

USING THE ROUGHNESSMETER FOR THE QUANTITATIVE STUDY OF CERAMIC GLAZE DEGRADATION BY ABRASION

A. Escardino, M.J. Ibáñez, A. Blasco, J.L. Amorós.

Instituto de Tecnología Cerámica. Universitat Jaume I. Castellón.
Asociación de Investigación de las Industrias Cerámicas (AICE). Castellón.

ABSTRACT

A roughnessmeter was used to quantitatively determine solid volume loss and variation in surface void volume (chips or pores) and roughness as a result of subjecting ceramic glazes to wear by abrasion.

A magnitude related to material volume loss for different degrees of abrasion, was determined from the Abbott curve, corresponding to a diametral profile of the circular wear surface. This information was used to calculate a coefficient representative of wear by abrasion, for the glazes.

An attempt was made to relate roughness values and those of the magnitude representative of void volume per unit surface area, to the change in surface quality of the glaze as a result of wear.

1.- INTRODUCTION.

1.1.- Abrasion of ceramic tile by the PEI classification method.

Resistance to wear by abrasion is one of the most important mechanical properties of glazed ceramic floor tile. Wear by abrasion is the material loss a solid surface undergoes as a result of a mechanical action (friction, impact, etc.). In the case of glazed ceramic tiles this wear takes place on the glazed surface.

Usually the PEI classification, described in European standard EN 154 "Ceramic tiles. Determination of resistance to surface abrasion. Glazed tiles.", is used to establish the degree of resistance to wear by abrasion in ceramic floor tile glazes [1]. The method involves visually appraising the change in surface quality of a glaze after being subjected to wear in a standard abrasion tester.

A drawback of this method is the visual assessment of the impaired surface appearance, which involves a certain subjectiveness in the evaluation. Moreover, there are other reasons for questioning its effectivity, some of which are set out below:

a) Influence of surface colour on loss of surface quality.

On abrading a glaze surface, its appearance tends to evolve towards lighter colours, as its texture changes.

Material loss, when dirt is absent, is therefore more easily appreciated in dark glazes than light-coloured ones, so that, with equal abrasion resistance, the former tend to yield a lower PEI classification than the latter.

However on true use of the floor tiles, dirt retention in the wear surface areas stands out much more in light-coloured glazes than in dark ones, where this partially hides the change in appearance as a result of wear. Application of the PEI method as set out in the standard, is therefore not comparable to the true action which causes loss of surface quality in installed floor tiles.

b) Influence of apparent porosity and wear-induced irregularities in the surface.

Most ceramic glazes show a certain internal porosity (sealed) which depends on the composition and manufacturing process. This porosity progressively becomes apparent with surface wear of the piece. Dirt enters these newly opened pores which is then retained irreversibly as it cannot be removed by washing. Another considerable amount of dirt is "reversibly" held in the surface roughness and chipping which arise from wear, and which also contributes to changing the surface appearance of floor tile on use. This can be removed by washing.

The standard procedure used in establishing the PEI classification, does not allow detection of these changes which are closely related to progressive loss of surface quality of the glaze.

c) Lack of wear uniformity.

The test carried out according to the PEI method does not uniformly wear away all the treated surface area, but affects its peripheral area most intensely. This ring with its greater degree of abrasion, widens as the test duration increases. This lack of wear uniformity, can be misleading when attempting to classify a glaze by this method.

P.A. Walters and R. Harrison [2], proposed replacing the abrasive charge used in this PEI method by one in which size distribution of the steel balls was modified and glass balls were introduced, in order to obtain more uniform wear.

1.2.- Modified PEI method.

With a view to studying the effect irreversible dirt retention has on loss of surface quality of the abraded glaze, the standard PEI method was modified by dirtying the wear surface area and then cleaning it with detergent, to remove the material not held in open pores [3].

The findings showed the modified PEI method to be more representative than the standard method, as it enables the influence of the irreversibly retained dirt to be appreciated on loss of surface quality as a result of wear, of the glaze surface. However, it is more effective for light-coloured glazes than for dark ones, in which retained dirt hides wear, yielding PEI classification values above the true values.

1.3.-Using a roughnessmeter for the quantitative study of abrasion resistance and the causes of loss of surface quality as a result of wear.

In order to attempt to quantitatively assess wear by abrasion in ceramic glazes, the possibility of using a roughnessmeter was considered. This apparatus allows a series of parameters to be determined by measuring surface roughness profiles, which must be related to loss of surface quality as a result of wear, in these glazes.

This technique is at present being utilized in studying wear in technical ceramics and metal surfaces.

1.3.1.- Abrasion resistance measurement.

The surface wear rate of a solid is defined as the amount of material lost (expressed in volume or mass units), per unit of the magnitude used to measure the duration of the abrasion process. This magnitude depends on the nature of the method used to bring about wear [4], [5], [6], [7]. If wear is produced in the abrasion tester used in applying the standard PEI method, the reference magnitude will be the number of revolutions to which the glaze surface has been subjected in the apparatus. Therefore, in this instance, the wear rate [8] can be defined in the form:

$$R = \frac{\Delta V_h}{N} \quad (1)$$

where:

R = wear rate (m³/no. of rev.)

ΔV_h = volume of material lost by abrasion (m³)

N = number of revolutions the glaze is subjected to in the abrasion tester (no. of rev.)

For metals, wear resistance (E) is defined as the inverse of the wear rate [8]:

$$E = 1/R \quad (2)$$

By applying this definition to ceramic glazes, from Eqs.(1) and (2), finding ΔV_h yields:

$$\Delta V_h = N/E \quad (3)$$

1.3.2.- Dirt retention capability measurement of a glazed surface.

i) Measurement of v_g .

A series of parameters may be obtained from which the so-called "oil retention volume" (v_g) [9] stems, from one of the different surface roughness profiles measured by means of a roughnessmeter, which represents "the void volume per unit area, capable of retaining oil on a scratched metal surface". This parameter, which was defined for engine cylinder liners, could be directly related to the dirt retention capability of ceramic glaze surfaces.

ii) Roughness measurement.

Surface roughness is defined in the literature as "the set of surface irregularities, conventionally defined, in a section where errors of shape and waves have been removed". [10]

Several parameters are used in measuring roughness with a roughnessmeter. The most commonly used one is the so-called "arithmetic mean roughness value" (Ra), computed as the arithmetic mean

of the absolute values of the point heights making up the profile, with regard to its reference line.

The value of this roughness parameter has been shown to increase with wear intensity on abrading the surface, in the case of technical ceramics [2], [6]. This increase in roughness, besides influencing some surface properties which are closely related to its appearance (variation in gloss and colour intensity), can contribute to retention of removable or "reversible" dirt, i.e. dirt removable by washing.

2.- AIM OF THIS STUDY.

As pointed out in the Introduction, the PEI classification method used to assess wear by abrasion in ceramic glazes, is qualitative and imparts a certain subjectivity to the findings. Moreover, some of the factors contributing to the loss of surface quality in ceramic tiles on true use, are not contemplated.

Using a roughnessmeter was therefore considered, in an attempt to measure quantitatively and instrumentally, the solid volume loss, roughness and the appearance of open pores, produced as a result of wear in ceramic glazes by abrasion. Furthermore, it was thought it might be interesting to try and relate these variables with the change in surface quality of the glaze as a result of this abrasion, as well as trying to calculate its own wear resistance from the solid volume loss.

With this aim, execution of the following stages was programmed:

- 1) Setting up a procedure involving a roughnessmeter, for measuring the amount of material lost from the glaze surface as a result of wear by abrasion according to the PEI test method.
- 2) Computing wear resistance in some ceramic glazes from the data obtained by the above procedure, applied to different degrees of wear.
- 3) Measuring the void volume capable of retaining dirt, per unit area, on the rough wear surfaces of glazes abraded in an abrasion tester (according to the PEI method).
- 4) Studying the variation in void volume per unit surface area potentially capable of retaining dirt and the variation in surface roughness with the degree of wear of the glaze.
- 5) Studying the possible relation between the values obtained for these last two parameters and the dirt retention capability of the glaze surface.

3.- EXPERIMENTAL TECHNIQUE.

3.1.- Preparation of the surfaces to be studied.

3.1.1.- Abrasion tests.

The abrasion tests were conducted according to the PEI method described in standard EN 154 "Ceramic tiles. Determination of resistance to surface abrasion. Glazed tiles.", which involves subjecting the glazed tile surface to the action of an abrasive charge consisting of steel balls, corundum and distilled water, in a standard abrasion tester.

3.1.2.- Surface dirtiness.

In order to explore possible dirt retention in the wear surfaces, these were dirtied after each test in the abrasion tester by pouring several drops of a mix of mineral oil and active powdered carbon on them, in a proportion of 85/15 in wt.

This mix was forced into the surface pores and cracks by means of a brush making a constant rotary movement and bringing to bear a uniform pressure. The remaining mix was then wiped off with a dry cloth.

3.2.- Surface roughness profile measurement.

A HOMMELWERKE, model T4000 roughnessmeter, with an traverse unit reaching up to 150 mm in its travel, supplied with software for conducting topographies, was used to determine the parameters derived from the surface roughness profiles of the materials studied.

Measurement was conducted by means of a laser pick-up with a 1 μm diameter, and a vertical measurement capability of $\pm 500 \mu\text{m}$.

3.2.1.- Abrasion resistance measurement.

The surface area abraded by the abrasion tester involved a circle wear face with an 82-mm diameter.

The profiles measured with the roughnessmeter to compute abrasion resistance lay over a travel of 100 mm, and at both ends of the pathlength, part of the original unworn surface was included, as well as diametrically scanning the circle wear area. On abrading the piece, the central part of the profiles lost height with regard to the starting surface, represented by the two profile outer ends which, as they remained unvarying, served as references to compute the magnitude related to the material volume loss in the wear area.

3.2.2.- Determination of v_s .

Ten roughness profile scans were run to determine this parameter, at a distance of 0.5 mm from each other, with a pathlength of 80 mm. In this way, an area of 80x5 mm was covered, corresponding to the central region of the wear circle. The parameter v_s was computed from each profile and the arithmetic mean of the ten values obtained, was calculated [9].

This determination was first carried out on the unabraded surface. It was then repeated with the same glaze after subjecting it to wear produced by applying 200 revolutions in the abrasion tester. This step was repeated several times. After each abrasion treatment, the wear surface was washed, first with a diluted hydrochloric acid solution to remove oxide residues from the steel balls in the abrasive charge used, and then with soap and distilled water, applying ultrasounds. Finally it was left to drain and was dried before measuring with the roughnessmeter.

Subsequently, the wear surface was dirtied with a mineral oil and active carbon mix, and it was repeatedly wiped with a dry cloth, examining the circle wear area to see if there was any darkening as a result of dirt retention.

3.2.3.- Surface roughness measurement.

The values of surface roughness parameter R_a were computed as the arithmetic mean of the values determined by the roughnessmeter, for each of the ten measured profiles, at each N value tested.

3.3.- Determination of scratch hardness.

The method described in European standard EN 101 "Ceramic tiles. Determination of scratch hardness according to Mohs.", was used in determining scratch hardness of the glaze surfaces, which involves attempting to scratch the surface with fragments of minerals belonging to Mohs scale.

Surface hardness is that of the mineral with the lowest hardness which produces no more than one scratch.

3.4.- Determination of Vickers microhardness.

Vickers microhardness was determined with a LECO microhardness tester, model M400, with a standard Vickers indenter.

Several indentations were carried out on a test piece of each of the materials tested, loading at 200 g for 15 s. The number of indentations performed depended on the dispersion of the microhardness values found for each material.

The arithmetic mean of the values obtained for each of the samples tested, was then computed.

4.- MATERIALS.

Four materials were chosen with a view to carrying out this study, whose surfaces were apparently different to each other: glass, two transparent glazes (crystalline) and a matt white glaze.

The characteristics of the pieces tested are briefly described below:

a) Glass.

Owing to the uniformity of glass surface properties and microstructure, it was thought to use it in the first place, to test the experimental method proposed in Section 4 and verify its effectivity.

The prepared test pieces consisted of 100x100 mm pieces cut from sheets of float glass (cooled on a tin bath). This kind of glass is used in calibrating the abrasion tester, according to European standard EN154 [1].

b) Transparent glaze obtained in the laboratory.

The test pieces of this glaze were obtained by cutting up fired bodies of industrial ware, which were first coated with an engobe layer. A transparent glaze composition, commonly used in ceramic wall tile manufacture, was then applied. The pieces were then fired in an electric laboratory furnace at 1100°C for 3 minutes.

c) Transparent glaze.

Test pieces of this glaze were obtained by cutting pieces measuring 100x100 mm from industrially manufactured wall tile.

In the photograph of Figure 1a, it may be observed that there are no crystalline phases in this glaze.

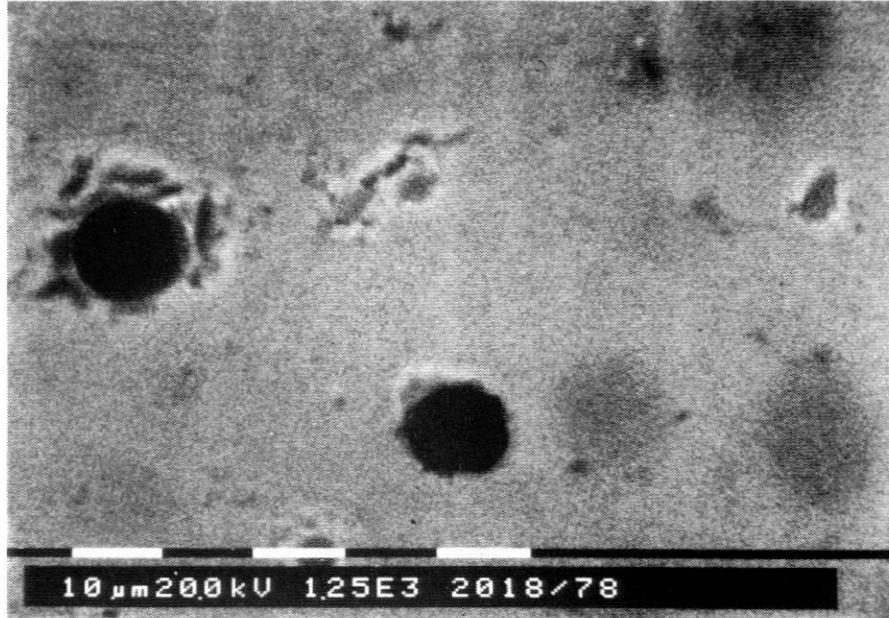


Figure 1a.- SEM photograph, magnification (1250x), revealing the absence of crystalline phases in the transparent glaze obtained from industrial ware.
d) Matt white glaze.

Industrially manufactured single-fired floor tile was used to obtain test pieces of this glaze.

In the photograph in Figure 1b, the crystalline phases included in the glassy phase of this glaze, as well as the open pores are to be observed, which, as will be seen below, are characteristic for this glaze.

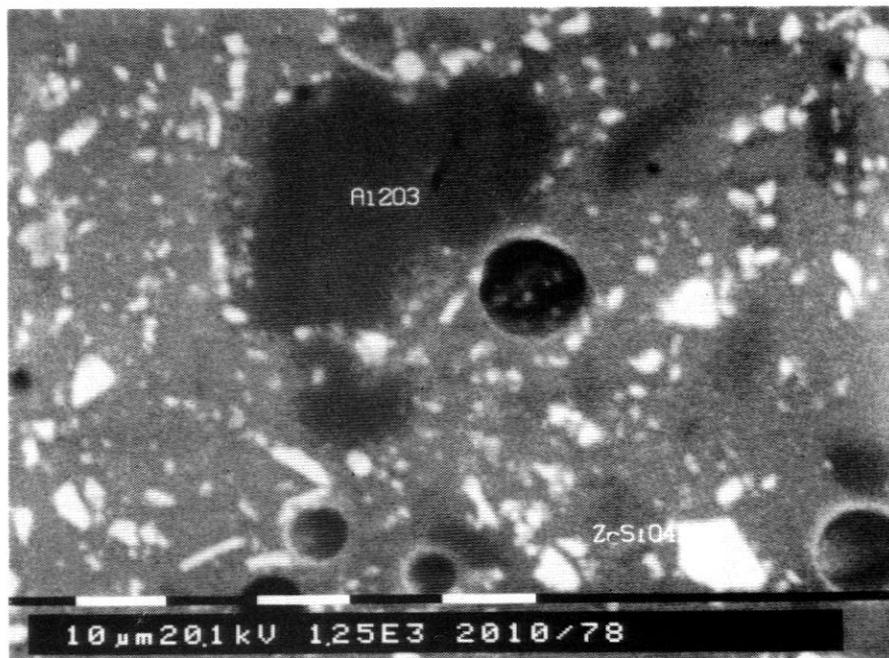


Figure 1b.- SEM photograph, magnification (1250x), revealing the presence of crystalline phases.

5.- RESULTS AND DISCUSSION.

5.1.- Wear resistance.

5.1.1.- Experiments with glass.

Figure 2 plots the values of x_v (see the ANNEX) versus the corresponding values of h for different degrees of abrasion, corresponding to different values of N . These values were deduced from the respective Abbott curves as set out in the ANNEX.

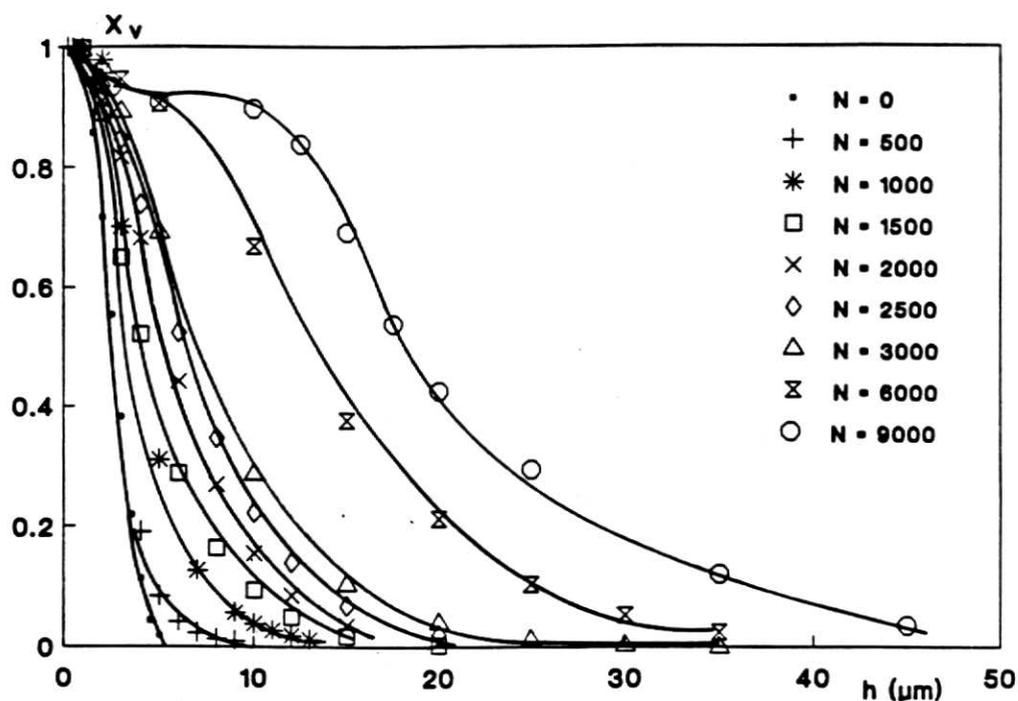


Figure 2.- Computation of the glass volume lost by abrasion.

From each of these curves, the values of $(A_h)_N$ and $(\Delta A_h)_N$ were computed by using Eq. (A-3) and the first and third members of Eq. (A-8) respectively (see ANNEX). The results are shown in Table I.

N (n.r.)	$(A_h)_N$ ($\mu\text{m}^2/\mu\text{m}$)	$(\Delta A_h)_N$ ($\mu\text{m}^2/\mu\text{m}$)
0	1.503	-
500	2.561	1.058
1000	2.997	1.494
1500	4.103	2.600
2000	5.133	3.630
2500	5.878	4.375
3000	7.921	6.418
6000	12.951	11.448
9000	18.633	17.130

Table I.

The values of N and $(\Delta A_h)_N$ in Table I are plotted in the form $(\Delta A_h)_N = f(N)$ in Figure 3. As may be observed, the points satisfactorily fit a straight line, virtually passing through the origin, having slope $1.923 \cdot 10^{-3}$, in accordance with the first and fourth members of Eq. (A-8).

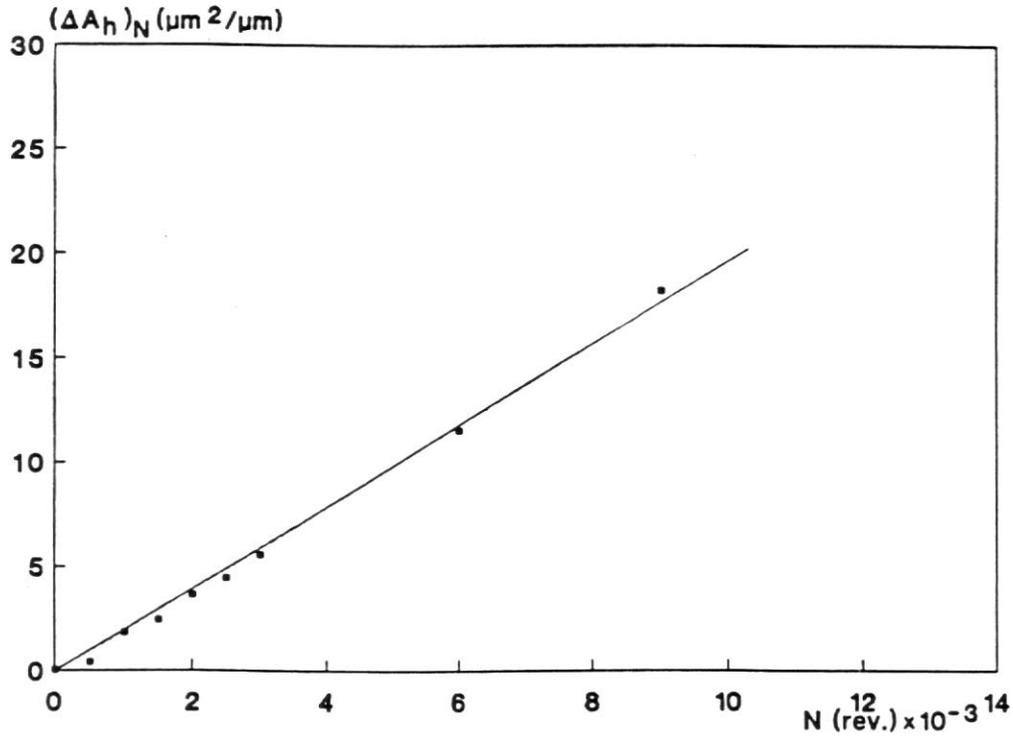


Figure 3.- Variation of the glass volume lost by wear, with the number of revolutions in the abrasion tester.

This outcome indicates that the behaviour of the glass, in the interval of N values studied, is analogous to that of metals, with regard to wear. A coefficient representative of wear resistance of the glass, was computed from the slope of the straight line found, with the value:

$$KE = 520,0 \frac{(n.r.) \mu m}{\mu m^2}$$

5.1.2.- Experiments with ceramic glazes.

5.1.2.1.- Homogeneous glazes.

a) Transparent glaze obtained in the laboratory.

Figure 4 reports the curves $x_v=f(h)$, obtained for different values of N to which the glaze was subjected in the abrasion tester. The values of $(A_h)_N$ and $(\Delta A_h)_N$, obtained from them are listed in Table II.

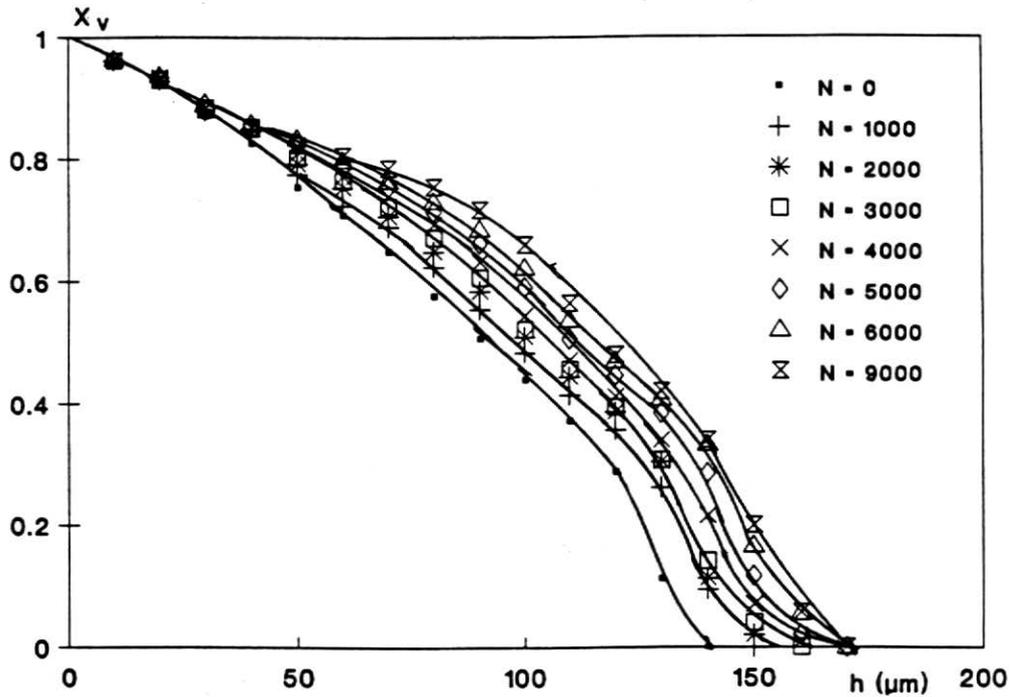


Figure 4.- Computation of the transparent laboratory glaze volume lost by abrasion.

N (n.r.)	$(A_h)_N$ ($\mu\text{m}^2/\mu\text{m}$)	$(\Delta A_h)_N$ ($\mu\text{m}^2/\mu\text{m}$)
0	84.74	-
1000	90.88	6.14
2000	93.67	8.93
3000	95.48	10.74
4000	98.22	13.48
5000	102.16	17.42
6000	105.24	20.50

Table II

On plotting these values in the form $(\Delta A_h)_N = f(N)$ (Figure 5), they may also be observed to fit a straight line with slope $2.857 \cdot 10^{-3}$. The value obtained for the coefficient of wear resistance $[KE = 350.0 \text{ (n.r.)} \cdot \mu\text{m}/\mu\text{m}^2]$, indicates that this glaze is less resistant to abrasion than the glass studied.

b) Transparent glaze of an industrial ware.

The values of $(A_h)_N$ and $(\Delta A_h)_N$ obtained for this glaze from the corresponding curves of $x_v = f(h)$, plotted in Figure 6, are listed in Table III.

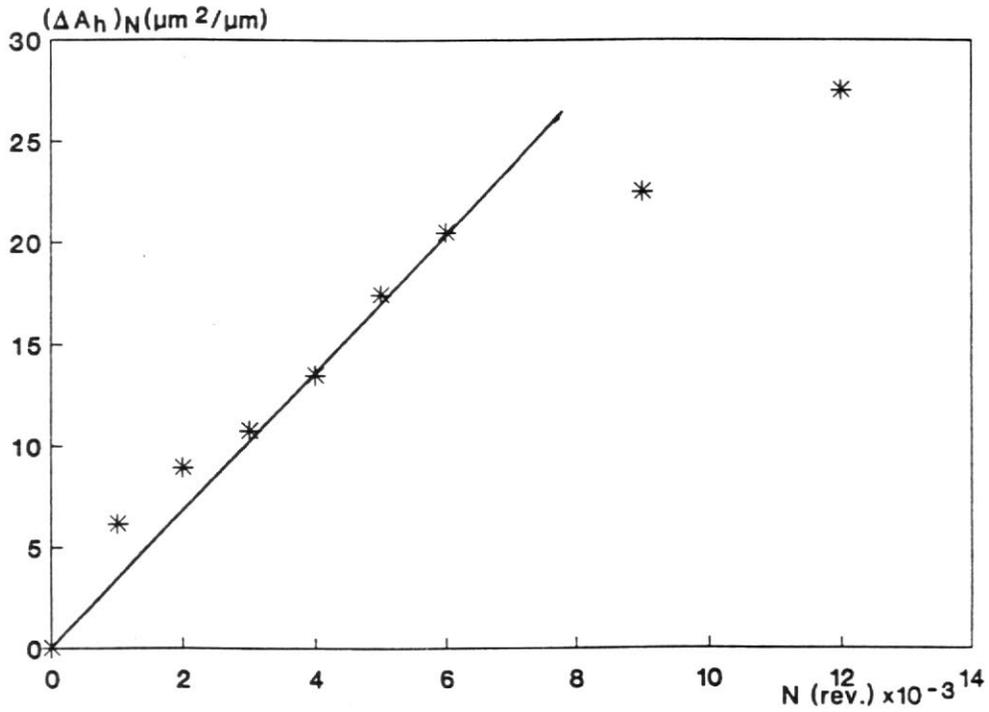


Figure 5.- Variation of the transparent laboratory glaze volume lost by wear, with the number of revolutions in the abrasion tester

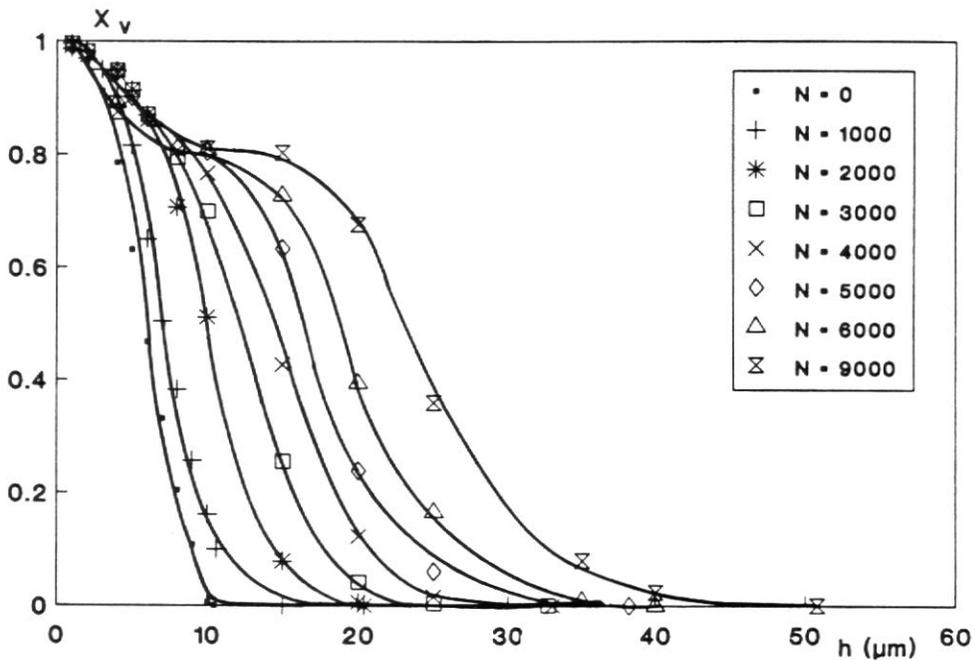


Figure 6.- Computation of the transparent glaze volume lost, obtained from industrial ware, by abrasion.

N (n.r.)	$(A_h)_N$ ($\mu\text{m}^2/\mu\text{m}$)	$(\Delta A_h)_N$ ($\mu\text{m}^2/\mu\text{m}$)
0	5.91	-
1000	7.36	1.45
2000	10.13	4.22
3000	12.01	6.10
4000	13.63	7.72
5000	15.72	9.81
6000	17.61	11.70
9000	21.61	15.70

Table III

The values of $(\Delta A_h)_N$ versus N are reported in Figure 7. These also fit a straight line passing through the origin, with slope $1.798 \cdot 10^{-3}$. From this curve, a value of $KE=556.2$ (n.r.) $\cdot\mu\text{m}/\mu\text{m}^2$ is deduced, of the same order as that obtained for the glass studied.

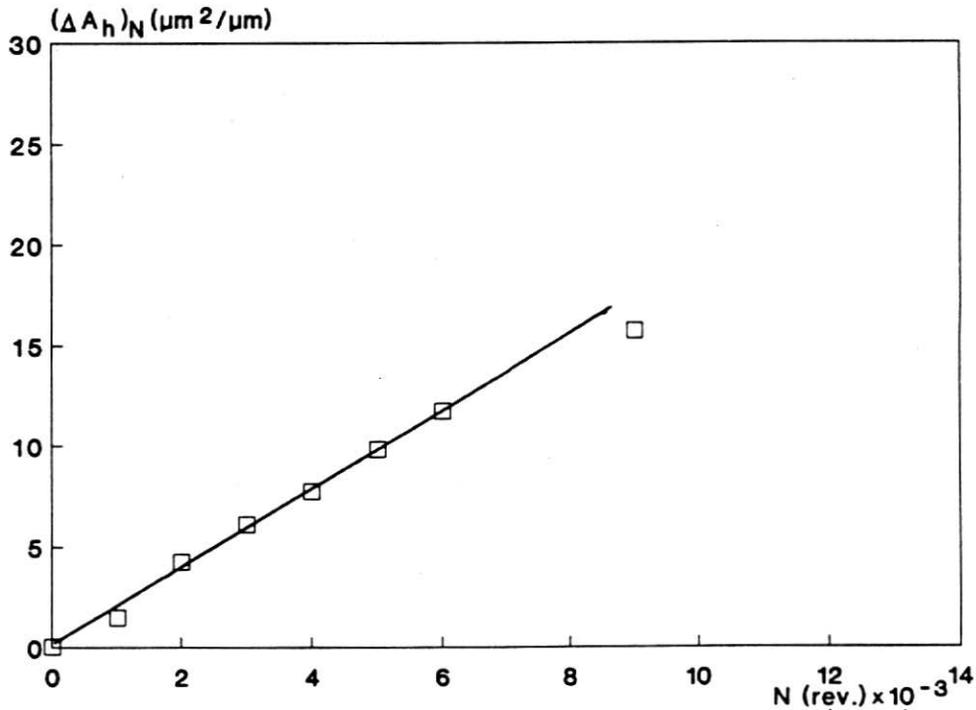


Figure 7.- Variation of the transparent glaze volume lost, obtained from industrial ware, by wear, with the number of revolutions in the abrasion tester.

5.1.2.2.- Matt heterogeneous glaze of an industrial ware.

The curves $x_v=f(h)$ for each value of N are plotted in Figure 8. The values obtained for $(A_h)_N$ are listed in Table IV together with the corresponding ones of $(\Delta A_h)_N$ for each value of N.

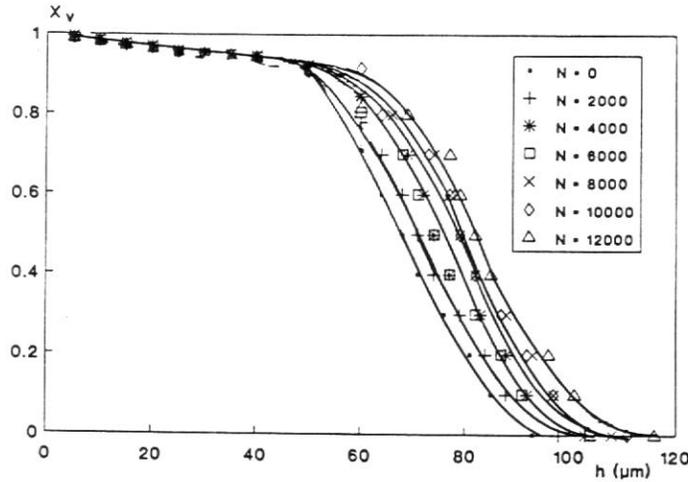


Figure 8.- Computation of the matt glaze volume lost, obtained from industrial ware, by abrasion.

N (n.r.)	$(A_h)N$ ($\mu\text{m}^2/\mu\text{m}$)	$(\Delta A_h)N$ ($\mu\text{m}^2/\mu\text{m}$)
0	65.88	-
2000	69.15	2.50
4000	73.23	5.03
6000	71.37	5.49
8000	77.39	8.97
10000	75.95	10.07
12000	78.27	12.39

Table IV

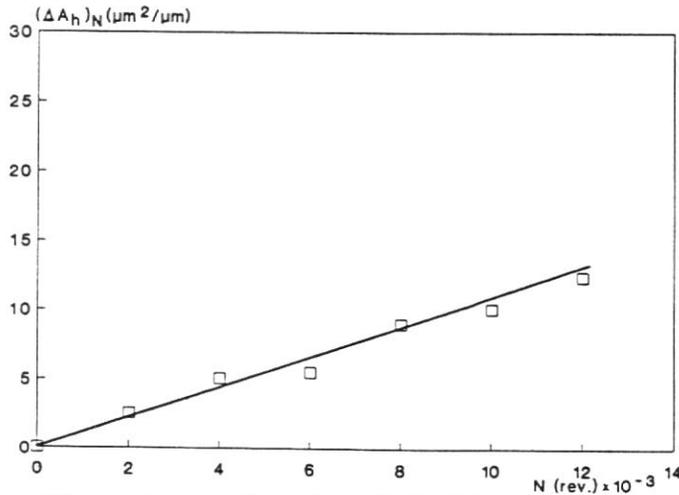


Figure 9.- Variation of the matt glaze volume lost, obtained from industrial ware, by wear, with the number of revolutions in the abrasion tester.

The plot of the values of $(\Delta A_h)_N$ versus those of N (Fig 9), fits a straight line with slope $1.04 \cdot 10^{-3}$, which yields:

$$KE = 961.5 \text{ (n.r.)} \cdot \mu\text{m}/\mu\text{m}$$

This result leads to the conclusion that the matt glaze has greater wear resistance than the glass or the transparent glazes.

5.1.3.- Mohs scratch hardness and Vickers microhardness. Comparison with the foregoing results.

Scratch hardness of each of the studied materials was determined, according to the method described in European standard EN 101 "Ceramic tiles. Determination of scratch hardness according to Mohs."

Vickers microhardness was also measured, by carrying out several indentations on the surface of each material and computing the arithmetic mean of the values obtained.

The results of these tests are reported in Table V, together with the values of the coefficient of wear resistance (KE) obtained previously.

Material	K.E (n.r.) $\cdot \mu\text{m}/\mu\text{m}^2$	Mohs hardness	Microhardness Vickers (kg/mm ²)
Glass	520,0	4	607
Trans.lab.glaze	350,0	3	-
Trans.ind.glaze	556,2	4	568
Matt ind. glaze	961,5	5	581

Table V

On comparing the values of the coefficient of wear resistance (KE) with the results obtained on testing scratch hardness, a certain parallelism between both properties may be observed, since the latter increases as wear resistance grows.

On comparing the values of the coefficient (KE) with those corresponding to microhardness, no kind of relation between these properties is to be observed. In the literature however [11], [12], there seems to be a certain relationship in the case of brittle materials, which include most ceramic materials (alumina, zirconia, etc).

The results obtained could be due to the fact that the mechanism used in conducting glaze scratching is more like that producing abrasion-induced wear than the mechanism producing the indent in the Vickers microhardness tester.

From the foregoing, it may be concluded that using the roughnessmeter to measure material loss caused by wear in a ceramic glaze, on treating it in an abrasion tester according to the PEI method, may be at least as representative as the method put forward in the standard, which is based on measuring material mass loss by weighing, brought about as a result of abrasion on applying 6000 revolutions in the abrasion tester.

The procedure tested here is based on the variations the wear surface profile undergoes and the error involved depends on the accuracy of the apparatus and the technique used. The method has one constraint: it can only be used when the starting glaze surface is completely flat or slightly concave.

On the other hand, the method involved in standard EN 154, is based on measuring very small changes in material mass, caused by the removal of part of this mass by wear, and measurement of which on sensitive scales, is subject to considerable errors and fluctuations in the case of ceramic ware. In fact, the conditions in which the test is carried out (relative atmospheric moisture, time the test pieces dwell in certain ambient conditions, etc.), can influence the findings.

5.2.- Variation of roughness, the "void volume dirt retention capability", and surface quality of the glaze with the number of revolutions it undergoes in the abrasion tester.

In this section, tests were solely carried out with the glass and with the glazes (transparent and

matt) of industrially fabricated ware.

With a view to studying the possible relation of the parameters R_a and v_s with the dirt retention capability, and its evolution with wear, these parameters were measured, first for the surface of the unabrased test piece and then for the surfaces resulting on sequential wear in the abrasion tester, at 200-revolution intervals. After each measurement, the test piece was dirtied as described in Section 4.1.2 and surface appearance was examined. The results of the measurements carried out, together with surface appearance appraisal of the test pieces on dirtying them, for each material studied, are reported in Tables VI, VII, and VIII, and are plotted in the form $v_s=f(N)$ and $R_a=f(N)$, in Figures 10 and 11 respectively.

N	R_a (μm)	v_s ($\mu\text{m}^3/\mu\text{m}^2$)	Surface appearance
0	0.10	0.010	Clean
200	0.17	0.024	Slightly dirty
400	0.21	0.062	Slight darkening
600	0.44	0.109	Clearly dirty
800	0.58	0.149	"
1000	0.76	0.194	Very dirty

Table VI.- Variation in the roughness and the volume of the potential dirt retention capability of the surface, and its visual appearance for the glass studied.

N	R_a (μm)	v_s ($\mu\text{m}^3/\mu\text{m}^2$)	Aspecto de la superficie
0	0.51	0.015	Limpia
200	0.57	0.046	Ligeramente sucia
400	0.58	0.081	Claramente sucia
600	0.74	0.134	Muy sucia
800	0.86	0.156	"
1000	0.93	0.237	"

Table VII.- Variation in the roughness and the volume of the potential dirt retention capability of the surface, and its visual appearance for the transparent glaze studied.

N	R _a (μm)	v _s (μm ³ /μm ²)	Surface appearance
0	2.32	0.149	Slightly dirty
200	2.32	0.143	Slightly dirty. Ring
400	2.29	0.148	Retained dirty
600	2.31	0.147	"
800	2.30	0.151	"
1000	2.28	0.148	"

Table VIII.- Variation in the roughness and the volume of the potential dirt retention capability of the surface, and its visual appearance for the matt glaze studied.

On comparing the values of R_a and v_s in Tables VI and VII, and the plots in Figures 10 and 11, these parameters are observed to have very low values at the beginning, increasing progressively as N increases (wear intensity) in the case of glass and transparent glaze. On the contrary, in the matt glaze R_a and v_s remain virtually constant, right from the start, having considerably higher values than the other two materials.

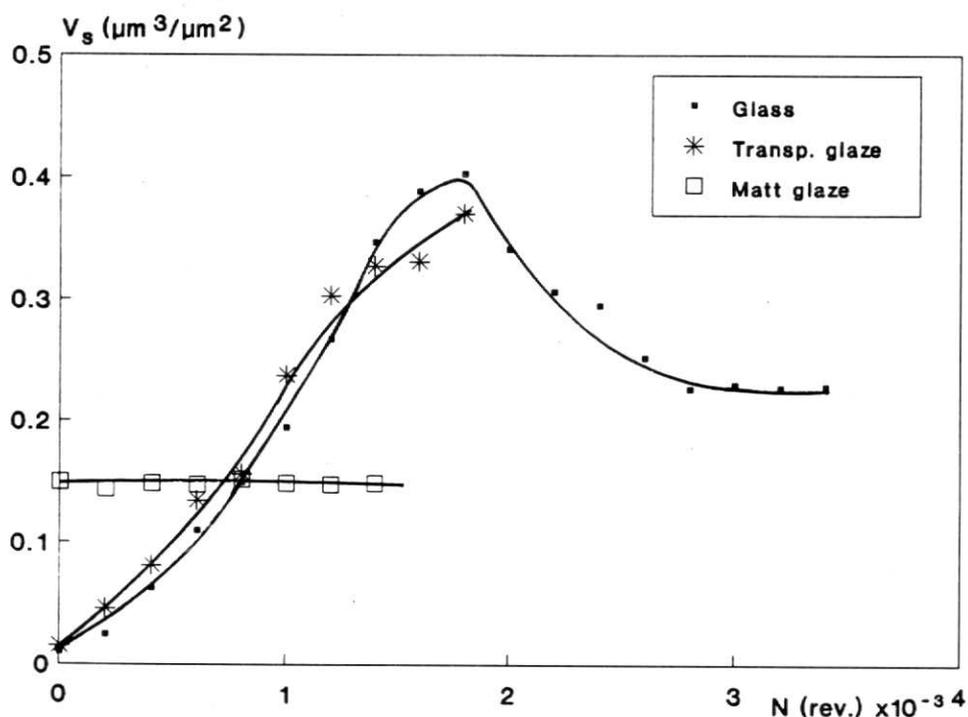


Figure 10.- Evolution of v_s with wear intensity.

Visual inspection showed that initial glass and transparent glaze surfaces did not retain dirt, but on increasing N (degree of wear), retained dirt became more and more noticeable in the abraded area. In the case of the matt glaze, it was observed to already retain dirt before wear, probably owing to the high roughness value and v_s of the starting surface.

On examining the wear faces through a stereoscopic magnifying glass, small cracks and fractures were already noticeable at low N values, in the form of chipping on the abraded surface. Figure 12 shows a photograph taken by SEM, in which these chips on the transparent glaze surface can be observed, after treatment at 800 revolutions in the abrasion tester. The photograph reveals that the impacts to which the surface was subjected, have broken off part of the glaze, giving rise to severe edge chipping and high roughness. These craters or flakes are clearly distinguishable from the circular, deeper open pores, appearing together with them in the photograph in Figure 13, corresponding to the matt glaze abraded at 400 revolutions.

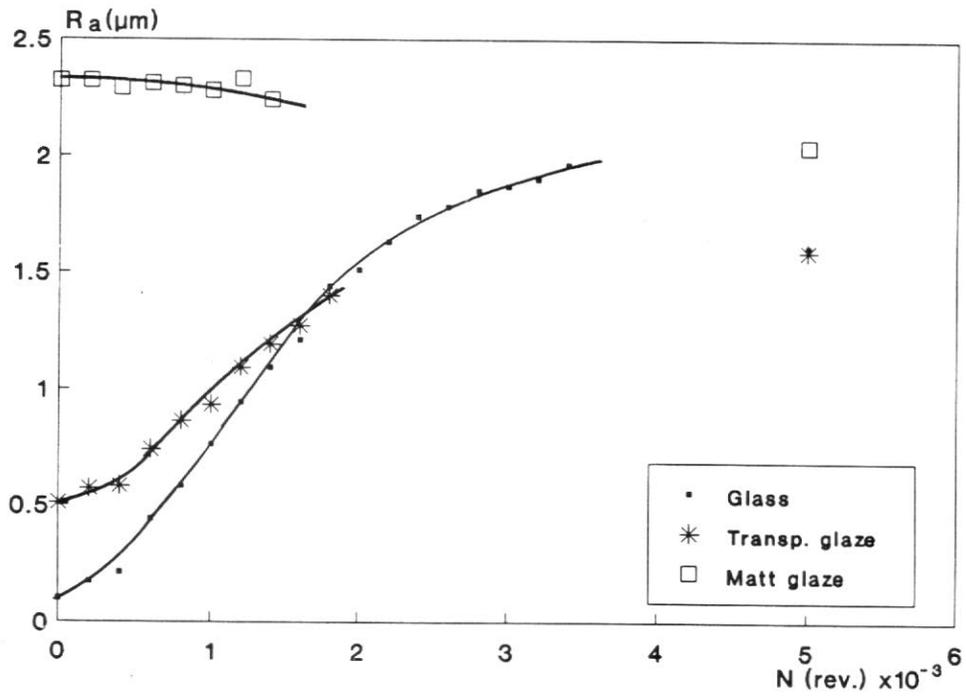


Figure 11.- Evolution of R_a with wear intensity.

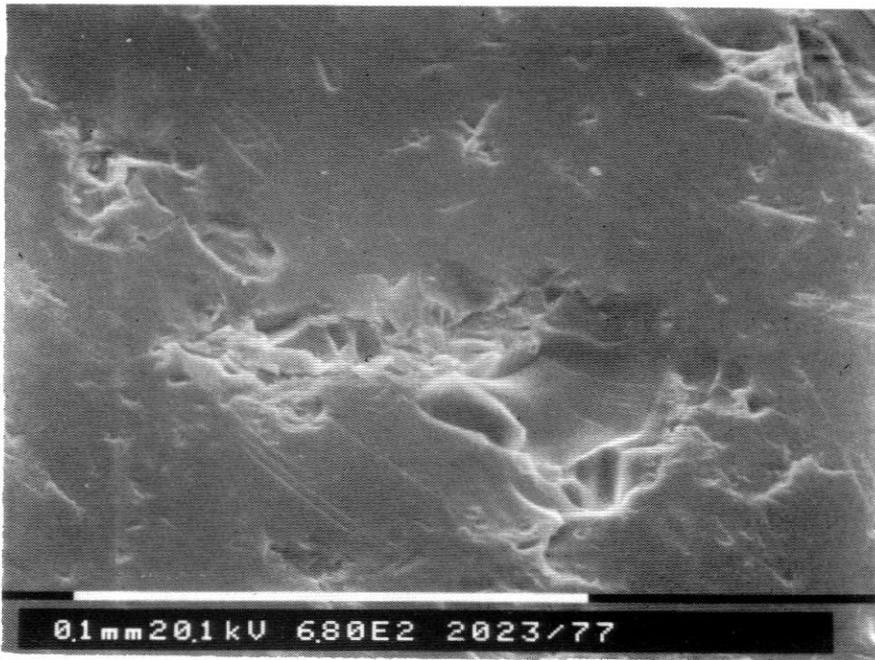


Figure 12.- SEM photograph, magnification (680x), of the surface of the transparent glaze, showing wear at 800 revolutions.

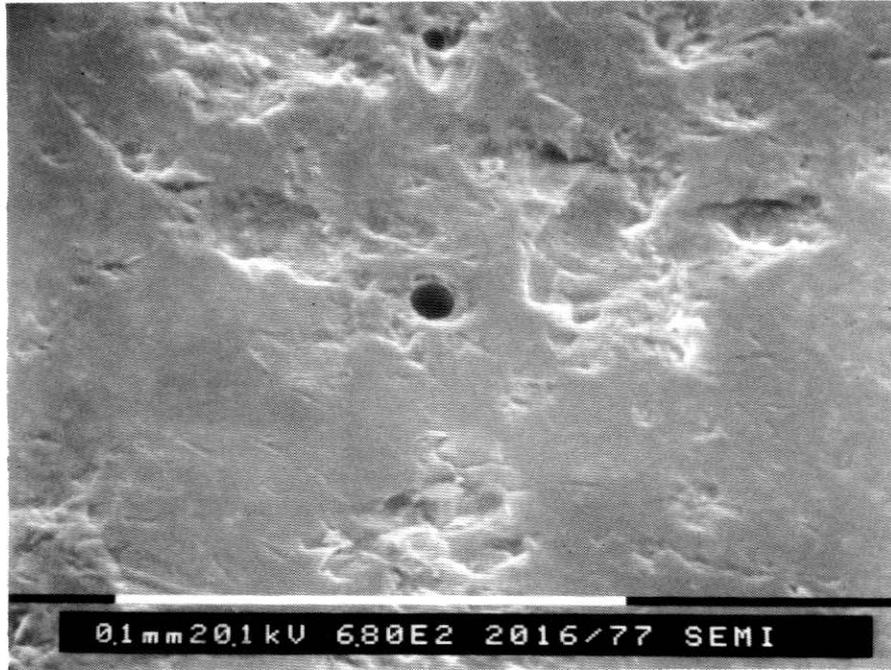


Figure 13.- SEM photograph, magnification (680x), of the surface of the matt glaze, showing wear at 400 revolutions.

On comparing surface appearance, in Tables VI, VII and VII, after “dirtying”, with the roughness values (R_a) and the void volume potentially capable of dirt retention, it may be concluded that R_a does not appear to maintain any coherent relation at all with the visual appreciation of glaze dirt retention. In fact, a slight darkening in the glass begins to become noticeable for $R_a=0.21 \mu\text{m}$, whilst this is observed in the transparent glaze for $R_a=0.57 \mu\text{m}$. The matt glaze does not allow drawing any conclusion, since a slight darkening is already observable in the starting surface before abrading, for $R_a=2.32 \mu\text{m}$.

On the other hand, the value of the parameter v_s might maintain a certain relationship to surface dirtiness, as this began to be detected in glass for $v_s=0.024 \mu\text{m}^3/\mu\text{m}^2$ and in transparent glaze for $v_s=0.046 \mu\text{m}^3/\mu\text{m}^2$. The matt glaze is dirtied, without abrading, and has an initial v_s of $0.149 \mu\text{m}^3/\mu\text{m}^2$, which seems to confirm that for v_s values greater than $0.024 \mu\text{m}^3/\mu\text{m}^2$, surface dirtiness becomes noticeable.

As in Tables VI and VII, v_s measurement and visual appreciation of dirtiness for the glass and the transparent glaze were carried out at 200 revolution intervals in the abrasion tester, it was decided to repeat this experiment, carrying out measurement and surface appearance inspection at 25 revolution intervals, in order to see if the values for v_s at which dirtiness in the glass and transparent glaze began, approached each other more closely, as dirtiness was not found in either of them for $N=0$.

The findings are listed in Tables IX and X.

N	v_s ($\mu\text{m}^3/\mu\text{m}^2$)	Appearance
0	0.008	Clean
25	0.007	"
50	0.012	"
75	0.023	Slight darkening
100	0.025	"

Table IX.- Variation of v_s with the number of revolutions in the abrasion tester for the glass.

N	v_s ($\mu\text{m}^3/\mu\text{m}^2$)	Appearance
0	0.011	Clean
25	0.019	"
50	0.023	Slight darkening
75	0.028	Darker
100	0.034	"

Table X.- Variation of v_s with the number of revolutions in the abrasion tester for the transparent glaze.

As may be observed, darkening of the surface begins to become noticeable for $v_s=0.023 \mu\text{m}^3/\mu\text{m}^2$, for both the glass as well for the transparent glaze. This v_s could therefore be considered the critical value, starting from which surface dirtiness becomes noticeable.

On abrading the matt glaze, besides chipping, open pores are seen to appear at 400 revolutions (Figure 13). These pores, besides contributing to increasing the dirty appearance of the wear surface, retain dirt irreversibly, unlike what is retained in chips, which can be removed by washing with soap and water.

In this matt glaze, the appearance of open pores in the wear surface does not appear to noticeably contribute to increasing the v_s parameter, from what may be deduced from Table VIII and Figure 10, where this parameter is hardly observed to vary with N, despite open pores appearing at N=400. The value of v_s appears to depend almost wholly on the void volume produced in the surface as a result of chipping.

In Figures 14, 15 and 16 photographs are shown of cross-sections of the three materials studied. In the case of the glass, no bubbles appear (Figure 14), in the transparent glaze (Figure 15) bubbles with a diameter of $\leq 10 \mu\text{m}$ appear and in the matt glaze (Figure 16) the bubbles are larger, some of them having a diameter in the order of 80 and 50 μm .



Figure 14.- SEM photograph, magnification (326x), of the cross-section of the glass.

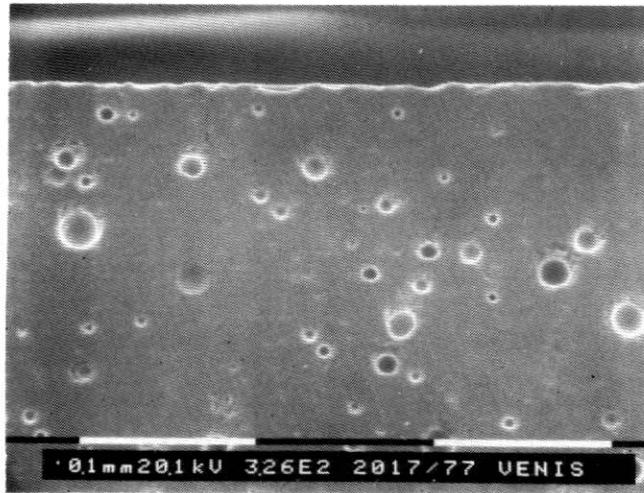


Figure 15.- SEM photograph, magnification (326x), of the cross-section of the transparent glaze.



Figure 16.- SEM photograph, magnification (326x), of the cross-section of the matt glaze.

On abrading the glaze, these bubbles appear at the surface and form the above-mentioned open pores, in which dirt enters irreversibly. On the wear surface of the matt glaze, these pores can be seen better, as well as the dirtiness they cause, owing to the bubbles being larger and therefore also the pores they give rise to.

6.- CONCLUSIONS.

The following conclusions may be drawn from the findings obtained in this study:

- i) Using a roughnessmeter in determining the material loss a ceramic glaze undergoes on

subjecting it to wear by abrasion, has led to promising results, as these satisfactorily fit the equations put forward for wear by abrasion of metals and technical ceramics.

- ii) A technique has been developed allowing quantitative values to be obtained for a coefficient of resistance to wear by abrasion for ceramic glazes. This technique, based on the progressive material volume loss the glaze undergoes, might give more representative values for this property than the method based on mass loss at 6000 revolutions, determined by weighing, as established by the PEI method.
- iii) Wear in ceramic glazes, in an abrasion tester, has been observed to arise as a result of progressive chipping of the abraded surface. The holes and craters appearing retain dirt which is removable by washing.
- iv) A parameter v_g (void volume or craters in the wear surface of a solid material, per unit area) has been related to the dirt-bearing capability of ceramic glaze surfaces. It was observed that dirtiness of these surfaces began to become visually noticeable for values of this parameter above $0.023 \mu\text{m}$.
- v) It was confirmed that the open pores formed in ceramic glaze surfaces from the internal bubbles appearing at the surface as a result of wear, contribute to dirtiness, retaining dirt inside themselves which is difficult to remove by washing.

ANNEX

COMPUTATION OF THE COEFFICIENT OF WEAR BY ABRASION

The Abbott curve [9], for a rough surface, is the plot of the solid fraction (x_s) existing on a plane located at a distance h from the highest peak of the measured profile (where $x_s=0$), versus the different values for h ranging from 0 to h_m (Figure A.1).

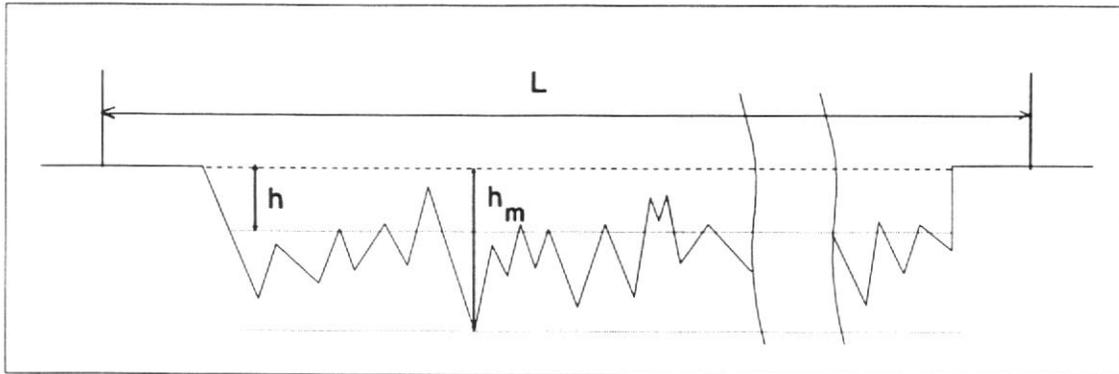


Figure A.1.- Definition of L , h and h_m .

If for a plane located at a distance h from the highest peak, on defining:

- L = Profile length scanned by the roughnessmeter pick-up (m).
- L_s = Profile length scanned occupied by solid material (m).
- L_v = Profile length scanned occupied by void (m).

the scanned profile fraction occupied by solid material on this plane will be:

$$x_s = \frac{L_s}{L} \quad (\text{A-1})$$

L

and the scanned profile fraction occupied by void

$$x_v = 1 - x_s \quad (\text{A-2})$$

The roughnessmeter can calculate the Abbott curve (Figure A.2), for a given profile and from it deduce the curve $x_v=f(h)$.

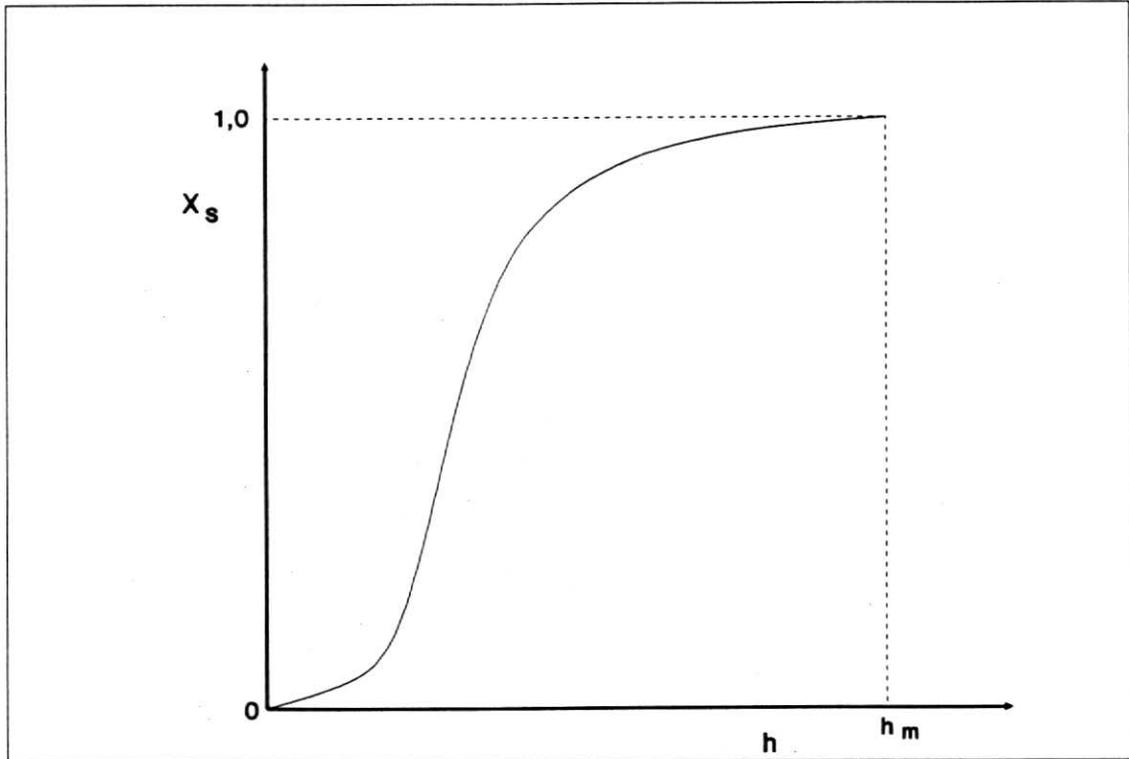


Figure A.2.- Abbott curve.

If wear of a glaze surface takes place in an abrasion tester according to the PEI method, and the profile is always measured for the same wear circle diameter (82 mm), for each value of N a curve of $x_s=f(h)$ will be obtained, from which the corresponding $x_v=f(h)$ curve can be deduced.

By operating like this a set of curves will found as shown in Figure A.3.

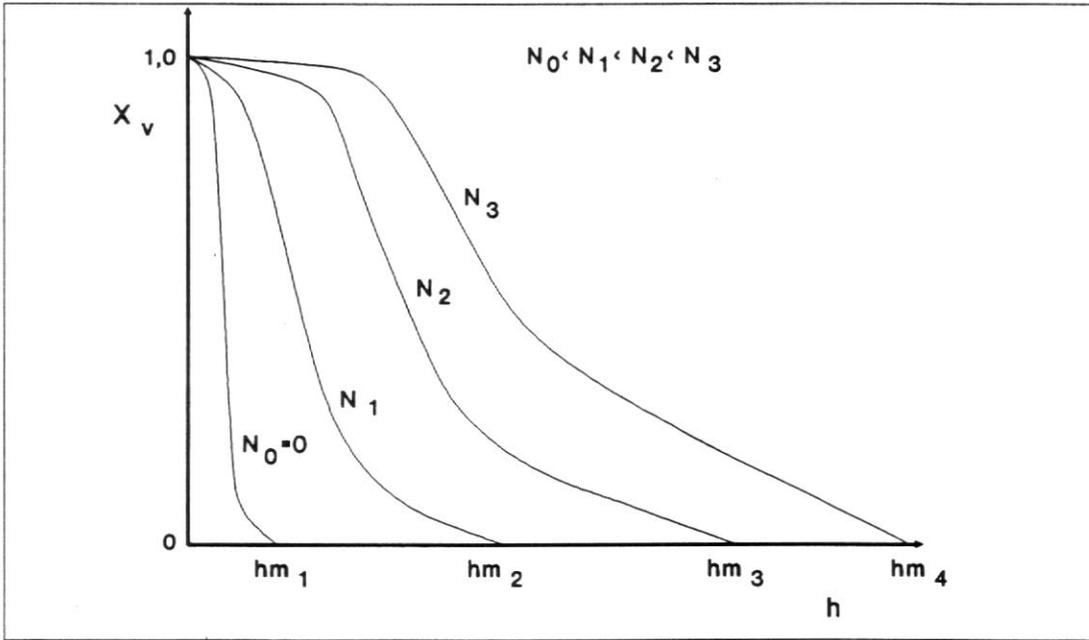


Fig A.3.- Variation of x_v with h_m for different degrees of abrasion (N).

The void area to be found in a vertical section, along the length of the profile scanned by the pick-up ($A_h \rightarrow$ m void/m scanned profile), can be computed from the curves $x_v=f(h)$ (Figure A.4). Therefore, the integral is to be computed:

$$(A_h)_N = \int_0^{(h_m)_N} x_v dh \quad (A-3)$$

where $(A_h)_N$ and $(h_m)_N$ are the values of A_h and h_m corresponding to each value of N applied to the glaze.

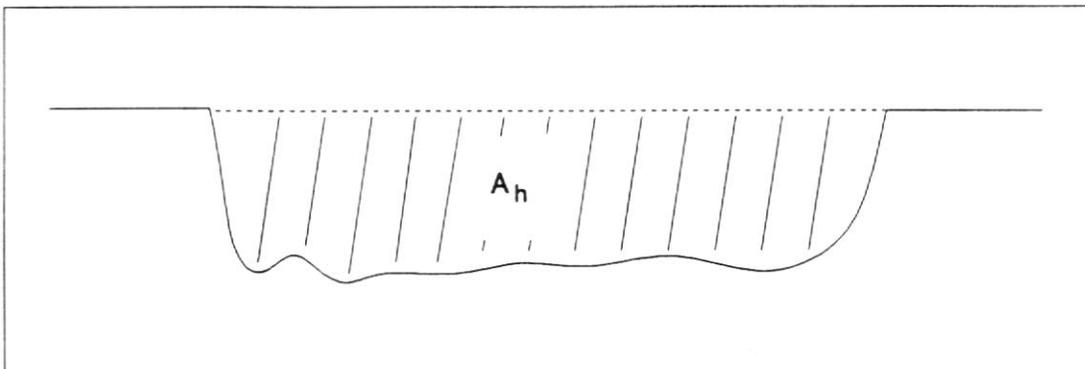


Fig A.4.- Definition of the variable A_h for a given section of the eroded area (m of void/m of scanned profile).

On abrading a glaze by the PEI method, abrasion is produced as mentioned above, in a circle with an 82-mm diameter. If the profile for which the curves $x_v=f(h)$, corresponding to different values of N , is always measured along the same circle diameter (always placing the square test piece in the same position on the roughnessmeter), and assuming that the value of $(A_h)_N$ obtained for this profile is representative of the behaviour of the whole eroded area, there must be a direct relationship between this magnitude and the material volume lost (or void volume produced) per unit surface area scanned, expressing it in the form:

$$(V_h)_N = K (A_h)_N \quad (A-4)$$

If $(V_h)_N$ has the dimensions of μm^3 void produced for each μm of surface area scanned by the roughnessmeter pick-up, the constant K must be dimensionless.

Eqs. (A-3) and (A-4) yield:

$$(V_h)_N = K \int_0^{(h_m)_N} x_v dh \quad (\text{A-5})$$

For each value of N , the material volume lost by wear will be:

$$(\Delta V_h)_N = (V_h)_N - (V_h)_0 \quad (\text{A-6})$$

where $(V_h)_0$ is the value of V_h computed for the profile corresponding to $N = 0$, which does not necessarily have to be zero, as the starting surface is not completely smooth.

In accordance herewith and taking into account Eq.(3) from the Introduction, yields:

$$(\Delta V_h)_N = K \left[\int_0^{(h_m)_N} x_v dh - \int_0^{(h_m)_0} x_v dh \right] = \frac{N}{E} \quad (\text{A-7})$$

or also:

$$(\Delta A_h)_N = \frac{(\Delta V_h)_N}{K} = \int_0^{(h_m)_N} x_v dh - \int_0^{(h_m)_0} x_v dh = \frac{N}{K E} \quad (\text{A-8})$$

From the first and fourth members of Eq.(A-8) is to be deduced that on plotting $(\Delta A_h)_N = f(N)$, a straight line will be found, having slope $1/(K \cdot E)$, that is, the inverse of a parameter representative of resistance to wear by abrasion.

7.- REFERENCES.

- [1] Baldosas cerámicas. Determinación de la resistencia a la abrasión. Baldosas esmaltadas. UNE 67-154-85 (EN 154). Recopilación de Normas UNE. Vol. 2 - Construcción. Pág. 188-197. AENOR. Madrid, 1988.
- [2] P.A. Walters y R. Harrison; Ceram Research RP 741; 3-15.
- [3] C. Felú, M.J. Ibáñez, A. Moreno, J.V. Carceller; Ponencia presentada al I Congreso de la Calidad de Pavimentos y Revestimientos Cerámicos QUALICER. Abril, 1990.
- [4] J.K. Lancaster; Wear; 141; 159-183; 1990.
- [5] J. Bijwe, C.M. Logani, U.S. Tewari; Wear; 138; 77-92; 1990.
- [6] D. Fingerle, W. Gundel, H. Opalinski; En: W. Bunk, H. Hausner. Ceramic Material and Components for Engines, 1191-1200. Lübeck-Travemünde, 1986.
- [7] S.A. Norton, J. Denape, D. Broussand, D. Dowson, F.L. Riley, N. Wallbridge; En: Stuart Hampshire. Non-oxide technical and engineering Ceramics. England. Elsevier Applied Science, 281-298, 1986.
- [8] J.H. Tylczak; Wear; 135; 305-318; 1990.
- [9] L. Mummery; Surface Texture Analysis. The Handbook; Hommelwerke GmbH, West Germany, 1990
- [10] Asociación Española para el Control de la Calidad. Comité de Metrología. Comentarios sobre la Medición actual de la rugosidad. 3. Consejos para la Práctica Metrológica.
- [11] Z. Gahr, Tribology Series, 10. Microstructure and Wear of Materials. Ed. Elsevier. New York, 1987.
- [12] K. Kato; Wear; 136; 117-133; 1990.

