INSTRUMENTED INDENTATION TEST FOR HARDNESS AND MATERIALS PARAMETER FROM MILLINEWTONS TO KILONEWTONS

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Abstract
The ISO/DIS 14577 Metallic materials - Instrumented indentation test for hardness and materials parameters – Part 1-3 (IIT) concerns test forces up to 30 kN. The paper reports on IIT at test forces ranging from 0.002 N to 1000 N on non – magnetizable steel X8 CrMnN 18-18 (1.386) with well polished surface using Vickers indenter and four hardness machines of different design (Nano Indenter XP, Fischerscope H 100, Zwick Z005 with Universal hardness head and a laboratory four-column set up materials testing machine) according to the standard. Using mostly identical test parameters the results of the different machines are almost in good agreement. Estimated small differences are caused by the different uncertainties of the used machines and by different methods of mathematical analysis of the detected raw data.

Introduction

The principle of IIT is described in the recently issued standard ISO/DIS 14577. It concerns continuous monitoring of the test force **F** and the relevant depth **h** of indentation during the penetration of an indenter of well known material and shape into the material tested. The test procedure can either be force or displacement controlled. The test result is a force/indentation depth curve. Because this curve includes the plastic and elastic response of the material tested, it my be used to derive a number of materials parameter describing the plastic and elastic behaviour of the material tested like Martens hardness, ***HM***, Martens hardness determined from the slope of increasing force/indentation depth curve, $\boldsymbol{HM_s}$, indentation hardness, $\boldsymbol{H_{IT}}$, indentation modulus, $\boldsymbol{E_{IT}}$, indentation creep, $\boldsymbol{C_{IT}}$, and the elastic part of indentation work, η_{IT}.
Martens hardness ***HM*** is determined from the force/indentation depth curve during increasing of test force, preferably after reaching and holding the specified maximum test force, and is defined as the test force ***F*** divided by the surface area $\boldsymbol{A_s(h)}$ of the indenter penetrating beyond the zero-point of the contact. The formula for the calculation of ***HM*** for a Vickers indenter is given in Equ. (1). This hardness definition includes both plastic and elastic deformation of the material tested.

$$HM = \frac{F}{A_s} \quad \text{with} \quad A_s = \frac{4 \times \sin\left(\frac{\alpha}{2}\right)}{\cos^2\left(\frac{\alpha}{2}\right)} \times h^2 \tag{1}$$

(α: angle between oppsite faces of vertex of the pyramid for Vickers indenter)

Today a variety of IIT instrument types is commercially available which have a maximum force ranging between10 mN and 2500 N. While a lot of studies has been published for the nano and micro range the application of IIT in the macro range above test forces of 2 N is relatively seldom. The aim of this study is the comparison of IIT machines working at test forces ranging from 2 mN to 1 kN. ***HM*** and $\boldsymbol{HM_s}$ were the values used for this comparison because for their calculation only the applied test force ***F*** and the surface area $\boldsymbol{A_s(h)}$ must be known. There is no need in further theoretical and analytical assumptions like for calculation of the stiffness at the beginning of unload, the contact depth or the contact area.

Calibration and corrections

All experiments have been done according to ISO/DIS 14577 using three commercially available testing instruments: Nano Indenter XP (MTS) Fischerscope H 100 (Fischer) and Zwick Z005 with Universal hardness head (Zwick AG) and one laboratory four-column set up materials testing machine (BAM). The test force, the displacement measuring devices and the machine compliance of all used testing machines were calibrated according to the standard. The main technical parameters of these instruments are given in ***Table 1***.

Table 1

	Nano Indenter XP	**Fischerscope H 100**	**Zwick Z005**	**Four-column set up**
Operation control mode	Force	Force	Displacement	Displacement
Maximum force	$5 \cdot 10^{-1}$ N	1 N	200 N	1000 N
Force resolution,	$75 \cdot 10^{-6}$ mN	$3\ 10^{-2}$ mN	1 mN	10 mN
Machine compliance	0.1 µm/N	Not explicit given	0.001 µm/N	0.002 µm/N
Displacement resolution	0,01 nm	1 nm	20 nm	50 nm

All indentation tests were carried out with Vickers indenters. For indentation depth < 6 µm the surface area $A_s(h)$ cannot be assumed to be that of an ideal shaped Vickers indenter because of the rounding at the tip. For the Nano Indenter XP and the Fischerscope H 100 the determination of the exact area function (Mathematical function relating the projected area $A_p(h)$ or the surface area $A_s(h)$ to the distance from the indenter tip) for the actually used indenter is necessary. The used area functions and methods of their determination are given in ***Table 2***. For the Zwick Z005 and the four-column set up the area function of an ideal shaped Vickers indenter was used because h > 6µm.

Table 2

Testing machine	**Type of area function**	**Method of determination**
Nano Indenter XP	$A_p = a_1 h^2 + a_2 h + a_3 h^{1/2} + a_4 h^{1/4} + a_5 h^{1/8} + a_6 h^{1/16}$	Indirect: Young's modulus of the reference material (Glass BK 7, E_{BK7}= 81,4 GPa) is assumed to be constant with indentation depth.
Fischerscope H 100	$A_S = 26{,}43 h^2 \left(1 + \frac{a_1}{h^{a_2}} + \frac{a_3}{h^2}\right)$	Indirect: Martens hardness of the reference material (Glass BK 7, HM_{BK7}= 4133 N/mm²) is assumed to be constant with indentation depth.

According to ISO/DIS 14577 the measured force/indentation depth data-sets must be corrected for the thermal drift, the machine compliance and the zero point (point of the first contact of the indenter with the test-piece surface).

The drift rate values determined during a hold period at 90 % unload were assumed to result only from temperature changes. The recommended procedure was that the holding time at 90 % unload is approximately the same as the time for application or removal of the test force. Because of the great variety of used instrument types, environments and software limitations, a uniform correction procedure of thermal drift could not be performed.

After correction for the thermal drift the data-sets were corrected for the compliance under the applied maximal test force of the machines itself.

For the Nano Indentor XP, the Fischerscope H100, and the Zwick Z005 the zero point was detected automatically during the indentation process by the software belonging to the instruments. The four-column set up recognises the surface if a preselected contact force is reached. The zero point correction is not done during the measurement but before analysing the results by the software belonging to this instrument.

Testing cycle

To make a comparison of the different testing machines possible, all indentation tests were performed with the same strain rate (1/h)(dh/dt) at maximum test force. The loading rate is proportional to the maximum force, which gives a constant (1/F)(dF/dt), so that the loading rates for all indentation tests can be calculated from the fixed strain rate at maximum test force. The same rate was used in the unloading segment.

When the hold period at maximum force is too short and the subsequent unloading rate is too slow, the material is still creeping under the indenter resulting in an initial increase of indentation depth upon unloading. Therefore, it is essential that the hold period at maximum force is sufficiently long to minimise time dependent deformation effects. The criterion was that the creep rate has decayed to a value where the depth increase in one minute is less than 1 % of the indentation depth.

Details of the realised testing cycles are given in ***Table 3***. Because of the great variety of used instrument types the definition of an identically uniform testing cycle was not possible.

Table 3

	Nano Indenter XP	**Fischerscope H 100**	**Zwick Z005**	**Four-column set up**
Maximum load, N	0,002; 0.005; 0,01; 0,02; 0,05; 0,1; 0,2; 0,5	0,01; 0,02; 0,05; 0,1; 0,2; 0,5; 1,0	1; 5; 10;20; 50;100; 200	100; 215; 464; 1000
Loading time, s	50	50	65 – 78	41 - 83
Holding time, s (at maximum force)	60	60	60	60
Unloading time, s	50	50	20 -35	55 - 90
Holding time, s (at 90 % unloading)	60	60	Not necessary	Not necessary
Strain rate, s^{-1} (at maximum force)	ca 0.01 at (?)	ca. 0.01	ca. 0.01	ca. 0.01

Specimen preparation

Indentation tests have been performed on specimens of non – magnetizable steel X8 CrMnN 18-18 (1.386) with a mechanically lapped or a well polished surface (R_a < 0,02 µm). It should be noted that the final surface roughness and also mechanical grinding and polishing of the surface may result in change of the residual stress state of the surface and consequently in change of the measured hardness parameters. Specimens of this steel were coated with a Chromium coating of 5 µm thickness in a sputter process. This specimens have been tested to demonstrate the influence of an additional surface coating on the measured hardness parameters. The high temperature (380°C) during the coating process may result in change of the properties of the surface near region and consequently in change of the measured hardness parameters. For a better comparison the not coated specimens were also thermal treated at the same conditions like the coated ones during the coating process.

Experimental results and discussion

After calibration and all corrections at each of the defined test forces ten measurements were made.

In Fig. 1 the average maximum indentation depths for all indentation tests on polished steel specimens as a function of the maximum test force are given. The calculated relative standard deviations for the maximum indentation depths (not indicated in Fig. 1) are between 1 % and 3 % for all testing machines.

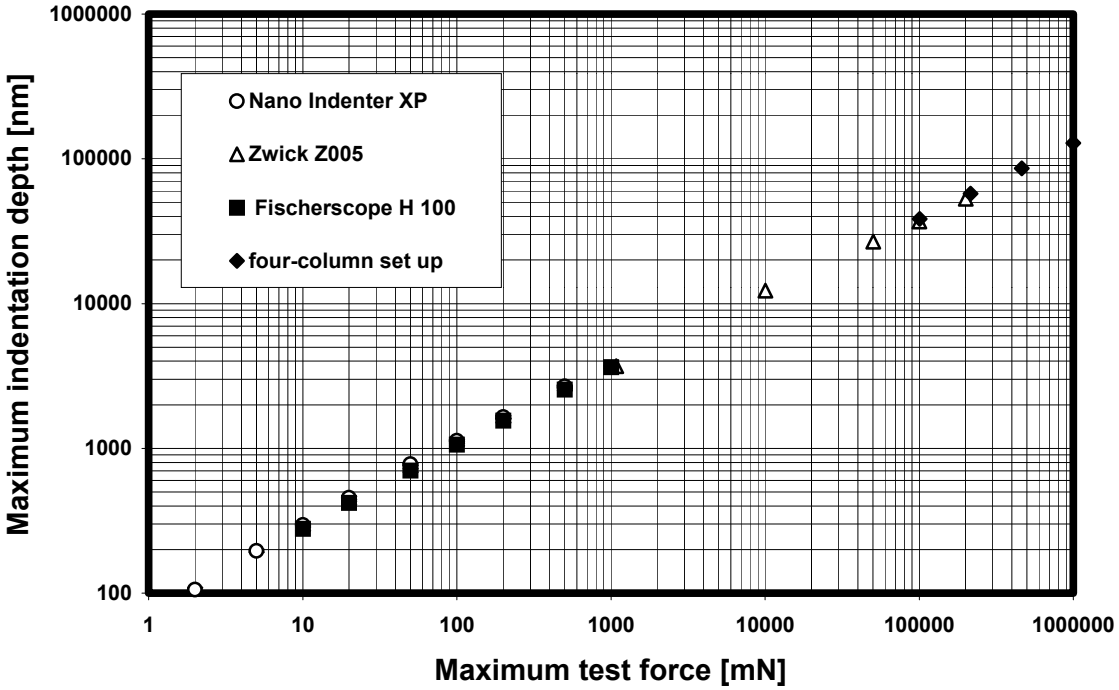

Fig. 1: Average maximum indentation depths as a function of the maximum test force for all indentation tests on polished steel specimens

For a first comparison indentation tests were performed on a glass BK7 reference block. The results for the Martens hardness ***HM*** are given in Fig. 2. For the Nano Indentor XP and the Fischerscope H 100 the values for ***HM*** are constant for test forces from 10 mN to 500 mN.
The constant difference in the ***HM*** values estimated with these two testing machines and also the decrease of ***HM*** values for smaller forces measured with the Nanoindenter XP can mainly be explained by the procedure of calculating A_s in the Nano Indentor XP software. A_s is calculated from A_p taken directly from the area function (see ***Table 2***), using the constant factor of 0.9269 given by the ratio A_p/A_s for an ideal Vickers indenter. There is no reason that this ratio is correct and constant with decreasing indention depth for the used real indenter. In the case of Fischerscope H 100 the A_s can be directly estimated from the area function. The reduced value for ***HM*** estimated with the Fischerscope H 100 at 1000 mN can be caused by a not correctly estimated area function at higher test forces.

The results for Martens hardness ***HM*** of all indentation tests on polished steel specimens are given in Fig. 3. In general a good agreement of the results estimated by all four testing machines over the force range from 2 mN to 1 kN was found. Like for the glass BK7 reference block also for the polished steel specimens a difference in the results estimated by the Fischerscope H100 and the Nano Indentor XP can be seen. The reason for this difference is the same like in the case of BK7. The increasing of ***HM*** values with decreasing indentation depth can be explained by a change of the residual stress state of the near surface region as result of mechanical grinding and polishing and also of thermal treatment.
The values for ***HM*** estimated with the Zwick Z005 and the four-column set up are not influenced by these changes.

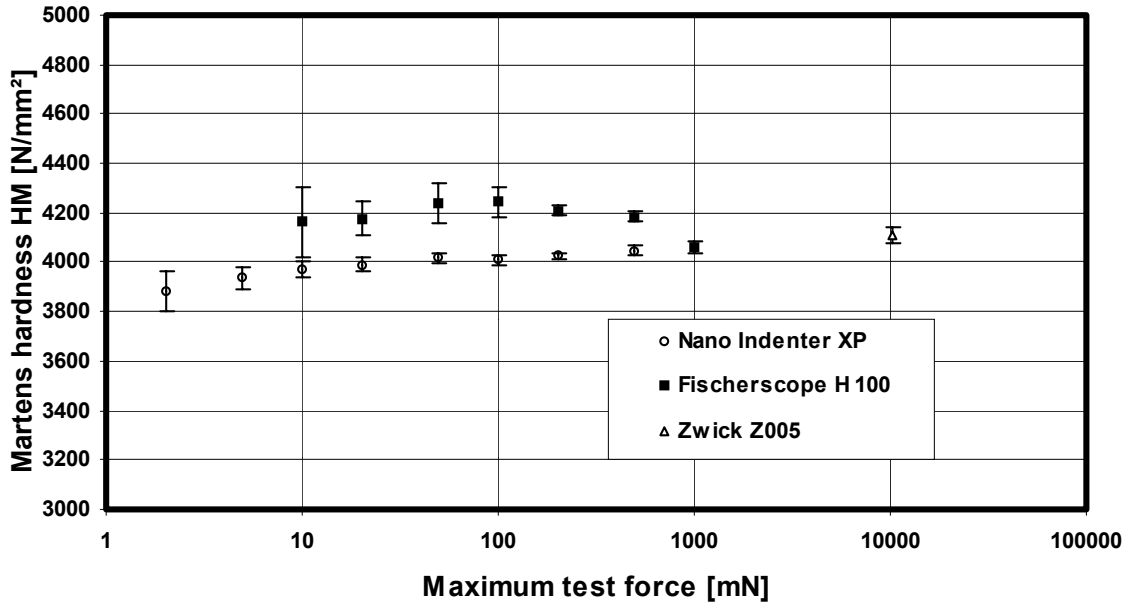

Fig. 2: Average Martens hardness as function of maximum test force for glass BK7 reference block

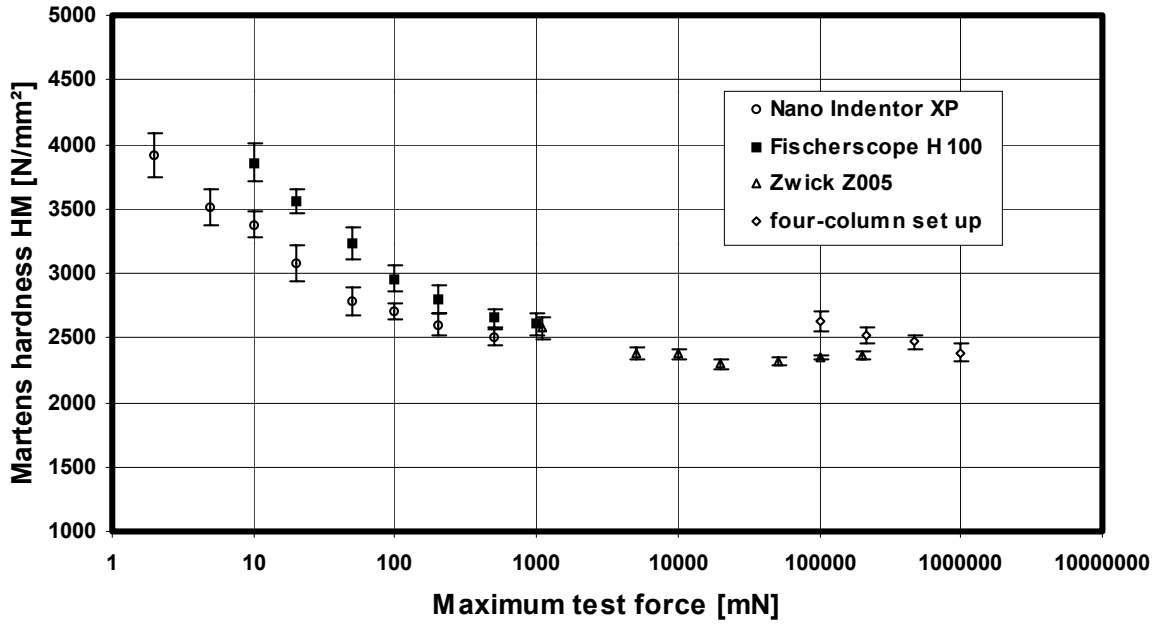

Fig. 3: Average Martens hardness as function of maximum test force for polished steel specimens

The results for Martens hardness ***HM*** of all indentation tests on Chromium coated polished steel specimens are given in Fig. 4. The values for ***HM*** estimated with the Zwick Z005 are only influenced by the Chromium coating for a test force of 1N. In general a good agreement of the results estimated by the Fischerscope H100 and the Nano Indentor XP can be seen. The Chromium coating leads to higher surface roughness because of random defects occurring during deposition like columnar growths, defects or droplets. This is the reason for the established increasing scatter in the results with decreasing indentation depth found for the Nano Indenter XP and the Fischerscope H 100.

Because of the limits of this short paper some details of the study are only given in the talk. The results of the estimation of Martens hardness determined from the slope of increasing force/indentation depth curve HM_s show in principal the same behaviour for all specimens investigated.

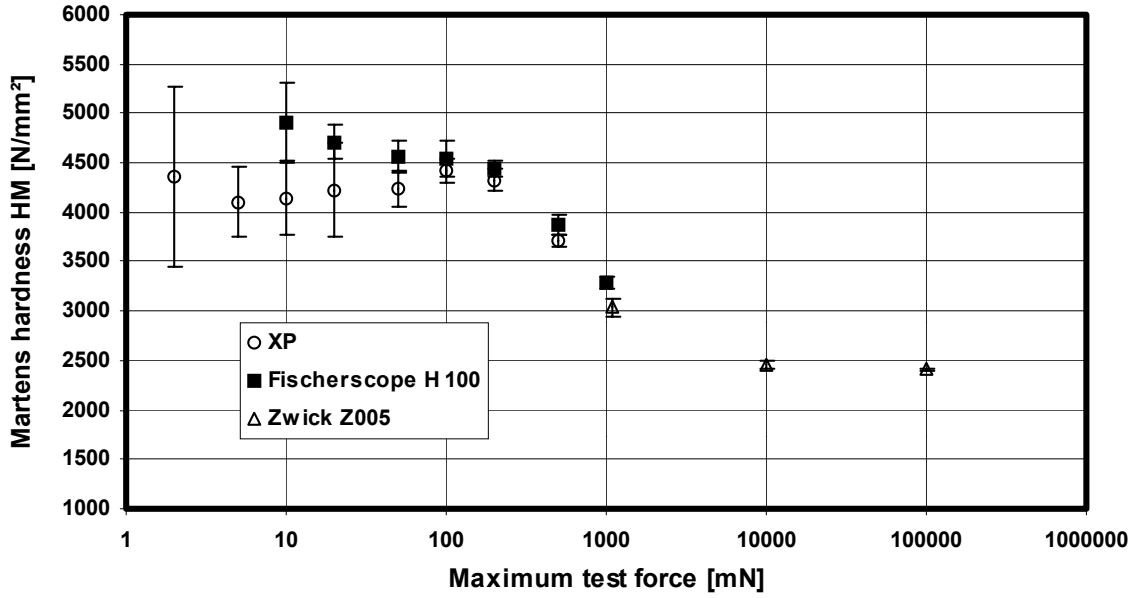

Fig 4: Martens hardness as function of maximum test force for polished steel specimens coated with 5 µm Chromium coating

The values estimated for the indentation modulus E_{IT} and the indentation hardness H_{IT} are not compared in this study because there is a large influence of the different used models for calculation (calculation of the stiffness at the beginning of unload, contact depth and contact area) and there was no way to evaluate the raw data in the same way by using the original software belonging the instruments. This can be done only by using the raw data and a software independent from the used machine.

Conclusions

If the indentation tests were performed according to the standard ISO/DIS 14577 the estimated values for Martens hardness ***HM*** arefound to be in a good agreement for all used testing machines used for the test forces from 2 mN to 1kN. Small differences are caused by different methods of calibration, by different mathematical analysis of the detected raw data and by different uncertainties of the used machines.

Specimens of steel with Chromium coating of 5 µm thickness allowed to demonstrate the advantage of using such a large range of test forces. In the nano and micro range (Nano Indenter XP and the Fischerscope H 100) the property of the coating can be studied while tests in the macro range (Zwick Z100, four-column set up) showed no influence of the coating on the determined parameter of the substrate. The macro range is also important for testing specimens with more realistic surface condition. The discussion of the reproducibility concerns the estimation of uncertainties caused by the different machines and the performance of a strongly identical mathematical analysis of the raw data recorded by the machines using mostly identical test parameters. This will be done in a further study.

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