Variation of the calibrating value and the range depending on the number of the calibrating indentations by the to Rockwell C hardness test.

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Abstract

Represented is the problem at the definition of measure values in the standards from the statistical point of view. The definition of the calibrating of hardness reference blocks in the standards will be translated to a statistical model formulations. The statistical model will be checked by experimental measurements. The problem of the determination of a quality feature for the hardness reference blocks will be discussed.

1. Introduction

The European Co-operation for Accreditation (EA) "Mechanical Measurements" has developed a concept for the definition and the transmission of measurement variables in a metrological chain [1]. Corresponding to this EA – concept a measurement variable will be transmitted by a primary reference material of the national metrology institute to the calibration laboratories. The transmission from the calibration laboratories to the industry occurs through reference materials.

The central element in this concept is the reference material of the measurement variable. For the transmission of the hardness scales the hardness reference blocks are the reference material. The requirements on the hardness reference blocks are determined for the different hardness tests in the ISO standards [2,3,4]. In accordance with definition in the standards [2,3,4], the calibrating value of a hardness reference blocks is the arithmetic mean value from the 5 hardness values of the 5 calibrating indentations. Characteristic value for homogeneity of material and quality feature of the hardness reference blocks is the relative range. A variation of each of the 5 hardness values is determined through:

- Hardness differences in the material of the hardness reference block
- Variations of the test parameters in the standardizing machine for calibration between the 5 test cycles during the calibration
- Variations of the environmental conditions.

2. Statistical and stochastic point of view

A technical value, measured under special determined conditions is from the statistical point of view a random variable (stochastic variable). If will be carried out a repeating experiment, mostly , the measured values deviate from each other. For n ->∞ repeating experiments it will be possible to calculate the distribution of the population with the characteristic statistical parameters mean value μ and standard deviation σ. In the technical application normally will be worked with random samples, therefore, the characteristically statistical parameters of the population cannot be calculated directly. For the statistical parameters of a representative random sample it will be possible to calculate a confidence interval for each parameter of the population. Results of the calculations which are based on measured values (random variables) again are random variables. For this reason the mean value, the relative range and the standard deviation of a random sample are also random variables. This can be clarified in a simple mind experiment: A random sample with a number of n values will be taken out of the population of all measured values. For this random sample are computed the mean value, the range and the standard deviation. So often as desired a new sample of n values will be taken out and their statistical parameters calculated. For all the parameters which were taken with the same sample size will be calculated the frequency distribution with the characteristical parameters, mean value and standard deviation. For the random variables mean value, relative range and standard deviation will be computed, depending on the sample size, the following parameters:

- The mean value of mean values $\bar{\bar{x}}$ and standard deviation of the mean values $s_{\bar{x}}$

- Mean value of the standard deviations $\overline{x}_s$ and standard deviation of the standard deviations s_s
- Mean value of the relative ranges $\overline{x}_{R_{rel}}$ and standard deviation of the relative ranges $s_{R_{rel}}$

By considering the problems which are described in the introduction, the individual hardness value is a random variable. Because the calibration of hardness reference blocks is a sample test, all values which will be computed from the calibration values like the statistical parameters, mean value, standard deviation and the relative range are also random values. Also all values which will be calculated based on the calibration values of the hardness reference blocks will have random character.

By every definition of a measurement variable in a standard will be defaulted a specific statistical model, e.g.

- sample size
- kind of the sampling
- kind of characteristic calculation e.g. (mean value, standard deviation etc.) onto the random sample or population
- limits of the high-quality values
- kind of distribution

In the actual ISO standards of the hardness test are defined the statistical parameter as follows:

- The sample size is defined to n=5
- For the sampling the surface of the hardness reference blocks will be subdivided into five sectors. In every sector will be made one indentation
- The mean value of the random sample will be equal with the mean value of the population µ and is identical with the correct value
- The basis for calculation of the uncertainty of measurement is the standard deviation of the population under the assumption of normal distribution
- The relative range R_{rel} of the random sample is a characteristic for material homogeneity and the limit value of high quality. The current ISO standards use the range and/or the relative range as the qualification feature for the hardness reference blocks. A qualification feature allows a good or bad decision for the hardness reference block at the limit value of high quality. In accordance with the definition the relative range is the variation width of the population. The relative range of a random sample is a random variable. In this context is the high quality decision for the hardness reference block a random variable. The definition of the relative range is very sensitive at outlier's values (values which will be not explained by the statistics). Also no procedure is estimate to the expectation of the relative range from a random sample to the population. With this background for the hardness reference blocks is determined as high quality feature a relative range on the basis of n=5.

3. Experimental destination and experimental plan

The conformity between the model definition from the standards and the reality will be investigated by the Rockwell C hardness test. In the investigation, the following questions of the statistical model formulation should be clarified:

- How representative are the results of a random sample based on n= 5 repeating experiments
- What variations will be possible for mean value, standard deviation and range
- Who is the probability that a bad hardness reference blocks will have good declaration at the quality limit
- Which is the lowest number n of testing indentations for normal distributed mean values?

3.1 Experimental plan

Testing method: HRC

Testing equipment: Standardizing machine of calibration of the MPA NRW

Test parameter: In accordance with ISO 6508-3

Test pieces: Hardness reference blocks of 3 different manufacturers

Test scope at each hardness reference blocks: 30 series of measurements; within each serie 5 single measurements.

The series of measurements will be occurred under repeatability conditions:

- The same hardness testing machine with unchanged test parameters
- The same user
- Measuring of all 150 values within one day.

The investigation of the measurements includes the following:

- Distribution population and the mean values with the sampling size n = 5, 10, 15, 20, 25 (probability net, asymmetry and excess)
- If it is given a normal distribution of the mean value for the different sample sizes will be checked the equality of the population by F-test and analysis of the variance
- The variations of relative range R_{rel} in a sample size of n = 5
- The variations of mean value, standard deviation in a sample size of n = 5, 10, 15, 20, 25

4. Results

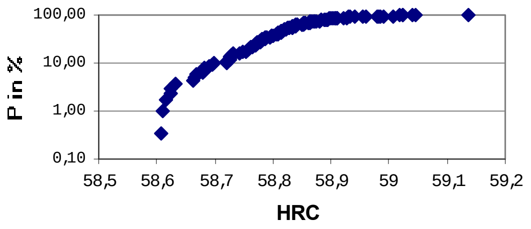
The investigations were carried out in the different ranges of the hardness scale Rockwell C. The experiments are not yet concluded. From this point of view it is an interim report. For a statistical significant correlation between different characteristics values in the hardness scale HRC the number of the previous investigation are too small. In the frame of this report a complete representation of the results would not be possible therefore the representations will be restricted to the standard hardness of 60 HRC.

4.1 Distribution

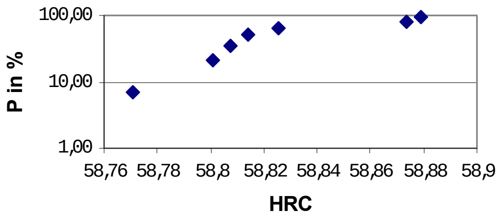
If the found distribution will agree with a well known statistical distribution it will be possible to calculate directly the interesting parameters. Therefore in the first step, the kind of distribution is investigated. In most tests series, the distribution of all measured values shows a small drift. In the beginning of the investigation, the drift of the original values was corrected. All investigations were carried out for the original and the corrected values. The type of distribution was checked for the population and for the mean values with the sample sizes 5, 10,15, 20, 25. The investigation at normal distribution occurred in the probability net and with calculation of the asymmetry and the kurtosis.

Results: In no one of the investigated tests series is the population normally distributed. Also, after a correction of the drift the values do not have a normal distribution. The expected normal distribution of mean values, according to the limit theorem of probability is not given. Examples of the investigated values of the population and of the mean values based on a samples based with n=15 in the probability net show the pictures 4.1 and 4.2.

Picture 4.1: Probability net of the population PI-No. 60-8



Picture 4.2: Probability net of the mean values n =15 PI-No. 60-8



The declaration for no normal distribution will be grounded in differences of the homogeneity of the hardness reference blocks and at the kind of sampling. The hardness distribution in the middle of the hardness reference blocks is mostly, depending on the production process, more uniform than at the edge sectors. In accordance with the definition in the standards, the hardness reference blocks are subdivided into 5 sectors. For every sampling one indentation is made into every sector. In the 4 edge sectors, the indentation generation occurs from the edge in direction to the middle. This effect produces systematical differences between the mean values and the variance parameters depending on the sampling. This explains the deviation from the normal distribution. The values of the asymmetry and the kurtosis confirm the results of the probability net.
Following from this result the interesting parameters cannot be deduced from a well-known statistical model distribution.

4.2 Variation of mean value

For the estimation of the variability of the mean values, of the random samples with the same sample size the mean values will be computed. Concerning these mean values will be computed the mean value and the standard deviation. From all measured values of a test will be calculated the mean value and the standard deviation of the population.
Results: Between the mean values from the random sample mean values on the basis of n = 5, 10, 15, 20, 25 hardness indentations and then mean value of the population, is not detectable a significant difference. In table 4.1 are listed the standard deviations of the mean values depending on the sample size.

Table 4.1 Results of the standard deviation of the mean values. (All values in HRC)

PI-No.	μ	σ	$s_{\bar{x}}$ n=25	$s_{\bar{x}}$ n=20	$s_{\bar{x}}$ n=15	$s_{\bar{x}}$ n=10	$s_{\bar{x}}$ n=5
PI 60-1	60,24	0,120	0,052	0,060	0,057	0,062	0,070
PI 60-2	59,73	0,126	0,062	0,065	0,062	0,070	0,075
PI 60-3	59,46	0,213	0,060	0,052	0,065	0,069	0,086
PI 60-4	61,20	0,112	0,057	0,057	0,057	0,058	0,068
PI 60-5	61,57	0,134	0,041	0,059	0,059	0,061	0,071
PI 60-6	59,60	0,192	0,061	0,054	0,065	0,064	0,100
PI 60-7	58,23	0,853	0,134	0,133	0,134	0,134	0,141
PI 60-8	58,88	0,096	0,046	0,044	0,048	0,050	0,057
PI 60-9	59,28	0,307	0,074	0,068	0,074	0,072	0,082
PI 60-10	59,58	0,204	0,028	0,029	0,044	0,055	0,078

Increasing with the sample size, the confidence interval of $\bar{x}$ gets smaller and closer to μ . The comparison for standard deviation of the population with the standard deviations of the different sample sizes confirms the validity of the root n law.

4.3 Variations of the range

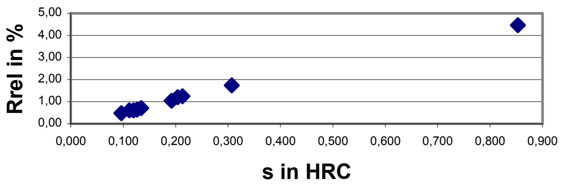
For the estimation of the variability of the relative range, of the random samples with the same sample size relative ranges will be computed. Concerning these relative ranges will be computed the mean value and the standard deviation.
Results: Following the normative definition (base value for calculation is the permanent depth of the indentation), the permissible high quality limit for the relative range will be exceeded more easily for higher hardness values. In table 4.2 are listed the results of the relative range. From the values of the column R_{rel} in table 4.2, the random character of the relative range it becomes clearly. A good example for the sensitivity of the relative range are the results of the hardness reference block PI 60-5. In the column of the failure is specified the part of bad decisions of measurements in test series of the material based on the 30 series.

Table 4.2 Results of the relative range. (All not declared values in HRC)

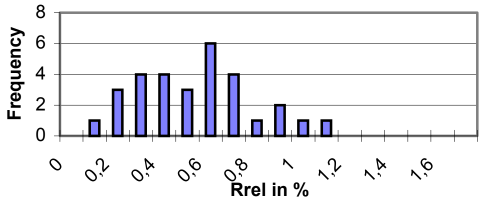
PI-No.	μ	σ	$\overline{x}_R$	s_R	$\overline{x}_{R_{rel}}$ %	$s_{R_{rel}}$ %	Failure %	Min. value R_{rel}	Max. value R_{rel}
PI 60-1	60,24	0,120	0,24	0,09	0,61	0,23	6,7	0,18	1,06
PI 60-2	59,73	0,126	0,26	0,08	0,64	0,21	3,3	0,30	1,09
PI 60-3	59,46	0,213	0,50	0,12	1,24	0,29	83,3	0,62	1,72
PI 60-4	61,20	0,112	0,24	0,08	0,61	0,21	6,7	0,23	1,03
PI 60-5	61,57	0,134	0,27	0,15	0,71	0,39	10,0	0,10	2,13
PI 60-6	59,60	0,192	0,42	0,12	1,05	0,31	53,3	0,50	1,58
PI 60-7	58,23	0,853	1,86	0,54	4,46	1,29	100,0	2,23	7,10
PI 60-8	58,88	0,096	0,20	0,10	0,48	0,25	0,0	0,07	1,00
PI 60-9	59,28	0,307	0,71	0,18	1,74	0,45	93,3	0,88	2,55
PI 60-10	59,58	0,204	0,49	0,14	1,20	0,34	73,3	0,47	1,88

The values are computed directly from the results of the relative ranges. The calculations on the basis of the mean values and standard deviations of the relative ranges according to the statistical model of the normal distribution supplies a similar result. In the field of standard hardness 60 HRC from the investigated 10 hardness reference blocks only one hardness reference block was examined as good. If the relative range of the population of all measurements would be the basis for a decision, all investigated hardness reference blocks would have been bad. If exists a connection between the relative range and the standard deviation based on the same sample size, it would be possible to estimate the probability of an outage at a quality feature. From the present values a linear connection can be derived between the mean value of the relative range and the standard deviation by a sample size of n = 5. But the width of distribution of the values of the relative range does not allow to draw conclusions on the basis of one value. In limited areas of the hardness scale the results show a linear connection between the mean value from the relative range Rrel at a sample size of n = 5 and the standard deviation of the population shows picture 4.3. The random character of the values of the relative range becomes clear in the pictures from frequency distributions of 3 different hardness reference blocks all from the field of 60 HRC in picture 4.4 to 4.7.

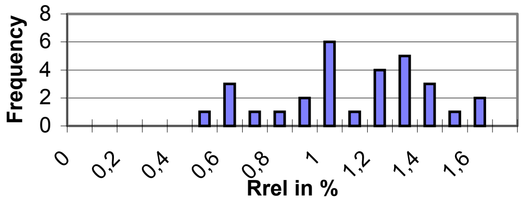
Picture 4.3: Correlation between the s and the mean value for Rrel at 60 HRC



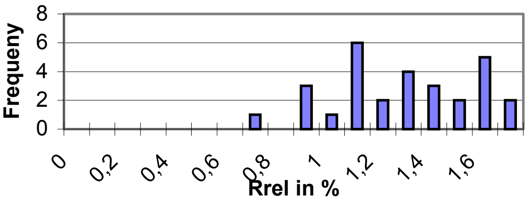
Picture 4.4: Frequency form Rrel PI 60-8 s = 0,096 HRC



Picture 4.5: Frequency form Rrel PI 60-6 s = 0,161 HRC



Picture 4.6: Frequency form Rrel PI 60-3 s = 0,213 HRC



The problems of the relative range as limit of the high quality will be clear from the frequency distributions. A significant statistical statement about a kind of distribution is not possible from the present measured values. The width of distribution of the relative range is by the best hardness reference blocks nearly as large as the limit in the standard [4]. This lets to appear that this rating is unsuitable as a criterion for a high quality feature of the hardness reference blocks. Another problem is that on the basis of 5 values an outlier value could not be clearly identified. Consequently, the 5 measured values will be the base for the calculation of the relative range. The experimental results show that it is possible that almost every hardness reference block is tested as good or as bad on the limit of the high quality feature.

4.4 Variation standard deviation

For the estimation of the variability of the standard deviations, from the random samples with the same number of indentation standard deviations will be computed. Concerning these standard deviations will be computed the mean value and the standard deviation.

Results: In table 4.3 are listed the mean values of standard deviation and the standard deviation of the standard deviation.

Table 4.3 Summary of results of the standard deviation. (All values in HRC)

Pl-No.	µ	σ	$\bar{x}_s$ n=25	s_s n=25	$\bar{x}_s$ n=20	s_s n=20	$\bar{x}_s$ n=15	s_s n=15	$\bar{x}_s$ n=10	s_s n=10	$\bar{x}_s$ n=5	s_s n=5
Pl 60-1	60,24	0,120	0,110	0,023	0,106	0,020	0,108	0,021	0,105	0,029	0,103	0,040
Pl 60-2	59,73	0,126	0,114	0,018	0,113	0,016	0,114	0,019	0,110	0,022	0,110	0,034
Pl 60-3	59,46	0,213	0,208	0,029	0,215	0,024	0,207	0,039	0,209	0,037	0,212	0,053
Pl 60-4	61,20	0,112	0,099	0,024	0,096	0,024	0,098	0,025	0,098	0,029	0,095	0,034
Pl 60-5	61,57	0,134	0,129	0,022	0,114	0,016	0,122	0,034	0,122	0,035	0,113	0,060
Pl 60-6	59,60	0,192	0,185	0,027	0,191	0,025	0,184	0,039	0,122	0,035	0,113	0,060
Pl 60-7	58,23	0,853	0,828	0,249	0,862	0,221	0,839	0,241	0,854	0,244	0,901	0,264
Pl 60-8	58,88	0,096	0,087	0,012	0,090	0,014	0,085	0,020	0,084	0,027	0,080	0,039
Pl 60-9	59,28	0,307	0,299	0,064	0,302	0,064	0,303	0,060	0,308	0,065	0,320	0,083
Pl 60-10	59,58	0,204	0,204	0,030	0,204	0,035	0,204	0,034	0,204	0,039	0,203	0,058

No significant differences exist between the mean value of the standard deviation of the different sample sizes among one another and of standard deviation of the population. If significant differences between these values exist, it will be an indication that they represent different populations (see section 4.1). Compared to the width of the distribution of the standard deviation of the standard deviations at a sample size of n = 5 in table 4.3 the width of the distribution for the standard deviation of the relative range table 4.2 is higher by a factor of 2.

5. Summary

The experimental results show the problems of the relative range and a sample size of n = 5 indentations as the high-quality feature of the calibrating for hardness reference blocks.

The better rating for the quality feature will be the standard deviation or the coefficient of variation. From the statistical point of view it will be recommended to increase the number of the calibrating indentations to at least n=10 or better to n=15. The limits of the high-quality feature for the different hardness tests and hardness scales should be elaborated for the next revision of the standards.

6. Literature

[1] EA Group “Mechanical Measurements” (1/99) Final Draft - Uncertainty in Hardness Measurement -
[2] ISO 6506: (10/99); Metallic materials Brinell hardness test part 1 - 3
[3] ISO 6507: (1/98); Metallic materials Vickers hardness test part 1 - 3
[4] ISO 6508: (10/99); Metallic materials Rockwell hardness test part 1 - 3