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Role of indentation size effect in micro-indentation testing on different materials

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Abstract:

In micro-indentation testing for several types of materials, the indentation size effect (ISE) was noted for a comparatively greater range of applied test loads. Heat-treated plain carbon steels, copper, aluminium, and tool steels were the samples used in this investigation. The analysis made use of a micro-hardness tester with loads ranging from 0.49N to 9.8N. The modified proportional specimen resistance model (MPSR i.e. $P = P_0 + a_1 d_1 + a_2 d_2$), where P0 accounts for the residual surface stresses generated during the sample preparation process, provided better correlation between literature-cited load-independent hardness values and the measured values among the existing models that relate the indentation load and indentation length. Regardless of the loads applied, the investigation produced the precise hardness values for all the materials. Surface strains or any brittle structures at the surfaces can occasionally cause inconsistencies in the hardness readings.

Keywords: indentation size effect, heat treated Copper, Aluminum, Tool steels, plain carbon steels, micro-hardness

Introduction:-

The ability of a material to resist indentation is known as hardness. There are numerous varieties of rebound, indentation, scratch, and hardness. [1] The microstructure of metals and alloys influences certain qualities. Hardness is the only attribute that a metallurgist or other materials engineer can use to comprehend the immediate behaviour of the material under consideration. To study these hardness, there is a variety of equipment available. [2] Vickers hardness, also known as a micro-hardness test, is one of the devices that is useful because it doesn't harm the specimen. One of these tools is the Vickers micro hardness tester, which employs an indenter made of diamond with an included angle of 136 degrees between the faces of the diamond indenter and uses a load range of 1-1000g. While studying the indentation tests at micron scales there are various phenomena arising regarding the generation of dislocation loop and its effect on various surfaces under study. This microstructural concept leads to ISE effect which infers about high hardness with low loads. [3, 4]

Hardness being a deformation parameter on a micro-scale is known as micro-hardness. The micro-hardness of solids generally depend on the indentation test load. This dependency is known as the indentation size effect (ISE), where a decrease in the micro-hardness is observed with increase in applied test load, i.e. with increasing indentation dimension.[5] Such ISE can be attributed to a different phenomena occurring at the material surface, including work hardening during indentation, [6,7] indentation elastic recovery, [6, 8] size of dislocation loops formed during indentation, [9] load to initiate plastic deformation, [10] indentation edges acting as plastic hinges,[11], mixed elastic/plastic deformation response of material, [12] strain gradients associated with dislocations and indenter/specimen friction resistance coupled with elastic resistance of the specimen. [13,14] A reverse sort of indentation size effect (RISE), where the apparent micro-hardness increases with increasing applied test load (Fig. 1), is also known. It differs from the aforementioned ISE, also known as standard ISE. It has been noted to occur in materials when plastic deformation is prevalent. The explanations offered include the occurrence of a deformed zone close to the crystal-medium interface, the effects of vibration, and the bluntness of the indenter as a result of specimen chipping surrounding the depression. [15] The phenomenon is still not fully understood, though. Fig. 1 presents a schematic illustration of the ISE and RISE. The graphic illustrates how the micro hardness varies depending on the test load for low indentation loads and becomes more constant for higher values.

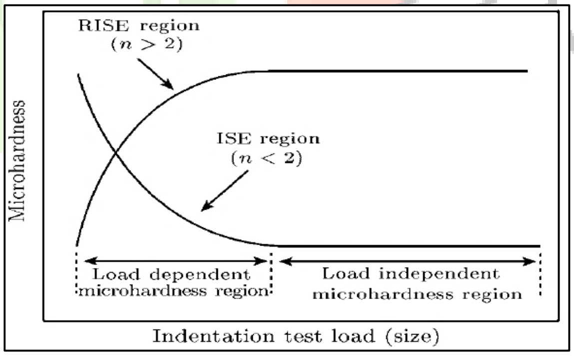

Fig 1:- Micro-hardness variation with test load, showing the indentation size effect (ISE) and reverse indentation size effect (RISE) [5]

To understand the concept behind ISE, various models have emerged and discussed. [16] The true hardness values are hindered by various surface and microstructural events. To obtain the true hardness values one has to really understand these vents which are related to the dislocation generation at the indentation volume and also the residual stresses while preparing the specimen for the indentation study. Each model provides a way to eliminate these events and help the engineer to obtain the errorless true hardness values. To investigate the cause of ISE, the study has been converted in different kind of materials right from softest to hardest. The underlying concepts behind the observed phenomena are discussed further in this paper.

Experimental Methods and Materials Details:

The goal of the current work is to investigate how micro-hardness varies with load for various steel samples made of a variety of materials, from soft to stiff. Additionally, the potential reason for the ISE observation will be emphasised. A variety of mathematical models will be used to assess how hardness varies with load, with a focus on how well Meyer's law and the Modified Proportional Specimen Resistance (PSR) model apply to a given range of test loads. Finally, it is possible to calculate each sample's True hardness (HT), also known as load independent hardness. The information about the samples collected for analysis is shown in Table 1:

Table 1: - Materials for the ISE study

Soft materials chosen	**Hard Materials**
1. Aluminum	1. Quenched - Mild Steel (MS)
	2. Carburized MS (60% Charcoal + 40% Barium Carbonate, Ba_2CO_3)
2. Copper	3. Carburized MS (80% Charcoal + 20% Ba_2CO_3)
	4. Tool Steel

Of the samples chosen two samples are soft and rest samples are harder. The carburized samples were carburized using standard methods with varying the composition of the energizer in our case Ba_2CO_3. (See Table 1). All the samples were polished up to 1 micron diamond polishing to remain scratch free and finally taken for the micro-hardness study. The indentation test were carried on (Machine details) for a range of loads from 50 gmf to 1000 gmf with a constant dwell time of 5 minutes.

Meyer's Law:

Meyer's law states that [5] when calculating hardness, the anticipated area of the indentation is taken into account. The formula is as follows:

$P = K_m d^n$ ……………………………………………………………………………………………Eq.1

Where P is the applied load, d is the diagonal of the indentation, K_m is the Meyer's law coefficient, and n is the Meyer's law exponent. The equation of the Meyer's law can be re-written in the form of a linear equation by applying "log" on both sides as mentioned below:

$\log P = n \log d + \log K_m$ …………………………………………………………..…………………Eq. 2

Here P is the Load, d is the indentation diagonal, K_m and n are material constants. K_m is known as Meyer's coefficient where n is known as Meyer's law exponent and this can be calculated by plotting the log-log plots of P vs d.

Modified Proportional Specimen Resistance model:

The ISE behaviour in various materials is investigated using the modified PSR model. The final MPSR model is described as: [17, 18]

$P = a_0 + a_1 \cdot d + a_2 \cdot d_2$ …… ……………………………………………………..……………..……Eq. 3

$P = a_0 + a_1 \cdot d + (\text{Hpsr}/1.854).d^2$ ……………...……………………………………………………Eq 4

Where,

a_0 is a constant that is connected to the leftover surface tensions from the surface grinding and polishing procedures used to prepare samples. Parameters a_1 and a_2 are constants as defined in the PSR model.

[17,18] The values of a_0, a_1, and a_2 can be evaluated by plotting the P data against d.

Results & Discussions:

Figure 2 (a) and (b) shows the micro-hardness (GPa) vs load of various materials tested. Each data point is the average of readings from three tests conducted at each load. The obtained results show that all materials clearly exhibit the load dependency of the hardness. The indentation size effect is responsible for the observed difference in hardness. Out of all the materials examined, the carburized (80-20) sample exhibits the RISE phenomena, increasing in hardness with increasing load.

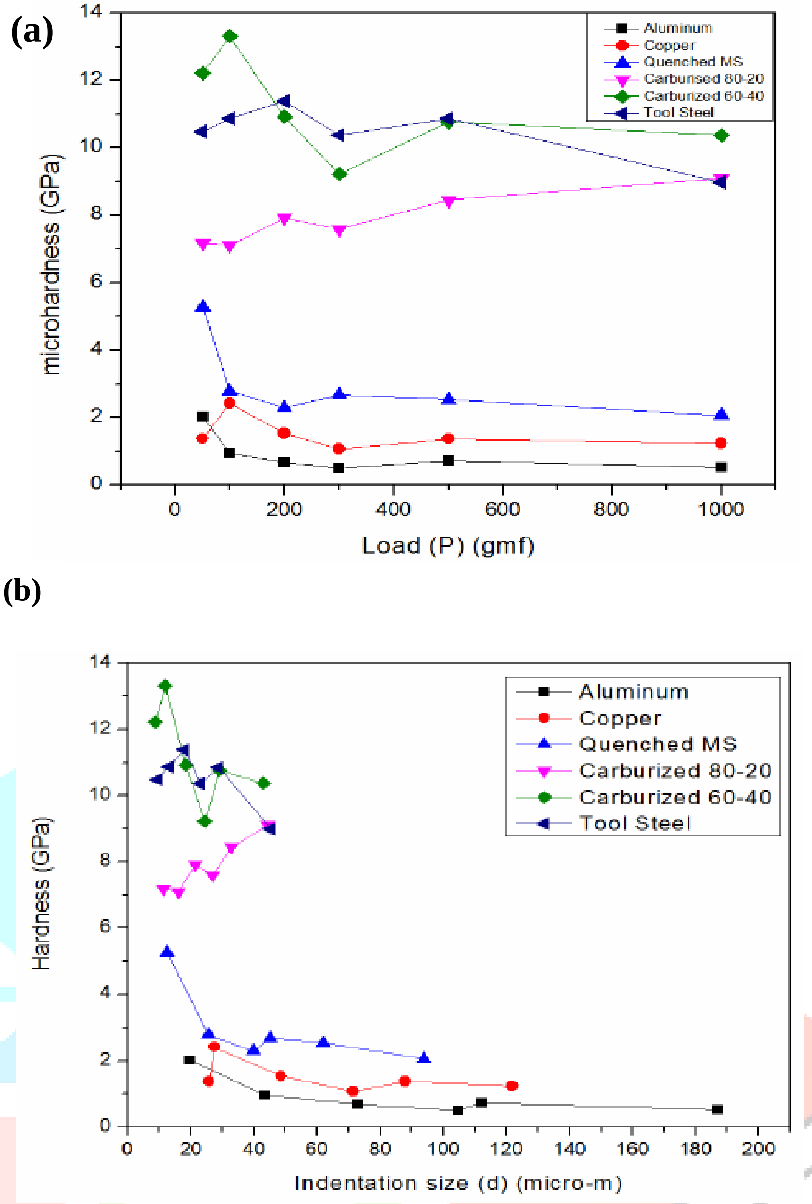

Fig 2:- a) Micro hardness (GPa) vs. load b) Hardness (GPa) vs Indentation size

From the figure it can be understood that harder samples have lesser indentation size (d) compared to the softer materials i.e. d for carburized (60-40)<Tool steel<carburized (80-20)<Quenched MS<Copper<Aluminum. This is a well-known trend which the samples are following confirming that the steel subjected to carburization was very well carburized and thus lesser indentation size.

To investigate the ISE, first the Meyer's law is used to tell about the work hardening of materials and establishes a relationship of hardness values with the strain hardening behavior of the materials. lnP vs lnd curves were plotted and fitted with straight lines to identify slope and intercept values. Similarly in case of MPSR, the P vs d was plotted and a nonlinear fit was used to identify the various parameters values.

Table 2: Best fit results of Parameters according to Meyer's Law and MPSR model

Sample	**Meyers Law**			**MPSR model**			
	n (slope)	**logA (intercept)**	**Adjusted R^2**	**a_0**	**a_1 (gmf/micron)**	**a_2 (gmf/micron^2)**	**Adjusted R^2**
Aluminum	1.32	-0.240	0.987	**14.154**	1.023	0.0228	0.966
Copper	1.68	-1.282	0.996	**90.02**	-2.480	0.0812	0.990
Quenched Mild Steel	1.51	-0.082	0.985	**-3.953**	2.241	0.0907	0.997
Tool Steel	1.91	-0.29	0.998	**-94.950**	11.883	0.273	0.996
Carburized Steel 60-40	1.82	0.025	0.995	**13.706**	0.248	0.531	0.993
Carburized Steel 80-20	2.18	-1.412	0.999	**70.90**	-9.079	0.674	0.999

From the Table 2 it can be understood that except carburized (80-20) samples every other sample exhibits clear ISE. Here the Aluminum is observed to have more prominent effect compared to other samples. The discrepancy in the results of carburized (80-20) is may be due to the uneven carbon diffusion on the surface, residual stresses developed due to carburization or mixture of energizer effect problem or even brittle nature of carbon layer. [19] Finally from the above obtained parameters from MPSR model the True hardness (load independent hardness) for all experimental samples has been determined. The HT values of all the samples are shown in Table 3.

Table 3: - True hardness values of different materials

Methods → **Samples ↓**	**Mayer's Law**	**MPSR Model**	**True Hardness (HT)**
Aluminium	$P=0.7866d^{1.32}$	$P= (14.154+1.023d+0.0228d^{2}$	42
Copper	$P=0.2774d^{1.68}$	$P= (90.02-2.480d+0.0812d^{2})$	150
Quenched Mild Steel	$P=0.9212d^{1.51}$	$P= (3.953+2.241d+0.0907d^{2})$	168
Tool Steel	$P=0.7482d^{1.91}$	$P= (-94.750+11.883d+0.273d^{2})$	506
Carburized Steel (60-40)	$P=1.0253d^{1.82}$	$P= (13.706+0.248d+0.531d^{2})$	984
Carburized Steel (80-20)	$P=0.2436d^{2.18}$	$P= (70.90-9.079d+0.674d^{2})$	1249

Conclusions: -

The behavior of the ISE may be observed in all experimental samples. It implies that indentation loads affect the measured Vickers hardness values. The test loads affect hardness. For all the samples, the experimentally determined Meyer's index n is less than 2, indicating normal ISE behavior, in which hardness decreases with increasing test loads. However, for the carburized steel (80% charcoal, 20% barium carbonate) sample, the Meyer's index n is greater than 2, indicating reverse ISE behavior (RISE), in which hardness increases with test loads. The Meyer's and MPSR models are used to assess the experimental hardness values, and regression analysis is used to find the parameters that fit the data the best. The Meyer Law and the modified PSR produced the highest correlation between measured data and mathematical models. True hardness which is independent of test loads is determined by using the parameters obtained according to MPSR model. The obtained real hardness is lowest because the aluminum sample is the softest of all the experimental materials. The maximum measured real hardness is found in carburized steel, which is carburized by 80% charcoal and 20% Ba_2CO_3. It's possible that residual stress, inappropriate carbon diffusion, or the brittleness of the carbon layer on the surface are the causes of the discrepancy in the values of carburized (80-20).

ACKNOWLEDGEMENT:

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References:

[1] Information on: http://www.substech.com/dokuwiki/doku.php?id=hardness_test_methods

[2] Information on: https://www.hardnesstesters.com/test-types/hardness-testing-basics

[3].Majić Renjo, M., Ćurković, L., Štefančić, S., & Ćorić, D. (2014). Indentation size effect of Y-TZP dental ceramics. Dental Materials, 30(12), e371–e376. doi:10.1016/j.dental.2014.08.367

[4] X. Ma, W. Higgins, Liang, Z. D. Zhao, G. M. Pharr, & K. Y. Xie, (2021). Exploring the origins of the indentation size effect at submicron scales. Proceedings of the National Academy of Sciences, 118(30), e2025657118.

[5] Information on: https://www.researchgate.net/publication/243415733-Indentation Size Effect and Microhardness Study of β-Sn Single Crystals

[6] Mott B W 1956 Micro indentation Hardness Testing (London: Butterworths Scientist) chap 4 p 101

[7] Buckle H 1973 the Science of Hardness Testing and Its Research Applications Ed Westbrook J H and Conrad H (Metals Park, OH: ASME) p 199

[8] Tate D R 1945 Trans. Am. Soc. Metals 35 374

[9] Upit G P and Varchenya S A 1986 Phys. Status Solidi 17 831

[10] Hays C and Kendall E G 1973 Metallography 6 275

[11] Ma Q and Clarke D R 1995 J. Mater. Res. 10 853

[12] Bull S J et al 1989 Phillos. Mag. Lett. 59 281

[13] Li H and Bradt R C 1993 J. Mater. Sci. 28 917

[14] Li H, Han Y H and Bradt R C 1994 J. Mater. Sci. 29 5641

[15] Sangwal K 2000 Mater. Chem. Phys. 63 145

[16] Ise :- Curkovi c L, Lalic M, Soli c S. Analysis of the indentation size effect on the hardness of alumina ceramics sing different models. Kov Mater – Met Mater 2009;47:89–93**.**

[17] Ronald Machaka, Trevor E. Derry, Iakovos Sigalas, and Mathias Herrmann, Analysis of the Indentation Size Effect in the Microhardness Measurements in B6O, Advances in Materials Science and Engineering Volume 2011, Article ID 539252, 6 pages

[18] J. Gong, Z. Guan, Load dependence of low-load Knoop hardness in ceramics: a modified PRS model, Mater.Lett. *47* (2001) 140-144.

[19] Dyonekura Effect of Residual Stress on Fatigue Strength of Steel Modified by WPC Process, N.E. LTD., 4-5-12, Sagamidai, Kanagawa, Japan