KN-77-10

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PIGMENT COLOR RESEARCH REPORT

TITLE: "AFFLAIR" - PRODUCT QUALITY

WORK DONE BY: D. P. SCHUSSLER

REPORT WRITTEN BY: D. P. SCHUSSLER

REPORT APPROVED BY: C. W. ANDERSON

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TECHNICAL ASSISTANTS: L. I. ASAY (APRIL, 1976 TO MARCH, 1977)
P. CERESINI (MARCH, 1977 TO JULY, 1977)

ABSTRACT

We have determined that the dominant factor governing "Afflair" Luster is the face area per unit weight of the mica flakes. The dominant factor governing opacity is the weight percent TiO_2 of the pigment.

The best examples to use to compare DuPont "Afflair" products with competitives are DuPont's NF-138-D and Mearl's Superpearl. Physical data shows that Mearl's Superpearl is superior in luster and opacity because its surface is free of the mudflat cracks (or crazing) found in DuPont "Afflair" products.

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OBJECTIVE

The purpose of this study is to determine those factors controlling "Afflair" quality, particularly luster and opacity.

SUMMARY AND CONCLUSIONS

- Relationships between physical data routinely gathered on "Afflair" products were examined. Through the use of the ESD program RAMAR, we have found that the opacity of an "Afflair" product depends on its TiO_2 content. The luster depends on its face area/unit weight. With some exceptions, it can be restated more simply that the opacity depends on the diameter of the place and the luster depends on the thickness of the flake. These factors account for the greatest differences between "Afflair" products.

- DuPont "Afflair" products are in general deficient to competitives. Comparison of products show that DuPont products have a higher surface area/unit weight. The higher surface area is due to crazing, or mudflat cracks, in the TiO_2 coating. Methods of hydrolysis that avoid crazing, demonstrated in the laboratory, result in products equivalent to the competitives.

PATENT STATUS

No patents or patent filings have been made.

PROGRAM

No further work is planned.

PUBLICATION STATUS

The material in this work is not suitable for publication.

SPECIAL SAFETY PRECAUTIONS

None.

ENVIRONMENTAL CONSIDERATIONS

None.

TABLE OF CONTENTS

	Page No.
INTRODUCTION	3
THE OPACITY VS. LUSTER GRAPH	3
EMPIRICAL DATA CORRELATIONS	4
DUPONT "AFFLAIR" VERSUS COMPETITIVES	6

INTRODUCTION

The work described in this report is in large part a continuation of studies initiated by R. D. Nelson. For additional background information, the reader is referred to in the Monthly Summaries, 1976, by R. D. Nelson.

This report is concerned exclusively with the properties of pearl (or white) "Afflair" and competitive products. No only do pearl "Afflairs" account for most of the sales volume, but they are also more easily studied than colored "Afflairs". Detailed descriptions of each DuPont and competitive code are contained in the Product Data Sheets issued in 1976 by R. D. Nelson.

The physical properties of "Afflair" of interest in this report are defined:

Luster is the amount of light reflected at the "mirror" angle or the shininess. It is measured as total specular reflectance, TSR.

Opacity is the amount of light reflected at an oblique angle. It is measured as total diffuse reflectance, TDR.

Interference hue is the color observed at the "mirror" angle only. It is measured as the ratio of blue reflectance at the "mirror" angle, SB/R.

Absorption hue is the color observed at an oblique angle. It may differ greatly from the interference hue. It is measured as the ratio of blue reflectance to red reflectance at an oblique angle, DB/R.

Particularity is the relative amount of graininess and is governed by the particle size distribution of the flakes. It is measured as the weight percent retained on a 30 micron sieve in wet screening.

THE OPACITY VS. LUSTER GRAPH

A construction found to be very useful in describing "Afflair" product quality is a plot of opacity, TDR, versus luster, TSR. Figure 1 shows such a plot for some DuPont products, some competitive products and some experimental samples. The points on this graph are concentrated in two areas along diagonals. The first group lies along a line where TSR +10 TDR = 1200. These products are made

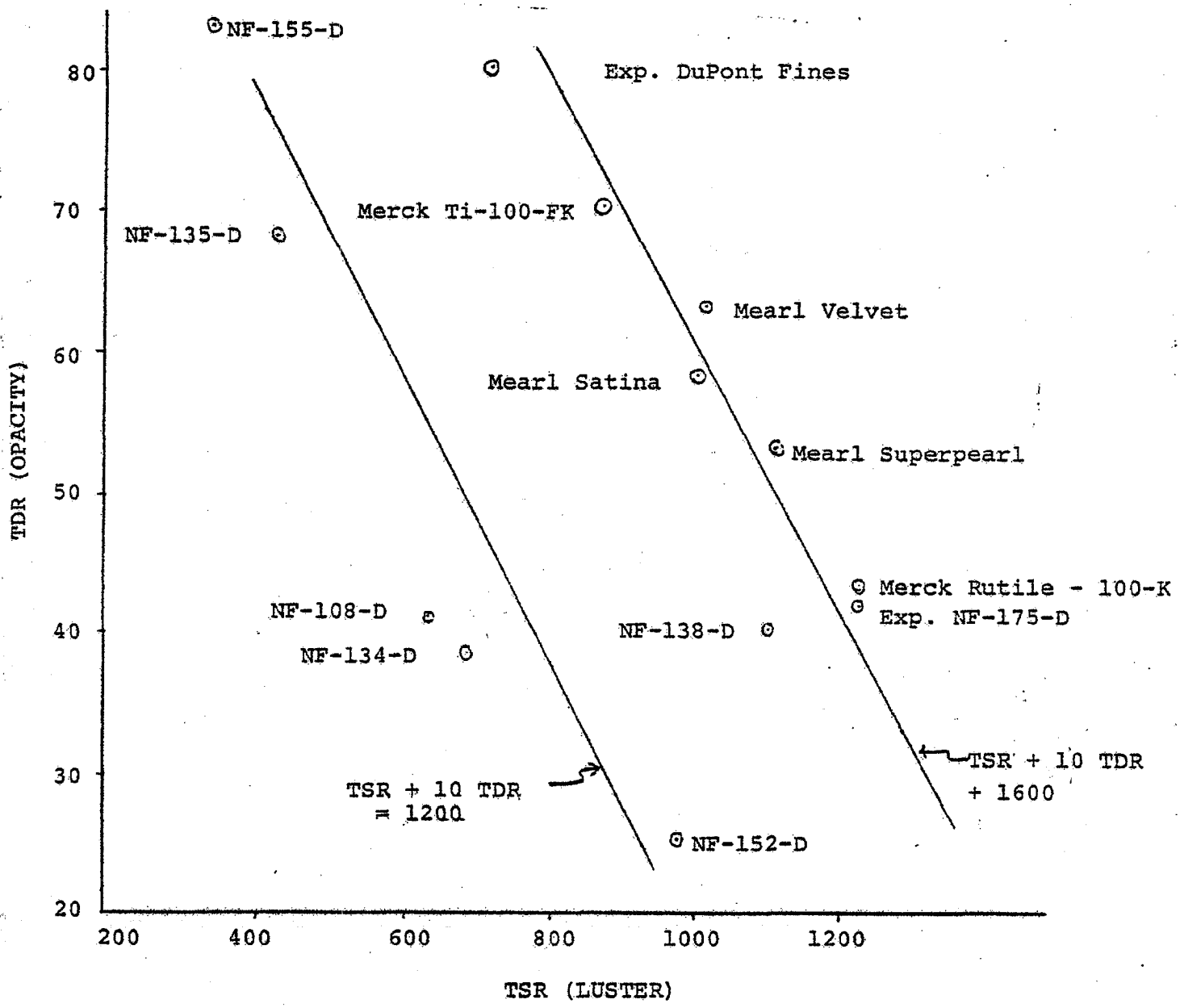

FIGURE 1 - OPACITY VERSUS LUSTER FOR REPRESENTATIVE "AFFLAIR" PRODUCTS

THE OPACITY VS. LUSTER GRAPH (Contd.)

from unclassified mica (excepting NF-152-D, see KN-77-11 and include the precalcined mica products. The second group lies along a line where TSR + 10 TDR = 1600. This group is composed of pigments made from classified mica.

Referring again to Figure 1, the opacity-luster graph, those pigments on the TSR + 10 TDR = 1600 line have a greater ability to scatter light per unit weight of pigment than those on the 1200 line. One might describe the diagonals on Figure 1 as "iso-quality" lines.

There is substantial variation along each of the iso-quality lines. Within a group of "iso-quality" pigments, with higher luster must be less opaque. It is shown in more detail in the next section that the variation along each line depends on the % TiO_2 of the sample. Ideally, those pigments of smaller particle size contain a higher percentage of TiO_2 and have more opacity (less luster) than larger particle size pigments.

EMPIRICAL DATA CORRELATION

We routinely make a number of physical measurements on our "Afflair" products including % TiO_2, face area, surface area, particle size, etc. Relationships between these quantities have been obscured by the large volume of data available and the tediousness of sorting through it all. With the help of V. Bacchetta of ESD and the use of the computer program, RAMAR, relationships between these variables have been revealed. Data from 14 DuPont pearl "Afflairs" and 5 competitives were used. RAMAR produced a table of correlation coefficients relating our quality parameters (TSR, TDR, etc.) to the various physical measurements (% TiO_2, face area, etc.).

A small portion of the table of correlation coefficients is reproduced in Table I. A correlation coefficient is a number between -1 and 1 that shows the statistical relation between two variables. If the correlation coefficient is 1, the two variables are perfectly, directly proportional. If it is 0, they have no statistical correspondence. Negative values show an inverse relation.

Examination of Table I shows that luster (TSR) is very dependent on face area/unit weight and, indirectly, thickness. Face area and thickness are not independent variables; they are two descriptions of the same variable. For convenience, we will use face area in developing this point.

TABLE I

RAMAR CORRELATION MATRIX FROM "AFFLAIR" DATA

VARIABLES	TSR	TDR
% TiO_2	-0.042	-.852
Face Area (m^2/g)	0.830	-0.420
Avg. Diam (μ)	-0.312	-0.603
Thickness (μ)	-0.760	0.077

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EMPIRICAL DATA CORRELATION (Contd.)

Table I also shows that TDR is highly correlated with % TiO_2 and average diameter. Again, these two variables are interdependent. Particles of smaller diameter have a larger surface area/unit weight and thereby require more TiO_2 to attain the same coating thickness. Exceptions to the interdependence of diameter and % TiO_2 are found in precalcined micas, which have TiO_2 contents out of proportion to their diameters. We will use % TiO_2 in the following discussion because its relationship with opacity is linear and direct.

Figure 2 shows a plot of TDR versus % TiO_2 for a large number of data. The equation for the correlation line through the points is:

$$TDR = \%\ TiO_2 \times 1.93\ (\pm 0.24)$$

The figure in parenthesis is the estimated error in the last two digits of the multiplier.

Figure 3 shows a similar plot for TSR versus face area. The data are more limited than for the TDR - % TiO_2 correlation. The equation for the correlation line is:

$$TSR = \text{Face Area}\ (m^2/g) \times 889\ (\pm 156)$$

The estimated error (in parenthesis) is larger, relatively, than the error in the TDR - % TiO_2 relation. The larger error is probably due to the difficulty in making accurate face area measurements.

There are certainly many other important factors in describing the luster and opacity of "Afflair" products in addition to face area/unit weight and % TiO_2. But the largest differences between "Afflair" products can be accounted for on the basis of % TiO_2 and face area alone. Alternately, with certain exceptions, the luster and opacity depend on the thickness and diameter of the flakes.

DUPONT "AFFLAIR" VERSUS COMPETITIVES

It is apparent from Figure 1 that DuPont "Afflairs" at the time of this writing do not have the high values of TSR + 10 TDR observed for many of the competitive products. NF-138-D, made from classified mica, approaches the TSR + 10 TDR = 1600 line more closely than the other codes. Because it is made from classified mica and has a high face area/unit weight, we expect that NF-138-D should fall on the 1600 line. It is apparent then that factors other than those so far discussed limit the quality of DuPont "Afflairs".

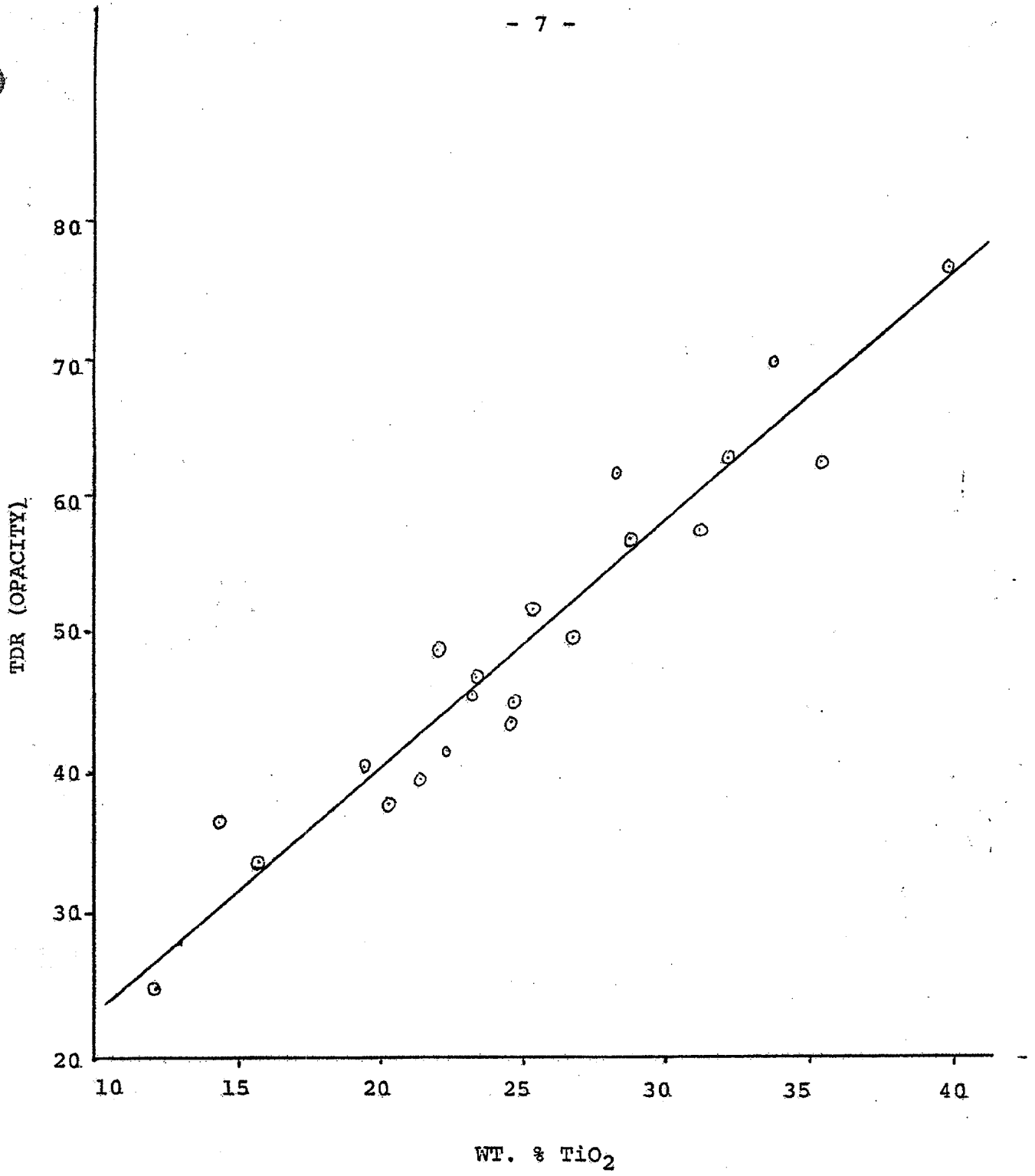

FIGURE 2 - TDR VERSUS % TiO_2 FOR PEARL "AFFLAIR"

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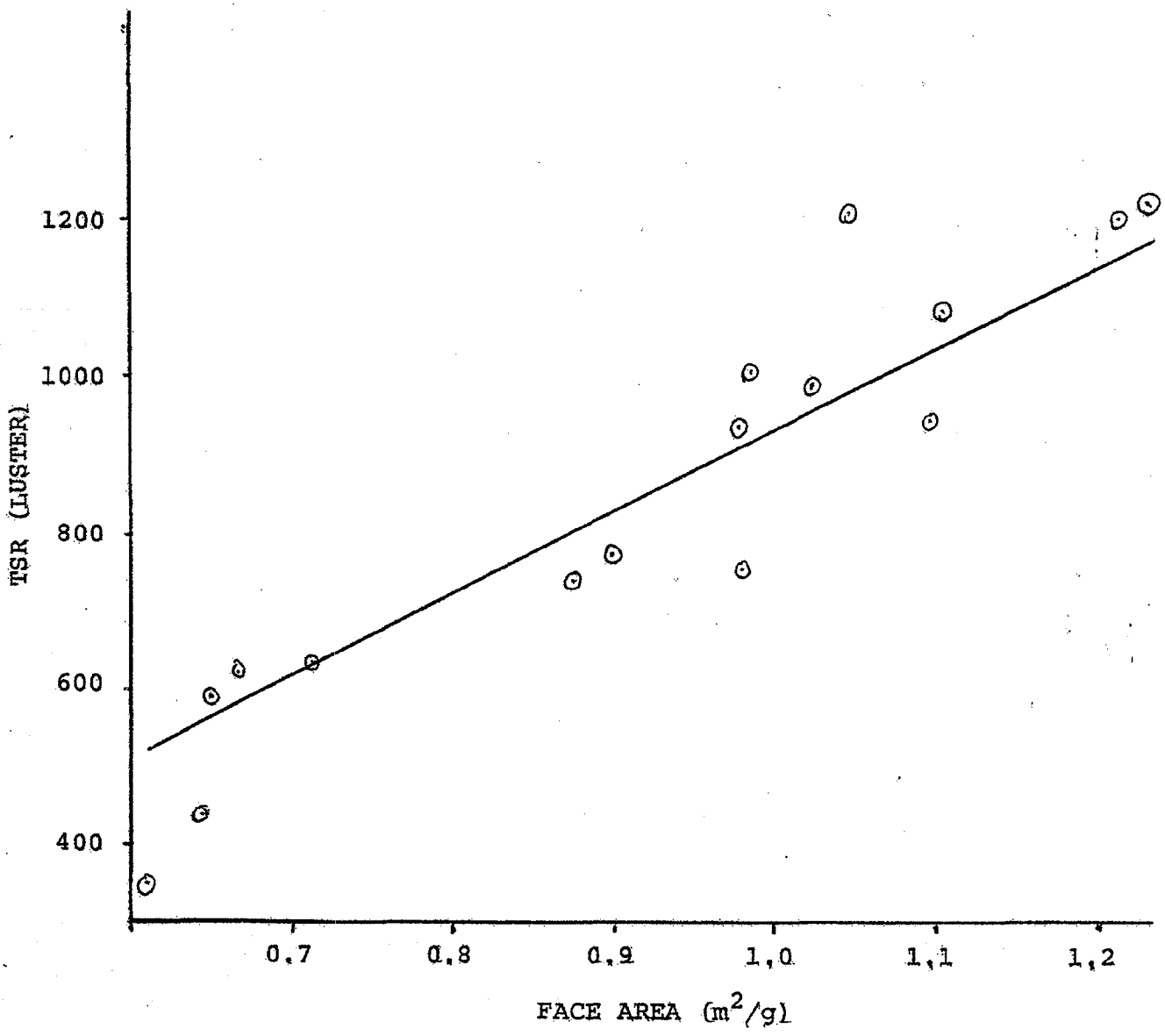

FIGURE 3 - TSR VERSUS FACE AREA FOR PEARL "AFFLAIR"

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DUPONT "AFFLAIR" VERSUS COMPETITIVES (Contd.)

Table II compares several of the physical properties of NF-138-D and Mearl's Superpearl. The fact that Superpearl is more opaque than NF-138-D follows from its higher TiO_2 content. However, since their face areas are similar (given the error in this measurement) and since they are both made from classified mica, we would expect NF-138-D to have a much higher luster. We finally note that the surface area of Superpearl is smaller than that of NF-138-D. The higher TiO_2 content of Superpearl would lead one to predict a higher surface area for Superpearl.

Figure 4 plots the % TiO_2 versus surface area (m^2/g) for some DuPont products and some Mearl products. We see that all of Mearl's products have a lower surface area than is expected from their TiO_2 content. One may think of % TiO_2 as a type of surface area measurement. Because the coating thickness must in all cases be the same in order to obtain a pearl (or white) pigment, a specific amount of TiO_2 is deposited per unit surface area. The Mearl products show a higher surface area than DuPont's by their TiO_2 content, but a lower surface area after the coating is applied. The most reasonable explanation is that the coating applied by Mearl is less porous.

Figure 5 compares SEM photographs of pigment flakes at 20,000X of Superpearl and NF-138-D. As we expect from the surface area measurements, there are very few pores and cracks in the Superpearl coating. The NF-138-D coating is heavily cracked.

KN-77-12 describes two methods for overcoming the deficiencies in NF-138-D. One method, developed by L. I. Asay, is to recoat NF-138-D with a thin TiO_2 layer to fill in the cracks. A second method, developed by E. F. Graewe, is to apply a coating that does not craze. Graewe's method uses rapid caustic addition during the TiO_2 coating application. Both recoated NF-138-D and the product of the base addition method show the craze-free coatings characteristic of the Mearl coatings. Table III compares the luster and opacity of NF-138-D, Superpearl and the two experimentals. Recoating NF-138-D results in a pigment more opaque than NF-138-D but with similar luster. The caustic addition method gives a high luster product with opacity similar to NF-138-D.

TABLE II

PHYSICAL PROPERTIES OF NF-138-D AND MEARL'S SUPERPEARL

	NF-138-D	Superpearl
TSR	1107	1117
TDR	41	53
% TiO_2	21.5	26.7
Face Area (m^2/g)	1.23	0.97
Surface Area (m^2/g)	5.5	4.2

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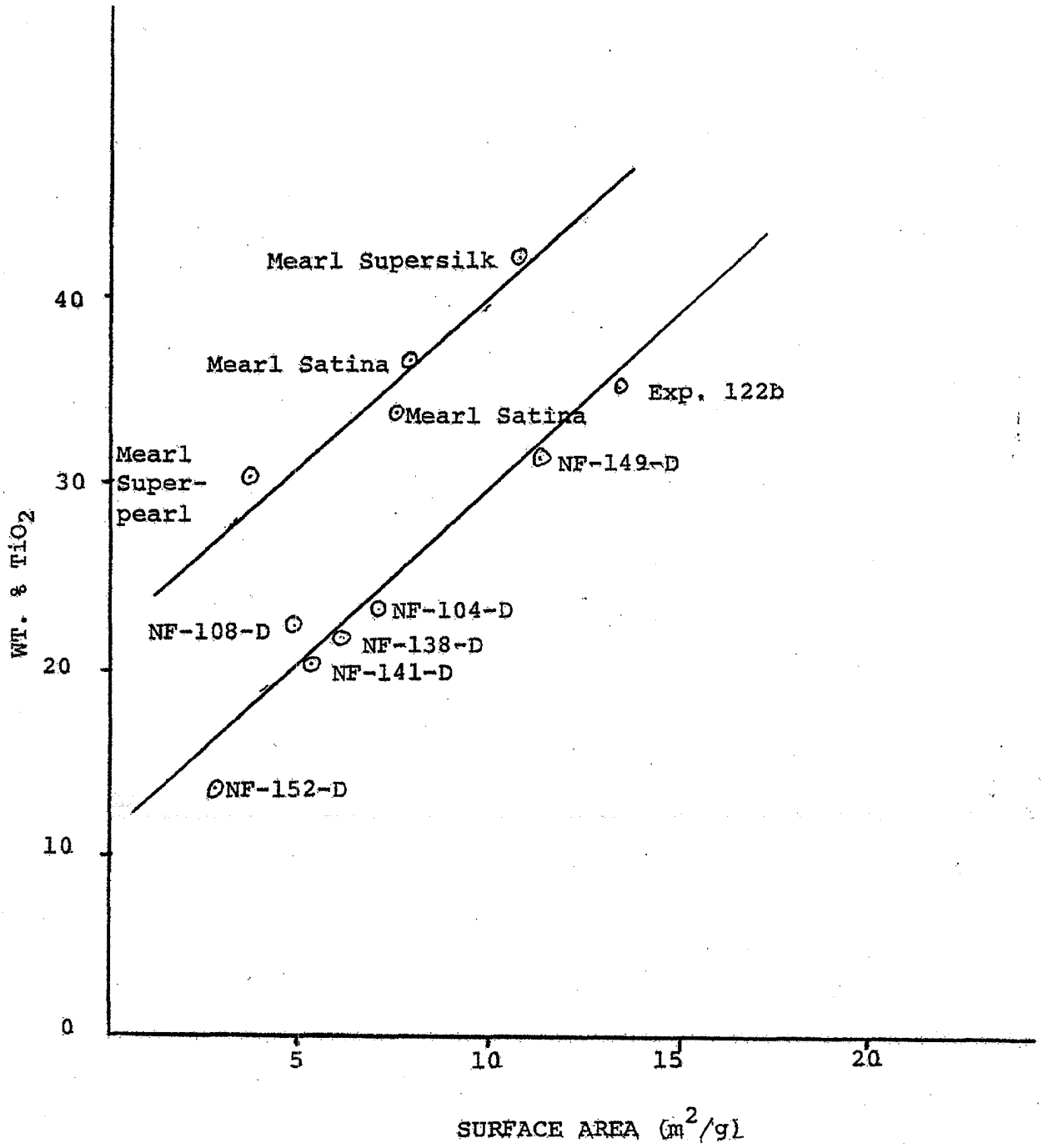

FIGURE 4 - WT. % TiO_2 VERSUS SURFACE AREA FOR DUPONT AND MEARL PRODUCTS

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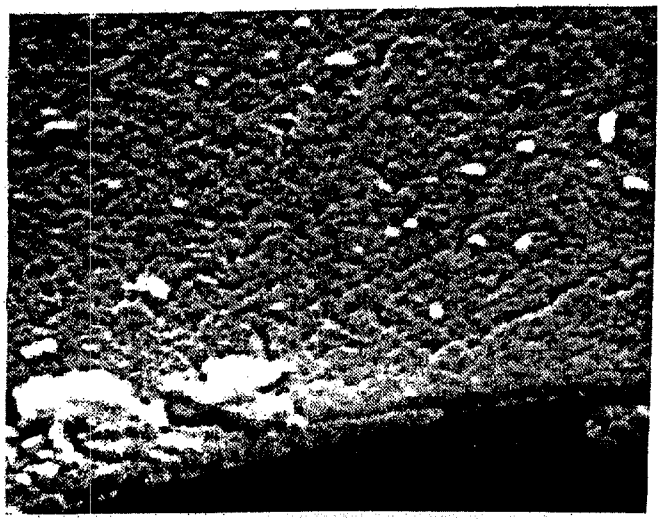
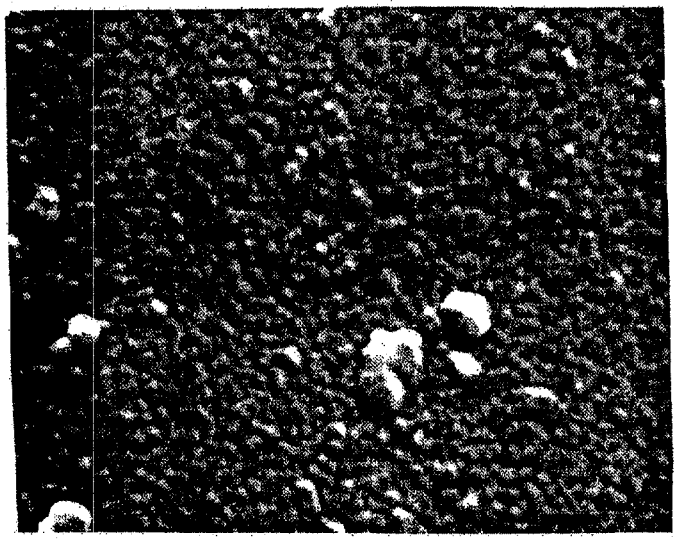


NF-138-D



SUPERPEARL

FIGURE 5 - SEM PHOTOGRAPHS OF SUPERPEARL AND NF-138-D AT 20,000X

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TABLE III

LUSTER AND OPACITY OF SUPERPEARL, NF-138-D AND TWO EXPERIMENTALS

	TSR	TDR
NF-138-D	1107	41
Superpearl	1117	53
Recoated NF-138-D (E-12474-24A)	1139	49
Caustic Addition Method (1915N-118)	1266	43

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