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EFFECT OF TIME RELATED PARAMETERS ON ROCKWELL HARDNESS MEASUREMENT OF POLYMERS

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Abstract - The effect of time-related parameters such as loading velocity, load application time, and gauge reading time on Rockwell hardness measurement was investigated for polymers such as polyethylene (PE), polypropylene (PP), polyvinylchloride (PVC) and acrylic plate. It was found that keeping the specific requirements of the test procedure, especially for the time parameter related one, is very important to obtain reliable and repeatable data in polymer hardness measurement. These characteristics of plastic hardness measurement are because of the good elastic properties of polymers and showed continuous deformation even at the time of data reading. The optimum condition for hardness measurement appeared to be 4 s for load application time, 15 s for test load application net time, and 15 s for gauge reading time after unloading. After this preliminary test for establishing protocol, a round robin test was carried out for six laboratories and it appeared that the testing machine should be one in which time-related variables are controllable. In other words, hardness tester with a function of plastic mode test showed much better repeatability in measurement data because of the automatic reading of data at a specific time. Verification of hardness tester using brass reference block was not enough to guarantee the reliability test results or reduced data scattering for polymer.

I. Introduction

The effect of time-related parameters such as loading velocity, load application time, and gauge reading time on Rockwell hardness measurement of polymers was not studied as much as metals. Most of the procedure for polymer hardness measurement is quite similar to a metal hardness test. [1,2,3,4]

[1] ISO 2039-2, Plastics-Determination of hardness. Part 2. Rockwell hardness (1987)
[2] ASTM D 785, Standard test method for Rockwell hardness of plastics and electrical insulating materials (1993)
[3] JIS K 7202, Method of Rockwell hardness test for plastics (1995)
[4] KS M3037, Rockwell hardness test methods for plastics (1983)

However, plastics usually show high elastic recovery properties and hence the control of time related parameters in hardness measurement is very important to avoid wide data scattering. In addition to this, depending on the polymers, elastic properties are different and hence the test data are different even when the same test procedure is applied. Because of this fact, information about the effect of time related parameters on polymer hardness measurement is necessary for a variety of polymers. In this study, polymers such as polyethylene (PE), polypropylene (PP), polyvinylchloride (PVC) and acrylic plate have been studied to observe the effect of time related variables on the hardness test results. Also results of the proficiency test have been reported.

I. Experiment

(1) Rockwell hardness measurement

In this study, hardness tester was modified for continuous monitoring of depth change while the hardness test is being carried out. Fig. 1 shows an example of a time-displacement curve obtained from the polymer hardness test.

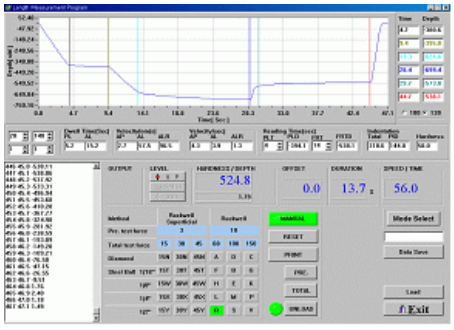

Fig. 1. An example of real time measurement of time-displacement during hardness test.

Test condition has been set as shown in Table 1 so that all of the test range specified in ISO and JIS are included. Specifically, the load application time of 2 s, 6 s, and 10 s, were chosen for test condition. For the test load holding time, 4 s, 10 s, 16 s and for the reading time 2 s, 10 s, and 20 s were chosen for test condition.

Table 1. Selected test conditions for hardness measurement.

Test condition	Load application time(s)	Test load holding time(s)	Reading time(s)
Standard	4	15	15
Variation of load application time	2	15	15
	6		
	10		
Variation of test load holding time	4	4	15
		10	
		16	
Variation of reading time after test	4	15	2
			10
			20

(2) Proficiency test

Six laboratories participated in this proficiency test. The test condition has been fixed to observe the variation among them. The load application time, test load holding time and reading time after load remove were set to be 4 s, 15 s and 15 s, respectively. The entire hardness testing machine was calibrated using brass reference blocks before testing the polymer specimens as specified in the ISO standard, 2039-2.

II. Results and Discussion

(1) Rockwell hardness measurement

The effect of load application time is shown in Fig. 2. The vertical axis shows

the difference of hardness value compared to the data obtained with the time, and reading time, respectively. There was almost no difference in hardness for brass and PVC specimens even the load application time has been changed from 2 s to 10 s. However, the softest materials among the four polymer specimens, PE, showed the largest difference in hardness value, from -2.5 HR to 3 HR. This means that strict control on the load application time for soft polymer is very important to get reliable data with a good repeatability.

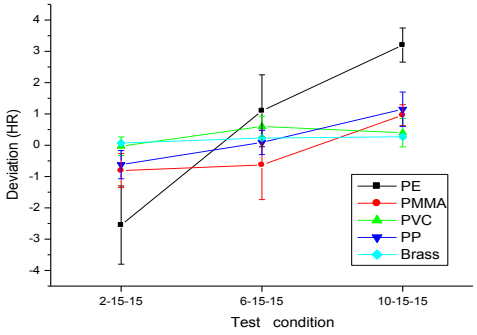

Fig. 2. Deviation of hardness values with the variation of load application time from 2 s to 10 s. The amount of deviation was calculated based on the hardness value obtained from the standard condition, 4-15-15

This means the load application time, total test force duration time, reading time after release of test force, respectively, in s unit.

For the effect of test load holding time, there was no effect for brass. However, all of the polymer materials showed that the hardness decreased as the load holding time increases and PE showed the largest amount of change for the range of test condition. This means that a standard test condition, 4 s, 15 s, and 15 s of load application time, test load holding slow elastic deformation has occurred during the load holding time and results in the decrease in hardness value. The results are shown in Fig. 3.

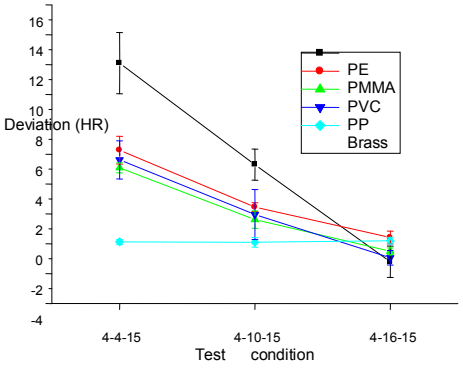

Fig. 3. Deviation of hardness values with the variation of total test force duration time from 4 s to 16 s. The amount of deviation was calculated based on the hardness value obtained from the standard test condition, 4-15-15.

When we consider the test range allowed in the ISO standard, between 11 s and 15 s, this may cause a serious difference in test data depending on polymer materials. For example, the difference of hardness for PE would be about 6.5 HRR for this range and this is about 10 % of the hardness value of PE which has about 50 HRR. Therefore, it is preferable to increase the test load holding time to obtain comparable test data. In this point of view, the test load holding time specified in JIS standard, 16 s is better than the ISO standard which specifies the test load holding time to be 10 s. The effect of reading time after the removal of test load is shown in Fig. 4.

As is expected, there is no change in the hardness value for brass hardness measurement. However, all of the polymer materials showed that hardness value increases as the reading time increases. This means that elastic recovery is going on for a while even after the test load removal. In this sense, it is preferable to keep the reading time longer than 5 s and better to be longer than 15 s. Even so, it is recommended to keep the reading time strictly to reduce scattering of data.

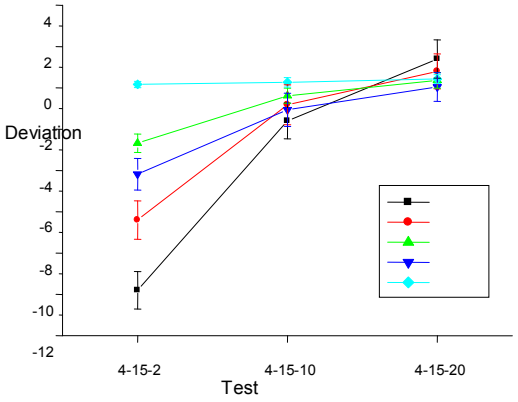

Fig. 4. Deviation of hardness values with the variation of reading time from 2 s to 20 s. The amount of deviation was calculated based on the hardness value obtained from the standard test condition, 4-15-15.

(2) Proficiency test

A proficiency test has been carried out for six laboratories. The test condition was set to be 4 s, 15 s, 15 s, for load application time, test load holding time, and reading time after test load removal, respectively. Before the test, all of the laboratories calibrated their machine with brass reference blocks.

Fig. 5 (a) and (b) shows the results of proficiency test for the four kinds of polymers. Laboratory C showed a very serious problem in test data. Except for this laboratory, all of the other laboratories showed satisfactory results and the difference of data compared to the reference value was within 5 % of reference value. However, for the PE, the data difference was almost 10 % for the laboratory C, E and F. This was the anticipated result since PE showed a highly sensitive property on the time relevant parameters.

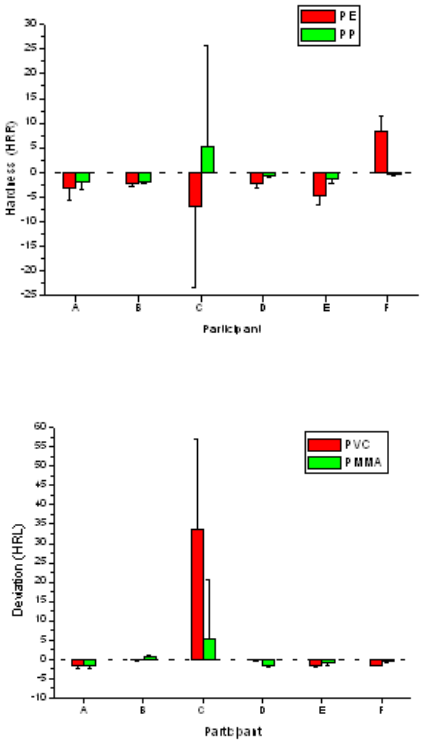

Fig. 5. Results of round robin test for HRR and HRL Rockwell hardness scale. (a) PE and PP, HRR scale. (b) PVC and PMMA, HRL scale. Deviations are calculated based on the KRISS value.

Overall, strictly controlling time parameters is very important, especially

for highly sensitive materials on time related parameters such as PE, to obtain reliable and repeatable test data with lower scattering range of data.

(3) Standards for Polymer hardness measurement

Table 2 is a summary of standards for polymer hardness measurement. It can be found that there are differences in the time related parameters among the standards.

For the load application time, ISO specifies that additional test load should be applied within 10 s after the preliminary test force was begun to be applied. A more detailed condition is that preliminary test force should be maintained within 3 s and the time for the application of additional test force should be between 1 s and 8 s. The duration time for the total test force is 4 s ± 2 s.

However, it is also required that the load should be released 15 s later after the additional test force was begun to be applied. Considering the fact that it takes about 4 s to 5 s for the application of additional test force, actual total test force duration time is about 10 s to 11 s. This is quite different compare to the requirements of total test force duration time, which should be 4 s ± 2 s. In other words, it is not possible to satisfy all of the requirements of ISO standards.

Reading time is specified also 15 s later after the beginning of release of additional test force for ISO, ASTM, and KS. However, it is specified 15 s later after the ending of the release of the additional test force for JIS. In this case, the reading time is about 2 s longer for JIS.

The overall effect of the difference in hardness measurement was compared by testing the four kinds of polymer materials according to the ISO and JIS standards. Fig. 6 shows the difference in hardness values and as it is expected the difference in PE is the largest and it reached to about 5 % of hardness.

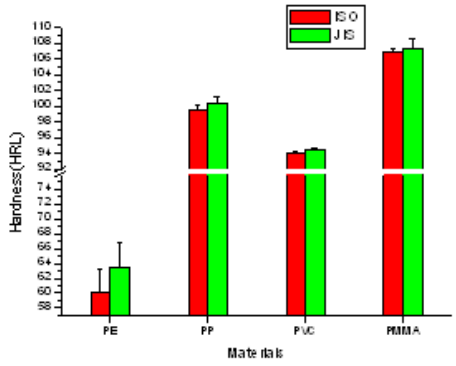

Fig. 6. Differences in hardness values obtained by JIS and ISO standards

Fig. 7 is the summary of the test results showing the effect of time related parameters for the four kinds of polymers. The vertical axis shows the total range of variation for the given variation of the parameters such as load application time, total test force duration time, and reading time. Overall, the variation was the largest for PE, followed by PP, PVC, and PMMA.

Table 2. Comparison of ISO, ASTM, JIS and KS standards for Rockwell hardness measurements.

Norm	**ISO** 2039-2 (1987)	**ASTM** D 785 (1993, Proc. A)	**JIS** K 7202 (1982), K 7202 (1995)	**KS** M 3037 (1983)
Indenter diameter (mm)	R: 12.7±0.015 L: 6.35±0.015 M: 6.35±0.015 E: 3.175±0.015	R: 12.7±0.0025 L: 6.35±0.0025 M: 6.35±0.0025 E: 3.175±0.0025 K: 3.175±0.0025	R: 12.700 L: 6.350 M: 6.350, deformation less than 0.002	R: 12.700 L: 6.350 M: 6.350
Preliminary load	98.07 N± 2%	10 kgf	10 kgf {98.07 N}	Same as left.
Calibration of preliminary load	Not specified.	Not specified	± 2% at (Measurement position; 130)	Same as left.
Test load(N)	R: 588.4 ±2% L: 588.4 ±2% M: 980.7 ±2% E: 980.7 ±2%	R: 60 kgf L: 60 kgf M: 100 kgf E: 100 kgf K: 150 kgf	R & L: 60 kgf {588.4 N} M: 100 kgf {980.7 N}	Same as left.
Calibration of test load	Not specified.	Not specified	60 kgf; ± 0.7 (Measurement position; 100 ± 5) 100 kgf; ± 2% (Measurement position; -70 ± 5)	Same as left.
Indicator accuracy	0.001 ㎜	0.001 mm	At 30, 50, 70, 90 & 110, Less than ±0.5 Hardness value	Same as left.
Adjustment of load application velocity	4 ~ 5 s with no specimen at 980.7 N load	4 ~ 5 s with no specimen at 100 kgf {980.7 N} load	About 5 s with no specimen at 100 kgf {980.7 N} load	4 ~ 5 s with no specimen at 100 kgf {980.7 N} load

Among the three parameters, the duration time of total test force was the largest factor affecting the test results. Compared to the results of brass reference block, it could be concluded that when testing polymer materials, strict control of total test force duration time and final reading time are very important to increase the repeatability of test data.

III. Conclusion

The effect of time related test parameters were studied for the four kinds of polymers. It was found that as the portion of elastic deformation is larger, the test data are more affected by those parameters. In this study, among the four kinds of polymers, PE, PVC, PP, PMMA, polyethylene showed the largest variation in test data for the given variation of test condition.

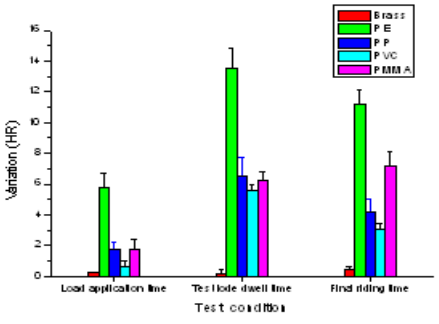

Fig. 7. Variation of hardness values depending on the variation of time related test parameters.

Considering the above test results, it can be concluded that the optimum test

condition is 4 s for additional test force application time, 15 s for total test force duration time, and 15 s for reading time after the release of the additional test force is finished.