Porcelain tile microstructure: implications for polishability

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Abstract

The present study examines the influence of porcelain tile microstructure on tile polishability and polishing efficiency. A range of different fired porcelain tile microstructures was obtained by varying the quartz particle size and content in the starting composition, and the peak firing temperature.

The polishability of these porcelain tile specimens was evaluated with a tribometer designed to simulate the industrial polishing operation, using cement-matrix silicon carbide abrasive tools. The wear rates of both the tile samples and the abrasive tools were measured. A new parameter, termed 'polishing efficiency', was defined as the ratio of tile specific wear rate to pin specific wear rate.

Quartz particle size and content in the starting composition, as well as peak firing temperature, strongly affected the fired tile microstructure, and hence the polishability. When the firing temperature lies below the optimum value (at which porosity is minimised) the polishing efficiency increases, while the polished surface quality decreases (i.e. porosity rises). Quartz particle size and content should be minimised in the starting composition to achieve maximum polished surface quality and polishing efficiency.
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1. Introduction

The high gloss and hardness of polished porcelain tiles, which far exceed those of most commercially-available natural stones, have led to strong growth in this product's market share.[1] Porcelain tile polishing is performed by the action of grinding tools on the tile surface. In this process, long polishing lines are used in which the tile thickness is reduced by up to 10% to produce a flat, smooth, final product with high optical reflectivity. This so-called polishing process actually consists of several operations. In the first process phase (levelling), tools with coarse abrasive grains (usually diamond) are used to dress and level the tile surface, removing a large amount of material. The grinding tools used in the following phase (the actual polishing steps) contain abrasives with progressively decreasing grain sizes to produce the desired final surface gloss. The main abrasive used in the polishing steps is silicon carbide, embedded in a magnesia-cement matrix.

Porcelain tiles consist of a glassy feldspathic matrix, containing disperse crystalline phases such as quartz and mullite. Mullite is typically found in low quantities (<7–8%) because of the fast firing cycle used in porcelain-tile manufacture.[2] The major crystalline phase is quartz. More than 70% of the starting quartz remains undissolved because of the fast firing cycle. The quartz content in the fired body can therefore amount to 30 wt.% or more, with a maximum particle size generally below 45 μm as result of the milling process applied to the raw materials mixture. It is consequently not surprising that porcelain tile microstructure and mechanical properties are largely defined by its quartz content.[3]

Porcelain tile polishing has received little scientific attention to date, despite the product's commercial significance. Some previous studies have focused on the evolution of the tile surface during the industrial polishing operation.[4,5] A recent study[6] analysed the influence of porcelain tile

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microstructure on tile behaviour during polishing, in which the wear rate was assessed using a tribometer with a pin-on-disc configuration. However, this configuration fails to reproduce typical industrial polishing conditions.

The authors have recently reported[7] the design and initial tests of a laboratory-scale tribometer which more accurately simulates the development of surface finish (roughness and optical gloss) in the industrial polishing process for porcelain tiles. This method has been used to investigate the influence of contact load and abrasive particle size, and it has been shown that the development of both roughness and gloss with polishing time is well described by quantitative empirical models involving a simple exponential function.[8]

The work described in the present paper has used the same laboratory-scale experimental method to analyse the influence of tile microstructure on polishability, evaluated in terms of specific wear rate. The variation in microstructure was established in two ways: by modifying the peak firing temperature, and by formulating compositions with different quartz particle size and content. The wear rate of the abrasive tools was also determined. This parameter, together with the tile wear rate, has allowed a new quantity, termed 'polishing efficiency', to be successfully defined and evaluated.

2. Experimental method and materials

2.1. Materials

Two types of compositions have been used. The first type consisted of industrial spray-dried powder used in porcelain tile manufacture, formulated from a mixture of ball clays, quartz and sodium feldspar. This type of composition was used to study the influence of peak firing temperature. The second type, which actually comprised a set of compositions containing the same raw materials, was developed in the laboratory due to the difficulty of obtaining industrial spray-dried powder with the desired variations in some of the compositional ingredients. In the second type of composition furthermore, quartz particle size and content were varied across (and beyond) the range found in industrial porcelain tile practice.

Table 1 details the porcelain tile compositions that were prepared (designated A–E) in the laboratory, together with their ingredients, expressed as a percentage by weight. Taking composition C (a standard porcelain tile composition) as a

Table 1
Compositions (wt.%) of the porcelain tile samples with different quartz grit 12 contents

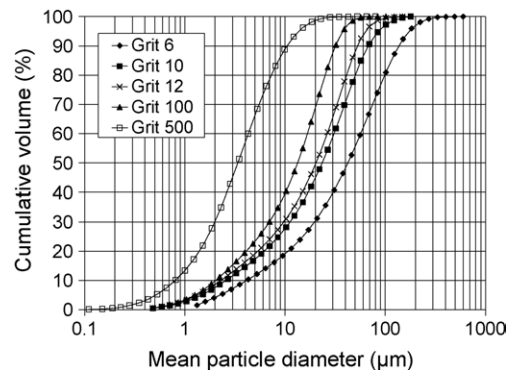| Raw material	A	B	C	D	E
Clay	35	32.8	30.6	26.2	17.5
Feldspar	55	51.6	48.1	41.3	27.5
Talc	10	9.4	8.7	7.5	5
Quartz grit 12	0	6.25	12.5	25	50

Fig. 1. Particle size distributions for the five sizes of quartz particles (grit 6 to grit 500).

basis, quartz grit 12 (i.e. nominal mesh size 12) was replaced by quartz samples of the same chemical composition, but with a different particle-size distribution (PSD): two quartz samples with a larger particle size (grit 6 and grit 10) and two quartz samples with a smaller particle size (grit 100 and grit 500). These yielded compositions F, G, H and I, respectively. As in sample C, these compositions all contained 12.5 wt.% quartz. Fig. 1 shows the PSD curves of the five quartz samples used.

2.2. Specimen preparation and characterisation

2.2.1. Pressing and firing

Each porcelain tile composition was used to form cylindrical test specimens (about 0.7 cm thick and 5 cm in diameter) and prism-shaped test specimens (80 mm long, 20 mm wide and about 7 mm thick) by uniaxial pressing at a pressing moisture content of 5.5% (dry weight basis) and pressing pressure of $350 \, kg \, cm^{-2}$. After drying, the specimens were fired in an electric laboratory kiln with a fast firing cycle and 6 min hold at each respective maximum densification temperature. The maximum densification temperature had been previously established from the composition vitrification diagram, constructed after determining the fired bulk density.[9]

In the experiments aimed at establishing the influence of peak firing temperature, in which industrial spray-dried powder was used as the starting composition, temperatures were tested across and beyond the typical range of peak firing temperatures used in industry (1180–1220 °C). The test temperatures were as follows: 1140, 1160, 1180, 1200, 1220, 1240 and 1260 °C. The heating rate was $25 \,^{\circ}C \, min^{-1}$ in every case.

To study the sample microstructures, specimen cross-sections were observed by optical microscopy (Olympus BX-60) and SEM (Philips XL30). Bulk porosity was determined with image analysis software (Olympus MicroImage) coupled to the optical microscope. Eighty images with a magnification of 20× were obtained in bright field mode to enhance the pore contrast. The image analysis software calculated the area occupied by the pores, as well as the pore size distribution.
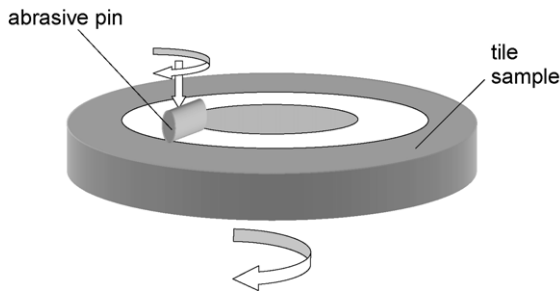
Fig. 2. Schematic diagram showing the relative motion between the upper abrasive pin and the lower tile sample.

Table 2
Experimental conditions used in the tribometer tests

Load/contact length	1.7 N mm^{-1}
Rotational speed of tile	300 rpm
Rotational speed of abrasive pin	150 rpm
Polishing time (s)	15 → 30 → 60
Abrasives (standard industrial abrasive)	#60, #600, #1000 (FEPA nominal grit numbers)
Recorded data (tile specimen)	Wear scar depth
Recorded data (abrasive specimen)	Wear scar width

2.2.2. Tribometer tests

The laboratory-scale polishing rig was designed to simulate industrial polishing conditions as closely as possible. A full description of the apparatus and the experimental conditions can be found elsewhere.[7]

The apparatus is based on an automatic metallographic polishing machine with sample drive head (Struers Roto-Force 3 and RotoPol 35). Abrasive pins, made from the same cement-based composite material as the industrial polishing tools, were mounted in the upper, rotating metallographic sample holder, and porcelain tile (discs) samples were mounted on the lower, rotating disc normally used for the metallographic polishing cloth. Cylindrical abrasive pins, 12 mm in diameter and 10 mm long, were produced from an SiC-cement composite with specifications conforming to nor-

mal industrial practice by Abrasivos de Castellón, Castellón, Spain. A single abrasive pin was used for each test. Fig. 2 shows a schematic diagram of the relative motion between the abrasive pin and the tile sample.

The abrasive pin was mounted with its axial axis parallel to the plane of the tile as shown in Fig. 2, and rotated about a vertical axis in the motorised head of the tribometer. The pin was pressed against the tile surface under a pneumatically controlled normal load. The tile sample, fixed to the lower disc, also rotated about a vertical axis. The inner radius of the annular polishing track was 23 mm.

Material removal from the tiles was assessed in terms of the depth of the centre of the annular wear scar relative to the neighbouring unworn surface, measured with a stylus profilometer (Rank Taylor Hobson Talysurf 10). The wear of the abrasive sample was calculated by measuring its wear scar width. Table 2 summarises the experimental conditions used in the tests performed with the tribometer.
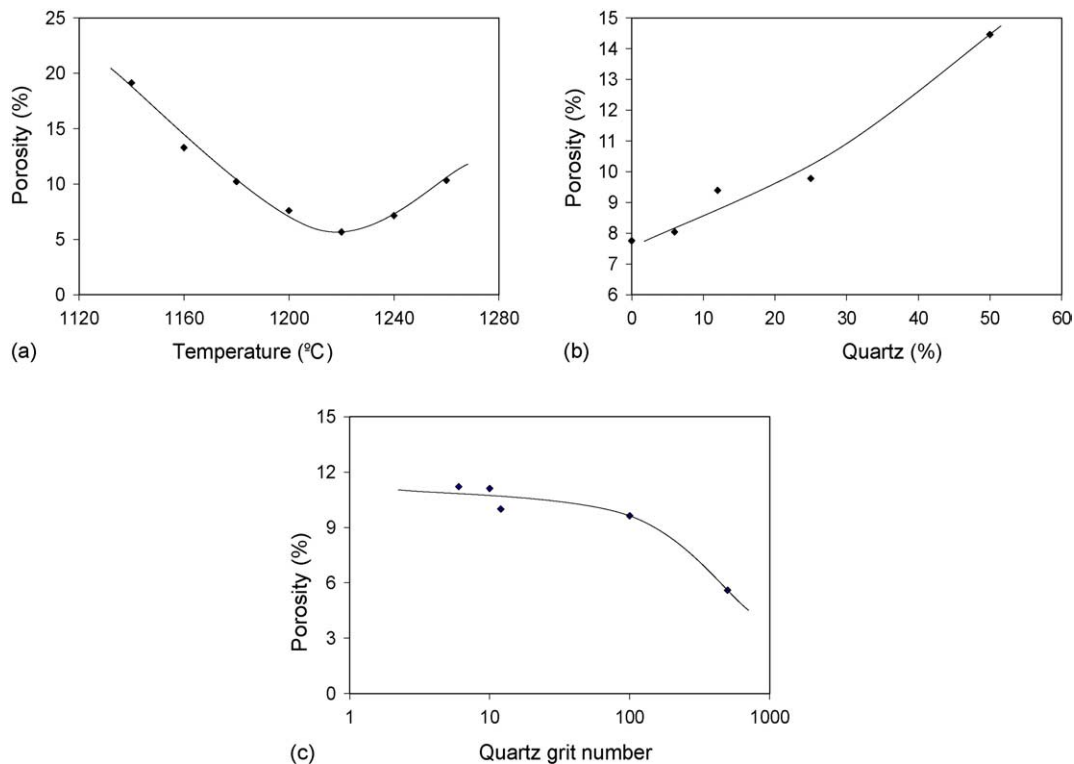

Fig. 3. Variation of tile specimen porosity with: (a) firing temperature, (b) quartz content in the starting composition and (c) quartz particle size (expressed as grit number) in the starting composition.

3. Results and discussion

3.1. Influence of firing temperature on porcelain tile microstructure

Fig. 3a shows the variation of porosity with firing temperature T, i.e. the vitrification diagram, for samples obtained from industrial spray-dried powder. The porosity of the fired specimens is observed to minimise at around $1220\,^{\circ}$C (T_{min}), as a result of sintering in the presence of liquid phase, which causes the tile porosity to decrease.[9] However, a further rise in temperature raises the porosity again because of bloating, though the pores are now round and isolated, characteristic of an over-fired condition. Optical micrographs of three specimens fired at $1160\,^{\circ}$C ($T < T_{min}$), $1220\,^{\circ}$C ($T = T_{min}$) and $1240\,^{\circ}$C ($T > T_{min}$) are presented in Fig. 4. They show that at lower temperatures the sample is characterised by an interconnected porous structure, which becomes more compact, with isolated closed pores, at higher firing temperatures.

3.2. Influence of quartz on porcelain tile microstructure

Fig. 3b and c respectively show the effects on porcelain tile porosity of quartz content and quartz particle size in the starting composition. The porosity value for each composition corresponds to the minimum porosity for that composition derived from its vitrification diagram. As reported in the literature,[2] porosity decreases as quartz content in the starting composition is reduced. At maximum densification temperature, the quantity of quartz that dissolves in the glassy phase during firing (and hence the quantity of non-dissolved quartz in the fired tiles) is proportional to the quartz content in the starting composition. Undissolved quartz dramatically reduces the sinterability. For this reason, and for simplicity, we used the starting composition quartz content (instead of the actual quantity in the fired specimens) in the 3b plot, as well as in the following representations.

Few studies are available on the effect of quartz particle size on porcelain tile microstructure.[10,11] Fig. 3c shows that this is similar to that of quartz content. Raising the quartz particle size leads to a larger quantity of undissolved quartz, which lowers body sinterability.[10] However, for the larger particles, above about $30\,\mu$m median size, quartz particle size no longer affects porosity, probably because when the particles are sufficiently large, their dissolution in the glassy phase is hardly affected. This last point was confirmed by quantitative XRD analysis using a calibrating straight line procedure.[2] The quantity of undissolved quartz in the fired specimens of compositions with different quartz particle sizes (compositions F, G, H and I) ranged from 58 to 68%. Note that for the two compositions with the largest quartz particle sizes (F and G) this amount only varied 1%.

The effects of quartz content and particle size on microstructure can be observed in the SEM micrographs in Fig. 5. For simplicity, only two micrographs from each series of experiments are shown: compositions C (12 wt.% quartz) and E (50 wt.% quartz) from the first series, and compositions F (grit 6) and I (grit 500) from the second series. Micrographs 5a and b illustrate the change in microstructure with increasing quartz content. The microstructural alteration leads to a rise in porosity, caused by non-dissolved quartz grains (marked A in Fig. 5b), together with a significant presence of typical circumferential cracks around quartz grains (B in Fig. 5b).[11] Cracking of the quartz grains during cooling of the tile is also observed (C in Fig. 5b).[3] Microstructural observation (Fig. 5a, c and d) confirms the effect of quartz particle size indicated above, as the specimen containing the largest quartz grains (grit 6) displays a heterogeneous microstructure, which contains more large-sized pores and cracks, mainly due to the presence of undissolved quartz particles in the glassy matrix.

3.3. Tribometer tests

3.3.1. Porcelain tile polishability

Material removal from the tiles was assessed in terms of specific wear rate (SWR), i.e. volume removed per unit load per unit sliding distance. The volume removed was calculated from the wear scar depth as discussed in Section 2.2.2. The sliding distance corresponded to a sliding time of 60 s, which was longer than the time required to obtain effectively steady-state roughness in the tribometer tests.[7]

Fig. 6a shows SWR for the tile samples obtained from industrial spray-dried powder, plotted against firing temperature for abrasives #60, #600 and #1000 (nominal silicon carbide grit numbers). The specific wear rate falls strongly with increasing firing temperature (decreasing porosity) to a minimum between $1220\,^{\circ}$C and $1260\,^{\circ}$C, depending on grit size. As expected, the SWR values are much greater for the larger abrasive grit sizes. The SWR values vary little at temperatures
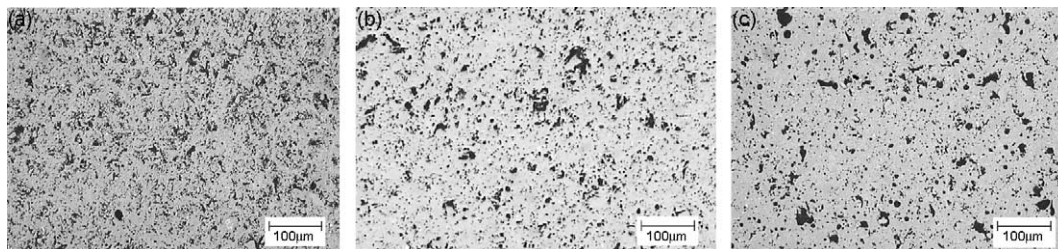

Fig. 4. Optical micrographs of three specimens with industrial powder fired at (a) $1160\,^{\circ}$C, (b) $1220\,^{\circ}$C and (c) $1240\,^{\circ}$C.

(a) **12.5% quartz (grit 12)**

(b) **50% quartz (grit 12)**

(c) **12.5% quartz (grit 6)**
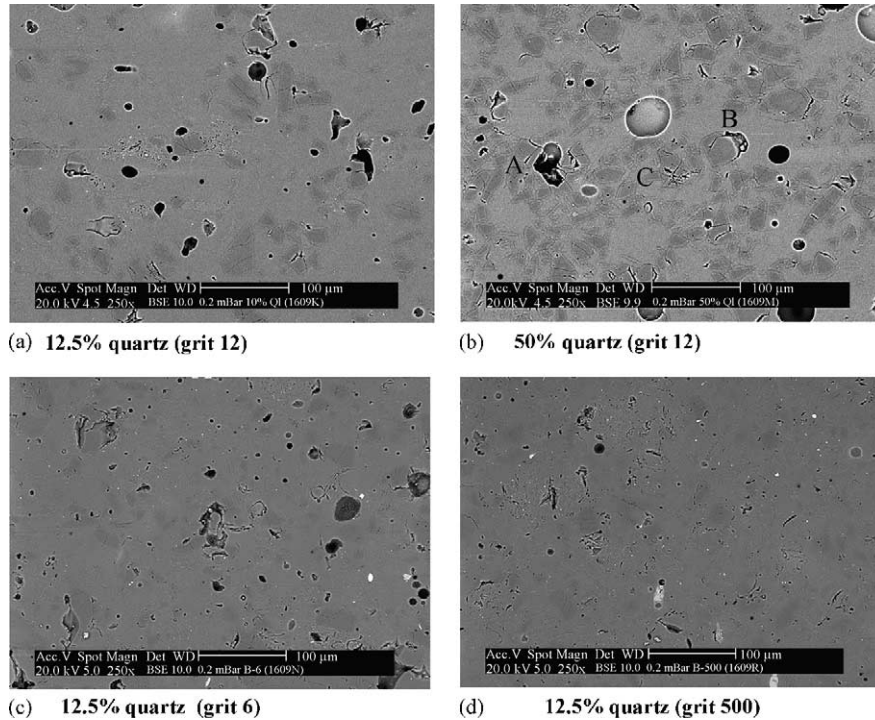
(d) **12.5% quartz (grit 500)**

Fig. 5. Microstructures of samples with different quartz content and quartz particle size in the starting composition.

above that for minimum porosity ($T_{min} = 1220\,°C$) because even though samples may have the same porosity, their microstructure will differ depending on whether it results from under-firing or over-firing. Under-firing gives rise to interconnected open pores, while over-firing produces isolated closed pores that provide the body with a more compact, continuous matrix, and hence greater wear resistance. This effect of porosity on wear rate is consistent with previously published data.[6,12]

Similar analysis of SWR was performed for the two other experimental series. Figs. 7a and 8a, respectively, depict the variation of SWR with quartz content and quartz particle size in the starting composition. A strong increase in SWR can be observed with quartz content for polishing by the largest silicon carbide grit size (#60). The variation of SWR with quartz content for the finer abrasives is practically negligible,

because of the much lower material removal rates associated with these smaller particles.[14] Little variation of SWR was found with quartz particle size, even for the largest abrasive grit size (Fig. 8a), probably because the variation in porosity was rather smaller than in the foregoing experimental series.

The results presented above illustrate the close relationship between porcelain tile polishability, evaluated in terms of specific wear rate, and specimen microstructure, described in terms of porosity. In view of the similarities between the trends shown in Fig. 3a–c and those in Figs. 6a, 7a and 8a respectively, a correlation was sought between SWR and porosity, independent of quartz content or quartz particle size. The resulting plot is shown in Fig. 9. The points corresponding to $T > T_{min}$ (i.e. for over-fired structures) have not been included, because of the particular characteristics of the resulting microstructures, as discussed above. Nor are the two
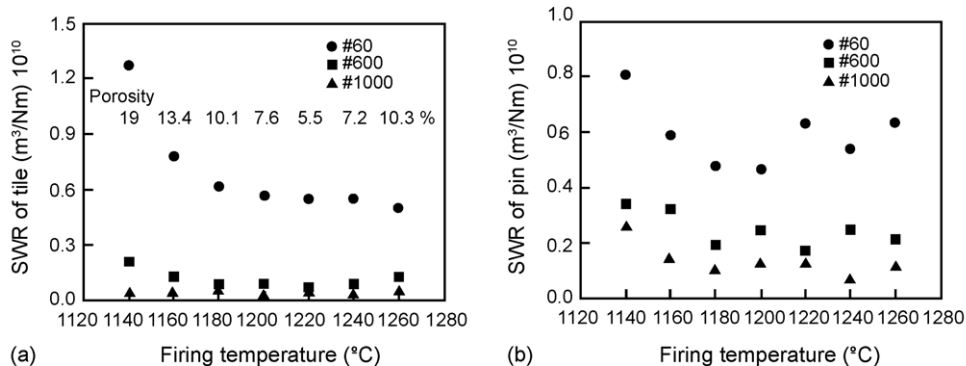

Fig. 6. Variation of SWR with tile firing temperature (a) for the tiles and (b) for the corresponding abrasive pins, for the three different SiC grit sizes.
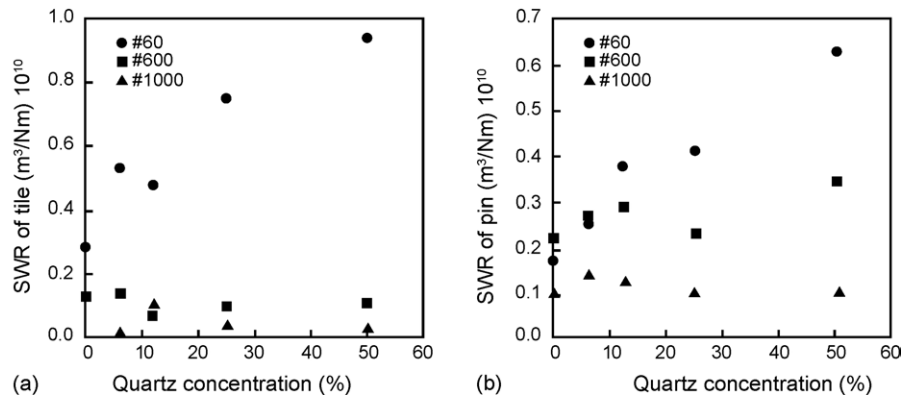
Fig. 7. Variation of tile SWR (a) and pin SWR (b) with quartz content in the starting composition, for the three silicon carbide grit sizes.
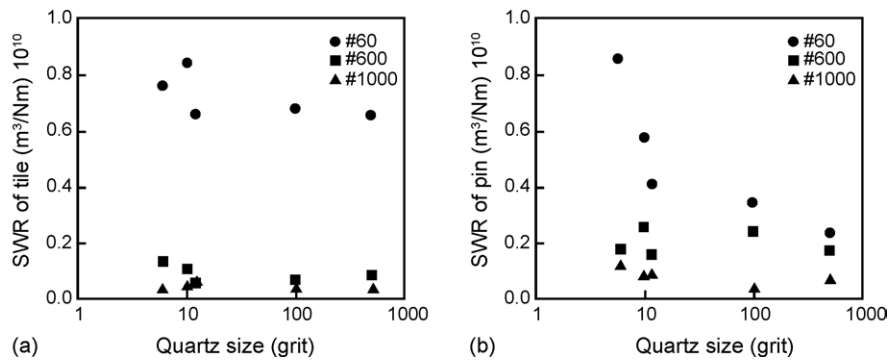

Fig. 8. Variation of tile SWR (a) and pin SWR (b) with quartz particle size (grit) in the starting composition for the three silicon carbide grit sizes.

experimental points included which correspond to the most extreme situations (composition A with no added quartz and composition I with grit 500 quartz, i.e. the finest quartz particle size), since both types of specimens lie very far from industrial practice.

Fig. 9 shows that, despite the simplistic approach of using porosity as the sole descriptive parameter for such a complex feature as microstructure, there is a clear relationship between tile SWR and porosity. Only the circled point (corresponding to composition C) departs significantly from this trend, probably due to the rather low SWR found with this composition (see Fig. 7a). The validity of this experimental correlation
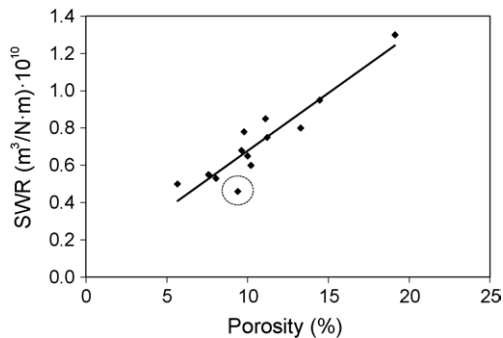
becomes even more relevant, if it is taken into account that the variations imposed on the process variables (firing temperature) and composition (quartz content and particle size) in these experiments far exceed the variations that might be expected in industrial practice. These results validate both the technique used (the laboratory-scale tribometer) and the parameter used here (SWR) for the study of industrial porcelain tile polishing.

Various models for the abrasive wear of brittle materials have been published, based on assumptions of lateral fracture, which predict non-linear relations between the wear rate and mechanical properties (generally modulus of elasticity, hardness and fracture toughness).[14,15] Such models give poor correlation with the results of these experiments, since, for example, the level of quartz in the tile composition causes insignificant variation in the small-scale hardness or toughness. In contrast, bulk mechanical strength and modulus of elasticity were observed to vary significantly, essentially depending on porosity and not on the mechanical properties of the toughening phase. The variation in tile SWR with quartz content is therefore largely due to the correlation between the wear rate and the porosity of the sintered body. Similar behaviour was observed in the other two series of experiments.

3.3.2. Polishing efficiency

From an industrial standpoint, the efficiency of the polishing process depends not only on the wear rate of the tile, but

Fig. 9. Relation between tile SWR and porosity for all the specimens tested with #60 silicon carbide pins.

also on the resulting wear of the polishing tool, which can be very high in the early stages of the process. In order to evaluate this efficiency, the SWR was measured for the abrasive pins, as well as for the tiles.

To evaluate the SWR for the abrasive, the wear volume was determined by measuring the width of the flat wear scar on the pin. Figs. 6b, 7b and 8b show, respectively, the variation of abrasive SWR with firing temperature, quartz content and quartz particle size in the starting composition. The data for the #60 pins indicate a general relationship between tile porosity and abrasive pin wear rate: the higher the porosity of the tile being polished, the greater the wear rate of the abrasive. This correlation is also seen when the wear rates for the three sizes of abrasive are compared: grit #60, which caused more rapid material removal from the tiles than the two smaller abrasives, was at the same time more rapidly worn. Both observations are consistent with the wear mechanisms identified in SiC abrasive-cement matrix tools,[13] in which progressive removal of the cement matrix is required to allow fresh, sharp-edged abrasive grains to wear the workpiece. The wear rate of the abrasive pins varied strongly with quartz particle size in the tile composition (Fig. 8b), probably because both a larger quantity and a larger grain size of hard quartz debris result from the porcelain tile, enhancing three-body abrasion of the pin. In contrast, the effect of firing temperature on pin wear (Fig. 6b) was smaller.

To explore the relative impact of the variables studied (peak firing temperature, quartz content and quartz particle size) a new parameter, termed polishing efficiency, E_P, was defined as the ratio of tile SWR to pin SWR. This is an important quantity for industrial practice because it determines the volume of tile removed by a certain volume of abrasive and can therefore be related directly to the cost-effectiveness of the abrasive. Fig. 10 shows the variation of polishing efficiency defined in this way with peak firing temperature, quartz content and quartz particle size for the #60 abrasive pins, which exhibited the highest material removal rates.

Fig. 10a clearly shows that polishing efficiency decreases as firing temperature rises, up to T_{min}. Above T_{min}, E_P appears to stabilise for the microstructural reasons indicated above. However, the highest polished surface quality (with minimum porosity) is obtained at temperatures around T_{min}. An industrial compromise may be required in this case to achieve an optimum balance between polishing efficiency and product quality. On the other hand, Fig. 10b shows that quartz concentration has little effect on polishing efficiency, because of the correlation between tile abrasion rate and pin wear. Raising the quartz content in the starting composition is therefore detrimental, since it impairs the final polished surface quality (greater porosity) without improving the polishing efficiency. Finally, in contrast to the above, polishing efficiency increases strongly for finer quartz particle sizes (Fig. 10c). This behaviour, together with the higher quality (lower porosity) found in tile specimens containing finer quartz particles, clearly demonstrates the need to minimise quartz particle size in manufacturing polished porcelain tiles.
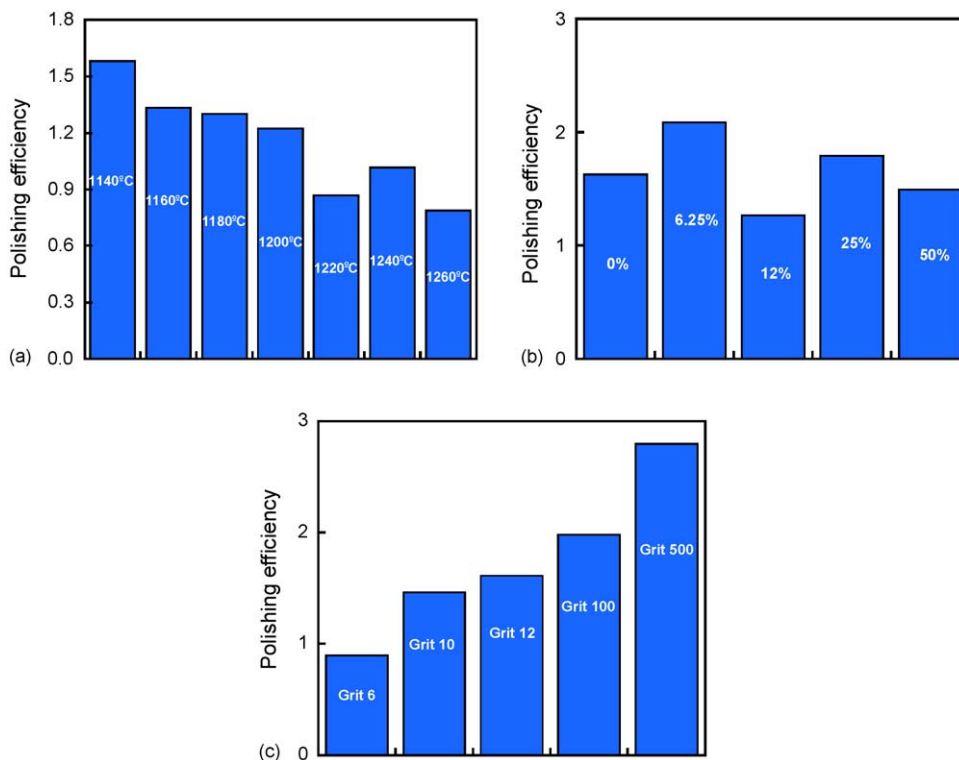

Fig. 10. Polishing efficiency (defined as the SWR for the tile divided by the SWR for the abrasive pin) for tile samples polished with #60 SiC pins at: (a) different peak firing temperatures, (b) different quartz content and (c) different quartz particle size.

4. Conclusions

- Porcelain tile specimens with different fired microstructures were produced by modifying the peak firing temperature. Closed porosity was minimised at temperatures around 1220 °C, increasing above this temperature, consistent with a typical liquid-phase sintering mechanism.

- Adding quartz and/or increasing quartz particle size in the porcelain tile starting composition raised the porosity of the fired specimens significantly, because this reduced the sinterability of the body.

- Tile specific wear rate (SWR) dropped significantly with increasing peak firing temperature (decreasing porosity) up to the temperature at which porosity was minimised. Polishing efficiency (tile SWR/abrasive SWR), E_P, followed a similar trend to tile SWR.

- There was a strong increase in tile SWR with increasing quartz content for larger abrasive particles, though raising the quartz content had little effect on E_P. This factor, together with the resulting deterioration in surface quality, suggests that the tile quartz content should be minimised.

- A slight fall in tile SWR was observed when the quartz particle size was decreased. The effect was much smaller than when the quartz content was varied. In contrast, increasing the quartz particle size led to a strong rise in pin SWR. Consequently, polishing efficiency rises markedly for finer quartz grain sizes.

- It was not possible to demonstrate a relationship between wear rate and the mechanical properties of fired porcelain tiles. However, a relationship between wear rate and porosity was clearly demonstrated.

- In order to improve polishing efficiency and enhance polished surface quality, compositions should therefore be used in which both quartz content and quartz particle size are minimised.

Acknowledgements

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