STUDY ON THE CAPABILITY OF MATERIALS AS REFERENCE BLOCKS FOR THE MACRO RANGE OF INSTRUMENTED INDENTATION TEST

Christian Ullner[1] and Thomas Reich[2]
[1] Federal Institute for Materials Research and Testing (BAM), 12000 Berlin, Germany
[2] Fraunhofer Institute for Ceramic Technologies and Sintered Materials (FhG-IKTS), 01277 Dresden, Germany

Abstract

Part 3 of the ISO 14577 "Instrumented indentation test for hardness and materials parameters" (IIT) concerns the calibration of reference blocks. Besides the widely spread activities for supplying reference blocks in the nano and micro range it is needed to be taken into account the macro range too. The paper reports on test results on ceramic materials which are tailored for reference blocks. The specimens made of Si_3N_4, SiC, or Al_2O_3 ceramics are highly homogenous and exhibit an increased quality with a roughness $R_a \leq 0.005$ µm. They are successfully used for reference blocks of the traditional Vickers and Knoop hardness. In comparison, the capability of hardened steel which is used for traditional hardness techniques is studied.

Introduction

Hardness is a technological quantity. It is affected by the interaction of the material with the real geometry of the indenter. From this point of view the instrumented indentation test (IIT) needs reference blocks like the traditional hardness techniques although the metrology of the quantities measured for the test (force and displacement) is quite clear. Part 3 of the ISO 14577 [1] concerns the calibration of reference blocks. Essential work was done in [2] for the nano and micro range of IIT regarding the test of coatings. For the macro range some features are different. An advantage is to take the bulk materials of the traditional hardness techniques but the question arises if the conditions and restrictions given in the IIT standard are optimal for the macro range. The paper is aimed to introduce in using known reference blocks and validating ISO 14577 for the macro range.

Materials

Reference blocks are plate-like specimens made of materials with a highly homogeneous microstructure and prepared to a precise flatness, parallelism, and very small surface roughness. The hardness value of such a block is very reproducible. They are used for the indirect verification of hardness machines according to part 2 of [1]. The specified conditions of hardness blocks according to part 3 of [1] are gathered in Table 1 for the macro range.

Blocks of different hardness levels are needed to verify a hardness machine completely especially to check its proficiency over a large range. Table 2 gives some properties of the investigated ceramic materials. The three advanced ceramics are developed and produced by

FhG-IKTS with the aim of high homogeneity and best surface quality. They are offered as hardness reference blocks with calibration certificate [3]. For comparison, a non-ceramic block made by Buderus AG and the optical glass BK7 are involved in the study.

Table 1. Requirements to the hardness blocks according to ISO 14577 Part 3
(Explanation of the symbols H_M, H_{IT}, E_{IT} see Table 3)

Property	Requirement
Structure	homogeneous, stable, uniform
Thickness	$\geq$ 16 mm
Flatness	$\geq$ 5 µm in 50 mm
Parallelism	$\geq$ 10 µm in 50 mm
Test surface	free from scratches
Surface roughness	$\leq$ 15 nm (test surface), $\leq$ 0.8 µm (support face)
Maximum permissible coefficient of variation for H_M, H_{IT}, E_{IT}	2 %

Table 2. Some properties of the advanced ceramics used for hardness blocks

Class of advanced ceramics	Si_3N_4	SiC	Al_2O_3
Type of processing	Gas pressure sintered	Liquid phase sintered	Sub-micro structured
Density (g/cm^3)	3.24	$\geq$ 3.21	3.94
Porosity (%)	$\leq$ 0.5	$\cong$ 2	$\leq$ 1
Thoughness (MPa m $^{1/2}$)	5.2	3.5	3.8
Youngs modulus	300	420	375

Method

IIT has been done using the two screw driven testing machine Z005 with the 200 N hardness test head (Zwick AG). The speed of the cross head can be reduced down to a continuous drive of 0.05 µm/s. The resolution of force and displacement are 1 mN and 20 nm, respectively. The machine is equipped by a computer controlled x,y – table M-525.22 with a resolution of 33 nm (Physik Instrumente) and an optical microscope BX30 (Olympus).

According to part 3 of [1] both times for application and removal of test forces are 10 s. The duration of the test force is 10 s. The machine is displacement controlled. The approach velocity of the indenter when contacting the specimen is 1 µm/s. The zero point of the indentation depth is calculated by extrapolation of a polynomial 2nd degree from 0.005 N to 5% of the maximum indentation depth.

To separate the main sources of scatter (machine, material) a statistical variation analysis is performed. It is based on an equilateral triangular grid for placing the indents according to the study on Rockwell hardness reference blocks [4]. Figure 1 shows the location of the indentations on the specimen. Up to five hexagons (35 indentations) are generated at the specimen.
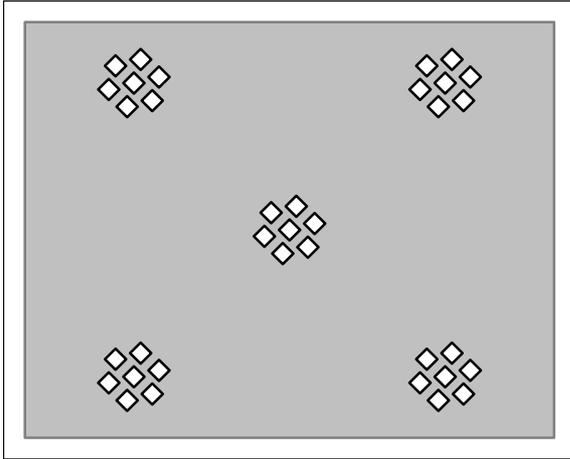
Figure 1. Location of the indentations

Uncertainty in measurement

The machine has been verified according to the part 2 of [1]. The force has been measured with force proving instrument of class 0.5 according to ISO 376. The machine is class 1 according to EN 10002 Part 2 from 2 N (F_{max}=10 N) or from 10 N (F_{max}=200 N). The machine compliance is measured using a specimen of steel X8CrMnN10-18. Since the indentation depth is greater than 6 μm the correction with the use of the indenter area function is not needed. However, it is taken into account for the estimation of the uncertainty in measurement. All information for the uncertainty estimation is collected in Table 3. Note it is distinguished between force application and force removal as well as between single values of force or displacement and slopes over a range of force or displacement. The uncertainty due to the extrapolation of the contact point is also estimated. The determination of the uncertainty in IIT for the macro range will be described in more detail in a future paper.

Table 3.   The sources and results of uncertainty in calculation of the Martens hardness, HM, the Martens hardness determined from the slope, HM_s, the indentation hardness, H_{IT}, and the indentation modulus, E_{IT}, from the measured indentation curve.

	Machine Force range Displacement range	Z005 200N head 4-10N >5μm	Z005 200N head 20-200N >5μm	According to ISO 14577 - 3
Force up	%	0.8	0.3	0.25
Force up and down	%	1.1	0.7	
Slope of force up	%	0.8	0.2	
Slope of force down	%	0.8	0.4	
Displacement up	μm	0.05	0.05	0.03
Displacement up and down	μm	0.15	0.15	
Slope of displacement up	%	0.04	0.04	0.5
Slope of displacement down	%	0.04	0.04	
Angle of the Vickers pyramid	°	0.14	0.14	0.3
Compliance of machine	μm/N	0.02	0.02	
Contact point h_0	μm	0.055	0.055	

Results and discussion

The results are collected in Table 4. The materials are presented in the order of decreasing scatter. The overall standard deviation represents the uniformity of the specimen over the whole test surface. The smaller values of the standard deviation within the hexagons indicate the experimental error in combination with the materials property in the micro range (roughness, microstructure).

Table 4. Results of the instrumented indentation test in the macro range

SiC; 10.24 N 28 indentations	HM N/mm²	HM$_s$ N/mm²	H$_{IT}$ N/mm²	E$_{IT}$ kN/mm²
Average over specimen	15538	14942	27124	375.0
Std.Dev. over specimen	371	373	811	9.3
Std.Dev. within hexagons	256	364	598	8.0
Expanded uncertainty	850	278	2076	29.2
Al$_2$O$_3$; 10.22 N 27 indentations	HM N/mm²	HM$_s$ N/mm²	H$_{IT}$ N/mm²	E$_{IT}$ kN/mm²
Average over specimen	14772	14965	25015	373.5
Std.Dev. over specimen	275	338	1000	15.3
Std.Dev. within hexagons	218	175	539	6.0
Expanded uncertainty	786	272	1860	28.5
Si$_3$N$_4$, 20.27 N 28 indentations	HM N/mm²	HM$_s$ N/mm²	H$_{IT}$ N/mm²	E$_{IT}$ kN/mm²
Average over specimen	11060	11282	18392	269.8
Std.Dev. over specimen	232	216	477	8.9
Std.Dev. within hexagons	130	118	304	3.4
Expanded uncertainty	357	94	836	21.2
Buderus 831HV10; 100.4 N 27 indentations	HM N/mm²	HM$_s$ N/mm²	H$_{IT}$ N/mm²	E$_{IT}$ kN/mm²
Average over specimen	6840	7085	9805	221.6
Std.Dev. over specimen	66	94	48	8.0
Std.Dev. within hexagons	22	44	36	1.6
Expanded uncertainty	88	46	173	30.6
BK7; 10.16 N 35 indentations	HM N/mm²	HM$_s$ N/mm²	H$_{IT}$ N/mm²	E$_{IT}$ kN/mm²
Average over specimen	4111	4206	7842	74.8
Std.Dev. over specimen	41	59	203	1.6
Std.Dev. within hexagons	30	23	103	0.7
Expanded uncertainty	69	70	150	1.6

The results on the metallic hardness blocks and on the optical glass BK7 show that a well-prepared commercial hardness machine could supply reference values in the macro range because in all cases of Table 4 the scatter caused by the materials is greater than the scatter caused by the machine. According to part 3 of [1] the coefficient of variation should not exceed 2 %. This requirement is fulfilled for the metallic hardness block and for BK7, partly for Si$_3$N$_4$. Cracking is starting in Si$_3$N$_4$ at the test force less than 20 N. The tendency to cracking is more pronounced for Al$_2$O$_3$ and SiC [5]. In this way the higher scatter within the hexagons

and over the whole test surface can be explained. However, it is important if cracking is generated before or after the hold period.

The expanded uncertainty in measurement given in Table 4 has been carefully calculated from the values in Table 3 using the equations of the standard. Since the uncertainty represents the range of the average the uncertainty can be smaller than the standard deviation. The higher uncertainty of HM in relation to the uncertainty of HM_s is caused by the uncertainty in the determination of the zero point. The expanded study of the uncertainty has indicated that the required precision of force, 0.25 %, is not needed, for example. On the other hand the uncertainty of the machine compliance is more important.

Conclusion

- The used testing machine has the potential to supply hardness blocks for the IIT in the macro range.
- The capability of materials for reference blocks in the macro range is comparable to the features regarding traditional hardness techniques.
- For certain cases the Martens hardness HM_s determined from the slope of the indentation curve is an appropriate parameter for characterizing hardness blocks in the macro range.
- In the range of higher forces the indentation modulus E_{IT} (partly indentation hardness H_{IT}) is not appropriate for characterizing hardness blocks.
- After an extended study on the uncertainty in IIT an optimisation of the ISO 14577 Part 3 will be useful for the macro range.

References

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Contact:
Dr. Christian Ullner
BAM – S.11
12200 Berlin, Germany
e-mail: christian.ullner@bam.de