Effect of solid particle impact on light transmission of transparent ceramics: Role of the microstructure

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ABSTRACT

Sand erosion was done on soda lime glass and transparent ceramics such as alumina and magnesium-aluminate spinel with different microstructures. Surface roughness and optical transmission were measured before and after erosion. The increase of surface roughness depends on both the hardness and grain size of the material. Nearly no surface degradation occurs on polycrystalline samples with HV3 > 15 GPa. The decrease of the real in-line transmittance (RIT) after sand blasting is linked to the increase of surface roughness. We have found that this RIT decrease is correlated to three parameters: incident light wavelength, nature of the material (mechanical properties like hardness) and material microstructure. The influence of these will be discussed. Finally, for all polycrystalline ceramics and single crystals, the RIT is only slightly or not altered after sand blasting either at IR or visible wavelengths.

1. Introduction

The erosion of brittle materials by solid particle impact depends on the properties of the particles (shape, mass, velocity, etc) and of the target (hardness, toughness, etc) [1,2]. Generally, the surface damage of brittle materials eroded by sandblasting occurs primarily by the formation and propagation of radial and lateral cracks [3,4]. The damage is essentially produced by scaling with formation and extension of lateral cracks corresponding to sharp indentation damage. The material removal is characterized by a chipping mechanism where grains are more easily ejected when the particle impact angle is high [5]. Surface roughness/surface morphology is known to decrease optical properties such as photoluminescence [6,7]. Surface roughness could be reduced and optical quality improved by CO2 laser irradiation [8] or by inductively-couple plasma etching [9].

For transparent materials such as glass, the optical transmission falls notably with the projected sand mass whereas the surface roughness is increased. The evolution of optical transmission of a soda lime glass during erosion by sandblasting has been studied by several authors [4,10–12]. This transmission loss is essentially caused by the light beam diffusion due to scattering by surface defects such as scratches and impact sites. Few studies concerning transparent ceramics have also been done. Mroz et al. [13] showed that grain size refinement of Y2O3-doped magnesium-aluminate spinel improves the sandblasting erosion resistance. Solid particle impacts on cermet materials [14] show that different erosion rates could be explained by different material microstructures rather than by their hardness which was shown to be of minor importance. Oka et al. [15] demonstrated that even if, generally speaking, the erosion rate decreases with an increase in both hardness and fracture toughness, this relationship should also be connected with their microstructure.

In this work, we follow of the evolution of optical transmission during sand particle impact erosion of a soda lime glass and transparent ceramics with different microstructures such as alumina or magnesium-aluminate spinel. Correlation between the degree of optical transmission and the sample characteristics (mechanical properties, roughness and grain size) is established.

2. Materials and methods

Seven transparent samples of different nature and microstructure were characterized in this study. They are the following:
The Gc sample is an ordinary soda-lime-silica glass with a 2.8 mm thickness which was used in its as-received state. Sc (orientation 0001) was supplied by Djeva (Switzerland) and PMSc fabricated by the Armorline Corporation (USA). For laboratory-made PCA and PMS samples, the starting materials were commercial powders from the Baïkowski Company. Their main characteristics are given in Table 1. The specific surface area (SSA) was measured by BET (Micromeritics) and the powder particle (or agglomerate) size distribution was measured with a laser diffraction apparatus (Horiba LA-920). The amount of impurity is very small at around 0.01 wt% for both powders to prevent light absorption by this impurity.

The commercial powders were put in a graphite die to be sintered by SPS (HP D 25, FCT System) without any further preparation. The temperature during sintering is measured 3 mm from the sample with an optical pyrometer focused on the non-through hole (3 mm diameter) in a graphite die. The sintering cycles were previously optimized to obtain transparent PCA samples [16,17]. Oka et al. [15] have shown that an impact angle of 90° significantly deteriorate the optical properties of soda lime glass [10,12]. For comparison purposes, all RIT measurements were calculated for the same thickness \( t_2 = 1\) mm (Eq. (1)).

\[
\text{RIT}(t_2) = (100 - R_0) \left( \frac{\text{RIT}(t_1)}{100 - R_0} \right)^{t_2/t_1}
\]

where \( R_0 \) is the total normal surface reflectance (\( R_0 = 14 \) for Sc and PCA samples, 13 for PMS samples and 8 for Gc sample). RIT\((t_0)\) is the RIT for sample thickness \( t_0 \).

A SEM ZEISS Supra55 was used to investigate the microstructure of the samples. Average grain sizes \( d_v \) were evaluated on fracture surfaces for PCA samples and on a polished surface after chemical etching for PMS samples (during 120 s in a concentrated phosphoric acid at its boiling temperature). A line-intercept method taking into account at least 200 grains was used and three-dimensional factors of 1.22 and 1.56 were applied to obtain a revised grain size for fractured and polished surfaces, respectively [19,20].

The sandblasting was done with a horizontal type sand blower apparatus (Fig. 1). A predetermined quantity of sand is placed in the sand hopper equipped with a flow rate control device. The sand is projected by airflow on the target surface with controlled incident angle and velocity. The selected experimental parameters are given in Table 2. From previous studies, they are known to significantly deteriorate the optical properties of soda lime glass [10,12]. Oka et al. [15] have shown that an impact angle of 90° is the worst condition for brittle material erosion like alumina. The sand particle angularity could also have a noticeable effect on material erosion as discussed by Hamblin and Stachowiak [21]. However, for our study the same kind of sand was used for all specimens (2 mm and 3 mm thickness for PCA and PMS specimens respectively) were carefully mirror-polished (down to 1 \( \mu \)m) on both sides using diamond slurries.
Finally, the Vickers Hardness (HV) of the samples was evaluated from the trace dimensions (diagonals d) left by a diamond square-base pyramid (136° at the top) using an indentation load \( P \) (Eq. (3)). Several measurements were performed on each sample to ensure reproducibility. The load selected for the indentation was 3 kgf and is labeled HV3 (as recommended by Swab [22]) except for the glass Gc (1 kgf) in order to avoid chip formation. For sapphire, 15 indents have been made for HV3 type for a successful measurement on 6 of these indents.

\[
HV = 2 \cdot \sin \left( \frac{136°}{2} \right) \cdot \frac{P}{d^2}
\]

(3)

3. Results and discussion

The microstructures, mechanical and optical properties of the samples were characterized before sand erosion. The results are presented in Table 3. As expected, low temperature laboratory-made samples (PCA1200, PMS1300) sintered by SPS present small average grain sizes that increase with increasing sintering temperature (compare with PCA1330, PMS1400). Hardness of PCA and PMS samples is decreasing when the grain size is increasing. However, it can be noticed that the hardness decrease of PMS is less important than observed by Krell and Bales [23]. That behavior could be related to carbon pollution from graphite mold which increases with the sintering temperature [24]. The presence of carbon at grain boundary may lead to an improvement of the mechanical properties [25]. As shown in Fig. 4a, for a birefringent material like PCA, the grain size increase from PCA1200 to PCA1330 results in a decrease of optical properties as predicted by the Apetz theoretical model [20]. Lower RIT values measured on the samples compared to the theoretical curve are due to residual porosity. For pure PMS which has a cubic structure (Fig. 4b), the decrease of optical properties can be due to the residual porosity (scattering) and/or carbon contamination (absorption). When increasing \( T_s \) from 1300 to 1400 °C, pore coalescence can occur at the end of densification. Thus, the large RIT decrease is due to the increase of the pore size from \( \sim 60 \) nm to \( \sim 150 \) nm which accompanies the grain growth from 0.25 \( \mu \)m to 13.6 \( \mu \)m. No whitish haze indicating a population of invisible small defects <1 \( \mu \)m has been observed for any PMS samples. However, the PMS 1400 sample exhibits a darker gray aspect compared to the PMS 1300, linked to the carbon contamination.

The RIT of glass (Gc) is close to its maximum theoretical transmission (92%) whereas that of the sapphire single crystal (Sc) corresponds to its theoretical value (86%). Those very high RITs indicate the absence of defects (pores, inclusions) within their structure. The PMS sample has very large grain growth that may slightly increase the porosity. This explains why its RIT is also very close to the maximum theoretical transmission (87%).

The measured hardness of each sample is in good agreement with the literature [26]. As expected for polycrystalline materials, higher hardness is found for fine-grained samples because of the limited dislocation mobility in smaller grains.

The Rq before and after sand blasting are given in Table 3. The difference is called \( \Delta Rq \). It decreases when the hardness increases as shown in Fig. 5. A plateau is reached from HV \( \sim 15 \) GPa and
nearly no degradation occurs on polycrystalline samples having superior hardness. Nevertheless, the Rq of a sapphire single crystal slightly increases after sand blasting despite its high hardness of 16.9 GPa. This suggests that the degradation of materials after erosion is also linked to the toughness (smaller for single crystals) and microstructure as already mentioned in the literature [1,2,14,15].

**Table 3**

<table>
<thead>
<tr>
<th>Samples</th>
<th>( \phi_c (\mu m) )</th>
<th>HV3 (GPa)</th>
<th>Before erosion</th>
<th>After erosion</th>
<th>( \Delta Rq (\mu m) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number</td>
<td>Name</td>
<td>RIT 550nm (%)</td>
<td>RIT 2000nm (%)</td>
<td>Rq (( \mu m ))</td>
<td>RIT 550nm (%)</td>
</tr>
<tr>
<td>1</td>
<td>Gc</td>
<td>90</td>
<td>88</td>
<td>0.015</td>
<td>4</td>
</tr>
<tr>
<td>2</td>
<td>Sc</td>
<td>86</td>
<td>86</td>
<td>0.703</td>
<td>84</td>
</tr>
<tr>
<td>3</td>
<td>PCA1200</td>
<td>5.5a</td>
<td>90</td>
<td>0.015</td>
<td>4</td>
</tr>
<tr>
<td>4</td>
<td>PCA1330</td>
<td>16.9</td>
<td>86</td>
<td>0.703</td>
<td>84</td>
</tr>
<tr>
<td>5</td>
<td>PMSc</td>
<td>1.67</td>
<td>5</td>
<td>0.647</td>
<td>85</td>
</tr>
<tr>
<td>6</td>
<td>PCA1200</td>
<td>0.55</td>
<td>37</td>
<td>0.041</td>
<td>37</td>
</tr>
<tr>
<td>7</td>
<td>PCA1330</td>
<td>13.6</td>
<td>18</td>
<td>0.249</td>
<td>18</td>
</tr>
</tbody>
</table>

RIT for 1 mm sample thickness.

\( \alpha \) With 1 kgf.

**Fig. 4.** Comparison of experimental and theoretical RIT550 nm values calculated for 1 mm sample thickness from the Apetz model as a function of (a) grain size \( \phi_c \) for PCA and (b) pore size \( \phi_p \) for PMSc (for 0.01% porosity).

**Fig. 5.** Difference between Rq after and before erosion as a function of material hardness.

**Fig. 6.** Micrographs showing the damage caused on the surfaces eroded by sandblasting: (a) Gc glass, (b) Sc sapphire and (c) PMSc spinel.

Fig. 6 shows some details of typical surface damage induced by sandblasting. The material removal takes place by the formation and propagation of lateral cracks which develop into chipping [4,5]. For our severe sandblasting conditions, the Gc glass shows
a totally eroded surface (Fig. 6a) corresponding to the large increase of roughness whereas the alumina, sapphire and spinel show very little indentation damage (Fig. 6b). These observations explain the low variation of roughness for the latter materials. Concerning the PMSc material, we observe some large impact flaws (with a size greater than 100 \( \mu m \)) which appear from the grain boundaries (Fig. 6c). This observation correlates to the relative increase of roughness for this ceramic which shows an effect of grain boundaries on the flaw generation. This effect of grain size can be related to the results obtained by Tokariev et al. [27] who observe that for a coarse-grained spinel the grain boundaries are weak and responsible for failure.

Optical properties tend to decrease after sand blasting due to the increase of surface light reflection (Table 3). In fact, according to the literature [28,29], \( R_q \) has a direct influence on the surface reflectance \( R_s \), which is more or less pronounced depending on the wavelength. If the defect size (\( R_q \)) is small compared to the wavelength, the \( R_s \) will only slightly increase. That is why optical properties decrease less at infrared (IR) wavelengths. In fact, the two materials having the smallest hardness show the highest \( R_q \) after sand blasting: 5.356 \( \mu m \) for Gc and 3.591 \( \mu m \) for PMSc. These deep eroded surfaces could induce an increase of surface light reflection in the IR whereas the optical properties of other materials having \( R_q \) inferior to 900 nm after sand blasting remains unaltered in the IR. However, even with a high \( R_q \), the PMSc sample does not present any RIT decrease in the IR because as shown in Fig. 6c the surface defects are deep but not numerous compared to the Gc sample in Fig. 6a.

In the visible range, the \( R_q \) after erosion is the same order as the wavelength and therefore induces more surface light reflection. As
a result, the greater ΔRq, the greater the decrease in optical properties (Table 3). We observe that the decrease of the RIT_{550nm} for PMSc is rather small compared to the increase of Rq (the variation of the RIT is comparable to the sapphire single crystal (Sc) or to the small-grain spinel (PMS1300) ones). The small RIT decrease of PMSc is linked to the particular surface degradation of that large grain microstructured sample. This result can be related to the observation of Tokariev et al. [27] who observed that the coarse-grained spinel is less prone to impact damage. In fact, for the large grain size of the PMSc the surface discontinuity (number of defects) is lower (less numerous defects) than for the smaller grain sized sample of PMS1300 (Fig. 7). Therefore, the Rq parameter is very sensitive to the impact depth whereas the numbers of defects seems to be greater. One can also observe on the profile in Fig. 7a that the space between two discontinuities is proportional to the sample grain size (300 μm). Furthermore, the defects observed in Fig. 6c have a similar size to the grain size which also corresponds to the large defect size in the profile. Increasing the grain size could be favorable in reducing the number of defects and consequently limiting the RIT decrease.

The decrease of optical properties due to surface roughness therefore depends on three parameters: incident light wavelength, material microstructure and mechanical properties (hardness) of the material.

4. Conclusion

To conclude, sand erosion of a soda lime glass and some transparent ceramics with different microstructures was done. We observed that after erosion the surface roughness increases as the hardness of the material decreases. For example, for HV3 > 14–15 GPa, nearly no degradation occurs on polycrystalline samples.

The light transmission decrease after sand blasting is linked to the increase of surface roughness. We have shown that this behavior depends on three different parameters. The first is the incident light wavelength. If it is high compared to the roughness, the light transmission will only slightly decrease. The second concerns the material properties, like hardness. In fact, the increase of surface roughness is material dependent and has a direct influence on the surface reflectance: the greater the hardness the smaller the RIT decrease. The third parameter concerns the material microstructure. Large grains minimize the density of surface defects, and even if these are large and greatly increase the roughness, they slightly reduce the RIT.

Acknowledgements

This work is a part of the CeraTRANS project. We would like to thank the ANR (French National Research Agency) for its funding support. We also thank Lionel Bonneau from Bailleul for providing the commercial samples and Guillaume Servin from Scream for the roughness measurements.

References