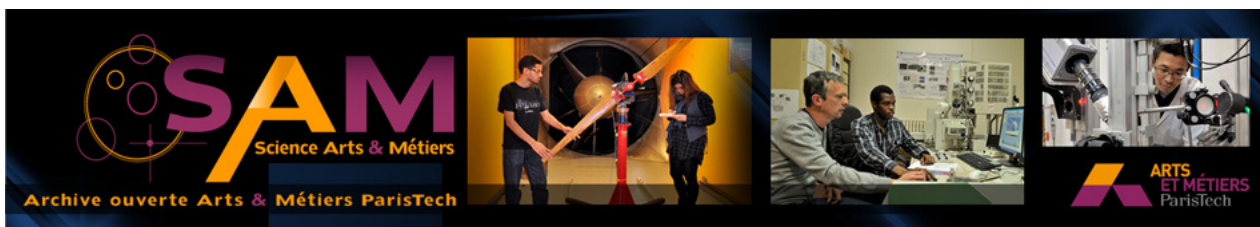**To cite this version :**

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Influence of the morphological texture on the low
wear damage of paint coated sheets

F. Hennebelle [a], D. Najjar [a,*], M. Bigerelle [a,b], A. Iost [a]

[a] *Laboratoire de Métallurgie Physique et Génie des Matériaux, CNRS UMR 8517, Equipe Caractérisation et Propriétés des Périsurfaces,*
ENSAM, 8 Boulevard Louis XIV, 59046 Lille, France
[b] *Laboratoire Roberval, FRE 2833/CNRS, Université Technologique de Compiègne, BP20529, 60205 Compiègne, France*

Abstract

The influence of the morphological texture (flat and structured) of a polyester based paint coating on the low wear damage is characterised by means of roughness and gloss measurements. Using statistical methods, the aim of the investigation is to determine, among about 60 surface roughness parameters, the most relevant of them with regard to the morphological texture and the wear behaviour of polymer coatings. The level of relevance of each roughness is quantitatively assessed through the calculation of a statistical index of performance determined by combining the two-way analysis of variance (ANOVA) and the computer based Bootstrap method (CBBM).

For the experimental conditions related to the present investigation, the fractal dimension and a roughness parameter directly related to the number of inflexion points of the profiles are shown to be the most relevant parameters for discriminating the different morphological textures of studied coatings and for characterising the low wear damage, respectively. Even if the gloss reduction related to the low wear damage is more visually perceptible at a macroscopic scale for the flat products than for the structured ones, the magnitude of this damage is shown to be however very similar at a microscopic scale whatever the morphological texture of the paint coatings.

Keywords: Polymer coatings; Mar damage; Gloss; Statistical indexes of performance; Roughness parameters; Fractal dimension; Number of inflexion points

1. Introduction

Paint coating is an economic way widely used to protect metallic substrates against aggressive and corrosive environments. However, the improvement of the resistance to mar damage is as much important as the resistance to corrosion for paint coatings used in many applications [1–9]. Indeed, mar damage is known to generate small scratches which affect the aesthetic appearance of automotive, building trade and domestic products. As a consequence, a major industrial concern is to develop paint coatings with morphological textures resistant to mar damage over time.

In a very recent paper [10], we assessed the influence of low wear damage on topography features and gloss reduction of several standard commercial paint coatings having different morphologies and surface hardness. Using a statistical methodology combining the analysis of variance (ANOVA) with the computer

based Bootstrap method (CBBM), it was shown that, among a large number of roughness parameters, the fractal dimension and the average curvature radius of peaks were the most relevant roughness parameters for discriminating, respectively, the morphological texture and the magnitude of a low wear damage of the different polymer coatings studied. Besides, from the experimental observations and the statistical analysis results, this low wear damage produced by means of 'the Scotch Brite test' was strongly thought to be the consequence of a ploughing mechanism affecting mainly the topography of asperities at a small scale whatever the initial morphological texture of the polymer coatings studied.

In this paper, the same experimental procedure and statistical methodology were reproduced to assess the influence of morphological texture of improved paint coatings developed to resist wear damage. For these new paint coatings, the aim is to determine which roughness parameters are the most relevant for discriminating the different morphological textures and the magnitude of the low wear damage. Besides, we might also wonder which roughness parameter is the most appropriate to

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provide an objective ranking of damaged products similar to that subjectively perceived at naked eye.

2. Materials and experimental procedure

2.1. Polymer coatings

The materials under study were pre-painted galvanised steel sheets with different morphological textures which provide different aesthetic appearances to the manufactured products. As aforementioned, the experimental paint coatings considered in this investigation were improved with regard to mar damage in comparison to standard commercial products. From a technological point of view, the formulations of these new experimental paintings were based on that of the more resistant commercial product. After their elaboration, these experimental paint coatings were supplied to us by the firm ARCELOR. These new products were polyester based paint coatings consisting of a primer and topcoat in which different additives were introduced to elaborate flat, grained and structured morphological textures having relatively high surface hardness. Microscopic observations of the morphological textures of these new products are shown in Table 1.

2.2. Wear testing

Because the affected area would be too small in comparison with the heterogeneity of the morphological texture of some polymer coatings under consideration in this study, the simple 'Scotch Brite test' has been preferred to usual scratch tests or micro-hardness tests [3,4,7,9,21]. While affecting a larger area during testing, the 'Scotch Brite test' is also more representative of damage encountered in real applications since it simulates a low wear mar damage (occurring for example during automatic car washing [3,4]). More precisely, the mar damage is due to a wear mechanism generating scratches of less than 1 μm depth [3,4,7,9,21] that may affect the surface topography and the aspect.
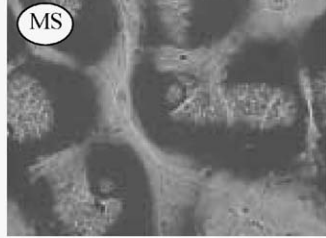
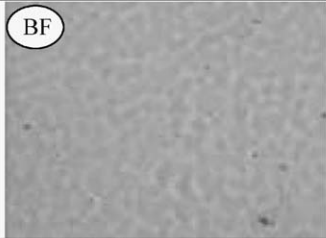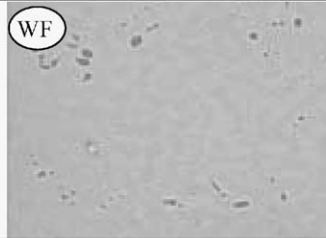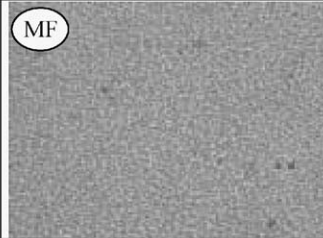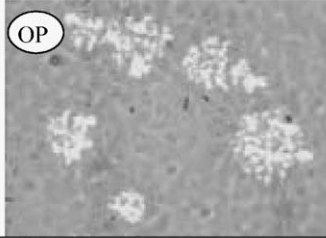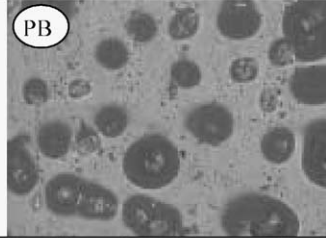Even if the 'Scotch Brite test' is a normalised test (BS EN ISO 11998 Standard), it was shown in our previous investigation [10] that the application of the normalised conditions was not adapted for the standard commercial paints. As a consequence, these normalised conditions were lightly modified and it was found that applying a 15 N load during 10 cycles allowed to obtain visually homogenous and reproducible wear damage. For each sample tested, the worn area was 4 cm large and 12 cm long. The same experimental conditions were retained for the new experimental hard paint coatings tested in this investigation.

It must be mentioned that the 'Scotch Brite test' provides only subjective information that depends in particular on the physiological perception of the practitioner since the magnitude of wear damage is visually assessed (high density of scratches, low density of scratches, no scratches). In this investigation, the mar damage has been quantitatively assessed by means of roughness and gloss measurements.

2.3. Roughness measurements

Preliminary measurements have been performed by means of a three-dimensional (3D) tactile roughness profilometer KLA
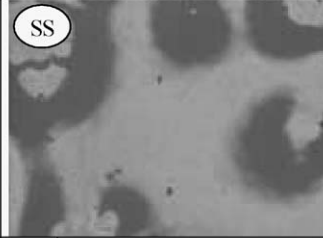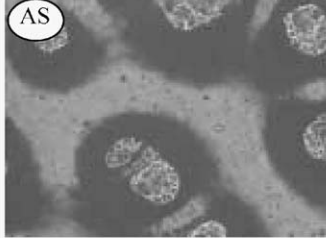
Table 1
Microscopic observations of the morphological textures of the different paint coatings under study

BF	Bright Flat
WF	White Flat
MF	Mat Flat (silica)
OP	Orange Peel
PB	Grained (polyamide balls)
SS	Standard Structured
AS	Alveolar Structured
MS	Mound Structured
▬	= 1mm

TENCOR P10 (diamond tip with a $2\,\mu$m rounded-end, 10 nm vertical resolution) to give an overview of both the as-received and worn surfaces' topographies of the polymer coatings. Whatever the polymer coating, these preliminary measurements revealed that, for scanned areas of $8\,mm \times 8\,mm$ in size, the topography was isotropic in each case. Confirmed by an analysis of the bidimensional autocorrelation functions led us afterwards to performing less time-consuming 2D measurements. Whatever the sample under consideration, 60 profiles of 8 mm length have been recorded using a load of 5 mg, a speed rate of $200\,\mu$m/s and an acquisition rate of $1000\,$Hz. Under these conditions, each recorded profile consists of 40,000 points equally spaced by a $0.2\,\mu$m distance. These profiles were randomly recorded over the surface in the case of the as-received samples and perpendicularly to the wear scratches in the case of the worn samples.

2.4. Gloss measurements

The gloss of polymer coatings has been assessed before and after the wear tests for the different polymer coatings. Measures of gloss have been performed on each sample by means of a three-angle glossmeter Dr. Lange Refo 3D. The main principle of this device is based on measuring the specular amount of reflected light directed to a surface at a specified angle from its normal. The amount of light reflected from the surface under investigation is divided by the amount of light reflected from the surface of a reference smooth black glass plate (delivered by the manufacturer of the glossmeter) and the specular gloss is obtained by multiplying this intensity ratio by 100. In our previous investigation [10], it was shown that a $60°$ angle was a good compromise to integer the effects of both the morphological texture and wear on specular gloss. As a consequence, only the $60°$ angle has been considered in the present investigation for specular gloss measurements.

3. Surface characterisation: relevance of roughness parameters

Fig. 1 presents typical 2D profiles recorded on the surfaces of the different as-received and worn polymer coatings. At first sight, it seems that, whatever the polymer coating under consideration, the 2D profiles of the as-received and worn samples are very similar. As a consequence, it can be expected that the values of usual amplitude roughness parameters like the average arithmetic roughness R_a or the total amplitude roughness R_t should not allow to discriminate the different surface topographies. If such amplitude roughness parameters are commonly selected to assess the surface morphology of manufactured products, it must be pointed out that, because of the various scientific and industrial interests, many other roughness parameters have been introduced. Specifying which of these existing parameters is the most relevant for characterising the topography of any manufactured surface is in general a real challenge. Several years ago, this "parameter rash" [11] was at the origin of the creation and the development in our laboratory of a specific computer software called MESRUG. This software enables, firstly to calculate

more than 60 roughness parameters and, secondly, to estimate statistically their relative relevance with regard to a correlation with any particular functional property [12–15]. The relevance of each roughness parameter P_i is estimated by calculating the value of the Treatment Index F_i^{Effect} by means of a one-way analysis of variance (ANOVA) procedure and by determining a related confidence interval on this variable by means of the computer based Bootstrap method (CBBM). It must be called back that, the higher the value of F_i^{Effect}, the higher the relevance of the roughness parameter, with regard to the treatment effect under consideration. For more details on the ANOVA, the reader should refer to ref. [16].

For the present investigation, we modify our specific computer software introducing the two-way ANOVA in combination with the CBBM with a view to finding the relevant roughness parameters for discriminating the morphological textures and the wear magnitude of the improved new paint coatings under consideration.

3.1. Traditional two-way analysis of variance and limits

Even if the relative relevance of more than 60 roughness parameters have been assessed by means of MESRUG, only the results related to three particular roughness parameters of interest are detailed for a sake of simplicity in a first time. These parameters are the average arithmetic roughness R_a, the fractal dimension Δ_{ANAM} calculated by a method developed by the authors [17] and a parameter denoted G directly related to the number of inflexion points of the studied profiles. The definitions of these roughness parameters are reported in Appendix A and their mean values are reported in Fig. 2 for the different experimental conditions. At first sight, it can be noticed that, as expected, the value of the average arithmetic roughness R_a is not modified after the wear testing for all the polymer coatings. On the contrary, the mean value of the parameter G increases whatever the morphological structure of the coating. As far as the fractal dimension Δ_{ANAM} is concerned, it can be seen that the mean value of this parameter decreases for flat products (except for the Mat Flat product denoted MF) while it increases lightly for structured and grained ones.

These conclusions are confirmed by the calculation of the values of the Treatment Index F^{Effect} resulting from the two-way ANOVA. Since $F_G^{\text{Wear}} \gg F_{\text{ANAM}}^{\text{Wear}} \gg F_{Ra}^{\text{Wear}}$, it can be deduced that, independently of the morphological texture of the polymer coating, the roughness parameter related to the number of inflexion points of the profile, seems to be the most relevant roughness parameter for describing the effect of wear on the topography of the as-received samples. In the same way, independently of any wear effect, the fractal dimension Δ_{ANAM} is the most relevant parameter for describing the morphological texture of the different polymer coatings since $F_{\text{ANAM}}^{\text{Text}} > F_{Ra}^{\text{Text}} > F_G^{\text{Text}}$. However, it must be emphasised that the reliability of these results theoretically depends on whether the inferred assumptions related to the ANOVA are valid or not (assumption of normality for recorded measurement in the different experimental conditions, assumption of homoscedasticity of variance for the different experimental conditions). Unfortunately, the number of statisti-

cal tests, which have to be performed to verify these assumptions is proportional to the number of roughness parameters under consideration. If only one of these tests was rejected, it should be concluded that the related variable F^{Effect} do not follow a Fisher–Snedecor probability density function and therefore the two-way ANOVA should not have been processed. Since the overall number of roughness parameters exceeds 60 in the present investigation, it is not conceivable to perform such a
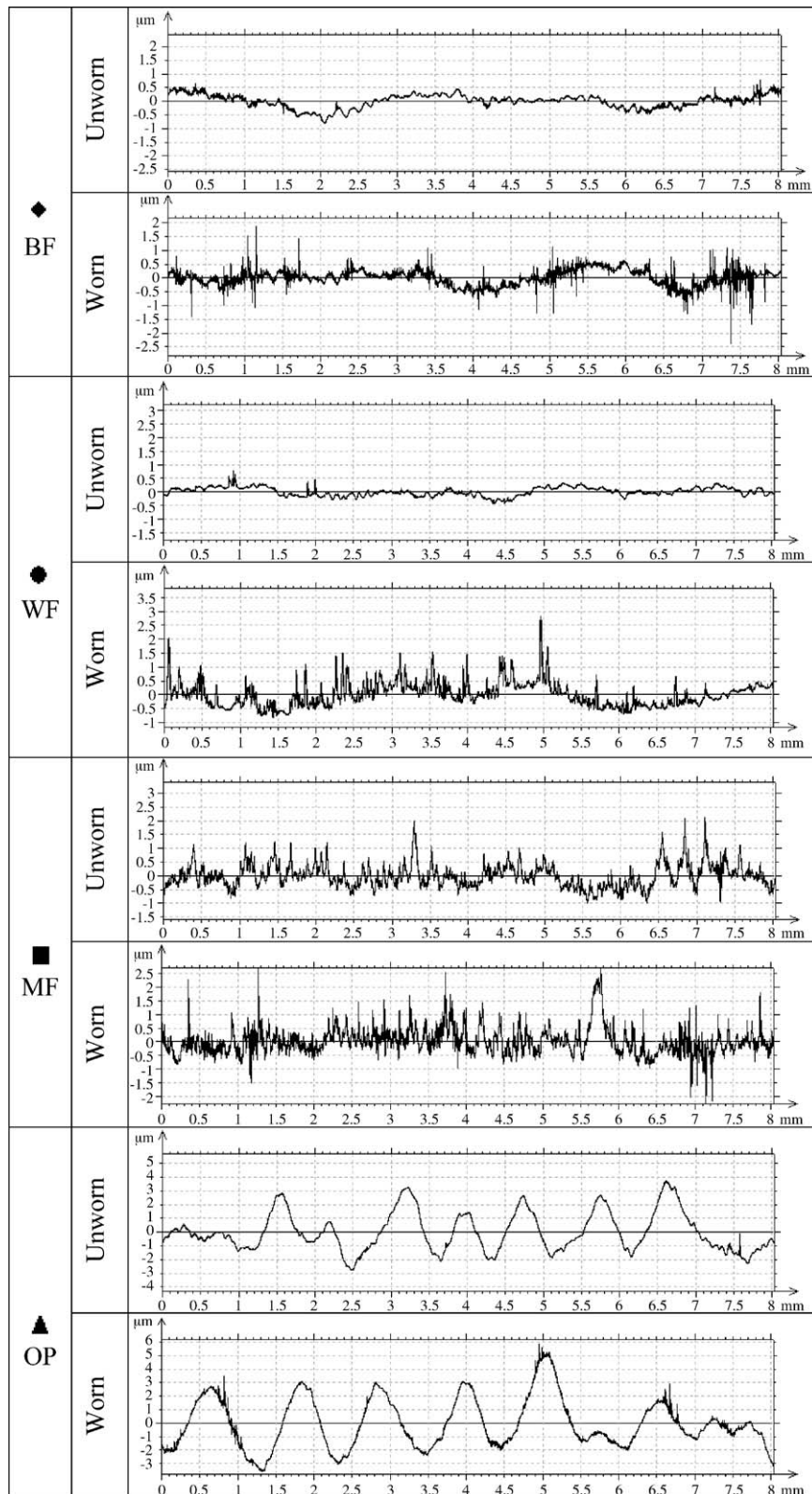
Fig. 1. Typical 2D profiles recorded on the surfaces of the different as-received (unworn) and worn polymer coatings.
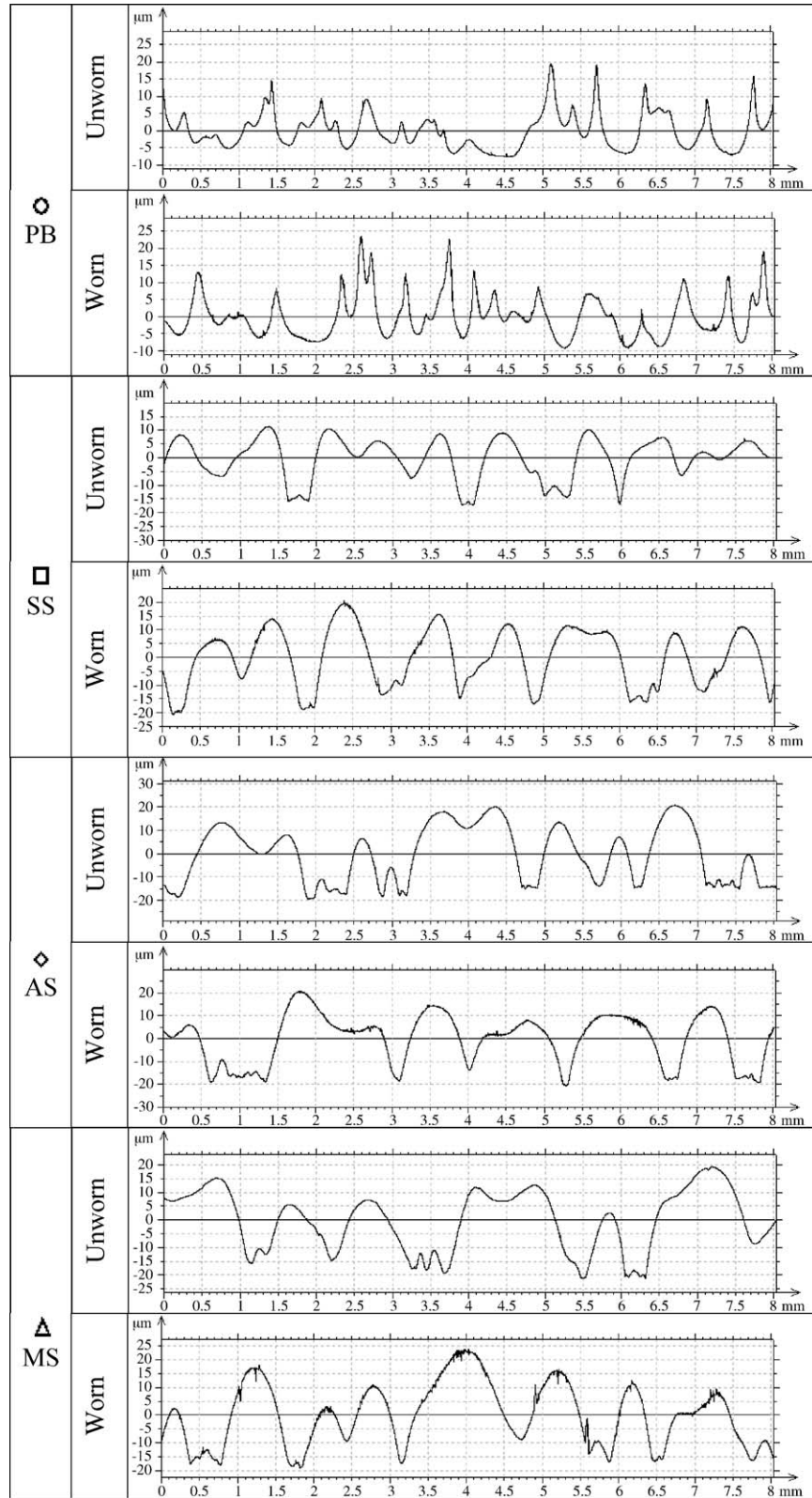
Fig. 1. (*Continued*).

high number of tests meaning that, in practice, the risk of violating the two-way ANOVA assumptions increases as well as the consequent risk of asserting false conclusions. Besides, even if the inferred hypotheses of ANOVA are supposed to be satisfied, this conventional statistical theory does not consider the subtle fact that a small perturbation in any original score may affect the value of the Treatment Index F^{Effect}; value resulting from the calculation of mean quantities. In other terms, the ANOVA

Fig. 2. Mean values of the three roughness parameters (R_a, G, Δ_{ANAM}) recorded for the different polymer coatings before and after the wear test.

Fig. 3. Empirical PDFs of the Bootstrapped F values ($N = 1000$) obtained for the three roughness parameters (Δ_{ANAM}, G, R_a) when analysing the effect of the initial morphological texture of the paint coating and assuming during the two-way ANOVA procedure that these parameters are irrelevant with regard to wear.
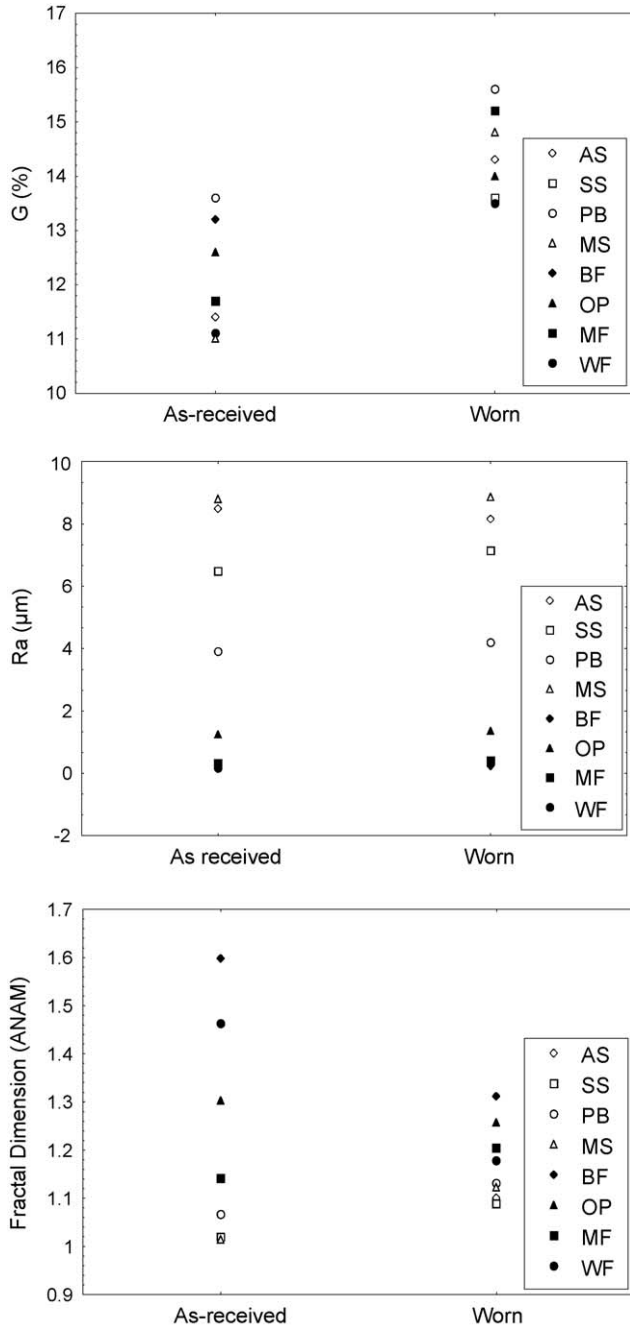
3.2. Application of the CBBM

Roughly speaking, the CBBM is based on the mathematical resampling technique and its main advantage is to allow the replacement of statistical inference assumptions about the underlying population by intensive calculations on a modern computer. Practically, the CBBM consists in generating a high number N of simulated Bootstrap samples by perturbing the scores of a given experimental data set of size J. A Bootstrap sample of size J indexed by $n (n \in [1–N])$, and noted $(x_1^{Boot.n}, x_2^{Boot.n}, \ldots, x_J^{Boot.n})$, is a collection of J values simply obtained by randomly sampling with replacement from the experimental data scores $(x_1^{Exp.}, x_2^{Exp.}, \ldots, x_J^{Exp.})$; each of them having a probability equal to $1/J$ to be selected. A Bootstrap sample contains therefore scores of the experimental data set; some appearing zero times, some appearing one, some appearing twice, etc. For more details on the CBBM, the reader should refer to [14,18–20].

In the present investigation, the CBBM has been combined with the two-way ANOVA for all the roughness parameters P_i under consideration. For each roughness parameter P_i and each treatment effect, the two-way ANOVA has been applied onto each simulated Bootstrap sample $n (n \in [1–N])$ to obtain an empirical probability density function (PDF) consisting of N ($N = 1000$, in this study) simulated values $\{(F^{Effect})_i^n; n \in [1, N])\}$. From this empirical PDF, it is finally possible to extract statistics like the mean $\overline{F_i}$ as well as the 5th and 95th percentiles F_i^{5th} and $F_i^{95\%}$ in order to assess both the central tendency and the deviation through the calculation of the 90% confidence level.

Fig. 3 shows examples of PDFs obtained for the three roughness parameters R_a, Δ_{ANAM} and G from which the statistics $\overline{F_{R_a}}$, $F_{R_a}^{5th}$, $F_{R_a}^{95th}$, $\overline{F_{\Delta_{ANAM}}}$, $F_{\Delta_{ANAM}}^{5th}$, $F_{\Delta_{ANAM}}^{95th}$, $\overline{F_G}$, F_G^{5th} and F_G^{95th} have been extracted. Fig. 4a and b present a summary of the values of $\overline{F_i}$, F_i^{5th} and F_i^{95th} extracted from the Bootstrapped PDFs related to each roughness parameter P_i when considering, respectively, either the effect of morphological texture independently of the fact that the coatings are worn or not, or

does not provide any confidence interval on the advanced value of Treatment Index F^{Effect}. Under these conditions, we might wonder if the values of F_{ANAM}^{Effect}, $F_{R_a}^{Effect}$ and F_G^{Effect}, we previously calculated and compared, for the two treatment effects (morphological texture and wear), were significantly different or not; in other words, if our conclusions were reliable or not in a statistical sense. These two limits of the usual two-way ANOVA were at the origin of our proposal to use an alternative statistical methodology taking into consideration the computer based Bootstrap method (CBBM).
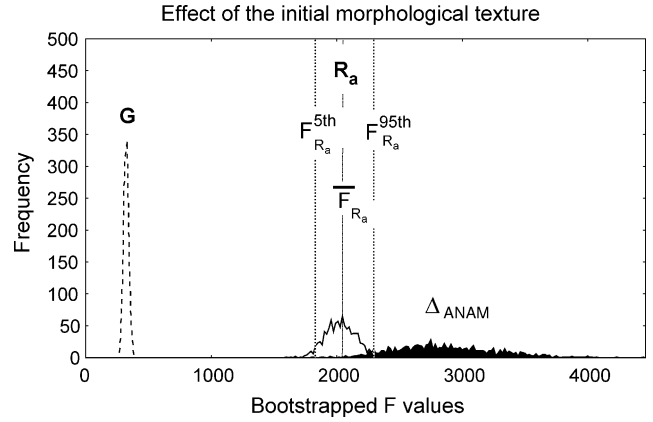
Fig. 5. Means of specular gloss recorded at a 60° angle for the different polymer coatings.
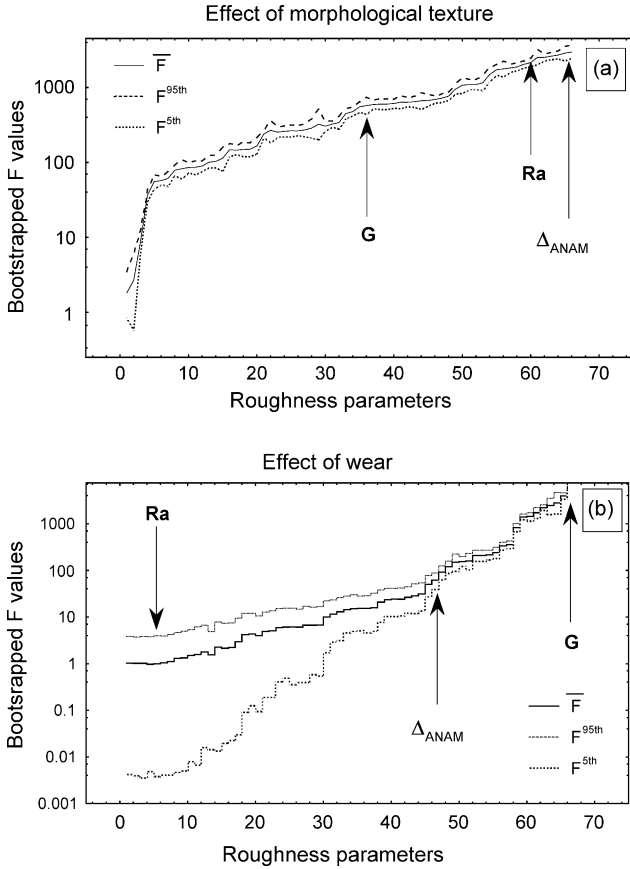

Fig. 4. Statistics (mean and 90% confidence interval) related to the Bootstrapped F values ($N = 1000$) obtained for all the roughness parameters when analysing the effect of: (a) the initial morphological texture of the polymer coating and (b) the wear.

the effect of wear independently of the morphological texture. It can be noticed in these figures that, whatever the effect analysed, there is an overlap of the 90% confidence intervals for many roughness parameters meaning that they have the same relevance level with regard to the effect considered. Besides, it can be seen that the most commonly used roughness parameter R_a does not allow to discriminate the wear effect while it is appropriated to discriminate the morphological texture of the studied polymer coatings. Finally, the overall results show that among more than 60 studied roughness parameters, the fractal dimension Δ_{ANAM} is the most relevant one for discriminating the different morphological textures of polymer coatings under study whereas the parameter G is the most relevant one for characterising the magnitude of the low wear damage.

4. Gloss of polymer coatings

Under the experimental conditions retained in this investigation, no debris were noticed at the surface of all the studied polymer coatings after their wear testing. In fact, only small depth scratches can be observed at the surface of flat products when observing them visually at a grazing angle. As far as structured or grained products are concerned, it is worth noting that none of them presents visible scratches at their surface. These subjective observations are confirmed by the mean val-
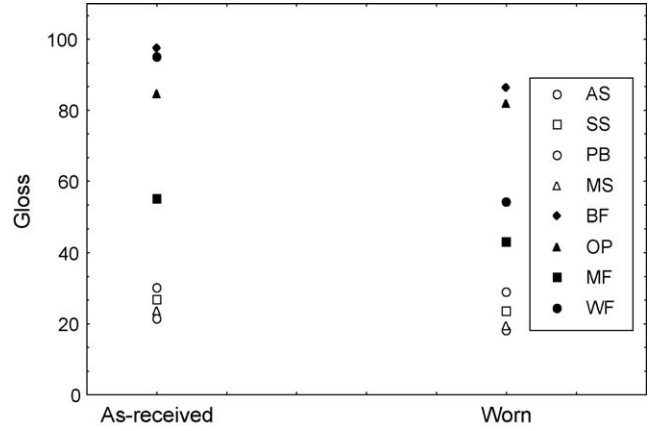
ues of gloss measurements reported in Fig. 5 for the different experimental conditions. Indeed, the reduction of gloss is only statistically significant for the flat morphological textures.

5. Discussion

Compared with the results obtained in our previous study on standard commercial products [10], the experimental paint coatings tested in this investigation show an improved resistance to mar damage. Only the experimental flat products are lightly sensitive to a mar damage observable at naked eye under the conditions retained for the 'Scotch Brite tests'. This mar damage operates at a small scale and it is at the origin of the significant gloss reduction observed for these products. Besides, the experimental structured and grained products are really interesting from a practical point of view since they do not present any visible damage at naked eye and any significant gloss reduction.

Under the experimental conditions of this study, the low wear damage is strongly thought to be due to the ploughing action of the abrasive particles contained in the 'Scotch Brite' sponge. Because of the large difference between the hardness of the abrasive particles and those of the different paint coatings studied, this ploughing mechanism is thought to affect mainly the roughness at a small scale with a similar degree of damage whether the initial morphological texture of the polymer coating is flat, grained or structured. Abrasive particles locally penetrate by plastic deformation in low hardness surfaces of these polymer coatings and new local peaks are likely to be generated (Fig. 6). This mechanism is particularly in agreement with the fact that, regardless of the initial morphological texture: (a) SEM obser-

Fig. 6. Schematic representation of the effect of hard abrasive particles acting at the top of a soft asperity during a wear test. A black arrow highlights the location where an abrasive particle penetrated at the top of the asperity. At a microscopic scale, this ploughing effect results in an increase of the number of inflexion points of the profile and therefore of the chaotic aspect of the profiles.

vations show similar scratches at a small scale, (b) the parameter G which characterises the tortuosity of the profiles is the most relevant roughness parameter for characterising the wear effect, (c) the variation of the parameter G due to the wear test is constant with regard to the measurements uncertainty and (d) the fractal dimension is modified.

As far as the most relevant roughness parameter for characterising the wear effect is concerned, it is worth noting that the difference noticed in this investigation in comparison with our previous study can be related to the higher hardness of the new experimental coatings tested in this investigation. Even if the degree of wear damage is similar for the different products, it has not the same impact on the value of the fractal dimension Δ_{ANAM} of the different polymer coatings. This value increases for grained and structured products while it decreases for flat ones. This is due to the different amplitudes related to these different morphological textures with regard to the scale on which the value of the fractal dimension is assessed. Moreover, it is interesting to note that this roughness parameter takes into account both the effect of the morphological texture and the magnitude of wear damage since it provides the same ranking as that perceived at naked eye.

6. Conclusion

This paper presents an original and efficient statistical methodology for assessing, without any preconceived opinion, the relative relevance of a high number of roughness parameters with regard to the morphological texture and the wear damage of experimental paint coatings.

This statistical methodology reveals that the commonly used arithmetic roughness parameter does not allow to discriminate the wear effect while it is sufficient to discriminate the morphological texture of the studied polymer coatings (or more precisely to rank them as a function of their macro-roughness). In fact, the most relevant roughness parameter for characterising the low wear damage (mar damage) affecting these improved experimental polymer coatings is the roughness parameter G related to the number of inflexion points of the profiles. This roughness parameter, which is a measure of the chaotic aspect of the profiles, is higher after than before the wear test whether the morphological texture of the paint coating is flat, structured or grained. It is worth noting that the fractal dimension is an interesting roughness parameter for taking into account both the effect of the morphological texture and the magnitude of wear damage since it provides the same ranking as that perceived at naked eye.

Finally, as in our previous study, the low wear damage is thought to be due to the ploughing action of the abrasive particle contained in the 'Scotch Brite' sponge. This ploughing mechanism affects mainly the roughness at the small scale with the same magnitude whether the initial morphological texture of the polymer coating is flat, structured or grained. However, a same degree of damage has not the same influence on the visual aspect with regard to this morphological texture. Flat products show a marked decrease of gloss whereas gloss of grained and structured product is not significantly affected. If the macro-roughness of the structured and grained products does not seem to have a real influence on the resistance to mar damage, it enables however to induce a window dressing effect that simply hides to the naked eye the small scratches generated at the small scale.

Appendix A

A.1. The arithmetic roughness parameter R_a

This most commonly used roughness parameter represents the absolute deviation from the profile heights and the reference mean line. This amplitude roughness parameter is defined such that:

$$R_a = \frac{1}{l} \int_0^l |z(x)| \, dx$$

where l is the evaluation length and $z(x)$ is the profile height relative to the reference mean line at position x.

A.2. The roughness parameter G

This roughness parameter is related to the chaotic aspect of a profile since it corresponds to the ratio between the number of inflexion points detected over the total length of the profile divided by the total number of points multiplied by 100.

To estimate this roughness parameter from a numerical point of view, the best fitting third-order polynomial curve ($z = a_1 x^3 + a_2 x^3 + a_3 x + a_4$) has been determined considering four consecutive points $[(x_i, z_i), (x_{i+1}, z_{i+1}), (x_{i+2}, z_{i+2}), (x_{i+3}, z_{i+3})]$ as the quantities $6a_1 x_{i+1} + 2a_2$ and $6a_1 x_{i+2} + 2a_2$. These two quantities correspond to the numerical value of the second derivative of the polynomial curve ($(d^2 z/d^2 x) = 6a_1 x + 2a_2$) at positions x_{i+1} and x_{i+2}, respectively. An inflexion point is supposed to exist between x_{i+1} and x_{i+2} if these two quantities have not the same sign.

The roughness parameter G is calculated by using the following formula:

$$G = \frac{n}{N - 3} \times 100$$

where N is the total number of points of the profile and n is the number of inflexion points detected over the length of the same profile when i is 1 to $(N - 3)$.

A.3. The fractal dimension Δ_{ANAM} estimated by the ANAM method [17]

Let us consider $a < b$ where a and b are two real numbers, f a C^0 function such that $f: [a - \tau, b + \tau] \rightarrow IR, x \rightarrow f(x)$. Let us

generalise the structure function with α a real number higher than unity and define $M_\tau^\alpha(f, x)$ as follows:

$$M_\tau^\alpha(f, x) = \left[\frac{1}{\tau^2} \int_{t_1=0}^{\tau} \int_{t_2=0}^{\tau} |f(x+t_1) - f(x-t_2)|^\alpha \, dt_1 \, dt_2\right]^{1/\alpha}$$

By averaging $M_\tau^\alpha(f, x)$ on the overall profile length L, the $K_\tau^\alpha(f, L)$ function is formally obtained:

$$K_\tau^\alpha(f, L) = \frac{1}{L} \int_{x=0}^{x=L}$$
$$\times \left[\frac{1}{\tau^2} \int_{t_1=0}^{\tau} \int_{t_2=0}^{\tau} |f(x+t_1)\right.$$
$$\left. - f(x-t_2)|^\alpha \, dt_1 \, dt_2\right]^{1/\alpha} dx$$

The numerical form of this equation is given below:

$$\hat{K}_{k\,\delta x}^\alpha(f, n) = \frac{(k+1)^{-(2/\alpha)}}{n-2k} \sum_{i=k+1}^{n-k} \left[\sum_{j=0}^{k}\sum_{l=0}^{k} |f_{i+j} - f_{i-l}|^\alpha\right]^{1/\alpha}$$

where $n = L\,\delta x$ is the number of discretised consecutive points equally spaced of a distance $\delta x = x_{i+1} - x_i$ on the overall profile length L.

If the considered function f is assumed to be Hölderian and anti-Hölderian, it can be shown that:

$$\Delta(f, L) = \lim_{\tau \to 0}\left(2 - \frac{\log K_\tau^\alpha(f, L)}{\log \tau}\right)$$

Finally, the fractal dimension is assessed using the linear least square method to calculate the slope of the plot $\log \hat{K}_{k\,\delta x}^\alpha(f, n)$ versus $\log(k\,\delta x)$ for various $k\,\delta x$ values; this slope corresponds to the Hölder exponant $H(f, L)$ and $\Delta(f, L) = 2 - H(f, L)$. The higher the fractal dimension, the higher the chaotic aspect of the profile.

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