Anton Paar

Advanced Mechanical Surface Testing

Characterization of the scratch resistance of ceramic tiles

Introduction

A novel approach to testing of scratch resistance of ceramic tiles and understanding of the mechanisms of material failure and removal is presented. The approach is based on a scratch test method that is routinely used in other industrial domains. The main reason for application of the scratch method for testing of ceramic tiles was replacement of the outdated 'EN 1071 Ceramic tiles - Determination of scratch hardness of surface according to Mohs hardness scale' standard, which proved to be too inaccurate and user subjective when applied to new advanced materials.

This Application Report reports on the experimental methods, testing conditions and results on both glazed and unglazed ceramic tiles.

A comprehensive test matrix was performed with the objective of proposing a replacement to the EN 1071 standard. The parameters of the presented test method were partially adapted from the 'ISO 20502 Standard Fine ceramics – Determination of adhesion of ceramic coatings by Scratch testing' [1].

Advantages of scratch tests on bulk materials

Though the scratch test technique is often used for characterization of adhesion of surface coatings, it can also be used for characterization of bulk materials. In such cases, the scratch resistance is usually obtained rather than adhesion. The scratch tests are often used to study the mechanisms that take place during abrasion or wear. Further, the scratch test offers a unique possibility of studying the effect of single particle abrasion, which is difficult or impossible to simulate by other abrasion or wear tests. Information such as elastic-plastic deformations, formation of median, radial and lateral cracks and material failure mechanisms occurring during scratch testing can be revealed during a scratch test. This information can substantially contribute to an understanding of the mechanisms involved in sliding wear and abrasion of brittle materials. Scratch testing is therefore an ideal candidate for simple and efficient evaluation of scratch resistance of various materials, including ceramic tiles.

Ceramic tiles, apart from their decorative aspects, are often used as protection against severe wear and abrasion. As such, they must exhibit surfaces with very high scratch and abrasion resistance. Previous studies [2, 3] showed that chipping and cracking of the tiles strongly decreases their wear resistance and negatively affects aesthetic properties (loss of brightness, discoloration effects, etc.). Besides, the presence of cracks and open voids leads to increase of surface roughness, which favors inlay of dust and dirt thus further decreasing the aesthetic aspects.

Experimental setup

Three types of stoneware tiles with different microstructure and surface finish were chosen for this study. The samples were divided into two groups:

- stoneware glazed tiles with different degrees of crysta linity,

- stoneware tiles with either as fired or polished surfaces.

Table 1 shows a summary of the stoneware samples with their main features. Stoneware tiles without glaze were used either with the as fired surface (group A) or polished surface (group B).

Sample	Color	Surface finish	Degree of crystallinity	Hardness Hit [MPa]
T1	yellow	glaze	high	7944
T2	black	glaze	low	8604
T3	white	glaze	high	9488
T4	brown	glaze	low	9416
T5	red	glaze	very low	7621

Table 1 – List of glazed tiles used in the study.

The surface characteristics were measured by surface profilometry and the Ra and RM parameters (according to EN 623-4) were determined. Hardness of the samples was measured using a Anton Paar Micro Indentation Tester (MHT) equipped with a diamond Vickers indenter. The indentation hardness was measured at a maximum load of 1 N. The hardness results for the glazed samples are shown in Table 1. The hardness of samples in groups A and B varied between 7230 MPa and 10120 MPa while no straightforward influence of the polishing on the hardness was found.

The phase composition of the glaze layers was determined by X-ray diffraction (XRD) analysis on the glazed surfaces. The XRD analysis allowed qualitative comparison of the degree of crystallization of each sample, a quantitative analysis being impossible due to the lack of suitable standards.

The scratch tests were performed on the top surface of the sample with an Anton Paar Revetest Scratch Tester with normal force range of 1 N to 200 N equipped with a Rockwell C diamond indenter of 200 μm radius. Progressive load scratching mode was used in all experiments. A summary of the scratch parameters is given in Table 2.

Prescan / Postscan procedure and Panorama imaging

The scratch tests were performed with the Prescan/Postscan procedure, which consists of three stages: first, the surface is scanned by the tip with minimal load. Secondly, the scratch test is made, during which the penetration depth (Pd) is recorded. Thirdly, another surface scan is made along the scratch track to reveal the true residual depth (Rd). In addition, for archive purposes and for a systematic analysis, panoramic imaging was used. The Panorama imaging consists of recording the entire scratch image, which is synchronized to all recorded signals and is saved with the data. One can recall the saved scratch results including the Panorama image and work with them as with a real scratch. An example of such Panorama image is shown on Fig.4

Minimal load	Maximal load	Scratch length	Scratch speed	Indenter radius
[N]	[N]	[mm]	[mm/min]	[μm]
1	80	8	10	200

Table 2 – Scratch test parameters.

Critical load determination

Loads corresponding to sudden events during the scratch are called critical loads (Lc) and are used for characterization of the scratch resistance of the material. The Revetest Scratch Tester allows direct determination of critical loads by using the implemented optical microscope and video software, which is synchronized with the scratch track. In our study, the scratch area corresponding to the critical load was subsequently inspected by Scanning Electron Microscopy (SEM) for detailed examination of the failure mechanisms.

The output of the progressive load scratch tests on all tested tiles were two critical loads Lc1 and Lc2, defined as follows:

- Lc1: the load at which the first cracking occurs (Hertzian cracks),
- Lc2 the load at which catastrophic failure begins.

Critical load values for each sample were calculated as an average from at least three tests made in different areas. Figure 1 shows typical morphology of areas corresponding to critical loads Lc1 and Lc2 on a polished sample.

Critical load Lc1: At loads below Lc1 the generated stressesresulted in tensile stresses which were not high enough to induce crack formation. At such loads the sample remained fully compact without showing signs of irreversible damage. At the first critical load the stresses were sufficiently high to initiatecracking of the material.

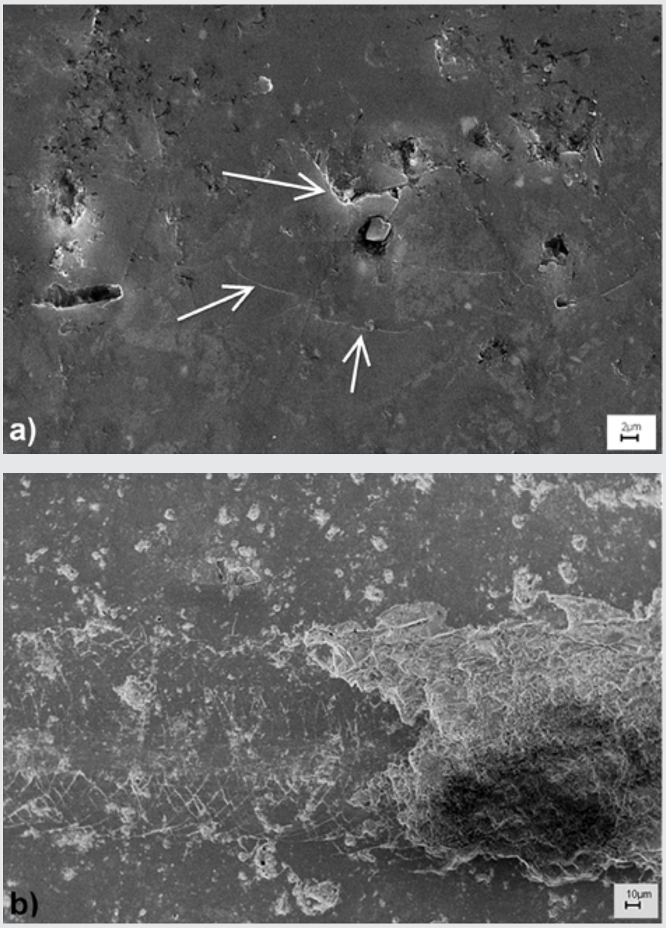


Fig 1 – Typical morphology of the first initiation of the Hertzian crack just before Lc1 (a). Catastrophic failure area corresponding to Lc2 (b). Both SEM images were taken on a polished sample.

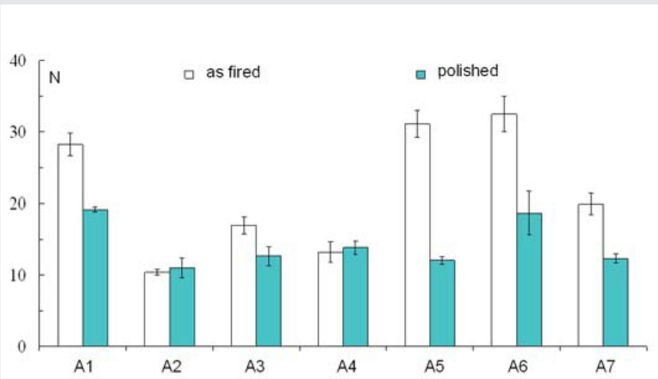
At loads between the first and the second critical load, the stresses in the material were increasing: cracking continued and the density of the cracks increased. Until reaching the second critical load however, no other failure mechanisms were observed.

Critical load Lc2: At the second critical load, the stresses in the vicinity of the indenter were high enough to cause spalling and chipping of the material and the catastrophic failure occurred. This failure mechanism and the corresponding morphology were then observed up to the end of the scratch. Similar failure behavior was observed on all tested samples, whether glazed, as fired or polished. However, the values of both critical loads were different for each group of samples. It was therefore possible to classify the tested ceramics by the critical load value obtained on the given sample. The critical load value was then completed by optical and SEM fractographic analysis.

Difference in scratch resistance of as fired and polished samples

One area of the unglazed stoneware samples was polished to evaluate the effect of polishing on the scratch resistance. The polishing decreased the surface roughness (Ra) from around 2.4 µm down to ~ 0.15 µm. Though significantly increasing the decorative properties of the tiles (colouring and gloss), polishing is known to negatively affect the wear and abrasion resistance. The scratch tests were therefore performed to find out whether such a trend can be detected by this method. The charts in Fig. 2 show the difference between the polished and the as fired samples for both first critical load (Fig. 2a) and second critical load (Fig. 2b).

There was an obvious difference between the scratch behavior for the first and the second critical load: while polishing had an important effect on most of the first critical loads, the second critical load remained practically unchanged. In most cases, the polishing led to a decrease of the first critical load, i.e. a decrease in scratch resistance. This is in good agreement with industrial experience. On the other hand, the Lc2 was unaffected by the surface treatment which indicates that the polishing procedure creates defects to only certain depth (scratch depth at Lc1). Above this depth the material remains unchanged and the critical load is very close for both polished and as-fired samples. This result confirms the utility of the scratch test for such materials and allows fast evaluation of the effects of surface treatment.



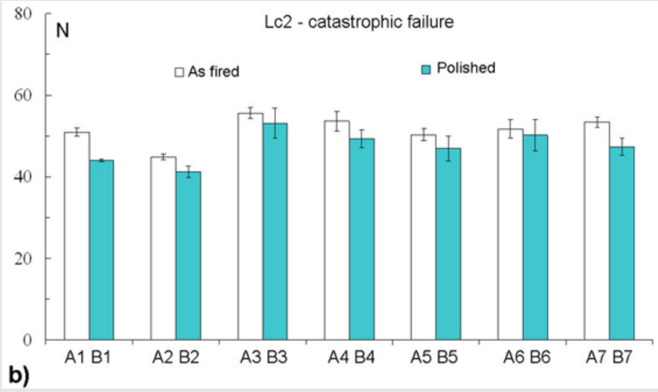


Fig 2 – Comparison of the first critical load (a) and the second critical load for the as fired and polished stoneware samples.

Glazed tiles: effect of the degree of crystallinity on the scratch resistance

The group of glazed tiles contained materials with different degrees of crystallinity (see also Tab. 1):

- 1 and T3 - high degree of crystallinity,
- T2 and T4 - low degree of crystallinity,
- T5 almost entirely amorphous.

The criteria for critical load determination were identical to those for the stoneware samples: Lc1 corresponded to the appearance of the first Hertzian cracks, Lc2 corresponded to the onset of catastrophic failure. The results of the progressive load scratch tests are summarized in Fig. 3. The scratch results show a very strong relationship between the degree of crystallinity and the second critical load.

While the difference between the Lc1 for the more crystalline and for the less crystalline samples is less pronounced, the difference between the Lc2 for the same samples is obvious. Samples with a high degree of crystallinity (T1 and T3) exhibited a higher second critical load, i.e. higher resistance to scratching. Samples with a lower degree of crystallinity (T2 and T4) showed the second critical load almost 50 % lower than the samples with a high degree of crystallinity. The amorphous T5 sample showed the lowest values for the first and second critical load out of all tested samples. Optical analysis and Panorama image was indispensable for critical load determination in all cases. An example of a Panorama image with the two critical loads is shown on Fig. 4. However, some of the recorded signals were well correlated with the optical observations: the drop in residual depth (Rd) corresponded to the second critical load. A typical example of a residual depth plot is shown in Fig. 5, where arrows point to the drop of the Rd signal.

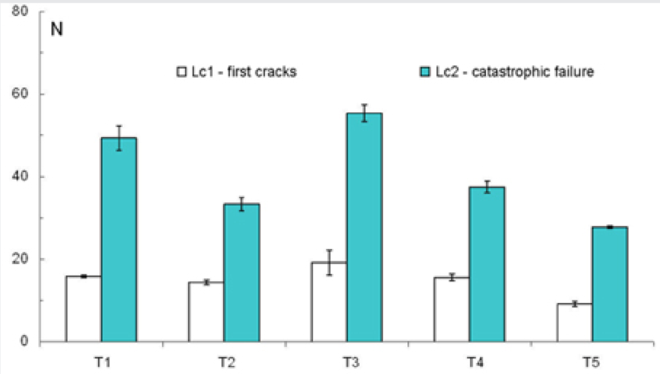


Fig 3 – Comparison of the first and the second critical load for the glazed samples. Samples T1 and T3 contained a high fraction of crystalline phase, samples T2 and T4 a low fraction of crystalline phase. Sample T5 was almost entirely amorphous.

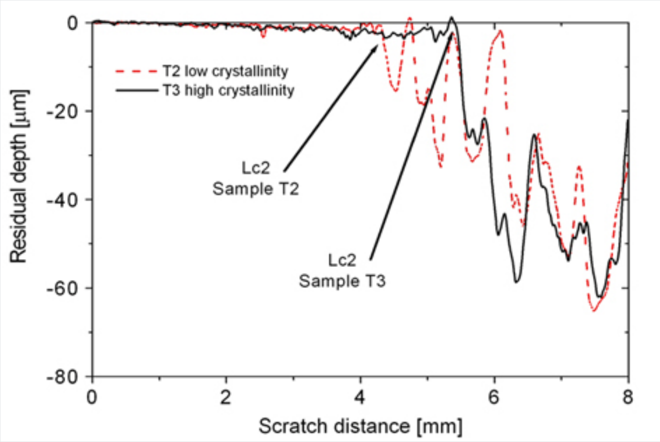


Fig 5 – Typical record of the residual scratch depth (Rd) for a highly crystalline sample (T3) and a less crystalline sample (T2). Arrows indicate drop in the Rd signal which corresponds to Lc2.

As further analysis by the SEM showed, the samples with a high degree of crystallinity contained crystals elongated along one axis, which effectively blocked crack propagation thus resulting in a higher value of Lc2 and thus better resistance to scratch failure.

Conclusions

The presented Application Bulletin presents a novel method for characterization of the scratch resistance of ceramic tiles. The method is based on the progressive load scratch test method with progressive load increase and it allows a fast and efficient determination of the scratch resistance of various types of tiles. The scratch test method clearly proves that the critical load depends on the degree of crystallinity of the material and on the surface finish:

- polishing leads to a decrease in the resistance of the tiles due to the formation of surface defects,

- an increase of the degree of crystallinity leads to an increase in the scratch resistance.

The progressive load scratch test method with diamond indenter proved to be a far superior method to the EN 101 standard with much higher repeatability and robustness.

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References

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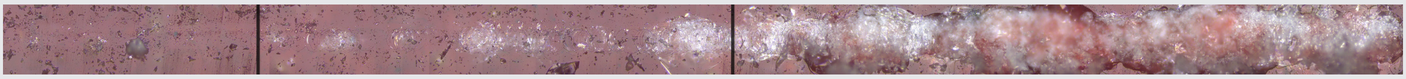

Lc1 - first Hertzian cracks   Lc1 - catastrophic failure

Fig. 4 – Panorama image of a scratch on sample T5. Vertical lines indicate areas corresponding to the critical loads.