# Fracture analysis of phosphate and silicate glasses by microscopy and nanoindentation: comparison of different glasses utilized for building engineering

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## INTRODUCTION

Traditional testing methods for fracture toughness determination are generally based on macroscopic crack extension experiments. However, evaluating the fracture toughness of glasses and ceramics is still a problematic task. As a consequence, a mechanism for measuring fracture toughness of these materials has not been well determined. Nanoindentation test is an option that has been used in order to assess the fracture toughness of brittle materials, such as glasses, ceramics, films and coatings. As long as the materials exhibit brittle fracture with minimal crack tip plasticity, this crack pattern can be used to establish fracture toughness values [1]. This property can be estimated by either crack-length-based method or crack-energy-based method, providing useful partially quantitative information. In this work, nanoindentation was used to assess the fracture toughness of different glass compositions. These innovative compositions are composed by potassium metaphosphate ($KPO_3$), aluminium oxide ($Al_2O_3$) and by-products (fly ash or slag). Their preparation was previously reported [2]. Different loads were tested in order to produce controlled cracks. The elastic modulus and the hardness of the samples were also determined. The crack lengths were evaluated by optical microscopy and scanning electron microscopy (SEM).

## MATERIALS AND METHODS

Berkovich nanoindentation was used for the measuring of elastic modulus and hardness of the samples. The load on sample was 400 mN and the average values of both properties were calculated between 1000 nm and 1400 nm. Each test was repeated

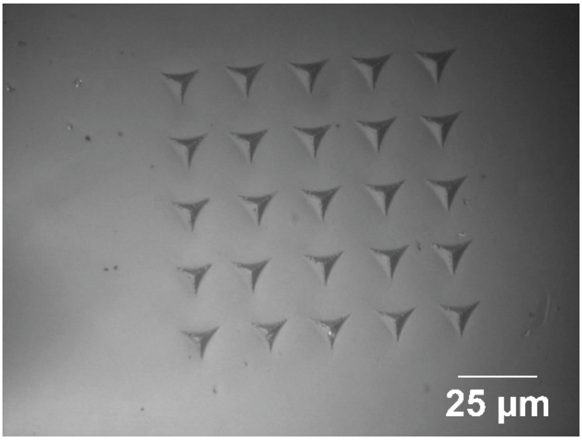


*Figure 1: Indents in the sample 40KPO₃-20B₂O₃-25fly ash-15Al₂O₃.*

25 times as 5 x 5 indents, as shown in Figure 1. High loads were used in order to produce controlled cracks and estimate the fracture toughness of the samples, also using a Berkovich indenter. The crack lengths were evaluated by optical microscopy. The first load-controlled experiments were performed using a load of 32 mN, loading and unloading time of 5 seconds, and hold time of 10 seconds. As these first tests did not generate visible cracks, the load was increased from 32 mN to 2.5 N, consisting of 30 seconds loading, 10 seconds unloading and hold time of 15 seconds. Each test was repeated at least 4 times. For the last fracture toughness tests a Knoop indenter was used under a load of 8.5 N. All the tests were performed using a Nano Indenter MTS G200.

## RESULTS AND DISCUSSION

The elastic modulus and hardness were calculated based on the load-displacement curves of the samples, via the Oliver–Pharr method [3]. The average values of both properties are shown in Table 1. The glasses containing fly ash and slag have elastic modulus and hardness much lower than the standard silicate glass, used here as reference.

*Table 1- Average elastic modulus and hardness of the samples.*

| Composition (wt. %) | Avg. E (GPa) | Avg. H (GPa) |
|---|---|---|
| $60KPO_3$-20fly ash-$20Al_2O_3$ | 41.6 | 3.94 |
| Reference (silicate) | 81.7 | 8.33 |
| $55KPO_3$-35fly ash-$10Al_2O_3$ | 43.5 | 4.18 |
| $60KPO_3$-30fly ash-$10Al_2O_3$ | 45.2 | 4.56 |
| $40KPO_3$-$20B_2O_3$-25fly ash-$15Al_2O_3$ | 48.4 | 5.02 |
| $75KPO_3$-12.5slag- $12.5Al_2O_3$ | 41.6 | 3.42 |
| $70KPO_3$-15slag-$15Al_2O_3$ | 43.7 | 4.19 |

Although somewhat problematic, fracture toughness evaluation can be quickly performed using indentation. If a sharp tip is forced into a bulk sample of a brittle material, radial cracking usually occurs after a critical load has been reached, originating from stress concentrations at the diamond facet edges, which allows the estimation of fracture toughness ($K_c$) based on the maximum indentation load and the crack length (Vella et al., 2003). An equation allows the calculation of $K_c$ based on indentation tests, measuring the length of cracks originating from the edges of the indent impression, determining that:

$$K_c = A\,(\frac{E}{H})^{1/2}\;\frac{P}{c^{3/2}}$$

In which $E$ = Youg's modulus, $H$ = hardness, $P$ = load, $c$ = crack length measured from the center of the indent to the crack tip. $A$ is an empirical constant, which varies according to the indenter and the crack geometry [4]. The representative dimensions of pyramidal indentation fracture testing method are shown in Figure 2(a). A crack is classified as well developed if $c >> a$. The different glass compositions were analysed by optical microscopy and presented huge variations in crack lengths. Some samples, such as 40$KPO_3$-20$B_2O_3$-25fly ash-15$Al_2O_3$ shows a crack length equivalent to the impression diagonal, as presented in Figure 2(b), while samples such as 70$KPO_3$-15slag-15$Al_2O_3$ exhibited well developed cracks (Figure 3).

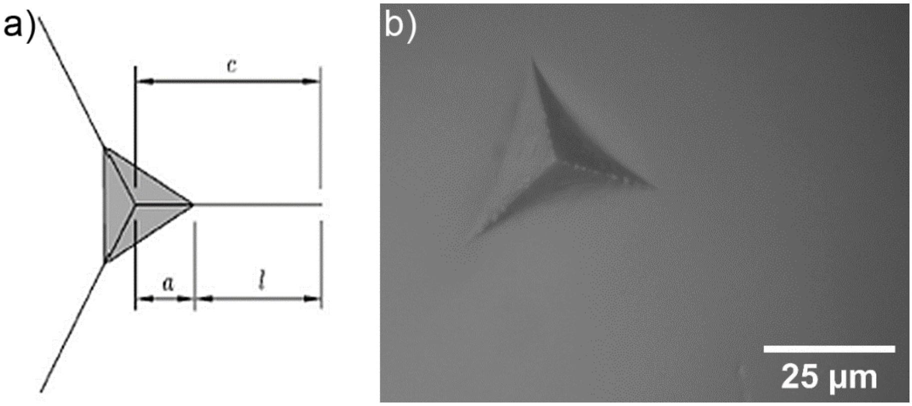


*Figure 2: (a) Schematic figure of crack pattern induced by a Berkovich indenter after Hyun et al. [5], and (b) indent in the sample 40$KPO_3$-20$B_2O_3$-25fly ash-15$Al_2O_3$ induced by a load of 2.5 N using a Berkovich indenter.*

Due to the absence of developed cracks in the samples 55$KPO_3$-35fly ash-10$Al_2O_3$, 60$KPO_3$-30fly ash-10$Al_2O_3$, reference (silicate), 50KPO3-20$B_2O_3$-15slag-15$Al_2O_3$ and 50$KPO_3$-20$B_2O_3$-15slag-15$Al_2O_3$, new tests were performed using a higher load and another geometry of indenter, the Knoop indenter. Then, the cracks were analyzed with optical microscopy and scanning electron microscopy. Different patterns of cracks were associated to different glass compositions (Figures 4 and 5).

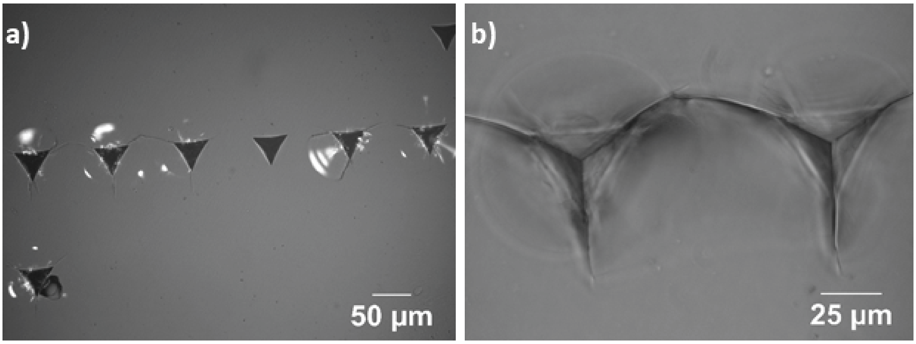


*Figure 3: Cracks in the sample 70KPO$_3$-15slag-15Al$_2$O$_3$ induced by a load of 2.5 N using a Berkovich indenter: (a) all seven indents, and (b) details of two indents.*

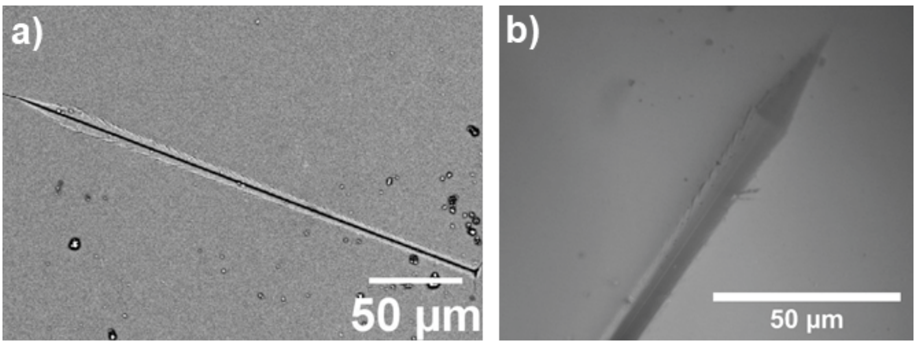


*Figure 4: Crack pattern of the sample 40KPO$_3$-20B$_2$O$_3$-25fly ash-15Al$_2$O$_3$ induced by a load of 8.5 N using a Knoop indenter indent (a) analyzed by SEM, and (b) close-up of the cracks analysed by optical microscopy.*

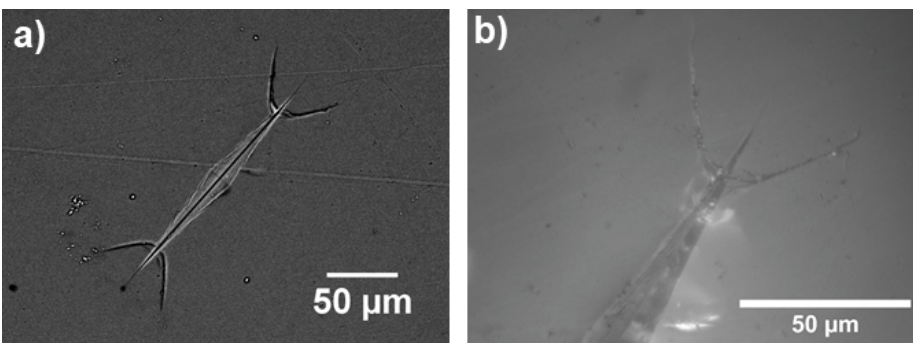


*Figure 5: Crack pattern of a silicate glass induced by a load of 8.5 N using a Knoop indenter (a) indent analyzed by SEM, and (b) close-up of the cracks analysed by optical microscopy.*

## CONCLUSIONS

The analysis allowed a comparison among the performance of the standard silicate glasses already used for construction purposes and new glass compositions, previously reported by the authors. Among the new compositions, the glasses containing $B_2O_3$ and highest amounts of fly ash and slag presented a higher fracture toughness than the samples with higher concentrations of phosphate and lower amounts of by-products. The results of the nanoindentation tests combined with microscopy and image analysis supported an evaluation of the potential of the new glass compositions to be utilized for building engineering purposes.

## ACKNOWLEDGEMENTS

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