Wasit Journal of Engineering Sciences

Journal homepage: https://ejuow.uowasit.edu.iq

Investigating the correlation between Nanoindentation and Vickers hardness of Ti6Al4V, pure copper and AISI316 stainless steel

Ammar D. G. Al-Rubaye[1], Muhaed S. Alali[2], Righdan M. Namus[1]

Affiliations
[1]Department of Mechanical Engineering, Wasit University, Kut, Iraq.
[2]Department of Material Engineering, Kufa University, Najaf, Iraq.

Correspondence
Ammar D. G. Al-Rubaye
ammardawood @uowasit.edu.iq

Accepted

6-January-2024

Doi: doi:https://doi.org/10.31185/ejuow.Vol12.Iss4.

Abstract

Nano-indentation test is the most popular hardness testing method because it can correlate to other materials' mechanical properties and is used in a narrow area or for materials with plastic deformation resistance that Vickers hardness cannot represent. Linking the results of different hardness tests could provide flexibility in utilizing various hardness tests. Therefore, this research investigated the practical results of Vickers Microhardness and Berkovich Nano-indentation for three different materials. A mathematical model was applied to describe the Vickers hardness - Nano-indentation correlation based on the projected surface area resulting from indenter penetration. The results showed that Nano-hardness and Vickers hardness can be correlated. However, for the accuracy of the correlation, the precise measurement of the projected area must be considered. Ti6Al4V exhibited the largest differential between practical and theoretical Micro-hardness and Nano-hardness results, 43% and 37%, respectively, whereas copper had the lowest, 30% and 33%.

Keywords: Nanoindentation, Microhardness, Mechanical properties, Correlation.

الخلاصة:

اختبار الصلادة النانوي هو أكثر طرق اختبار الصلابة شيوعًا لأنه يمكن أن يرتبط بالخواص الميكانيكية للمواد الأخرى فضلا عن امكانية تطبيقه في منطقة ضيقة أو للمواد ذات مقاومة التشوه البلاستيكي التي لا يمكن لصلادة فيكرز تمثيلها. الربط بين نتائج اختبارات الصلابة المختلفة يمكن ان يعطي مرونة في استخدام اختبارات الصلابة المختلفة. لذلك، يحقق البحث الحالي في النتائج العملية لصلادة فيكرز المايكروية وصلادة اختبار بيركوفيتش النانوي لثلاث من المواد مختلفة، فضلاعن تطبيق نموذج رياضي لوصف وايجاد علاقة لارتباط صلابة فيكرز مع اختبار الصلادة النانوي بناءً على مساحة السطح المتوقعة الناتجة عن اختراق المتغلغل. أظهرت النتائج أنه يمكن ربط نتائج الصلابة النانوية وصلادة فيكرز. ومع ذلك، من أجل دقة الارتباط، يجب مراعاة القياس الدقيق للمساحة المتوقعة. أظهر Ti6Al4V أكبر فرق بين نتائج الصلادة الدقيقة والصلادة النانوية لكلا النتائج العملية والنظرية وبنسبة 43٪ و 37٪ على التوالي، بينما كان للنحاس أدنى نسبة وبنسبة 30٪ و 33٪.

1. INTRODUCTION

Hardness is one of the most important mechanical properties as it represents the material resistance to the applied loads. The hardness values of materials have long been used to describe their mechanical properties. New technological advancements have made depth-sensing instrumented micro and Nanoindentation tests widely available [1]. Nano-indentation is recognized as one of the techniques for evaluating the mechanical properties of materials even thin films, which are critical to the advancement of technology, and demand for the technique will continue to rise [2]. The advantage of Nanoindentation is the high resolution, which allows for the estimation of mechanical properties depending on the theories of analysis indentation using the theoretical framework that allows the measurement of a material's elastic and plastic characteristics regarding the indenter penetration depth and real contact area [3]. The correlation between mechanical properties and hardness value is known and established accurately. Shen and Tan [4] used Nanoindentation to examine the main mechanical properties such as yield strength and modulus of elasticity. This is to establish the link between Nanohardness values and elastic and plastic materials properties. In addition, Giannakopoulos and Suresh [5] developed a basic theoretical framework for sharp indentation and employed the inverse approach to derive material attributes via penetration depth and true contact area for a sharp indenter of Nanoindentation, which could provide enough information to determine the requisite qualities. Poon et al. [6] re-evaluated the assumptions utilized in calculating the linearly elastic material properties of an elastic-plastic indentation. Furthermore, the study provided a new way of estimating material yield stress using

Nanoindentation by directly measuring the anticipated contact area. On the other hand, the micro-hardness test can calculate only the hardness value in the micro-scale that can express the resistance of the material to penetrate the Vickers indenter that is directly proportional to the residual stress resulting from the deformation [7]. However, the use of any of the two methods is subject to the availability of the devices or the state of the testing area. Thus, it is crucial to comprehend how the outcomes of the two indenters relate to one another. However, it was found that the Vickers hardness value for the same material, is not identical to the Nanohardness value, according to earlier investigations and several studies have been conducted to establish this relationship, but none of them were successful in providing a clear correlation [8]. For instance, Yang et al. [9], investigated the correlations between Vickers and Nanohardness for different materials where a linear relationship had beenestablished, and a correlation factor was calculated. Also, LU et al [10] studied the relationship between the hardness tests in different scales and explain that the correlation between the hardness values does not correspond to the mathematical geometric relationship due to sink-in and pile-up effects resulting from the effect of the penetration of the indenter into the surface of the material. The two types of hardness become similar with this improved modification in the projected region of the Vickers hardness and Nanohardness. Therefore, the current work aims to continue exploring the relationship between Nanoindentation and Vickers micro-hardness methods to give flexibility of obtain as much information as possible about the examined material.

2. THEORETICAL ANALYSIS

The correlation between Nano-indentation and micro-hardness can be achieved based on indenter's projection area at same indentation depth. For micro scale hardness testing, a common method used is Vickers. While, for Nano-indentation test, a typically method used is Berkovich indenter tip [11].

Vickers hardness (HV) indenter consist from diamond pyramid with a square-based, thus it has four sided with center line to face angle α is 68°, as shown in Figure 1:

$$\mathrm{HV} = \frac{\mathrm{F(Peak\ force)}}{\mathrm{A\ (Area)}}\ (\mathrm{Kgf/mm^2}) \tag{1}$$

$$\mathrm{A} = \frac{\mathrm{d^2}}{2\times\sin 68^\circ}\ \mathrm{mm^2} \approx 1.8544\ \mathrm{d^2} \tag{2}$$

Where the indenter angle is 68° or 136/2, and A is the surface contact area of the resulting indentation, d is the average of indenter tip diagonals that equal to the $(d_1+d_2)/2$. By submitted equation 2 in 1:

$$\mathrm{HV} = \frac{2\mathrm{F}\sin 68^{\mathrm{o}}}{\mathrm{d^2}} \tag{3}$$

Or $$\mathrm{HV} \approx 1.8544\frac{\mathrm{F}}{\mathrm{d^2}}\ (\mathrm{Kg\ f/mm^2}) \tag{4}$$

Berkovich hardness (H) indenter consists also from diamond pyramid but with a triangle-based. So it has three sided only with centreline to face angle α is 65.3°, as shown in Figure 1:

$$\mathrm{H} = \frac{\mathrm{F(Peak\ force)}}{\mathrm{A\ (project\ contact\ area)}} \tag{5}$$

Where: A, or $\mathrm{A(h_p)}$ is the project contact area of the resulting indentation and equal to the $(d^2)/2$. Therefor:

$$\mathrm{H} = \mathrm{F/A(h_p)}\quad (\mathrm{MPa}) \tag{6}$$

$$\mathrm{H} = 2\mathrm{F}/\ \mathrm{d^2}\quad (\text{or } \mathrm{F} = \frac{\mathrm{H.d^2}}{2})\quad (\mathrm{MPa}) \tag{7}$$

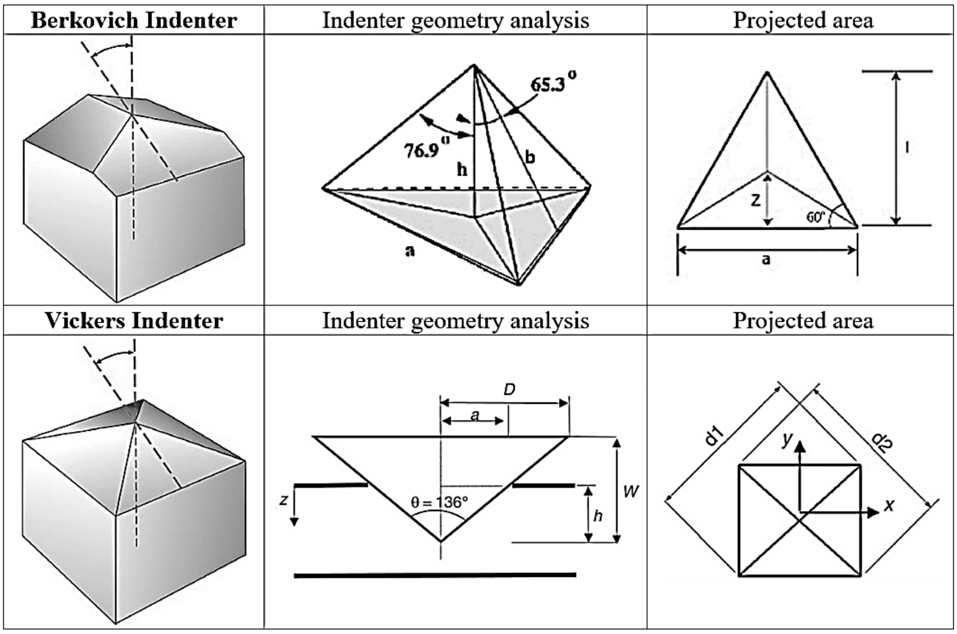


Figure 1. Berkovich and Vickers Indenters analysis [11].

The projected area of the tips for both Berkovich and Vickers indenters are designed to match each other at same indentation depth:

For Berkovich indenter: $A_{proj} = 3\sqrt{3h^2}.\tan^2 65.3^o = 24.56h^2$ (8)

For Vickers indenter: $A_{proj} = 4h^2 \tan^2 68^o = 24.50h^2$ (9)

By substitute (7) in (4):

$$HV(\frac{Kgf}{mm^2}) \approx 1.8544\frac{H}{2}(MPa) \quad (10)$$

$$\frac{2HV\left[\frac{Kgf}{mm^2}\right]\times 9.81\left[\frac{N}{Kgf}\right]}{1.8544} = H\ [Mpa] \quad \text{, (multiple by } 9.81\frac{N}{Kg\,f}) \quad (11)$$

$$\therefore\ H[Mpa] \approx 10.58\ HV\left[\frac{Kgf}{mm^2}\right] \quad (12)$$

$$\text{or, } HV\left[\frac{N}{mm^2}\right] \approx 94.518\ H[Gpa] \quad (13)$$

This finding is consistent with the theoretical models of [12]

3. EXPERIMENTAL WORK

The practical side included conducting micro and nanohardness tests for each of the applied materials at the same test area to ensure the precision in the results and compare it with the theoretical results by applying the correlation equations between the two testing methods.

3.1 Material

Ti6Al4V, commercially pure copper, and AISI 316L stainless steel are the metals used in this research. All the metals were received in the as-rolled and mill-annealed condition. Tables 1 and 2, respectively, list the chemical composition and mechanical properties of the three metals. The AMG Super alloys UK Limited Corporation conducted an analysis of the material's chemical composition.

Table 1. Chemical composition of the utilized materials.

Element (wt %)	Cr	Ni	C	Si	Mn	Mo	Cu	Co	Ti	Al	V	Fe
Ti6Al4V	--	--	--	--	--	--	--	--	Bal.	6	4	--
Cu	99.7	0.02						0.03	--	0.02	--	0.02
316L	16.63	10.14	0.03	0.46	1.32	1.87	0.67	0.11	--	--	0.07	Bal.

Table 2. Mechanical properties of ti6al4v, commercial pure cu, and AISI 316L [13-14].

Material	Tensile Strength, MPa	Yield Strength, MPa	Elongation %, (at 50 mm/min)	Hardness VH
Ti6Al4V	895	828	10	396
Cu	221	69	55	40
316L	485	170	40	220

3.2 Tests techniques and Indents maps

The DuraScan G5 hardness testing device was used to conduct the Micro-hardness test. Vickers hardness measurements were made in the middle of the metal in a two-dimensional grid with about 25 indents, forming a matrix with five rows and five columns. 68-degree angle Vickers hardness (HV) indenter was used on a pyramid-shaped diamond. The Triboscope Nano-mechanical Test Instrument, which was provided by the Hysitron Corporation, was used on the other side to conduct Nanoindentation testing. The test was conducted in the center of the metal with around 25 indents in the shape of a matrix with five arrows and five columns, with a 10 m gap between the indenters. In order to test the overall hardness values of the material, it is necessary to make sure it is not distorted or impacted by any residual stress. Every single indent received a fixed load of 5000 N applied with a 15-second dwell duration and a trapezium load and unload operation. In the current test, Berkovich indenter tip with a half angle of 65.35 degrees was employed [11].

As shown in Fig. 2, the Nanoindentation test involves pushing the indenter into the sample's surface to cause both elastic and plastic deformation of the material. The primary distinction between the micro and Nanoindentation setup tests is that the displacement h and the load L are continually and precisely measured in the Nanoindentation test. Values for Nanohardness are determined by analyzing the load P generated curve as a function of the depth of the indenter's penetration into the material h. In the load cycle, the load must reach its maximum value (P_{max}) in order to provide the planned contact area A_{max}. Next, it must be held and then unloaded. Equation 14 describes the curve during loading. A measurement of a material's resistance to indentation is the indentation curvature (C). The hardness value H from equation 15 is utilized to determine the curve's unloading portion. E from equation 16 is calculated using the unloading curve's slope S [5, 15].

$$Pmax = C\ .h_{max}^2 \tag{14}$$
$$H = P_{max}/A \tag{15}$$
$$E = S\ \sqrt{\pi}\ /\ 2\sqrt{A_{max}} \tag{16}$$

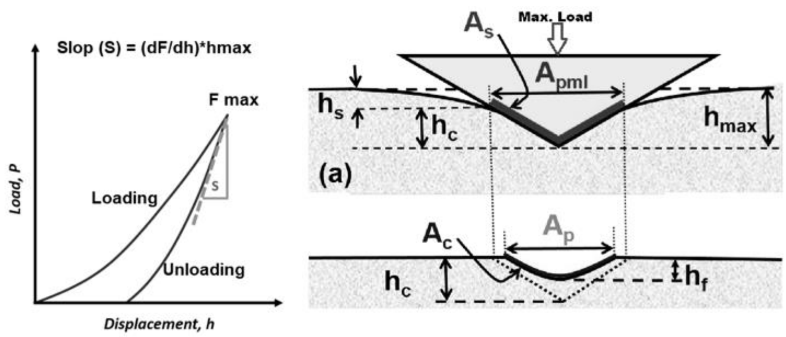
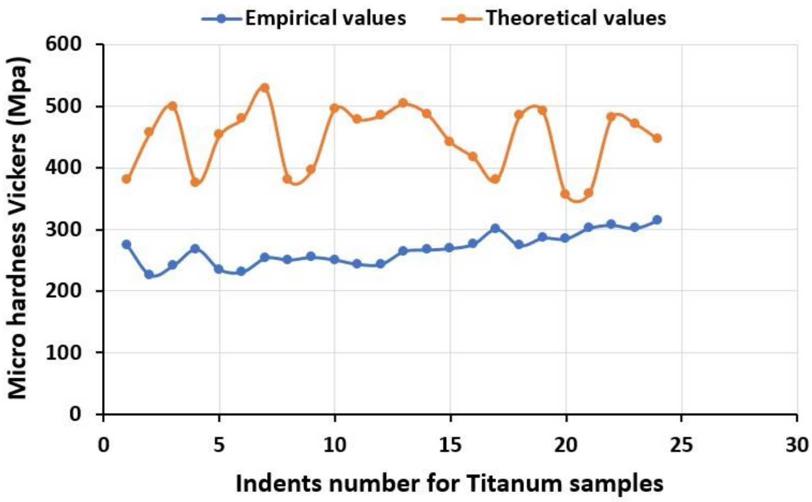


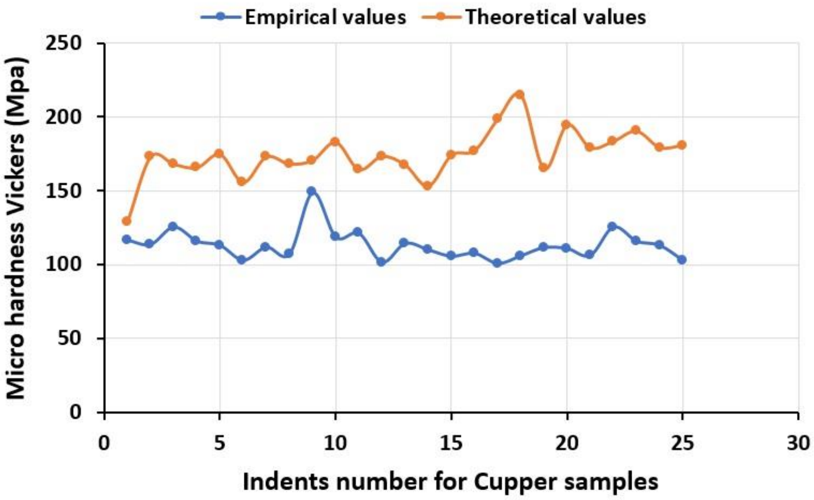
Figure 2. Nanoindentation load cycle and elastic-plastic deformation at the maximum load. Adapted from [16].

It is worth noting that the contact area, projected area, and, in some circumstances, the indenter's penetration depth can all be used to determine the hardness. Due to these variations induced by the influence of elasticity and plasticity of the material impact, it is quite hard to compare hardness evaluated by various methods and scales [16].

4. RESULTS AND DISCUSSIONS

Figures 3 and 4 show the empirical and theoretical values that were measured and calculated using hardness tests for both micro and Nano scales and for all materials used in the current research (Ti, Cu, and stainless steel). It can be observed from the figures that the Micro-hardness values are closer and more stable than the Nanohardness values. That might be due to the Vickers indenter tip size is bigger than the Berkovich indenter tip and therefore it is not greatly affected by the test location. Despite the differences in hardness values, this may be normal because the hardness depends on the state of the test location, which is affected by the type of grains and grains boundaries or exists of impurities and defects in the material. Also, the theoretical values that were calculated from the conversion equations (the relationship between the two scales) took the same trend as the measured practical values, but they do not match them because of the approximation that was adopted in calculating the projection area. Generally, the measured and calculated hardness values for titanium material are the highest, followed by steel, then copper, and this is identical to what is known.





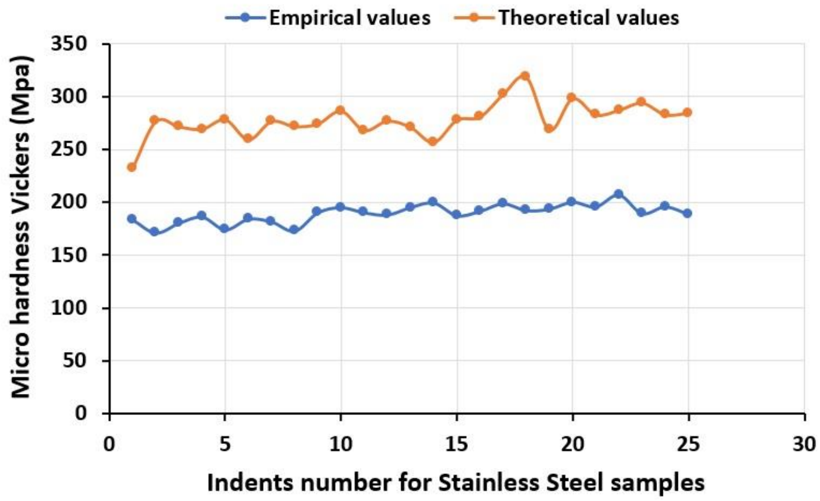
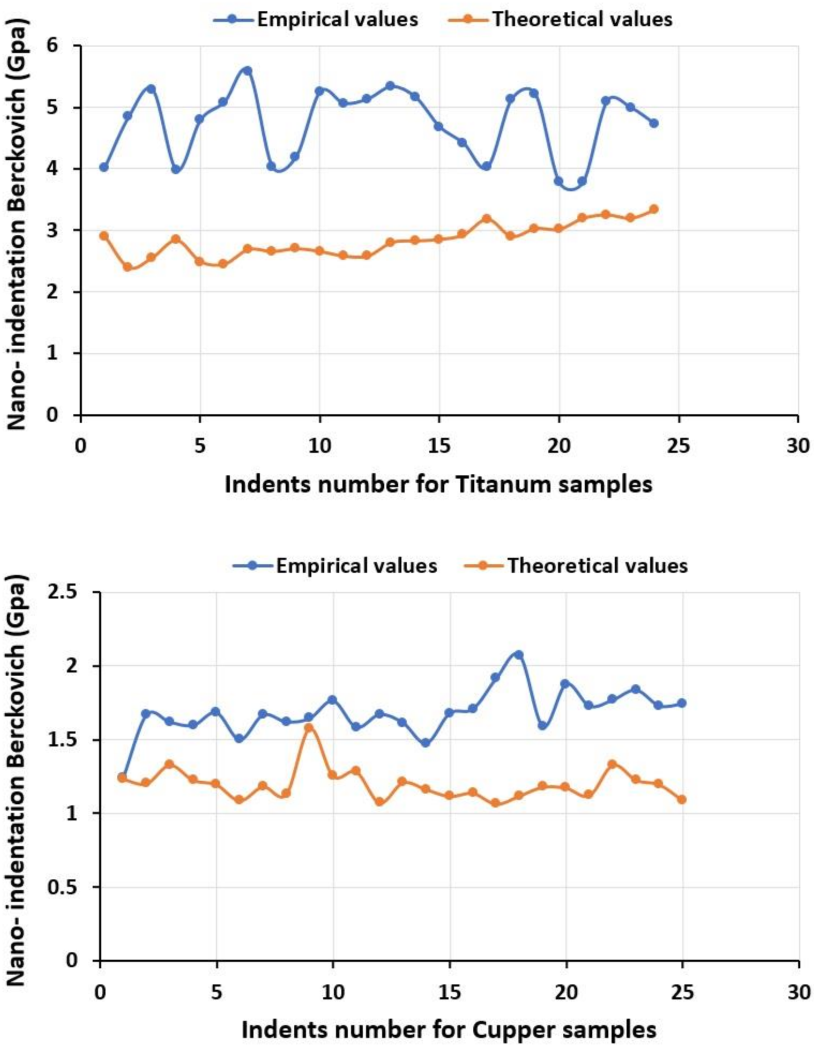


Figure 3. Empirical and Theoretical of Micro-hardness values for samples (Ti, Cu, St.St.) respectively.



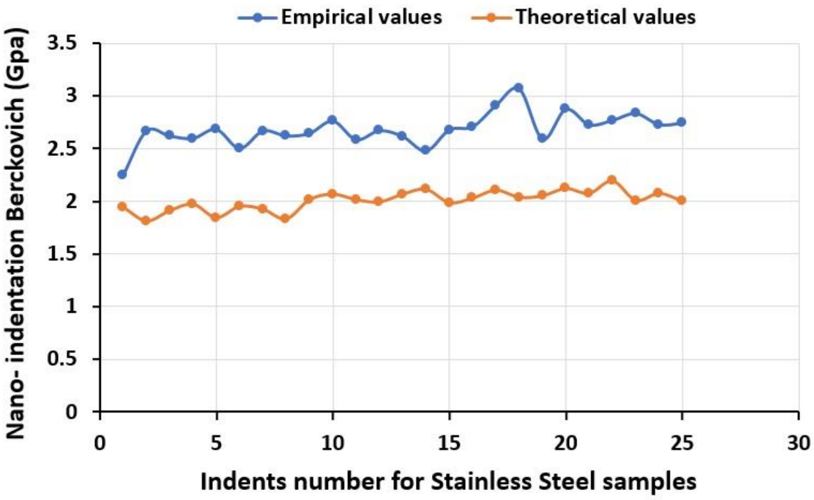


Figure 4. Empirical and Theoretical of Nano hardness values for samples (Ti, Cu, St.St.) respectively.

It can be seen obviously that there are clear convergence in the theoretical and experimental figures between the two scales (Nano and Micro) and vice versa. This illustrated that the equations used to make the correction between the two scales are correct, but at different values, which means that only correction factor is required. Figure **5** shows the variation in measuring and calculating the experimental and theoretical values of the average Microhardness for samples (Ti, Cu, St.St.) using the standard deviation. The figure shows that the differences are uneven between the applied materials, as they were higher in titanium, especially when calculating the theoretical values, this means that the amount of disparity increases with the increase in hardness values of the material. The figure (5) also shows that there is some disparity between the theoretical and practical values of all measured samples, which is reflected in the accuracy of the equations and hypotheses that were adopted in the study. This might be due to the dependence of the approximation in matching the projected area of each of the Micro and Nano indenters at the same depth of penetration despite the existence of a slight difference in the angle of the indenters tips (68^o for Vickers and 65.3^o for Berkovich, or in the number of faces (Berkovich indenter consists pyramid but with a triangle-based and Vickers hardness indenter consists of a pyramid with a square-based, thus it has four-sided). Therefore, the stress distribution when applied to the load test by the indenter on the surface of the material causes elastic and plastic deformation that directly affects the material's resistance to penetration and the calculated hardness values.

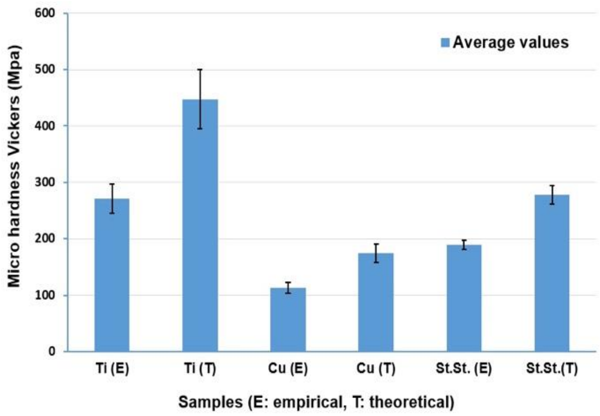


Figure 5. Uncertainty of empirical and theoretical of Microhardness average values for samples (Ti, Cu, St.St.).

Fig. 6 shows the variation in measuring and calculating the experimental and theoretical average Nanoindentation values for samples (Ti, Cu, St.St) using the standard deviation. The figure illustrates that the differences are uneven between experimental and theoretical results for the applied materials, as they were higher in titanium, for both experimental and theoretical values, this means that the amount of disparity increases with the increase in the hardness values of the material. This means that the type of tested material can affect the measured projected area due to the difference in the resulting elastic and plastic deformation of the material when penetrating and removing the indenter.

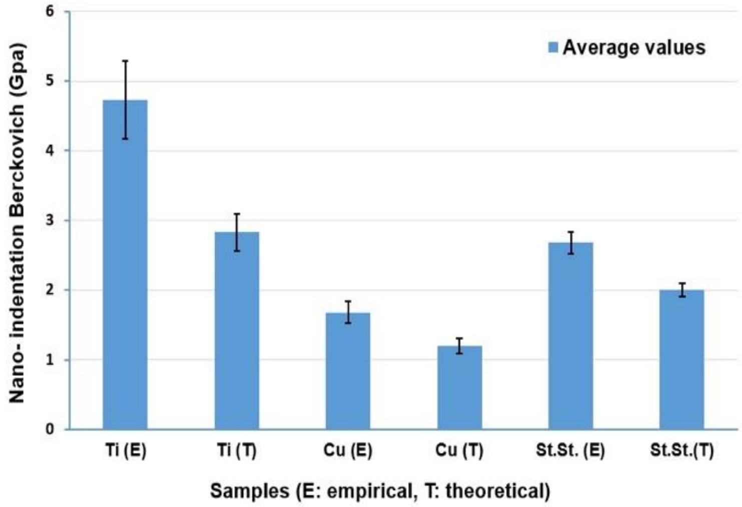


Figure 6. Uncertainty of empirical and theoretical Nanoindentation average values for samples (Ti, Cu, St.St.).

Fig. 7 shows the accuracy ratio of the conversion between experimental and theoretical values for the average micro and Nanohardness of the samples (Ti, Cu, and St.St) It is noted that the conversion ratio between the two scales is disparity, as it is high with titanium metal relative to the rest of the metals, and this means that the correlation coefficient between the hardness values has limited applications, but it parallel to the practical values. Unexpectedly, the conversion rate is slightly lower in stainless steel than in copper, and this gives a good impression of the stability of the relationship between hardness values.

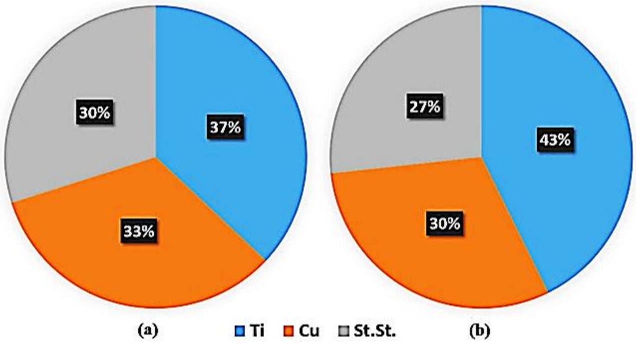


Figure 7. The accuracy (differences) of converting between the empirical and theoretical results for (Ti, Cu, St.St.) samples (a) Microhardness values (b) Nanoindentation values.

5. CONCLUSION

Linking the results of different types of hardness tests has an important role in improving the accuracy of the testing results. Although the operating conditions and hardness test settings such as the applied force, indenter type or size, and tip angle, are different, the results are supposed to be identical or close and represent the real specifications of the examined material. A comparison investigation has been made between the practical results of Vickers's Microhardness and the Berkovich indenters' Nanoindentation for three different materials. Then a mathematical model was applied based on the projected surface area resulting from the penetration of the indenters. Accordingly, the following conclusions were reached:

- It is possible to link the results of micro and Nano hardness tests depending on the projected area resulting from the penetration of the indenters or the indenter geometry.
- The accuracy of the correlation between micro and Nano hardness tests increases with increasing the accuracy of calculating the projected area due to the influence of material elastic and plastic properties.

The differences between experimental and theoretical results increase with increasing the hardness of the tested material as Ti6Al4V showed the highest difference of 43% and 37% between experimental and theoretical results for the Microhardness and Nanohardness tests, respectively. While Cu showed the lowest differences with only 30% and 33%.

REFERENCES

1. Dao, M., et al. "Computational modeling of the forward and reverse problems in instrumented sharp indentation." Acta materialia 49.19 (2001): 3899-3918.
2. Sawa, Takeshi. "Correlation between Nanoindentation test result and Vickers hardness." Proceedings of the IMEKO. 2010.
3. Wego Wang, “Reverse engineering, technology of reinvention,” Taylor and Francis Group, LLC, 2011.
4. Y. L. Shen and X. H. Tan, “On the correlation between hardness and yield strength in multilayered elastic-plastic materials,” J. Mater. Sci., vol. 40, no. 17, pp. 4683–4686, 2005.
5. A. E. Giannakopoulos and S. Suresh, “Determination of elastoplastic properties by instrumented sharp indentation,” Scr. Mater., vol. 40, no. 10, pp. 1191–1198, 1999.
6. B. Poon, D. Rittel, and G. Ravichandran, “An analysis of Nano indentation in elastic-plastic solids,” Int. J. Solids Struct., vol. 45, no. 25–26, pp. 6399–6415, 2008.
7. A. Pulnikov, P. Baudouin, and J. Melkebeek, “Induced stresses due to the mechanical cutting of non-oriented electrical steels,” J. Magn. Magn. Material, vol. 254–255, pp. 3, 2003.
8. Mphahlele, M. R., et al. "A correlation between nano and micro-hardness properties of TiN nanoparticles strengthened SAF 2205." IOP Conference Series: Materials Science and Engineering. Vol. 499. No. 1. IOP Publishing, 2019.
9. Yang, Yitao, et al. "A correlation between micro-and Nano-indentation on materials irradiated by high-energy heavy ions." Journal of Nuclear Materials 498 (2018): 129-136.
10. Youping, L. U., et al. "Conversion between Vickers hardness and Nanohardness by correcting projected area with sink-in and pile-up effects." Plasma Science and Technology 22.6 (2020): 065602.
11. Anthony C. Fischer-Cripps, "Nanoindentation", Third Edition, Mechanical Engineering Series, springer, 2011.
12. Q. Luo and M. Kitchen," Microhardness, Indentation Size Effect and Real Hardness of Plastically Deformed Austenitic Hadfield Steel" MDPI, Materials 2023, 16, 1117. https://doi.org/10.3390/ma16031117.
13. A. H. Volume, "2: Properties and Selection: Nonferrous Alloys and Special-Purpose Materials," ASM international, pp. 889-896, 1990.
14. B. ASTM, "265-99—Standard Specification for Titanium and Titanium Alloy Strip, Sheet, and Plate," West Conshohocken, Pennsylvania: American Society for Testing and Materials, 1999.
15. T.A. Venkatesh, K.J. Van Vliet, A.E. Giannakopoulos and S. Suresh, “Determination of elastoplastic properties by instrumented sharp indentation: guidelines for property extraction,” Scripta mater. 42 (2000) 833–839, Pergamon, elsevier, 2000.
16. E. Broitman, "Indentation Hardness Measurements at Macro, Micro, and Nanoscale: A Critical Overview" Tribol Lett, Springer, 65:23, 2017.