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THE DEPENDENCE OF MICROHARDNESS MEASUREMENT SYSTEM CAPABILITY ON TIME

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ABSTRACT:
Measurement systems analysis and control charts of micro hardness and capability indices were used for evaluation of long-term capability of micro hardness HV0.02 measurement process. The measurement process of copper micro hardness is not capable. Analysis of the control charts indicate that the measurement process capability is stable since there are no obvious special causes effects visible.
KEYWORDS:
Micro hardness, copper, long-term capability

1. INTRODUCTIONS

Micro hardness tests are used with loads lighter than 200 g, which typically creates indentation with the diagonal on the order of 50 μm. The Vickers test is carried out in a similar manner to the Vickers macro indentation tests with the same pyramid. Due to their specificity, micro hardness testing can be used to observe changes in hardness on the microscopic scale. Unfortunately, micro hardness values vary with load and work-hardening effects of materials. Additionally, it is difficult to standardize micro hardness measurements.

The capability of a process is a measure of its ability to satisfy customer's requirements. Process capability studies are carried out to compare customer's requirements with process performance and to identify the stakes-in-the-ground for process monitoring.

Repeated measurements of micro hardness [1], but also hardness [2, 3] process capability indicated time variability of capability indices. The aim of submitted work is to evaluate micro hardness HV0.02 measurement process capability by analysis of its system, as well as long-term variability of system capability. The long-term variability of the measurement system will also be reviewed using graphical analyses over time.

2. EXPERIMENTAL

The semiconductor grade copper Cu - K3A – 534 EG, manufacturer VUK Panenské Břežany, Czech republic, was used as experimental material. The copper was delivered as a cylinder ϕ 40 mm. The surface for metallographic analysis was prepared by standard way by grinding through a series of gradually finer silicon carbide water-cooled papers. The sequence was 220, 240… and 3000 grit

(ANSI/CAMI grit size scale). Finally, it was mechanically polished with Al_2O_3, moistened with water and cleaned with ultrasonic cleaning equipment. Polished surface was etched with 4 g $FeCl_3$ - 30 cm^3 HCl - 1000 cm^3 CH_3OH. The material has coarse - grained structure with grain diameter 8 – 15 mm. The samples for micro hardness analysis No. 1 – 4 were taken from the grain No. 1, the samples No. 5 – 9 from the grain No. 2 and sample 10 was part of the grain No. 3. The dimensions of samples were 3 × 5 mm with thickness 6 mm. Before micro hardness measurement the samples were mechanically polished as well as before etching.

The optical microscope NEOPHOT 32 with micro hardness tester Hanemann, type Mod D32 were used as measurement equipment. The smallest graduation on the scale of the equipment's measurement system equals 0.3058 μm. The micro hardness was measured according to standard STN EN ISO 6507 – 1 with load 20 g and loading time 10 s. The linearity requirement for the tester was satisfied between loads of 10 and 50 g.

The number of samples (parts) and trials (repeated measurements) depends upon the significance of the characteristic being measured an upon the confidence level required in the estimate of measurement system variation. As with any statistical technique, the larger the sample size, the less the sampling variation and the resultant risk will be present. As a rule, 10 samples, 3 trials (repeated measurements on each sample) and 2 appraisers are used for tests. If possible, the appraisers who normally use the measurement equipment should be included in the study [4].

Discrimination (readability or resolution) is the minimum amount of change from a reference value that an instrument can detect and faithfully indicate. The measure of this ability is typically the value of the smallest graduation on the scale of the equipment's measurement system. A general rule of thumb is that the measuring instrument discrimination ought to be at least one-tenth the process variation. If the measurement system lacks discrimination (sensitivity or effective resolution), it may not be an appropriate system to identify the process variation.

This requirement was not satisfied, because the value of the smallest tester graduation was 2.61 HV 0.02 and average standard deviation (SD) for all micro hardness values was 8.66 HV 0.02.

The measurement was carried out by the two (A and B) same skilled appraisers. Each of them carried out 3 trials on each sample. The measurements were made in a random order to ensure that any drift or changes that could occur will be spread randomly throughout the measurement.

Grubbs' test (with significance level α = 0,05 %) detected no outliers. The statistical outliers would indicate, that the process is suffering from special disturbances and is out of statistical control. Ideally, the causes of outiers should be eliminated and new data is obtained [5].

The standard methods of MSA assume normal probability distribution. In fact, there are measurement systems that are not normally distributed. When this happens and normality is assumed, the MSA method may overestimate the measurement system error. Therefore, before use, the data should be checked to confirm that its distribution is approximately normal. The simplest check is probability plotting, which give indications of unusual and non-normal distributions.

The normality was evaluated by normal probability plot, using software Freeware Process Capability Calculator by Symphony technologies. The normality of all samples**,** measured by particular appraisers was confirmed.

The quality - capability of analyzed process is defined by the statistical properties of multiple measurements obtained from a measurement system operating under stable conditions. The measurement system with a large amount of variation may not be appropriate for use in the analyzing a manufacturing process because the variation of measurement system may mask the variation in the manufacturing process.

The GRR method - combined estimate of measurement system repeatability and reproducibility, described in [4] with significance level 1 % and confidence level 1 % (5,15 σ) was used for capability evaluation. Periodic GRR studies make it easy to establish and monitor the performance of the equipment. A GRR study can quickly establish the short-time performance of the equipment, including appraiser influence. The method will allow the measurement system's variation to be decomposed into two separate components, reproducibility and repeatability, but can not express their interaction.

Table 1. The values of HV0.02

Stage No.	1	2	3	4	5	6	7	8	9	10	AVERAGE
A	86	94	93	91	94	92	87,8	91,3	86,5	87,6	90,32
B	87	96	95	91	92	91	88,3	91	85	86,6	90,29
A+B	87	95	94	91	93	91	88	91,2	85,8	87,1	90,31

As well as %GRR value, determining the process capability, partial indices %EV, %AV and %PV were evaluated. For evaluating a long-term variability of capability indices, the microhardness tests were repeated 10 times in a week intervals (stages). The results were evaluated by control charts.

Measurement system performance is a long-term estimate of measurement system variation (e.g. long-term control chart method).

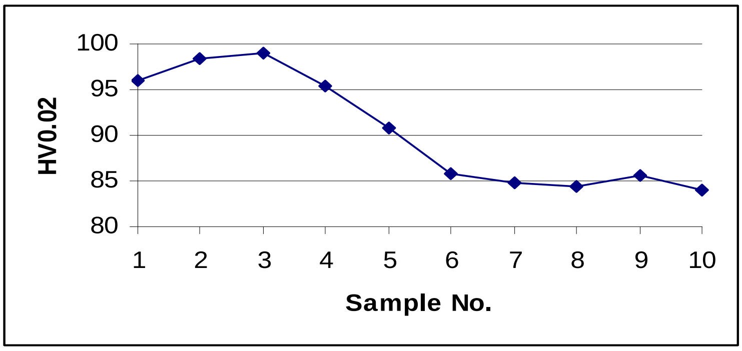

FIGURE 1. THE AVERAGE MICROHARDNESS OF SAMPLES.

3. RESULTS

The measurement system ought to be under statistical control before capability is assessed. This means that under repeatable conditions, the variation in the measurement system should be due to stochastic causes only not due to determinable causes.

The range control chart is used to determine whether the process is under control. If all ranges are in control, all appraisers are doing the same job. If one appraiser is out of control, his method differs from the others.

If all appraisers have some out of control ranges, the measurement system is sensitive to appraiser technique and needs improvement to obtain useful data. With respect to the table 2 (%R), the condition of system statistical control was not fulfilled, except for the 2th and the 8th – 10th stages. Only one point, measured by appraiser B, was out of control limits in these cases.

The area within the control limits represents the measurement sensitivity („noise“). Since the sample group used in the study represents the process variation, approximately one half or more of the averages should fall outside the control limits. If the data show this pattern, then the measurement system should be adequate to detect part-to-part variation and the measurement system can be provide useful information for analyzing and controlling the process. If less than a half of them falls outside the control limits then either the measurement system lacks adequate effective resolution or the sample does not represent the expected process variation. With respect to the table 2 (%X), this condition, except for the 6th stage, was satisfied.

Table 2.: Statistical control of measuremet system.

stage	1	2	3	4	5	6	7	8	9	10
%R	5 (B)	0	5 (B)	5 (B)	5 (B)	5 (B)	5 (B)	0	0	0
%X	80	60	80	55	70	35	55	85	50	90

The criteria as to whether a measurement system's capability is satisfactory depend on the rate of the manufacturing production process variability that is „consumer“ by measurement system variation. %GRR<10 % is generally considered to be an acceptable measurement system, %GRR > 30 % is considered to be not acceptable - every effort should be made to improve the measurement system.

The charts with sample size 1 were used for evaluating of statistical control of capability indices. There are times when, as in this case, it is inappropriate to consider samples with size greater than 1. In this case the ranges are calculated by taking the difference between one value and the next. Effectively, each sample size for range is 2. The plotted values are the individual values of capability indices; the central line (CL) is the mean of all indices of the same type. Note, that in this particular chart the range values are not independent.

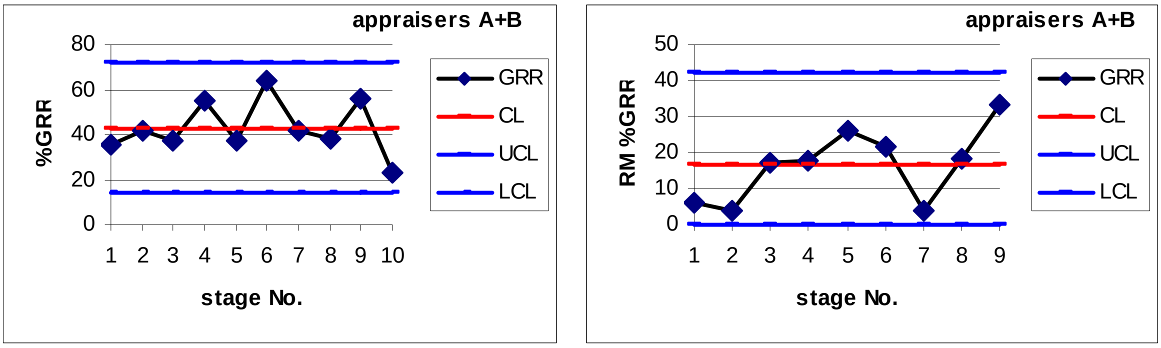

FIGURE 2. CONTROL CHARTS OF %GRR.

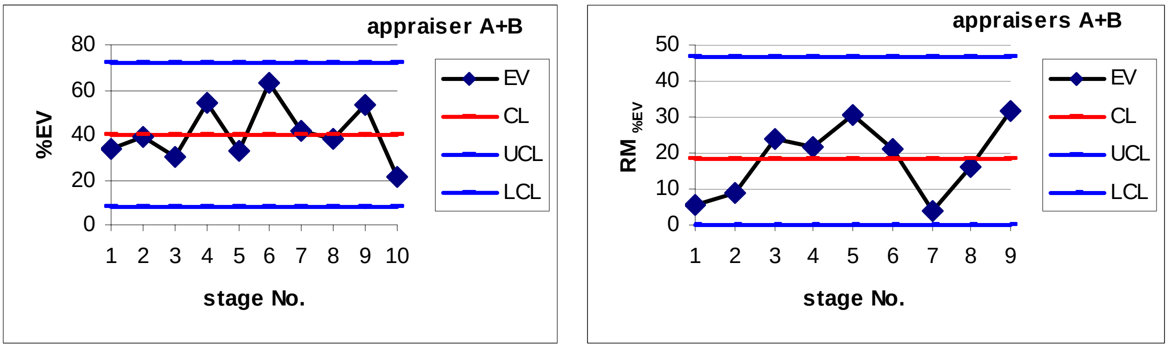

FIGURE 3. CONTROL CHARTS OF %EV.

%GRR index refers to contribution of measurement instrument to the variability. Its value represents the process capability. Good %GRR results show variations of 10 % or less. This translates directly into lower uncertainties.

Analyzed system was not capable in all stages beside the 10th stage, as it was conditionally capable (%GRR = 21.7 %). It is possible, that non – capability is typical for micro hardness, but also hardness measurement [6, 7]. However, it is difficult to achieve only 10 % variation in hardness testers. Dead-weight testers typically active results of 15 to 20 %. Older testers in poor condition give much worse results [8].

With respect to the fig. 2, from long-term viewpoint, capability for this measurement is stable.

Consistency is the difference in the variation of measurements taken over time. It may be viewed as repeatability over time. Repeatability is the inherent variation or capability of the equipment itself. Repeatability is commonly referred to as equipment variation (EV), although this is misleading. In fact, repeatability is common cause (random error) variation from successive trials under defined conditions of measurement.

Possible causes for repeatability are equipment, standard, method, appraisers lack of experience, environment, wrong gage for the application [4, p. 56]. In consideration of measurement according to standard method and standard measurement environment, %EV value is evaluation of micro hardness tester properties. With respect to the control chart on fig. 3, the metrology characteristics of tester, specified by %EV, are steady without trend to drift.

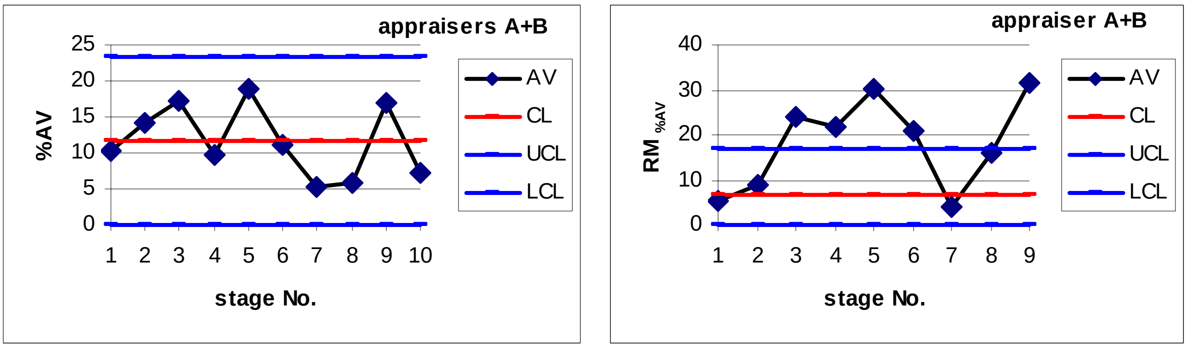

FIGURE 4. CONTROL CHARTS OF %AV.

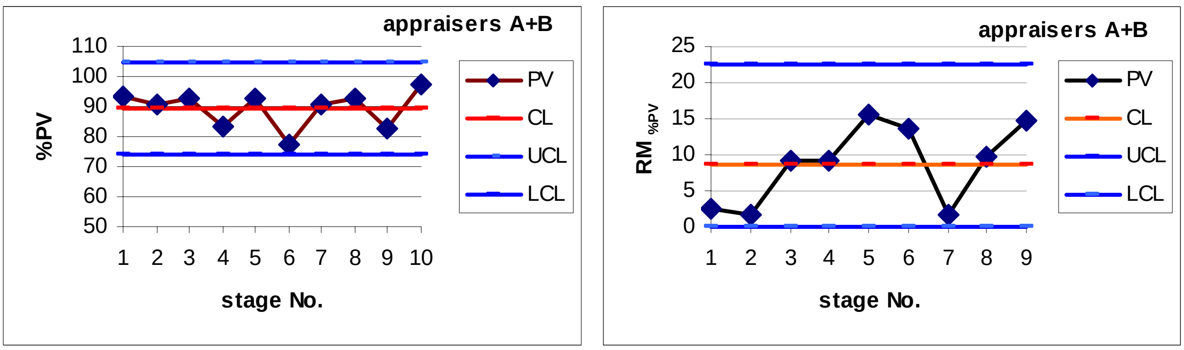

FIGURE 5. CONTROL CHARTS OF %PV.

%AV index represents the influence of appraisers on variability, for example their competence, perceptions, skills disciplines and vigilance. It is function of average values for individual appraisers. A control chart, based on long-term data, has been drawn in fig. 4. The range control chart indicates partial instability of appraiser influence.

%PV index is a function of range of average micro hardness of individual samples. It is sensitive to the variability influence between measured samples. Its value indirectly defines propriety of equipment used for measurement [9].

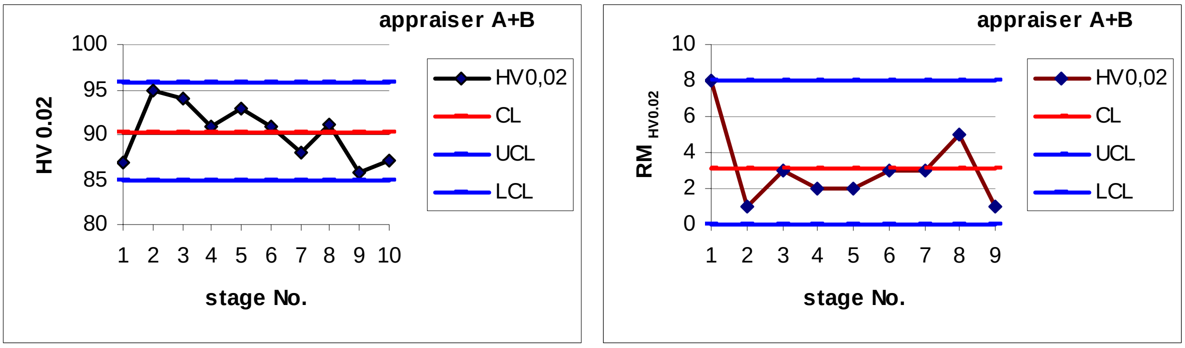

FIGURE 6. CONTROL CHARTS OF HV 0.02.

The long-term value of %PV index is stabile, used micro hardness tester is, on the base of %PV, satisfactory for measurement.

Micro hardness measurement, as such, is stable with "decreasing" of hardness in time and consideration of %EV index stability. It is obvious that the changes in micro hardness resulted rather from samples material changes than from drift of tester.

4. CONCLUSIONS

1. The measurement process of copper micro hardness is not capable.
2. Analysis of the control charts indicate that the measurement process capability is stable since there are no obvious special causes effects visible.

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