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Ferrography에서 샘플희석률이 마모입자 정량분석에 미치는 영향

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The Effect of Dilution on Particle Deposition in The Entry Deposit of The Ferrograms

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요 약

Ferrography 방법은 유체중 마모입작를 Substrate glass 위에 Van der Waals 힘에 의하여 분산교착 시키므로써 윤활Systems에서의 기계요소들의 동특성적 마모진행과정및 mechanism의 정성적및 정량적인 분석을 가능케 한다. 정성적 방법으로는 광학현미경으로부터 SEM, X-Ray Emissiom, EDAX 등의 분석방법이 적용되고 있으나, 정량적으로는 마모입자의 크기별 분포함수에 따른 Variance, Kurtosis, Skewness와 같은 인자함수의 변이가 마모진행 과정과 진행될 마모형태기구의 예측인자로서 연구되고 있다. 수치적 Model로 마모진행기구를 예측할 경우 third moment의 Skewness가 Size 및 Number 분포에 따라 예민하게 반응을 나타내는 결과를 보이고 있다. 이러한 정량분석을 위하여는 실험 Sample에 따라서, 즉 오염도에 따른 Sample 희석이 따르는데, 이러한 Sample 희석이 정량적 분포에 미치는 영향에 관한 연구결과는 아직 없다. 따라서 본연구에는 용제희석이 정량적 마모입자 분포에 미치는 영향을 image analyser인 Quantimat720을 이용하여 검토하였으며 전영역의 희석률에 적용될 수 있는 Standard Calibration function에 따른 수학적 model을 유도하였다.

ABSTRACT

Ferrograms prepared from oil samples collected during testing in the transition region were originally diluted at 20:1. To obtain some information about the effect of dilution on the analysis procedures, a series of measurements were made on ferrograms prepared to different dilutions in the range 6 to 30:1 from oil samples collected after testing in the four ball machine at the 51 kg and 55 kg load, respectively, Fig. 1. The variations in area covered, perimeter, intercept and particle count were then plotted as a function of dilution level and appropriate mathematical expressions established such that the results obtained at any dilution level specified within the range can then be corrected back to an equivalent undiluted value. The effect of dilution on the variance of the particle size distribution was also investigated.

The main results are tabulated, Tables 1-5 and also plotted as a function of dilution, level Figs. 2-9.

1. Sample Preparation and analysis in Quantimet

Six oil samples were prepared with the 55 kg-load sample in different ratios of dilution, and four samples with the 51 kg-load sample. The same lubricant, SAE # 10 base oil, supplied straight from its container, was used as the diluent for the samples. Initially, a ferrogram of the diluent oil was prepared and examined to determine whether it was contaminated by any foreign particles. Having confirmed that it was quite clean, preparation of ferrograms from diluted test samples was carried out in the normal manner and then analysed in Quantimet. Throughout the analysis, the field of view was located in the dense entry region and measurements were made in accordance with the following conditions of analysis:—

Conditions in Quantimet 720

Magnification: object lens 25 x, condenser 6.3 x

Lighting : reflected light source

Detection : lighter than background

1 picture point is equivalent to 0.65 μm

Ferrogram slide details

Slide Nos. 51- 5, 51-10, 51-15, 51-20 — ferrograms prepared from a single oil sample of 51 kg-load. (The last numbers indicate the dilution ratios.)

Slide Nos. 55- 0, 55- 5, 55-10, 55-15, 55-20, 55-30 — ferrograms prepared from a single oil sample of 55 kg-load.

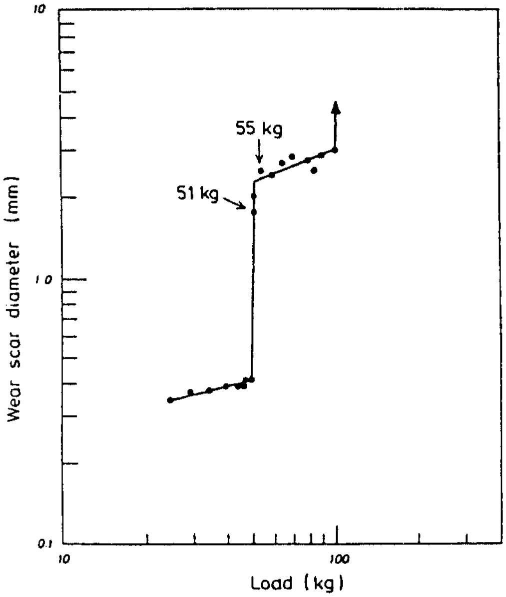

Fig. 1. Location of oil samples used to determine the effect of dilution on particle distribution 4 ball test (SAE 10)

The repeatability of measurement in Quantimet analysis was very sensitive to the detection level used, but it was usually within 3% for the same position of detection under the conditions of optimum adjustment of detection level. The measurements of number of particles, area, intercept and perimeter were limited to the 90° orientation setting on the microscope camera head.

2. Data analysis and processing

The results obtained by Quantimet analysis are tabulated in Tables 1 and 2, and plotted as shown in Figs. 2-5. Fig. 2 shows the variation in the total number of particles with sample dilution. The total particle count in this instance represents the sum of the count for particles $\leqslant 1$ pp and > 1 pp, respectively. The variation, with dilution, of particle count within specific size bandwidths is shown in Fig. 3. Methods for analysing the wear debris in lubricated systems

Table 1. Initial measurements obtained for evaluation of dilution effect – 51kg

Sample diluton	Total No. of particles, (<0.5m + >0.5 μm)	Area covered(A) (pp)	Intercept(K) (pp)	Perimeter(P) (pp)	Weibull variance $(pp)^2$
5	362	2,303	667	3,116	3.4529
10	78	726	142	661	6.0820
15	60	432	114	518	2.5943
20	78	360	119	549	1.0946

Table 2. Initial measurements obtained for evaluation of dilution effect – 55kg

Sample dilution	No. of particle sized					Total No. of particles	A (pp)	I (pp)	P (pp)	Weibull variance $(pp)^2$
	≤0.5μm	0.5–2	2–3	3–5	>5					
0	404	345	137	51	36	973	5144	1712	8019	7.4578
5	179	124	69	36	50	458	4753	1140	4900	9,7084
10	110	77	44	10	11	252	1336	474	208	4.6852
15	76	49	30	13	8	176	1008	331	1521	3.6846
20	22	23	9	6	0	60	269	104	487	4.7760
30	37	21	12	7	6	83	611	173	757	6.6265

all depend on the transporting properties of the lubricant when functioning as a carrier medium to transfer the debris from the source of wear to the sampling point and are also all size-dependent. Hence the particle size variation within the particle population is of primary

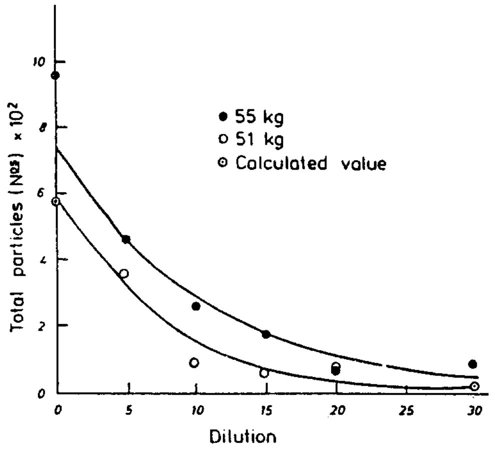

Fig. 2. Total Particle count vs dilution 51 and 55 kg oil samples from 4 ball test (sae 10).

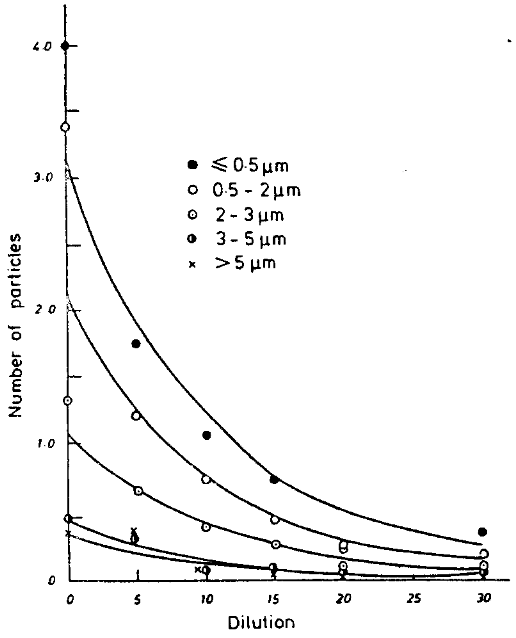

Fig. 3. Particle count within specific size bandwidths vs dilution.

consideration in establishing the wear state of a system from the information which can be obtained from a sample. Hence any variation in particle size distribution, will tend to influence the interpretation of the results obtained in relation to determining the true nature of the wear situation. In Fig. 3, each particle size distribution within a specific particle size bandwidth is exponentially decreasing with increasing sample dilution. The effects of dilution on Area, Intercept and Perimeter for the 51 and 55 kg-load conditions are shown in Fig. 4 and 5.

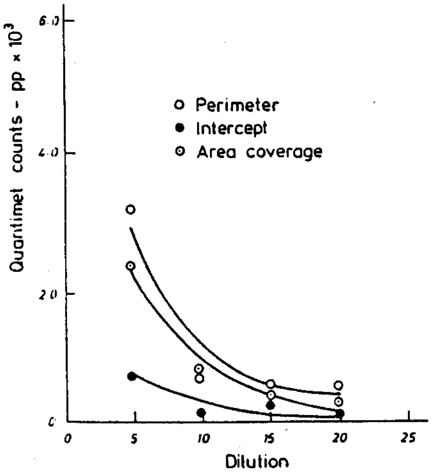

Fig. 4. **Variation in geometric parameters with dilution – 51 kg sample.**

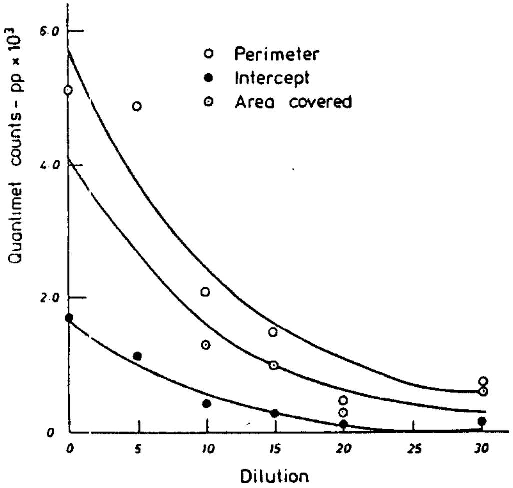

Fig. 5. **Variation in geometic parameters with dilution – 55 kg sample.**

In establishing a general function in relation to sample dilution, the results of Fig. 2-5 can be generally expressed in the form of a hyper-exponential function, as follows,

where

$$y = a \exp(-bx) \tag{1}$$

y = number of particles or a specific parameter (Area, Intercept or Perimeter)

x = degree of sample dilution

a and b = constants being calculated from the measurements.

The general expression in eq. (1) can be linearised by a logarithmic transformation as follows, where

$$\ln y = -bx + \ln a \tag{2}$$

b = slope of the function

a = particle count at any position of x dilution

By using equation (2), the results shown in Fig. 2 and 3 were linearised and plotted against sample dilution as shown in Fig. 6 and 7 in which the least squares method was used to determine the best straight line. Table 3 shows the results

Table 3. Calculation of slopes of particle distribution functions due to sample dilution

51kg load sample (Fig. A6)	**slope, b**	γ
Total No. of particles	0.097	0.7659
$\leqslant 0.5\ \mu m$	0.098	0.7443
$> 0.5\ \mu m$	0.098	0.7916
	Avg. 0.098	0.7673

55kg load sample (Fig. A7)	**slope, b**	γ
Total No. of particles	0.089	0.9140
$\leqslant 0.5$m	0.088	0.8922
0.5 – 2 μm	0.094	0.9456
2 – 3 μm	0.089	0.9245
3 – 5 μm	0.071	0.9668
$> 5\ \mu m$	0.095	0.7260
	Avg. 0.088	0.8800

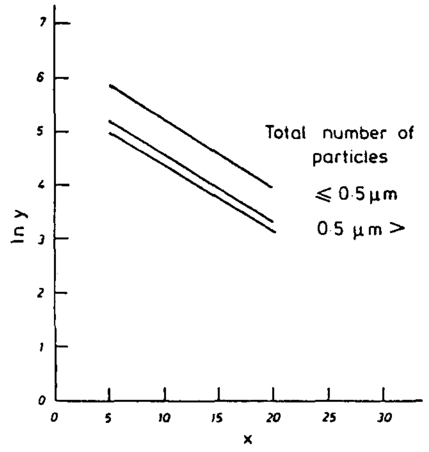

Fig. 6. Exponential plots of particles vs dilution (x) at 51 kg.

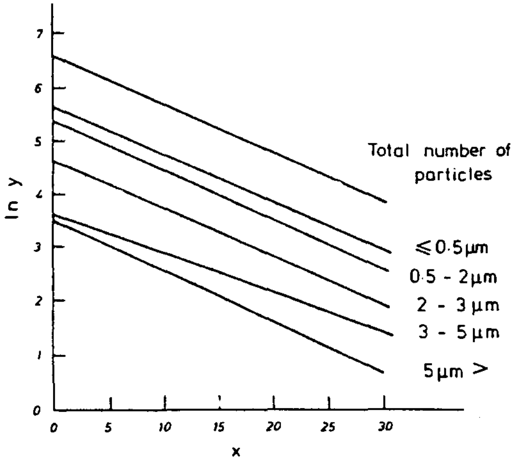

Fig. 7. Exponential plots of particles vs dilution (x) at 55 kg.

of calculation by eq. (2), in which the correlation coefficient indicates the goodness of fit.

As can be seen from these results, the slope value is quite small with the values obtained for the 51 kg-load being slightly greater than those obtained at the 55 kg-load, although there is a noticeable reduction in the correlation coefficients of the former. The variation in slope for different size range at the same load is fairly small, and this indicates that the particle distribution is not markedly affected by sample dilution.

In Table 3 and Fig. 6, the variation in slope value of the larger particle distribution (i.e. 3 – 5 μm, > 5 μm) from the average value of the smaller particle size distributions may have resulted because there were relatively few particles deposited in the larger size range as dilution was increased. Furthermore, in making measurements in Quantimet using a ferrogram in which very few particles are deposited, it is extremely difficult to select a field of view which is truly representative of the particles' distribution.

3. The effect of sample dilution on the Weibull distribution function

The Weibull function provides an indication of the possibilities for relating parameters such as the slope and scale parameter of the particles distribution to the wear state. The variance of the distribution in particular shows promise as a means for obtaining important information in respect of wear transitions in terms of a single quantitative parameter.

The effect of dilution on the Weibull variance and the slope has been evaluated and the results are shown in Fig. 8. The variance and the slope are seen to vary but not markedly as

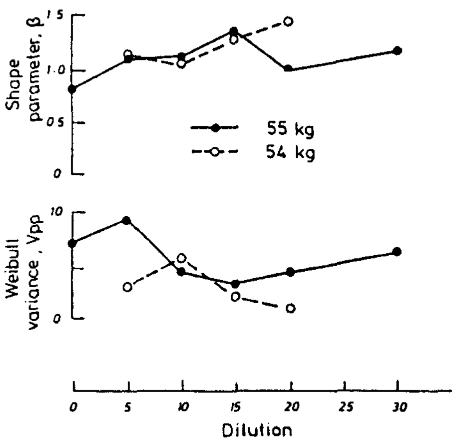

Fig. 8. Weibull variance and shape parameter, β (slope of distribution) vs dilution.

dilution changes.

To evaluate the extent to which the slope value derived from the dilution function influenced the Weibull distribution function, the number of particles for an equivalent non-diluted sample was calculated using eq. (2) for varying slope values, ranging from 0.090 to 0.150, and based on the data of slide No. 51-5 which were directly obtained by Quantimet count. The calculated particle distributions with different slope values of dilution function are shown in Table 4. Using this data, the parameters of the Weibull distribution function were calculated to establish the slope values of the Weibull dilution function, Table A5.

Table 4. The effect of slope value on correcting data to undiluted state – 51kg

No. of particles (pp) slope values	1.	3.	5.	7.	9.	11.	13.	15.	17.	19.
0.090	267	116	31	11	5	0	0	0	0	0
0.095	273	119	32	11	5	0	0	0	0	0
0.098	277	120	32	11	5	0	0	0	0	0
0.100	280	122	53	12	5	0	0	0	0	0
0.120	310	135	36	13	5	0	0	0	0	0
0.150	359	157	42	15	6	0	0	0	0	0
*slide No. 51-5	170	74	20	7	3	0	0	0	0	0

*The basic data of slide no. 51 − 5 by Quantimet count for the computation of Table 4

Table 5. Weibull Parameters calculated from the data of Table A4

Slope, b in the dilution function	Weibull slope, β	Scale parameter, α	Variance γ	γ
0.090	1.1405	2.2312	3.4992	0.9929
0.095	1.1489	2.2403	3.4637	0.9933
0.098	1.1461	2.2257	3.4399	0.9932
0.100	1.1467	2.2449	3.4941	0.9941
0.120	1.1612	2.2448	3.3860	0.9948
0.150	1.1608	2.2521	3.34107	0.9945
Slide No. 51-5	1.1510	2.2419	3.4529	0.9941

4. Effect of sample dilution on shape

To establish the effect of sample dilution on shape factor, a dimensionless grouping of the basic parameters Area (A) and Intercept (I) is obtained from using A/I^2 and this represents the simplest form of shape parameter. It offers the advantage that the results are obtained directly with only two measurements. Although it is independent of size, it is, however, seen to be dependent on the number of particles when relating it to an average aspect ratio.

The shape factor A/I^2 has been applied to the results obtained for the various dilutions. Its variation with sample dilution is shown in Fig. 9 in which it is seen that A/I^2 tends to increase exponentially with sample dilution. The scatter at higher dilution probably resulted from the low particle count.

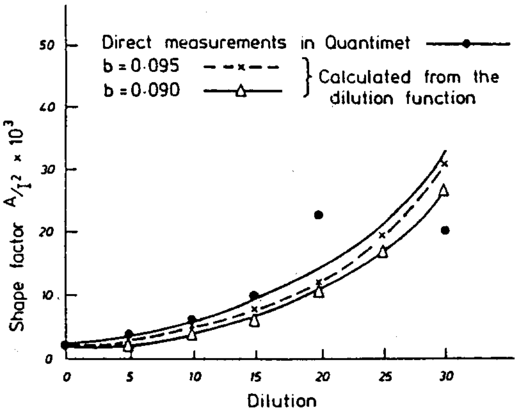

Fig. 9. **Effect of sample dilution on shape factor.**

The exponential change of the shape factor with sample dilution was confirmed from the results of the empirical dilution function. As seen from the previous results, the variation of the basic parameters, viz., number of particles, Area, Intercept, and Perimeter, with sample dilution, follow stringently the empirical equation (6)

By using the empirical eq. (6), Area and Intercept can be expressed in the form of a generalized dilution function as follows,

i.e.

$$A_x = A_i e^{-b(x-x_i)} \quad .... (3)$$

$$I_x = I_i e^{-b(x-x_i)} \quad .... (4)$$

A_x = estimated value of Area with x dilution

I_x = estimated value of Intercept with x dilution

A_i = measurement of Area by direct Quantimet count with x_i sample dilution

I_i = measurement of Intercept by direct Quantimet count with x_i sample dilution

b = constant (slope of the logarithmic dilution function).

Now, let us assume that A_i and I_i were directly counted in Quantimet under the same conditions and from the same field of view in a ferrogram, and also that the constant in eqtns. (3) and (4) have the same value in both equations, (it has been found that the constant, b value in the empirical dilution function of eq. (6) was the same for the basic parameters, such as e.g. Area, Intercept, Perimeter and Number of particles, when their measurements in Quantimet were compared with a sequent ferrogram prepared from a single oil sample.) Then the nondimensional shape factor, $A/_{I^2}$ can be expressed in terms of a general dilution function from eq. (3) and (4) as follows,

$$(A/_{I^2})_x = A_x/_{I^2_x} = \frac{A_i \cdot e^{-b(x-x_i)}}{I_i - e^{-b(x-x_i)^2}}$$

$$= \frac{A_i \cdot e^{-b(x-x_i)}}{I^2{}_i \cdot e^{-2b(x-x_i)}}$$

$$= A_i/_{I^2{}_i} \cdot [\exp -b(x-x_i) + 2b\,(x-x_i)]$$

$$= (A/_{I^2})_i \cdot e^{b(x-x_i)} \quad ... (5)$$

From eq. (5), it can be seen that the nondimensional shape factor is directly affected by sample dilution, and its value would be exponentially changed by sample dilution in terms of the exponential fraction term on the RHS in the equation (5). This has been verified with the results obtained from the data of direct Quantiment countings, Fig. 9. By using eqtn. (5), the values of $(A/_{I^2})_x$ for the various dilutions were computed from the data of slide No. 55-O, which had previously been measured in Quantimet. The computed results are plotted in Fig.

9 and compared with the results obtained from the direct measurements made at each dilution. It is seen that by using a slop value, b, of 0.095 there is quite good agreement with the experimental results.

5. Discussion and conclusions

When analysing ferrograms in conjunction with the Quantimet, sample dilution is required whenever the sample population in the dense region becomes such that the features of the particles are no longer properly distinugishable from one another. Heavy, or overlapped, deposition of particles on a glass substrate reduces the reliability of the measurements and hence increases the difficulties of interpreting the results in terms of the wear situation. It has been found that the measurement of particles in Quantimet when using reflected light overcounts as much as 25% the true value when the number of particles deposited in a single field of view exceeds five hundred particles. This is attributed to light scattering effects. The variation of the slope in the empirical dilution function varied from 0.090 to 0.100. However, the variation within the above range has not affected significantly the Weibull distribution analysis, as confirmed in Table 5.

In conclusion, these experimental results provide a useful indication of the relationship existing between diluted oil samples and an equivalent undiluted sample in which the empirical equation is of the form

$$y_x = y_i \cdot e^{-b(x-x_i)} \qquad \text{........ (6)}$$

where

y_x = estimating value of parameters (no. of particles, Area, Intercept or perimeter) at x sample dilution

y_i = Quantimet counting value of parameters at x_i sample dilution

b = constant ranging from 0.09 to 0.10 (usually b = 0.095)

x_i = sample dilution of the ferrogram for Quantimet counting

x = expecting sample dilution

(N.B.) Expecting sample dilution, x is defined as,

$$x = \left(\frac{\text{Volume fraction of original sample oil}}{\text{Total volume of sample oil diluted}} \right)^{-1}$$

These results from a useful basis for more work being undertaken to establish more precisely the basic laws of particle distribution on a ferrogram.

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