DESIGN AND DEVELOPMENT OF AN APPARATUS TO MEASURE THE SCRATCH HARDNESS OF CERAMIC FLOOR TILES

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ABSTRACT

Wear strength is an important criterion for classifying different kinds of floor tiles. Many techniques were developed in order to estimate the durability of a particular type of floor tile according to the severity of traffic to which it will be subjected. In Brazil, the PEI and Mohs tests are widely used, in spite of the subjectivity and poor accuracy of the final data inherent to these methods. Sclerometry, or the scratching test, is an alternative experimental technique that allows achieving quantitative measurements of the scratch hardness of materials. The method basically consists of simulating the effects of a single abrasive particle, where an indenter with known geometry is drawn against the surface of the sample to promote the scratch. During the test, normal and tangential loading are monitored while the indenter velocity is controlled. This work proposes to describe the main details of the development of a sclerometric apparatus at The Laboratory of Materials of The Mechanic Engineering Department of The Federal University of Santa Catarina. The equipment is already running and is fitted with a clamp device in order to use commercial hardness indenters. Piezoelectric sensors measure the loading in both normal and tangential direction. The indenter movements are computer commanded, with up to 10 newton normal loading capacity. Either constant or variable loads can be applied. The latter is useful for detecting the critical load condition in which the rupture mode changes from ductile to brittle. The load applied, and the corresponding width of the scratch at each point, are evaluated to determine the Scratch Hardness and the Specific Energy required to remove a unit volume from the material surface. The harder the material, the narrower is the scratch. In order to highlight the potential of the sclerometric technique, a comparative study has been carried out on several tiles with known PEI and Mohs results. The advantages and limitations of this technique with regard to brittle materials are discussed, as well as the contribution to a more suitable characterization criterion for a reliable classification of ceramic floor tiles.

1. INTRODUCTION

Tribology is the science that studies friction phenomena. It is a complex branch in which several factors of the whole tribological system must be considered. Geometric, mechanical, chemical and physical characteristics of the entire friction region need to be taken into account.

The main point in tribology is the estimation of the useful life of components while subjected to given service conditions. In the case of ceramic tiles, as well as in many others, durability is a decisive classification criterion, since it can encourage the consumption of those products considered most suitable for the severity of the intended use. A wide consensus is therefore necessary with regard to the method used for evaluating durability. A consistent classification must take into account not only a technical and scientific point of view, but also the end-user's acceptance.

Nevertheless, the methods that best simulate the real conditions of use, which obviously would characterize the samples better, are usually unfeasible because of demands of time, price, or even the limitation of the results. Therefore, the use of more general methods becomes necessary for the classification of products in relation to their tribological properties, of which abrasive resistance is the most important and hence will be the property dealt with below.

Among the techniques for studying abrasive phenomena, testing by abrasive wear and sclerometry are the most usual. The abrasive wear test evaluates the deterioration of the surface tested. In other words, the tribological system is examined before and after the test, and any differences are attributed to the abrasive process. The results are usually expressed in mass or volume loss[1-4].

For ceramic tiles, the PEI abrasion test described in ISO 10545 and in EN 154[2-4] is internationally recommended and widely adopted, including in Brazil. The name is taken from the initials of the "Porcelain Enamel Institute ". Basically, the PEI test places in one class all the products which present visually perceptible changes in their surface after a test stage. An observer makes the visual inspection under standard viewing and illumination conditions, in order to minimize the problems of subjectivity. The higher the PEI class, the greater is the abrasion resistance.

Sclerometry, or the scratching test, consists basically of promoting a scratch at the surface of the sample knowing the applied load, the scratching speed and some geometric characteristics of the indenter. The results are related to the width of the scratch and to the average of the normal and tangential components of the force used during the test. The average penetration pressure is defined by scratch hardness, expressed in force by unit of area (pascal). It should be pointed out, that while sclerometry requires a measurement system for the systematic collection and treatment of the measurements, which can raise the cost because of the accuracy needed, subjectivity is avoided.

Abrasive wear testing is considered to provide an overall insight in the sense that it computes the interaction of several abrasive particles acting on an entire surface of the sample, whereas sclerometry, in which the test is limited to the effect of a single particle, is more related to the relative hardness between the indenter and the sample and, therefore, is considered a local insight[2-3].

However, the possibility that the sclerometric results can be interpreted with greater accuracy than the abrasion test data allows sclerometry to offer a broader range for classification process, since many products of similar behaviour in abrasion tests can then be differentiated through sclerometry. In fact, an example of such case will be observed below, in the experimental part of this work.

Although both techniques have different approaches, the literature suggests good correlation between the results of PEI and sclerometry tests[4]. However, an easier adaptation to the factory environment and perhaps the lesser requirements of technological support have contributed to the current level of acceptance of the PEI test.

Nowadays, however, the subjectivity of the PEI method is making it more and more inadequate. This is due to the increasing modernization of industries and the availability of technological devices, followed by the search for quality and diversity and a more demanding market.

In this sense, the Laboratory of Materials of the Department of Mechanical Engineering of the Federal University of Santa Catarina - Brazil, has designed and built an computerized sclerometer, with a view to studying the abrasive process, and eventually suggesting test conditions that best characterize the different types of ceramic floors, thus contributing to the standardization of sclerometry and a reliable classification of products according to use.

With a view to exemplifying the potentiality of sclerometry in the several types of ceramic tiles, the following samples have been tested: a porcelainized stoneware tile, two single-fired wall tiles, one single-fired stoneware floor tile, and a glass-ceramic product with high abrasive resistance. The scratch hardness, the energy required to remove a unity volume from the sample surface, and the Coulomb coefficient of attrition for these materials were determined.

2. ABRASIVE WEAR AND SCLEROMETRY

Abrasive wear is defined as the progressive loss of surface material due to a relative movement between two bodies. According to literature[1-4], as result of geometric characteristics, and rheological and physico-chemical factors, three basic mechanisms of material loss can occur:

(a) micro-ploughing,

(b) micro-cutting and

(c) micro-cracking,

schematically displayed in Figure 1.

Figure 1. Material loss mechanism

In micro-ploughing, the action of the abrasive particles causes plastic deformations that result in material accumulation long its trajectory, leaving a kind of furrow. The material loss is very small and it is due either to an interaction among effects of the different abrasive particles, or of repeated plastic deformations. In micro-cutting, the shear stress at the front of the abrasive particles causes the separation of fragments in the form of micro-shavings.

The mechanism of micro-cracking is limited to brittle materials, and it is characterized by the sudden pullout of great fragments from the surface. This is caused by the crack formation and propagation. The mechanism is governed essentially by the classic theories of the fracture mechanics, and occurs when the material is subjected to loads higher than critical values.

Each of the mechanisms mentioned above has it own degree of severity, so that any attempt to estimate durability is extremely partial. In other words, abrasive resistance is not an intrinsic material property, but one of the whole tribological system, which again evidences the complexity of the phenomena.

Therefore, there is a great difficulty of adopting general models in order to enable establishing satisfactory analytical formulations, which makes experimental analysis the basic tool in the study of the abrasive process.

When simulating a scratch in the polished surface of a sample using an indenter of simple geometry, under idealized conditions, the sclerometric test allows development of models that, although simplified, can make real contributions to the understanding of the underlying mechanisms of the abrasive process. The following contributions may be noted:

- the determination of properties such as scratch hardness, toughness, specific energy, and brittleness index, for each constituent phase of the material and,
- the identification of the conditions for transition from a ductile to a brittle mechanism of material loss, as well as the contribution of each of these mechanisms in the abrasive process.

This is done by considering the forces acting on the surface of the sample while under scratching, illustrated in Figures 2 (a) and (b). The direction of this resulting force can only be known in indenters of simple geometries. As soon as the preset normal load (Fn) is reached, the indenter begins a parallel relative movement to the surface. This movement produces tangential loading (Ft), which in turn is recorded.

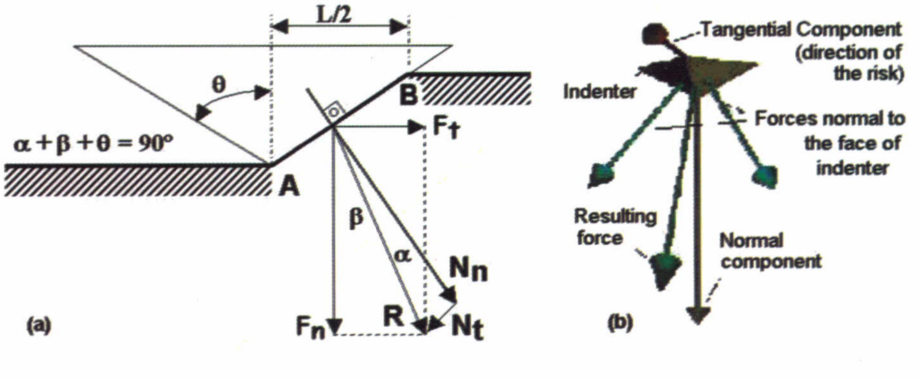

Figure 2. Forces acting in a (a) general and (b) pyramidal indenter

Among the several sclerometric parameters to be defined, scratch hardness (H_r) and specific energy (e) are to be highlighted. Figure 2 also gives:

$$H_r = F_n / A_n = K_1 F_n / L^2 \qquad (1)$$

$$e = F_t / A_t = K_2 F_t / L^2 \qquad (2)$$

Where A_n and A_t are horizontal and vertical projections of the contact area AB, K_1 and K_2 are constants related to the geometry of the indenter and L is the width of the scratch.

The forces in the contact plane also reveal the existence of a normal force N_n, responsible for the plastic deformation of the material, and of the tangential force N_t, responsible for the relative slipping between the indenter and the contact surface. The ratio among components N_t and N_n furnishes the Coulomb surface friction coefficient ($= tg\beta$).

3. CONCEPTION AND DESIGN DETAILS

In Brazil the State of Santa Catarina leads the production of wall and floor tiles. The Federal University has played an important rule in this achievement. Ceramic tiles have been the subject of several researches, some of them suggesting changes in manufacturing, and some of them studying phenomena as the wear strength.

The national current criterion for the wear strength classification is the PEI test, despite its subjectivity and the poor quality inherent to this method. In an attempt to improve this classification, and also to study the abrasive phenomena in greater depth, a sclerometric apparatus has been designed and developed at the Laboratory of Materials of The Mechanic Engineering Department. According to the literature[4], a commercial apparatus can be bought for about US$ 60,000, without any computerization. This has been an important motivation in trying to develop a complete one.

From the outset, the initial structure was based on previous knowledge of many available models, in particular, the one mounted at the Federal University of Uberlândia. The apparatus was mounted on a rigid table, solid enough to avoid external influences. This table is in turn supported by a steel structure with vibration-free feet.

Sclerometric tests require measurement of force in two axes, in both normal forces (Z) and tangential forces (X). As very small loads are used during the tests, a measurement system with compatible sensitivity is needed, usually in a range of 0.10 to 0.20 newton. Piezoelectric sensors were therefore used due to their response capability under dynamic loads. The software was adapted to compensate a well-known decrease in their sensitivity during the test period.

The displacement on the X axis is the principal movement of a sclerometric test, since it causes the relative movement between the sample and the indenter, so that this movement must be as straight as possible. A linear guide with numerical control was adopted. This control allows variations of speed during the test and also allows the positioning of the indenter on a specific area of the sample.

The axial loading system, on the Z-axis, also consists of an automatic linear guide, which moves the indenter and sensor group. Vertical positioning allows using samples of 50 mm or less. A closed command circuit compares the instant axial load furnished by the piezoelectric sensor with the programmed one so that the correct load tends to be maintained throughout the sample surface even at slight undulations. Either constant or variable axial loadings can be applied. The latter are useful for detecting the critical load condition in which the rupture mode changes from ductile to brittle. Brittle scratches remove more surface material: therefore, the higher the transition load, the higher will presumably be the durability.

The equipment is schematically illustrated in Figure 3. It shows many details: the sample holder, the two movement directions travelled by the indenter during a test, the circuit of the electric signal emitted by the sensors to the microcomputer, including the amplifiers and control interface.

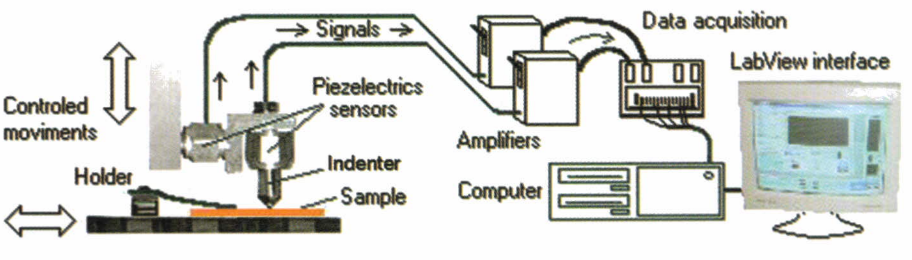

Figure 3. Schematic illustration of the sclerometric apparatus

A precision cramp device was made in order to hold the same indenters used in the Vickers hardness tests, which are commercially available. The signals from the piezoelectric sensors are processed by software with a control interface with a 1.25 MHz acquisition rate. The software controls the movements of the linear guides, paying special attention to the control of the linear guides for the positioning and loading device. LabView was chosen in view of its user-friendly interface. A control interface was thus developed in order to conduct all the foregoing actions, in addition to documenting the tests. For each test, a program option helps the user to follow the several steps until the end of the test. The interface can be seen in Figure 4.

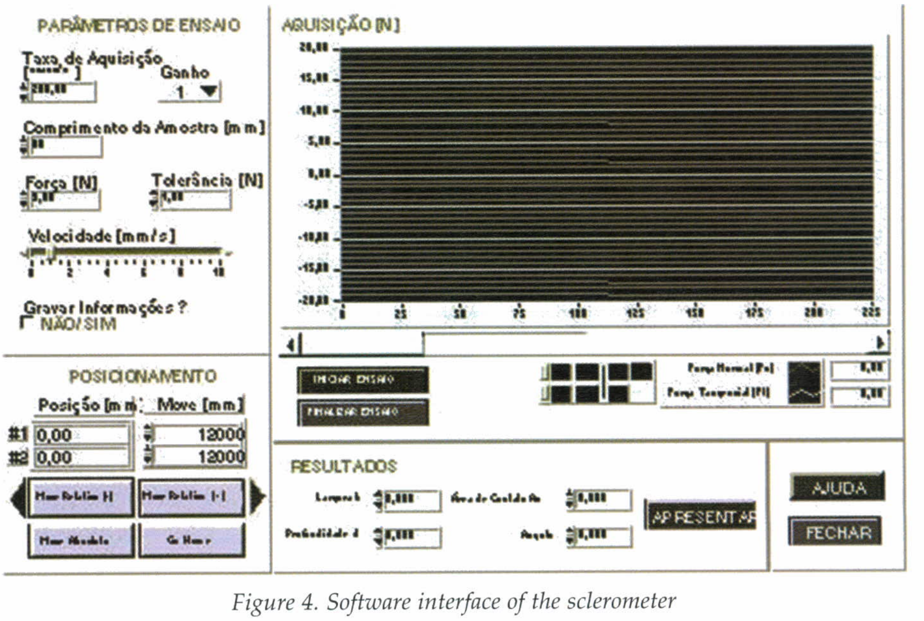

Figure 4. Software interface of the sclerometer

The following fields may be noted: control of the acquisition rate, control of the amplifiers, length of the scratch, axial force (normal) to be used in the test and its tolerance, option of saving the test, control of guide positioning, and finally, graphics of the forces monitored during the test. Moreover, auxiliary tools for visualizing the information on the graphics are also available, as well as fields for extra information about the scratches, and buttons to run the tests and request help.

4. EXPERIMENTAL

In order to highlight the potential of the sclerometric technique, the following types of tiles with known PEI or Mohs results were subjected to sclerometric tests: a porcelainized stoneware tile, a single-fired floor tile, two single-fired wall tiles, and a glass-ceramic tile. Table I lists these sample.

Sample	Mohs hardness	PEI Class
1 Porcelainized stoneware	5	<150 mm^3
1 Single-fired floor tile	5	4
2 Single-fired wall tiles	4	4
1 Glass-ceramic	6	6

Table 1. Description of the samples

The two single-fired wall tiles were actually different types, but had the same classification either in the Mohs or PEI test. They were tested in order to verify any difference regarding their abrasive behaviour when using the sclerometer. The glass-ceramic tile was the product of a recent research[5] accomplished at the Laboratory of Materials. All the others samples came from commercial tiles of the company Cecrisa S.A.

It is not necessary here to present correlations regarding these wear characterization tests. In fact, studies[4-5] have already reported the good correlations between PEI and scratch hardness for several floor tiles. The intention here is to show, however, that the latter is considerably more sensitive and accurate.

For the width of the scratch, the average was taken of at least eight measurements obtained at ductile points chosen at random and observed by scanning electron microscopy. The averages of the normal and tangential forces were automatically calculated, computing all the values furnished by the corresponding sensor during the test.

All the samples were equally tested using constant loading of 0.5 newton, scratching speed of 0.5 mm/s and length of scratch of 6 mm. In the glass-ceramic sample, an extra test with 1.0 N was performed to verify the influence of this test parameter. In addition, an increasing loading test was also carried out, from 0.5 N to 2.5 N, on the glass-ceramic, in order to determine the critical load of the ductile/brittle transition. Table 2 summarizes the measurements and the calculations deriving from a sclerometric test. The geometric parameters K for scratch hardness and specific energy were 4000 and 14278, respectively.

Measures [units]	Description	Evaluation
F_n [N]	Normal load	Average value
F_t [N]	Tangential load	Average value
μ^* [-]	Friction coefficient	$\mu^*=F_t/F_n$
L [μm]	Width of the scratch	Average value using micrographs of the scratch
H_r [KPa]	Scratch hardness	$H_r = 4000F_n/L^2$
e [KPa]	Specific energy	$e = 14278F_t/L^2$

Table 2. Details of the measured properties

The small value of normal loading was used to assure ductile scratches, which are necessary to make the correct width measurements of the scratch.

5. RESULTS AND DISCUSSION

Table 3 presents the sclerometric results. The two last columns should be noted. As expected, the greater values of scratch hardness were achieved for the glass-ceramic, and these values were in agreement with those from the literature[5]. The difference in the specific energy for different levels of force is well known. The mechanism involved is not well understood yet, but is generally attributed to the scale factor[4]. The discussion concerning the other types of tile is presented along with the corresponding micrograph.

Kind of tile	F_n [N]	F_t [N]	μ [N/N]	L [μm]	Hr [GPa]	e [GPa]
Glass-ceramic - 0.5 N	0,9	0,1	0,1	16,6	12,5	5,9
Glass-ceramic - 1.0 N	1,4	0,2	0,1	21,0	12,6	4,9
Porcelainized stoneware - 0.5 N	0,7	~ 0,0	0,1	15,8	11,4	2,8
Single-fired wall tile - 0.5 N	0,8	0,1	0,1	18,2	9,4	3,9
Single-fired wall tile - 0.5 N	0,6	0,1	0,1	15,9	9,8	3,2
Single-fired floor tile - 0.5 N	0,6	0,1	0,2	14,0	11,6	8,7

Table 3. Sclerometric results

As may be observed, although 0.5 N and 1.0 N were stated as nominal loading, higher levels of force were used. This was caused by computer restrictions, whose performance is about to be updated so that a higher data acquisition rate could be adopted and closer real loads are expected.

Figure 5 refers to the porcelainized stoneware tile. Different phases are noted throughout the depth of the scratch, and all of them accounted for the scratch hardness. In such materials, the definition of scratch boundaries is often not sharp, even with the aid of a scanning electron microprobe. However, in ductile scratching, reliable measurements can be made, and image analysis can be used in order to increase the accuracy. The real limitation is the measurement at brittle regions.

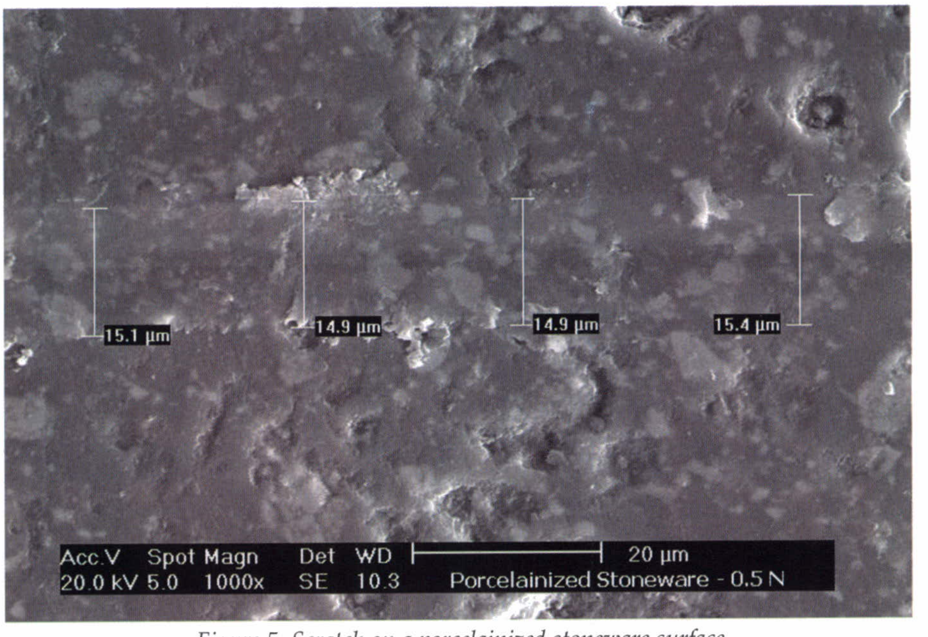

Figure 5. Scratch on a porcelainized stoneware surface

An example of homogeneous material is seen in Figure 6. The ductility achieved was assured by the impression of the indenter apex at the beginning of the scratch, where no cracking occurred. One should highlight the flatness of the sample, which enhances great reliability in either H_r or e values.

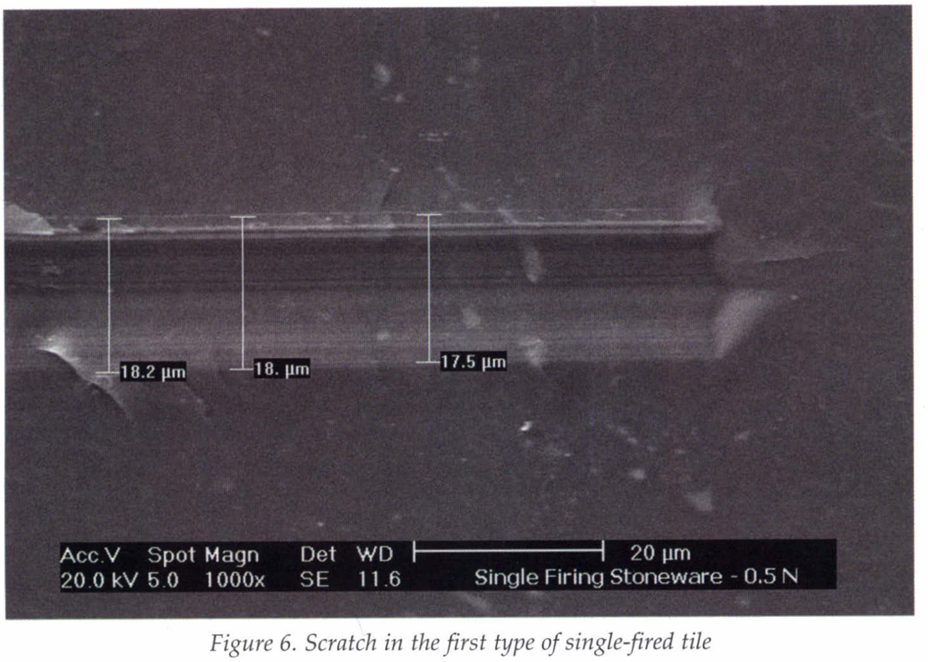

Figure 6. Scratch in the first type of single-fired tile

The second single-fired tile tested had quite an undulated heterogeneous surface, as can be seen in Figure 7. This also holds for the single-fired floor tile surface presented in Figure 8. In such cases, a very fast response circuit with regard to the indenter positioning is needed in order to maintain the programmed load. The current equipment was not able to do that, and loads were maintained only in an average sense, resulting in a broad variation of applied force. This loading variation causes scratches with a few islands of ductile behaviour between many brittle ones. Therefore, in the force measurements in the second single-fired and stoneware floor tile tests, only the ductile lengths were considered. The mapping of such ductile regions was made using micrographs of the whole scratch. The results suggest slightly higher scratch hardness for the tile with undulated surface.

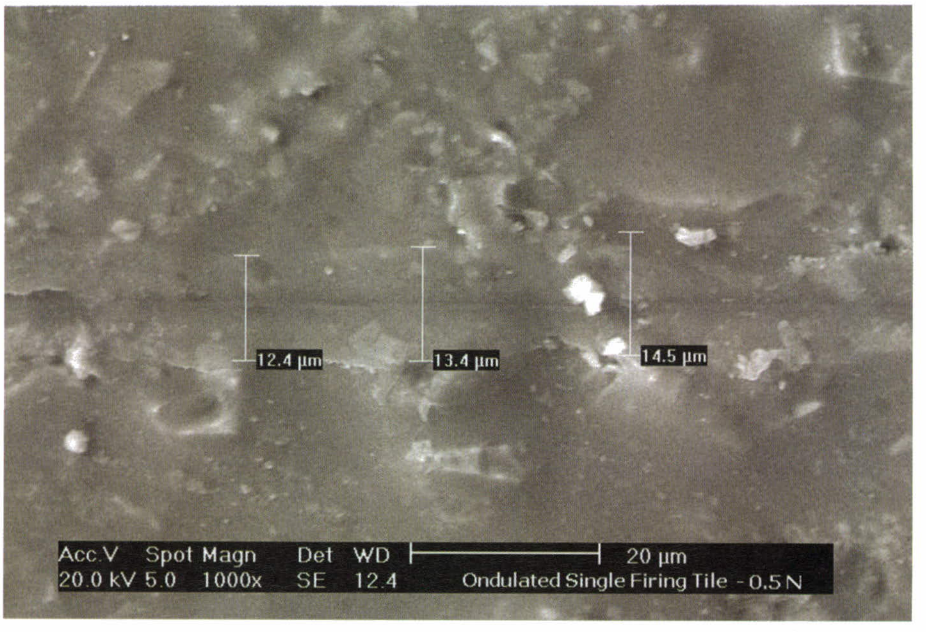

Figure 7. Scratch in the second type of single-fired wall tile

Particles encountered in the indenter path, as observed in Figures 7 and 8, also hindered the force measurement. Besides, such particles tend to increase the scratch hardness of these materials. The chemical composition of the particles present, determined by the EDX technique, revealed the presence of aluminium and zircon, which likely had accounted for the high values obtained for H_r, due to the hardness of alumina and zircon silicates.

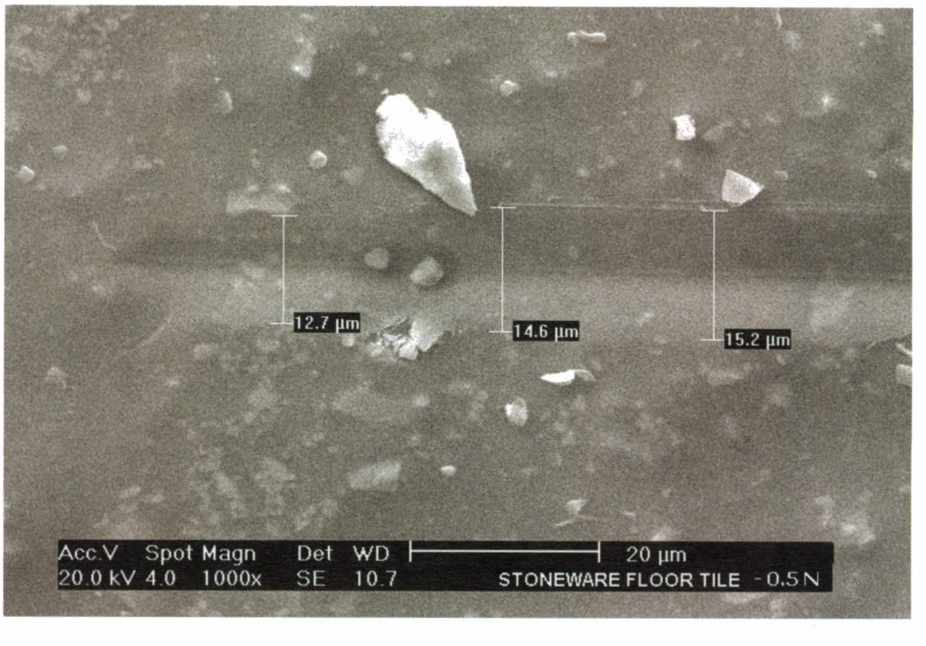

Figure 8. Scratch in the stoneware floor tile

The scratch formed in the glass-ceramic tile using 0.5 N and 1.0 N is shown in Figure 9 (a) and (b) respectively, with the same magnification (1000x). Both scratch hardness and specific energy results have already been discussed.

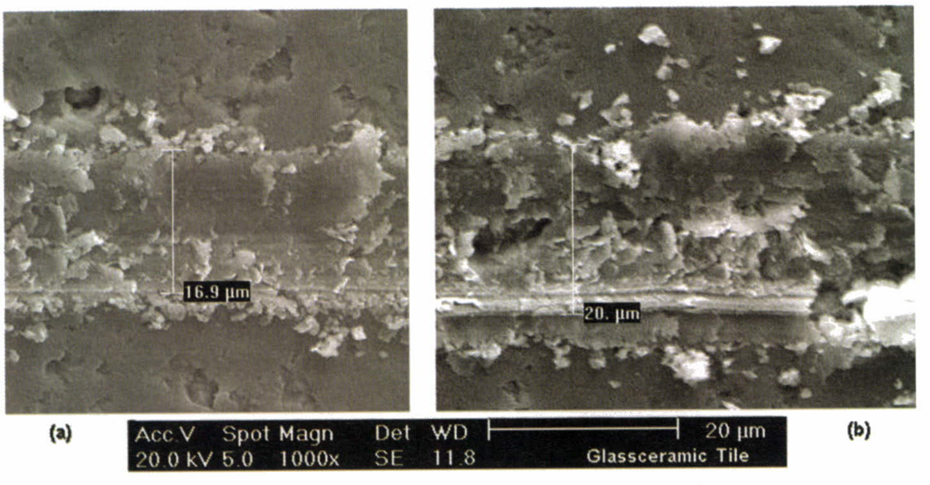

Figure 9. Scratch in the glass-ceramic tile using: (a) 0.5 N and (b) 1.0 N

It can be observed that the scratch corresponding to 1.0 N has a slightly higher fissure concentration along the scratch, which could suggest an approximation to the critical load condition. This phenomenon is presented in Figure 10, while critical load is determined with the graph shown in Figure 11.

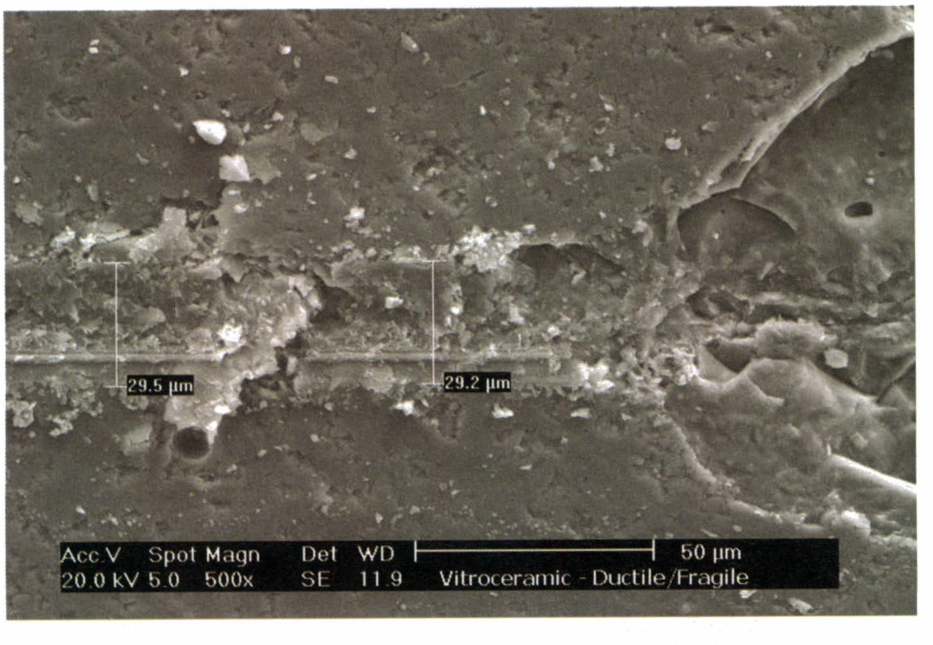

Figure 10. Ductile/brittle transition of the scratch in the glass-ceramic tile

A ductile behaviour is observed until the vicinity of the crater. As soon as the load reaches a critical value, depending on several factors mentioned above, a great amount of material was suddenly removed from the material surface, characterizing the ductile/brittle transition of the material. Besides, the occurrence of the transition can also be clearly identified in the normal force diagram. In this case, the pronounced kink in Figure 11 indicates that the critical load is about 2.5 N (in Y axis).

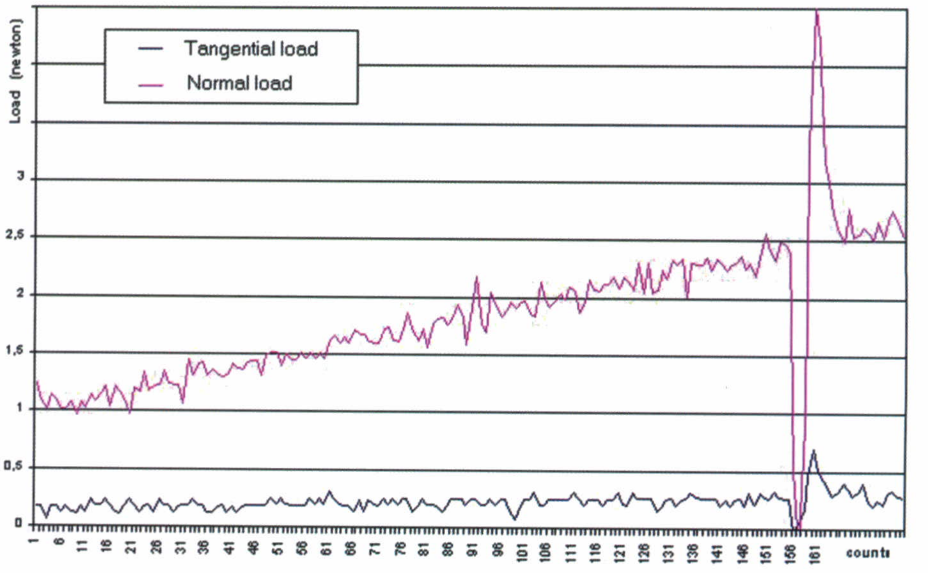

Figure 11. Identification of the critical load for scratch transition of the glass-ceramic tile

One should note that the tangential force is kept during the whole test, even with increments of normal loading. This finding agrees with the previous conclusions that specific energy decreases as the amount of removed material increases, since this volume increases with the width of scratch, which in turn increases with normal force.

6. CONCLUSIONS

Some advantages and limitations of this technique regarding brittle materials are discussed. The Federal University of Santa Catarina can count on a useful apparatus for studying abrasive phenomena, despite the system being in an early stage, while the tile industries have an alternative tool for the characterization of wear resistance. In addition, an effective contribution to the classification criteria for the durability of ceramic tiles can be expected.

However, many improvements must be implemented to assure reliability of the apparatus, or even to increase its performance. Besides, ceramic tiles are not that easy to test. Very small normal loading, often less then 0.5 N, should be used to avoid brittle behaviour. The values of scratch hardness and specific energy are in the region close to 10 GPa.

Scratching a homogeneous and flat surface results in a sharp continuous scratch, which makes the width measurement easier. On the other hand, measurements on surfaces with many phases and embedded particles, or with undulations, represent a truly hard task, requiring extra time during the image analysis, and higher performance equipment.

The study shows moreover that scanning electron microscopy is very useful, not only as a measurement system but also to identify the scratch mechanism, and map the forces at the different phases present.

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