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APPRAISAL OF THE METHOD OF DETERMINING MICRO-HARDNESS BY MEANS OF THE REICHERT MICRO-HARDNESS TESTER

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SUMMARY:

For measuring the micro-hardness of material like magnetite, the time consuming production of a large number of impressions is shown to be unnecessary and all the impressions need not be made on the same individual grain.

Although the accuracy of the average values improves with the number of data included in the average, a limit is imposed by the experimental error (S est = ± 0,6 and r = ± 0,9985) at about 50 measurements per load, or a total of about 400 measurements.

The correlation coefficient r is an independent criterion for the accuracy of a series of measurements.

INTRODUCTION:

During tests on the characteristics of magnetites use was made of the Reichert Micro-Hardness Tester.

In order to explain the results obtained with the instrument, it was necessary to evaluate this method of determining micro-hardness and it was decided to make this the subject of a separate memorandum.

As no explicit instructions are given in the pamphlet* as to how many indentations are required, or how many times a single indentation should be remeasured, these points had to be clarified.

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* "Micro-Hardness - Its Theory and Practice with the Reichert Micro-Hardness Tester" - Optische-Werke C. Reichert, Wien 1950.

METHOD:

To find the optimum number of measurements the approach was as follows:

It was thought that the larger the number of measurements that are combined to produce an average value, the more accurate this average value is likely to be, within the bounds set by unavoidable experimental errors.

For small loads, over a relatively short range, the relation of the load to the indentation diagonal is: $P = a.d^n$, where P is the load in ponds**, d the indentation diagonal in microns and a and n material constants (page 11 of the pamphlet*). This relation may be expressed as a straight line in logarithmic coordinates: $\log P = n \log d + \log a$.

The method was to calculate the regression line from averages of all the values. The cumulative averages made up of increasing numbers of measurements were then compared with the values calculated from this regression line by means of computing the standard deviation S_{est} from the estimated values.

The correlation coefficient r was also calculated separately for the different series of average values.

Though the pamphlet* (page 36) instructs that all the indentations be made on a single individual, this may not be possible where the grains are relatively small and a large number of indentations need be made. It was therefore decided also to test whether magnetite has an appreciable anisotropy of hardness by the calculation of standard deviations. If its hardness anisotropy is small, indentations may be made on

/different .....

** One pond is the force exerted by a mass of 1 gram in the earth's gravity field.

different grains. A large number of orientations are also preferable for the determination of an average micro-hardness for an individual specimen made up of smaller grains.

THE DATA:

Eight points were considered sufficient for the calculation of the regression line, so that indentation diagonal measurements were made at eight different loads. All the tests were done on a powder mount of Allanwood magnetite.

The data consist of the following:-

(i) A total of 200 measurements of 200 impressions on five individual grains, i.e. 40 measurements, five for each different load, were made on each grain.

(ii) A total of 280 measurements of 80 impressions on 80 individual grains. The impressions were re-measured from two to six times.

All the readings were combined and tabulated at random for a particular load. The standard deviation and deviation from the mean for the data above, for each load are given in Table 1, together with measurements on a single indentation diagonal, repeated 60 times.

In Table 2 appear the average values calculated from progressively larger numbers of measurements. Values calculated from the regression line are given ("estimated values") as well as the standard deviation from the estimated values. The correlation coefficient was computed separately for each series of average values.

Figure 1 is a graphical representation of Table 2.

DISCUSSION:

From Table 1 the standard deviation of 60 measurements of a single impression is ± 3,28 drum scale units. This falls within the range ± 3,00 to ± 4,04 computed from the same number of measurements per load, but for 35 impressions per load. An identical result is obtained for the other parameters.

It is apparent that the standard deviation of the measurements of a single impression is indistinguishable from that of 35 impressions at a particular load. Thus it appears, that for magnetite, the inherent error in measurement overshadows differences in micro-hardness due to different grain orientations or to errors in applying the load.

The conclusion may be drawn that it is not necessary to confine all the impressions to a single grain (single orientation). It is also unnecessarily time consuming to make a large number of impressions, as fewer impressions may be remeasured, to yield the same result.

In Table 2 average values for progressively increasing numbers of measurements are compared with values calculated from the regression line for an average of 60 measurements per load. The standard deviation from the estimated values for a single measurement per load is ± 3,27 drum scale units. For an average of four measurements per load, the deviation is reduced to ± 1,44 drum scale units. Thereafter the improvement is less and less pronounced, approximating to the curve in Figure 1.

It is significant that at 49 measurements per load (total number of measurements 392) a minimum deviation of ± 0,57 drum scale units occurs, which is slightly better than the deviation at 60 measurements per load (total number of measurements 480) of ± 0,59 drum scale units. At 49 measurements per load the point has already been reached where increasing the number of measurements does not improve the averages. This must be due to the inherent experimental error and as a general rule, it is probably pointless increasing the total number of measurements to beyond 400, or an average of about 50 measurements per load.

The correlation coefficient $r = \pm\sqrt{\frac{\Sigma(Yest - \bar{Y})^2}{\Sigma(Y-\bar{Y})^2}}$, where Yest is the value calculated from the regression line, $\bar{Y}$ the mean of the experimental values and Y the experimental value, is a measure of the spread of points about a straight line. When all the experimental values fall on the line r = 1,0000. As the slopes of the regression lines in the present instance vary from + 1,6482 to + 1,8114, the influence of this factor on the correlation coefficient may be safely ignored.

The correlation coefficients given in Table 2 were calculated independently from the estimated values given in the table. For each series of average measurements, the drum scale values were converted to d, the regression line: log P = n log d + log a calculated, and the spread of the points about this particular line computed.

A single measurement per load had a correlation coefficient of + 0,9540, at an average of four measurements per load it had improved to + 0,9929, whereafter the increase was relatively smaller to a maximum of + 0,9985 at an average of 49 measurements per load, or a total of 392 measurements.

Although calculated independently from the estimated values given in Table 2, the improvement of the correlation coefficient with increasing number of measurements in the average is similar to the improvement noted for the standard deviation from the estimated values and the same conclusions apply. Also, it seems that on its own, the correlation coefficient gives a very good measure of the accuracy of the average data.

/CONCLUSIONS .....

CONCLUSIONS:

Comparison between standard deviations of measurements for a single impression with 35 impressions shows that for a material like magnetite, it is unnecessary to produce a large number of impressions. Also all of the impressions need not be made on the same grain.

The standard deviation from the estimated values and the correlation coefficient, calculated independently, both indicate that no improvement of the average values occurs when the total number of measurements is increased to more than about 400. Limiting values of about $\pm$ 0,6 drum scale units for the standard deviation from the estimated value and a correlation coefficient of r = + 0,9985 are reached. These limits are the result of the unavoidable errors in the measurement of indentation pyramids.

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RESEARCH OFFICER

Pretoria.
13th December, 1972.
JLG/EMc

TABLE 1

Standard Deviations $S = \pm\sqrt{\frac{1}{N-1}\ \Sigma(x-\bar{x})^2}$, and Deviation from the Mean (in Drum Scale Units) for Different Numbers of Impressions. 60 Measurements were made for each Load.

Number of Impressions	35 Impressions per Load[1]								One Impression[2]
Load (ponds)	19,00	22,41	25,82	29,23	32,63	36,01	39,41	42,80	34,33
Arithmetic mean $\bar{x}$	57,60	62,46	66,99	72,02	75,19	78,32	81,21	85,17	82,23
Standard Deviation S	± 3,66	± 3,30	± 4,04	± 3,08	± 3,00	± 3,05	± 3,41	± 3,53	± 3,28
" " S %	± 6,35	± 5,28	± 6,04	± 4,28	± 3,99	± 3,89	± 4,20	± 4,15	± 3,99
Std. Dev. from Mean $S\bar{x}$	± 0,47	± 0,43	± 0,52	± 0,40	± 0,39	± 0,39	± 0,44	± 0,46	± 0,42
" " " " $S\bar{x}$ %	± 0,82	± 0,68	± 0,78	± 0,55	± 0,51	± 0,50	± 0,54	± 0,54	± 0,51

1) i.e. each impression was measured from 1 to 2 times for a total of 60 measurements.

2) i.e. a single impression was measured 60 times for a total of 60 measurements.

TABLE 2

Cumulative Average Values for Increasing Numbers of Measurements, the Estimated Value, Standard Deviation from the Estimated Value Sest (all in drum scale units) and the Correlation Coefficient r

Total	Average per Load	Load in ponds								Sest*	r**
		19,00	22,41	25,82	29,23	32,63	36,01	39,41	42,80		
8	1	59,90	61,90	62,50	74,80	76,60	74,90	81,40	88,40	± 3,27	+ 0,9540
32	4	59,43	61,93	66,80	71,48	77,80	78,73	82,73	85,25	± 1,44	+ 0,9929
72	9	57,22	61,67	68,07	72,27	76,00	78,04	80,37	85,20	± 1,20	+ 0,9935
128	16	56,89	61,96	68,19	71,64	75,81	77,68	80,47	85,18	± 1,12	+ 0,9943
200	25	56,44	62,22	67,06	72,30	75,70	78,21	81,02	84,69	± 1,04	+ 0,9952
288	36	57,55	62,00	67,01	72,51	75,11	78,29	81,25	85,00	± 0,78	+ 0,9972
392	49	57,68	62,16	67,12	72,08	75,03	78,17	81,52	85,21	± 0,57	+ 0,9985
480	60	57,61	62,47	67,00	72,04	75,20	78,34	81,23	85,18	± 0,59	+ 0,9985
Estimated Value***		57,98	62,60	66,91	70,97	74,82	78,46	81,98	85,35	± 0,00	+ 1,0000

NOTE: * Standard deviation from the estimated value calculated with two degrees of freedom as the points represent a line $Sest = \pm\sqrt{\frac{1}{N-2}\ \Sigma(x - x_{est})^2}$

** The correlation coefficient was computed from the spread of points around the individual regression lines for each average measurement independently of the estimated value $r = +\sqrt{\frac{\Sigma(Yest - \overline{Y})^2}{\Sigma(Y - \overline{Y})^2}}$

*** The estimated values lie on the regressive line calculated for an average of 60 measurements per load i.e. log P = 1,8112 log d - 0,3305. Drum scale units are converted to d by subtracting 9,41 and multiplying by 0,1594.

FIGURE I

CUMULATIVE AVERAGE VALUES vs. ESTIMATED VALUES. STANDARD DEVIATION FROM THE ESTIMATED VALUES Sest. CORRELATION COEFFICIENT r