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MECHANICAL PROPERTIES OF REUSED LITHIUM DISILICATE GLASS-CERAMIC OF IPS EMPRESS 2 SYSTEM

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This investigation was designed to estimate the biaxial flexure strength and fracture toughness of lithium disilicate glass-ceramics of IPS Empress 2 system pressed with as-received ingots and their sprue buttons. Two groups of the lithium disilicate glass-ceramics were prepared as follows: group 1 is ingot-pressed group; group 2 is sprue button-pressed group. A ball-on-three-ball test was used to determine biaxial flexure strength (BFS) of disks in wet environment. Scanning electron microscopy(SEM) analysis was conducted to observe the microstructure of the ceramics. Unpaired t-test showed that there were no differences in the mean biaxial flexure strength (BFS) and KIC values between group 1 and 2 ($p > 0.05$). Two groups showed similar values in the KIC and the strength at 5% failure probability. The SEM micrographs of the IPS Empress 2 glass-ceramic showed a closely packed, multi-directionally interlocking pattern of numerous lithium disilicate crystals protruding from the glass matrix. The lithium orthophosphate crystals could not be observed on the fracture surface etched. There was no a marked difference of the microstructure between group 1 and 2. Although there were no tests including color stability, casting accuracy, etc., the results of this study implied that we could reuse the sprue button of the pressed lithium disilicate glass-ceramic of IPS Empress 2 system.

Key Words

Biaxial flexure strength, Fracture toughness, Ceramic microstructure, Lithium disilicate glass-ceramics, IPS Empress 2 system

In the past decade, the glass-ceramic has been used as a material with variable crystals that affect the appearance, characteristics, and mechanical properties of metal-free fixed restorations. Since 1990, the IPS Empress glass-ceramic has been successfully used to fabricate veneers, inlays, onlays, and anterior and posterior crowns for dental esthetics.[1,2] However, the IPS Empress glass-ceramic was not suitable for the fabrication of bridges because the flexural strength of the ceramic was about 200 MPa.[3] To overcome this limitation, the lithium disilicate glass-ceramic of IPS Empress 2 was recently developed to sig-

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nificantly elevate the strength beyond the original leucite material of the IPS Empress and enable the fabrication of 3-unit fixed partial denture in the anterior and premolar region.[4] The IPS Empress 2 glass-ceramic replaced the conventional IPS Empress layering ceramic.

Heat-pressing technique is widely employed as the fabrication method of all ceramic restoration because of its convenience and good dimensional accuracy. The lithium disilicate glass-ceramic is one of the heat-pressable materials using lost wax technique. Ceramic sprue button which is necessarily remained after the heat-pressing procedure can be reused like a noble metal sprue button in economic reason. The purpose of this study was to estimate biaxial flexure strength and fracture toughness of lithium disilicate glass-ceramic pressed with as-received ingots and their sprue buttons.

MATERIALS AND METHODS

The lithium disilicate glass-ceramic ingots (IPS Empress 2 ingot, shade no. 500, Ivoclar) were used for this investigation. The disk specimens were divided into two groups: group 1 is ingot-pressed group; group 2 is sprue button-pressed group.

For group 1, the paraffin wax disks were made a little bigger than the required dimension that is 15mm in diameter and 1.4mm in thickness. One wax wire (2.8 mm × 7.0 mm) was attached at the end of the specimen for spruing. Investment was carried out with the IPS Empress 2 special investment powder(200 g) and liquid(44 mL). After setting time(1 hour), the stabilizer ring, ring base, and paper ring were removed, and the bottom of the investment cylinder was trimmed with a plaster knife and sand paper. Without ingots, the investment ring and AlOx plunger were preheated in a furnace(MicroThurm, Fink and Hilburg). After preheating, the investment ring positioned the cold ingot and AlOx plunger were transferred from a preheating furnace to a special heat-press furnace(EP500, Ivoclar) that was already preheated at 500℃. The investment cylinder was immediately removed from the furnace and allowed to cool to room temperature on an elevated grid after the program was automatically completed. After divesting and sprue cutting, the final preparation of all specimens(n=20) were done with careful grinding to ensure the required fine surface and uniform thickness using a metallurical grinding machine (Knuth-Rotor, Stuers) with SiC disks (grit size 320 to 1200). For group 2, the specimens(n=20) were made with ceramic sprue buttons that were remnants of the heat-pressing for group 1 by the same procedure.

A ball-on-three-ball test was used to determine biaxial flexure strength (BFS) of disks in wet environment. The specimens were loaded in a universal testing machine (Z020, Zwick) at a crosshead speed of 1.0 mm/min until fracture (Fig. 1). The

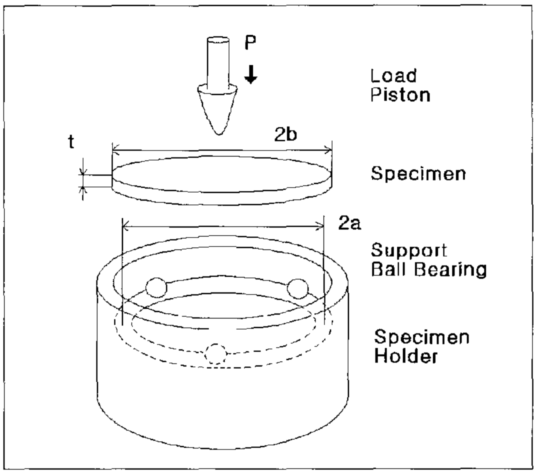

Fig. 1. Schematic illustration of ball-on-three-ball bi-axial flexure test. a: the radius of the support circle; b: the radius of disc specimen; c :is radius of the region of uniform loading at the center (c = t/3); t:: the thickness of the specimen.

fracture load was used to calculate the biaxial flexure strength with the following formula:

$S(BFS) = - 0.2387 P(X-Y) / t^2$

Where P = fracture load (N), $X = (1+\acute{\imath})\ln(c/b)^2 + [(1-\acute{\imath})/2](c/b)^2$, $Y = (1+\acute{\imath})[1+\ln(a/b)^2] + [(1-\acute{\imath})(a/b)^2]$, $\acute{\imath}$ is Poissons ratio (0.25), A is radius of the supporting circle (10 mm), B is radius of the disk (mm), C is radius of the region of uniform loading at the center ($c = t/3$), and T is specimen thickness (mm).

Fracture toughness (KIC) was measured on 10 disk pieces per group by an indentation method. Seven indentations on each piece were made with a Vickers indenter (MXT-70, Matsuzawa Seiki) at a load of 9.8 N for 15 seconds. The KIC was calculated by the following equation:

$KIC = 0.016(E/H)^{0.5} (P/C\ 1.5)$

Where E/H is the elastic-modulus-to-hardness ration, P is the load applied, C is the crack length measured from the center of indentation. The E/H ratio was determined from three Knoop indentations on each specimen. Biaxial flexure strength and KIC data were analyzed by unpaired t-test.

The Weibull distribution on the strength data was described by following formula:

$Pf = 1-\exp[-(\sigma / \sigma o)m]$, $Pf = Rank/(N+1)$

where *Pf* is the probability of failure, $\sigma$is the strength at a given *Pf*, σo is the characteristic strength, m is Weibull modulus, and *N* is the number of specimens. The Weibull regression analysis provided the Weibull modulus (m) and their 95% confidence intervals, and strength predicted at the 5% level of failure.

For observation of the microstructure using a scanning electron microscope (LEO 145VP, LEO, England), the polished surface was etched with the watery mixture containing 30% H_2SO_4 and 4% HF for 10~15 seconds and immediately cleaned with water and ethanol, and coated by gold sputtering.

RESULTS

Mean biaxial flexure strength and fracture toughness of the groups are shown in Table 1. Unpaired t-test showed that there were no differences in the mean biaxial flexure strength (BFS) and KIC values between the group 1 and 2 ($p > 0.05$).

The results of Weibull analysis are listed in Table 1. Weibull plot of the groups is displayed in Fig. 2. Two groups showed similar values in the m-value and the strength ($\sigma 0.05$) at 5% failure probability.

In SEM observation, IPS Empress 2 glass-ceramic showed rod-shaped lithium disilicate crystals. This crystals exhibited a closely packed, multi-directionally interlocking pattern of numerous lithium disilicate crystals protruding from the glass matrix. However, the lithium orthophosphate crystals could not be observed on the fracture surface etched. In the microstructure, there was no a marked difference between group 1 and 2 (Fig. 3).

Table Ⅰ. Mean Biaxial Flexure Strength(BFS) and Fracture Toughness Values of the Specimens

Group	No	BFS (MPa)	m-value	$\sigma 0.05$ (MPa)	KIC (MPa · $m^{1/2}$)
1	20	334.2±27.6	12.4	275.4	3.19±0.19
2	20	338.5±29.3	11.8	276.1	3.10±0.12

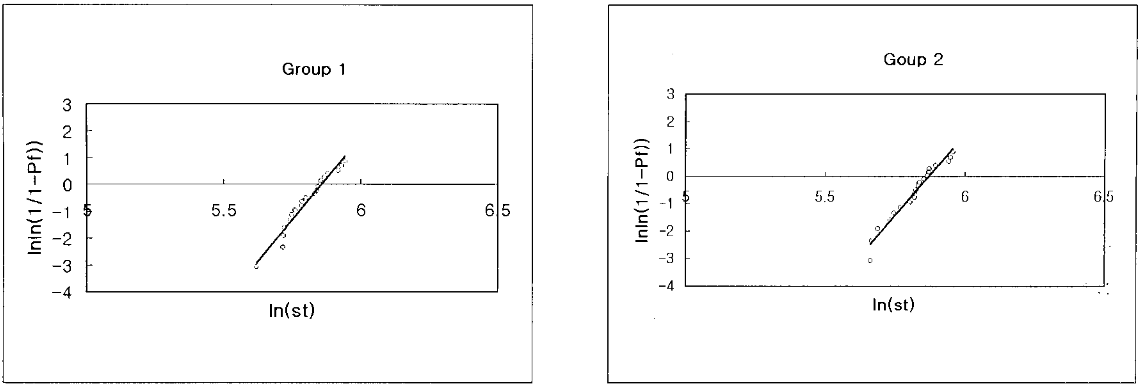

Fig. 2. This Weibull plot shows two groups have similar values in the m-value and the strength at 5% failure probability

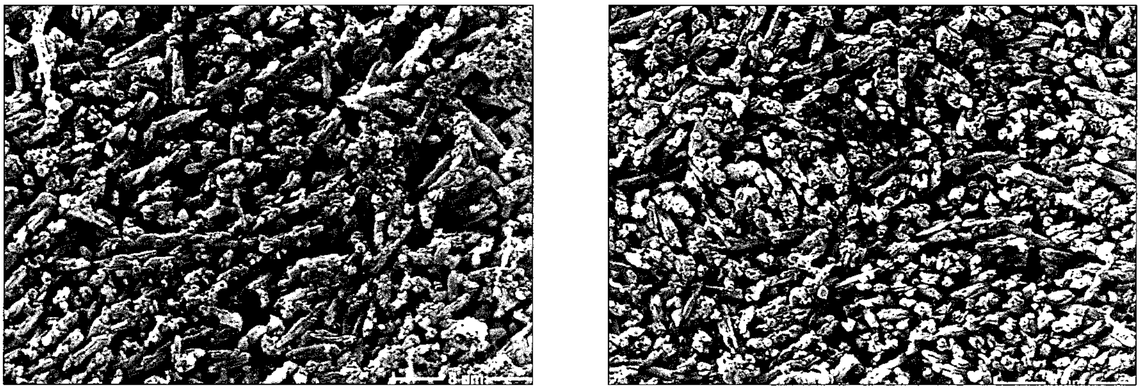

Fig. 3. The SEM micrograph of IPS Empress 2 glass-ceramic pressed with as-received ingots(group 1) and their sprue buttons(group2). A: group 1; B: group 2 (Original magnification × 5,000).

DISCUSSION

Glass-ceramics are polycrystalline materials consisting of a glassy matrix and one or more crystal phases produced by the controlled nucleation and growth of crystals in the glass.[5] The first lithium disilicate glass-ceramics were developed as early as in the fifties by Stookey.[6] Following his fundamental discovery, lithium disilicate glass-ceramic became the subject of a considerable amount of research. Although the ceramics of IPS Empress and IPS Empress 2 are a glass-ceramic produced by the Ivoclar company[7,8], they have completely different chemical compositions as follows: 1) IPS Empress ceramic (leucite reinforced glass-ceramic): 63 SiO_2, 17.7 Al_2O_3, 11.2 K_2O, 4.6 NaO_2, 0.6 B_2O_3, 0.4 CeO_2, 1.6 CaO, 0.7 BaO, and 0.2 TiO_2 in wt%. 2) IPS Empress 2 ceramic (lithium disilicate glass-ceramic): 57.0~80.0 SiO_2, 11.0~19.0 Li_2O, 0~13.0 K_2O, 0~11.0 P_2O_5, 0~8.0 ZnO, 0~5.0 MgO, 0.1~6.0 La_2O_3, 0~5.0 Al_2O_3, and 0~8.0 additional components in wt%.[4] This means they have different physical properties. That is, the IPS Empress 2 ceramic has much higher flexure strength than IPS Empress ceramic.[9] This high strength of IPS Empess 2 ceramic could be explained by the high crystal content of more than 60 vol%, the multi-directionally, densely packed interlocking microstructure of elongated rod-shaped crystals, and the increase in the size of the lithium disilicate crystals after the heat-pressing operation.

The IPS Empress 2 glass-ceramic is a heat-pressed, lithium disilicate material using the "lost-wax" principle.[7] The heat-press procedure is conducted in the EP500 heat-press furnace. A pressing temperature of 920℃ at a holding time of 20 min and an effective pressure of 20 bar lead to viscous flow of the glass-ceramic ingot. The flexure strengths of IPS Empress 2 glass-ceramic pressed with as-received ingots(334 MPa) and their sprue buttons(338 MPa) were a little lower than the results estimated by Frank et al(350 MPa)[10], Fischer and Marx[11], and Oh et al(357 MPa).[9] This difference was result depending on the test method and fabrication of the test specimens. In the present study, the fracture toughness of both group was no significant difference and was in agreement with the result(3.2 MPa · $m^{1/2}$) found by Kappert.[12]

The SEM micrograph of IPS Empress 2 glass-ceramic pressed with as-received ingots and their sprue buttons showed no difference and exhibited a similar microstructure as Frank et al[10] reported. The IPS Empress 2 glass-ceramic has two kind of cystal: the main rod- shaped lithium disilicate crystals (length up to 6 ㎛; diameter up to 1㎛) and the secondary lithium orthophosphate crystals (0.1 to 0.3 ㎛). We observed the lithium disilicate crystal with an interlocking pattern, but could not observed the lithium orthophosphate crystals. This result might be explained as the glass matrix dissolving the thin surface layer during the etching procedure.

CONCLUSION

In the respect of mechanical property, this study demonstrated the reliability of reused lithium disilicate glass-ceramic of IPS Empress 2. Although there were no tests including color stability, casting accuracy, etc., the result of this study implied that we could reuse the sprue button of the pressed lithium disilicate glass-ceramic of IPS Empress 2 system.

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