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Institute for Reference Materials and Measurements

The certification of critical coating failure loads: a reference material for scratch testing according to ENV 1071-3: 1994 BCR-692

Report EUR 20986

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REFERENCE MATERIALS

The certification of critical coating failure loads: a reference material for scratch testing according to ENV 1071-3: 1994

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SUMMARY

Scratch testing procedures for the testing of adhesive and cohesive strength of hard coatings, described in the European standard prEN 1071-3, require the measurement of “critical load” values associated with characteristic failure events occurring when a stylus is drawn across the coating with a normal load applied. The CRM BCR-692 has been produced and certified for verification and monitoring of the proper functioning of scratch test instruments. The reference samples are (30x30x5) mm steel coupons with a diamond-like carbon coating (DLC) applied by plasma-assisted chemical vapour deposition. Certification of critical load values was carried out by nine laboratories using three different types of scratch testing instruments and specially prepared Rockwell C diamond styli. A study of heterogeneity and stability was made and the sensitivity of critical load to stylus tip radius was also investigated. Certified values were assigned for three critical loads Lc_1, Lc_2, Lc_3 (corresponding to three particular failure events) with uncertainties arising from the evaluation of the material properties. In addition, machine “verification ranges” (wider than the certified ranges), to be used for verification of scratch test instruments were assigned, including additional components of uncertainty related to stylus variability and machine type. The verification ranges can be used to verify any scratch test instrument compliant with, and calibrated according to, prEN 1071-3. The certified and verification ranges are:

Failure event[1)]	Certified range[2)] (N)	Verification range[2)] (N)
Forward chevron cracks at the borders of the scratch track (Lc_1 shall be taken at the closest end of the event to the scratch track start).	Lc_1 (13.6 ± 1.9)	Lc_1 (13.6 ± 3.1)
Forward chevron cracks at the borders of the scratch track, with local interfacial spallation or with gross interfacial spallation (Lc_2 shall be taken at the failure event that occurs first, and at the closest end of the event to the scratch track start).	Lc_2 (17.0 ± 2.0)	Lc_2 (17.0 ± 5.4)
Gross interfacial shell-shaped spallation (Lc_3 shall be taken at the first point where the substrate can be seen at the centre of the track in a crescent that goes completely through the track).	Lc_3 (27.9 ± 3.6)	Lc_3 (27.9 ± 5.7)

[1)] Micrographs of the critical failure events are shown in Annex A of this certification report.

[2)] The uncertainty is the expanded uncertainty estimated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM) with a coverage factor k = 2.

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GLOSSARY

DLC	diamond-like carbon coating
OMS	optical measurement system
PACVD	plasma-assisted chemical vapour deposition
SEM	scanning electron microscopy
GD-OES	glow discharge optical emission spectroscopy
XPS	X-ray emission photoelectron spectroscopy
KE	knife-edge profilometry
u_{bb}	“between-batch” uncertainty component
u_{bs}	“between-samples” uncertainty component
u_{ws}	“within-samples” uncertainty component
u_{ss}	“between-styli” uncertainty component
u_{I}	“between-instruments” uncertainty component
p	no. of accepted sets of results
s_c	standard deviation of means of p sets of results
Lc	critical load
L_i, R_j, S_k	laboratory/instrument, sample and stylus off-sets in the regression model of data treatment.
e_{ijk}	random uncertainty in the regression model.
μ	critical load reference values in the regression model.
$E(L_i)$, $E(R_j)$, $E(S_k)$, $E(e_{ijk})$	sums of off-sets in the regression model.
R	stylus tip radius
RSD	relative standard deviation
U_c	(certified) expanded uncertainty
VR	verification range
CV	certified value

1. Introduction

Surface engineering through the use of applied coatings has become a well-established technology for improving component performance, e.g. wear resistant coatings on cutting tools or optical coatings on glass. Coating adhesion can be tested using the quick and simple ‘scratch test’ in which a diamond stylus is drawn across a surface with the normal load increased step-wise (on different scratches) or continuously (on a single scratch) until well defined failures occur, usually through thickness cracking followed by spallation of the coating or other events at critical loads Lc_1, Lc_2… Lc_n. The failure events are detected by examination of the scratch track after scratching using an optical microscope. The scratch stylus is usually a Rockwell C diamond with (200 ± 10) µm tip radius, and the scratch test method is described in the (prospective) European Standard prEN 1071-3 [1].

The reliability of the scratch-test method was investigated as part of a previous European project (FASTE) [2], studying the influence of stylus properties, cleaning procedures, ambient conditions and transfer layers (from previous scratches) on critical load values. The main conclusion of that work was that variation in stylus tip shape, either due to damage or incorrect radius, was the main source of uncertainty in the test method. The REMAST project described here (REference MAterial for the Scratch Test, contract SMT4-CT98-2238) was therefore set up with the objective of developing and certifying a ‘real world’ reference material for the verification of the proper functioning of scratch test instruments by detecting deviations in stylus tip shape and errors in load or displacement calibrations or other instrument malfunctions. Secondary objectives were improvements in stylus manufacturing and characterisation methods, because FASTE had shown that many styli in use deviated significantly from those specified [3] for the scratch testing method. The REMAST project included the selection of suitable coating material, the preparation of special ‘qualified’ styli of tip radius close to 200 µm, and an investigation of uncertainties due to variations in scratch test instrument compliance. This certification report describes the certification of three critical loads corresponding to characteristic failure events in the DLC (diamond-like carbon coating) selected for the reference material, and the associated homogeneity and stability studies and describes the use of the CRM for instrument verification and monitoring. A guide [4] to be used as a companion to this report has been produced by the National Physical Laboratory, (UK).

2. List of participants

Vlaamse Instelling voor Technologisch Onderzoek (VITO), Mol (BE): coordination, certification, control measurements, stylus sensitivity measurements, evaluation
Teer Coatings Ltd. (TCL), Hartlebury (GB): sample preparation, certification
National Physical Laboratory (NPL), Teddington (GB): stylus characterisation, certification, evaluation
Laboratoire de Science et Génie des Surfaces (LSGS), Nancy (FR): stylus characterisation, certification
Mößner GmbH-Hamödia, Pforzheim (DE): stylus production, stylus characterisation
Instituto Pedro Nunes (IPN), Coimbra (PT): homogeneity, certification
VTT Manufacturing Technology (VTT), Espoo (FI): certification
Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin (DE): homogeneity, stability, certification
European Commission, Joint Research Centre, Institute for Reference Materials and Measurements (IRMM), Geel (BE): evaluation, CRM storage and distribution
Hull University (RSCE), Hull (GB): certification
Université de Poitiers (LMS), Futuroscope (FR): certification
CSEM, Neuchâtel (CH): certification

3. Feasibility study and selection of candidate material

3.1 Substrates and coating preparation

Two candidate coating types, DLC and TiN, were selected for the feasibility study. The TiN is a very widely used and established coating type and DLC coatings represent an important emerging technology. Three batches of 25 samples (three deposition runs) of each type of coating were carried out. The DLC coatings were produced by rf PACVD at VITO. The substrates were first cleaned with argon plasma in the deposition chamber, then a PACVD adhesion layer was deposited and finally the DLC was made using CH_4 and H_2. The TiN layers were made by a reactive sputtering process at TCL. The substrates used were coupons (30x30x5) mm, vacuum hardened, plane-parallel ground and polished from Böhler S790 ISO MATRIX powder metallurgy steel. The substrate surface roughness after polishing was R_a=0.01µm. This type of substrate was selected for its isotropic properties and fine carbide dispersion. Some difficulties were experienced however with void formation during surface preparation of the substrates. Void density was eventually reduced, to less than five voids of diameter 40 µm or larger per substrate, by improved polishing methods. Hardnesses of the TiN and DLC coatings were typically 27 and 21 GPa respectively.

3.2 Stylus production

Sixteen styli were specially ground for the feasibility study by Mößner and the radii were measured by a number of techniques: an optical measurement (shadow) system (OMS), SEM, knife-edge profilometry (KE) and two further optical methods based on interference. The supplier's data (OMS) indicated radii within the range 191-207 µm. Two styli of other radii (132 and 322 µm by KE) were also used. Significant improvements in stylus manufacturing, particularly in grinding, were made during the feasibility study.

3.3. Failure events and critical loads

The three failure events identified for DLC were:

- forward chevron cracks at the borders of the scratch track,
- forward chevron cracks at the borders of the scratch track, with local interfacial spallation or with gross interfacial spallation,
- gross interfacial shell-shaped spallation.

Typical critical failure loads measured by one lab were Lc_1=17 N, Lc_2=20 N, Lc_3=33 N. Failure events for DLC are shown in Annex A (with descriptions slightly revised after the feasibility study). Some instances of false critical load values for Lc_2 due to coating defects were observed (Annex A). Four failure events (not shown in this report) were identified for TiN coatings:

- longitudinal cracks at track edges (Lc_1=25 N),
- semicircular coating cracks inside the scratch tracks (Lc_2=42 N),
- adhesive chipping at the tracks edges (Lc_3=64 N),
- spallation at the track edges (Lc_4=65 N).

3.4 Homogeneity of test batches

One laboratory tested eight samples from each of 3 batches of DLC coatings and eight samples from each of three batches of TiN. A total of 90 scratches were made over each set of eight samples to investigate potential differences within samples and between samples originating from different locations in the reactor. A large amount of critical load data was generated (540 scratches per material, each with 3 or 4 critical load values) and this is summarised for each batch in Table 1.

Table 1: Homogeneity of test batches. Critical load values (Lc in N) of DLC and TiN with standard deviation s in N and RSD in %.

DLC												
Batch	Lc_1	s	RSD	Lc_2	s	RSD	Lc_3	s	RSD			
B1708	13.9	0.5	3.8	18.3	2.2	12.1	26.0	0.9	3.4			
B1709	14.2	0.6	4.2	17.7	0.9	4.8	26.2	1.1	4.1			
B1725	14.2	0.5	3.7	19.2	2.9	15.2	26.8	0.9	3.3			
TiN												
Batch	Lc_1	s	RSD	Lc_2	s	RSD	Lc_3	s	RSD	Lc_4	s	RSD
B035	30.3	1.4	4.7	44.4	4.4	10.0	54.6	6.4	11.7	58.5	6.7	11.4
B037	29.9	1.7	5.7	41.7	6.1	14.6	51.8	4.3	8.3	56.4	5.2	9.2
B038	28.4	1.5	5.4	36.8	5.9	15.9	43.1	5.2	12.1	45.7	7.7	16.8

The DLC samples showed lower scatter in critical loads, for Lc_1 and Lc_3 in particular, than the TiN, indicating better homogeneity or more easily detected failure events. There are no significant differences between critical loads for DLC batches, indicating that the deposition technique is reproducible. In the case of TiN, one of the batches, B038, showed lower values than the other two. No differences between samples originating from different locations within a batch were detected.

In a number of cases, outliers on the high side were observed and those outside the 99% confidence interval for each batch were eliminated. Such outliers are not entirely unexpected in scratch testing and may be due to the build up of debris in front of the indenter, or to poor definition of failure events. The number of eliminated data or missing data (no event detected) was small, but was significantly less for DLC than for TiN (26 compared with 140).

Chemical heterogeneity was studied by XPS, Auger and GD-OES. No significant heterogeneity was detected, either for DLC or TiN.

3.5 Stability of test batches

A series of tests was carried out to determine stability during cycled exposure to high temperature (40℃, 23 h x 10) and cycled exposure to low temperature (-50℃, 48h x 2) and after storage in laboratory air (6 months). No evidence of instability of critical loads was detected, although changes in the stylus during the course of testing reduced the reliability of these data.

3.6 Selection of candidate material

The feasibility study data summarised in this report and other REMAST data on stylus wear, reproducibility and uncertainty due to variations in instrument

compliance allowed a comparison of the two candidate coatings, DLC and TiN. The most important factor taken into account was the sensitivity of critical load to tip radius *R* relative to the standard deviation of the data ($\Delta Lc/(\Delta R.s)$). The DLC was much superior in this respect, for all critical loads. The DLC was also found to be more homogeneous, as described above, with fewer outlying or missing values and to cause less wear of the stylus than the TiN. The homogeneity study also indicated that batch to batch variability would be less with DLC than with TiN.

The load range for DLC (up to about 35 N) was half that of the TiN (up to 65 N) resulting in a smaller probe depth (4-6 μm), but this was considered sufficient for the evaluation of stylus shape, especially bearing in mind that ‘multi-mode’ scratch testing, now being developed [5] and likely to become the standard method in scratch testing, will use lower loads than those generally used in current practice.

On the basis of the above considerations DLC was selected as the coating to be used for the reference material.

4. Production of the reference material

Steel coupons (30x30x5) mm of Böhler S790 powder metallurgy steel were prepared and polished following the procedure developed during the feasibility study. The deposition of the DLC was carried out as for the test batches in eleven deposition runs, each of 100 specimens. The coupons were laser engraved (before polishing and coating) on one edge with the identification numbers ‘REMAST 0001’ to ‘REMAST 1099’. The first two digits indicated the batch number (0 to 10) and the second two digits indicated the sample number (0 to 99). The production of the CRM samples was carried out in August and September 2000.

Microscopic inspection of the CRM samples revealed some coating defects, usually voids, similar to those observed on the samples produced for the feasibility study, mainly close to the specimen borders rather than in the centre.

After production, the samples were stored in desiccator cabinets. At the time of sale, the samples are packed, into reusable plastic boxes containing desiccant.

5. Homogeneity of the reference material

The reference material was prepared in eleven separate deposition runs giving potential for differences between samples originating from different batches. In addition, the local plasma and deposition conditions within the chamber are possibly different from one area on the substrate table to another and there may even be differences between different areas on the same sample. An extensive homogeneity study was therefore carried out (finished September 2001) in which 6 samples from each batch were tested (5 scratches per sample) in laboratory 8. A parallel study was carried out by laboratory 5 testing 3 samples from each batch (10 scratches per sample) for a more detailed examination of the “within-sample” effect.

Two-way ANOVA was then carried out for each data set (33 samples and 66 samples), with an appropriate nested design and the insertion of a small number of dummy values (and corresponding reduction in the number of degrees of freedom) to estimate the three uncertainty components (as standard uncertainties): between-batches u_{bb}, between-samples u_{bs} and within-samples u_{ws} for each failure event (Table 2). The large volume of data from the homogeneity study (1880 values minus 14 missing values in total) is not reproduced in this report.

Table 2: Heterogeneity components (N) determined by lab 5 (33 samples) and lab 8 (66 samples). The values in bold are those carried forward to the calculation of the final combined uncertainty.

Heterogeneity component	Lc_1		Lc_2		Lc_3	
	Lab 5	Lab 8	Lab 5	Lab 8	Lab 5	Lab 8
between batches u_{bb}	0.59	**0.40**	0.66	**0.44**	1.39	**0.80**
between sample u_{bs}	0.34	**0.49**	0.44	**0.49**	0.84	**0.81**
within sample u_{ws}	**0.55**	0.53	**0.53**	0.49	**1.30**	0.64

Comparison of variances (F-test) produced by the ANOVA calculations showed that between-batch and between-sample differences were both statistically significant. Laboratory 5 made more scratches per sample, therefore sampling a larger surface of each sample tested than lab 8, and this may be the reason for the higher u_{ws} (within-sample) values. The u_{ws} value from lab 5, which is more representative of the entire sample area, is therefore carried forward to the calculation of the combined uncertainty. The real within-sample effect, cannot of course be separated from the natural scatter of the method (repeatability), which also makes a contribution to u_{ws}. The u_{bb} and u_{bs} values preferred for the final uncertainty calculation are those from lab 8, which tested more samples per batch. Note that the u_{ws} values given above were, as expected, similar to the standard deviations given in Table 1 for the test batches, except for Lc_2, where they were considerably lower.

6. Stability of the reference material

A stability study was carried out during 2001 on samples similar to that done during the feasibility phase. Again results were influenced by changes in stylus characteristics and conclusive data were not obtained. Diamond-like carbon coatings, however, are increasingly used in the engineering world for their wear resistance and stability and there is no indication from other sources that the DLC coatings used for the BCR-692 samples could suffer from instability that would affect critical loads in scratch testing, provided the samples are properly stored (see section 10). None of the reference samples showed any visible signs of deterioration after two years of storage in desiccator cabinets. Stability monitoring during the lifetime of the CRM will be performed.

7. Certification of the reference material

7.1 Production of styli

It was essential for the certification exercise to use styli conforming to the specifications for Rockwell C indenters in order to reduce as far as possible variability in measured critical load values due to stylus irregularities or damage. The Rockwell C indenter [3] is a conically shaped diamond with an included cone angle of 120° and a spherical tip of (200 ± 10) µm radius (mean of four measurements). Eighteen styli were prepared by Mößner, using the improved methods developed during the feasibility study. Natural dodecahedral diamonds were sintered into the holders with the <001> axis parallel to the indenter axis and <110> along the scratching direction. The forward direction of the indenter was identified by a mark on the holder. The crystal orientations were checked by Laue diffraction. The tip radii (over a 150 µm window) were determined by OMS by Mößner and by knife-edge profilometry (KE) (both calibrated using a ruby ball), by LSGS in the 0° direction (parallel to the scratch direction) and 45, 90 and 135°. Average radii were also determined by interferometry by NPL over a window width of 50 µm. NPL also carried out Twymann-Green interferometry to look at tip shape and found evidence that the stylus tips often showed "nodes" or local irregularities, with the consequence that stylus radius values depended on the "window" or tip area examined. Styli were allocated to individual laboratories for the certification exercise as indicated in Table 3, where the radius data are also shown.

7.2 Instrument calibration

The ten laboratories participating in the certification campaign used three different types of instrument (CSEM Revetest, VTT, TCL). Instructions (see Annex B) were issued for calibration, consistent with the requirements of prEN 1071-3. The calibration procedures included verification of sample planarity, load and load rate, horizontal displacement and displacement rate. For the load and displacement calibrations of all instruments, a calibrated load cell and a calibrated displacement transducer were made available and circulated amongst the participants. Calibration data were reported by all participants, together with the test results.

7.3 Certification measurements

Each of the ten laboratories was supplied (October 2000) with two samples selected randomly from the entire production and one of the qualified styli described in 7.1. After calibration, each laboratory made 40 scratches on each of the two samples with a load rate of 100 N/min, starting load 5 N, maximum load 45 N and displacement rate of 10 mm/min. Critical loads Lc_1, Lc_2, Lc_3 corresponding to the failure events in Annex A were reported (the definition of Lc_3 was slightly revised after the feasibility study to be the first shell-shaped crack that goes completely across the track). Each laboratory was also required to monitor load and displacement rate during the course of the testing, together with stylus wear to detect any drift in performance of the equipment. Instructions were also given to interrupt the testing if any cracks were detected in the stylus. Detailed instructions to participants are reproduced in Annex C.

The results of the certification campaign (completed in September 2001), corrected for each of the calibration factors, are presented in Annex D. The certification data are also displayed in Fig.1. The results of lab 10 were not accepted because of the lack of proper calibration data and the use of a scratch stylus, which was not in conformity with the Rockwell C standard.

Table 3: Styli used for the certification analyses. Radius measurements made in different orientations by three methods before scratch testing.

Stylus code	Measurement method	0°	45°	90°	135°	Mean radius (µm)	Destination
2238-33	OMS	197	206	198	200	200	lab 1
	KE	208	209	197	212	207	
	Interference					210	
2238-39	OMS	200	197	198	198	198	lab 2
	KE	201	209	200	205	204	
	Interference					210	
2238-43	OMS	200	201	200	200	200	lab 3
	KE	204	213	199	205	205	
	Interference					199	
2238-46	OMS	201	203	200	201	201	lab 4
	KE	199	199	193	206	199	
	Interference					195	
2238-50	OMS	201	193	204	200	200	lab 5
	KE	199	205	193	203	200	
	Interference					203	
2238-56	OMS	199	193	200	200	198	lab 6
	KE	199	205	193	208	201	
	Interference					202	
2238-57	OMS	199	201	199	195	199	lab 7
	KE	203	204	199	207	203	
	Interference					204	
2238-58	OMS	202	201	199	206	202	lab 8
	KE	193	203	186	189	193	
	Interference					198	
2238-36	OMS	203	198	198	193	198	lab 9
	KE	202	204	198	204	202	
2238-23	OMS	203	209	200	205	204	lab 10
	KE	208	219	204	215	212	
	Interference					208	

In addition to the measurements above, one control laboratory (lab 9) carried out a further 5 scratches on each of the samples supplied to the other participants using, in each case, the same stylus used by the respective participant and an additional 5 scratches using a single “control stylus”. The results are given in Annex E.

A specific stylus sensitivity test was also carried out by laboratory 9, taking eight styli with radii in the range (200 ± 10) µm, as required by ISO 6508-2 [3] and making nine scratches with each one on a single sample (no. 0445). The results of this sensitivity study are given in Annex F together with the stylus radii.

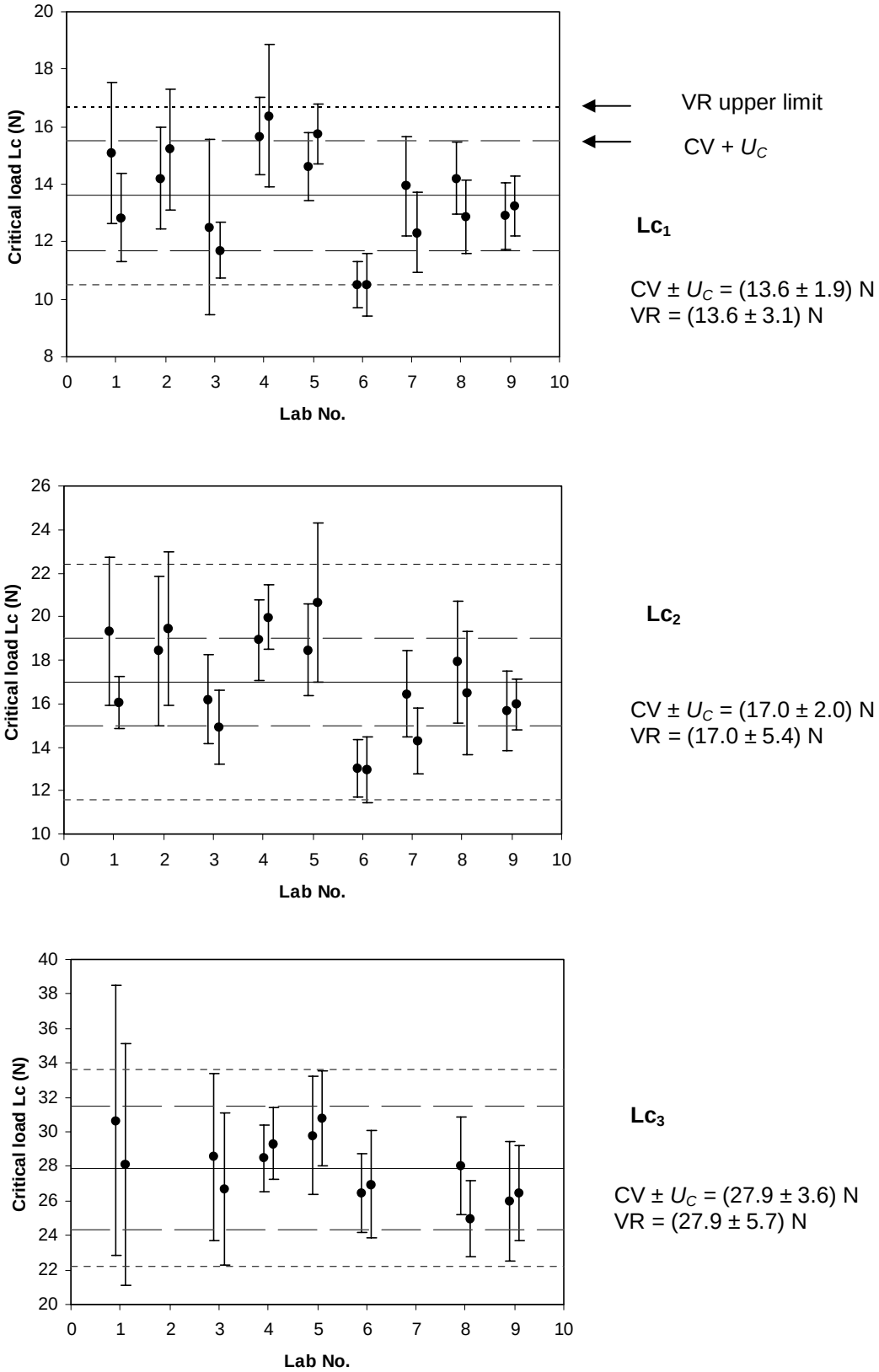


Fig. 1 Certified values (CV), expanded uncertainties (U_C) and verification ranges for the critical loads Lc_1, Lc_2 and Lc_3. Certification data are shown with ± 2s (type A) uncertainties.

8. Technical and statistical evaluation

For each critical load, the complete set of certification data and control data L_{ijk} can be modelled in terms of a reference value μ, laboratory/instrument, stylus and sample offsets (L_i, R_j and S_k, respectively) and random measurement error e_{ijk}:

$$Lc_{ijk} = \mu + L_i + R_j + S_k + e_{ijk} \qquad \text{- (1)}$$

with the assumption that the sums of the off-sets:

$E(L_i) = E(R_j) = E(S_k) = E(e_{ijk}) = 0, \quad \mathrm{var}(e_{ijk}) = \sigma^2,$

with σ^2 an (unknown) constant, and e_{ijk} independent. The uncertainty components to be combined to give the total uncertainty are given by estimates of the standard deviations of these offsets, and are evaluated from the data as described in this section.

It is apparent that there are significant differences between some of the sets of certification data, particularly for Lc_1 and Lc_2, and that samples giving low results for Lc_1 also gave low results for Lc_2. The control laboratory, however, using the same styli and the same specimens as the certifying laboratories produced a similar pattern of results (compare the data in Annex D with that in Annex E), showing that the bias noticed for each data set is largely explained by the combination of samples and styli assigned to the certifying labs.

As mentioned, the control testing generated data similar to that from each of the certifying laboratories except for the Lc_3 data for labs 2 and 7 where the differences in mean values from the control laboratory were substantial (5 N or more). The reason for these discrepancies was not definitely established, but it is probably significant that labs 2 and 7 were using the same type of instrument (and were the only ones with this instrument). In view of the fact that good agreement between certifying labs and the control lab was achieved in other cases, it was decided not to use the Lc_3 values from labs 2 and 7 for the certification and these are omitted from Fig. 1. The Lc_1 and Lc_2 values from labs 2 and 7 were retained because these relate to different failure modes in different load ranges.

Another feature of the certification data, not visible in Fig. 1, but which can be detected by examination of the tabulated data in Annex D, was tailing of the Lc_2 values on the high side, giving an asymmetric distribution of values. It seems that the Lc_2 track edge spallation is sometimes delayed to higher loads, maybe dependent on the spacing of the track edge cracks (Lc_1) which have to coalesce to produce the Lc_2 failures.

The styli used in the “stylus sensitivity” study were assumed to represent, in terms of radius, the population of all styli with radii (200 ± 10) μm that may be used for scratch testing. The actual radii of the styli measured after testing are given in Table F1. The only sources of variation in the results of this study are a) the different styli and b) scatter related to the precision of the testing or to small differences in the coating across the single specimen used. These two components can be separated by a one-way ANOVA of the data in Table F1, allowing the uncertainty component arising from stylus effects u_{ss} to be estimated (Table F2 or Table 4). Note that Lc_2 appears to

be more sensitive to stylus radius (higher u_{ss}) than the other critical loads, and that the “within-sample” values (u_{ws}) estimated in the stylus sensitivity study were higher than those measured in the homogeneity study, indicating that different styli may not only give different mean values of critical load, but also that some tip shapes may produce more scatter than others.

One remaining uncertainty component to be considered is that which arises from the use of different types of instruments in the certification campaign, which all met the requirements of prEN 1071-3 in terms of operation and calibration, but may produce different critical load values as a result of differences in mechanism or compliance. In order to evaluate this component (u_l), a regression analysis was carried out, based on the model given by equation (1) above, using all the certification data. Offsets from the regression fitting were then evaluated and the standard deviation of the laboratory offsets was taken as the estimate of u_l. Note that this uncertainty component includes contributions both from the variations in the instruments and variations in lab performance and these two cannot be separated. The values of u_l estimated in this way are given in Table 4.

Table 4 shows that the largest component of uncertainty for Lc_1 and Lc_2 is the stylus-related term u_{ss}, which confirms the high sensitivity of the test to stylus effects as observed in the FASTE programme [2]. It should be remembered, of course, that the DLC coating was chosen specifically for its high sensitivity to stylus radius. Instrument/laboratory effects (the u_l term) also play a major role.

9. Certified values and verification ranges

It was decided to assign, for each critical load:

- A **certified value** (CV) with an uncertainty containing only components arising from the material properties (mainly heterogeneity). The certified value for each critical load was the mean of means of the sets of data in Annex D (excluding labs 2 and 7 in the case of Lc_3), giving equal weight to each laboratory. The expanded uncertainty (U_c) was calculated according to the method given in ISO 35 [6]:

  $$U_c = \pm k.(s_c^2/p + u^2_{bb} + u^2_{bs} + u^2_{ws})^{1/2}$$

  where s_c is the standard deviation of the set means of the certification data, (Table 4), p is the number of accepted sets, and k is a coverage factor. The certified values with uncertainties and the uncertainty components are given in Table 4.

- A **verification range** (VR) to be utilised by users of the CRM for verification of their own scratch testing machines. This verification range (based on five scratches) is larger than the certified range and includes components arising from stylus differences and from the use of different machines:

  $$VR = \mathrm{CV} \pm k.(s_c^2/p + u^2_{bb} + u^2_{bs} + u^2_{ws}/5 + u^2_{ss} + u^2_{l})^{1/2}$$

  where the additional terms u_{ss} and u_l are defined as above and are given in Table 4.

A more detailed explanation of the above approach is that the certified ranges are the ranges within which approximately 95% of critical loads should fall when testing a specimen on an average machine with an average stylus of those used in the certification campaign. The decision to assign certified ranges excluding the stylus (u_{ss}) and machine (u_l) effects was based on the fact that these effects are test-related rather than material-related. They are also rather less well-defined than the material properties, and may well change in future due to improvements in machine performance and stylus production or to redefinition of the range of acceptable styli. The certified ranges, based on the materials' properties only, although not directly relevant to the user, will not change.

The user, however, would want to verify one particular type of (calibrated) test machine fitted with any stylus conforming to ISO 6508-2, possibly at the extreme edge of the allowable radii, and would be using one sample that could come from any one of the 11 batches. Thus a range including the stylus (u_{ss}) and machine (u_l) effects is necessary for the purposes of verification.

The uncertainties and verification ranges are shown in Fig. 1 together with the certified values.

Table 4: Certified values, expanded uncertainties (U_c) verification ranges (VR) and uncertainty components (all in N). A coverage factor k=2 has been applied.

Crit. load	u_{bb}	u_{bs}	u_{ws}	s_c	p	u_{ss}	u_l	**CV ± U_c**	**VR**
Lc_1	0.40	0.49	0.55	1.72	18	1.05	0.8	**13.6 ± 1.9**	**13.6 ± 3.1**
Lc_2	0.44	0.49	0.53	2.31	18	2.26	1.11	**17.0 ± 2.0**	**17.0 ± 5.4**
Lc_3	0.8	0.81	1.30	1.78	14	1.34	2.11	**27.9 ± 3.6**	**27.9 ± 5.7**

10. Instructions for use

The CRM can be used for two main purposes (as described in detail in ref. 4):

- for verification of (calibrated) scratch-test instruments, giving a good indication of overall instrument performance.
- for establishing control charts for monitoring instrument performance (including stylus) over an extended period.

The CRM should not be used to calibrate the scratch tester. The verification ranges are relatively wide, due to the combination of material and test-related uncertainties, and it is possible that the instrument and stylus may have significant, but counteracting defects, that keep the measured critical load values within the verification ranges. The use of the CRM for verification as described below is therefore not an absolute guarantee that the scratch-test instrument is operating in an optimum way.

10.1 Verification

1. Ensure that the instrument is calibrated according to prEN 1071-3.

2. Clean the stylus (before each scratch):

   a) Wipe with a soft tissue soaked in petroleum ether.
   b) If adhering debris are still observed under an optical microscope (recommended magnification 200x), mechanically clean with #1200 and #2400 SiC paper, and wipe with a soft tissue soaked in petroleum ether.
   c) Allow at least 1 minute equilibration time before testing.
   d) Check using the optical microscope that the stylus is not chipped or cracked.

3. Clean the BCR-692 reference sample (before each series of scratches, or at least every day):

   a) Ultrasonic bath during 5 minutes in pro analysis petroleum ether.
   b) Allow at least 3 minutes equilibration time before testing.
   c) If drying stains are observed, wipe with a soft tissue soaked in petroleum ether until the surface is clean and free of loose particles.
   d) Allow at least 3 minutes equilibration time before testing.

4. Carry out five parallel scratches on the sample with a load rate of 100 N/min, horizontal displacement rate of 10 mm/min, starting load of 5 N and maximum load of 45 N. The scratches should be not less than 1 mm apart, parallel to one of the sides of the reference sample, but not within 3 mm of the edge. The stylus should be cleaned between scratches as described above.

5. Examine the scratch tracks with an optical microscope (recommended magnification 200x) and determine the critical loads Lc_1, Lc_2 and Lc_3 corresponding to the failure events shown in Annex A of this report. The critical

load values (in N) are calculated from the measured distance between the scratch track start and the failure event, x_m, using the following equation:

$$Lc = 10x_m + 5$$

(where 10 N/mm is the nominal loading rate and 5 N is the initial load). Careful attention must be paid to the exact definition of each failure event. In the unlikely event of a scratch track crossing a coating defect (see Annex A), the scratch should be repeated.

6. If the average of the five Lc values for each critical fall within the relevant verification range range, then this gives a high degree of confidence that the instrument is functioning well. If this is not the case, then the user should more carefully check the instrument components, particularly the stylus, and the calibration.

7. The user should also ensure that the repeatability of the results (the standard deviation of the five values for each critical load) are similar (after correction for different samples sizes – see ref. 6) to the u_{ws} values measured during the BCR-692 certification (Table 4) representative of an instrument of good performance.

10.2 Monitoring

The reference sample can provide a sensitive monitor of instrument performance over time (control chart). To do this, the user should carry out the steps given in 10.1 above and monitor the evolution of mean critical loads measured and the standard deviations (see ref. 6). This procedure obviously only has value provided the same reference sample and stylus are used for the duration of the monitoring. The frequency of the monitor measurements depends on the other uses of the instrument being tested, bearing in mind that the expected stylus lifetime on the BCR-692 DLC coatings is about 200 scratches, and is likely to be less on higher friction coatings and with higher loads.

10.3 Specimen care

The samples should be stored in the box supplied with desiccant or in a laboratory desiccator and only removed for the duration of the tests. Care must be taken to maintain the cleanliness of the test samples; in particular, finger marks must be avoided, as these are corrosive.

References

[1] European Standard prEN1071-3:1999, Advanced technical ceramics - methods of test for ceramic coatings - Part 3: Determination of adhesion and other mechanical failure modes by a scratch test.

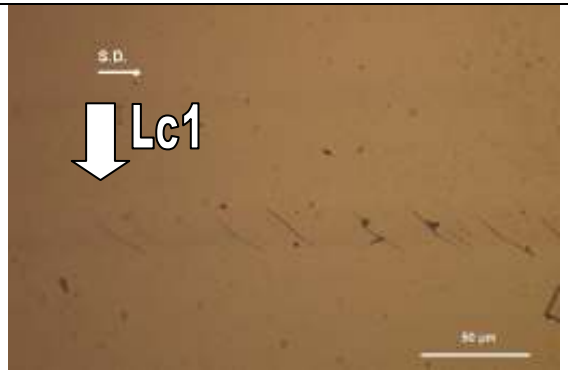
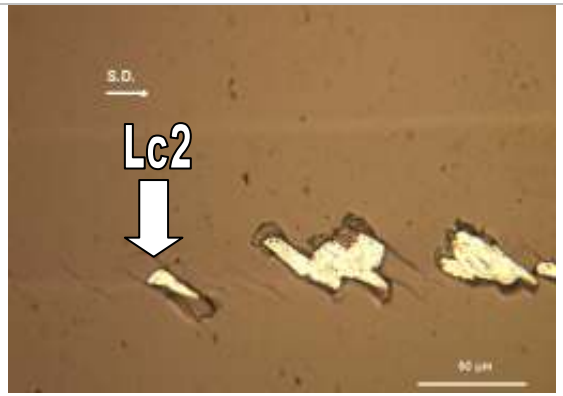
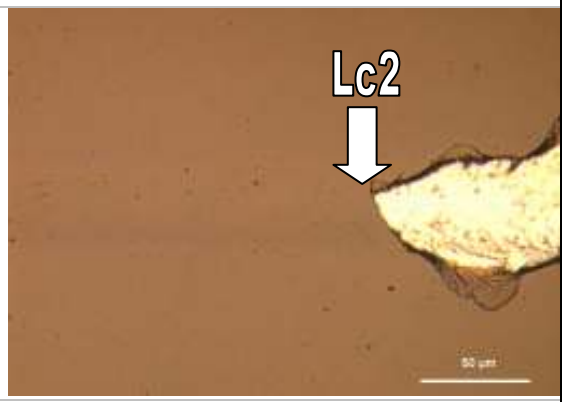
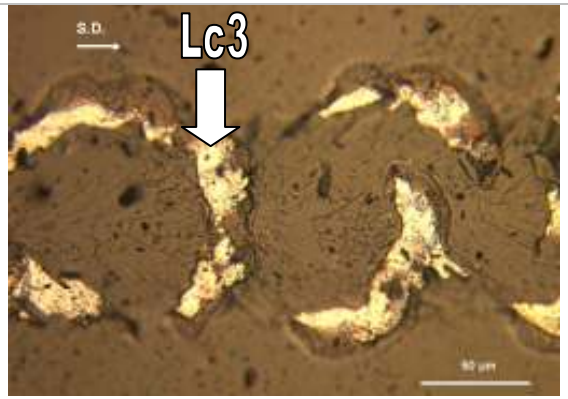
[2] Scratch adhesion testing of coated surfaces - Challenges and new directions, J. Meneve et al., Adhesion Measurement of Films and Coatings, Vol.2, pp. 79-106, Ed. K.L. Mittal, 2001.

[3] Standard EN ISO 6508-2:1999, Metallic materials – Rockwell hardness test – Part 2: Verification and calibration of testing instruments (scales A, B, C, D, E, F, G, H, K, N, T).

[4] The Scratch Test: Calibration, Verification and the Use of a Certified Reference Material, N.M. Jennett and S. Owen-Jones, Measurement Good Practice Guide No 54, National Physical Laboratory, Teddington, United Kingdom, December 2002.

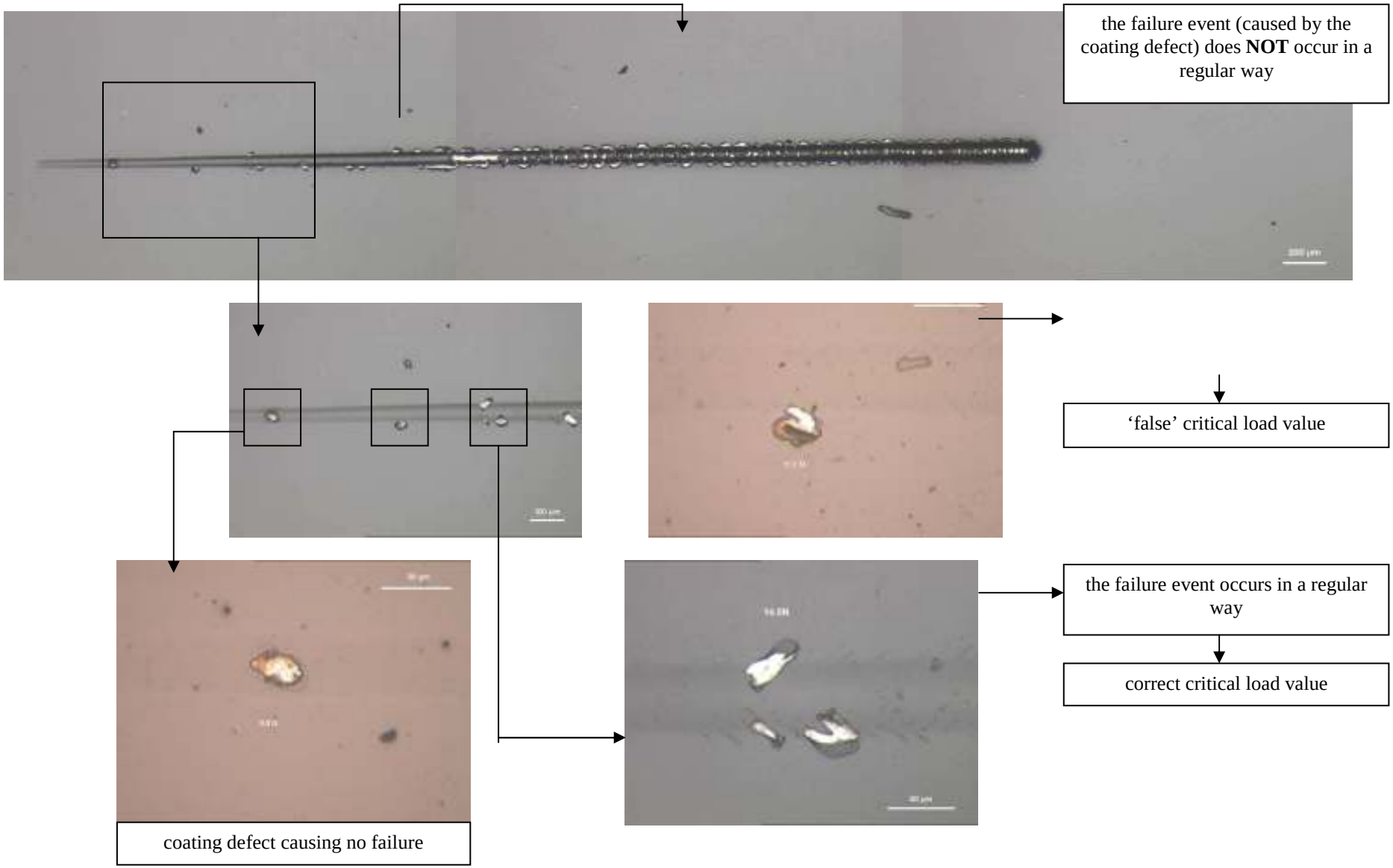
[5] European Commission - Standards, Measurements and Testing Programme, Project ‘Multimode Scratch Testing (MMST): Extension of Operation Modes and Update of Instrumentation’, contract SMT4-CT97/2150, completed 2001.

[6] ISO document REMCO/WG 1 N 41 (Draft Guide 35, Certification of Reference Materials - General and statistical principles), ISO, Geneva, Switzerland, February 2002.

	Description of failure events	**Micrograph** (S.D. = scratch direction)
LC_1	Forward chevron cracks at the borders of the scratch track. ***Lc_1 shall be taken at the closest end of the event to the scratch track start.***	
Lc_2	*Forward chevron cracks at the borders of the scratch track,* with *local* interfacial spallation or with *gross* interfacial spallation ***Lc_2 shall be taken at the failure event that occurs first, and at the closest end of the event to the scratch track start.***	 
Lc_3	Gross interfacial shell-shaped spallation ***Lc_3 shall be taken at the first point where the substrate can be seen at the <u>centre</u> of the track in a crescent that goes completely through the track.***	

(1) The critical load value corresponds to the first incidence of <u>REGULAR OCCURRENCE</u> of the associated failure event

Illustration of coating defect initiating a ‘false’ critical load value for Lc_2



ANNEX B

Instrument calibration for BCR-692 certification measurements

Each scratch test instrument shall be calibrated according to the calibration guidelines established in the European Standard, prEN 1071-3, "Advanced technical ceramics - Methods of test for ceramic coatings - Part 3: Determination of adhesion and other mechanical failure modes by a scratch test". In order to enable the participants to perform the calibrations, a calibrated displacement transducer, a calibrated load cell (accompanied by a hardened steel plate M2-521, to prevent damage to the load cell), and additional parts for mounting (these parts can be modified (e.g. drilling holes) if necessary for mounting) will be circulated between participants

The mandatory calibration procedure will consist of calibration of sample planarity, applied load and load rate, horizontal displacement and displacement rate. The calibration procedure to be used in the certification exercise is described briefly here. A more comprehensive presentation can be found in European Standard, prEN 1071-3.

The results of the calibrations shall be reported using the certification exercise proforma (distributed to participants).

<u>Procedure to assess the sample planarity</u>
The DLC coated sample with the lowest specimen number shall be used to assess the planarity of the sample stage. The flatness parameter, F_l, corresponds to the greatest deviation from the recorded normal load, expressed in N. Instructions can be found in European Standard, prEN 1071-3.

<u>Calibration of applied load and load rate</u>
Normal load shall be calibrated using the travelling load cell. For adjusting the load rate to (100 $\pm$ 1) N/min, the DLC coated sample with the lowest series number clamped in the specimen holder shall be used. The time base used (e.g. recorder plot length) must be checked by means of a chronometer. Where possible, the sample stage motor movement should be blocked to prevent damage to the specimen. Instructions can be found in European Standard, prEN 1071-3.

<u>Calibration of the horizontal displacement and displacement rate</u>
The in-built displacement transducer, which measures the movement of the sample, is calibrated by comparing its output with the output from a displacement transducer, which is calibrated and traceable to national standards. Instructions can be found in European Standard, prEN 1071-3.

ANNEX C

Procedure for certification measurements

Cleaning procedure
The samples should be stored in the box supplied with desiccant or in a laboratory desiccator and only removed for the duration of the tests. Care must be taken to maintain the cleanliness of the test samples, in particular, finger marks must be avoided as these are generally very corrosive.

Specimen: (before each series of scratches, or at least every day):
a) Ultrasonic bath during 5 minutes in pro analysis petroleum ether.
b) Allow at least 3 minutes equilibration time before testing.
c) If drying stains are observed, wipe with a soft tissue soaked in petroleum ether.
d) Allow at least 3 minutes equilibration time before testing.

Stylus: (before each scratch):
e) Wipe with a soft tissue soaked in petroleum ether.
f) If adhering debris are still observed under an optical microscope (recommended magnification 200x), mechanically clean with #1200 and #2400 SiC paper, and wipe with a soft tissue soaked in petroleum ether.
g) Allow at least 1 minute equilibration time before testing.
h) Check using the optical microscope that the stylus is not chipped or cracked.
.

Stylus wear
Each participant should take a photograph (recommended magnification: 200x) of the indenter before carrying out the scratch testing. Subsequent photographs shall be taken after every fifth scratch and after the scratch testing has been finished. This is necessary in order to document the condition of the diamond styli during the certification exercise.

Load rate
A continuous loading cycle, up to maximum load of 100 N should also be performed and plotted against a time base, every tenth scratch. This will verify the loading rate, which is equal to the slope of the load-time graph. The loading rate should be (100 ± 1) N/min.

Horizontal displacement rate
The horizontal displacement rate should be 10 mm/min and, in order to verify this, the length of every scratch will be verified with a travelling microscope, and shall be reported. The length should be 4 mm for a scratch made with a minimum load of 5 N and a maximum load of 45 N.

Scratch test procedure
Forty scratches shall be made on each reference material. The distance between the scratches shall be at least 1 mm. A mark is applied permanently to the stylus holder showing the scratching direction ('upstream', see Figure C1). Each scratch must be made parallel with the mark, ‘upstream’, and with the other scratches.

A start load of 5 N should be used to allow a better determination of the start of the scratch track. Caution has to be taken not to start the scratches too close on the edge and therefore at least 3 mm from the specimen edges should be left free of scratches. If for any reason some scratches should fail, there should be sufficient space available to redo those tests. All scratches made, including the erroneous ones, must be indicated schematically.

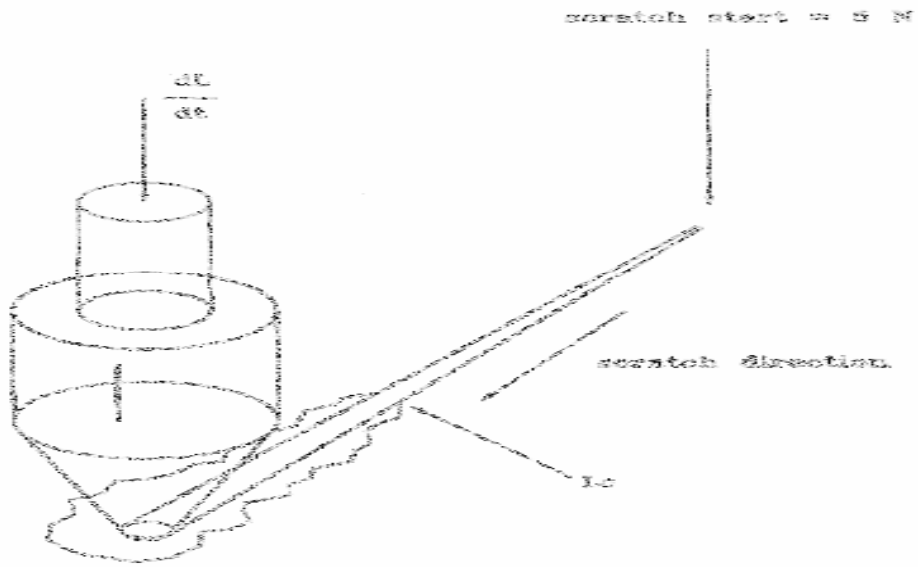


Figure C1. Scratch test set-up for the certification exercise: Start from 5 N, scratch parallel with the mark on the indenter holder and with the mark upstream.

For each scratch, the critical load values Lc_1 to Lc_3 corresponding to the clearly distinguishable failure events shall be determined. Micrographs of the failure events are shown in Annex A. The critical load values shall be determined by using an optical microscope with a recommended magnification of 200x. The critical load values (in N) are calculated from the measured distance between the scratch track start and the failure event, x_m, using the following equation:

$$Lc = 10x_m + 5$$

(where 10 N/mm is the nominal loading rate and 5 N is the initial load). The total length of the scratch shall also be measured and reported.

ANNEX D Table D1:Certification data for critical load Lc_1 (N). Sample numbers are given in the second row**.**

	Lab 1		Lab 2		Lab 3		Lab 4		Lab 5		Lab 6		Lab 7		Lab 8		Lab 9	
Sample	**0077**	**0711**	**0436**	**1064**	**0235**	**0635**	**0350**	**0986**	**0104**	**0134**	**0613**	**0588**	**1029**	**0560**	**0383**	**0649**	**0512**	**0851**
	14.4	12.3	13.2	16.9	11.2	11.8	13.4	11.1	13.9	15.6	10.5	11.8	14.8	13.1	14.4	11.3	12.9	12.1
	15.0	13.1	12.9	16.8	11.6	11.9	15.2	17.1	13.9	15.8	10.9	11.1	14.3	13.6	13.9	12.8	12.4	13.4
	14.9	13.8	14.2	16.6	11.9	12.0	15.4	17.0	14.4	15.2	10.2	10.9	14.8	13.0	14.5	13.2	12.7	13.4
	14.6	13.3	13.9	15.6	12.6	11.5	15.6	16.7	14.6	16.4	9.9	10.9	14.0	12.8	13.6	11.5	12.3	13.5
	15.0	14.4	15.0	15.3	12.1	12.0	15.1	16.1	14.6	14.8	10.3	10.6	14.9	11.9	14.4	11.0	12.4	13.4
	15.0	13.3	13.6	15.9	12.5	10.9	15.3	15.2	14.6	16.2	10.7	10.2	14.9	12.8	13.9	12.2	12.5	12.8
	14.5	14.5	13.9	13.7	12.2	11.8	16.1	16.2	14.0	16.9	10.5	10.0	14.3	14.3	14.3	12.3	13.2	13.5
	15.3	13.1	14.0	15.4	12.6	11.5	17.2	15.8	14.3	16.0	9.9	12.0	13.6	13.3	14.8	12.9	13.0	12.6
	14.9	12.4	13.7	15.0	12.8	11.4	14.6	14.4	14.0	16.0	11.9	10.5	12.7	12.4	13.5	13.5	12.5	13.5
	14.9	14.5	13.9	14.4	12.3	11.5	16.1	14.5	15.0	16.3	10.7	10.3	13.6	12.8	15.7	13.7	14.1	13.1
	14.4	11.9	15.5	14.2	12.3	12.1	16.1	15.9	15.0	15.7	10.7	9.2	13.4	12.9	14.2	13.1	13.1	13.6
	14.2	13.3	11.5	16.2	12.0	11.4	15.8	18.0	14.8	16.3	10.1	10.4	13.1	13.3	14.1	13.7	12.7	13.4
	15.2	13.3	13.4	15.6	12.5	11.3	15.9	16.7	14.4	15.7	10.7	10.0	13.3	12.4	15.1	12.8	12.1	13.0
	13.8	13.2	13.5	14.3	13.2	11.6	15.9	17.1	14.7	15.1	10.3	10.8	12.4	12.3	13.5	12.5	13.1	13.4
	15.4	12.5	14.8	14.2	13.0	11.5	16.5	16.5	14.7	15.0	10.3	11.0	13.1	12.4	13.9	13.6	12.4	13.2
	13.2	12.0	12.8	15.0	12.9	12.1	16.2	16.0	14.1	15.6	10.8	11.3	14.2	12.1	14.6	13.3	12.5	13.1
	14.0	12.1	14.1	16.2	12.6	11.6	16.3	15.8	13.8	16.1	11.0	9.7	15.4	12.7	13.6	12.1	12.5	12.5
	14.7	12.8	14.2	13.7	12.4	11.6	16.2	16.1	14.1	15.2	10.7	10.5	14.4	10.9	13.2	13.1	12.7	12.9
	14.6	13.1	14.0	14.0	12.0	11.9	15.9	16.8	14.7	15.6	10.7	9.8	14.5	12.2	14.1	13.1	12.8	13.0
	15.4	13.7	14.4	15.4	12.0	11.3	14.9	17.4	13.7	15.3	10.6	9.9	14.0	11.7	13.8	13.1	13.2	13.0
	14.3	11.5	13.3	15.8	11.6	11.6	16.5	15.4	13.7	15.7	10.2	10.3	15.0	11.8	16.0	12.5	12.5	12.6
	14.1	12.7	15.0	16.6	12.3	11.4	15.9	17.9	14.4	15.0	9.5	10.7	13.4	11.8	14.5	13.0	13.3	13.3
	16.9	13.0	13.8	16.9	12.4	10.7	15.4	17.0	15.4	16.2	10.8	10.7	14.6	12.2	14.9	12.6	11.9	12.3
	17.9	13.6	14.8	15.0	12.8	12.6	15.8	16.1	14.5	16.3	10.7	10.7	13.1	11.8	14.4	13.5	12.7	12.9
	17.3	12.3	14.6	13.8	12.3	11.5	15.0	16.8	15.9	14.7	10.9	10.3	12.8	12.2	13.9	13.1	13.0	13.1
	15.9	11.5	14.4	15.4	12.6	11.5	16.2	14.9	14.9	15.0	10.1	10.3	13.8	12.6	13.4	13.7	12.7	12.7
	13.6	13.2	13.1	15.0	21.5	11.7	15.3	18.3	15.4	15.6	10.5	10.6	13.0	12.7	14.1	12.9	12.9	12.7
	13.5	11.9	14.1	14.5	12.0	11.3	15.2	17.2	15.2	15.7	10.2	10.9	13.6	12.1	13.7	12.4	13.4	12.6
	14.7	12.3	14.8	14.5	11.8	12.4	15.7	17.6	14.2	15.6	10.6	10.1	13.2	12.7	14.1	13.5	12.3	13.8
	14.5	13.3	14.3	15.0	12.9	11.7	15.6	16.7	14.3	15.6	10.9	10.4	13.6	12.6	14.2	13.3	13.6	13.1
	15.5	12.6	14.3	15.6	12.2	11.8	15.3	15.8	15.3	15.0	10.3	10.4	15.8	12.1	13.4	13.4	13.2	13.7
	14.1	12.2	15.4	16.4	12.2	11.3	14.9	16.5	15.0	16.8	10.1	10.8	15.4	11.5	13.8	12.6	13.6	13.3
	17.2	13.5	13.8	15.5	12.7	12.0	16.7	16.7	14.4	15.8	10.6	10.0	15.0	11.1	14.6	12.4	13.3	14.2
	16.6	12.1	14.7	11.9	12.9	11.5	16.3	17.6	15.3	15.6	10.3	10.5	14.2	12.5	14.0	13.0	14.3	14.5
	16.8	13.0	15.4	15.2	11.9	12.7	15.8	17.0	14.9	15.8	10.2	9.7	14.8	12.0	14.3	13.2	12.0	13.5
	16.9	11.3	14.9	16.7	12.0	12.1	15.0	16.2	14.3	15.7	10.9	10.8	14.2	12.0	13.7	13.4	12.8	13.7
	14.3	12.8	16.2	15.3	12.3	11.5	15.2	16.9	14.7	16.2	10.3	10.3	12.9	11.9	14.8	13.5	14.1	12.9
	17.6	12.7	14.9	15.3	12.0	13.2	15.6	17.4	13.6	15.8	10.5	10.8	12.8	11.2	14.3	13.0	13.6	13.7
	13.3	12.7	15.0	15.2	12.8	11.8	16.3	15.8	14.7	16.1	9.9	10.5	13.0	11.4	14.7	13.1	13.1	13.8
	15.2	13.0	15.1	15.1	12.3	12.6	15.8	17.0	16.2	16.1	10.7	10.2	13.8	11.2	13.9	13.2	12.4	14.1
Mean	15.09	12.84	14.20	15.22	12.51	11.70	15.67	16.38	14.60	15.74	10.51	10.52	13.94	12.31	14.21	12.87	12.89	13.23
Std dev	1.22	0.77	0.89	1.05	1.52	0.48	0.67	1.24	0.59	0.52	0.41	0.54	0.86	0.70	0.61	0.63	0.57	0.52

ANNEX D Table D2:Certification data for critical load Lc_2 (N). Sample numbers are given in the second row.

	Lab 1		Lab 2		Lab 3		Lab 4		Lab 5		Lab 6		Lab 7		Lab 8		Lab 9	
Sample	**0077**	**0711**	**0436**	**1064**	**0235**	**0635**	**0350**	**0986**	**0104**	**0134**	**0613**	**0588**	**1029**	**0560**	**0383**	**0649**	**0512**	**0851**
	18.2	15.8	18.4	20.3	15.2	15.9	15.9	19.3	17.5	20.9	12.5	11.8	16.6	14.1	17.5	18.9	14.7	15.9
	18.2	15.9	17.1	19.8	18.3	14.9	18.6	20.4	17.6	25.4	12.6	13.3	16.3	14.6	23.0	19.4	15.1	16.5
	19.2	16.7	16.9	20.6	15.2	14.3	18.4	20.4	16.6	22.9	11.8	14.0	17.0	15.3	17.2	15.9	15.4	15.8
	18.4	16.3	17.9	19.7	17.3	16.5	17.9	18.8	21.3	20.9	13.8	12.9	16.4	14.8	17.2	13.9	15.4	16.0
	19.1	17.1	18.5	19.2	14.4	14.5	17.6	19.8	18.8	20.0	12.8	13.6	17.2	13.5	18.0	16.0	16.0	17.5
	17.5	16.6	17.9	21.1	15.2	14.6	18.6	19.3	18.5	22.3	12.4	12.7	17.3	14.3	16.2	14.6	15.3	15.7
	18.2	15.7	17.5	18.3	16.1	15.0	18.8	18.7	18.4	26.4	13.5	11.9	17.1	16.3	17.2	16.7	14.5	16.2
	18.3	16.5	18.7	18.5	15.6	15.4	19.6	19.9	18.6	19.3	13.3	13.5	16.3	15.4	17.8	15.2	15.7	16.9
	18.9	15.5	16.8	19.2	16.1	14.5	18.6	18.5	17.2	20.4	13.7	13.7	15.6	14.4	18.8	15.8	15.7	15.9
	17.6	17.0	18.3	23.0	17.6	14.0	19.5	20.2	17.6	19.8	13.9	13.1	15.9	14.9	17.4	17.9	16.7	15.2
	19.6	15.5	19.0	22.8	15.4	14.2	18.7	19.9	18.3	19.7	12.5	11.5	16.0	15.2	17.1	16.5	15.8	17.1
	17.3	17.2	14.8	20.4	16.2	14.2	21.0	20.5	18.4	20.7	13.2	12.0	15.4	15.2	17.9	16.0	15.5	15.5
	18.3	15.9	18.6	18.9	17.8	15.1	19.1	20.4	18.9	19.7	14.3	13.0	15.2	13.6	22.7	18.5	16.2	14.9
	17.7	16.2	18.9	17.9	17.3	14.8	19.5	20.9	18.0	18.3	12.5	13.5	14.6	14.3	18.9	15.1	15.6	16.2
	18.3	16.5	16.9	17.8	18.1	14.2	20.7	20.2	18.1	20.4	12.6	13.7	15.3	14.0	16.9	15.2	15.4	15.9
	17.4	15.9	19.2	17.9	16.5	15.1	19.0	20.6	17.2	19.3	13.2	13.2	16.9	13.8	18.2	18.0	15.4	15.7
	17.6	16.1	18.3	19.4	17.2	14.1	19.5	21.7	18.1	20.8	12.7	12.5	17.3	14.4	18.7	15.0	15.4	15.3
	20.4	15.8	17.1	16.7	17.0	14.7	19.2	19.4	18.2	24.7	13.6	12.4	18.1	13.1	17.1	15.4	14.8	15.7
	17.6	15.7	17.2	17.3	16.2	15.4	18.9	19.3	18.4	20.4	12.9	12.1	18.0	14.9	17.6	16.3	15.8	16.0
	18.8	16.1	17.8	18.5	15.9	14.7	18.4	19.7	18.5	19.8	12.8	13.5	17.0	13.5	17.1	16.3	16.9	16.9
	19.8	15.8	24.6	20.7	16.1	13.6	18.7	20.3	18.6	19.0	12.7	13.7	17.8	14.2	18.1	17.7	15.0	14.7
	17.3	16.4	21.8	20.4	16.2	15.3	17.9	20.4	19.6	20.2	13.4	14.1	16.1	13.6	18.1	16.4	14.8	16.0
	21.6	15.9	16.4	21.7	17.1	16.5	19.0	21.2	18.1	20.6	12.8	12.8	16.9	13.9	18.4	15.4	14.5	15.1
	20.4	16.1	17.3	18.9	16.8	17.7	19.1	19.8	16.8	20.4	12.2	15.3	15.9	13.9	17.1	15.4	16.0	15.7
	20.2	16.1	17.7	19.1	16.5	15.4	19.5	19.4	18.1	19.0	13.9	13.0	17.8	13.9	16.4	17.5	15.7	15.5
	19.8	16.3	19.7	20.3	17.1	14.0	19.1	18.7	18.5	21.5	13.6	12.2	16.3	15.5	17.7	16.5	14.4	15.9
	22.4	16.6	18.7	17.5	14.3	15.3	18.5	20.2	18.7	18.7	13.6	11.9	15.1	15.0	17.3	16.5	17.8	16.3
	19.5	15.8	23.5	18.7	15.0	14.0	17.6	20.4	18.0	20.5	13.9	12.9	15.2	14.0	19.2	15.3	15.4	15.4
	16.9	15.1	19.5	26.2	16.7	15.4	20.4	20.2	18.8	21.9	12.7	12.8	16.8	14.6	17.4	16.1	15.4	16.9
	21.9	17.1	18.3	19.0	16.2	14.7	18.5	21.5	20.0	19.4	13.7	12.9	16.7	14.2	18.3	16.2	15.7	15.8
	19.4	16.0	17.9	19.0	16.0	14.7	18.4	18.5	19.0	19.0	11.7	12.9	18.3	13.7	17.0	17.0	14.7	15.3
	16.8	14.9	18.5	19.7	15.7	15.1	20.4	19.7	21.0	21.1	12.1	13.5	17.2	14.0	18.2	16.4	15.2	16.1
	22.5	16.7	17.2	19.0	16.1	14.7	19.5	20.2	18.4	19.8	13.7	12.9	17.4	15.2	19.3	16.4	15.6	16.4
	19.9	16.1	17.6	17.2	15.7	16.1	19.2	20.7	17.8	22.7	12.7	12.6	17.0	13.7	17.4	18.8	15.6	16.1
	22.0	16.2	18.1	18.6	14.7	15.1	19.5	19.7	18.7	22.1	12.5	12.9	16.7	14.9	16.5	15.4	18.4	16.1
	20.2	14.2	18.7	19.0	14.7	16.1	19.4	19.8	16.4	21.1	13.0	14.0	16.8	14.7	18.7	16.1	15.9	16.6
	22.2	15.0	19.0	19.1	16.4	14.1	18.0	20.3	19.8	19.6	12.0	13.2	14.9	13.7	16.4	15.9	15.4	16.1
	21.8	16.1	19.2	20.6	16.6	15.5	19.0	20.1	19.4	17.7	13.1	12.8	15.2	13.3	18.1	16.4	17.0	15.8
	21.5	16.1	18.7	18.6	15.6	14.5	19.9	20.3	20.3	20.0	14.5	12.1	14.7	13.5	16.6	16.7	15.5	16.2
	20.5	16.3	18.0	18.9	15.4	15.1	18.3	19.8	17.6	18.7	12.7	12.3	15.7	12.7	16.8	21.1	18.0	15.9
Mean	19.32	16.07	18.41	19.47	16.19	14.94	18.93	19.98	18.46	20.64	13.03	12.97	16.45	14.30	17.87	16.50	15.68	15.96
Std dev	1.69	0.60	1.73	1.76	1.02	0.85	0.93	0.75	1.06	1.83	0.66	0.76	0.99	0.75	1.40	1.41	0.91	0.58

ANNEX D Table D3:Certification data for critical load Lc_3 (N). Sample numbers are given in the second row.

	Lab 1		Lab 2		Lab 3		Lab 4		Lab 5		Lab 6		Lab 7		Lab 8		Lab 9	
Sample	**0077**	**0711**	**0436**	**1064**	**0235**	**0635**	**0350**	**0986**	**0104**	**0134**	**0613**	**0588**	**1029**	**0560**	**0383**	**0649**	**0512**	**0851**
	28.7	23.9	38.1	35.4	27.2	25.7	30.3	28.6	27.1	33.5	26.4	29.7	27.2	21.4	28.2	23.0	28.3	25.2
	30.9	25.7	36.0	36.6	30.6	27.6	28.2	29.0	28.0	32.2	26.5	30.0	25.7	20.8	28.1	26.0	27.8	27.6
	30.3	27.4	37.2	34.1	28.3	27.4	28.7	31.2	29.3	30.4	26.8	27.8	25.9	24.0	30.5	25.6	29.3	26.4
	30.4	24.5	35.1	41.2	29.4	26.0	26.8	28.7	30.0	31.5	25.6	26.1	22.5	22.6	26.5	21.6	26.0	25.4
	31.7	28.4	30.8	35.6	29.5	29.2	27.2	29.6	28.2	31.6	26.3	28.3	25.2	22.7	30.0	23.4	28.6	26.6
	30.7	28.8	30.4	31.2	26.2	28.7	27.6	29.4	30.4	32.3	24.4	28.9	25.1	22.5	28.1	25.3	27.0	23.5
	29.3	29.5	34.6	33.2	26.5	27.6	27.5	30.6	29.8	30.0	27.0	25.0	24.3	23.3	27.1	24.1	25.6	25.4
	29.3	27.4	32.8	38.4	25.7	25.9	28.9	29.6	29.6	30.8	27.6	26.9	21.1	23.5	30.0	25.9	24.7	25.8
	29.6	28.7	38.1	39.1	28.3	27.6	27.8	30.7	26.7	31.8	27.9	28.8	21.2	23.5	30.2	24.0	22.2	27.1
	19.8	28.1	34.9	32.8	30.4	27.1	28.2	28.6	28.6	31.9	26.4	28.0	20.6	20.4	29.3	24.1	24.9	28.0
	30.8	29.2	30.8	36.9	26.3	28.8	28.9	30.5	27.1	28.7	27.2	25.9	22.9	24.5	26.2	23.4	29.1	25.3
	27.8	24.8	28.0	37.9	32.0	25.7	29.1	28.5	32.7	29.2	25.4	24.1	25.7	21.3	26.2	24.3	26.5	26.9
	25.8	31.5	31.2	35.6	29.7	25.0	29.5	29.2	29.0	30.0	26.3	28.8	23.7	20.5	27.9	24.7	26.2	24.9
	29.6	27.4	40.5	29.2	29.7	24.9	29.5	29.8	31.8	30.7	26.9	28.3	21.2	20.2	27.7	24.3	24.5	25.1
	30.3	29.5	31.4	33.4	27.5	24.7	27.1	28.1	28.1	31.3	25.2	29.7	23.1	23.6	28.2	25.1	25.5	26.1
	27.9	33.7	40.1	35.8	24.8	28.3	29.4	29.6	31.1	30.2	28.0	27.0	24.6	20.8	29.3	22.9	27.9	24.6
	30.1	30.2	33.9	37.3	26.6	29.7	28.0	28.1	29.4	32.5	27.4	25.3	24.3	22.5	26.5	25.1	26.0	26.0
	31.4	27.9	39.5	35.3	26.6	29.4	29.3	27.5	31.2	31.9	26.1	26.8	23.8	20.6	25.2	25.1	28.4	28.9
	31.1	29.0	32.6	29.3	31.4	26.9	28.3	27.6	30.5	31.7	27.0	25.2	23.0	23.1	27.9	25.6	24.2	27.6
	26.9	27.0	29.8	33.1	28.8	25.3	29.4	28.7	31.4	30.5	26.0	27.5	25.9	20.3	28.5	25.9	28.1	26.6
	29.2	36.5	42.1	38.7	25.4	27.1	28.5	27.9	33.8	28.7	26.8	26.6	23.5	20.8	29.7	25.4	27.4	30.1
	36.9	25.7	31.2	41.4	29.3	27.0	27.2	29.1	27.9	31.6	28.0	27.8	22.4	21.1	26.6	25.0	27.3	25.0
	34.9	31.8	32.8	38.3	27.8	26.4	29.2	29.0	30.3	29.2	28.1	27.1	25.1	21.2	28.7	24.8	27.0	25.6
	33.7	26.9	31.8	32.8	29.7	24.0	28.5	29.9	31.5	29.6	27.4	24.5	21.2	19.8	27.3	24.7	29.1	27.9
	33.8	27.6	36.3	31.8	26.6	27.3	29.9	28.9	30.5	31.7	24.7	26.3	21.0	21.2	27.1	24.7	26.1	27.4
	34.9	27.9	40.5	36.9	26.9	24.1	26.7	28.6	30.8	32.8	25.5	24.7	25.7	19.0	27.4	25.5	25.7	28.1
	33.1	27.1	31.3	38.9	27.1	23.3	28.7	30.8	29.2	31.9	27.3	25.9	22.0	20.0	28.4	25.5	24.5	27.4
	28.8	26.3	33.3	31.7	27.6	30.1	26.7	29.5	30.1	27.9	25.0	28.3	20.4	21.6	32.4	26.7	25.3	25.5
	40.5	28.3	36.7	35.6	26.2	26.2	28.3	30.5	29.0	31.0	27.6	24.8	21.4	19.8	28.2	26.3	24.9	28.1
	28.7	27.4	33.5	35.6	29.4	24.8	28.9	30.5	30.8	30.0	27.4	26.4	21.8	20.3	28.0	25.5	25.5	27.6
	27.9	24.4	28.8	35.0	27.6	30.0	30.3	27.8	30.4	31.2	25.4	26.5	26.8	18.7	27.0	26.3	23.5	25.7
	25.3	26.4	36.4	34.8	27.5	27.5	29.6	28.5	31.3	29.1	25.1	27.8	25.3	19.6	28.0	25.5	23.9	24.8
	30.5	25.2	30.2	38.5	27.7	18.8	28.4	32.1	32.6	28.4	26.9	26.4	22.2	19.7	27.8	24.5	24.5	26.9
	36.6	26.9	35.2	32.5	26.9	25.7	29.6	30.0	31.3	28.7	27.1	27.4	25.7	19.2	26.4	25.6	24.1	27.7
	35.7	24.7	36.4	32.5	28.8	25.8	28.1	28.3	31.3	29.2	23.5	27.6	23.2	18.8	26.5	25.1	24.2	25.0
	26.6	18.5	29.8	43.8	28.1	25.6	28.2	30.2	28.3	30.4	26.3	24.6	26.1	21.2	27.8	26.3	24.9	25.6
	27.9	36.8	38.8	35.5	34.3	27.7	28.0	28.3	28.9	30.8	28.3	25.8	23.5	21.5	28.3	26.6	24.8	28.4
	26.2	26.0	39.0	38.5	29.0	25.4	28.8	29.6	28.4	31.0	24.8	26.2	20.6	18.7	28.9	25.7	25.0	26.9
	36.2	32.3	39.3	36.7	33.2	30.7	28.9	29.9	27.0	32.5	26.6	28.7	24.1	20.3	29.5	24.0	26.2	26.5
	36.6	34.9	33.0	40.2	36.4	26.4	27.4	29.4	27.5	32.7	26.4	26.3	21.8	21.5	28.3	25.8	24.6	26.1
Mean	30.66	28.10	34.55	35.75	28.55	26.67	28.49	29.31	29.79	30.80	26.46	26.94	23.52	21.15	28.09	24.99	25.98	26.49
Std dev	3.91	3.49	3.73	3.26	2.42	2.20	0.96	1.05	1.71	1.39	1.13	1.55	1.96	1.55	1.42	1.10	1.74	1.38

ANNEX E

Table E1**: Lc_1 control measurements (N) made by lab 9 on samples used for the certification analyses. Each sample was tested with the certifying stylus used by the original lab and with a control stylus 2238-36.**

Lc_1																		
Orig. lab	1	1	2	2	3	3	4	4	5	5	6	6	7	7	8	8	9	9
sample	0077	0711	1064	0436	0235	0635	0986	0350	0104	0134	0613	0588	0560	1029	0383	0649	0512	0851
Cert. stylus	2238-33	2238-33	2238-39	2238-39	2238-43	2238-43	2238-46	2238-46	2238-50	2238-50	2238-56	2238-56	2238-57	2238-57	2238-51	2238-51	2238-36	2238-36
	13.1	12.7	13.6	13.9	11.7	11.5	15.1	16.1	13.4	14.1	10.5	11.4	13.2	14.1	13.6	12.6	12.7	13.5
	12.7	13.0	12.8	12.0	12.1	10.5	15.6	15.5	13.2	12.4	10.7	11.1	13.5	13.4	13.5	12.2	12.2	12.6
	13.2	12.1	12.9	13.8	12.8	12.2	16.1	15.3	13.6	13.7	12.6	10.6	12.8	14.3	14.7	12.3	12.4	12.9
	13.4	12.4	12.9	14.2	11.6	11.2	15.7	17.1	13.7	13.5	10.6	10.4	13.4	14.3	14.8	12.8	12.7	12.6
	12.9	12.6	15.1	14.5	12.1	11.8	16.2	16.2	13.6	13.6	10.4	11.0	13.0	13.8	14.6	12.6	12.8	13.5
Mean	13.05	12.56	13.45	13.67	12.07	11.44	15.74	16.06	13.51	13.46	10.95	10.90	13.18	13.96	14.23	12.51	12.53	13.01
Std dev	0.28	0.35	0.94	0.97	0.48	0.66	0.44	0.69	0.18	0.63	0.93	0.39	0.32	0.39	0.64	0.24	0.24	0.44
Control stylus	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36
	15.3	13.9	16.2	13.0	12.9	13.2	13.1	13.8	12.7	13.0	11.9	12.2	11.5	14.2	15.1	13.5	11.3	11.7
	14.2	14.3	13.5	13.3	13.9	12.7	13.8	13.1	13.2	14.2	12.2	13.0	11.9	13.5	15.0	13.4	10.7	12.1
	15.9	13.3	12.7	13.5	12.8	12.6	14.5	13.5	13.0	13.2	12.8	11.9	12.5	13.1	15.3	14.0	11.1	11.7
	15.5	13.4	15.3	13.6	13.2	12.5	13.2	13.8	13.6	14.3	12.3	12.6	13.0	14.0	14.6	13.4	10.1	11.2
	14.6	14.4	12.1	13.2	13.7	12.9	14.5	13.7	13.0	12.7	12.9	13.4	12.7	13.5	15.9	13.5	11.1	11.9
Mean	15.10	13.86	13.95	13.31	13.30	12.78	13.81	13.56	13.09	13.47	12.40	12.64	12.33	13.65	15.20	13.57	10.87	11.71
Std dev	0.67	0.51	1.75	0.21	0.49	0.30	0.69	0.30	0.34	0.73	0.44	0.59	0.61	0.44	0.46	0.27	0.46	0.34

ANNEX E

Table E2**: Lc_2 control measurements (N) made by lab 9 on samples used for the certification analyses. Each sample was tested with the certifying stylus used by the original lab and with a control stylus 2238-36.**

Lc_2																		
Orig. lab	1	1	2	2	3	3	4	4	5	5	6	6	7	7	8	8	9	9
sample	0077	0711	1064	0436	0235	0635	0986	0350	0104	0134	0613	0588	0560	1029	0383	0649	0512	0851
Cert. stylus	2238-33	2238-33	2238-39	2238-39	2238-43	2238-43	2238-46	2238-46	2238-50	2238-50	2238-56	2238-56	2238-57	2238-57	2238-51	2238-51	2238-36	2238-36
	16.6	15.3	16.3	16.2	14.0	15.7	19.5	18.9	21.4	16.7	13.2	12.8	19.1	16.3	17.5	17.3	14.1	15.2
	16.8	15.9	17.1	15.9	14.6	12.4	19.1	18.6	21.6	16.3	13.1	13.1	15.7		17.4	15.6	13.9	15.2
	17.1	15.8	15.9	16.2	16.0	13.4	19.5	20.2	18.2	22.5	16.9	12.4	16.1	17.1	25.0	16.7	18.1	15.6
	15.9	15.4	16.3	15.5	13.4	13.8	19.7	19.1	16.4	16.9	13.2	13.4	18.5	16.5	15.8	17.8	13.8	15.4
	16.2	15.3	17.4	15.7	15.0	14.7	18.8	19.2	16.9	17.1	13.3	13.3	17.8	21.3	17.0	15.2	14.5	14.9
Mean	16.51	15.53	16.61	15.92	14.60	13.99	19.31	19.20	18.90	17.90	13.95	13.01	17.45	17.78	18.52	16.51	14.89	15.25
Std dev	0.45	0.28	0.63	0.31	0.97	1.27	0.37	0.57	2.48	2.57	1.62	0.40	1.46	2.35	3.67	1.10	1.84	0.24
Control stylus	2238-39	2238-40	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-38	2238-36	2238-36
	18.4	16.5	17.8	16.1	16.6	15.4	19.3	16.4	16.9	16.4	14.0	15.2	15.4	17.2	18.3	16.4	13.4	13.4
	17.7	15.9	17.1	18.4	16.4	14.7	19.9	16.7	15.8	15.9	14.8	14.8	14.5	16.0	18.5	15.9	12.8	18.4
	18.2	15.9	16.0	17.0	15.5	15.1	16.1	17.0	17.0	16.3	14.3	15.3	15.8	16.4	17.6	16.1	13.3	14.4
	18.3	16.6	17.7	19.4	17.4	17.5	16.7	16.1	16.0	15.7	14.5	14.9	16.0	15.8	18.2	16.5	13.7	13.7
	19.2	15.6	17.5	16.3	19.4	15.6	20.4	16.8	16.0	15.6	15.1	14.8	15.8	15.7	18.3	16.3	13.6	13.8
Mean	18.38	16.10	17.21	17.44	17.05	15.65	18.47	16.59	16.35	15.97	14.54	15.00	15.49	16.22	18.19	16.21	13.36	14.72
Std dev	0.52	0.40	0.74	1.40	1.47	1.09	1.98	0.36	0.57	0.36	0.42	0.24	0.63	0.62	0.34	0.25	0.33	2.07

ANNEX E

Table E3**: Lc_3 control measurements (N) made by lab 9 on samples used for the certification analyses. Each sample was tested with the certifying stylus used by the original lab and with a control stylus 2238-36.**

Lc_3																		
Orig. lab	1	1	2	2	3	3	4	4	5	5	6	6	7	7	8	8	9	9
sample	0077	0711	1064	0436	0235	0635	0986	0350	0104	0134	0613	0588	0560	1029	0383	0649	0512	0851
Cert. stylus	2238-33	2238-33	2238-39	2238-39	2238-43	2238-43	2238-46	2238-46	2238-50	2238-50	2238-56	2238-56	2238-57	2238-57	2238-51	2238-51	2238-36	2238-36
	33.5	24.8	34.3	28.3	31.7	24.5	29.2	29.4	25.6	27.9	29.6	27.6	28.8	29.8	35.8	21.0	24.4	23.2
	28.6	24.6	29.5	31.3	31.7	24.7	29.0	26.3	28.4	30.7	27.7	29.5	33.3	29.3	30.1	29.2	23.1	23.2
	30.9	26.3	28.0	25.9	30.1	26.0	29.8	30.1	27.2	28.0	31.9	28.8	28.8	27.8	39.5	26.8	22.2	23.9
	29.9	35.9	27.2	33.0	28.6	24.5	33.1	30.3	27.0	29.4	29.1	30.5	30.0	28.5	33.7	25.2	21.3	26.3
	28.1	23.6	32.3	27.5	31.9	26.9	30.4	32.3	26.9	27.3	26.6	30.9	29.0	26.9	39.7	21.9	23.2	25.0
Mean	30.19	27.06	30.27	29.21	30.78	25.32	30.28	29.68	27.02	28.68	28.98	29.47	29.99	28.44	35.76	24.80	22.82	24.31
Std dev	2.16	5.05	2.97	2.90	1.44	1.10	1.66	2.18	0.99	1.37	2.00	1.34	1.91	1.17	4.03	3.38	1.15	1.33
Control stylus	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36	2238-36
	39.1	30.0	26.1	25.1	26.4	25.0	28.7	27.5	29.6	28.3	23.6	26.7	23.0	26.4	30.2	26.4	22.4	23.9
	33.2	26.8	27.9	27.8	26.6	27.0	28.7	31.2	25.6	28.1	26.2	26.6	26.0	26.1	30.5	32.6	22.3	27.6
	33.2	26.3	28.5	27.5	28.4	23.5	26.9	25.9	25.9	30.5	23.0	29.8	28.0	27.1	32.1	27.3	22.7	24.6
	33.8	28.5	36.0	27.6	28.5	24.1	30.8	28.4	28.0	27.4	22.6	24.3	27.2	28.6	33.1	26.6	25.0	22.5
	30.9	25.9	28.1	26.0	26.3	23.7	29.4	28.4	27.4	24.5	23.7	28.3	26.9	28.1	33.6	25.7	23.1	25.9
Mean	34.06	27.51	29.32	26.81	27.24	24.66	28.89	28.26	27.29	27.76	23.83	27.12	26.23	27.26	31.91	27.74	23.10	24.92
Std dev	3.05	1.73	3.85	1.20	1.11	1.43	1.38	1.94	1.62	2.17	1.41	2.04	1.94	1.07	1.51	2.79	1.08	1.94

ANNEX F. Stylus sensitivity data.

Table F1. Critical loads Lc_1 - Lc_3 in N for eight styli determined on sample 0445 by lab 9 and stylus radii (µm) determined by three methods (OMS, KE, Interference) measuring in the 0° orientation.

Stylus	2238-26			2238-31			2238-39			2238-43			2238-46			2238-50			2238-56			2238-57		
	Lc_1	Lc_2	Lc_3	Lc_1	Lc_2	Lc_3	Lc_1	Lc_2	Lc_3	Lc_1	Lc_2	Lc_3	Lc_1	Lc_2	Lc_3	Lc_1	Lc_2	Lc_3	Lc_1	Lc_2	Lc_3	Lc_1	Lc_2	Lc_3
	15.9	19.0	33.1	15.6	17.9	30.9	14.6	17.6	31.5	15.7	15.7	30.4	17.6	20.8	32.8	16.5	-	38.7	14.0	15.3	34.4	16.7	24.2	34.3
	15.3	19.0	32.4	14.7	18.5	37.7	15.2	19.0	38.9	16.4	16.4	32.1	17.7	19.9	33.5	17.2	-	35.7	14.2	15.6	35.8	15.7	19.9	35.9
	16.4	19.1	34.3	15.1	17.8	27.0	14.3	17.7	34.8	16.0	16.0	36.5	17.4	21.1	32.5	16.0	-	32.1	13.8	15.5	40.3	16.4	25.6	31.8
	16.5	19.3	33.4	16.6	19.4	34.9	14.6	18.7	40.7	17.6	17.6	42.8	17.7	20.9	35.5	17.5	-	39.1	14.0	15.9	36.5	15.7	25.2	35.7
	15.7	19.2	34.1	15.4	19.2	29.4	13.8	17.9	32.3	17.0	17.0	32.5	16.2	21.3	35.2	16.2	-	33.7	14.4	16.8	40.3	16.2	19.8	39.5
	16.5	19.2	40.8	15.8	18.5	29.1	15.9	18.4	33.4	16.8	16.8	31.3	17.3	20.8	31.5	15.9	-	33.9	13.8	15.9	34.0	16.5	20.0	31.8
	15.8	18.5	32.6	15.1	18.5	26.3	14.8	19.3	34.5	15.5	15.5	30.0	17.6	21.9	33.4	16.7	-	37.9	13.5	16.0	32.8	16.7	18.4	40.1
	16.6	18.7	34.2	16.1	18.3	34.9	15.7	17.4	28.7	15.6	15.6	30.0	18.6	22.3	35.1	14.1	-	35.9	14.0	15.8	41.9	15.7	23.3	34.9
	16.4	19.0	32.1	14.6	18.3	33.4	14.7	17.6	35.0	15.5	15.5	31.6	17.9	20.5	36.1	16.9	-	42.4	14.3	15.9	39.3	14.5	22.6	36.5
Mean	16.1	19.0	34.1	15.4	18.5	31.5	14.8	18.1	34.4	16.2	16.2	33.0	17.6	21.0	34.0	16.3	-	36.6	14.0	15.9	37.2	16.0	22.1	35.6
Std dev	0.5	0.2	2.6	0.7	0.5	3.9	0.7	0.8	3.7	0.8	0.8	4.2	0.6	0.7	1.6	1.0	-	3.2	0.3	0.4	3.3	0.7	2.7	2.9
OMS	200			195			198			200			201			200			198			199		
KE	194			190			204			205			199			200			201			203		
Interf.	195			191			210			199			195			203			202			204		

Table F2. ANOVA of stylus sensitivity data

Lc_1	*Source of Variation*	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
	Between Groups	72.308	7	10.33	22.914	3E-15	2.1564
	Within Groups	28.851	64	0.4508			
	Total	101.16	71				

MS between = $n.u^2_{ss}+u^2_{ws}$ u_{ss} = 1.05 N

MS within = u^2_{ws} u_{ws} = 0.67 N

n = no. of replicates = 9

Lc_2	*Source of Variation*	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
	Between Groups	285.71	6	47.62	36.151	2E-17	2.2656
	Within Groups	73.763	56	1.317			
	Total	359.47	62				

MS between = $n.u^2_{ss}+u^2_{ws}$ u_{ss} = 2.26 N

MS within = u^2_{ws} u_{ws} = 1.15 N

n = no. of replicates = 9

Lc_3	*Source of Variation*	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
	Between Groups	222.14	7	31.73	2.984	0.0090	2.1564
	Within Groups	680.59	64	10.63			
	Total	902.74	71				

MS between = $n.u^2_{ss}+u^2_{ws}$ u_{ss} = 1.53 N

MS within = u^2_{ws} u_{ws} = 3.26 N

n = no. of replicates = 9

European Commission

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The certification of critical coating failure loads: a reference material for scratch testing according to ENV 1071: 1994 – BCR-692
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Abstract

Scratch testing procedures for the testing of adhesive and cohesive strength of hard coatings, described in the European standard prEN 1071-3, require the measurement of "critical load" values associated with characteristic failure events occurring when a stylus is drawn across the coating with a normal load applied. The CRM BCR-692 has been produced and certified for verification and monitoring of the proper functioning of scratch test instruments. The reference samples are (30x30x5) mm steel coupons with a diamond-like carbon coating (DLC) applied by plasma-assisted chemical vapour deposition. Certification of critical load values was carried out by nine laboratories using three different types of scratch testing instruments and specially prepared Rockwell C diamond styli. A study of heterogeneity and stability was made and the sensitivity of critical load to stylus tip radius was also investigated. Certified values were assigned for three critical loads Lc_1, Lc_2, Lc_3 (corresponding to three particular failure events) with uncertainties arising from the evaluation of the material properties. In addition, machine "verification ranges" (wider than the certified ranges), to be used for verification of scratch test instruments were assigned, including additional components of uncertainty related to stylus variability and machine type. The verification ranges can be used to verify any scratch test instrument compliant with, and calibrated according to, prEN 1071-3. The certified and verification ranges are:

Failure event[1)]	Certified range[2)] (N)	Verification range[2)] (N)
Forward chevron cracks at the borders of the scratch track (Lc1 shall be taken at the closest end of the event to the scratch track start).	Lc1 (13.6 ± 1.9)	Lc1 (13.6 ± 3.1)
Forward chevron cracks at the borders of the scratch track, with local interfacial spallation or with gross interfacial spallation (Lc2 shall be taken at the failure event that occurs first, and at the closest end of the event to the scratch track start).	Lc2 (17.0 ± 2.0)	Lc2 (17.0 ± 5.4)
Gross interfacial shell-shaped spallation (Lc3 shall be taken at the first point where the substrate can be seen at the centre of the track in a crescent that goes completely through the track).	Lc3 (27.9 ± 3.6)	Lc3 (27.9 ± 5.7)

[1)] Micrographs of the critical failure events are shown in Annex A of this certification report.

[2)] The uncertainty is the expanded uncertainty estimated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM) with a coverage factor k = 2.

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