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DETERMINATION OF THE COMPLIANCE OF AN INSTRUMENTED INDENTATION TESTING MACHINE

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Abstract – A major factor in calibrating a depth-sensing indentation tester is to determine its load-frame compliance. In this work a simplified theoretical approach and a computerized iterative method were developed to calculate the load-frame compliance of our laboratory commercial instrumented indentation tester. In addition, this research discusses many of the problems associated with the calibration of this type of testing machine. Three materials were used for the load-unload tests: fused silica, 6066-O aluminum and electrolytic copper. A value of load-frame compliance of 0,23 nm/mN was obtained with fused silica. This value was considered acceptable because the calculated elastic modulus for fused silica was comparable to those found by other researchers using a similar strategy of unload curve analysis. This calculated load-frame compliance is in the range found in literature for other instrumented indentation testers. The load-frame compliance values obtained with the two metals were unacceptable because of errors probably associated with pile-up and strain hardening.

Keywords: instrumented indentation testing, load-frame compliance, elastic modulus.

1. INTRODUCTION

Instrumented indentation testing is a powerful tool to measure the hardness and elastic modulus of materials. However, to accurately measure these mechanical properties, the machine compliance must be considered because during indentation test the applied force acts on the sample and also on parts of the testing machine. It leads to an increase of the measured indentation depth and causes an underestimation of the hardness and elastic modulus.

The machine compliance is generally named the load-frame compliance, which includes the loading column along with any additional compliance associated with the mounting which supports the sample [1]. The most accepted methods to determine the load-frame compliance involve the mathematical analysis of unload curves of materials with well-known elastic modulus [1-3]. In these approaches the load-frame compliance is linked to the indenter area function and this second parameter must also be determined. Oliver and Pharr [2] developed an iterative procedure to simultaneously determine the load-frame compliance and the indenter area function but their method is mathematically and time intensive. Other methods use image analyses of indentations by transmission electron microscopes [1], scanning force microscopes [3] or atomic force microscopes [4] to determine the indenter area function. VanLandingham [5] described the difficulties of determining the load-frame compliance.

In this work a computerized method was used to estimate the load-frame compliance of an instrumented indentation machine recently installed in our laboratory.

2. THEORETICAL APPROACH

The present modeling is a combination of the theoretical approach developed by Doerner and Nix [1] and the iterative procedures used by Oliver and Pharr [2] and others researchers [3]. Moreover some assumptions were made in order to simplify the calculation. The total compliance of the system, C_t, is:

$$C_t = C_f + C_m \quad (1)$$

where C_f is the load-frame compliance and C_m is the compliance of the sample expressed as:

$$C_m = \frac{\sqrt{\pi}}{2} \frac{1}{E_R \sqrt{A_p}} \quad (2)$$

where E_R is the reduced elastic modulus and A_p is the projected area of contact between the indenter and the material. The reduced modulus accounts for elastic deformation of both the indenter and the sample and is given by:

$$\frac{1}{E_R} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu_d^2)}{E_d} \quad (3)$$

where ν and E are the Poisson's ratio and elastic modulus of the sample material and ν_d and E_d are the Poisson's ratio and elastic modulus of the diamond indenter.

The theoretical area function for a Berkovich indenter is:

$$A_p = 24{,}5 h_c^2 \tag{4}$$

where h_c is the contact depth between the indenter and the material. It is important to note that, due to tip rounding, the theoretical area function is valid only for large indentation depths.

Combining (1), (2) and (3) provides a single equation to directly calculate the frame-compliance:

$$C_t = C_f + \frac{0{,}179}{E_R}\frac{1}{h_c} \tag{5}$$

A minimum of two indentations in a well-known monolithic material at different loads needs to be made to obtain a C_f by plotting C_t versus $1/h_c$. However, the initial h_c values are over estimated because they also depend on the frame-compliance C_f. For this reason, an iteration procedure needs to be used to converge onto the correct depth and correct frame-compliance. Since that:

$$C = \frac{\Delta h}{\Delta P} \tag{6}$$

equation (1) can be re-written as:

$$h_m = h_t - C_f P \tag{7}$$

where h_m is the actual maximum indentation depth on the sample and h_t is the maximum indentation depth measured during the test obtained from the curve. The contact depth h_c is proportional to maximum depth and thus:

$$h_c^m = h_c^t - C_f P \tag{8}$$

A more general formula for (8) is:

$$h_c^{i+1} = h_c^i - C_f P \tag{9}$$

So, using (5) and (9) new C_f and h_c values can be calculated. The calculation procedure is repeated until the frame-compliance value converges.

3. COMPUTERIZED CALCULATION METHOD

A C++ program was developed to automatically analyze the load-unload curves, extracting the values of h_c and C_t for each indentation test, and then applying the above iterative procedure to calculate the final C_f.

During the indentation process the force and displacement data points are saved to a computer file. The computer file is converted into an ASCII file with two columns force and displacement. This file has both the load and unload data. The unload curve starts at the maximum displacement data point after the hold-time was complete and ends where the indenter last makes contact with the sample surface. To measure the compliance only the unload portion is needed and the experimental software automatically separates the unload curve from the total force-displacement data.

Next step is to extract from the unload curve the total compliance C_t and contact depth h_c. There are several strategies to analyze the unload curve and measure the system compliance and contact depth. This part of the software was made so that it can be easily modified to apply any unload curve analysis strategy. As a starting point this research used the Doerner-Nix method because it is simple to program.

Doerner and Nix [1] uses the top 30% of the unload curve assuming that this portion is linear. In the software least-squares analysis was applied to fit a linear regression curve to the top 30% of the unload data points. The inverse slope of this curve is the total-compliance C_t and the intercept on the depth axis is the contact depth h_c (Fig.1). The remaining 70% of the data points were disregarded.

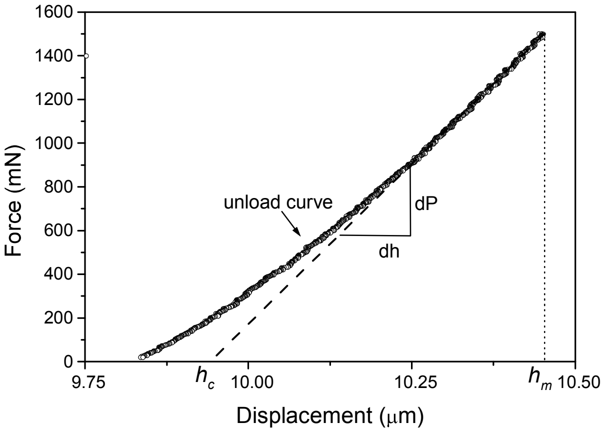

Figure 1. Doerner-Nix analysis of unload curves.

Both the system-compliance and indent depth change with load making this a multi-variant system. Therefore, the contact depth and system-compliance must be calculated simultaneously. Many unload curves need to be analyzed to measure the frame-compliance and determine the effects of depth and system-compliance together. Several (10 or more) indents must be made at each of the desired forces in order to achieve confidence in the measurements. Every unload curve gives one pair of data points of total-compliance C_t and contact depth h_c.

Using a Microsoft^{TM} Windows function several unload curves can be selected together and then the software calculates and saves C_t and $1/h_c$ for each unload curve. After this, linear regression is used to fit the set of data points for C_t versus $1/h_c$. According to (5), the point where the line crosses the compliance-axis is the frame-compliance C_f. This initial frame-compliance is used to calculate a new value of the contact depth h_c in all unload curves according to (9). Again a linear regression curve is fit to the new data set for C_t versus $1/h_c$ and a new frame-compliance is calculated. The software continues to calculate the frame-compliance value until convergence.

4. EXPERIMENTAL PROCEDURE

The studied equipment is a Shimadzu DUH-W201S instrumented indentation machine. This machine uses a magnetic coil to apply forces up to 1,961 N having an accuracy of ±19,6 μN. A pyramidal triangular Berkovich 115° indenter was used to make the indentation tests. The indenter tip radius was determined as 31±11 nm from residual indents imaged by an atomic force microscope [4].

The indentation experiments were carried out using three materials: fused silica, aluminum 6066-O and electrolytic copper. The samples were mechanical polished with SiC wet sand paper, diamond paste and suspended colloidal silica. Forces between 0,05 and 1,5 N were used. The displacement minimum measurement unit was 0,001 μm. The force and displacement accuracy were obtained from the owners manual.

Simple one-cycle load-unload tests were used to make the indentations. The loading speed was proportional to applied force varying from 2,2 mN/s for 0,05 N up to 14,5 mN/s for 1,5 N. The holding time was of 50 s for all tests. The total time for each indentation was of approximately 6 minutes. There were 70 indentation tests performed in each metal sample and 50 indentation tests performed in fused silica (always 10 tests for each force condition). The laboratory condition during the tests was 20°C and 60% humidity.

5. RESULTS AND DISCUSSION

The load-unload indentation test curves for the three materials were used, first, to evaluate the correct functioning of the Shimadzu depth-sensing testing machine and, secondly, to calculate the load-frame compliance.

5.1. Machine evaluation

The primary function load-unload was analyzed. Fig. 2 shows the load-unload curves for fused silica, electrolytic copper, and 6066-O aluminum. Each of these curves contains all 456 raw data points captured during the test. Outlying data did not have to be eliminated. As expected the indentation depth into fused silica was much less than copper and aluminum. Force and displacement results were produced by the Shimadzu testing machine and software during testing and initially are not considered accurate. The accuracy of the displacement will depend on the following calibration of the frame-compliance.

Fig. 3 shows four randomly selected load-unload curves for 6066-O aluminum between maximum loads of 0,2 and 1,5 N. It was observed that the load curves were superimposed and repeatable. Similar results were observed for the fused silica and electrolytic copper. This indicates that the testing machine provides repeatable results.

It is important to know how the machine behaved under its full range of testing conditions. The operation manual states that the machine can apply forces between 0,0001 up to 1,961 N. Fig. 4 shows C_f versus $1/h_c$ for seven loads comprising almost the full range of our indentation testing machine.

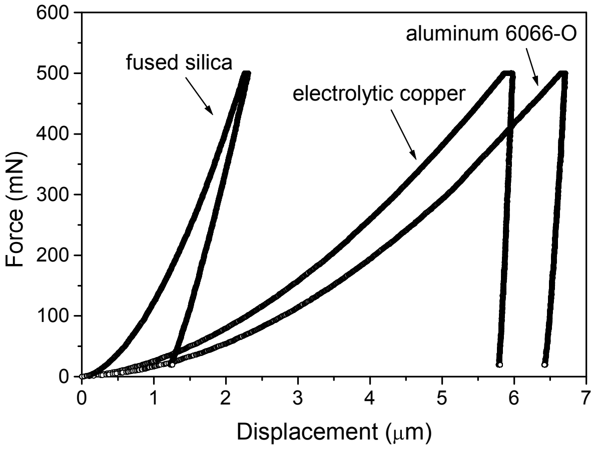

Figure 2. Typical load-unload curves for the three experimental materials.

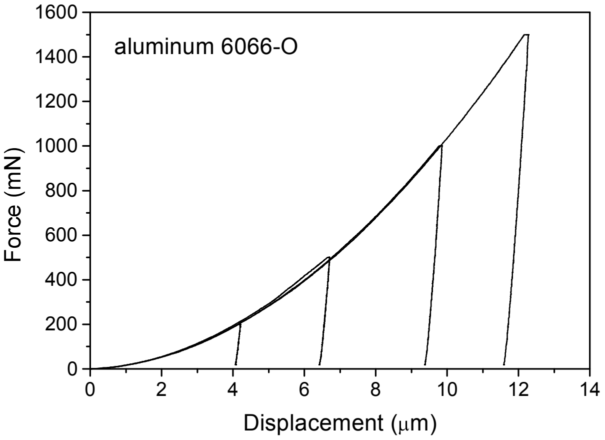

Figure 3. Load-unload curves for the same material at different maximum forces.

At the extreme limits of high and low forces there is a significant amount of error. From fig. 4 it is observed that there are two regions for the system-compliance that vary with the applied load. The transition point was approximately at 4 μm and 0,5 N. For the region of large forces, above 4 μm in the left side of fig. 4, the machine has a high C_f value. Below 4 μm the C_f is less than found for large forces.

The reason for the transition is uncertain. First, it could be caused by the way the machine components are designed and mounted. Here it appears that the load-frame presents a higher elastic spring constant when large forces are applied. Other factors could be due to material phenomena during indentation as pile-up and strain hardening. Moreover, the control software could also be responsible for this transition.

On the right side of fig. 4, in the low load region, it also is observed large scatter in the data. The tip rounding influence, crystallography, microstructural effects or surface roughness could cause this scatter. The effects of vibrations could also have influenced the load-unload curves because it was noticed that there is some scatter in the displacement data during tests at low loads. This could be linked to machine sensibility at small loads and displacements measurements. Like the high loads tests the software algorithms could create vibration scatter artifacts.

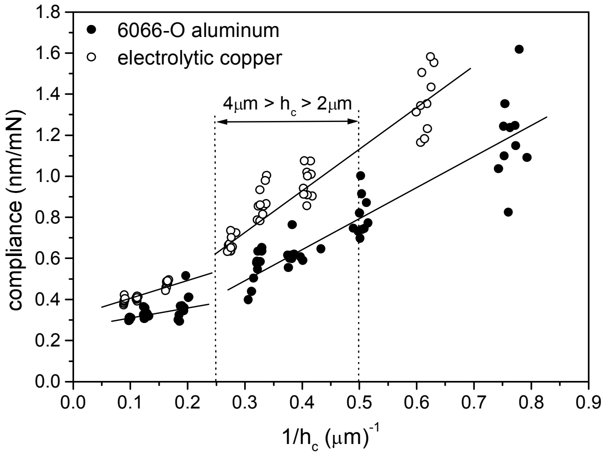

Figure 4. Values of C_f versus $1/h_c$ for 6606-O aluminum and electrolytic copper showing a transition point at approximately 2 μm indentation depth.

In order to reduce displacement vibration effects the velocity of indenter approach and retraction was analyzed. It was found that at slow approach and retraction rates the scatter was more pronounced. However, at fast retraction rates more than 6 or 7 mN/s the unload curves cannot be used to correctly measure the elastic recovery at low loads below 0,5 N because the number of data point captured, that depend on the indentation velocity, is small. For this reason a velocity of 2,2 mN/s was chosen for low load tests.

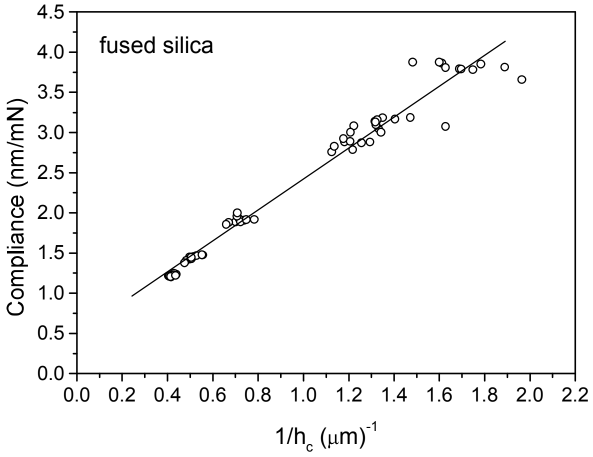

Figure 5. C_f versus $1/h_c$ for fused silica obtained using loads between 0,12 and 1,5 N.

For fused silica the transition point is not observed as shown in figure 5. On the other hand, it is noted that the maximum depth reached for fused silica at 1,5 N of load is lower than transition depth of 4 μm. This indicates that not only the large load is critical but large displacement distances can also contribute to the error of C_f measurements. However, this hypothesis is inconclusive because the material is monolithic and does not present pile-up or strain-hardening that would cause the transition observed for the metals.

5.2. Frame-compliance calculation

Considering the machine limitations discussed above specific force ranges were chosen to calculate the load-frame compliance. For 6066-O aluminum and electrolytic copper loads between 0,1 and 0,5 N were used. For fused silica loads between 0,2 and 1,5 N were applied.

Fig. 6 illustrates the iteration procedure for fused silica. As expected the C_f values oscillated about the final convergent C_f value. The convergence of fused silica was obtained in 15 iterations to 2 significant figures. For the two metals tested convergence occurred in 4 iterations to 4 significant figures. The C_f values calculated from software are shown in Table 1.

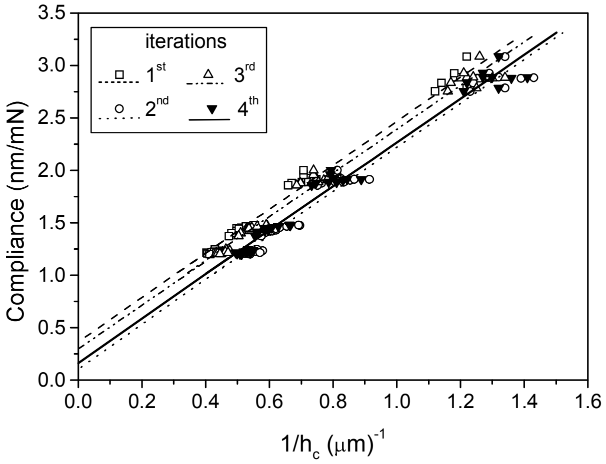

Figure 6- Load-frame compliance calculation results for fused silica showing the first 4 iterations.

Fig. 7 shows the final results for the three materials. The frame-compliance C_f obtained for the metals had approximately the same value, averaging 0,074 nm/mN. However, the C_f calculated using fused silica was much higher 0,23 nm/mN. This discrepancy could have been caused by the sources of error discussed earlier.

Comparisons were made between the elastic modulus calculated for the three materials for this analysis and other published researches. The slope of the final linear fit was used to calculate the reduced elastic modulus according to (5) and the elastic modulus of the material was calculated using (4). The Poisson's ratio for fused silica is 0,17 and for diamond it is 0,07; the elastic modulus of diamond is 1141 GPa [2]. For the metals the Poisson's ratio was assumed to be 0,3. The actual modulus of fused silica is 72 GPa and the calculated modulus E from the linear fit shown in Fig. 7 was 90 GPa. For aluminum the bulk modulus is 70 GPa and the calculated for the tested 6066-O alloy was 69 GPa. For the electrolytic copper, the bulk modulus is 110 GPa and the calculated modulus was 105 GPa.

Table 1. Iterative procedure results for the load-frame compliance.

iterations	load-frame compliance (nm/mN)		
	fused silica	electrolytic copper	6066-O aluminum
1	0.3699	0,0689	0,0831
2	0.1193	0,0673	0,0808
3	0.3055	0,0674	0,0809
4	0.1756	0,0674	0,0809
5	0.2706		
6	0.2034		
7	0.2521		
8	0.2174		
9	0.2424		
10	0.2246		
11	0.2374		
12	0.2282		
13	0.2348		
14	0.2300		

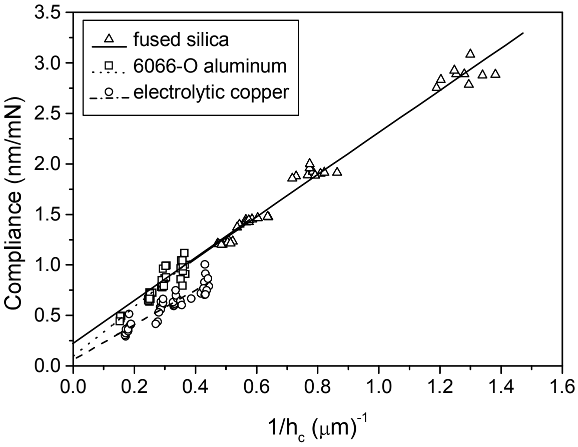

Figure 7. Final computerized iteration results for the three materials. Where the linear regression fits cross the compliance axis is the load-frame compliance.

The calculated elastic modulus for each of the materials was slightly less than the actual value. It is interesting to note that the modulus measurement for fused silica in this research is almost identical to the value obtained by Doerner and Nix [1]. For aluminum (111) and (100) single crystals Doerner-Nix also found elastic modulus values about 70 GPa. These results give evidence that all the experiments and analyze strategies were equivalent. This also shows that the strategy does not depend on the force scale because Doerner-Nix results using low loads up to 0,1 N where the same as the present experiments using higher loads up to 1,5 N. Doerner-Nix used a TEM to measure the contact area while this software method used the theoretical area function for Berkovich indenter and an iteration procedure to correct the contact depth. So it appears that the strategy used to analyze the unload curve is a more crucial factor than the contact area measurement method when determining the load-frame compliance.

Finally, there is more supporting evidence for the demonstrated methodology. Oliver and Pharr [6] stated that when the convergence happens in a few cycles its calibration method works well. In our case, for two significant figures, the two metals required 2 iterations and the fused silica converged in 14 iterations.

5. CONCLUSION

The load-frame compliance C_f of an instrumented indentation machine was calculated using three test materials. The C_f values were not the same. The load-frame compliance calculated using fused silica was 0,23 nm/mN while using the metals the C_f values were much less at only 0,07 nm/mN. However, several possible sources of errors were discussed about the metals. So, the load-frame compliance value obtained with fused silica was chosen as acceptable. This value compared to those obtained to other commercial depth-sensing indentation machines. For example Gong et al. [7] reported a C_f of 0,196 nm/mN. Moreover, the computerized method provided elastic modulus results for fused silica and aluminum comparable to those found by Doerner and Nix[1].

In order to more accurately determine the load-frame compliance of instrumented indentation machines other strategies of unload curve analyze are being investigated.

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