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COMPARISON OF NANOINDENTATION TESTERS IN TAIWAN

Yi-Ching Lin [1], *Ya-Ko Chih* [2]

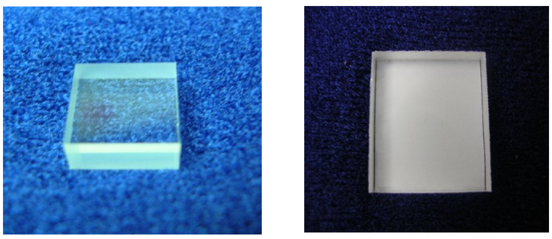
[1] Industrial Technology Research Institute (CMS), Hsinchu, Taiwan, email：franceslu@itri.org.tw
[2] Industrial Technology Research Institute (CMS), Hsinchu, Taiwan, email：JacobChih@itri.org.tw

Abstract – The Center for Measurement Standards provides two test pieces for this exercise to compare a set of common data including elastic modulus and indentation hardness. Participating laboratories will perform the common test items on their systems and follow the recommended instructions. The distribution chart compiled from the experiment data provided by each laboratory can then be used to fully demonstrate the current state of nanoindentation testers. The experimentation method shall take the testing capabilities of each laboratory into consideration, and a full analysis shall be provided. The providers of experiment data shall be represented by an index code in the report. By participating in this comparison exercise, participants will be able to understand the differences and similarities between their own results and those of other laboratories; thus the results can be used as a reference basis for evaluation of the participant's testing capabilities.

Keywords:nanoindentation、elastic modulus、hardness

1. Test Pieces for Comparison

The two materials used for comparison are the **Fused Silica** and **Polycarbonate** (FIG 1(a) – FIG 1(b)) both produced by ASMEC from Germany. The dimensions of the test pieces are as indicated in TABLE 1. The test pieces used for comparison will be stored in a 85mm x 83mm x 25mm carrying case to prevent damage to the objects.



(a) Fused silica test piece (b) Polycarbonate test piece

FIG 1. Comparison test pieces and storage case

TABLE 1. Dimensions of the test pieces for comparison

Material	Area	Thickness
Fused silica	8mm x 8mm	(3.0 ± 0.2) mm
Polycarbonate	16mm x 16mm	5.0 mm

Before mailing out the test pieces for comparison, the following tasks will be conducted by the Center for Measurement Standards. This is for simplification of results comparison, where the uniformity test and homogeneity test on the subjects are taken into consideration (FIG 2) to provide sufficient information on the test piece itself.

I. Uniformity testing of test pieces

Before mailing out the test pieces to each respective laboratory, the pieces at the center are preferably chosen as the test pieces. Each piece is tested for five repetitions and analyzed for its uniformity.

II. Homogeneity testing of test pieces

Before mailing out the test pieces to each respective laboratory, the pieces at the center are preferably chosen as the test pieces. Each piece is tested for five repetitions and analyzed by an ANOVA model to find the ratio of variance between samples to variance of individual samples. A higher ratio value indicates excessive variation between sample test pieces.

The test pieces are to be affixed individually to a metal block after testing and then stored in the carrying case. The test pieces will come with gripping pliers and dust-free gloves to avoid unnecessary pollution caused by the user. See FIG 3.

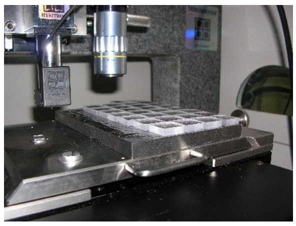


FIG 2. Uniformity testing

FIG 3. Storing test pieces in the carrying case

2. Testing Method

Participating laboratories in the comparison exercise shall conduct the experiment at the time when environmental disturbance is at a minimum for the system performing the indentation. If the system can be customized and programmed to run the experiment at the designated timeslot and location, then it is recommended to run it late at night. If the system cannot be programmed to run according to the conditions mentioned above, requiring the operator to manually conduct the experiment, then the process is to be carried out at a time when the environment is more stabilized. In addition, we recommend that the operator is

the only person remaining in the laboratory in which the experiment is conducted. Furthermore, other test equipment in the same room that may become a possible source of disturbance should be disabled as much as is permissible during the indentation process. This will reduce external factors that may influence the test results. The test pieces are to be affixed by the supplied quick-drying cement, and must be allowed to set for at least 24 hours before the indentation experiment. Do not use double-sided tape to attach the test pieces. The experiment operator must be wearing the supplied gloves and must use the gripping clippers to hold the test pieces during the affixing and indentation process. Do not use bare hands to touch the surface of the test pieces.
This experiment uses the Berkovich indenter to carry out 11 repetitions of indentation procedures on different positions within the central region of the test pieces, where the experiment data is recorded in the datasheet. Before participating in this comparison exercise, each laboratory must perform calibration on the area function of the indenter that is to be used. No other indentation experiments before the official trial shall be conducted. If the system can be calibrated for machine compliance, the participating laboratory shall run the compliance tests on the system before the comparison exercise begins. The resulting compliance parameters shall be used for the comparison experiments. Due to the fact that the nanoindentation testers included in this exercise are produced by different manufacturers, the maximum load for indentation experiments shall be set to 9000 μN for all participating laboratories for the goal of result comparison. The load vs. time function as shown in FIG 4 is recommended to be used in the indentation experiment. The indentation experiment shall be conducted with the most reliable control method of the system, for example the load control mode on Hysitron systems.

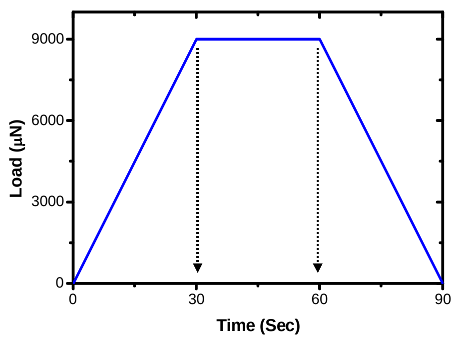


FIG 4. Recommended load function for nanoindentation

3. Analysis of Comparison Results

Z _ Score can be used to calculate the performance index of the comparison results, as shown by the equation below:

$$Z\_\text{Score} = \frac{X_i - X_{\text{assigned}}}{S}$$

Wherein,
X_i is the mean value from the participating laboratory with index code *i*, and then deducted by the mean value tested at the Center for Measurement Standards;
X_{assigned} is the median of X_i;
S is the normalized interquartile range of X_i.

If $|Z\_\text{Score}| \leq 2$, test results from the participating laboratory are consistent.
If $2 < |Z\_\text{Score}| < 3$, test results from the participating laboratory are questionable.
If $|Z\_\text{Score}| \geqq 3$, test results from the participating laboratory are not consistent.

During the analysis and comparison process of test results, the test results from participating laboratories are labeled by an index code. The source of experiment data for this exercise not only comes from laboratories of different organizations, but also includes two test methods (quasi static and CSM) and equipment from three manufacturers (Hysitron, MTS, and UMIS). Discarding system errors or situations where the test pieces cannot be placed in the system for testing, full data sets from 17 laboratories are provided with results completed by 19 test systems. The mean of the test results from participating laboratories is deducted with the reference value as tested by the Center of Measurement Standards, ITRT, and analysis is performed on the resulting data. To begin with, experiment data from 16 laboratories and 18 test systems minus the reference value of the specific test piece as measured by the Center of Measurement Standards is used to plot the distribution chart of mean and standard deviation values (FIG 5 ~ FIG 8)

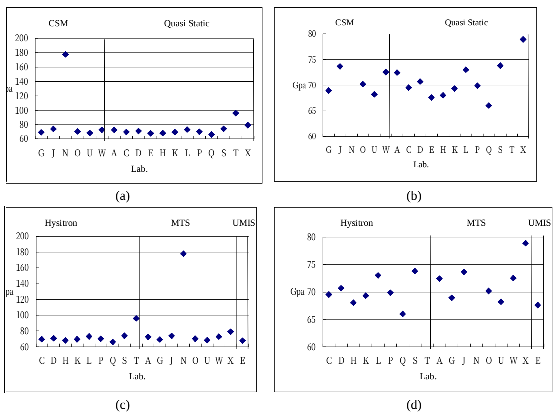


FIG 5. Mean and standard deviation of complex modulus for fused silica from each laboratory

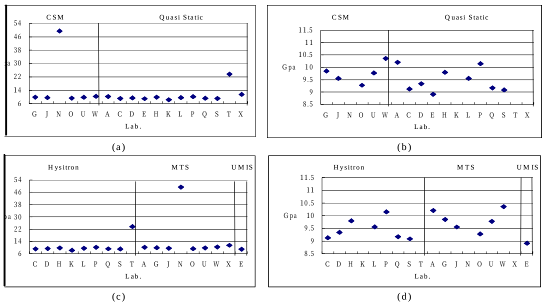
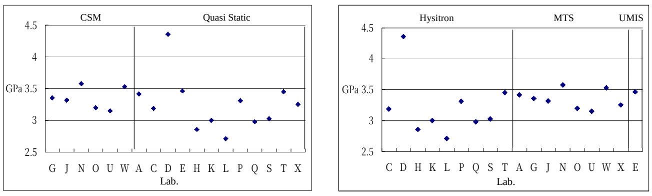
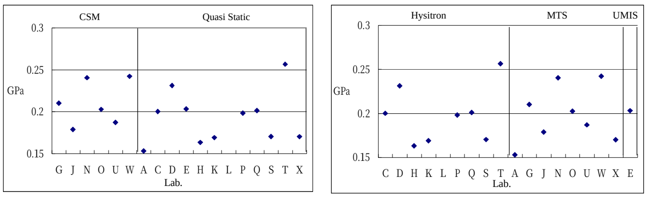


FIG 6. Mean and standard deviation of indentation hardness for fused silica from each laboratory



(a) (b)

FIG 7. Mean and standard deviation of complex modulus for polycarbonate from each laboratory



(a) (b)

FIG 8. Mean and standard deviation of indentation hardness for polycarbonate from each laboratory

As data distribution shows significant groups of outliers, the median used by the evaluation method to represent the reference value is therefore not dependent on the outliers. The *Z _* Score bar chart indicates the distribution of testing capabilities for laboratories participating in this exercise.

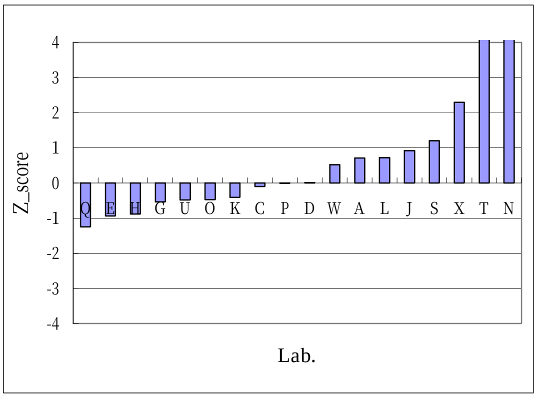


FIG 9. Z _ score distribution of complex modules for fused silica

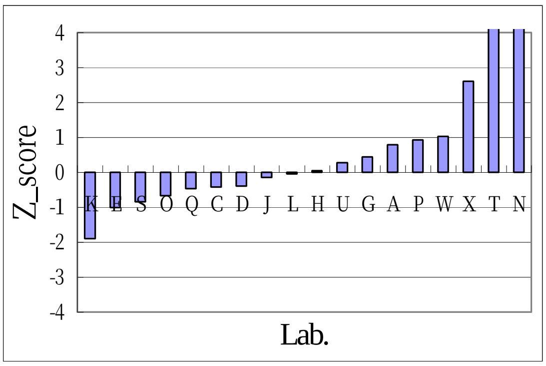


FIG 10. Z _ score distribution of hardness for fused silica

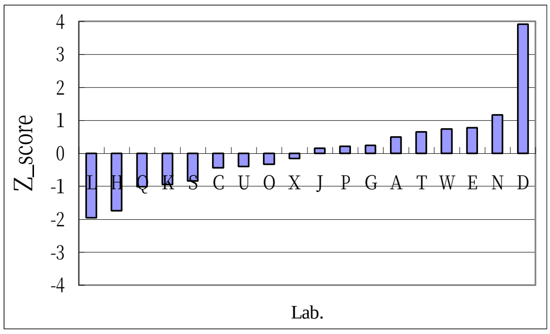


FIG 11. Z _ score distribution of complex modulus for polycarbonate

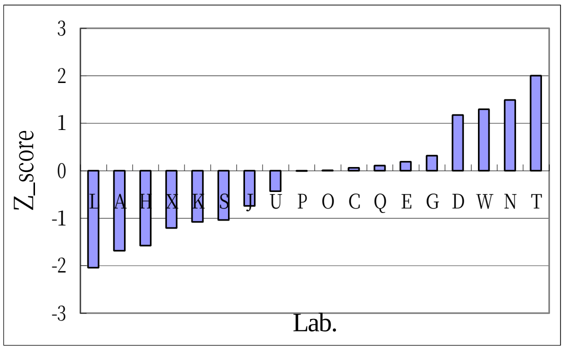


FIG 20. Z _ score distribution of hardness for polycarbonate

4. Conclusion

FIG 5~ FIG 8 indicates that fused silica with higher hardness, with the exception of N and T laboratories who produced greater variance in test results, will produce better consistency from all laboratories for both the complex modulus and hardness data. However, the softer polycarbonate will produce higher variance in test results between different laboratories for both the complex modulus and hardness properties. Looking at the distribution of experiment data on the softer polycarbonate material, the ratio of standard deviation to the mean value (a higher ratio indicates a higher uncertainty in the experiment data) produced by each laboratory is as high as 12.3% for the complex modules and 17.7% for the indentation hardness. The complex modulus for fused silica is at 4.2% if data with greater variance from two laboratories is removed from consideration, and the indentation hardness is at 6.3%. This indicates that polycarbonate exhibits a greater variance factor in the indentation experiment. For example the different sizes of indenter used by each laboratory will result in different sink-in levels in the material, thus influencing the experiment results. Therefore, for test pieces with lower hardness such as polycarbonate, more test data is required in order to reproduce the actual properties of the material. Alternatively, system parameters can be adjusted to test pieces with lower hardness for better representation of the test results.

FIG 9 ~ FIG 12 includes the Z _ score bar charts; Z _ score less than 2 indicates consistent test results from the participating laboratory; scoring between 2 and 3 indicates questionable test results from the participating laboratory; and a score higher than 3 indicates inconsistent test results from the participating laboratory. FIG 9 and FIG 10 shows the comparison of complex modulus for fused silica, including questionable test results from one participating laboratory (X) and inconsistent test results from two participating laboratories (N and T). Z _ score distribution of complex modulus for polycarbonate shows inconsistent test results from one participating laboratory (D), and the Z _ score distribution of hardness for polycarbonate shows questionable test results from only one participating laboratory. As for the participating laboratories T and N with higher variance for the complex modulus and hardness data on the harder material fused silica, their test results on the softer material polycarbonate exhibit relatively less variation when compared to the other data groups. A possible explanation is that before conducting the experiment, these two laboratories calibrated the area function of the indenter for a larger range of indention depth, thus affecting the accuracy of the test due to the shallower indentation depth on the harder fused silica material. However, under similar conditions of the indentation experiment, the deeper depth of the indenter on the softer polycarbonate will result in a more accurate test measurement.

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Author:
Ms.Yi-Ching Lin
Center for Measurement Standards
Bldg. 8, 321 Kuang Fu Road, Sec. 2, Hsinchu, Taiwan 300, R.O.C.
TEL: 886-3-5743790
FAX: 886-3-5724952
E-mail: franceslu@.itri.org.tw

Dr. Ya-Ko Chih
Center for Measurement Standards
Bldg. 8, 321 Kuang Fu Road, Sec. 2, Hsinchu, Taiwan 300, R.O.C.
TEL: 886-3-5743745
FAX: 886-3-5724952
E-mail: JacobChih@itri.org.tw