SCLEROMETRIC ANALYSIS AND MICROSTRUCTURAL CARACTERIZATION OF A SINTERED Li_2O-Zr_2O-SiO_2 GLASS-CERAMIC

C. Lira*, R. M. do Nascimento, L. Pandini, A. P. Novaes de Oliveira, O. E. Alarcon

Universidade Federal de Santa Catarina - Departamento de Eng. Mecânica - LabMat
Cx. Postal: 476, CEP: 88035-900, Florianópolis (SC) - Brazil
*claudia@pg.materiais.ufsc.br

INTRODUCTION

Areas like the floors of shopping centres, supermarkets and commercial buildings require products exhibiting suitable mechanical properties such as high strength and wear resistance. Some commonly used products for such applications are Marble, Granite and more recently Neoparies and Porcelanized Stoneware. The research of new structural products, mainly hard ceramic tiles, has pointed out the potential of glass-ceramic materials in architectural applications. Features like high hardness and mechanical strength can be easily reached in glass-ceramics. Because of their good properties, glass-ceramic materials could be applied instead of traditional tiles and products in such areas where a better performance is required [1].

In recent studies [2,3] a sintered glass-ceramic belonging to the Li_2O-Zr_2O-SiO_2 system (LZS) with interesting features for use as floor tile was obtained. The LZS glass-ceramic presented a higher modulus of rupture and also a higher resistance to deep abrasion.

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[1] P. Novaes de Oliveira, C. Leonelli, T. Manfredini, Actas de Qualicer/98, , Castellón, (1998), P.GI: 193.

[2] P. Novaes de Oliveira, T. Manfredini, L. Barbieri, C. Leonelli, G. C. Pellacani, J. Am. Ceram. Soc, 81, 3 (1998) 777.

[3] P. Novaes de Oliveira, T. Manfredini, G. C. Pellacani, Anais do IV Congresso Nazionale AIMAT - Cagliari, (1998), 3.

The main purpose of this work is to evaluate the scratch hardness of a LZS glass-ceramic, using the sclerometric technique and to relate the results to its microstructure and compare it with some reference materials. Glass-ceramic samples were obtained by sintering LZS glass powders and the microstructure of the sintered samples was analysed.

EXPERIMENTAL PROCEDURES

Glass powder compacts were formed from frits with a composition of 23,12% mol Li_2O, 11,10% mol ZrO_2 and 65,78% mol SiO_2. Samples were sintered in an electric furnace at 850°C, 900°C and 950°C, with a constant heating rate of 20°C/min and 10-min hold at maximum temperatures. The microstructure and scratch surfaces were examined using scanning electron microscopy (SEM) (Model Philips XL 30). Furthermore, samples from the reference materials Marble, Granite, Porcelanized Stoneware and Neoparies were prepared by cutting and polishing for the sclerometric tests and the results were compared to those of the LZS sintered glass-ceramic.

Sclerometric tests were performed using a normal load of about 300g, at a constant scratching rate of 100 $\mu m.s^{-1}$. Scratch measurements were made with the aid of an image analyzer software on micrographs from the scratched samples. Scratch hardness was calculated through the equation:

$$H_r = K_1 \cdot \frac{F_n}{L^2} \qquad (1)$$

(Hr = scratch hardness [N/mm^2]; Fn = normal load applied [N]; K_1 = constant due to indenter geometry; L= scratch width [mm]

RESULTS AND DISCUSSION

Table 1 relates the results from sclerometric tests. The obtained scratch hardness values indicate a better performance of the LZS sintered glass-ceramics when compared to the other materials, similarly to the deep abrasion results found.

LZS glass-ceramic – 850°C			
Load (g)	Fn (N)	L (μm)	Hr (N/mm²)
300	2,50	23,91 (σ = 2,25)	17492,05
LZS glass-ceramic – 900°C			
300	2,56	26,92 (σ = 1,23)	14119,76
LZS glass-ceramic – 950°C			
300	3,07	15,79 (σ = 1,41)	49253,16
Granite			
300	2,37	62,04 (σ = 5,89)	2363,00
Neoparies			
300	2,40	149,80 (σ = 21,69)	427,81
Porcelanized Stoneware			
300	2,54	84,86 (σ = 8,59)	1410,87

Table 1. *Sclerometric test results.*

The best results were obtained for the glass-ceramic sintered at 950°C and the lowest scratch hardness was found for Neoparies. Higher scratch hardness values were obtained for the glass-ceramic sintered at 950°C. From the analysis of scratch morphology, the appearance was observed of lateral cracks in sample surfaces of Granite, Marble, Neoparies and Porcelanized Stoneware, leading to poor accuracy of the measured scratch widths and consequently of the calculated scratch hardness. On the other hand, this analysis allows inferring the higher brittleness of these materials when compared to the LZS glass-ceramic, which did not present the lateral crack nucleation effect.

CONCLUSIONS

The sintered LZS glass-ceramic presented a higher scratch hardness than the other tested materials, under the same test conditions. A micro-cracking effect due to the scratching tests was observed on the surfaces of the reference materials. The glass-ceramic LZS did not present the micro-cracking effect when tested under the same conditions. This reveals a higher brittleness of the reference materials when compared with the studied glass-ceramic. The better results of the sintered glass-ceramic are probably related to the formation of the crystalline phases: $Li_2Si_2O_5$ and $ZrSiO_4$. The large quantity of small precipitated crystals works against crack propagation, improving material toughness and scratch resistance.

The sclerometric technique can be a useful method for the quantitative evaluation of the abrasion resistance of materials. Nevertheless, with regard to the non-comparable aspects, further study is necessary to analyze scratch hardness results as absolute values.

The lateral crack nucleation effect can occur when scratching tests are performed on very brittle materials. Scratch width cannot be accurately measured under these conditions. Therefore, the quantitative evaluation of scratch hardness on these materials remains inconclusive under such conditions.