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STANDARDIZATION – THE SON EXPERIENCE

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Abstract - Standards Organisation of Nigeria (SON) was established by Decree No. 56 of 1971 and statutorily charged with the responsibility for National Policy on Metrology, Standards, Testing and Quality Control (MSTQ) in Nigeria. Standardization activities carried out by SON range from development and approval of standards, quality assurance, inspection and testing, certification and calibration.

Hardness measurement plays a vital role in Standardization. Product and System certification programmes of SON have recorded a considerable number of hardness measurements in industries, oil & gas facilities, laboratories and research institutions.

This paper gives a detailed report of the Standardization activities in SON with emphases on Metrology. It started by giving a full description of the corporate entity, legal status, organizational structure, human and technical resources of SON. The paper goes further to show the functions and detailed activities of SON, including limits of capability. The role of hardness tests and values in quality assurance activities is given a considerable portion in this paper. The development of Metrological Infrastructure, which enhanced the promotion of Standardization activities in SON, is also highlighted. Fields of Measurements available in SON Metrology laboratory are enumerated. Institutional support and international co-operation with various foreign bodies and international organizations are included in the paper.

In conclusion, the paper outlines the way forward to ensure the growth of metrology, hardness measurement in particular, and effective Standardization activities in SON.

1. INTRODUCTION

Standards Organisation of Nigeria (SON) was established by Decree No. 56 of 1971, and with three amendments in 1976, 1984 and 1990, the body corporate, has the sole responsibility for National Policy on Metrology, Standards, Testing and Quality Control (MSTQ).SON is the custodian of all product, process and metrology Standards in Nigeria. The Organisation is governed by a Standards Council but the day – to – day running of the Organisation rests on the Director General/Chief Executive.

The following Directorates exist in SON: Standards, Quality Assurance, Laboratories, Metrology, Technical services, Human resources, Finance and supplies and Legal Services.

2. METROLOGICAL FACILITIES AND CALIBRATION

Metrological facilities are being gradually developed in SON under the UNIDO integrated Programme. This programme commenced with skeletal training of personnel on some fields of measurements in various parts of Europe. The project document for the laboratory, which is to be located in the capital city, Abuja has been written by the UNIDO Consultant and approved by UNIDO. The setting up of the laboratory infrastructure is the responsibility of the government of Nigeria but yet to commence. While the establishment of the laboratory in Abuja is being awaited, SON carries out skeletal calibration services from the operational headquarters in Lagos and the engineering laboratory in Enugu with low accuracy standards. These are in the fields of mass, volume, temperature and pressure. The calibrations are carried out using the following metrological standards being maintained;

(a) Standard weights: 1gm to 20kg of class m1
(b) Standard flasks made of glass 0.5 litres to 5 litres.
(c) Precision digital thermometer
(d) Standard pressure gauges

In order to render calibration services to the industry as well as carry out the legal metrology activities in the remote areas of Nigeria, which is not included in the UNIDO Integrated Programme, SON considered it advantageous to procure a vehicle with built – in calibration equipment and operate it as Mobile Calibration Service. The process of acquiring the mobile calibration facilities is going on now. The vehicle has been designed to have the following features; Minimum power output of 90kw;Minimum load carrying capacity of 7.5 tons; Sleeping cabin for 2 people; Air conditioning system for laboratory area and sleeping cabin; Rigid work tables firmly installed; Power supply stabilizer 220 V, 50 Hz, 10 k VA; Tool set.

The modules proposed for the Mobile Calibration Service include;

Mass measurement

Volume/Flow measurement

Dimension measurement

Pressure measurement

Force measurement

Hardness measurement

Temperature measurement

Electrical measurement

Viscosity measurement

The project will be completed in 2004.

3. MEASUREMENT SUPPORT SERVICES: TRAINING, MAINTENANCE, REPAIR, AND CALIBRATION

Measurement is the act of measuring, which is the way of discovering exact size, amount and characteristics of things encountered in all fields of human endeavour. Measurements are encountered in the fields of medicine, engineering, physical sciences, arts, culture, music, commerce, agriculture, education, communication, etc.

The safe operation and effectiveness of measuring instruments need support services. These support services are essential to ensure that reliable measured values are obtained. The basic support services needed for adequate and effective use of measuring instruments are training, maintenance, repair and calibration.

4 TRAINING

Training is generally given a high priority in SON. Training is carried out in areas of Quality Assurance, Testing, Standards development and Metrology. The Organisation has trained engineers in various fields of measurements, which afforded them the skill, and expertise in selection of appropriate measuring methods and correct use of measuring equipment. It cannot be expected from even the most prominent experts working in various fields of profession to solve every diversified new measuring task that may arise in their fields of activity in an expert – like manner. In view of this, training and retraining are given in different specific areas of measurements in SON to satisfy the metrological needs in Nigeria and keep experts abreast of technological developments.

Metrologists in SON were trained in PTB, Germany; KBS, Kenya; NML, South Africa; NWML, United Kingdom; NMI, Holland and OIML,
France.

5 REGIONAL CO-OPERATION IN METROLOGY

It is the resolve of SON to establish and maintain a strong co-operation in metrology with other regions of Africa. This is to harmonize and rationalize our policies and strategies with other regions of Africa and the world for sustainable development. This has been manifested in our determination to become an associate member of Southern African Development Community Cooperation in Measurement Traceability (SADCMET) and Southern African Development Community Cooperation in Legal Metrology (SADCMEL).

SON signed a Memorandum Of Understanding (MOU) with the National Metrology Laboratory (CSIR) of South Africa in August 2002. The areas of agreement include, training, provision of equipment, exchange of expertise, exchange of standards, inter – comparison of measurements, etc. It is the wish of SON to co-operate with the National Institute of Standards and Technology (NIST) of USA.

6 PROPERTY TESTING IN SON

Testing is an essential part of any engineering or standardization activity. Inspection and testing are key functions that take place in SON. They are preformed during conformity assessment (CA) processes.

Inspection and testing are carried out on metals, polymers, ceramics or composites, and during the forming of these materials into composites, and assembling the components to create an engineering product, to satisfy some specific requirements.

The requirement for testing does not automatically cease when the product has been manufactured. It is frequently necessary to test and check the product during its service life in order to monitor changes, such as the possible development of fatigue or corrosion damage.

The types of test carried out in SON can be classified into two categories:

(a) Tests to establish the properties of the materials, and

(b) Tests to establish the integrity of the materials or component.

Those tests in the first category are generally of a destructive type. They are performed on samples of materials and the test piece is damaged, dented or broken in the process as in the process when determining the tensile strength or hardness of a material. In this case, samples taken are indicative of the properties of entire batch of materials. E.g. batch or lot testing of steel bars, etc.

The tests in the second category are of a non-destructive nature and are used to detect the presence of internal or surface flaws in a material, component or finished product. These tests do not damage the parts being tested and sampling is not required. If necessary, every item can be checked. E.g. testing of unfired pressure vessels.

In Nigeria, SON publishes the standards and codes of practices, which cover most aspects of the testing and utilization of materials.

Many test procedures have been devised for the determination of some of the properties of materials, for

example, the hardness test. This is to ensure that results of tests have values.

Some of the physical property testing carried out in SON include, tensile strength tests, hardness tests, bend tests, yield strength tests and impact tests.

6.1 HARDNESS TESTS

The hardness of a material can be determined either by making a surface indentation or a scratch test. Materials that are capable of plastic deformation are tested for hardness by surface indentation method. These include metals and thermoplastic materials.

The resistance of a material to indentation is not necessarily the same as its resistance to abrasion, but a hardness measurement obtained from an indentation test can be used as an empirical check for abrasion resistance In general, metals having high hardness will have high resistance to abrasive wear.

Indention – type hardness tests are widely used for the checking of metals samples, as they are easy to make and yield information on heat treatment condition. There are also empirical relationship between hardness of a metal and its tensile strength.

6.2 HARDNESS TESTING IN SON

In SON, hardness tests are carried out on metallic and non-metallic materials using the Brinell test method, Vickers test method, Rockwell test method, and Micro hardness test method.

The hardness of ceramic materials are obtained using a scratch method. In this case, Mohs' scales of hardness, which was devised for assessing the relative hardness of minerals and rocks is based on ten naturally occurring minerals ranging from very soft talc to diamond.

Fig I shows the Mohs' scale of hardness. In this test, an attempt is made to scratch the surfaces of the standards with the materials under test. The hardness of the unknown lies between the numbers of the mineral it just fail to scratch and that of the mineral it just scratches.

Fig 1- Mohs' scale of hardness

Number	Mineral
1	Talc
2	Gypsum
3	Calcite
4	Fluorite
5	Apatite
6	Orthoclase Felspar
7	Quartz
8	Topaz
9	Corundum
10	Diamond

Metallic materials are tested for hardness in SON using a Universal Hardness Tester, Model DV5B – M serial No 0109, and manufactured by CV- England.

The technical specifications for the Hardness Tester is shown in Table 2.

Table 2 Technical Specifications

Hardness parameters	Brinell, Vickers, Rockwell
Optics	High precision optics, screen diameter 135mm
Objectives	Interchangeable 20x, 44x, 70x, 140x, 250x, magnification.
Scales Resolution	Incremental scale / better than 1 micron
Display	Integrated hardness calculator, determination of hardness values for all procedures, statistic functions, data transfer.
Standard	Conform to 1S0 6506, 6507, 6508 and ASTM
Test Load Type	Dead weights, dial selector
Test Cycle	Manual lever system/semi – automatic
Test Loads	Brinell 1-5-10-15,625-30-31,25-62,5-125-87,5 -250kg Vickers 1-3-5-10-20-30-40-50-100kg Rockwell 60-100-150kg
Indentor types optional	Brinell Balls 1-2,5-5-10mm Vickers Diamond cone 136^0 Rockwell Diamond cone $120^{0,}$ Balls $^1/_{16}$–$^1/_8$–$^1/_4$-1/2 (inches).
Load duration	0.1-255sec
Data output	RS 232 serial interface (to printer or PC)
Specimen accommodation	Maximum test height 300mm, maximum throat 150mm
Specimen access	External surfaces, cylindrical surfaces down to 3mm diameter
Power supply	220v /50Hz
Machine Dimensions	Width 250mm, depth 567mm, height 1018mm
Machine net weight	300kg

Table 3 shows materials versus range for samples tested in SON over the past 10 years.

Table 3 -Materials versus range

Materials	HRC	HB	HV
Steel and cast steel	20-68	80-650	80-940
Cold work tool steel	20-68		80-940
Stainless steel	20-62	80-650	80-800
Grey cast iron	-	90-380	
Cast aluminium alloys	-	20-160	
Brass	-	40-170	
Bronze		60-290	
Copper	-	45-315	

The regular Rockwell scales, loads, indenters and applications are shown in Table 4.

Table 4 – Regular Rockwell Scales, Loads, Indenters and Applications.

Scale	Indenter	Test force	Applications
A	Diamond	588,4N (60kgf)	Case hardness steel, cemented carbide,thin steel sheet, copper
D	Diamond	980,7 (100kgf)	Case hardness steel, cemented carbide, thin steel sheet, copper
C	Diamond	1471N (150kgf)	Case hardness steel, cemented carbide, thin steel sheet, copper
F	Steel ball diameter $^1/_{16}$ (inches)	588,4N (60kgf)	Annealed Steel, bearing metal, hard – drawn aluminium alloys, brass, beryllium copper, Phospor bronze.
B	Steel ball diameter $^1/_{16}$ (inches)	980,7N (100kgf)	Annealed Steel, bearing metal, hard – drawn aluminium alloys, brass, beryllium copper, Phospor bronze.
G	Steel ball diameter $^1/_{16}$ (inches)	1471N (150kgf)	Annealed Steel, bearing metal, hard – drawn alluminium alloys, brass, beryllium copper, Phospor bronze.
H	Steel ball diameter $^1/_8$ (inches)	588,4N (60kgf)	Bearing metal, Grinding Stone
E	Steel ball diameter $^1/_8$ (inches)	980,7N (100kgf)	Bearing Metal, Grinding Stone
K	Steel ball diameter $^1/_8$ (inches)	1471N (150kgf)	Bearing metal, grinding stone
P	Steel ball diameter ¼ (inches)	588,4N (60kgf)	Extra mild metal (e.g. aluminium, zinc, lead
M	Steel ball diameter ¼ (inches)	980,7N (100kgf)	Extra mild metal (e.g. aluminium, zinc, lead
L	Steel ball diameter ¼ (inches)	1471N (150kgf)	Extra mild metal (e.g. aluminium, zinc, lead
R	Steel ball diameter ½ (inches)	588,4N (60kgf)	Tin, Plastics, Cardboard
S	Steel ball diameter ½ (inches)	980,7N (100kgf)	Tin, Plastics, Cardboard
V	Steel ball diameter $^1/_2$ (inches)	1471 N (150kgf)	Tin, Plastics, Cardboard

Hardness tests are carried out on samples of materials during conformity assessment activities.

These conformity assessment activities are covered under the routine product certification exercise and the quality system certification scheme, which lead to award of NIS product quality award and 1SO 9001 standard certification.

7 CALIBRATION OF HARDNESS TESTING MACHINES

Hardness reference blocks are used for verification and calibration of hardness testing machines, as well as for periodic check and sometimes for overtaking of hardness scales on a hardness testing machine. Hardness reference blocks are essential and a necessary help of industrial Quality Management.

Only the use of high quality, precise hardness reference blocks calibrated to applicable standards can ensure the functionality and relative reliability and accuracy of measurement of a hardness testing machine.

Standardized blocks used in the indirect verification of hardness testing machines are also calibrated. This does not necessarily apply to the calibration of test blocks, which are used in the routine checking of the testing machine by the user. Standardized blocks used for Rockwell B and C scale hardness testing machine are calibrated in a standardizing machine complying with the requirements of sections 4 to 8 of ISO/R674-1968 (E) at a temperature of 27 $\pm$ 2 (for tropical climates).

7.1 REPEATABILITY

For each standardized block, let e_1, e_2, e_3, e_4, e_5 be the values of the measured increase in depth of indentation, arranged in increasing order of magnitude, where e is in units of 0.002mm, as defined in ISO 6508.

The repeatability of the testing machine under the particular verification conditions is determined by the following quantity:

$$e_5 - e_1 \quad - \quad (1)$$

The repeatability of the testing machine being verified is not considered satisfactory unless the repeatability at each hardness at which the machine is verified is:

- For the Scale A, less than 0,03ē
- For the Scale B, less than 0,06ē
- For the Scale C, less than 0,03ē
- For the Scale D, less than 0,03ē
- For the Scale E, less than 0,06ē
- For the Scale F, less than 0,06ē
- For the Scale G, less than 0,06ē
- For the Scale H, less than 0,06ē
- For the Scale K, less than 0,06ē

Where $\bar{e} = \frac{e_1 + e_2 + e_3 + e_4 + e_5}{5}$ - (2)

8 INDENTERS FOR ALL HARDNESS SCALES

There are wide ranges of indenters, which are certified and traceable to internationally recognized standards. The indenters used in SON are traceable to UKAS. It is a policy in SON to protect all indenters from defects. Experience has shown that a number of initially perfect indenters can become defective after use for a comparatively short time.

This is due to small cracks, pits or other flaws in the surface. If such flaws are detected in time, many indenters may be reclaimed by grinding. If not, any small defects on the surface rapidly worsen and make the indenter useless.

In view of the above, the following steps are taken in SON to ensure the adequacy and suitability of indenters;

(a) The condition of indenters are checked at frequent intervals using optical devices (microscope or magnifying glass).

(b) if the flaw is not on the active part of the indenters, it is ignored, but if it is on the active part, even if small, the indenter is reground before use.

(c) the verification of the indenter is no longer valid when the indenter has become unusable because of defects.

(d) reground or otherwise repaired indenters are re-verified.

9 LIMITATIONS

Internal tests on rings, tubes and annular parts of inside diameters plus the wall thickness, less than 50,8mm or 2 inches are not performed in SON due to non-availability of the required accessories. The necessary adapters that will permit regular or superficial Rockwell hardness testers to perform internal tests are not available in the Organisation. These adapters will fit any of the Standard Rockwell hardness testers.

It can be clamped into the bottom of the plunger rod (in the same manner as an indenter) and is not heavy enough to affect a reading due to increasing the applied load. Minimum diameters of 11,5mm or 7/16 inch are often required to be tested. Another major limitation in SON is the non-availability of hardness reference blocks for calibration of the universal hardness testing machine. This has affected the functionality and relative reliability and accuracy of measurements. To ensure relative accuracy of measurements, the Organisation embarked on inter-laboratory comparisons with other laboratories in Nigeria where hardness tests are carried out.

The results of inter-laboratory comparison showed average deviations of less that 1%.

10 MICRO HARDNESS TESTING

The micro hardness testing carried out in SON is the Vickers diamond test. This test is performed with a micro hardness tester manufactured by Buehler (USA).The principle of Vickers diamond micro hardness test is basically the same as for the standard Vickers test but the indenting loads used are measured in grammes rather than kilograms. This type of hardness testing is performed on a metallurgical microscope adapted for the purpose. The small pyramidal diamond indenter is embedded in the surface of a special objective lens. The surface of the test sample is prepared to a high polish and etched for micro-examination. When viewed under the microscope with a high magnification, usually some value between 200x and 2000x, any particular micro-constituent or feature can be centred in the field of view and a micro-sized diamond indentation made using a small indenting load.

11 CONCLUSION

Standards Organisation of Nigeria has successfully carried out standardization activities in the past 23 years. They covered Quality Assurance, Inspection and Testing, Calibration, and Standards development. Hardness testing formed a significant part of these activities.

The Rockwell, Brinell, Vickers and micro Vickers scales of Hardness tests carried out in SON has gone a long way in ensuring stability, accuracy and repeatability of results obtained in industries in Nigeria.

Industries seeking product and system certification have over the years relied on SON for reliable hardness values for their materials and products.

In the absence of hardness reference blocks for calibration of the universal hardness testing machine, SON engaged in inter-laboratory comparisons to ensure acceptable accuracy level.

Knowing fully well that this type of inter-laboratory comparison cannot substitute the calibration or verification of hardness testers, SON is making arrangement to purchase hardness reference blocks for all scales with official calibration certificates that are traceable to internationally recognized standards.

When the hardness reference blocks are available in SON, and the universal hardness testing machine calibrated and verified annually, the functionality and relative reliability and accuracy of measurements will be achieved.

It is also the intention of SON to enlarge the field of application of the hardness measurements. This can be achieved by purchasing special adapters in various sizes to permit hardness testers to perform tests on rings, tubes and annular parts where the inside diameter, plus the wall thickness, is less than 50,8mm or 2 inches. This will enable SON render services to numerous manufacturers of the products in Nigeria.

REFERENCES

[1] V.B. John, "Introduction to Engineering Materials", Macmillan Press Ltd, Houndmills, Basingstoke, Hampshire RG21 6XS and London, 1992.

[2] Wilson Wolpert, "Materials Testing and Inspection", Catalogue for materials, Testing and instruments, Wolpert Europe, Netherlands, 2003.

[3] International Organisation for Standardization, "Mechanical testing of metallic materials" Geneva,1988.

[4] ASTM International, "Nondestructive and Mechanical Testing" Standardization News, Volume 31, Number 10, West Conshohocken, PA, 2003.

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