HELLENIC SOCIETY OF NON DESTRUCTIVE TESTING
4th ICNDT of HSNT, Chania-Greece, 11-14 October 2007

National Society, Member of EFNDT and ICNDT
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CASE DEPTH DETERMINATION BY USING VICKERS MICRO - HARDNESS TEST METHOD AT TRSC / PPC SA

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ABSTRACT

The laboratory hardness tester supported by relative software equipped with a microscope giving magnification between 10 to 40x, applied the Vickers Test method on ferrous materials. The hardness difference between the core and the outer surface of a material gives the case depth. Thus the case depth can be easily determined by measuring the diagonals of the (micro) indentations comparing it with the basic metal.

The aim of this paper is to present our experience on determining the case depth in mechanical equipment and machinery tools of carburised parts providing information on its mechanical properties.

Tests Research & Standards Centre (TRSC) is the testing organization of Public Power Corporation (PPC) S.A. involved with Destructive and NDT including the Metallurgical laboratory.

Keywords*: Carburization, Case Depth, Hardness, Microscope, Non Destructive Testing (NDT), Vickers, Hellenic Accreditation System, National Accreditation Body of Greece (NAB).*

1. Introduction - The TRSC

The Metallurgical Laboratory is a subsection of the Mechanical and Metallurgical Section which is in charge of NDT for Tests Research & Standards Centre (TRSC). TRSC is the Public Power Corporation (PPC) S.A. inspection organization and employees personnel of high expertise and uses state of the art machinery and equipment.

Among our duties is to test the PPC equipment and parts that have been purchased, to carry out the Electric Power Stations NDT and provide the relative consulting engineering. In addition the aim of this work is to inform the engineering society and especially NDT engineers about the case depth examination that the laboratory initiates.

The TRSC headquarters are for more than 25 years now located in Kantza, Pallini, a suburb on the north east that is almost 15 kms far from the centre of Athens. Moreover it is easily accessible via exit 17 'Kantza' of the new major road named 'Attiki Odos' and also via exit Y6 'Glyka Nera' from the branch connecting the Rafina with Ymitos. Besides that it is easily accessed by using the new Athens metro and the 'proastiakos' suburban railways. Free and plenty of parking is available for the visitor's and the staff's cars as well. A map below navigates the visitors driving on ATTIKI ODOS.

Fig. 1 Area map. Source: http://www.attiki-odos.gr

2. Literature survey

2.1. Hardness has typically been defined as the resistance of a material to permanent penetration by another one that is harder. This can be achieved by using an intender with hardness similar to that of the diamond. In combination with the applied force on it and the velocity of the application and finally the total time of the penetration applied [9]. The most popular hardness methods are being used the inventor's names and are described below:

The results obtained when performing Rockwell, Vickers, Knoop and Brinell tests are determined after the test force has been removed. Thus the consequence of the elastic deformation occurred during the test has been ignored.

2.1.1. The Vickers (HV) test was developed in England is 1925 and was formally known as the Diamond Pyramid Hardness (DPH) test. The method described in detail in ELOT EN ISO 6507.01 [10].

2.1.2. Knoop (HK) hardness was developed by at the National Bureau of Standards (now NIST) in 1939 (USA). The indenter used is a rhombic-based pyramidal diamond that produces an elongated diamond shaped indent.

2.1.3. Stanley P. Rockwell (HRB-C) invented the Rockwell hardness test. He was a metallurgist for a large ball bearing company and he wanted a fast non-destructive way to determine if the heat treatment process they were doing on the bearing races was successful.

2.1.4. Dr. J. A. Brinell (HB) invented the Brinell test in Sweden in 1900. The oldest of the hardness test methods in common use today, the Brinell test is frequently used to determine the hardness of forgings and castings that have a grain structure too course for Rockwell or Vickers testing (http://www.instron.co.uk).

2.2. Case hardening improves both the wear resistance and the fatigue strength of parts under dynamic and/or thermal stresses. The characteristics of case hardening are primarily determined by surface hardness, the effective hardness depth, and the depth profile of the residual stress.
Case hardness depth - or the thickness of the hardened layer - is an essential quality attribute of the case hardening process. Until recently, the quality of the hardening process could only be

evaluated by random sampling using destructive testing methods, which were expensive and time consuming. In addition there is an NDT ultrasonic method that is less expensive. Finally the micro hardness test is suggested method as the most promising thus presented in details in this paper.

2.2.1. The perpendicular distance from the surface is defined as:

2.2.1.1. Effective case depth of a hardened case is the depth up to a further point, for which a specified level of hardness is maintained.
2.2.1.2. Total case depth of the hardened or unhardened case is the depth to a point where no differences in chemical or physical properties of the case and core can no longer be distinguished [7] [16].

2.3. Case hardening methods

2.3.1. Carburized cases
2.3.2. Cyanided cases
2.3.3. Carbonitrided cases
2.3.4. Nitrided cases
2.3.5. Flame or induction hardened cases [16].

The International Standard that defines the case-hardened depth and specifies methods for the determination of this depth in steels is the ISO 2639:2002. The applications applied are:

- Carburized and carbonitrided cases

- Heat treated parts to the final hardness meeting a hardness of less than 450 HV1 at a distance of three times the case hardened depth from the surface [8].

3. Case depth determination

3.1. Methods employed for determining the depth of the case are the following:

3.1.1. Chemical Methods

This method is generally applicable only for carburized cases, but may be used for cyanided or carbonitrided cases. The procedure consists of determining the carbon content at various depths below the surface of a test sample. This method is considered as the most accurate for measuring the total case depth.

3.1.2. Mechanical Methods
In general this method is considered to be one of the most useful and accurate of the case depth measuring methods. It is the preferred method for determination for effective case depth and can be used effectively on all types of hardness cases.

The following table suggests the hardness levels for various nominal carbon levels.

TABLE 1. CARBON CONTENT. Source: [16][4]

Carbon content % C	Effective Case Depth Hardness	
	HRC	HV
0.28 – 0.32	35	345
0.33 – 0.42	40	392
0.43 – 0.52	45	446
0.53 and over	50	513

3.1.3. Visual Methods
This method in general applies visual procedure with or without the aid of magnification for reading the depth of case procedure by any of the various processes. Samples may be prepared by combinations of fracturing, cutting, grinding and polishing methods. Etching with a suitable reagent is normally required to produce the contrast between the case and core. Nital (concentrated nitric acid in alcohol) of various strengths is frequently used for this purpose.

3.1.3.1. Macroscopic
Magnification methods for determination of case depth measurement are recommended for routine process control, primary because of the short time

required for determinations, and the minimum of specialised equipment and trained personnel needed. They have the added advantage of being applicable to the measurement of all types of cases.

3.1.3.2. Microscopic

Microscopic methods are generally for laboratory determination and require a complete metallographic polish and an etch suitable for the material and the process. Usually the magnification is 100x, in our case the used magnification is 40x which can be extra magnified by 2 or 3 times.

Microscopic method may be used for laboratory determination of total case and effective case depths in the hardened condition.

The distinction between macro and micro is based on the test forces in relation to the indentation depth. Attention is drawn to the fact that the micro range has an upper limit given by the force of 2N and a lower depth limit given by the indentation of 0.2 μm.
Finally it is important to bear in mind that the method of case depth determination should be carefully selected on the basis of specific requirements in terms of economy.

4. Vickers Microhardness test measurements principles

The continuous monitoring of the force and the depth of the indentation can permit the determination of hardness and materials properties. The Diamond indenter is an orthogonal pyramid with a square base and in an inner angle α=136º between the opposite faces at the vertex. An indenter probe made of a material harder than the specimen may have the following shapes:

The below picture (Fig. 2) depicts the indenter on the left, illustrating the angles of the pyramidal form probe. On the right is obvious the depth ‘h’ of the indentation. Finally on the bottom depicts the indentation and the diagonal ‘D’.

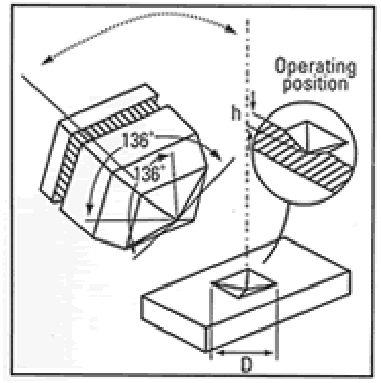

Figure 2: Schematic presentation of Vickers probe and its indentation
Source: http://www.instron.co.uk

4.1. Terms and definitions

The used software by the microhardness equipment calculates the Vickers Hardness using the following formula:

$$HV = 1854.4\ P/d^2$$

Where: P: the test load (gf)
d: the average diagonal length (μm)

The Vickers hardness value is calculated with the formula **HV = 0.102·F/A,** where F is the applied force and A is the contact surface area A of the resultant indentation after the indenter has been withdrawn. The area is computed from the mean diagonal d of the indentation (d = mean average of the two measured diagonals d1 and d2) [17].

5. Testing equipment

The equipment that participates in Case Depth Determination playing the central role is the micro hardness tester that is equipped with a microscope as well. The Hardness tester made in Denmark by Struers A/S, is Computer-controlled and produces HV, Knoop and Brinell methods of testing materials. The year of manufacture is 2003 and it is the unique in Greece. The software program used is STRUERS HMS32 [18] and the test parameters that could be modified are:

- Test mode: Vickers, Knoop and Brinell
- The load (Test force) : from 10 p (98.12 mN) up to 2.00 Kp (19.6 N)
- The lens magnification: 40x and 10x
- Time: sec

- The mode of data series: Line, Zigzag, Polyline, xy-List, manual and Teach_ln
- Calculations are according to: DIN 50190/1, /2, /3 and Hardness profile [5].

The used mode of indentations is the zigzag path which enables the indentations to be made with a small point-to-point separation along the x-axis though without contravening the 2.5 D rule [3],[5],[8]. The 'a' - displacement concerns the y-direction. The 'Changing parameters' inputs have been modified by interacting to the mask shown below:

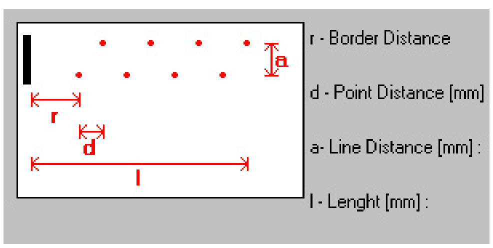

Fig. 3: The measurement line modifications view. Source: [18]

In our case the following modifications have been used:

- r- Border distance of first indentation from edge of specimen (r ≥ 2.5 D): 0.100
- d- Point Distance (d ≥ 2.5 D) [mm]: 0.200
- a- Line Distance [mm] : 0.100 and
- l- Total length [mm]: 6.000

(D = indentation diagonal).

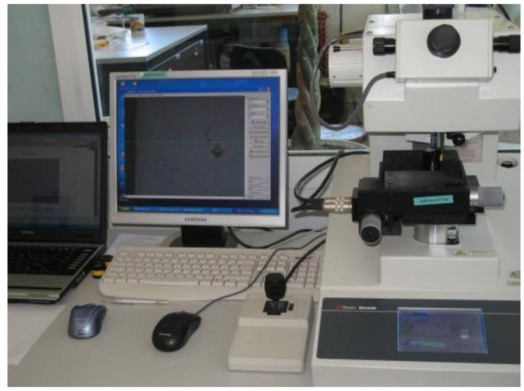

Fig. 4 The micro hardness equipment view

The above picture depicts the microhardness test equipment on the right with the sample on the 'test sample stage', the joystick and the monitor on the centre depicting the test measurement view and an additional laptop supporting the computation needs.

We will focus on using the micro hardness test by using the Vickers method in order to determine the materials case depth [10], [11] [14].

5.1. Calibration of reference hardness blocks

The available reference blocks have been calibrated by EURO PRODUCTS CALIBRATION LABORATORY on May 2005 gained accreditation by UKAS CALIBRATION under the code number 0441. The two reference blocks data are identified with the following data:

5.1.1. Serial number: EP0581110 Mean hardness value: 249.4 HV1

5.1.2. Serial number: EP0581087 Mean hardness value: 96.64 HV0.5

Fig. 5 Micro Vickers Reference Hardness Blocks

Both the above mentioned Micro Vickers Reference Hardness Blocks comply with the requirements of BS EN ISO 6507-3 clause 7. (1997) [12] [15].

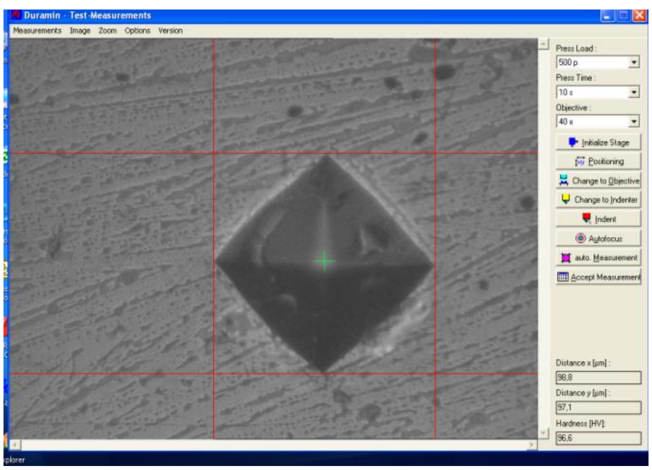

Fig. 6 The reference block test indentation [18]

The above picture depicts the test measurement which have made with the reference hardness block EP0581087 giving a hardness of 96.6 HV 0.5 which is fully complied with the referred on the calibration certificate. Thus the micro hardness testing equipment no need further calibration.

5.2. Verification of testing machines

Since along with our objectives is the laboratory development by expanding the activities through the growth of the new tests and definitely our most important scope is to gain accreditation by NAB. The scope of this work is to validate the microhardness Vickers method. Thus the laboratory has organised a technical leaflet which is currently under development by the author ([1]).

6. Experimental part - CASE STUDY

Among the applications concerning case depth determination carried out in the metallurgical laboratory is the case depth of a pin in a diameter of 16 mm used in conveyor belts at PPC / SA Open Cast Lignite Mines. From the metallurgical analysis is concluded that the material structure is of perlite and ferrite.

Fig. 7 Picture of the referred test sample

The above picture (Fig. 7) depicts the plan view of the sample that has been prepared using the metallographic preparation practices mounted in a cylindrical form in a diameter of Ø 30 mm made of epoxy resin [2]. It is obvious that after the etching process has displayed a distinguished area which is the transition zone.

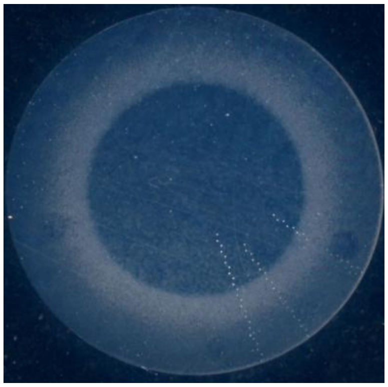

Fig. 8 Macro view of the test sample

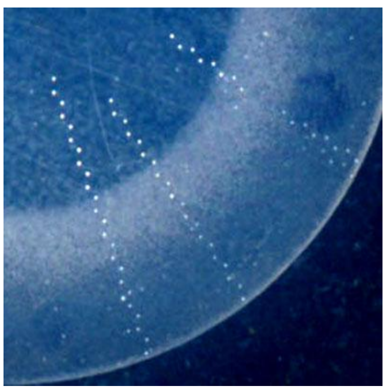

Fig. 9 Detail of the indentation series

The above picture (Fig. 8) on the top in macro mode depicts brightly the transition zone contrast which is similar to that of the sun's corona phenomenon. Figure 9 (on the bottom) represents the lower right part of the fig. 8 magnified to show the indentations through the transition zone in detail. Apart from that it represents the length of indentations towards to the core.

The 'effective case' depth at this point is determined by measurement of microhardness at the distance from the surface to a designated hardness level. By a further metallographic investigation to the distance from the surface to the core it could be defined as 'observed case depth' [1].

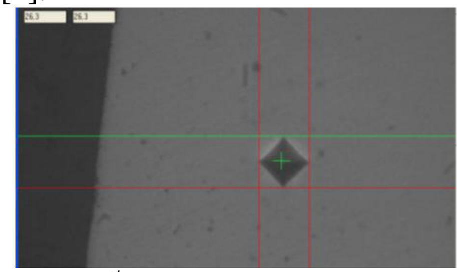

Fig. 10. The 1st Indentation up to the case (40x).

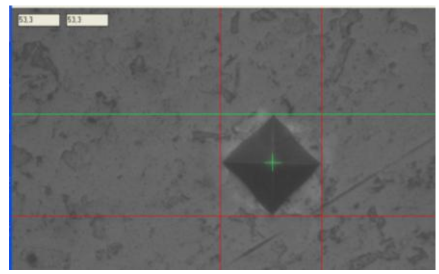

Fig.11. The 21st Indentation up to the core (40x).

The pictures above have been obtained from the micro hardness tester camera. The indentations presented are among the series of the test. The above picture on the top (Fig. 10) depicts the 1st indentation of the indenter close to the edge of the test piece. On the bottom (Fig. 11) is the 21st indentation to the core which is definitely larger than the 1st and the largest. The size difference confirms the hardness by giving the hardest to the surface.

Further investigation on material through data if it takes place such as the chemical composition, the mechanical properties or both will determine the treatment the material has undergone.

7. Results

Since among the objectives are to conclude whether the derived case depth corresponds to effective or the total case depth, the specific method should be decided [9].

Table 2 Data acquisition series from the measurements

Nr	X (mm)	Z (mm)	d1 (mm)	d2 (mm)	HV0.3	Material status
1	0,100	0,000	0,026	0,026	831,7	CASE DEPTH
2	0,350	0,008	0,026	0,027	748,8	
3	0,600	0,008	0,027	0,027	739,4	
4	0,850	0,001	0,029	0,029	681,6	
5	1,100	0,008	0,028	0,029	690,8	
6	1,350	0,003	0,028	0,029	690,8	
7	1,600	0,010	0,030	0,030	615,1	TRANSITION ZONE
8	1,850	0,010	0,032	0,034	510,7	
9	2,100	0,001	0,033	0,034	502,5	
10	2,350	0,017	0,034	0,037	445,5	
11	2,600	0,006	0,036	0,036	426,8	
12	2,850	0,026	0,039	0,039	367,2	
13	3,100	0,014	0,033	0,037	446,0	
14	3,350	0,035	0,044	0,044	290,5	
15	3,600	0,031	0,052	0,051	213,5	CORE
16	3,850	0,045	0,052	0,054	196,0	
17	4,100	0,045	0,052	0,053	200,6	
18	4,350	0,052	0,052	0,053	203,1	
19	4,600	0,050	0,047	0,053	223,7	
20	4,850	0,063	0,051	0,052	209,2	
21	5,100	0,059	0,053	0,053	195,8	
22	5,350	0,075	0,048	0,051	225,2	
23	5,600	0,070	0,052	0,052	205,1	
24	5,850	0,081	0,050	0,051	220,1	

Problems occurred during collating the results by the supporting software program due to its weaknesses to respond. Thus, as a consequence, while waiting for the updated edition of the specific software, there was a need for repeated measurement courses to be done [18].

To convert HV to MPa multiply by 9.807
To convert HV to GPa multiply by 0.009807

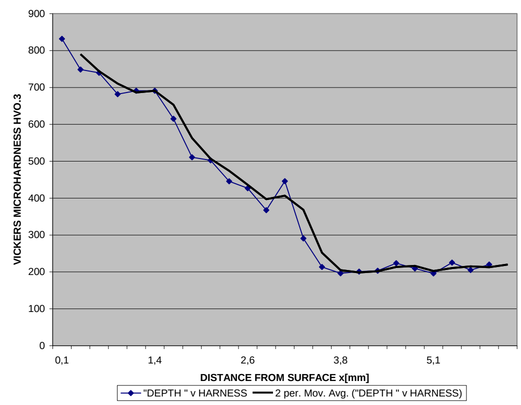

Fig 12 Case depth graph

The above graph Fig. 12 depicts the variation of the Hardness versus the case depth. The resource of the data based on the above Table 2 giving two curves. The light curve with the markers depicts the exact value of the hardness commencing at the height of 831.7 HV 0.3, position Nr 1. determining the case depth on x-axis given up to the Nr. 6 where the hardness drops to 690.8 HV 0.3 up to the depth of 1.350 mm. From Nr. 7th the curve inclination drops steeply up to Nr. 14th giving hardness varying to the range of 615.8 down to 290.5 HV 0.3 thus commencing the transition zone at the depth of 3.350 mm. Finally the last 10 spots are made under the pressure of the indentations to the core structure down to the depth of 5.850 mm with the hardness dropping down to 195.8 HV 0.3 at the depth of 5.100 mm as lowest.

The bold line is the moving average thus producing a smooth trend. This curve seems similar to that shown in DIN 50190 -4 Bild 1.a thus, concluding that our methodology is fully compatible with practices used in international standards [6].

Tilt should be included in the uncertainty calculation [13]. Typically test surface tilt is less than 1° and sin 1°=0.017. Tilt grade concerning the z-axis difference, depicted on table 2 above, through the indentation series it is obvious that it is on z_{24} =0.081 z_2 =0.008 giving a difference of 0.073 mm. Thus the tilt from beginning to end of the series rises up to 5.85- 0.1 = 5.75.

In our case is calculated that the inner angle θ derived from the tilt giving in sin is: sin θ =0.073 / 5.75 = 0.013 and comparing with that of the permitted above 0.013<0.017 consequently it is in the safe area.

In order to determine the hardness it is necessary to calculate the total uncertainty of the measurements based in mathematical methods which are irrelevant to this paper. The uncertainty determination is important for the accreditation process (regulation of the NAB).

This ability to relate measurements back to appropriate measurement standards, through an unbroken chain of calibrations, is referred to as traceability of measurement.

8. Conclusions

From the experimental data obtained from the tests referred above we take the chance to present the results appear both on Table 2 in combination with the Case Depth Graph on Fig. 12. It is obvious that the graph derived using current methodology is similar to those shown in the international standards e.g. DIN 50190-4. In addition the effective case depth of 1.350 mm has been determined. Conclusively the adopted method is considered as appropriate.
Finally a future investigation related with the 'observed case depth' determination would be useful to be taken into consideration.

9. References

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Acknowledgements

Our grateful acknowledgements to Prof Dr Ing N.G.Orfanoudakis, of TEI of Chalkis Greece for his enthusiasm to the valuable and unprofitable contribution on fulfilling this paper.