Paper No. 4

"Reducing the Duration of IRHD Hardness Tests"

by S. Lackovic[1] Bsc PhD, R. Morgans[2] Bsc and D. Reynolds[1]*

1. H W Wallace & Co. Ltd
Curtis Road Industrial Estate
Curtis Road
Dorking
RH4 1EJ
United Kingdom

2. University of Greenwich
School of Engineering
Medway Campus
Chatham Maritime
Kent
ME4 4TB
United Kingdom

Presented at a meeting of the

Rubber Division, American Chemical Society

Cleveland, Ohio

October 21-24, 1997

*speaker

ABSTRACT

Hardness measurement is a practical way of determining the degree of vulcanisation of a rubber sample. The IRHD (International Rubber Hardness Degree) method is one of the most important and commonly used measurements for non-destructive hardness testing.

International testing standards require a primary load of 5 seconds and a secondary load of 30 seconds. H W Wallace has developed a predictive technique, enabling these times to be significantly reduced.

A programme of research was carried out on different rubber types (and thicknesses) at varying temperatures using a Wallace Cogenix Micro Hardness Tester. Further investigations were made using a Cogenix Dead Load Hardness Tester (H14).

The tests were conducted to determine the relationship between indentation (IRHD) and time. A general equation was derived which enabled the 30 second hardness value to be calculated after a few seconds of "secondary" load application. The effects of the "primary" load time was also investigated.

The predictive technique is incorporated into the newly launched Data Collection Software, producing a rapid, accurate prediction of the standard result, with a high confidence level.

INTRODUCTION

Hardness measurement is one of the most commonly used methods of quality and product control and has been in existence since the early 1900's. The standard (non-destructive) IRHD (International Rubber Hardness Degree) method (such as ISO 48:1994[1]) uses an annular foot to hold the sample in place. A spherically tipped indentor is then applied under a constant "primary" load to the sample surface for 5 seconds. A "secondary" load is then applied for an additional 30 seconds, giving the indentor an incremental displacement which is measured and scale-converted to an IRHD reading.

The "secondary" load period was originally set at 60 seconds, but it was felt that a reduction in this time would be desirable, especially when testing large numbers of samples. The influence of the period of application of the load was investigated by Gurney[2] and later by Dock[3]. As a result of this work, the 30 second time was commonly adopted.

It was observed that skilled operators could 'predict' the hardness of a sample much earlier in the measurement cycle. For this to be the case, there has to be a common family of hardness curves for a majority of sample types and ambient measurement conditions. This would allow a general equation to be derived, which in turn would lead to a specific, predictable curve for any one measurement. This curve could then be used to predict the 30 second value at a much earlier point in the measurement cycle.

Ambient temperatures are specified in the standard to be 23 ± 2 °C. However, since these conditions are not always observed in practice, the effects of temperature on the prediction accuracy were also studied.

EXPERIMENTAL

Both micro hardness and dead load instruments were used to perform tests on a variety of samples, in the hardness range 30-95 IRHD. The results were recorded every 0.5 seconds, giving 60 stored data points for each test, which were then logged on a PC. The standard load times of 5 and 30 seconds were used.

Although the standard[1] specifies a temperature of 23 ± 2 °C, tests were also conducted at varying temperatures to determine what, if any, effect the temperature would make to the predictability of the sample's endpoint. The instrument and samples were tested from 30°C to 45°C in 5° steps and at 12°C. The samples only were heated to 65°C and tested on an instrument at room temperature.

Samples of thicknesses other than those specified in the standard[1] (2mm ± 0.5mm for the micro test and 8-10mm for the dead load test) were also tested to determine the effect of sample thickness on the predictability of the sample's endpoint.

All data were plotted and a random selection of measurements was chosen for a variety of samples and temperatures. The data were normalised and superimposed to test the assumption of the similarity of the curves' shape. The normalised data were then used to fit a general equation of the form:

$$y = a + bt^{-c}\,, \qquad \text{where } c \text{ is a constant} \tag{1}$$

Each sample will have a unique solution to equation (1). For each sample, two equations in the form of equation (1) can be created, using two t (time) values (t_1 and t_2) with their corresponding y (hardness) values (y_1 and y_2). These can then be solved simultaneously to provide unique values of *a* and *b* for a particular sample. These values can then be substituted into equation (1) to give a curve for that sample.

The general equation was fitted at different *t* values to see which two values of *t* gave the best fit for the majority of samples. Although it was desirable to fit as small a *t* value as possible, it was also important for the equation to fit as large a number of samples as possible. This ensured a high degree of accuracy whilst giving the greatest possible time reduction. The equation, along with the chosen *t* values, was re-checked using a selection of data.

For each sample, the equation and two *y* values at given *t* values, can be used to produce a 'predicted 30 second' value. This was compared for each sample to the actual value measured after the full 30 seconds during the same test on the same sample. This gave a measurement of prediction accuracy.

The primary load provides a datum point from which to measure the indentation depth. The standard specifies 5 seconds; investigations of this time were carried out.

RESULTS

As each sample was tested to the standard 30 second load time, it was possible to calculate a predicted 30 second value using the previously agreed t_1 and t_2 values with their corresponding y_1 and y_2 values. This was after only 6 seconds (t_2) of secondary load time. This enabled the actual 30 second values and the predicted 30 second values to be compared. Out of the 380 samples measured, 366 of the predicted values were in agreement with the actual 30 second values to within 0.5 IRHD. The samples within this agreement included samples from the entire hardness range (30-95IRHD) as well as those of non-standard thickness and those that were tested at varying temperatures. The 14 samples that did not agree to within 0.5 IRHD did, however, agree to within 1 IRHD. This was found to be as a result of the sample not being completely flat whilst being tested.

The compounds that were tested are listed in table 1 and covers the full range of hardnesses tested, as well as including the non-standard samples used. Each of the samples was also tested more than once to ensure prediction repeatability.

In figure 1, a selection of samples of varying hardnesses are plotted, indicating their actual 30 second point as compared to their predicted 30 second value. It can be seen that there is good agreement between the predicted and actual endpoints over the entire hardness range, 30-95 IRHD.

Although a majority of the tests carried out were on samples of a standard thickness relevant to the instrument used (2mm ± 0.5mm for the micro and 8-10mm for the dead load) some tests, where possible, were carried out on samples of varying thickness. This ranged from 1-4mm for the micro and from 4-10mm for the dead load. However, the thickness of the sample made no observable difference to the predictability of the 30 second endpoint.

A typical graph can be seen in figure 2. The sample shown here is neoprene, and it demonstrates the predictability of the indentation curve with respect to time. The graph shows the standard 30 second data (illustrated in fig. 2 from 2.5 seconds for clarity of the curves shape) along with a predicted curve, which begins at 6 seconds and culminates with a predicted 30 second value. This graph allows a comparison to be drawn of the actual against predicted data for one particular data series, and in this case, these endpoints are within 0.5 IRHD.

Investigations were carried out to determine the effect of reducing the primary load time. It was found that reducing the standard 5 second primary load time to 1 second made no significant difference to the endpoint.

DISCUSSION

The investigation was carried out on both Dead Load and Micro Hardness instruments. Many samples were tested to ensure a repeatable system. The compounds tested are listed in Table 1.

The curve shape was predictable in all cases including the use of non-standard tests. The hardness prediction accuracy can be determined for a given compound by running the predictive software and the standard test to conclusion and comparing the endpoints. In this way, consistency is achieved because with only one test being carried out, only one position on the rubber is used. Hence the sample variability does not interfere with the results allowing a true comparison of the accuracy of the prediction.

This method provides a significant reduction in the test cycle time in situations where it is not necessary to adhere strictly to the standard, for example, in-house comparative testing. A time reduction may be beneficial to users for two reasons - the same number of tests can be carried out more quickly or more tests can be carried out in the same time.

When a new sample type is used, the prediction validity can be proved by the user. As well as running one test to the reduced time period and producing a predicted endpoint, a test can be carried out to the full 30 seconds. In this way, a comparison can be drawn as to the reliability of the prediction. However, care must be taken since each new test is in a new position on the sample. When testing rubber, sample hardness usually exhibits variations of a few IRHD units across the surface. As a result, a predicted test carried out to a shorter time in order to make a prediction of the 30 second value will also be subject to this. So agreement to within the normal amount of fluctuation is expected and is observed in practice.

The equation and mathematics used to fit the curve, and hence predict the hardness value, is incorporated as an additional extra in to the newly launched Wallace Data Collection Software.

CONCLUSION

The standard 35 second IRHD test time (5 second primary load and 30 second secondary load time) for a rubber sample can be significantly reduced by fitting a general equation of the form given in Equation (1). The equation can be solved for each sample to produce an accurate prediction of the ‘final’ value with a high confidence level. Furthermore, the standard 5 second primary load time can be reduced to 1 second.

The prediction accuracy required determines the minimum test time. To ensure an agreement between the predicted value and the actual value to within 0.5 IRHD, the total test time can be reduced to 7 seconds (1 second primary time and 6 seconds secondary load time). For less accuracy, such as agreement within 1 IRHD between the predicted and actual values, the total test times can be reduced to 5 seconds. This time can be reduced still further but with less accuracy between the actual and predicted endpoints.

REFERENCES

1. ISO 48: 1994, Physical Testing of Rubber, Method for determination of hardness
2. H. P. Gurney, India Rubber Journal, p17-22, (1921).
3. E.H. Dock, J. Rubber Res., Vol. 13, p19-22 (1944)

TABLE 1
SAMPLES TESTED

Sample Type	Micro	Dead Load
Chloro compound	3	3
CR compounds	3	3
EPDM compounds	3	3
EPM compounds	3	
FKM compounds	3	
H W Wallace calibration blocks	3	3
H W Wallace samples	3	3
IIR compounds	3	
NR compounds	3	3
NBR compounds	3	3
Nitrile/PVC compound	3	
Polyurethane compounds	3	3
Q compounds	3	3
Thiokol compound	3	

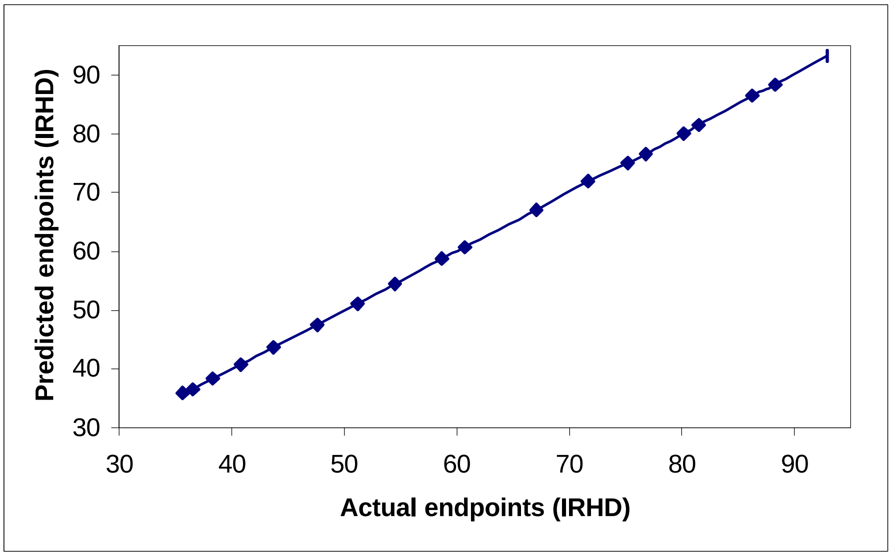

Fig. 1.– Graph demonstrating the agreement between the actual and predicted 30 second endpoints

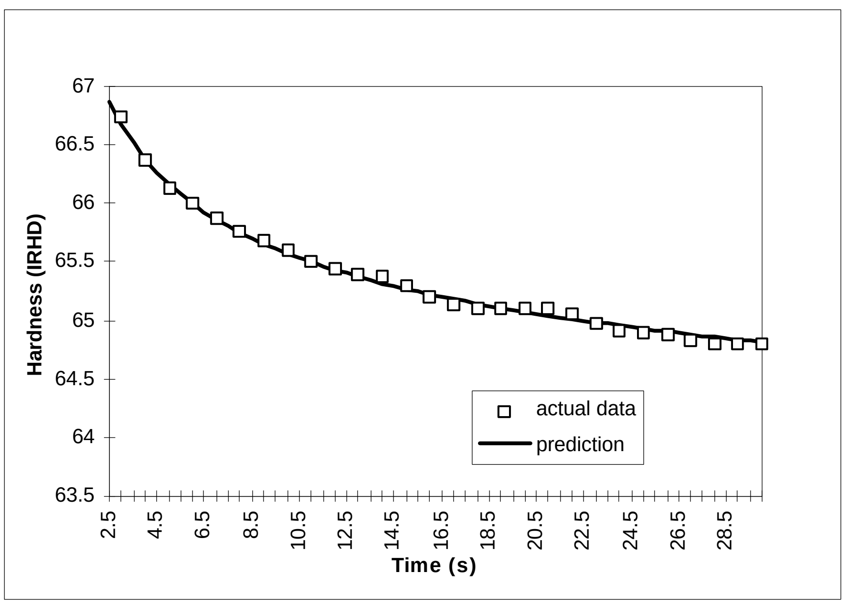

Fig. 2.– Graph showing data collected from a sample of neoprene, with its predicted curve