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QUALITY OF MICROHARDNESS MEASUREMENT PROCESS

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ABSTRACT

The capability of microhardness measurement process was assessed by MSA - Gear Repeatability and Reproducibility method. Experimental sample materials were: annealed ferrite, semiconductor-grade copper and aluminium. The measurement system consisted of microhardness tester Hanemann, type Mod D32 coupled with Neophot 32 optical microscope. Measurements were conducted by two appraisers. Obtained experimental data was evaluated by standard MSA process, using software Palstat CAQ, modul MSA. The assessment of the GRR capability indexes led to the conclusion that measurement process non-capability is typical for microhardness testing and it is also influenced by the type of tested material.

KEYWORDS: measurement capability, repeatability, reproducibility, microhardness, MSA, GRR

1. Introduction

The microhardness measurement method is frequently used for determination of hardness of small objects, thin layers, and phase identification in metallography. Its principle is identical to Vickers method, except for considerably smaller loads used. The determined value of microhardness depends on the load, accuracy of scale reading, and the indentation size. As for every test of mechanical properties, there is a natural requirement for reliability of measurement results, which is unthinkable without sufficient capability of measurement process. There are two ways of measurement process capability assessment. The first one is based on analysis of control processes according to uncertainty of measurement. The second is based on the measurement system analysis (MSA). In this case it is possible to use repeatability and reproducibility (GRR) or the variability (ANOVA). Due to simpler realization, the GRR method was used. In the Gear Repeatability and Reproducibility method, individual components of the measurement system (measuring device, operator, measured material...) are analyzed, and subsequently the capability of the whole system is numerically evaluated by indices.

Thus it is probable that the process of measurement taking place in capable system is also capable.

2. Experimental materials

Experiments were realized with three materials: iron, copper, and aluminium.

1. Pure iron - annealed ferrite was divided into 10 samples with size of 5×10×2 mm. Those were cast into epoxide resin Dentacryl. The grinding was done on SiC papers with water cooling on gradually decreasing grain size (according to ANSI/CAMI) 220 - 3000. Water suspension of Al_2O_3 was used for mechanical polishing. The sample was etched with 5% nital (5% HNO_3 in CH_3OH) for 10 seconds. The microstructure was uniform throughout the whole sample surface, consisting of isoaxial grains. The grain size was determined according to standard STN 42 0462 by grain counting method. Mean grain size 36.3 μm (standard deviation 2.58 μm) was determined from 30 analyses. Before microhardness measurement samples were grinded on No. 3000 paper and polished as stated above.

2. The semiconductor 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 in standard way by grinding through a series of finer and 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 equipmnent. Polished surface was etched with 4 g $FeCl_3$ - 30 cm^3 HCl - 1000 cm^3 CH_3OH.

The material had coarse - grained microstructure with grain diameter of 8 - 15 mm. The samples for microhardness 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 of 6 mm. Before microhardness measurement the samples were mechanically polished as well as before etching.

3. Electroconducting aluminium STN 42 4004 cast at 760 °C with cooling rate 2 $°Cs^{-1}$ was used as experimental material. Ten samples with size of 10×10×5 mm were taken from the casting. The sample surface was processed the same way as for iron samples. For microstructure development etching solution of 0.7 % HF in water was used. The semiproduct had a coarse microstructure with grain diameter gradually decreasing from 170 µm in sample No. 1 to 90 µm in sample No. 10.

3. Measurement equipment and method

The optic microscope NEOPHOT 32 with microhardness tester Hanemann, type Mod D32 were used as measurement equipment. The microhardness was measured according to standard STN EN ISO 6507 - 1 with load of 20 g and loading time of 10 s. The load was chosen so that the size of the imprint diagonal would not exceed 50 % of the grain size. The tester was up to standard of linearity between loads 10 - 50 g. Discrimination (readability or resolution) is the 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.

Table. 1. *Discrimination and standard deviation*

Material	Fe			Cu			Al		
Measurement	1	2	3	1	2	3	1	2	3
Discrimination	5.3	5.0	6.5	3.18	3.33	2.45	0,69	1.6	0.67
SD (HV0.02)	9.29	9.54	11.56	9.49	8.84	7.84	2.09	1.17	1.68

The measurement system is said to have sufficient sensitivity threshold if its resolution is small compared to the process variability. The discrimination and corresponding SD are listed in the Tab. 1. The discriminations for all measurements and materials were not sufficient.

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

For evaluating a term variability of capability indices, the microhardness tests were repeated 3 times on each material.

The results are in Tab. 2 and Fig. 1.

Table 2. *The hardness and standard deviations*

Microhardness HV0.02									
Material	Fe			Cu			Al		
Measurement	1	2	3	1	2	3	1	2	3
Appraiser A	135.3	135.8	140.3	99.9	97.0	95.6	34.9	34.8	32.7
Appraiser B	143.0	139.5	147.7	100.9	97.7	94.9	34.9	34.6	32.3
A and B	139.2	137.6	144.0	100.4	97.4	95.2	34.9	34.7	32.5
Standard deviation SD (HV 0.02)									
Material	Fe			Cu			Al		
Measurement	1	2	3	1	2	3	1	2	3
Appraiser A	8.13	9.59	8.43	9.62	8.77	8.06	2.39	1.36	1.8
Appriaser B	8.89	9.29	13.1	9.49	9.06	7.74	1.8	0.96	1.55
A and B	9.29	9.54	11.56	9.49	8.84	7.84	2.09	1.17	1.68

Grubbs' test (with significant level α = 0,05 %) detected no outliers. The statistical outliers indicate that the process is out of statistical control. Again ideally, the causes of outiers are eliminated and new data is obtained. Normal probability distribution is an assumption of the standard methods of MSA. 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.

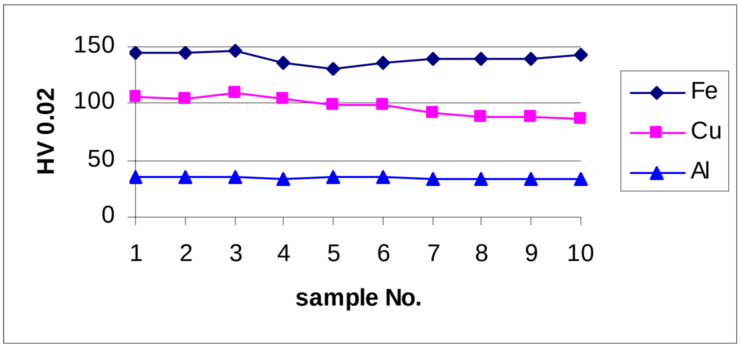

Fig. 1. *The hardness of individual samples.*

Therefore, before use, the data should be checked to confirm that its distribution is approximately normal.

The most simple check is probability plotting, which gives indications of unusual and non-normal distributions [1]. 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 GRR method - combined estimation of measurement system repeatability and reproducibility, described in [2] with confidence 99 % and coverageve 99 % (5,15 σ) was used for capability evaluation. A GRR study can quickly establish the short - time performance of a tester, including appraiser influence. The method will allow the measurement system's variation to be decomposed into two separate components, reproducibility and repeatability, but does not describe their interaction [4]

As well as %GRR value, determining the process capability, partial indices %EV, %AV and %PV were evaluated. Software Palstat CAQ, module MSA, was used for calculation.

4. 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 is due to common causes only. The range control chart is used to determine whether the process is under statistical control. If all ranges are in control, all appraisers are doing the same job. If one appraiser is out of control, his/her 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.

As shown in the Tab. 3, the condition of system statistical control was not satisfied, except for the second and the third measurement on the aluminium. All values outside control lines were measured equally by both appraisers.

Table 3. *Statistical control of measuremet system*

Range control chart (% $\overline{R}$ - % values outside control lines)									
Material	Fe			Cu			Al		
Measurement	1	2	3	1	2	3	1	2	3
Appraiser A	0	0	0	10	10	10	10	0	0
Appraiser B	10	10	10	0	0	10	10	0	0
A and B	5	5	5	5	5	10	10	0	0
Average control chart (% $\overline{X}$ - % values outside control lines)									
Material	Fe			Cu			Al		
Measurement	1	2	3	1	2	3	1	2	3
Appraiser A	10	30	10	60	70	80	20	10	10
Appriaser B	0	30	30	70	80	70	0	0	0
A and B	5	30	20	65	75	75	10	5	5

The resulting chart for average shows "usability" of the measurement system. The area within the control limits represents the measurement sensitivity ("noise"). Since the group of samples 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 shows this pattern, then the measurement system should be adequate to detect part - to - part variation and the measurement system can provide useful information for analyzing and controlling the process. If less than half 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, this condition was satisfied only for Cu measurements.

The number of distinct categories ("ndc" - number, based on Wheeler's discrimination ratio) should be more than, or equal to 5, but values between 2-5 may be conditionaly used for rough calculations.

As it can be seen from Fig. 2, low values of "ndc" bear witness of the low capability of evaluated process. The criteria as to whether a measurement system's capability is satisfactory depend on the rate of the manufacturing production process variability that is "consumed" by measurement system variation. This consumption is characterized by index %GRR.

The value of %GRR < 10 % is generally considered to be an acceptable measurement system, the value between 10 % and 30 % may be acceptable according to the importance of application and value > 30 % is considered to be not acceptable - every effort should be made to improve the measurement system.

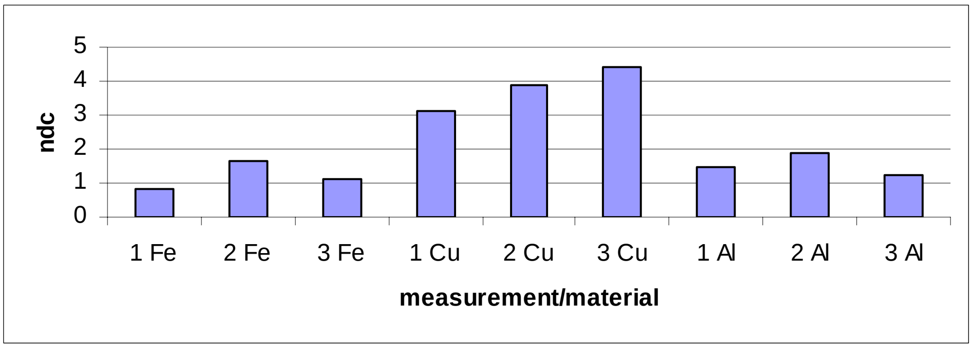

Fig. 2. *The values of "ndc".*

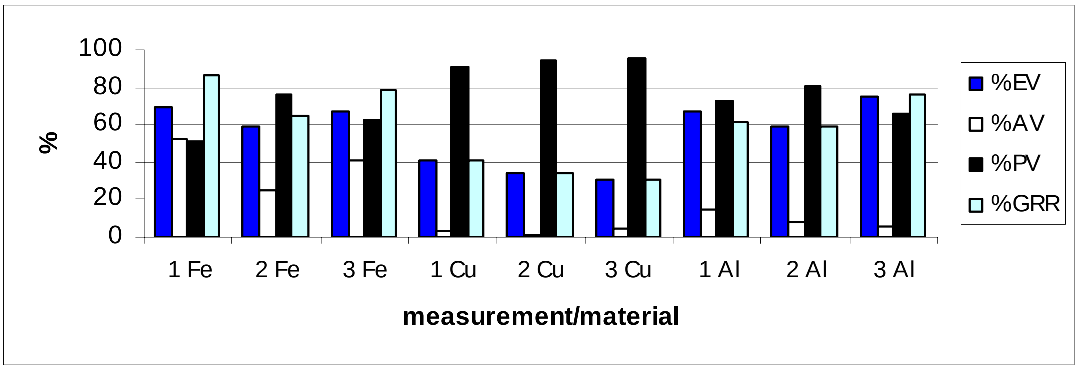

Fig. 3. *The capability indices.*

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 non - acceptable repeatability are equipment, standard, method, appraisers lack of experience, environment, wrong gage for the application.

Considering the same micrometer and standard measurement environment, %EV value depends on the relation between load and measured materials eventually getting the method under control by appraisers. The load is related to the imprint diagonal size, which should be comparable to the grain size. %AV index represents the influence of appraisers on variability, for example their competence, perceptions, skills discipline and vigilance. It is a function of average values of individual appraisers.

%PV index is function of range of average microhardnes of individual samples. It is sensitive to the variability influence among measured samples.

Its value indirectly defines propriety of used equipment for measurement.

The value of %PV above 99 % is for very accurate equipment, above 90 % for suitable, above 70 % for satisfactory and above 50% for inaccurate one. The equipment with value up to 50 % is unsuitable [3].

Analyzed process was non - capable for all materials and measurements. It is possible, that non - capability is typical for microhardness, but also hardness measurement [4,5].

However, it is difficult to achieve only 10 % variation in hardness testers. The dead - weight testers typically achieve results of 15 to 20 %. The older testers in poor condition give much worse results [6].

Fig. 3 shows that the value of capability indices depends more on material type than repeated measurements; considerably lower values of %GRR were obtained for copper (average 42,1 %) than for iron (average 76.5 %) and aluminium (average 77.9 %). Two-factor analysis of variance between groups (ANOVA) without repeating confirms this conclusion for indices %GRR, %EV and %EV.

The lower values for copper may have been positively influenced by lower ratio of imprint diagonal to grain size, i.e. lower uneven influence of the results by grain interfaces.

5. Conclusions

1. The microhardness measurement process is non-capable for all three materials.
2. Non-capability is typical for microhardness (and hardness) measurement.
3. Capability is influenced by the measured material.

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