Nanomechanical Method Development for UNiT: Universal Nanoindentation Toolkit

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A Major Qualifying Project

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Abstract

This project aims to create a general evaluation toolkit for traditional nanoindentation data. While similar toolkits have been developed in the past, such as NIGET, they lack many important and relevant methods which we plan to implement. This project plans to implement three less accessible data analysis methods: Nix-Gao's relation of hardness and depth, Dao et al.'s evaluation of stress-strain curves, and Chen-Bull's "Relation between the ratio of elastic work to the total work of indentation and the ratio of hardness to Young's modulus for a perfect conical tip." Once complete, this toolkit will allow users to calculate parameters such as energy density as fracture toughness for ductile materials, hardness in the limit of infinite depth, and inversely estimated stress-strain curves from single indentation tests.

How this project is carried out is split into two components: data science and materials science. The data science component is responsible for implementing algorithms, methods, and other technical pieces into the kit. More specifics can be found in a separate report located in Worcester Polytechnic Institute's Major Qualifying Project archive, as a 2022 MQP titled *UNiT: A Universal Toolkit for Metallic Nanoindentation Data Analysis and Mechanical Properties Exploration* by Aaron Krueger and Eric Schmid, in the Computer Science department. The materials science component of this project is highlighted in this paper. This portion evaluates and verifies the data science methods, and nanoindentation data is gathered. Then, extensive tests are conducted using this nanoindentation data, which is related to and compared with the algorithms used in the data science segment.

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Introduction

The need for vetted, validated, and verified methods of quantifying mechanical properties of engineering materials through high-throughput experimental techniques coupled with data-driven solutions as well as methods of data analysis have become ever more relevant within the materials science, processing, and engineering communities. The need for high-throughput experimental modalities for acquiring datasets comprised of materials properties stems from the cost-prohibitive, labor-intensive, and time-consuming nature of conventional approaches to acquiring mechanical properties as a function of processing history and composition. For example, Profilometry-based Indentation Plastometry (PIP) has emerged as one such approach to technique implementation within the field of materials science and engineering that significantly reduces the amount of time, effort, cost, and material required to obtain nominal stress-strain curves (up to the point of ultimate tensile stress) as well as true stress-strain curves with a simple indentation approach (Tang et al., 2021). However, PIP testing is only one such tool. Thus, implementing additional tools that can reduce the time and effort needed to gather and analyze data would be particularly useful in materials science and open new doors in research.

Nanoindentation is a method of finding the hardness and elastic modulus of materials by applying a load to create a sub-micron indent on the surface of a material (Campbell et al., 2019). Creating a tool that can evaluate the properties of materials can be highly beneficial in many aspects, including reducing testing and data analysis time, primarily since nanoindentation provides a massive amount of data in one single test. Take NIGET, or Nanoindentation General Evaluation Toolkit, which is a toolkit that can analyze data gathered through nanoindentation to infer material properties. It uses well-known and standardized methods such as the Oliver-Pharr method, pop-in detection, and elastic and plastic work calculations to evaluate nanoindentation data (Campbell et al., 2019). With the success of NIGET, this kind of tool proves to be helpful in the field - since nanoindentation alone is only typically used to gather hardness and modulus of elasticity (Schuh, 2006). Any additional properties, such as yield and tensile strength, dislocation density, and toughness, must be calculated manually by the user. These properties are a limitation of NIGET that we plan to implement into UNiT and maximize its usability. A tool like NIGET offers greater insight into this nanoindentation data without the need for manual calculations. Furthermore, NIGET has the added measure of showing the uncertainties of standard evaluation methods – which many researchers find vital (Campbell et al., 2019).

The tool we are developing, UNiT (Universal Nanoindentation Toolkit), will take many of the most beneficial aspects of NIGET and add additional features focused on data analysis. Our tool will reduce or replace many tests using extensive algorithms. We plan to implement several testing methods into UNiT, used to gather many common properties of a material. A few of these methods are tensile testing and compression testing for stress-strain curves, Vickers hardness testing across the entire range of applied loads, and indentation plastometry for stress-strain curve extraction wherein necking is neglected. With UNiT, bulk levels of data can be analyzed quickly, and the properties gathered from these tests can all be found with one tool.

Background

Nanoindentation

Nanoindentation is a method of obtaining force-displacement data via load-controlled or displacement-controlled electromotive or MEMs actuators (Nanoscience Instruments, 2022). Engineers and researchers typically employ nanoindentation to calculate the modulus of elasticity, mean contact pressure, or hardness as a function of load or depth, among other properties, such as fracture toughness in the case of brittle materials (Oliver & Pharr, 1992). While there are many ways in which a nanoindenter can be used, depending on the system available and hardware capabilities associated with a given nanoindenter, nanoindentation has been recognized as being industrially significant enough to warrant standard formulation by ASTM (Nanoscience Instruments, 2018) and ISO (Nanoscience Instruments, 2018). During nanoindentation testing, force-displacement data is constantly collected throughout a given load cycle or individual test within a given experimental batch of measurements, allowing load-depth curves to be generated via the recorded force-displacement data. The modulus of elasticity, mean contact pressure, or hardness can be calculated using these curves. Given the high-throughput nature of many modernized nanoindenter systems, the ability to collect a wide range of interpretable datasets makes nanoindentation data analysis through data science techniques and algorithms ripe for application. For example, nanomechanical mapping methods have been complimented by data science and analysis techniques such as neural networks (Lee et al., 2019), k-means clustering (Sousa et al., 2022), and probability density function deconvolution (Sousa et al., 2022).

In addition to purely data-driven analytics, algorithmic load-displacement nanoindentation data analysis can be implemented relatively quickly to explore non-traditional material property quantification to expand nanoindentations utility within the materials science and engineering discipline. For example, Weaver et al. presented a framework for estimating spherical nanoindentation stress-strain curves for metallic material systems in his SPIN program (Weaver et al., 2016). At the same time, Kossman and Bigerelle presented a deep learning approach to pop-in detection in nanoindentation data (Kossman & Bigerelle, 2021). Mercier et al. has also implemented a technique for automated pop-in detection in a MATLAB-based PopIn Toolbox (Mercier et al., 2015). As illustrated by said examples, nanoindentation is well suited for further consideration through the lens of algorithmic data analysis to automatically extract more material properties from instrumented indentation testing (IIT) data. Nanoindentation-unveiled properties sought after by the materials science and engineering community include dislocation density, depth-independent hardness, yield strength, stress-strain curves, strain-rate sensitivity, ductility, toughness, and more (Nanoscience Instruments, 2022).

The process of using a nanoindenter is relatively simple and can take minutes to hours for data acquisition and collection, depending on outside parameters such as the level of background noise and the test method implemented. For example, when using KLA Instruments nanoindentation systems, the NanoBlitz 3D method can record data at a rate of 1 indent per second. On the other hand, dynamic nanoindentation tests using an iMicro, G200, iNano, or another such system, can take multiple hours for 30 indentation force-displacement datasets to be measured. From an operational perspective, mounted and polished samples are first loaded into a nanoindenter, followed by test parameter selection, such as the number of indents and suitable thermal drift rates. Next, the system takes each indent and populates the data set. It finally begins to unload once the tip has reached either the input load or depth. To better conceptualize hardware assembly for a typical load-controlled nanoindentation system, readers may consult Figure 1.

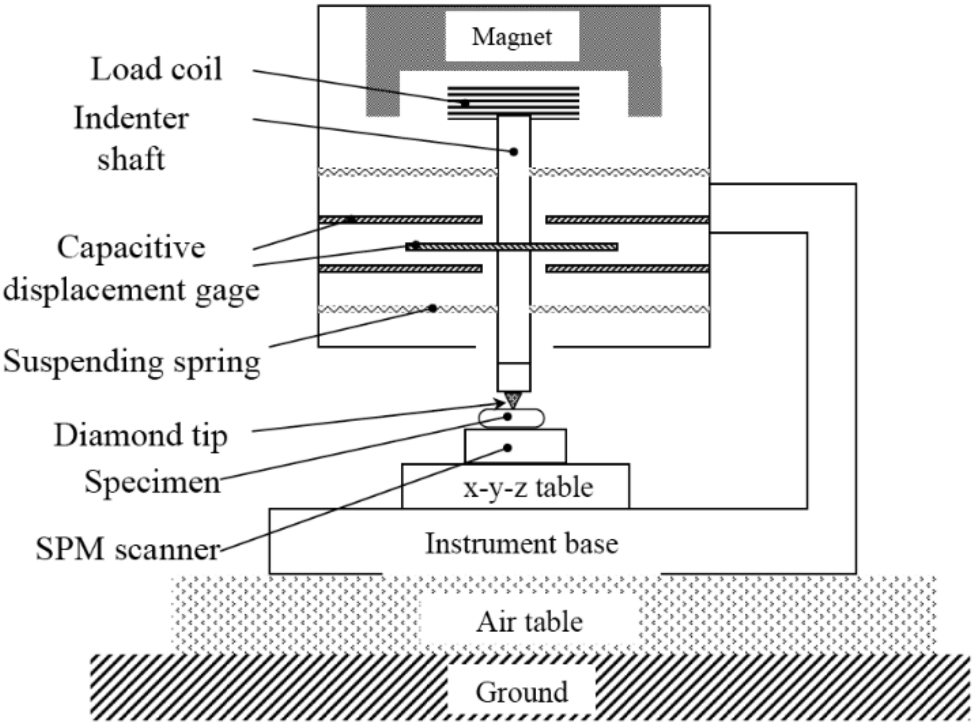


Figure 1: Assembly and hardware for a typical load-controlled nanoindentation system (Write et al., 2013)

Nanoindentation was chosen as the testing method to implement UNiT because it works for a wide range of materials – even when the grains are too small to be imaged well (Oliver & Pharr, 1992). Many other tests used imaging to measure material properties, and small grain size can be a source of measurement issues. Since it has also been in development for many years and has shown itself to be reliable, it proves to be a great candidate for our tool. As mentioned before, the bulk data set that nanoindentation creates is another advantage of this testing method. Bulk data gives many reference points to utilize the same data to analyze more material properties. These characteristics of nanoindentation have made it a suitable match for the work we would like to implement with UNiT.

Current Data Analysis Methods

Material Properties

The properties of a material are its defining characteristics that ultimately specify how suitable the material is for an application. Thus, there is a constant drive to find and create materials that have the best properties for a situation in research. The following are essential properties in materials science, which UNiT will calculate: hardness, quantification of statistically stored dislocations, energy density, yield strength, ultimate tensile strength, and stress-strain behavior. All these properties have multiple methods in which they will be collected to validate the results output by UNiT.

Data Collection and Verification Methods

The goal of UNiT is for each of the properties mentioned above to be collected via nanoindentation, with our tool being the steppingstone between nanoindentation and these properties. Without UNiT, the properties are found through many different testing methods that can be timely to complete. While our tool may not wholly replace current methods for finding material properties, it can reduce the time needed to evaluate the feasibility of particular materials for a given application. Each material property implemented into this toolkit has its testing method, which we will also be using to verify the accuracy of our tool.

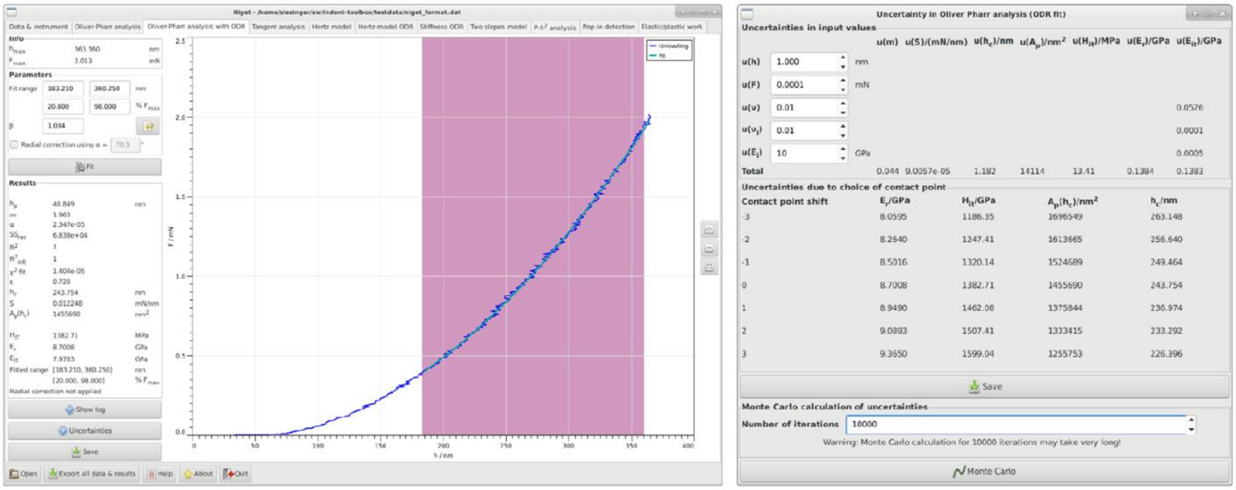
Hardness can be measured in many ways. However, the two standard methods are Brinell and Vickers hardness testing. Brinell hardness testing works by making small impressions into a metal, while a microscope measures the indent size. Vickers testing works similarly to Brinell; however, it places more minor indents, which can be more accurate as it focuses on microelements within the material's surface. Indentation plastometry, an indentation test that finds load vs. displacement data, will also be used with finite element analysis through the built-in SEMPID software to verify Brinell hardness further (Hughbanks, 2020). Both Brinell and Vickers hardness testing is widely used and trusted methods to find the hardness of materials and will provide validation results for our toolkit with high confidence.

Yield strength, tensile strength, and stress-strain relations can also be validated with the above three methods of Charpy impact testing, toughness-ductility relations, and indentation plastometry. Aside from that, we will also use existing tensile test data, literature, flat-punch nanoindentation stress-strain analysis, and empirical relations between hardness and strength and the Hall-Petch relation.

Nanoindentation General Evaluation Tool (NIGET)

NIGET is a tool that has a similar purpose as our tool UNiT; both aim to evaluate nanoindentation data to expand its use. NIGET implements several methods such as the Oliver-Pharr method, pop-in detection, linear stiffness determination, and elastic and plastic work calculations. This data is gathered through several relations, all of which are implemented into the NIGET toolkit. This data is quickly recorded and processed by using the C programming language.

NIGET has inspired UNiT since having free-to-use software providing valuable data with one machine and tool is extremely useful. The idea can be expanded indefinitely by adding any potential method, but a few should be included first; integrating more valuable and challenging to gather methods into this software can reduce any excess testing and quickly prove to be helpful. Figure 2 below highlights the ODR curve fitting tool and uncertainty toolbox (Write et al., 2013).



Info

h_{max}	363.360	nm
F_{max}	2.013	mN

Parameters

Fit range	183.210	360.250	nm
	20.000	98.000	% F_{max}
β	1.034		

☐ Radial correction using α = 70.3 °

Results

h_p	40.849	nm
m	1.963	
α	2.347e-05	
SS_{res}	6.838e+04	
R^2	1	
R^2_{adj}	1	
χ^2 fit	1.404e-05	
ε	0.728	
h_c	243.754	nm
S	0.012248	mN/nm
$A_p(h_c)$	1455690	nm²
H_{IT}	1382.71	MPa
E_r	8.7008	GPa
E_{IT}	7.9783	GPa
Fitted range	[183.210, 360.250]	nm
	[20.000, 98.000]	% F_{max}

Radial correction not applied

Uncertainties in input values

			u(m)	u(S)/(mN/nm)	$u(h_c)$/nm	$u(A_p)$/nm²	$u(H_{it})$/MPa	$u(E_r)$/GPa	$u(E_{it})$/GPa
u(h)	1.000	nm							
u(F)	0.0001	mN							
u(ν)	0.01								0.0526
$u(\nu_i)$	0.01								0.0001
$u(E_i)$	10	GPa							0.0005
Total			0.044	9.0057e-05	1.182	14114	13.41	0.1384	0.1383

Uncertainties due to choice of contact point

Contact point shift	E_r/GPa	H_{it}/GPa	$A_p(h_c)$/nm²	h_c/nm
-3	8.0595	1186.35	1696549	263.148
-2	8.2640	1247.41	1613665	256.640
-1	8.5016	1320.14	1524689	249.464
0	8.7008	1382.71	1455690	243.754
1	8.9490	1462.08	1375844	236.974
2	9.0893	1507.41	1333415	233.292
3	9.3650	1599.04	1255753	226.396

Monte Carlo calculation of uncertainties

Number of iterations 10000

Warning: Monte Carlo calculation for 10000 iterations may take very long!

Figure 2: NIGET's ODR curve fitting tool and its uncertainty toolbox

Material Selection

It is vital to gather a range of metals to validate that the tool gives accurate data analysis when selecting materials. We selected several different aluminum and steel alloys and copper for our purposes. We chose three different aluminum alloys: Al 6061, Al 3003, and MIC 6. Three different steel alloys were chosen as well, being 304 Stainless Steel, 4340 Steel, and Annealed Maraging 350 Steel. Lastly, oxygen-free low conductivity (OFHC) copper was chosen. These metals have different material properties, which help provide a well-rounded pool of materials for testing and validation purposes. Various kinds of metals and alloys with ranging material properties help verify that UNiT can be universal to a wide range of materials. Figures 3 and 4 below show the materials' true range through stress-strain graphs.

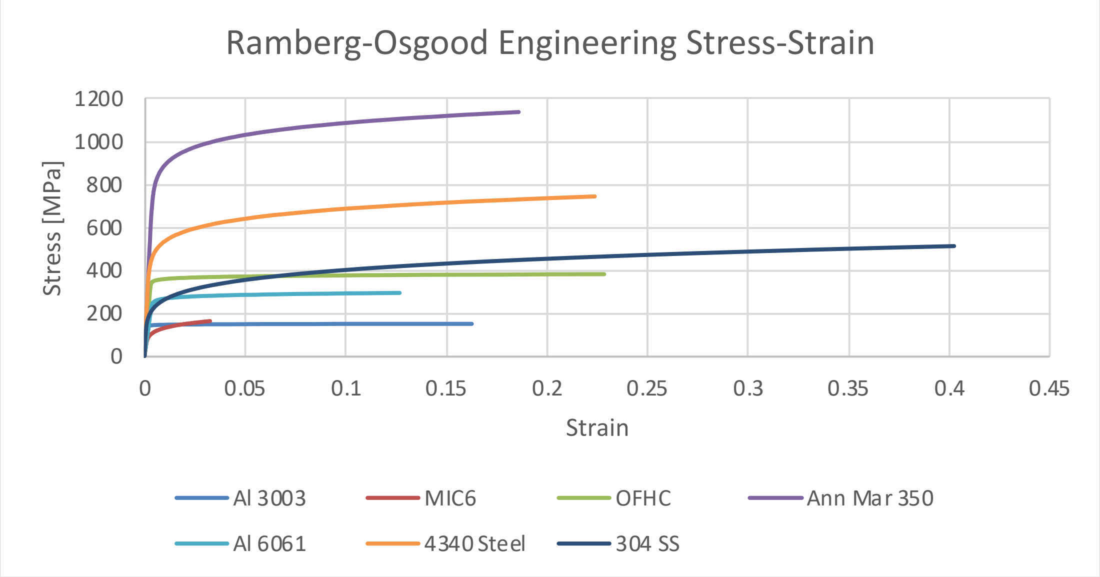


Figure 3: Engineering stress-strain graph formulated using the Ramberg Osgood method containing all the materials used to test UNiT

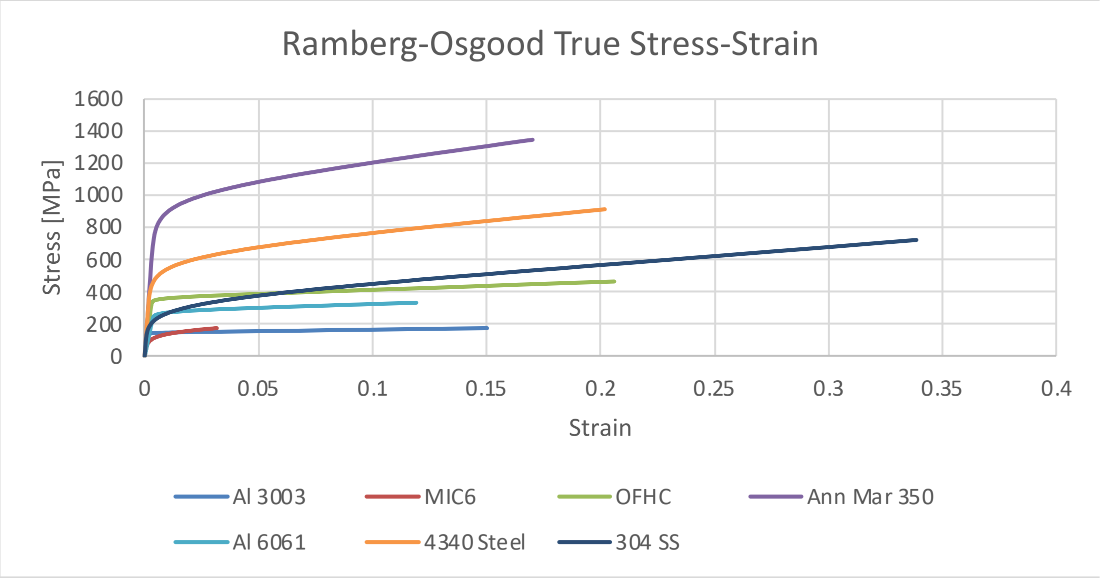


Figure 4: True stress-strain graph formulated using the Ramberg Osgood method containing all the materials used to test UNiT

Algorithms in UNiT

The relations we chose to implement into UNiT are Nix-Gao's “Indentation size effects in crystalline materials,” Dao et al.’s “Computational modeling of the forward and reverse problems in instrumented sharp indentation,” and the Chen Bull’s “Relation between the ratio of elastic work to the total work of indentation and the ratio of hardness to Young’s modulus for a perfect conical tip." These were chosen to gather the specific properties that UNiT provides, as well-tested and reliable methods.

Algorithm 1: Determining Vickers Hardness and h*

The Nix-Gao Model,

$$\frac{H}{H_o} = \sqrt{1 + \frac{h^*}{h}}$$

Equation 1 – The original Nix Gao method of determining Vickers Hardness

Calculates hardness at an infinite depth or actual hardness. In the equation written above, H is the hardness for a given depth of indentation, h, and $h*$ are a characteristic length that depends on the shape of the indenter, the shear modulus of the material, and the hardness in the limit of infinite depth. UNiT is capable of determining both H_0 and $h*$ given any nanoindentation dataset containing hardness, H, and depth, h, readings and simply requires the user to input values for a material's shear modulus, μ, and Burgers vector, b. The user is also given the option to input a value θ that represents the angle between the surface of the indenter and the plane of the surface. θ is a necessary value to calculate the contact radius of the indenter and the material (2) UNiT also provides a default of $\theta = 65.03°$, the angle for common Berkovich tips.

$$a = \frac{h}{\tan(\theta)}$$

Equation 2 – The calculation of a, contact radius

This same model can calculate the quantification of statistically stored dislocations with the following equation:

$$\rho_s = \left(\frac{H_0}{3\sqrt{3\alpha\mu b}}\right)^2$$

Equation 3 - The calculation of ρ_s, the density of statistically stored dislocations

$$L_s \cong \sqrt{\frac{1}{\rho_s}}$$

Equation 4 - The calculation of L_s, the mean spacing between statistically stored dislocations

UNiT also performs further calculations to determine the total dislocation density of the indentation, ρ_T, which requires the calculation of geometrically necessary dislocations, ρ_G (5 and 6).

$$\rho_G = \frac{3h}{2ba^2}$$

Equation 5 - The calculation of ρ_G, the geometrically necessary dislocations

$$\rho_T = \rho_G + \rho_s$$

Equation 6 - The calculation of ρ_T, the total dislocation density of the indentation

A secondary method also exists to determine hardness in the limit of infinite depth, as described in “On the breakdown of the Nix-Gao model for indentation size effect” (7) (Haušild, 2021).

$$H = H_0 \sqrt{1 + \frac{h^*}{h}\left(1 + re^{-\frac{h}{h_1}}\right)^{-3}}$$

Equation 7 - The calculation of Vickers hardness using Hausild’s method

The variables r and h_1 are described as fitting parameters, meaning UNiT must solve for them based on other available data. In this case, H is the dependent variable, and h, H_0, and h^* are independent variables. Using a curve fit function, UNiT can determine the values of r and h_1 and subsequently use their values in its calculation of hardness in the limit of infinite depth. In this case, the fitted function is that in (7), uses h, h^*, H_0, and H values to determine the values for r and h_1.

Algorithm 2: Evaluating Stress-Strain Curve

Dao et al.’s 2001 paper “Computational modeling of the forward and reverse problems in instrumented sharp indentation” describes a forward and reverse method by which users can calculate stress and strain values given nanoindentation data (Dao et al., 2001). While both methods are valuable, the team decided to use the reverse analysis method as it proved more applicable to UNiT’s domain and required less user input to operate than the forward analysis method. UNiT takes h (depth), P (load), E (Young’s modulus), and ν (Poisson’s ratio for the material) as user input. P and h form a load-depth curve, which, in nanoindentation applications, describes the general path of the indenter during its loading and unloading phases (Figure 5).

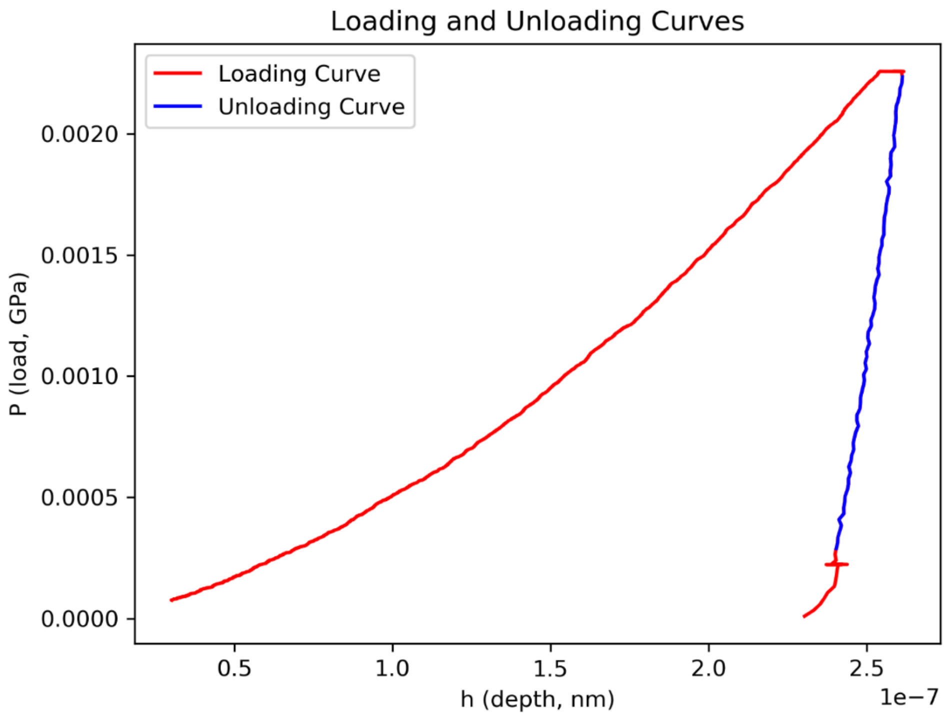


Figure 5: The load-unload curve created by h and P values calculated during a nanoindentation test

Given this input, UNiT can calculate several other parameters for the data, including stress and strain values and computations specified in Dao et al.'s paper required for analysis. To achieve this, UNiT once again utilizes curve fitting functions like the, as several of the equations cannot be solved without fitting for specific parameters. UNiT starts by using the unloading curve created by h and P to solve for the residual depth after indentation, h_f, when $P = 0$. This function fits an n-degree polynomial to x and y data, which, in this case, is used to solve for where $P = 0$. UNiT then solves the loading curvature coefficient C based on h and P (8).

$$P_m = C * h_m^2$$

Equation 8 - Kick's Law for determining loading curvature

Next, UNiT calculates E^*, a value representing the reduced Young's modulus of the material (9). Finally, UNiT provides default values for ν_i and E_i based on a diamond indentation tip, a standard tip used during nanoindentation.

$$E^* = \left(\frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}\right)^{-1}$$

Equation 9 - The calculation of E^*

Using E^* and h_f, UNiT determines the average pressure p_{ave} before further calculating stress, σ, and strain, ϵ, data values. Specifically, UNiT calculates values for $\sigma_{0.082}$ and $\sigma_{0.033}$, which are described in Π_7 (10) and Π_1 (11) respectively in Dao et al.'s paper (Dao et al., 2001). In this case, UNiT uses a curve fit to solve for both $\sigma_{0.082}$ and $\sigma_{0.033}$, as all other variables are already known in Π_7 and Π_1 but cannot be solved outright without the use of fitting.

$$\frac{p_{ave}}{\sigma_{0.082}} = -15.4944\left(\frac{\sigma_{0.082}^2}{{E^*}^2}\right) - 15.1699\left(\frac{\sigma_{0.082}}{E^*}\right) + 2.7497$$

Equation 10 - Dao et al.'s Π_7, used to solve for $\sigma_{0.082}$

$$\frac{C}{\sigma_{0.033}} = -1.131\left(\ln\left(\frac{E^*}{\sigma_{0.033}}\right)\right)^3 + 13.635\left(\ln\left(\frac{E^*}{\sigma_{0.033}}\right)\right)^2 - 30.594\left(\ln\left(\frac{E^*}{\sigma_{0.033}}\right)\right) + 29.267$$

Equation 11 - Dao et al.'s Π_1, which is used to solve for $\sigma_{0.033}$

Using these values, UNiT once again utilizes a curve fit to solve for the initial yield stress σ_y, which it can use to calculate further ϵ_y (12).

$$\sigma_{0.033} = \sigma_y\left(1 + \left(\frac{E}{\sigma_y}\right) * 0.033\right)^n$$

Equation 12 - The equation used to solve for σ_y

UNiT then uses $\sigma_{0.033}$ and E^* to solve for the strain hardening exponent n, which must be in the range of 0 to 1 and is calculated through Dao et al.'s Π_2 formula (13).

$$\begin{aligned}\Pi_2 = {} & (-1.40557n^3 + 0.77526n^2 + 0.15830n - 0.06831)\left[\ln\left(\frac{E^*}{\sigma_{0.033}}\right)\right]^3 \\ & + (17.93006n^3 - 9.22091n^2 - 2.37733n + 0.86295)\left[\ln\left(\frac{E^*}{\sigma_{0.033}}\right)\right]^2 \\ & + (-79.99715n^3 + 40.55620n^2 + 9.00157n - 2.54543)\left[\ln\left(\frac{E^*}{\sigma_{0.033}}\right)\right] \\ & + (122.65069n^3 - 63.88418n^2 - 9.58936n + 6.20045)\end{aligned}$$

Equation 13 – Dao et al.'s Π_2, which is used to solve for n

Lastly, UNiT begins creating the material's stress-strain curve by calculating the strength coefficient R (14).

$$\sigma = R\epsilon^n$$

Equation 14 - Calculation of R, the strength coefficient, and the calculation of σ where $\sigma \geq \sigma_y$

This formula is also used in the following system of equations to create and output the final stress-strain curve for the data. Specifically, σ values less than σ_y are calculated as in (15), and σ values greater than σ_y are calculated as above in (14). ϵ values are simply created and plotted between 0 and 0.3 with a step value of 0.0001.

$$\sigma = E\epsilon$$

Equation 15 - The calculation for σ where $\sigma \leq \sigma_y$

Algorithm 3: Identifying the Relationship Between Work of Indentation and Hardness and Reduced Modulus

In their 2008 paper "Relation between the ratio of elastic work to the total work of indentation and the ratio of hardness to Young's modulus for a perfect conical tip," Chen and Bull identify several formulas to calculate the ratio of a material's hardness, H, and its' reduced modulus, ER (Chen & Bull, 2009). As input, the algorithm requires load P, depth h, and stiffness S from nanoindentation data, as well as the material's Poisson's ratio and the indenter's half-angle. UNiT starts by calculating the loading and unloading curves by finding the maximum load and separating each half of the curve. UNiT also finds the unloading stiffness of the curve, S_U, which is simply the stiffness value at max load. The unloading curve is then translated in the negative direction to remove the "holding" period used during nanoindentation. From here, UNiT can use the load-depth curve to calculate several relevant values. First, work of indentation is calculated by integrating and adding the areas below the loading and unloading curves. Figure 6 corresponds to the red and blue curves below, respectively. Then, the value h_r is calculated by finding the x-intercept of the unloading curve. When calculating stress-strain values, this can be found using a polynomial fit. The above is summarized in Figure 6.

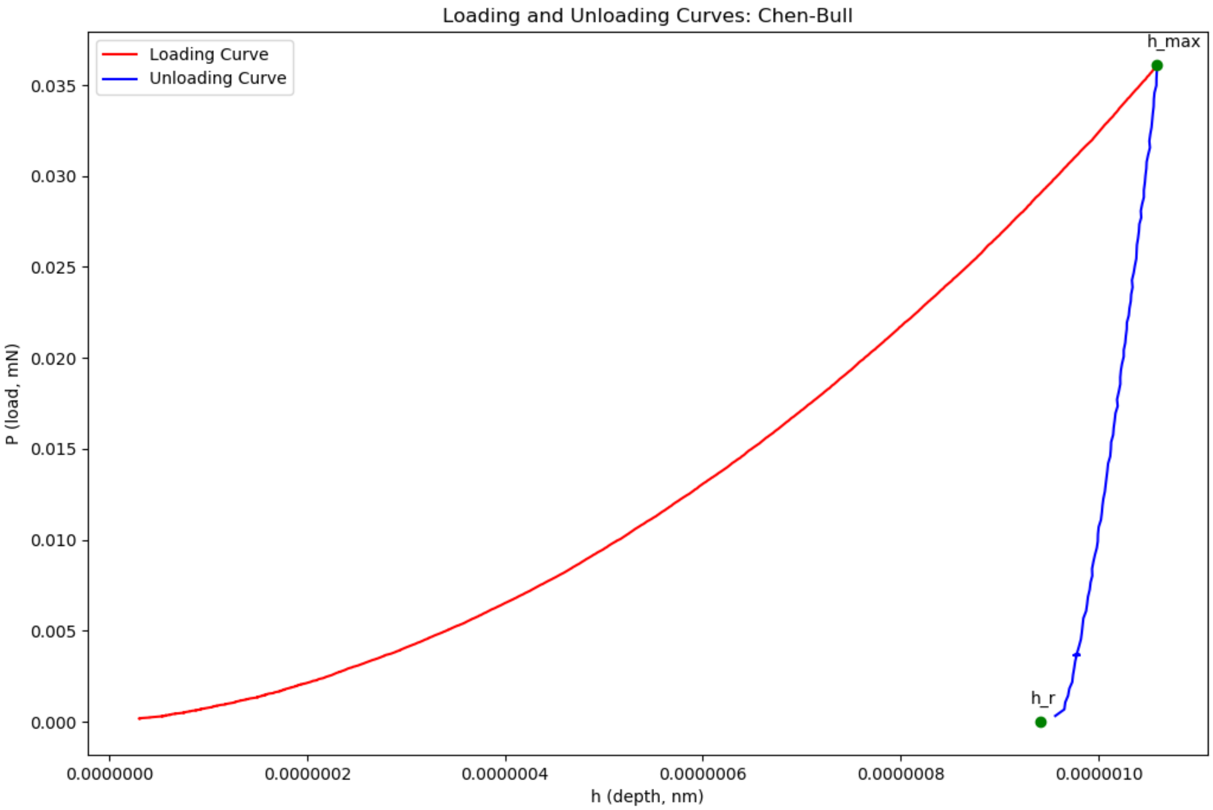


Figure 6: Chen-Bull's loading and unloading curves, with labels describing h_{max} and h_r

Next, UNiT uses a curve fit to solve for m, the exponent in the power-law described in (16). P_m is the max load value, B is a parameter used in the power law, and δ values correspond to depth values described above.

$$P_m = B(\delta_m - \delta_r)^m$$

Equation 16 - Calculation of P_m, as described in Equation 4 of (Bull & Chen, 2009)

Finally, UNiT can calculate values for H and E_r of the material. To do this, it utilizes the following equations described in Chen and Bull's paper:

$$E_r = \frac{\left(1 - \frac{\delta_r}{\delta_m}\right) S_u^2 \cot\theta}{2\beta P_m[m - \left(1 - \frac{\delta_r}{\delta_m}\right)\epsilon]}$$

Equation 17 - Calculation of E_r, as described in Equation 28a of (Bull & Chen, 2009)

$$H = \frac{\left(1 - \frac{\delta_r}{\delta_m}\right)^2 S_u^2 \cot^2\theta}{\pi\beta P_m \left[m - \left(1 - \frac{\delta_r}{\delta_m}\right)\epsilon\right]^2}$$

Equation 18 - Calculation of H, as described in Equation 28b of (Bull & Chen, 2009)

Chen and Bull also describe other methods by which to calculate E_r and H (19 and 20), respectively. However, the team found that these methods were not as accurate as the methods described above, likely due to differences in calculating work of indentation. So, UNiT simply offers the methods above to calculate E_r and H.

$$E_r = \frac{W_e/W_t}{\frac{1.5\pi m}{1+m} - \pi\epsilon W_e/2W_t} \cot\theta \frac{4\beta P_m}{\pi S_u^2}$$

Equation 19 - Calculation of E_r, as described in Equation 29a of (Bull & Chen, 2009)

$$H = \left(\frac{\frac{W_e}{W_t}}{\frac{1.5\pi m}{1+m} - \frac{\pi\epsilon W_e}{2W_t}}\right)^2 \frac{4P_m}{\pi S_u^2}$$

Equation 20 - Calculation of H, as described in Equation 29b of (Bull & Chen, 2009)

Methodology

Test Procedures for Validation of Material Properties

Various tests were used to validate the results of the Universal Nanoindentation Toolkit (UNiT). Several test methods were needed to create a solid foundation of validation data, meaning that ideally, two to three trusted methods are backing up each material property UNiT finds. The methods used were: Nanoindentation testing, indentation plastometry, hardness testing, data from the literature, and computationally simulated tests.

Nanoindentation

The nanoindentation tests were performed using an iMicro Pro from Nanomechanics, Inc., now part of KLA Instruments. This test machine utilizes the Oliver-Pharr method for its internal calculations and results. Continuous stiffness measurements were collected from 20-30 targeted indents using Berkovich and flat punch tips for validation. The variation in the number of indents is due to environmental circumstances and inherent error from the limitations of small-scale measurements.

The Berkovich tip (Figure 7) is made of diamond and manufactured by Micro Star Technologies. Berkovich is one of the most well-understood tips, as it dates back decades and has thus gained popularity. One benefit of this tip is its versatility since the three-sided pyramid shape can also be geometrically reframed as a Vickers tip for hardness. Its ease in manufacturing and resistance to wear also makes it desirable. However, one downside to the method is that the non-homogenous strain experienced throughout indentation results in improper stress-strain calculations. One set of data from each test material was taken using a Berkovich tip alongside the measurement type and number of indents mentioned above.

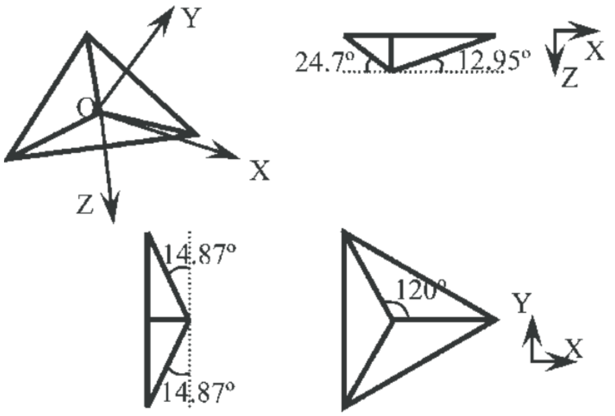


Figure 7: The geometry of the Berkovich tip in detail, being a three-sided pyramid with unique contact angles (Shi et al., 2013)

Another set of data using the same materials was taken using a diamond flat punch tip manufactured by Synton-MDP (Figure 8). The same methods as above were used for this test. Like the Berkovich tip, the flat punch tip is easy to manufacture; it has a simple, circular design with a diameter of 10.44 microns. The flat end of this tip allows for a constant contact area throughout the indentation process, which can be beneficial for a few reasons. The flat tip allows applied strain to stay constant while a higher volume of material is tested. These factors led to more accurate results and provided accurate stress-strain calculations, which was a downfall of the Berkovich tip.

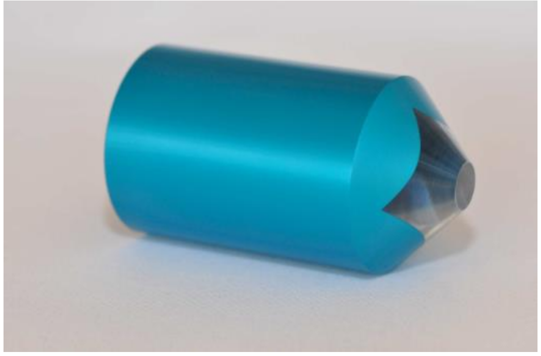
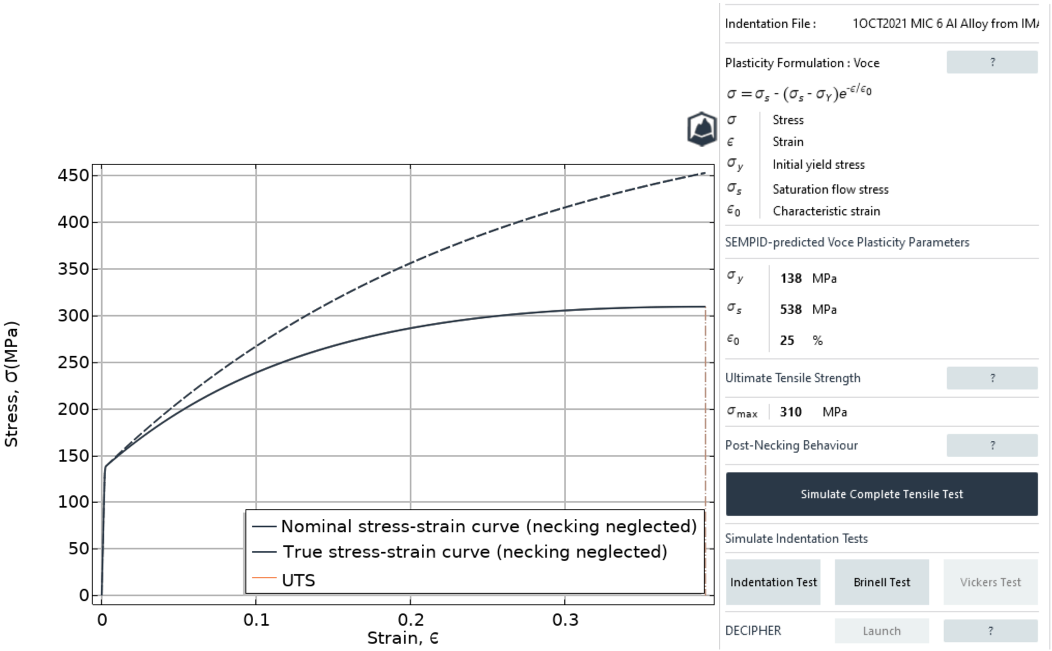


Figure 8: Diamond flat punch tip produced by Synton-MDP (Synton-MDP, n.d.)

Indentation Plastometry

The test machine used to conduct the indentation plastometry test was the Plastometrex Indentation Plastometer. The tip used was the WC-Co cemented carbide tip with a spherical geometry and 1 mm radius. To begin the indentation plastometry test, the machine was set up as directed by Plastometrex, and the profilometer was calibrated using the manufacturer-provided test sample to ensure the highest measurement accuracy possible. Once calibrated, the corresponding material class for each material was chosen, as prompted by the software. Next, the sample was positioned under the machine and started the test. During the testing process, the tip applies a load onto the material until yielding, when it is then released. Next, the profilometer measures the size and depth of the newly created indent by dragging a small tip along with the indent’s profile. The built-in software can output the test’s results (Figure 9). Each material was tested three to five times to ensure ample data points for accuracy.



Indentation File : 1OCT2021 MIC 6 Al Alloy from IM

Plasticity Formulation : Voce

$\sigma = \sigma_s - (\sigma_s - \sigma_Y)e^{-\epsilon/\epsilon_0}$

σ	Stress
ϵ	Strain
σ_y	Initial yield stress
σ_s	Saturation flow stress
ϵ_0	Characteristic strain

SEMPID-predicted Voce Plasticity Parameters

σ_y	138	MPa
σ_s	538	MPa
ϵ_0	25	%

Ultimate Tensile Strength

σ_{max}	310	MPa

Post-Necking Behaviour

Simulate Indentation Tests

DECIPHER

Figure 9: Indentation plastometry results from one location on the MIC 6 alloy from IMAT

Hardness Test

The machine used to conduct the Vickers hardness test was the DiaMet Hardness Tester from Buehler. To begin the test, a mounted sample was placed firmly in the sample holder of the machine. Next, a three-by-four array was specified with distances between each indent far exceeding the ASTM (American Society for Testing and Materials) standard, which outlines the minimum distance test points can be placed from each other without disrupting the test data. Particular care was taken when choosing the location of the points to ensure they were not near an edge or points from other tests, which would also skew the test results (Figure 10). Next, a load holding time of ten seconds was selected. To achieve the hardness value at an infinite depth, each material was tested twice, once at HV (Vickers Pyramid Number) 0.1 and once at HV1. HV1 was chosen since it is in the range known to find the hardness value at an infinite depth of each material in the test. The addition of HV0.1 was to investigate whether this load was sufficient to find this same value.

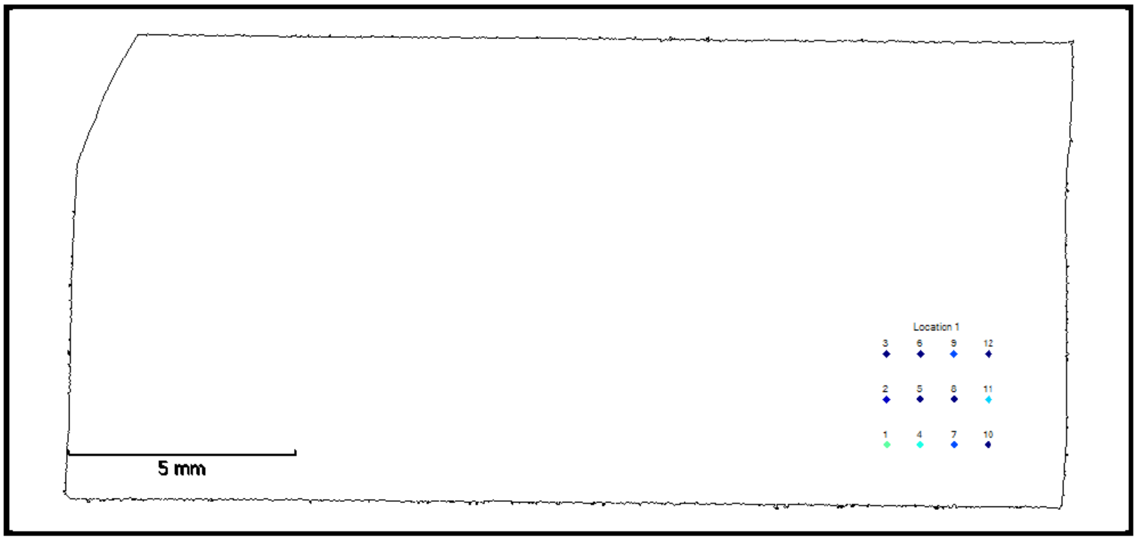


Figure 10: Results from an HV0.1 hardness test of the 304 Stainless Steel

Values from Literature

Data from literature is essential for validation because it brings previously tested and validated results to use and compare. Granta Edupack (CES, 2009) is a trusted resource developed by ANSYS, mainly used in educational settings to gather characterization data about materials. This software was used to find the majority of the material properties of the selected range of materials, specific to their material class, kind of surface treatment, and extrusion method, as seen in Table 1. If the specific material could not be located in Granta Edupack, MatWeb was another trusted resource used.

Material	Hardness	YS	UTS	Elongation to Failure	Treatment	Source
Al 6061	95-105 HB 100-107 HVN	35-40.8 ksi 241.3-281.3 MPa	39.7-46.4 ksi 273.7-319.9 MPa	10-14.4 % strain	T651	Granta Edupack
Al 3003	25-40 HB 25-40 HVN	21 ksi 144.8 MPa	22 ksi 151.7 MPa	16%	H14 Temper, cold worked, ASTM B209	MatWeb, McMaster Carr
304 Stainless Steel	201 HB 210 HVN	29.7 ksi 205 MPa	74.7 ksi 515 MPa	40%	Annealed, ASTM A240, A480	MatWeb
AISI 4340 Steel	190-233 HB 200-245 HVN	60.9-76.1 ksi 419.9-524.7 MPa	97.2-119 ksi 670.2-820.5 MPa	17-27%	Annealed	Granta Edupack
Maraging 350	276-314 HB 290-330 HVN	120 ksi 827.4 MPa	165 ksi 1137.6 MPa	18%	Annealed, round extrusion	MatWeb
OFHC	48-109 HB 48-115 HVN	50.8 ksi 350 MPa	55.1 ksi 380 MPa	22.50%	Extruded, annealed, hard	Granta Edupack
MIC 6	65 HB 65 HVN	15.2 ksi 104.8 MPa	23.9 ksi 164.8 MPa	3%		MatWeb

Table 1: Collected literature values of material properties

UNiT Software Development

These validation methods ensure the effectiveness of the three algorithms presented in UNiT. A Python backend was used to implement these algorithms, precisely Nix-Gao's relation of hardness and depth, Dao et al.'s evaluation of stress-strain curves, and Chen-Bull's "Relation between the ratio of elastic work to the total work of indentation and the ratio of hardness to Young's modulus for a perfect conical tip." The user interface was built using TypeScript, React, and Material UI and had the goal of being a straightforward system to use. The user can begin the analysis by selecting a Microsoft Excel sheet of nanoindentation data. Then, the user is immediately brought to a new page and prompted to select the algorithm(s) they would like to execute and specify any key parameters relevant to them (Figure 11). Once entered correctly, the software interprets the nanoindentation datasheet through the selected algorithms and outputs values or plots into a simple modular interface (Figure 12).

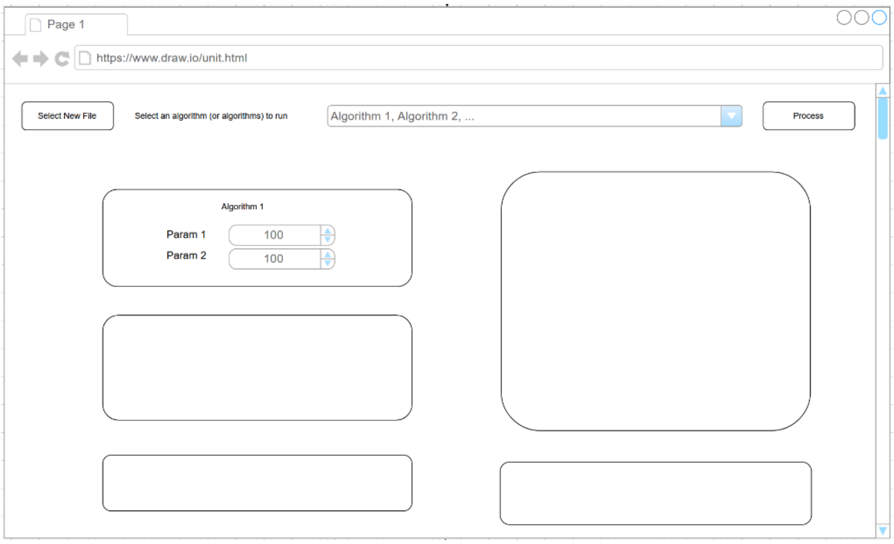


Figure 11: A mockup of UNiT's algorithm selection page

Figure 12: A mockup of UNiT's processing results page

Particular emphasis was placed on UNiT's ability to be improvised by the user and for new algorithms to be added with ease, regardless of coding fluency. Because of this, future users of UNiT can improve upon and develop the tool as they see fit.

Results and Discussion

Accuracy of Results

To evaluate the accuracy of the methods in UNiT, values collected from validation testing methods, which are outlined below, and literature are compared with results from the two algorithms in the program. A bar chart was chosen to visualize this comparison because it directly compares the several validation methods with UNiT's algorithms sorted by each material. For example, the main output from the Nix Gao method was the Vickers hardness of the material, and one of the main numerical outputs of Dao et al. was the material's yield strength. As such, Vickers hardness data was gathered from two different microhardness tests at 1 kgf and 0.1 kgf, flat punch nanoindentation, and literature. In addition, yield strength data from indentation plastometry, flat punch nanoindentation, and literature were used to compare with the Dao et al. method. Again, these values are considered the most well-known of the outputs and are therefore more significant when evaluated against other methods.

Figure 13 displays the first attempt at evaluating the Vickers hardness results output by UNiT's implementation of the Nix Gao method. Each value was within reason of the literature and tested values and in the correct order of magnitude between each material, which was the goal for UNiT. While this goal was reached, it became clear that the Nix Gao method initially overestimated the Vickers hardness of all the materials. Adjustments were then made to UNiT's algorithm implementation to counter the overestimation and provide more accurate results. Specifically, this was done by adjusting input depth and hardness values so that the Nix Gao method only utilized data where hardness is generally decreasing after a depth of 200nm, as described in (Write et al., 2013). In doing this, the estimations of Vickers hardness from Nix Gao were made notably more accurate, with only a slight overestimation (Figure 14).

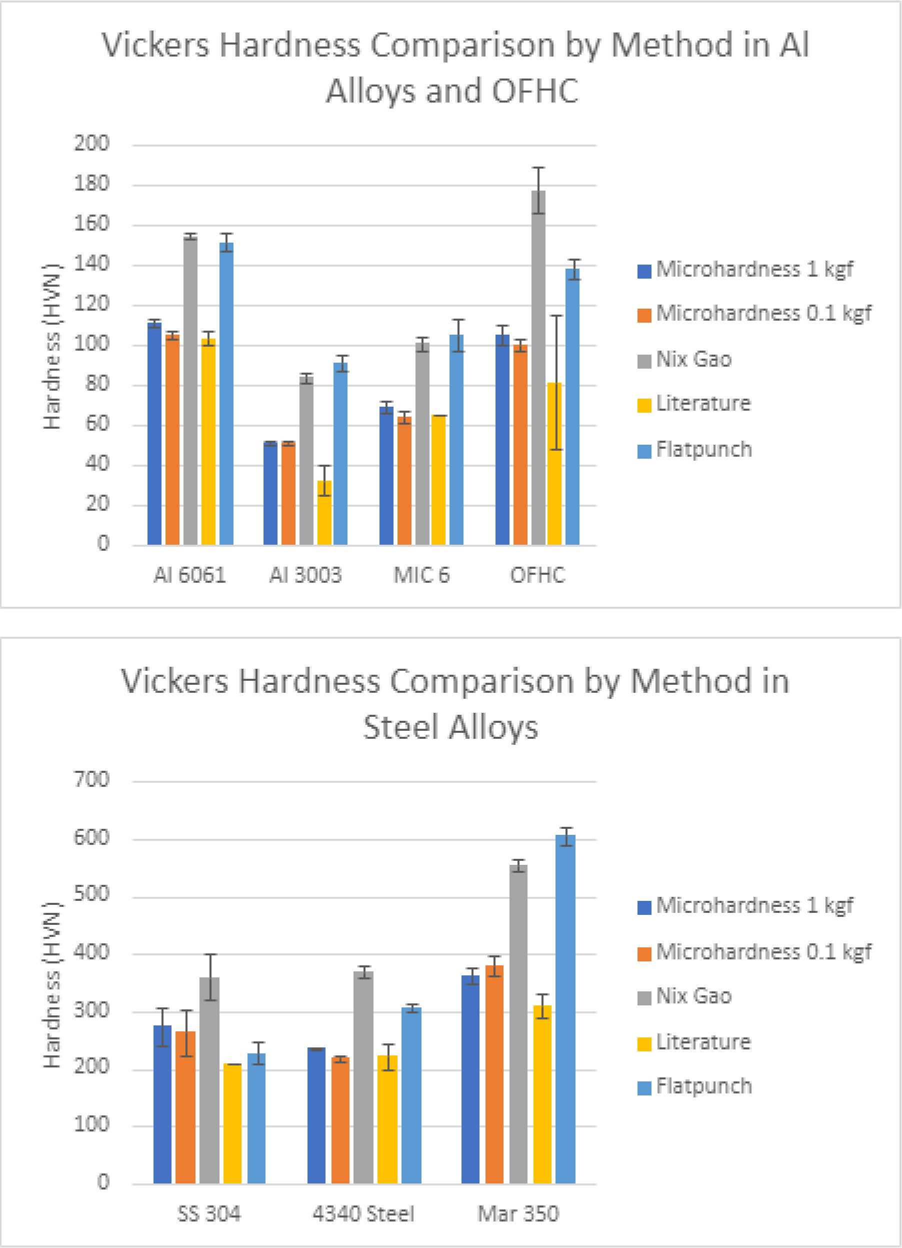


Figure 13: Comparison of Vickers Hardness across different testing methods and Nix Gao

It is also essential to look at the variance in values between the different validation methods. This is something to keep in mind when using test data, as it shows the value of using multiple testing methods for validation purposes. While each testing method is popular in materials science research and well-validated for finding Vickers hardness, they do not precisely align with each other. As expected, both Vickers microhardness values are nearly the same, with 0.1 kgf being slightly lower than 1 kgf. However, the flat punch data is higher than the microhardness tests, closer to the overestimated Nix Gao values. Again, this variance is expected between methods; however, it is interesting to see and note how they compare from a materials science standpoint.

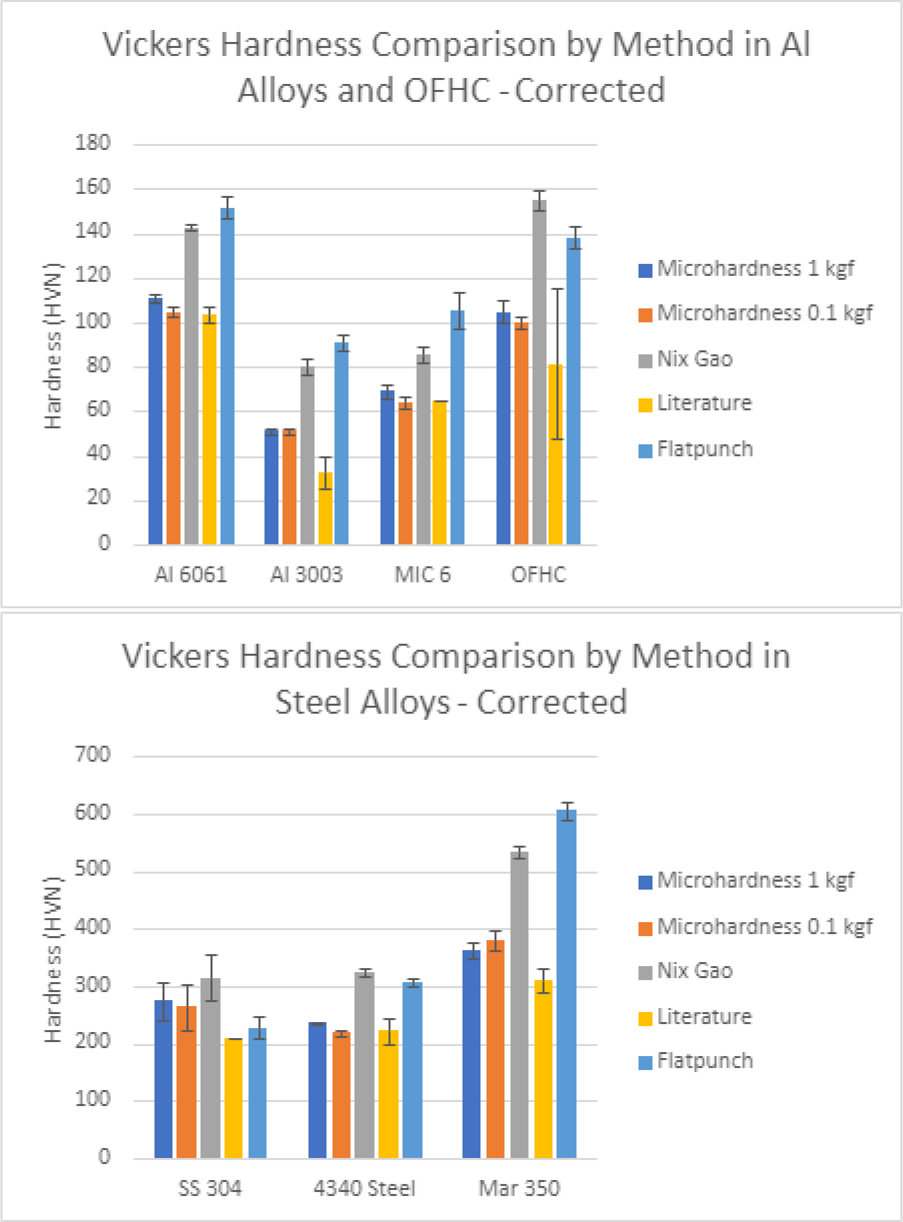


Figure 14: Comparison of Vickers Hardness across different testing methods and corrected Nix Gao

UNiT's implementation of Dao et al.'s stress-strain calculation is evaluated below in the form of yield strength (Figure 15). This method gave differing accuracies for each material and was not as consistent as the Vickers hardness results. For Aluminum 6061, 304 Stainless Steel, and Maraging 350 Steel, UNiT's Dao et al. provided fairly accurate results that matched closely to the validation values. However, for the remainder of the material set (i.e., Aluminum 3003, MIC 6, and 4340 Steel), the yield strength showed to be extremely low. For example, Al 3003 and MIC 6 had yield strengths less than 1 MPa, while they both should have been in the 100-150 MPa range according to literature and other values. This shows a limitation of UNiT that is important to note for future work. So, while the Dao et al. method has relatively accurate findings in the areas it works well in, it falls short for specific materials. This stark inconsistency is easy to spot if a user checks the results against values from other methods, but it could lead to some issues if it goes unnoticed.

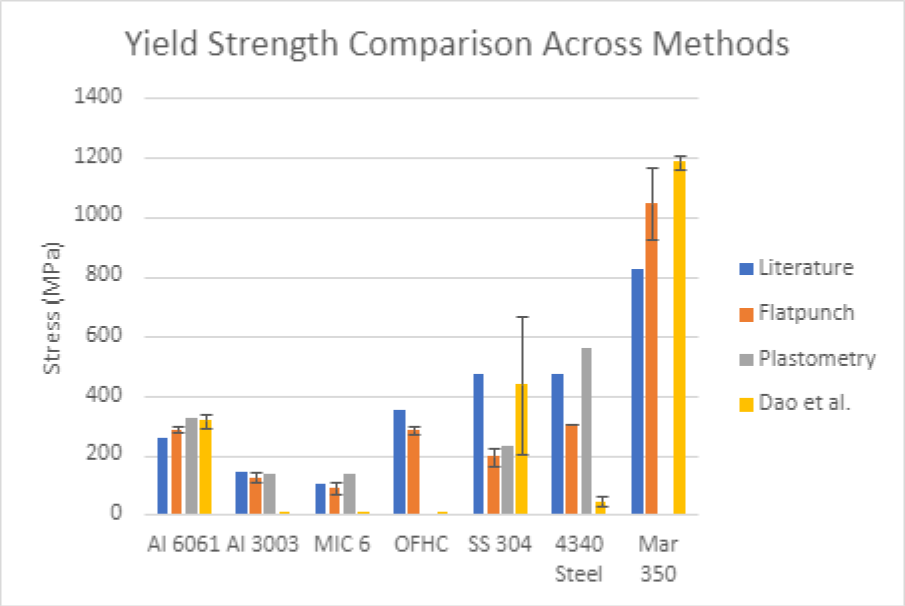


Figure 15: Comparison of yield strength across different testing methods and Dao et al.

The Dao et al. method seemed to align best with the literature values. When only looking at the difference in validation values, the variance is apparent in 304 Stainless Steel, 4340 Steel, and Maraging 350 Steel. In many cases, the Dao et al. And literature data are more similar and more prominent than the other methods, while the flat punch data appears to be at the lower end. Again, this shows the value in the multiple validation methods, as it gives insight into the variance of the tested values for material properties.

Intertest Variations

Method	**Standard deviation**
Vickers microhardness 1 kgf	8.57
Vickers microhardness 0.1 kgf	10.14
Literature	12.43
Flat punch	6.26
Nix Gao	11.56

Table 2: Comparison of standard deviation of Vickers hardness (across all materials) across different testing methods and UNiT's Nix Gao implementation

Method	**Standard deviation**
Flat punch	11.71
Dao et al.	42.53
Dao et al. (SS304 outlier removed)	10.99

Table 3: Comparison of the standard deviation of yield strength (across all materials) between flat punch and UNiT's Dao et al. implementation

The computed material properties can vary from test to test when running the same test multiple times on the same material. The variability of the results obtained through UNiT's Nix Gao implementation is on par with Vickers tests and the literature. However, the variation between each test is higher than that of the flat punch method (Table 2). While the indenter tip for the flat punch and nanoindentation is the same size, they have a different shape. The difference between a pointed and a flat surface on the tip could explain this difference in variation. UNiT's implementation of Dao et al. had much higher variability precisely due to testing on a sample of 304 stainless steel, which appears to be an outlier (Table 3). If we control for this outlier, the variation falls very close to that of the flat punch method. Multiple tests were run on each material to counter this variation, and the average was taken.

While we used the average of the computed values when determining the accuracy of the algorithms we include in UNiT, it is also worth considering the relevance of the standard deviation of these results. Materials are not entirely homogeneous in terms of their properties. Instead, they have an underlying microstructure that can vary across the material at a nanometer scale. Many traditional methods, such as Vickers used to compute material properties operate on a much larger region of the sample material than nanoindentation. For example, Nanoindenter tips are 10 µm while traditional methods for indentation testing operate on the 20 µm – 1 mm scale. Therefore, the standard deviation of the results can be used as an accurate measure of the distribution of material properties where the deviation is intrinsic to where the indentations were made. However, considering the underlying crystalline microstructure of a material must be taken when choosing the number of indentations. An adequate number of indentations can be selected per material basis by increasing the number of indentations until statistical significance ($p<0.05$) in the resultant material properties is reached. Due to the deterministic nature of the underlying algorithms implemented in UNiT, the intramaterial variations in its output can be attributed to the materials' underlying microstructure and not the result of any variation in the algorithms themselves.

User Testing

User testing was performed to determine the level of the Universal Nanoindentation Toolkit's (UNiT's) overall ease of use. UNiT team members administered user tests on four materials science researchers familiar with nanoindentation. These users were identified as ideal candidates due to their knowledge of nanoindentation and proximity to materials science research, meaning they would likely use UNiT if it were commercial software. Also, these users would benefit from having such a program because they do not have the solid technical computing backgrounds required to implement the algorithms that UNiT offers. User tests were administered remotely and involved the user manipulating the software through Zoom's remote-control feature while UNiT was open on a team member's device. Users were given a description of the software and its intention and were subsequently asked to perform various tasks using UNiT. While performing these tasks, users were given minimal instruction other than being told to perform said task. With this setup, users would be able to give feedback on the program without influencing team members' instructions or opinions. In addition, other UNiT team members observed and recorded feedback from the users' actions and comments.

Participants were given the initial UNiT interface to start the user test and select a specific Excel spreadsheet containing nanoindentation data. Then, users were asked to select an algorithm, input the parameters, and initialize the test data processing. After this processing was complete, users were asked to save and view the algorithm's output on the Results page. Users were then asked to go back to the initial UNiT landing page to select an additional algorithm to process the data with. Again, users processed the data like before, this time running both algorithms simultaneously, then saved and viewed the output of each algorithm. Lastly, users were allowed to use the toolkit for any further testing they desired and the chance to give any general feedback they felt was necessary.

As a result of user testing, the UNiT team was able to identify areas for improvement in the software prototype. For example, several users pointed out that buttons used in the interface were slightly tricky to notice at first, and some users visibly struggled for a moment to locate the button required to process the data. As a result, UNiT would benefit from having larger, centered buttons that users can more easily interact with. Users also requested that various other information be displayed in more detail, such as adding more information about each algorithm and improving the data layout on the Results page. Lastly, users requested that the display be more fine-tuned regarding the user interface design, such as having a less minimalistic design and using appropriate Greek lettering rather than the phonetic names for Greek letters. These results are summarized in Figure 16. Many of these areas were addressed by the UNiT team and added to the final version of the program.

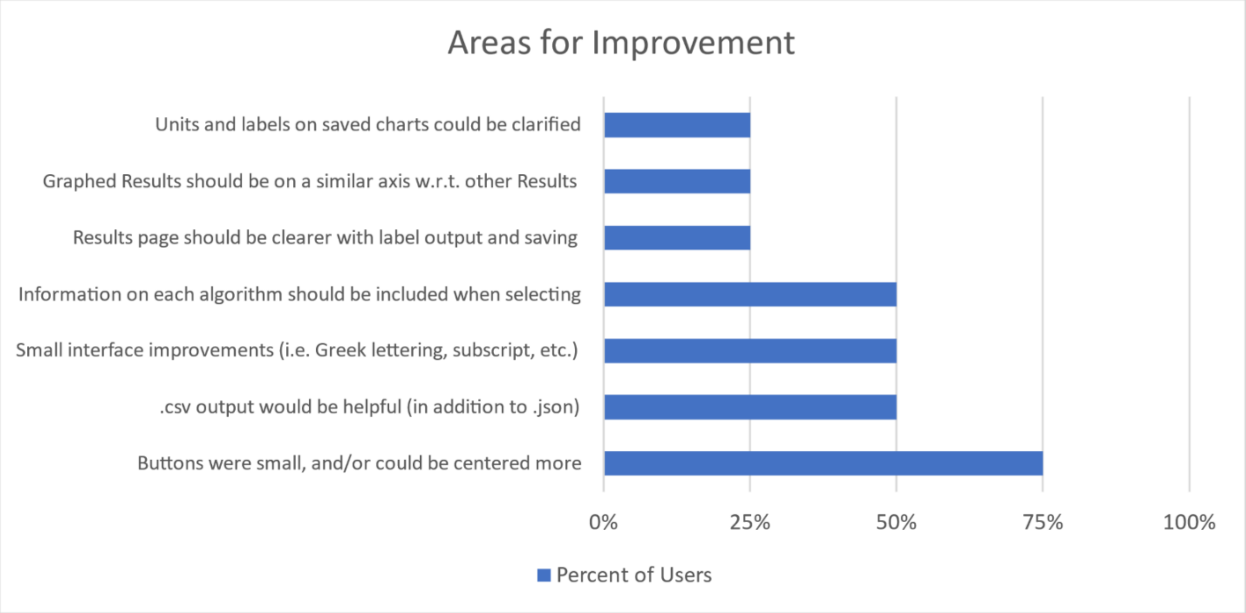


Figure 16: Areas for improvement identified with user testing

User testing also identified multiple areas of success for UNiT. First and foremost, all users appreciated the feature allowing them to select and run multiple algorithms simultaneously. Alongside this fact, most users appreciated the program's simple design and felt interacting with it was straightforward. As for calculating results from each algorithm, multiple users made the point that they liked the output format of the Results page and liked that they could save their results in raw data formats that were easy to analyze. Finally, users also recognized that using UNiT would save time than other methods (i.e., manual calculation) of obtaining results from the same algorithms. These results are summarized in Figure 17. The success areas match UNiT’s design philosophy of providing a straightforward interface that allows users to quickly calculate the results of various complex algorithms on nanoindentation data. User testing helped identify areas where UNiT could improve and where its design proved successful.

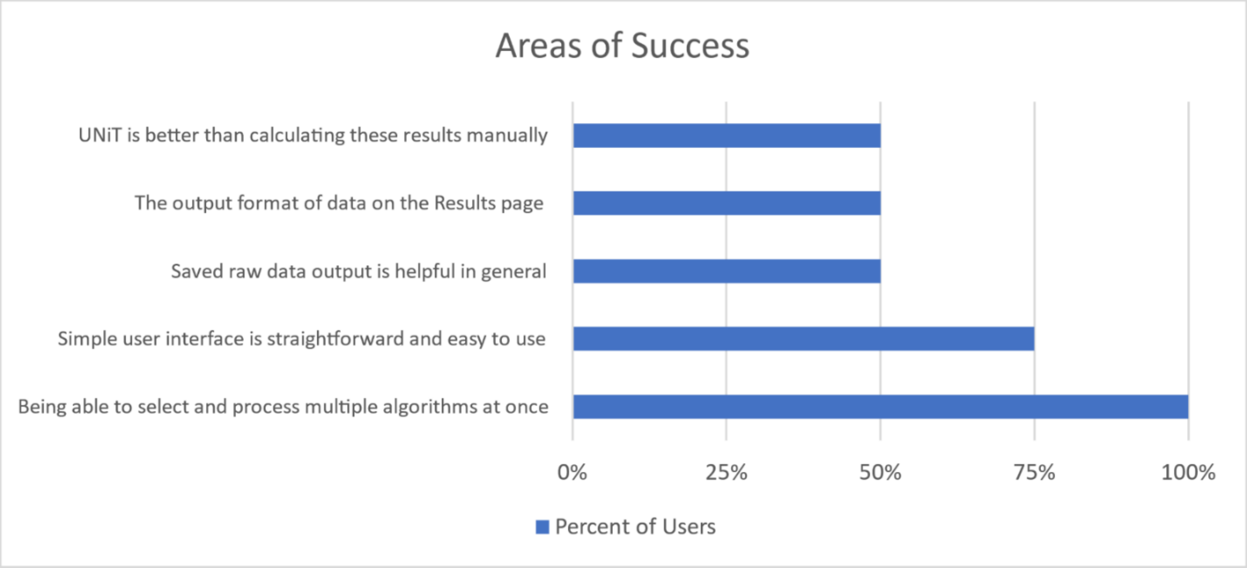


Figure 17: Areas of success identified with user testing

Discussion

The Universal Nanoindentation Toolkit (UNiT) achieved its goal of creating accurate implementations of complex algorithms that are generally unavailable to materials science researchers. User tests determined that while some changes to the interface would improve the overall user experience, users generally approved of several of the design decisions UNiT incorporated into its user interface. A simple, minimalist design meant that users could quickly understand the tool and process large amounts of nanoindentation data in ways that could not have feasibly been done before. The ability to save results in standard formats also proved helpful, as users would easily report on such data or otherwise utilize it how they please. Lastly, processing multiple algorithms simultaneously saved users a significant amount of time and improved accessibility to complex algorithms for users without a significant technical background. Overall, user tests demonstrated that UNiT's goal of providing a smooth user experience for materials science researchers was successful.

There was a lot to learn in building UNiT, and limitations were undoubtedly reached. Unfortunately, some goals that were initially set for this project could not be completed for several reasons, including unexpected issues with algorithms and time constraints. The inherent inaccuracies of the Chen Bull method, for example, limited it from being a contender for UNiT because it did not satisfy the level of accuracy that was needed. So, while it was implemented purely for example purposes, only the Nix Gao and Dao et al. methods were officially implemented into UNiT. It is hoped that more algorithms and improvements to UNiT can be made shortly to counter the limitations faced in this project.

Conclusion

The main goals that were set out for the Universal Nanoindentation Toolkit (UNiT) were met through this project. Three algorithms were implemented into a user-friendly interface that allows a range of metals to be evaluated with minimal time and effort compared to traditional methods. These algorithms were validated through multiple traditional testing methods to ensure that the quality of UNiT's results was on par with scholarly sources. The culmination of these efforts is a fully functional software program that supplies materials scientists with a faster and easier way to calculate material properties for a range of materials. There is future work that can be done to improve the toolkit, which is why UNiT was also designed to be fully modularized, allowing for straightforward changes and additions. Overall, UNiT successfully meets its goals of providing materials scientists with a tool that allows them to analyze nanoindentation data through complex and previously inaccessible methods quickly.

Broader Impacts Chapter

Engineering Ethics

The Fundamental Principals

I. Enhancement of Human Welfare: UNiT is free, buildable, and highly useful software. Creating this software was an effective and efficient way to analyze materials and ultimately improve the research process.

II. Be Honest and Impartial: Our goal is to provide an honest and impartial analysis of UNiT's capabilities. Both the advantages and disadvantages of UNiT are essential to our analysis which is why user testing was carried out on top of our observations to provide an unbiased point of view.

III. Increase the Confidence and Prestige of Engineering: Our goal is to create software that increases engineering capabilities by opening doors to research. We hope that UNiT will raise the bar in materials science and assist in reaching new findings at a higher rate.

The Fundamental Canons

Fundamental Cannon 2. Areas of Competence: Each developer of UNiT has the experience that qualifies them for their position. Each member is in the fourth and final year of their undergraduate studies and feels confident in their skills. In many areas where experience or understanding is doubtful, qualified professors and a Ph.D. candidate stepped forward to guide the work.

Fundamental Cannon 3. Continued Professional Development: This project was able to be carried out with the help of two professors and a Ph.D. candidate, who advised each decision that was made. Through these individuals and the opportunities given, everyone involved was able to grow their professional skillset and experiences.

Societal and Global Impact

As mentioned before, research on an individual or group basis can be expedited with UNiT. However, if overlooked, the inaccuracies in UNiT could lead research astray or cause reliability issues down the line. The user must keep in mind the limitations of the capabilities of UNiT to avoid such issues. While inevitable, it is essential to mention that anything that grows the research capabilities could further unethical research devices such as weaponry.

Environmental Impact

UNiT by nature does not impact the environment, plants, or animals.

Codes and Standards

Indentation Plastometry

Being a relatively new kind of test, there is no published testing standard for indentation plastometry. However, we followed the procedure provided by the manufacturer to ensure the best possible results.

Microhardness Test

The Standard Test Method for Vickers Hardness of Metallic Materials (ASTM E92-17) was followed during the microhardness testing process. This standardizes collecting Vickers hardness data to ensure accurate and consistent results.

Nanoindentation

The Standardized Nanoindentation standard (ISO 14577) was observed to collect all nanoindentation data. While not precisely followed, this standard inspired and guided our method for collecting nanoindentation data.

Economic factors

UNiT is free to consumers, and therefore the cost associated with the software is fundamental. The user must pay for or by some means provide their materials to test. Other economic factors may be the cost of the nanoindenter to collect data for UNiT and the lab space and utilities needed to hold the equipment. On the other hand, the user may save money compared to traditional methods since less equipment is needed and less testing material is used to conclude similar results.

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