Institute for Reference Materials and Measurements

ERM European Reference Materials

CERTIFICATION REPORT

Certification of Charpy V-notch reference test pieces of 150 J nominal absorbed energy

Certified Reference Materials ERM®-FA415p and ERM®-FA415r

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CERTIFICATION REPORT

Certification of Charpy V-notch reference test pieces of 150 J nominal absorbed energy

Certified Reference Materials ERM®-FA415p and ERM®-FA415r

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Disclaimer

Summary

This certification report describes the processing and characterisation of ERM®-FA415p and ERM®-FA415r, two batches 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 testing machines [2]). The certified value for *KV* (= energy required to break a V-notched test piece using a pendulum impact test machine) and the associated uncertainty ($k = 2$ corresponding to a confidence level of about 95 %) calculated for the mean of a set of five test pieces, are:

Batch-code	**Certified KV-value**	**Expanded uncertainty** (k = 2, confidence level of about 95 %)
ERM®-FA415p	155 J	6 J
ERM®-FA415r	151 J	7 J

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 traceable to the International System of Units (SI) through the corresponding Master Batch ERM®-FA415b of the same nominal absorbed energy (150 J).

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Glossary

AISI	American Iron and Steel Institute
ASTM	American Society for Testing and Materials
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, Joint Research Centre, European Commission
ISO	International Organization for Standardization
JRC	Joint Research Centre, European Commission
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
MB	Master Batch
m	Mass of pendulum
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_{MB} Standard deviation of n_{MB} results obtained on samples of the Master Batch tested for the certification of the Secondary Batch

s_{SB} Standard deviation of n_{SB} results obtained on samples of the Secondary Batch 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_h Homogeneity contribution to uncertainty

u_{MB} Standard uncertainty of KV_{MB}

$\overline{X}_{MB}$ Mean KV value of the n_{MB} measurements on samples of the Master Batch tested when characterising the Secondary Batch

$\overline{X}_{SB}$ Mean KV value of the n_{SB} results of the samples of the Secondary Batch 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.

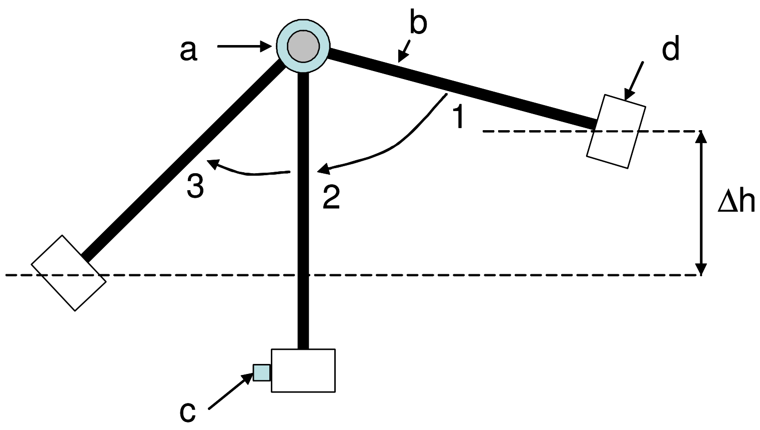

Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft onto which is fixed d: the hammer, of mass m. The hammer is released from a well-defined height (position 1). The hammer strikes c: the test sample, when the hammer has reached maximum kinetic energy (shaft in vertical position 2). 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 (= $m \times g \times \Delta h$, with g = gravitation acceleration), and is a measure of the energy required to break the test sample.

The energy absorbed by the test sample depends 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, ISO and American 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.

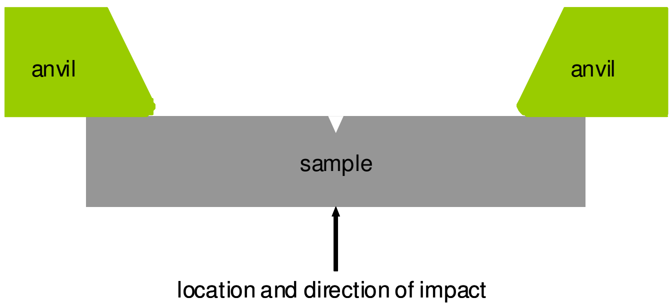

Figure 2: Schematic drawing of a V-notched Charpy sample (top-view), indicating the place and direction of impact.

2 The certification concept of Master Batch and Secondary Batch

2.1 Difference between 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 interlaboratory comparison.

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 (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 Guide to 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 ($\overline{X}_{\mathrm{SB}}$) tested on a single pendulum. This value $\overline{X}_{\mathrm{SB}}$ 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 ($\overline{X}_{\mathrm{MB}}$), 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]:

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

For this approach to be reliable, the pendulum used for the tests on MB and SB in repeatability conditions, must be well performing. This can be checked by comparing the certified value of the MB, KV_{MB}, with the results obtained on the MB samples when comparing SB and MB, $\overline{X}_{MB}$. IRMM allows a difference of 5 % (if $KV_{MB} > 40$ J) or 2 J (if $KV_{MB} < 40$ 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 steel with nominally the same chemical composition, and similar heat treatments. These precautions have to result in a ratio $\frac{KV_{CRM}}{KV_{MB}}$ 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}.

3 Participants

The processing of the SB test pieces was carried out by Cogne Acciai Speciali, Aosta (IT). The MB samples used in the characterisation of the SB were provided by IRMM, Geel (BE). 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®-FA415p and ERM®-FA415r test pieces were prepared from ASTM 565 steel. The steel was cast and rolled into bars at Cogne Acciai Speciali (see section 4.1). Heat treatment and production of the test pieces from these bars were performed later, also at Cogne Acciai Speciali (see sections 4.2 and 4.3).

4.1 Processing of hot-rolled bars

Bars were produced at Cogne Acciai Speciali according to a contractually agreed internal Cogne procedure PQU-483 [12].

The base material consisted of ASTM 565 – Grade XM32 steel, produced at Cogne Acciai Speciali [13]. To limit the amount of impurities potentially affecting the homogeneity of the fracture resistance, composition tolerances stricter than generally allowed for ASTM 565 were imposed on the selected steel batch (tolerances specified in Table 1).

Table 1: Adapted composition tolerances of ASTM 565 Grade XM32.

composition (mass %)						
C	S	P	Si	Mn	Cr	Ni
0.11 - 0.13	< 0.003	0.018	0.15 – 0.3	0.15 – 0.3	11.25 – 11.65	2.55 – 2.75
Mo	Cu	Al	V	W	N	
1.55 – 1.7	< 0.2	< 0.01	0.25 – 0.3	< 0.1	0.025 - 0.04	

For the ERM®-FA415p and ERM®-FA415r batches, steel was used from ingot number 360404. The ingot billets were hot rolled, resulting in bars that were 4 m long and with a squared cross-section of 14 mm.

4.2 Cutting, heat-treatment and final machining of test pieces

At Cogne Acciai Speciali, the hot rolled bars were turned into accurately machined Charpy test pieces of the appropriate impact toughness according to a contractually agreed internal Cogne procedure PQU-484 [14].

The hot rolled bars were cut into rectangular beams of 58 mm length. These rectangular beams were machined to 55 mm length and 11 mm x 11 mm cross-section. The batch code (3, P (or R), 160) was engraved on one end face of each sample ('3' indicates the steel type (ASTM 565); '160' indicates the target absorbed energy level; 'P (or R)' is the letter as assigned consecutively to batches of nominally the same absorbed energy).

The heat treatment of these test pieces was performed in salt baths. Samples were put into steel wire baskets containing 16 samples each. The baskets were fixed at regular spacing on a frame, collecting nominally 420 samples, which were immersed into two salt baths. Prior to immersion in the salt baths, the homogeneity of the temperature in these baths was checked with a calibrated mobile thermocouple unit.

In the first salt bath an austenisation treatment was performed (nominally 30 min at a temperature near 1000 °C, inside the austenite area in the phase diagram). From this bath, samples were quenched into oil at 40 °C, and further cooled down in air. After the oil-quench, the samples were annealed twice in a second salt bath at temperatures around 700 °C, each time for about 120 min. After this annealing treatment, the samples were cooled down in oil. The suitability of the temperatures of the second bath, which strongly affect the KV value of the resulting samples, was checked in a dedicated test run.

After the heat treatment the samples were machined to dimensional tolerances imposed in EN 10045-2 [1]. First, the cross-section was reduced to 10 mm x 10 mm, respecting the orthogonality of the four long faces of the bar. Then the surface was finished to roughness $R_a < 0.8$ µm. Finally, the V-notch was introduced using a diamond grinding tool with the appropriate V-profile.

4.3 Packaging and storage

Samples were cleaned and packed in sets of 5, in oil-filled plastic bags. These oil-filled bags were closed and, together with a BCR-label, again packed in a sealed plastic bag, and shipped to IRMM in 2001. After arrival, the samples (~ 80 sets of 5 samples for each batch) were registered and stored at room temperature, pending distribution.

After approval of the Charpy Secondary Batch approach by the European Reference Materials consortium, in 2004, the original BCR-labels were replaced at IRMM by ERM®-labels, keeping the same batch-code (415p and 415r) and set numbers.

5 Characterisation

5.1 Characterisation tests

20 samples from batch ERM®-FA415p (sets 1, 40, 52 and 67) and 20 samples from batch ERM®-FA415r (sets 1, 22, 39 and 66) were tested under repeatability conditions with 30 samples from MB ERM®-FA415b (sets 38, 42, 46, 68, 72 and 80), 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]. Half of the tests were performed on February 24 (10 samples of the secondary batches, 15 samples of the Master batch) and the other half on February 25 (laboratory temperature 20 ± 1 °C), in accordance with EN 10045-1 [3] and ISO 148-1 [4]. The measured absorbed energy values were corrected for friction and windage losses as described in ISO 148-2 [2]. Data obtained on individual test pieces are shown in Figure 3 and in Figure 4, and in Annex 1. The results of the measurements are summarised in Table 2.

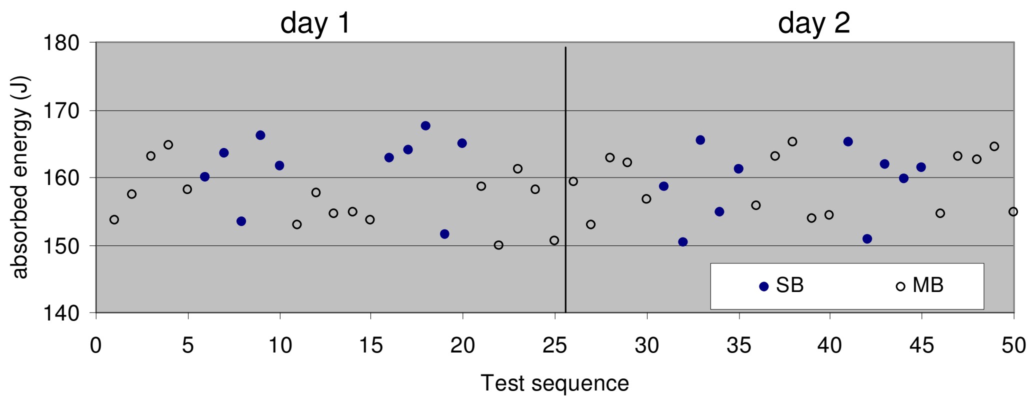

Figure 3: Absorbed energy values of the 20 test pieces of ERM®-FA415p and 30 test pieces of ERM®-FA415b displayed in the actual test sequence.

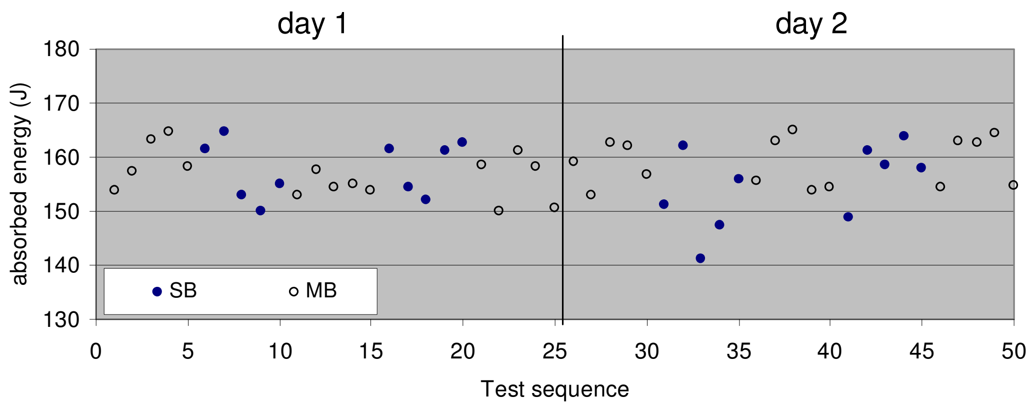

Figure 4: Absorbed energy values of the 20 test pieces of ERM®-FA415r and 30 test pieces of ERM®-FA415b displayed in the actual test sequence; Note: the MB results in Figure 3 and Figure 4 are identical: in between every set of 5 MB test pieces two set of 5 test pieces corresponding to the 2 SBs were tested.

Table 2: Characterisation measurements of Batches ERM®-FA415p and ERM®-FA415r.

	Number of test pieces	**Mean value**	**Standard deviation**	**Relative standard deviation**
	n_{MB} , n_{SB}	$\overline{X}_{MB}$, $\overline{X}_{SB}$ (J)	s_{MB} , s_{SB} (J)	*RSD* (%)
ERM®-FA415b (MB)	30	157.82	4.45	2.8
ERM®-FA415p (SB)	20	160.23	5.32	3.3
ERM®-FA415r (SB)	20	156.17	6.36	4.1

The relative standard deviations of the SB-results meet the EN 10045-2 [1] and ISO 148-3 [11] acceptance criteria for a batch of reference materials (s_{SB} < 5 %). In addition, the difference between $\overline{X}_{MB}$ and $\overline{X}_{SB}$ is smaller than 20 %, the level used to assess the similarity of Master Batch and Secondary Batch behaviour (see section 2.2).

5.2 Data from Master Batch ERM®-FA415b

To calculate KV_{CRM} for ERM®-FA415p and ERM®-FA415r one needs KV_{MB} of the MB used, i.e. ERM®-FA415b. Table 3 shows the main MB-data, taken from the Certificate of Analysis of ERM®-FA415b (Annex 2), which is the revised, ERM®-version of the originally issued certificate, based on the certification report of the MB [7]. The certified value was obtained from an interlaboratory comparison with 9 laboratories.

Table 3: Data from the certification of Master Batch ERM®-FA415b [7].

	Certified absorbed energy of Master Batch	**Standard uncertainty of KV_{MB}**	**Standard uncertainty of KV_{MB}**
	KV_{MB} (J)	u_{MB} (J)	u_{MB} (%)
ERM®-FA415b	152.4	1.1	0.7

The values KV_{MB} (Table 3) and $\overline{X}_{MB}$ (Table 2) are less than 5 % different, confirming that the pendulum used for the characterisation of the secondary batch is functioning with a sufficiently low bias (see section 2.2).

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

From the data in Table 2 and Table 3, and using Eq. 1, one readily obtains KV_{CRM}. The results are summarised in Table 4.

Table 4: Results of the calculation of KV_{CRM} from Eq. 1.

	ERM®-FA415p	**ERM®-FA415r**
KV $\boldsymbol{KV_{CRM}}$ **(J)**	154.7	150.8

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 appearing in Eq. 1:

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

$\overline{X}_{SB}$ 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 5 and Table 6 summarise the input quantities of the u_{char} uncertainty budget, their respective statistical properties, and show how they were combined. The effective number of degrees of freedom for u_{char} is obtained using the Welch-Satterthwaite equation [8].

Table 5: Uncertainty budget for u_{char} of ERM®-FA415p

symbol	measured value (J)	source of uncertainty	uncertainty value (J)	probability distribution	divisor	sensitivity coefficient	relative standard uncertainty (%)	degrees of freedom
KV_{MB}	152.4	certification of MB	1.1	Normal	1	1	0.72	8
$\overline{X}_{SB}$	160.23	comparison of SB and MB under repeatability conditions	1.19	Normal	1	1	0.74	19
$\overline{X}_{MB}$	157.82		0.81	normal	1	1	0.52	29
						u_{char} (%)	1.16	34
						u_{char} (J)	1.79	

[1] Divisor: number used to calculate standard uncertainty from non-standard-uncertainty expression of uncertainty (e.g.: coverage factor to adapt expanded uncertainty to standard uncertainty, or factor to transform bounds of rectangular distribution into standard uncertainty of equivalent normal distribution).
[2] Sensitivity coefficient: partial derivative $\partial f / \partial x_i$ describing how an output estimate (here KV_{CRM}) varies with changes in the values of input estimates (here KV_{MB} , $\overline{X}_{SB}$ and $\overline{X}_{MB}$). If the functional relationship is a product or quotient, as in Eq. 1, it is advantageous to combine relative instead of absolute standard uncertainties, as in this case the sensitivity coefficients are equal to 1 [15]; this has been done here.

Table 6: Uncertainty budget for u_{char} of ERM®-FA415r

symbol	measured value (J)	source of uncertainty	uncertainty value (J)	probability distribution	divisor	sensitivity coefficient	relative standard uncertainty (%)	degrees of freedom
KV_{MB}	152.4	certification of MB	1.1	normal	1	1	0.72	8
$\overline{X}_{SB}$	156.17	comparison of SB and MB in repeatability conditions	1.42	normal	1	1	0.91	19
$\overline{X}_{MB}$	157.82		0.81	normal	1	1	0.52	29
						u_{char} (%)	1.27	35
						u_{char} (J)	1.92	

[1] Divisor: number used to calculate standard uncertainty from non-standard-uncertainty expression of uncertainty (e.g.: coverage factor to adapt expanded uncertainty to standard uncertainty, or factor to transform bounds of rectangular distribution into standard uncertainty of equivalent normal distribution).
[2] Sensitivity coefficient: partial derivative $\partial f / \partial x_i$ describing how an output estimate (here KV_{CRM}) varies with changes in the values of input estimates (here KV_{MB}, $\overline{X}_{SB}$ and $\overline{X}_{MB}$). If the functional relationship is a product or quotient, as in Eq. 1, it is advantageous to combine relative instead of absolute standard uncertainties, as in this case the sensitivity coefficients are equal to 1 [15]; this has been done here.

6 Homogeneity

The five test pieces constituting a CRM unit are taken at random from the SB, which is sufficiently homogeneous (s_{SB} < 5 %, as required in EN 10045-2 [1] and ISO 148-3 [11]), but not perfectly homogeneous. Therefore, as for most reference materials, a separate homogeneity contribution u_h to the uncertainty of the certified value is required.

Here, u_h is estimated from s_{SB}, the standard deviation of the results shown in Table 2. As is required for a homogeneity test, the samples were randomly selected from the whole batch. The number of samples tested (20) is largely sufficient to reflect the homogeneity of the full SBs (nominal batch sizes 450 samples).

The effect of s_{SB} on the uncertainty of the certified value depends on the number of samples over which the KV-value is averaged. EN 10045-2 [1] and in ISO 148-2 [2] specify that pendulum 'indirect verification' with CRMs must be performed using 5 test pieces. Therefore, a CRM-unit consists of 5 test pieces, and $u_h = \frac{s_{SB}}{\sqrt{5}}$. 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. The u_h values are reported for each batch in Table 8 and Table 9.

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 [16]. 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 during the International Master Batch (IMB) project [17]. 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 0.05 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 the batches ERM®-FA415p and ERM®-FA415r were characterised in February 2009, the validity of the respective certificates stretches until February 2019. It can be noted that tests were performed in July 2001 on the ERM®-FA415p and ERM®-FA415r secondary batches and on the ERM®-FA415b master batch at Cogne (Aosta, IT). The results obtained then were used to prepare a BCR-certificate. The resulting certified values agree well with the certified values proposed in this report, within their uncertainty.

Table 7: Comparison of values based on test results obtained in 2001 and in 2009

	Results July 2001	**Results February 2009**	
	BCR-certified value (J)	*ERM-certified value* (J)	*Expanded Uncertainty* (J)
ERM®-FA415p	152.1	155	6
ERM®-FA415r	150.9	151	7

8 Evaluation of results

8.1 Calculation of certified value, combined and expanded uncertainty

As shown in 5.3, KV_{CRM} = 154.7 J for ERM®-FA415p and 150.8 J for ERM®-FA415r. 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 budgets (Table 8 and Table 9).

The relevant number of degrees of freedom calculated using the Welch-Satterthwaite equation [8], is sufficiently large (ν_{RM} = 39 for ERM®-FA415p and ν_{RM} = 36 for ERM®-FA415r) 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 smaller than the verification criterion of 10 % (for industrial pendulums [1, 2]) or even 5 % (for reference pendulums [1, 11]).

Table 8: Uncertainty budget of KV_{CRM} for ERM®-FA415p

symbol	source of uncertainty	absolute value (J)	Divisor	sensitivity coefficient	u_i (J)	degrees of freedom
u_{char}	characterisation of SB	1.79	1	1	1.79	34
u_h	homogeneity of SB	2.38	1	1	2.38	19
				Combined standard uncertainty, u_{CRM}	2.98 J	39
				Expanded Uncertainty, k = 2, U_{CRM}	6 J	ν_{RM}

[1] Divisor: number used to calculate standard uncertainty from non-standard-uncertainty expression of uncertainty (see Table 5).
[2] Sensitivity coefficient: partial derivative $\partial f / \partial x_i$ describing how the output estimate (here u_{CRM}) varies with changes in the values of the input estimates (here u_{char} and u_h), evaluated at their actual values. Since the functional relationship between certified value and the characterisation, stability and homogeneity terms, is an addition, the sensitivity coefficients are equal to 1.

Table 9: Uncertainty budget of KV_{CRM} for ERM®-FA415r

symbol	source of uncertainty	absolute value (J)	Divisor	sensitivity coefficient	u_i (J)	degrees of freedom
u_{char}	characterisation of SB	1.92	1	1	1.92	35
u_h	homogeneity of SB	2.84	1	1	2.84	19
				Combined standard uncertainty, u_{CRM}	3.43 J	36
				Expanded Uncertainty, k = 2, U_{CRM}	7 J	ν_{RM}

[1] Divisor: number used to calculate standard uncertainty from non-standard-uncertainty expression of uncertainty (see Table 5).
[2] Sensitivity coefficient: partial derivative $\partial f / \partial x_i$ describing how the output estimate (here u_{CRM}) varies with changes in the values of the input estimates (here u_{char} and u_h), evaluated at their actual values. Since the functional relationship between certified value and the characterisation, stability and homogeneity terms, is an addition, the sensitivity coefficients are equal to 1.

8.2 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®-FA415b 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 [18].

The certified values of ERM®-FA415p and ERM®-FA415r are 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 *values* of ERM®-FA415p and ERM®-FA415r are traceable to the International System of Units (SI) via the corresponding Master Batch ERM®-FA415b of the same nominal absorbed energy (150 J).

8.3 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. The certified values are not to be used when the test pieces are broken with an ASTM-type striker of 8 mm tip radius.

8.4 Summary of results

The certified values and associated uncertainties are summarised in Table 10.

Table 10: Certified values and associated uncertainties for ERM®-FA415p and ERM®-FA415r.

	Certified mean value for set of 5 test pieces KV_{CRM} **(J)**	**Combined standard uncertainty** u_{CRM} **(J)**	**Expanded uncertainty (*k* = 2)** U_{CRM} **(J)**
ERM®-FA415p	155	2.98	6
ERM®-FA415r	151	3.43	7

9 Instructions for use

9.1 Intended use

Samples of ERM®-FA415p and ERM®-FA415r correspond with the '(certified) BCR test pieces' as referred to in EN 10045-2 [1], as well as with 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. This bias assessment can be used in the calculation of the measurement uncertainty of Charpy tests on the pendulum after indirect verification. 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 1 of the certificate.

9.2 Sample preparation

Special attention is drawn to 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 into the test laboratory at least 3 h before the tests.

9.3 Pendulum impact tests

After cleaning, the 5 samples which together constitute one CRM unit, need to be broken with a pendulum impact testing 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 comparison of the indirect verification results with the certified value and uncertainty must be based on the mean of the 5 measured *KV* values, because the calculation of the uncertainty of the certified value is based on this sample size.

10 Acknowledgements

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Annex 1

Results of characterisation measurements of ERM®-FA415p and ERM®-FA415r measured according to EN 10045-1 [3] and ISO 148-1 [4] (IRMM, Geel, 24-25/2/2009).

	Master Batch	**Secondary Batches**	
	ERM®-FA415b	**ERM®-FA415p**	**ERM®-FA415r**
	***KV* (J)**	***KV* (J)**	***KV* (J)**
1	153.70	159.96	161.40
2	157.43	163.49	164.66
3	163.10	153.50	152.92
4	164.66	166.02	149.97
5	158.21	161.54	155.07
6	152.98	162.90	161.34
7	157.62	163.95	154.49
8	154.49	167.45	151.93
9	154.88	151.41	161.14
10	153.70	164.92	162.57
11	158.66	158.47	151.22
12	149.97	150.43	162.05
13	161.20	165.51	141.20
14	158.21	154.94	147.49
15	150.63	161.20	155.93
16	159.25	165.12	148.72
17	152.98	150.82	161.21
18	162.78	161.99	158.47
19	162.19	159.65	163.75
20	156.64	161.41	157.89
21	155.73		
22	162.97		
23	165.12		
24	153.77		
25	154.36		
26	154.54		
27	162.97		
28	162.58		
29	164.53		
30	154.75		
Mean (J)	**157.82**	**160.23**	**156.17**
Standard deviation (J)	**4.45**	**5.32**	**6.36**
***RSD* (%)**	**2.8**	**3.3**	**4.1**

Annex 2

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CERTIFICATE OF ANALYSIS

ERM®- FA415b

Steel Charpy V-notch test pieces (nominal absorbed energy[1)] 150 J, Master Batch)		
Parameter	Certified value [2)] (J)	Uncertainty [3)] (J)
Absorbed energy (KV) at 20 ± 2 °C, according to EN 10045-1 and ISO 148	152.4	1.1

1) The term absorbed energy is defined in EN 10045-1 and ISO 148 and refers to the impact energy required to break a V-notched bar of standardised dimensions.

2) Mean absorbed energy of test pieces from batch ERM®-FA415b. The certified value was obtained as the mean of means of absorbed energies measured at 9 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. The certified value is valid only for impact hammers with a 2 mm striker tip radius.

3) Half-width of the 68 % confidence interval of the mean absorbed energy defined in 2), estimated as $\frac{\sigma_m}{\sqrt{9}}$, with σ_m the standard deviation of the mean of the mean values obtained at the 9 participating laboratories.

This certificate is valid until October 2009; this validity may be extended as further evidence of stability becomes available.

NOTE

European Reference Material ERM®-FA415b was originally certified as BCR-415 B. 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, July 2005

Signed: ____________________

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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 dimensions 55 mm x 10 mm x 10 mm, with one V-notch, accurately machined to tolerances 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

The ERM-FA415b batch is intended to be used as a 'Master Batch'. Master Batch test pieces are not for sale. They are used to traceably certify Secondary Batches of Charpy V-notch reference test pieces of the same type of steel with the same nominal absorbed energy (here 150 J).

To characterise a secondary batch, a selection of samples from the secondary batch have to be broken under repeatability conditions together with Master Batch test pieces. The certified value of the Master Batch and its associated uncertainty are used in the calculation of the certified value and its combined and expanded uncertainty 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.

Special attention is drawn to cleaning and conditioning 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 5 min.

3. Wipe the specimens with cellulose paper and let dry.

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 12 h before the tests.

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 or indentation of the specimen edges should be avoided, as this can result in obtaining erroneous data.

The cleaned samples need to be broken with an impact pendulum in accordance with EN 10045-1 or ISO 148 standards.

Unlike Charpy test pieces of lower nominal impact energies, samples from ERM-FA415 batches sometimes leave debris on the Charpy pendulum anvils. Therefore, after each impact, the anvils must be checked and if debris is found, it must be removed.

After testing, the user is recommended to inspect the traces/imprints left behind by the anvils and hammer on the two halves of the broken specimen. Asymmetry of these marks can indicate problems with the machine geometry or the positioning of the sample prior to impact. If so desired, broken samples can be stored for later inspection of the anvil and striker marks.

STORAGE

Specimens should be kept at ambient temperature in their original packing until used. The European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

SAFETY INFORMATION

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

METHOD USED FOR CERTIFICATION

Charpy pendulum impact tests in accordance with EN 10045-1 and ISO 148, using pendulum impact machines with a 2 mm striker tip radius.

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- National Physical Laboratory (NPL), Teddington (UK)
- Rolls Royce Ltd., Derby (UK)
- Studiecentrum voor Kernenergie (SCK•CEN), Mol (BE)
- Universidad Carlos III, Madrid (ES)

LEGAL NOTICE

NOTE

A detailed technical report of the Master Batch certification project (“The certification of two new Master Batches of V-notch Charpy impact toughness specimens in accordance with EN 10045-2: 1992”, R. K. Varma, bcr information, EUR 18947 EN, 1999) can be obtained from IRMM on explicit request. In this report, the ERM-FA415b batch is called ‘CRM 415 B’.

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Abstract

This certification report describes the processing and characterisation of ERM®-FA415p and ERM®-FA415r, two batches 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 testing machines [2]). The certified value for *KV* (= energy required to break a V-notched test piece using a pendulum impact test machine) and the associated uncertainty (*k* = 2 corresponding to a confidence level of about 95 %) calculated for the mean of a set of five test pieces, are:

Batch-code	Certified KV-value	Expanded uncertainty (k = 2, confidence level of about 95 %)
ERM®-FA415p	155 J	6 J
ERM®-FA415r	151 J	7 J

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 traceable to the International System of Units (SI) through the corresponding Master Batch ERM®-FA415b of the same nominal absorbed energy (150 J).

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