The Influence of Operators and Applied Load on Micro-Hardness of the Standard Block

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Abstract: The aim of the submitted work is to study the influence of operator on measured micro-hardness if the applied loads are ranging from 0.09807 N to 0.9807 N. The ISE effect, i.e. the influence of the load on the micro-hardness is expected. Whereas standard reference block with defined specified hardness and its uncertainty was used as a specimen, individual measurement involved indirect calibration of a tester. Five operators have measured in five trials the diagonals of the same indentations. The measurement was evaluated by Meyer's index n, ANOVA – two factor analysis, Post Hoc Analysis, Total Dispersion Zone SM%, Cgm index and Measurement Systems Analysis. The participating operators have a statistically significant effect on the value and type of the ISE effect.

Keywords: Micro-hardness; operator; load; ISE

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1 Introduction

Indentation hardness testing is a convenient mean of investigating the mechanical properties of a small volume of materials. The principle of Vickers micro-hardness method is identical to macro-hardness test, except for considerably smaller loads (or test forces). It is frequently used for determination of hardness of small items or thin layers and identification of individual phases in metallography.

The advantage of Vickers test is the hardness independence (defined) on the applied load, because indentations with various diagonals are geometrically similar. In contrast to the (macro)hardness, it is well known that the micro-hardness of solids depends on the load. This phenomenon is known as the indentation size effect (below ISE).

When a very low load is used, the measured micro-hardness is usually high; with an increase in test load, the measured micro-hardness decreases. Such a phenomenon is referred to as "normal" ISE [1, 2]. It may be caused by the testing equipment. For example, the experimental errors resulting from the measurement of indentation diagonals as a result of the limitations of the resolution of the objective lens, inadequate measurement capability of small areas of indentations and determination of the applied load belongs in this group [2, 3, 4]. Other potential causes are related to the intrinsic structural factors of measured material (work hardening during indentation; load to initiate plastic deformation or elastic resistance) [1, 3, 4]. In addition, the effect of indenter/specimen friction resistance coupled with elastic resistance of the specimen friction could be significant. Lubrication weakens the ISE [5, 6].

In contrast to the above “normal” ISE, a reverse type of ISE (inverse ISE or RISE), where the apparent micro-hardness increases with increasing applied test load, is also known. In the literature, there are many examples, which reveal that, the “normal” ISE occurs in brittle materials including glass while the reverse ISE essentially takes place in materials in which plastic deformation is predominant [3].

$$P = Ad^n \tag{1}$$

The constant “n” - Meyer’s index or work hardening coefficient is used as a measure of ISE. It is the slope and coefficient A is the y-intercept of the linear line in a straight line graph of ln d (the diagonal length) versus ln P (applied load). When n = 2, the micro-hardness is expected to be independent of the applied load and is given by Kick’s law. However, $n < 2$ indicated “normal” ISE behavior. Reverse ISE occurs if $n > 2$ [3].

When evaluating the of Meyer’s index “n” and subsequently the presence and type of ISE (“normal” or reverse) with the same micro-hardness tester on the same specimen, variance of results (even with the occurrence of both types ISE on the same sample) was observed. The variance was also observed when comparing results of several operators in conditions of repeatability [7] and when one operator repeated the measurements [8, 9]. Uneven loading rate applied by individual operators as a result of the manual control on the used type of micro-hardness tester is one of the possible sources of variance. To eliminate this factor, the each of the operators measured the diagonals of the same prearranged indentations. The influence of operators was evaluated by Meyer’s index “n”, two factor Analysis of Variance (ANOVA), Post Hoc Tests, Total Dispersion Zone S_M%, index C_{gm} and Measurement Systems Analysis (MSA).

2 Experimental Details

Micro-hardness was measured by tester Hanemann; type Mod D32 fitted to microscope Neophot-32. A standard reference block (or certified reference material CRM) for indirect calibration with specified hardness H_c = 195 HV0.05 and standard uncertainty u_{CRM} = 4.0 HV0.05 was the specimen. The tester was calibrated in the course of measurement according to standard [10] regarding the results obtained at load P = 0.4903 N (50 g).

The indentations were carried out on a surface polished to a mirror finish by applying loads P between 0.09807 N (10 g) and 0.9807 N (100 g) with a 0.09807 N step size. The load duration time was 15 seconds and loading rate 0.15 N s^{-1} (15 g s^{-1}). The indentation velocity of indenter in the reference block was 1 μm s^{-1} in average. The result was ten indentations. Their diagonals measured five operators (A – E). An operator measured the diagonals five times in random order. The ambient temperature was between 20.3°C and 21.9°C. The magnification of the optical device of micro-hardness tester was 375 ×.

The number of outliers and the normality were determined for files involving all values measured by one operator (n = 50 indentations). The outliers were detected by Grubbs' test (significance level $\alpha = 0.05$). Their presence would indicate measurement process suffering from special disturbances and out of statistical control. The normality was detected by Anderson – Darling test (Quantum XL software). It was confirmed only for results of operator D. The results of operator A satisfy the conditions for three parameter gamma distribution (normal distribution is rejected), B and C satisfy the two parameter logistic distribution (but normal distribution is not fully rejected) and E satisfy the two parameter Weibull distribution (also with possible normal distribution).

The number of the outliers, average micro-hardness value of 50 indentations (HV), micro-hardness HV0.05, repeatability r_{rel}, the maximum error of tester E_{rel} and relative expanded uncertainty of calibration U_{rel} for individual operators are in Table 1.

Table 1

The number of outliers, micro-hardness - average value of 50 indentation (HV), micro-hardness HV0.05, repeatability r_{rel}, the maximum error of tester E_{rel}, relative expanded uncertainty of calibration U_{rel}, Meyer's index "n" and constant A

operator No.	outliers	HV	HV0.05	$r_{rel0.05}$ (%)	$E_{rel0.05}$ (%)	$U_{rel0.05}$ (%)	"n"	A
A	3	299	284	22.1	45.8	72.5	1.6100	5.7179
B	2	195	188	6.7	-3.5	11.3	1.9749	6.8317
C	0	196	199	6.9	2.2	9.4	1.9841	6.8800
D	1	194	204	6.0	4.6	12.1	2.0891	7.2718
E	0	206	213	4.1	9.3	15.3	2.1475	7.5642

The box – plot of the values of micro-hardness, measured by individual operators are in Figure 1. The results of the operator A are significantly different from other. The difference between values micro-hardness measured by operator A and other operators is visible in Figure 2, which illustrates the influence of the load on the micro-hardness. The results of operator A are the most significantly affected.

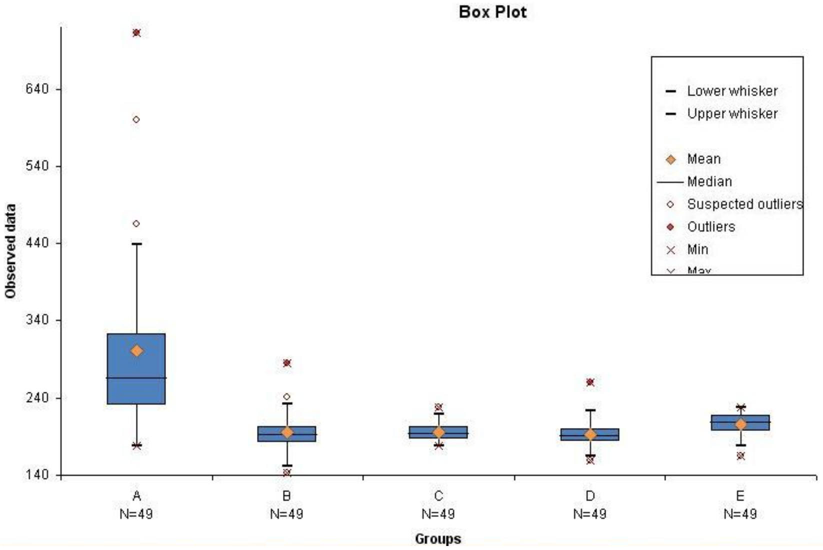

Figure 1

Box plot: the values of micro-hardness

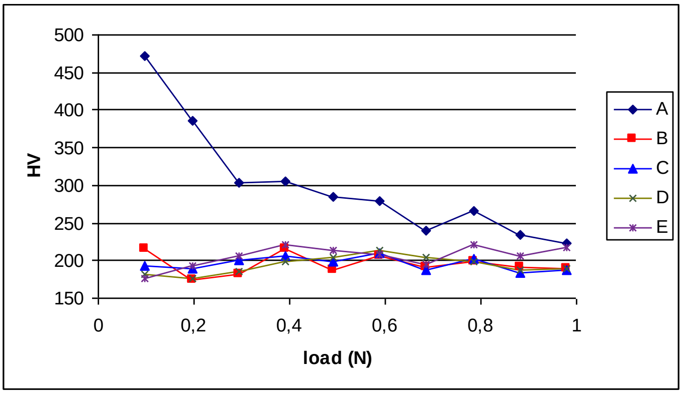

Figure 2

The influence of load on the average values of micro-hardness

The results of calibration were used for calculation of relative expanded (k = 2) uncertainty U_{rel} of the hardness values, Figure 3, according to standard [10]. Required repeatability (maximum r_{rel} = 9%) and the maximum permissible error (maximum E_{rel} = 10%) does not meet only operator A. Because the standard allows a maximum value U_{rel} = 10%, only operator C meets the requirement. As can be seen in Figure 3, U_{rel} significantly increases with decreasing of load for operator B and partially for operator A.

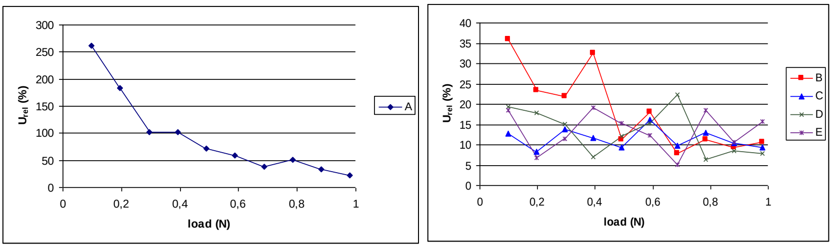

Figure 3

The values of uncertainty U_{rel}

As for the sources of uncertainty, the standard uncertainty of the reference block remains constant for all measurements and loads. The maximum dispersion of the diagonals length usually occurs at the low loads and is reduced as the applied load increases. The ambiguity in the measurement of small indentation areas, particularly when pile-up or sink-in effects are present, can lead to over- or underestimation of the indentation area [11]. The experimental error related to the size of the indentation is the significant at low loads, the most important for the determination of Meyer's index.

3 Meyer's Index

As for Meyer's index "n", the specimen has „normal" ISE typical for brittle materials ("n" = 1.61, Table 1, for example n = 1.46-1.90 for blast furnace slag) according to operator A, behaves close to Kick's law according to operators B, C and D or has moderate reverse ISE according to operator D. Because the material, and the tester are always the same, stated differences are the result of differences in quality of the measurement of diagonals, carried out by individual operators.

4 Two Factor Analysis of Variance (ANOVA)

Analysis of Variance (ANOVA) is a statistical method often used in designed experiments (DOE), to analyze variable data from multiple groups in order to compare means and analyze sources of variation.

Two factor ANOVA with replication (five times repeated measurement of the diagonals) was used for evaluation of statistical significance of the operator (factor 1) and applied load (factor 2) on the measured value of micro-hardness. The operator ($p = 1.73\ E^{-28}$) and load ($p = 0.013244$) have both statistically significant effect. The interaction between the operator and the load ($p = 1.75\ E^{-7}$) was found. It can be defined as a combined effect or outcome resulting from two or more variables that are significant. Operator differences depend on the applied load.

5 Post Hoc Tests

Tukey's Honestly Significant Differences Test (HSD) is a Post Hoc Test, meaning that it is performed after an ANOVA test. This means that to maintain the integrity; a statistician should not perform Tukey's HSD test unless he/she has first performed an ANOVA analysis.

The purpose of Tukey's HSD test is to determine which groups in the sample differ. While ANOVA can tell the researcher whether groups in the sample differ, it cannot tell the researcher which groups differ. That is if the results of ANOVA are positive in the sense that they state there is a significant difference among the groups, the obvious question becomes: Which groups in this sample differ significantly? It is not likely that all groups differ when compared to each other, only that a handful have significant differences. Tukey's HSD can clarify to the researcher which groups among the sample in specific have significant differences [12, 13, 14].

Files are equal to the extent; $n_1 = n_2 = ... = n_r$

The test for individual loads was carried out for

$$\frac{r(r-1)}{2} = \frac{5(5-1)}{2} = 10 \qquad (2)$$

pairs of files; the number of operators r = 5. Any difference $\left|HV_i - HV_j\right|$ (where i, j = A, B,…E, the 3rd and the last rows of Table 2) was compared with the critical value (quantile q_γ, the 2nd row in Table 2):

$$q_\gamma(r; n-r)\sqrt{\frac{MSE}{n_i}} = q_{0.95}(5; 20)\sqrt{\frac{MSA}{5}} \qquad (3)$$

Where n = 25 is the sum measured values of all 5 operators; $n_i = 5$ is number of repeated measurements (trials) by one operator, $q_{0.95}$ (5;20) = 4.23.

$$MSE = \frac{SSE}{n-r} \qquad (4)$$

$$SSE = \sum_{i=1}^{\gamma}(n_i - 1)s_{\Delta i}^2 \qquad (5)$$

$s_{\Delta i}$ = standard deviation of 5 repeated measurements by one operator at particular load.

If the value of $\left|HV_i - HV_j\right|$ is greater than the critical value (quantile q_γ), statistically significant difference between the two operators under consideration was demonstrated. As can be seen in Table 2, a statistically significant difference was observed between the operator A and other operators under load below 0.294 N. At higher loads, the differences were not detected.

Table 2

The results of Post Hoc Tests

load (N)	0.0981	0.196	0.294	0.392	0.490	0.588	0.686	0.784	0.883	0.981
q_γ	238	171	130	126	99	98	89	87	62	59
A-B	256	211	122	90	96	72	48	68	44	34
A-C	278	195	103	99	85	70	52	64	50	36
A-D	291	210	118	107	80	65	35	68	47	35
A-E	296	192	99	85	71	72	44	45	28	7
B-C	22	15	19	10	11	2	4	4	6	2
B-D	35	1	4	18	16	7	13	0	3	1
B-E	5	18	20	23	9	7	8	23	19	28
C-D	12	14	15	8	5	5	16	4	3	1
C-E	18	4	4	15	14	2	8	19	22	29
D-E	5	18	20	23	9	7	8	23	19	28

6 Total Dispersion Zone

The aim of the Total Dispersion Zone S_M % is to define if operators can achieve the same values of measurement using the same measuring equipment. It is necessary to calculate the average values HV_A, HV_B..HV_E (Table 1) and to calculate their standard deviations $s_{\Delta A}$, $s_{\Delta B}$ …$s_{\Delta E}$ for 5 repeated measurements of a particular operator at particular load [15, 16, 17].

Total scatter area S_M will be calculated as follows:

- average standard deviation of the measuring device (Table 3)

$$\bar{s}_\Delta = \frac{s_{\Delta A}+s_{\Delta B}+s_{\Delta C}+s_{\Delta D}+s_{\Delta E}}{5} \tag{6}$$

$$\bar{s} = \frac{\bar{s}_\Delta}{\sqrt{2}} \tag{7}$$

Table 3

Output data for calculation by total dispersion zone method

Load (N)	0.0981	0.196	0.294	0.392	0.490	0.588	0.686	0.784	0.883	0.981
$s_{\Delta A}$	226.86	161.72	87.18	84.76	49.89	27.99	27.04	26.17	23.53	12.91
$s_{\Delta B}$	46.25	22.19	27.31	40.21	11.76	20.84	7.44	16.06	10.62	12.52
$s_{\Delta C}$	18.89	2.61	17.50	6.18	10.27	13.94	7.68	15.21	4.70	6.34
$s_{\Delta D}$	19.75	10.65	17.38	4.36	11.04	5.19	32.29	3.35	3.04	1.40
$s_{\Delta E}$	10.91	4.99	6.03	5.54	6.95	7.08	2.71	3.96	2.57	1.73
s_ν	126.27	90.88	50.45	43.44	38.24	31.27	20.90	29.05	20.63	17.60

The sign tolerance T = 39 HV, the same for all test loads, was calculated pursuant to maximal permissible error (10% of 195 HV 0.05) according to standard [10]. S_M % values up to 20% are "good", the difference between the operators is negligible. Values above 30% indicate a statistically significant difference between operators and are unacceptable.

standard deviation s_v (Table 3) is the standard deviation of 5 average values HV_A, HV_B,…, HV_E

- total scatter zone of the measuring device S_M:

$$S_M = 6\sqrt{\bar{s}^2 + s_v^2} \tag{8}$$

$$S_M\% = \frac{S_M}{T} \cdot 100\% \tag{9}$$

The value of S_M% for particular loads are in Figure 4. As can be seen, it is "good" for all applied loads and its value improves with increasing load. The difference between values, measured by operators participating in the test appears to be negligible in the full range. Is likely that the total dispersion zone method is less sensitive than the methods above.

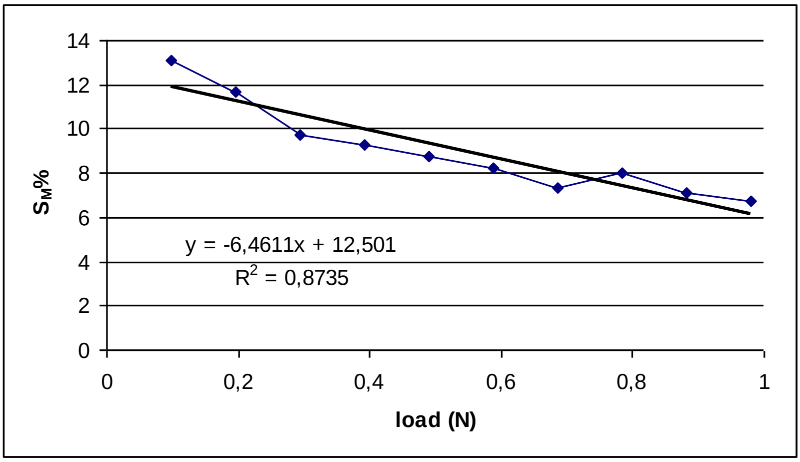

Figure 4

The values of S_M% for particular loads

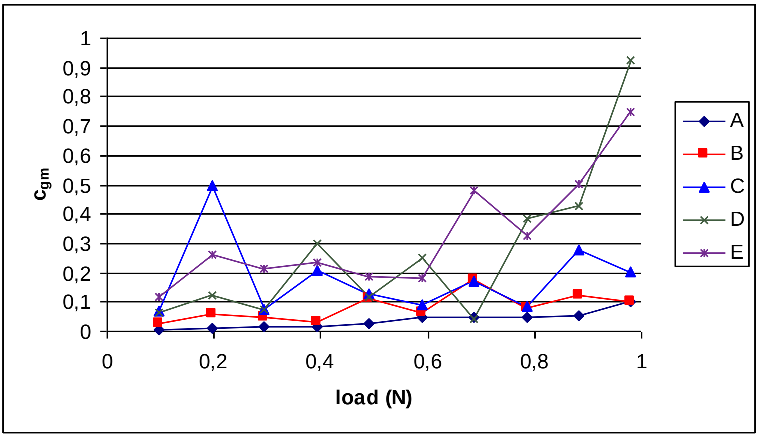

Figure 5
The values of index C_{gm}

7 Index C_{gm}

As described above, the calibration of the micro-hardness tester was carried out simultaneously with measurement. The functionality of the tester can be expressed by index C_{gm}.

$$C_{gm} = \frac{0.2 \cdot T}{6 \cdot s_{\Delta}} \tag{10}$$

Where: $s_{\Delta A}, s_{\Delta B} \ldots s_{\Delta E}$ is standard deviation (Table 3)

T - sign tolerance (39 HV).

The value of the index has to be more than 1.33. If the index $C_{gm} < 1.33$; afterwards will be proposed corrective measures. It will eliminate the cause of the detected non-compliance or other unwanted situations (changes in the surrounding temperature, adjust production equipment, collection of products, used material, a human factor…). As can be seen in Figure 5 (C_{gm}), the values of indices are insufficient with some improvement in increasing load (operators D and E).

8 Measurement Systems Analysis (MSA)

The Measurement Systems Analysis (MSA) was originally designed for the engineering industry. It is not standardized yet but is recommended in the reference manuals for the automotive industry. It helps to conform with ISO/TS 16 949:2009 [18] requirements, as well as AIAG standards. MSA is an experimental and mathematical method of determining how much the variation within the measurement process contributes to the overall process variability. If the analyzed measurement system (consists of measurement equipment, parts, environment, method, appraisers…) is capable, it is likely that the measurement process, taking place in it is capable, as well.

GRR (gauge repeatability and reproducibility) is one of MSA methods. It is an approach that will provide an estimate of both repeatability and reproducibility for a measurement system. This approach will allow the measurement system's variation to be decomposed into two separate components, repeatability and reproducibility [19].

Table 4

The indices of MSA analysis

ndc	%EV	%AV	%PV	%GRR
1.0	24.02	67.79	8.19	91.81

The number of distinct categories ("ndc", based on Wheeler's discrimination ratio) is connected with the resolution of equipment. It indicates the number of various categories, which can be distinguished by the measurement systems. It is the number of non-overlay 97% confidence intervals, which cover the range of expected variability of product. The ndc ≥ 5 for capable processes, the processes with ndc between 2-5 may be conditionally used for rough estimations. The values of ndc and other indices of MSA are in Table 4.

%EV index represents cumulative influence of measurement equipment, used measuring method and environmental conditions on the variability. It is a function of the average range of trials of all appraisers. %AV index represents the influence of operators on the variability. It is a function of the maximum average operator difference. High value of the index signalizes the differences in the work of operators. %PV index is a function of the range of micro-hardness values of the loads. It expresses the sensitivity of the measurement system on the difference between the loads and indirectly defines the suitability of equipment for specific measurement. %PV above 99% suggests extremely accurate, above 90% suitable, above 70% satisfactory and above 50% inaccurate equipment. Used equipment is inaccurate, and impact of the test load is small because %PV is only 8.19%. %GRR index represents the process capability in practice. For acceptable measurement system %GRR < 10%, the system with %GRR > 30% is considered not acceptable. Used system of measurement is not capable, especially due to the difference between operators.

9 Discussion

Temperature is one of the most significant influence quantities in metrology. The influence of temperature on the measured values of micro-hardness and consequently on ISE effect could be statistically significant [9, 20]. Vickers test method allows calibration in relatively broad interval of temperatures (23°C ± 5°C) [10]. The ambient temperature of the laboratory varied between 20.3°C and 21.9°C during the experiment. The influence of temperature in the said range on the mechanical properties of the reference block, the tester (thermal expansivity) or operator (personal sense of comfort) is practically negligible.

Indirect calibration of micro-hardness testers is not routinely practiced process, unlike the (macro)hardness testers. The largest source of error is probably the manual measurement of the indentation. It is affected by several factors including the operator's subjective decision in determining the indentation edge as well as operator fatigue and eye strain due to long time needed for each measurement. Optical image analysis systems have been used for some time in hardness testing and are considered adequately to estimate a measurement made by the human eye [21].

Two opposite trends affect the evaluation of the load influence on measured micro-hardness value. The low-load indentations are the most significant for confirmation of ISE occurrence. The length of diagonals measured on these indentations are most affected by uncertainty at the same time (note the extremely high uncertainty for small load for the operator A). The uncertainty of the length measuring system of tester u_{ms} and standard uncertainty of hardness testing machine, when measuring reference block u_H, are the most significant sources of uncertainty [11, 22, 23].

Small dimensions of diagonals and indentations with irregular shape are measured with difficulty. Small difference in reading has a significant effect on the value of micro-hardness and makes possible influence of individuality and the skill of operator. Unsatisfactory calibration results could be improved by greater magnification (with demands of the quality of metallographic specimen), selection of operators (their competence, including education, preparation and experience), higher quality of the reference block (with low uncertainty), strict observance of operating instructions (standardized methods), the conditions of the environment [24]. It is possible that the high value of uncertainty of calibration is a result of low capability (high value of Gauge Repeatability and Reproducibility index %GRR) [19].

Computerized methods (for example software ImageJ, Impor or TechDig. 1.1.b) used for treating of pictures of indentations significantly facilitate and accelerate the measurement of diagonals without significant effect on the presence and type of ISE effect [25].

Conclusions

1) The operators affect character of Meyer's index "n".
2) The influence of the operator is statistically significant according to ANOVA, Post Hoc Tests (operator A) and MSA (high value of % AV).
3) On the contrary, the method of Total Dispersion Zone did not confirm the influence of operators. This method is not difficult to calculate, on the other hand, and it seems not sufficiently sensitive.
4) Analyzed process is not capable according to high value of % GRR and low value of C_{gm} indices, primarily as a result of operators.

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