
3. Once samples are removed from the solvent, only manipulate the samples wearing clean gloves. This is to prevent development of corrosion between the time of cleaning and the actual test.
4. Before testing, bring the specimens to the test temperature (20 ± 2 °C). To assure thermal equilibrium is reached, move the specimens to the test laboratory at least 3 h before the tests.

12.3 Pendulum impact tests

After cleaning, the 5 samples constituting a CRM-unit need to be broken with a pendulum impact test machine in accordance with EN 10045-2 [1] or ISO 148-2 [2] standards. Prior to the tests, the anvils must be cleaned. It must be noted that Charpy test pieces sometimes leave debris on the Charpy pendulum anvils. Therefore, the anvils must be checked regularly and if debris is found, it must be removed. The uncertainty of the certified value applies to the mean of the 5 *KV*-values obtained from one CRM unit.

Acknowledgements

The authors wish to thank M. Ricci, A. Bernreuther, R. Koeber, T. Linsinger and H. Emons (all IRMM) and the experts of the Certification Advisory Panel "Physical and physicochemical properties', J. Dusza (Institute of Materials Research, Slovak Academy of Sciences, Kosiče, Slovakia), N. Jennett (National Physics Laboratory, Teddington, United Kingdom) and M. Stintz (T.U. Dresden, Germany) for reviewing of the certification report.

References

1. *EN 10045-2 Charpy impact test on metallic materials - Part 2. Method for the verification of impact testing machines*, European Committee for Standardization, Brussels (BE), 1993.
2. *ISO 148-2: Metallic materials - Charpy pendulum impact test - Part 2: Verification of testing machines*, International Organization for Standardization, Geneva (CH), 2008.
3. *EN 10045-1: Charpy impact test on metallic materials - Part 1. Test method (V- and U-notches)*, European Committee for Standardization, Brussels (BE), 1990.
4. *ISO 148-1: Metallic materials - Charpy pendulum impact test - Part 1: Test method*, International Organization for Standardization, Geneva (CH), 2006.
5. *ASTM E 23 - 02a Standard Test Methods for Notched Bar Impact Testing of Metallic Materials*, C. E-28, Editor. ASTM International, West Conshohocken (PA, USA), 2002.
6. Marchandise, H., Perez-Sainz, A., Colinet, E., *Certification of the impact toughness of V-notch Charpy specimens*, in *BCR information series*. Community Bureau of Reference - BCR, Brussels (BE), 1991.
7. Varma, R.K., *The certification of two new master batches of V-notch Charpy impact toughness specimens in accordance with EN 10045-2: 1992*, in *BCR information series*. Office for the Official Publications of the European Communities, Luxembourg (LU), 1999.
8. *ISO/IEC Guide 98-3:2008; Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*, International Organization for Standardization, Geneva (CH), 2008.
9. Ingelbrecht, C. and Pauwels J., *EC Reference Materials for Impact Toughness - Traceability and uncertainty*. Presentation at Eurachem Eurolab symposium on Reference Materials for Technologies in the New Millennium, Berlin (DE), May 22-23, 2000.
10. Ingelbrecht, C., Pauwels, J., and Gyppaz, D., *Charpy specimens from BCR for machine verification according to EN 10045-2*. Poster presentation at Charpy Centenary Conference, Poitiers (FR), October 2-5, 2001.
11. *ISO 148-3: Metallic materials - Charpy pendulum impact test - Part 3: Preparation and characterization of Charpy V-notch test pieces for indirect verification of pendulum impact machines*, International Organization for Standardization, Geneva (CH), 2008.
12. Gyppaz, D., *Elaborazione e verifica acciaio SAE 4340 per provette Charpy V di riferimento - colata 960133*, Cogne Acciai Speciali, Aosta (IT), 1999.
13. Lefrançois, S., *Certificat d'étalonage F120699/CQPE/2*. Laboratoire National de métrologie et d'essais, Trappes (FR), 2007.
14. Pauwels, J., Gyppaz, D., Varma, R., Ingelbrecht, C. *European certification of Charpy specimens: Reasoning and observations*. in *Pendulum impact testing: A century of progress,* eds. Siewert, T. A., Manahan, M. P. Sr., Seattle (Washington), American Society for Testing and Materials, West Conshohocken (PA, USA), 1999.

15. McCowan, C. N., Roebben, G., Yamaguchi, Y., Lefrançois, S., Splett, J. D., Takagi, S., Lamberty, A., *International comparison of impact reference materials (2004).* J. ASTM International, Vol. 3 (2), 2006.
16. *ISO Guide 35: Reference materials - General and statistical principles for certification,* International Organization for Standardization, Geneva (CH), 2005.

Annex 1

Results of characterisation measurements of ERM®-FA014q as measured according to EN 10045-1 and ISO 148 at IRMM, October 6, 2009.

	Master Batch **ERM®-FA014c**	**Secondary Batch** **ERM®-FA014q**
	***KV* (J)**	***KV* (J)**
1	55.53	62.93
2	56.66	61.43
3	55.69	61.43
4	54.89	62.09
5	56.34	60.93
6	58.45	63.56
7	57.38	62.30
8	57.70	61.47
9	59.82	61.97
10	57.21	62.97
11	58.64	61.80
12	57.17	61.97
13	56.36	62.63
14	55.08	62.47
15	56.43	61.47
16	55.86	62.40
17	57.86	62.90
18	58.19	62.40
19	56.73	62.40
20	57.38	62.07
21	57.08	62.97
22	57.40	61.97
23	55.69	62.30
24	59.10	62.63
25	59.10	62.63
26		61.43
27		62.76
28		61.33
29		61.83
30		62.16
Mean (J)	**57.11**	**62.19**
Standard deviation (J)	**1.30**	**0.61**
***RSD* (%)**	**2.28**	**0.98**

Annex 2

Institute for Reference Materials and Measurements

European Reference Materials

CERTIFICATE OF ANALYSIS

ERM®- FA014c

CHARPY V-notch test pieces - Master Batch (nominal absorbed energy 60 J)		
Parameter	Certified value [1] (J)	Uncertainty [2] (J)
Absorbed energy (KV) at 20 ± 2 °C according to EN 10045-1 and ISO 148	56.8	0.4

1) Mean absorbed energy of test pieces from batch ERM-FA014c, estimated as the mean of means of absorbed energies measured at 12 laboratories. At each laboratory, 10 test pieces were broken. The certified value is traceable to the Charpy impact test method as described in EN 10045-1 and ISO 148.

2) Standard uncertainty *u* of the certified mean absorbed energy of batch ERM-FA014c, estimated as the standard deviation of the mean of the 12 laboratory mean values, corresponding with a level of confidence of about 68 %.

This certificate is valid until December 2010; this validity may be extended as further evidence of stability becomes available.

NOTE

European Reference Material ERM®-FA014c was originally certified as BCR-014C. It was produced and certified under the responsibility of IRMM according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-IRMM-LGC. Information on these guidelines is available on the Internet (http://www.erm-crm.org).

Accepted as an ERM®, Geel, November 2004

Signed: ______

Prof. Dr. Hendrik Emons
Unit for Reference Materials
EC - JRC- IRMM
Retieseweg 111
2440 Geel, Belgium

All following pages are an integral part of the certificate.

Page 1 of 3

DESCRIPTION OF THE SAMPLE

A unit consists of five Charpy V-notch test pieces, which are rectangular steel bars of nominal 55 mm x 10 mm x 10 mm, with one V-notch, accurately machined to dimensions imposed in EN-10045-1 and ISO 148. The five specimens are packed together in a plastic bag filled with oil to prevent oxidation.

INSTRUCTIONS FOR USE

Samples of ERM-FA014c correspond with the '(certified) BCR test pieces' as referred to in EN 10045-2 (Method for the verification of impact testing machines), as well as with the 'certified reference test pieces' as defined in ISO 148-3 (Preparation and characterisation of Charpy V reference test pieces for verification of test machines).

The ERM-FA014c batch is one of the 'Master Batches'. Master Batch test pieces are not for sale. They are intended solely to traceably certify Secondary Batches of the same nominal absorbed energy (here 60 J). The certified value and associated uncertainty of the Master Batch are used in the calculation of the combined uncertainty of the certified value of a set of 5 specimens from a Secondary Batch. Sets of 5 samples of Secondary Batches are distributed as certified reference test pieces for the verification of Charpy impact test machines in accordance with EN 10045-2 and ISO 148-2.

When characterising a secondary batch, (a minimum of) 25 Master Batch test pieces are broken in repeatability conditions together with a selection of samples from the secondary batch. Special attention is drawn to cleaning of the specimens prior to testing. The following procedure is recommended:

1. Wipe excess oil from the specimens with cellulose paper.
2. Immerse the specimens in a clean bath of degreasing solvent for about five minutes.
3. Wipe the specimens with cellulose paper and allow to dry in still air.
4. Before testing, allow the specimens to equilibrate to laboratory temperature for about 12 hours.

After cleaning, the user must avoid touching the specimens with the fingers (wear clean gloves). Vigorous cleaning methods affecting the roughness of the specimen surface or possibly causing deformation/indentation of the specimen edges should be avoided, as this can result in obtaining erroneous data.

Charpy test pieces sometimes leave debris on the Charpy pendulum anvils. Therefore, the anvils must be checked regularly and if debris is found, it must be removed.

STORAGE

Specimens should be kept at ambient temperature in their original packing until they are used.

SAFETY INFORMATION

Precautions need to be taken to avoid injury of operator by broken specimens while operating the Charpy impact pendulum.

METHOD USED FOR CERTIFICATION

Charpy pendulum impact tests in accordance with EN 10045-1 and ISO 148.

PARTICIPANTS

- Aubert et Duval, Gennevilliers (FR)
- Aubert et Duval, Les Ancizes (FR)
- Bundesanstalt für Materialforschung und –prüfung (BAM), Berlin (DE)
- Centre de Recherches et d'Etudes d'Arceuil (FR)
- Cermet, Bologna (IT)

- Cogne Acciai Speciali, Aosta (IT)
- Equipos Nucleares, Santander (ES)
- Franco Tosi Industriale, Legnano (IT)
- GTS Industries, Dunkerque (FR)
- Laboratoire National d'Essais, Paris (FR)
- National Physical Laboratory, Teddington (UK)
- Sollac, Dunkerque (FR)
- Staatliches Materialprüfungsamt, Dortmund (DE)
- Ugine, Isbergues Research Centre (FR)

LEGAL NOTICE

Neither IRMM, its contractors nor any person acting on their behalf:

(a) make any warranty or representation, express or implied, that the use of any information, material, apparatus, method or process disclosed in this document does not infringe any privately owned intellectual property rights; or

(b) assume any liability with respect to, or for damages resulting from, the use of any information, material, apparatus, method or process disclosed in this document save for loss or damage arising solely and directly from the negligence of IRMM.

NOTE

A detailed technical report of the Master Batch certification project ("Certification of the impact toughness of V-notch Charpy specimens", H. Marchandise, A. Perez-Sainz, E. Colinet, bcr information, XII/323/91-EN, 1991) can be obtained from IRMM on explicit request. In this report, the ERM-FA014c batch is called '60 C' (60 J is the nominal energy of the ERM-FA014 batches).

European Commission – Joint Research Centre
Institute for Reference Materials and Measurements (IRMM)
Retieseweg 111, B - 2440 Geel (Belgium)
Telephone: +32-(0)14-571 722 - Telefax: +32-(0)14-590 406

European Commission

EUR 24407 EN – Joint Research Centre – Institute for Reference Materials and Measurements
Title: Certification of Charpy V-notch reference test pieces of 60 J nominal absorbed energy, ERM®-FA014q
Author(s): G. Roebben, A. Dean, A. Lamberty
Luxembourg: Publications Office of the European Union
2010 – 23 pp. – 21.0 x 29.7 cm
EUR – Scientific and Technical Research series – ISSN 1018-5593
ISBN 978-92-79-07561-2
DOI 10.2787/26083

Abstract

This certification report describes the processing and characterisation of ERM®-FA014q, a batch of Charpy V-notch certified reference test pieces. Sets of five of these test pieces are used for the verification of pendulum impact test machines according to EN 10045-2 (Charpy impact test on metallic materials, Part 2. Method for the verification of impact testing machines [1]) or according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of test machines [2]). The certified value for *KV* (= energy required to break a V-notched test piece using a pendulum impact test machine) is 61.9 J. The associated expanded uncertainty (1.2 J, k = 2 corresponding to a confidence level of about 95 %) is calculated for the mean of a set of five test pieces.
The certified property is defined by the Charpy impact test procedure as described in EN 10045-1 [3] and ISO 148-1 [4]. The certified values are made traceable to the International System of Units (SI) through the corresponding Master Batch ERM®-FA014c of the same nominal absorbed energy (60 J), by testing samples of ERM®-FA014c and ERM®-FA014q in repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at (20 ± 2) °C.

How to obtain EU publications

Our priced publications are available from EU Bookshop (http://bookshop.europa.eu), where you can place an order with the sales agent of your choice.

The Publications Office has a worldwide network of sales agents. You can obtain their contact details by sending a fax to (352) 29 29-42758.

The mission of the JRC is to provide customer-driven scientific and technical support for the conception, development, implementation and monitoring of EU policies. As a service of the European Commission, the JRC functions as a reference centre of science and technology for the Union. Close to the policy-making process, it serves the common interest of the Member States, while being independent of special interests, whether private or national.

LA-NA-24407-EN-C

EUROPEAN COMMISSION

ISBN 978-92-79-07561-2

Publications Office