# **Biaxial Flexural Strength and Microstructure Changes of Two Recycled Pressable Glass Ceramics**

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<u>Purpose</u>: This study evaluated the biaxial flexural strength and identified the crystalline phases and the microstructural features of pressed and repressed materials of the glass ceramics, Empress 1 and Empress 2.

<u>Materials and Methods</u>: Twenty pressed and 20 repressed disc specimens measuring  $14 \text{ mm} \times 1 \text{ mm}$  per material were prepared following the manufacturers' recommendations. Biaxial flexure (piston on 3-ball method) was used to assess strength. X-ray diffraction was performed to identify the crystalline phases, and a scanning electron microscope was used to disclose microstructural features.

<u>Results</u>: Biaxial flexural strength, for the pressed and repressed specimens, respectively, were E1 [148 (SD 18) and 149 (SD 35)] and E2 [340 (SD 40), 325 (SD 60)] MPa. There was no significant difference in strength between the pressed and the repressed groups of either material, Empress 1 and Empress 2 (p > 0.05). Weibull modulus values results were E1: (8, 4.7) and E2: (9, 5.8) for the same groups, respectively. X-ray diffraction revealed that leucite was the main crystalline phase for Empress 1 groups, and lithium disilicate for Empress 2 groups. No further peaks were observed in the X-ray diffraction patterns of either material after repressing. Dispersed leucite crystals and cracks within the leucite crystals and glass matrix were features observed in Empress 1 for pressed and repressed samples. Similar microstructure features—dense lithium disilicate crystals within a glass matrix—were observed in Empress 2 pressed and repressed materials. However, the repressed material showed larger lithium disilicate crystals than the singly pressed material.

<u>Conclusions</u>: Second pressing had no significant effect on the biaxial flexural strength of Empress 1 or Empress 2; however, higher strength variations among the repressed samples of the materials may indicate less reliability of these materials after second pressing.

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INDEX WORDS: lithium disilicate, leucite, biaxial flexural strength, repressing

THE POSSIBILITY of producing more vibrant dental restorations using all-ceramic systems has gained considerable attention from many clinicians and patients, because of these materials'

unique features, including esthetics, low thermal conductivity, abrasion resistance, and biocompatibility.<sup>1</sup> However, the widespread application and reliability of these materials has been dictated, until recently, by the credibility imparted to traditional porcelains by the metallic substrates.<sup>2</sup> Furthermore, all-ceramic dental materials, like any other ceramics, are inherently fragile in tension, and may be affected by microcracking, flaws, and defects that may be introduced during thermal treatment or fabrication procedures.

All-ceramic restorations are submitted to intermittent forces during fabrication and mastication. It is, therefore, important to evaluate their behavior under load. Mechanical properties such as strength and fracture toughness are the first parameters assessed to understand the clinical potential and limits of a dental ceramic;<sup>3</sup> however, other factors, including fatigue during

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functioning, restoration design, precise fabrication process, and skills of individual dental technicians, may affect the reliability and clinical performance of all-ceramic restorations.<sup>4</sup>

A wide range of materials and systems is currently available for the construction of all-ceramic restorations. One class among these systems requires hot pressing by means of a special furnace to produce the required shape (pressable materials). During the last 10 years, heat pressing has become a common technique in dentistry for the fabrication of all-ceramic prostheses.<sup>5</sup> In addition to its simplicity, this technique promotes better crystalline dispersion within a glass matrix,<sup>6</sup> less porosity,<sup>7,8</sup> and better marginal adaptation<sup>9</sup> compared to other techniques, such as sintering. Empress 1 (E1; leucite reinforced glass-ceramic; Ivoclar-Vivadent, Schaan, Liechtenstein) and Empress 2 (E2; lithium disilicate glass-ceramic; Ivoclar-Vivadent, Schaan, Liechtenstein) are well known and increasingly utilized all-ceramic dental materials. The reliability of El as an all-ceramic material suitable for the fabrication of single units, such as inlays, onlays, and crowns, has been recommended by many mechanical and clinical investigations.<sup>8,10-22</sup> According to the manufacturer's recommendations, E2 has been used as a core material suitable for the construction of 3-unit fixed partial dentures up to the second premolar. The improved mechanical properties of this material, 20-25 compared to most other pressable ceramics, are attributed to its chemical composition, which is comprised of dense multielongated lithium disilicate crystals within a glass matrix. In such a structure, a crack would be trapped by these distributed crystals, resulting in an improved strength and fracture toughness.<sup>21</sup> In previous studies, this material also showed good results in vivo and in vitro.<sup>26-30</sup>

Both E1 and E2 are available in ingots of several different shades and transparencies to match various clinical situations. These ingots are pressed into a mold by an Alumina plunger under pressure from a pneumatic press furnace. After pressing and cooling, the sprues are removed, along with the remaining material (button). The buttons should be discarded and a new ingot should be used for a new pressing. However, it has been reported that these materials are recycled in some dental laboratories; sufficient knowledge about the safety and consequences of such treatment is not available. Whether these buttons can be repressed and recycled successfully has been questioned. Concerns have also been expressed regarding the change in the microstructure and possible degradation of the mechanical properties of these materials, as a result of multiple processing and subsequent heat firing.

The aim of the present study was to appraise the biaxial flexural strength and describe the microstructural features and crystalline phases of repressed materials of E1 and E2, and to compare them to those of singly pressed materials.

# **Materials and Methods**

Twenty perspex disc samples measuring 14 mm (diameter)  $\times$  1.1 mm (thickness) were sprued and attached to muffle bases with surrounding paper cylinders. The samples were then invested using a total of 200 g investment powder and 52 ml of liquid, 40 ml special investment liquid and 12 ml of distilled water. Mixing was carried out for 60 seconds manually and 60 seconds under vacuum, then the mixtures were poured into a cylinder under vibration to prevent the formation of air bubbles. The cylinders were allowed to set for 30 minutes. The refractory molds with the E1 ingots and the Alumina plunger were heated at a rate of 9°C/min from room temperature to 850°C; this temperature was held for 90 minutes. When the preheating cycle was complete, the ingots were inserted into the molds, and the preheated plunger was placed on top and then transformed to the pressing furnace (Programat EP 500, Ivoclar-Vivadent) and the pressing cycle was started. The pressing was performed at a temperature of 1,175°C and a pressure of 5 bars, with a 20 minute hold and 40 minute pressing. After pressing, the investment molds were removed from the furnace and allowed to air cool. The specimens were then carefully devested using an air abrasion unit (Kavo EWL, Type 5423; Kavo, Biberach, Germany) with 50  $\mu$ m glass beads at a pressure of 3 bars. The sprues were separated from the disks using a diamond-cutting wheel saw (Isomet, Buehler Ltd., Lake Bluff, IL). E2 disk specimens were prepared following the same procedure utilized for E1. However, E2 ingots were not preheated along with the moulds, but inserted at room temperature into the preheated refractory mould and the pressing cycle was started at 920°C and pressure of 5 bars with a 20 minute hold and 30 minute pressing. To remove the reaction layer of the E2 specimens, they were immersed in invex liquid (Invex liquid, Ivoclar-Vivadent) in an ultrasonic unit (Ultrasonic cleaner; Unisonics, Manly Vale, New South Wales, Australia) for 10 minutes and then rinsed and dried; this was followed by aluminum oxide sandblasting 50  $\mu$ m at 1 bar pressure. The remaining buttons of E1 and E2 were adjusted by grinding to allow proper insertion into the refractory moulds. The same procedures were then followed to press these buttons.

All specimens were serially wet ground with 220, 320, 500, and 600 grade silicon carbide paper mounted on a metallographic lapping machine (RotoPol-22, Struers A/S, Rodovre, Denmark). All specimens were cleaned using an ultrasonic bath for 15 minutes at 90°C, followed by washing in detergent and boiling water. Specimen dimensions were 14 mm (diameter) × 1 mm (thickness). All specimens were fired in a porcelain furnace (Programat P100; Ivoclar-Vivadent) according to the manufacturer's recommended firing cycles to simulate laboratory procedures and release all stresses associated with polishing procedures.

For microstructure investigations, 2 specimens of E1 and E2, pressed and repressed, were finely polished further using 4, 2, and 1  $\mu$ m diamond paste, cleaned in ethanol, etched with HF acid, 0.1% for E1 and 10% for E2, and coated with platinum 20 nm. A field emission (scanning electron microscope; JSM 6000 FSEM, Joel, Tokyo, Japan) was used for the microstructural examination.

#### **Biaxial Flexure Strength**

Piston on 3-ball test was utilized to determine the biaxial flexure strength. The test was carried out using a universal testing machine (Shimadzu Autograph AG-G, Kyoto, Japan) at a crosshead speed rate of 0.5 mm/min until failure occurred. The disk specimens were supported on 3 symmetrically spaced balls (6 mm distant from the center of the jig). A thin plastic sheet (0.05 mm thick) was placed between the piston (1.5 mm diameter) and the specimen to facilitate even load distribution. Testing was performed at room conditions (22°C, and 66% relative humidity). The maximum tensile stress, which corresponds to the biaxial flexure strength, was calculated according to the equation suggested by the test standard (ASTM F 394-78)<sup>31</sup> as follows:

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

where S is the maximum tensile stress, P is the load at fracture and d is the specimen thickness at fracture origin. X and Y were determined as follows:

$$X = (1 + \nu) \ln(B/C)^{2} + [(1 - \nu)/2](B/C)^{2}$$
$$Y = (1 + \nu)[1 + \ln(A/C)^{2}] + (1 - \nu)(A/C)^{2}$$

where  $\nu$  is the Poisson's ratio, A is the radius of the support circle, B is the radius of the tip of the piston, and C is the radius of the specimen. Poisson's ratio for both materials, 0.23 and 0.24 for E1 and E2, respectively, was taken from a previous investigation.<sup>20</sup>

#### Weibull Modulus

Strength variation among each group was evaluated by calculating the Weibull modulus (m). A computer was used to rank the biaxial strength data in ascending order and appoint a rank over the range 1 to N (N is the number of specimens); a straight line was then fitted through the points using the median rank regression method. The following equation was used to calculate the Weibull modulus:

$$P_f = 1 - \exp\left[-(\sigma/\sigma_0)^m\right] \tag{1}$$

where  $P_f$  is the failure probability,  $\sigma$  is the strength at a given  $P_f$ ,  $\sigma_0$  is the characteristic strength, and *m* is the Weibull modulus. However, since  $P_f$  can be identified by the following relation:

$$P_f = j/(N-1) \tag{2}$$

where j is the rank in strength and N is the number of specimens, equation no (1) can be rewritten as follows:

$$1/(1 - P_f) = 1/\exp\left[-(\sigma/\sigma_0)^m\right]$$
(3)

Accordingly, plotting  $\ln[1/(1-P_f)]$  against  $\ln$  (strength) will provide a slope with the value of the Weibull modulus.<sup>2,32</sup>

#### X-ray Diffraction

X-ray diffraction (Diffractometer D5000, Siemens, Germany) was carried out to investigate and determine the crystalline phases in the pressed and repressed samples of both materials. Samples measuring 14 mm (diameter)  $\times 1$  mm (thickness) were placed in the holder of a Siemens diffractometer and scanned using Cu K $\alpha$  X-ray from 20° to 40° 2 $\theta$  degrees; a step size of 0.04° and 5 s-step interval was used.

#### **Statistics**

One-way analysis of variance (ANOVA) was used for the result comparisons. Pairwise *t*-tests were also carried out at overall significance level 0.05 respecting the Bonferroni adjustment.

### Results

Biaxial flexural strength results, Weibull modulus, standard deviation, and coefficient of variation for the 2 materials are listed in Table 1. The biaxial strength results were: E1: [148 (SD 18), 149 (SD 35)], and E2 [340 (SD 40), 325 (SD 60)] MPa for the pressed and repressed groups, respectively. One-way ANOVA revealed no significant difference between the 2 tested pressed and repressed groups for the 2 materials E1 and E2 (p > 0.05). All biaxial strength data were ranked in an ascending order and the resultant Weibull modulus for E1 and E2 specimens was tested as pressed and repressed, respectively were: E1 (8, 4.7) and E2 (9, 5.8). Probability of survival for all data was plotted versus the ranked strength values (in ascending order) for the 2 groups of each material (Figs 1A and B).

X-ray diffraction patterns of E1 groups showed that leucite was the main crystalline phase, with the background intensity signals indicating the presence of an amorphous phase, the glass matrix. The main leucite peaks were detected at  $2\theta$  values of 25.89°, 27.22°, 30.41°, and 31.36° with the main peak at 27.22° matching the (400) crystallographic plane of the tetragonal phase (Fig 1C). After repressing, peaks remained unchanged and no further peaks were detected, which denotes no change in the crystalline phase.

Lithium disilicate was the main crystalline phase for E2 groups. The major peaks of the lithium disilicate crystals ( $\text{Li}_2\text{Si}_2\text{O}_5$ ) were observed at  $2\theta$  values of  $23.79^\circ$ ,  $24.33^\circ$ , and  $24.84^\circ$ , with the dominant peak (highest intensity) at  $24.33^\circ$ , which corresponds to the (040) crystallographic plane of the monoclinic phase, as predicted from the X-ray diffraction standards file (40-0376), lithium silicate. The lithium orthophosphate ( $\text{Li}_3\text{PO}_4$ ) peaks were detected at  $2\theta$  values of 22.3 and 23.1. X-ray diffraction patterns of the pressed and repressed materials are shown in Figure 1D.

Scanning electron microscopic observations of the E1 pressed material show a dispersal of various shapes and sizes (0.5 to 3.5  $\mu$ m in diameter) of tetragonal leucite crystals in a glass matrix (Fig 1E). Microfracturing was frequently seen in the glass matrix, and in areas of larger leucite crystals (Fig 1F). Twinning of the leucite crystals

**Table 1.** Biaxial Flexural Strength, Standard Deviation, Coefficient of Variation, and Weibull Modulus of E1 and E2

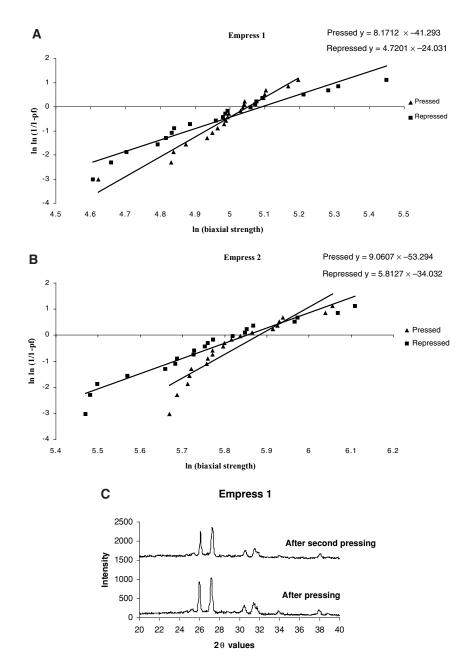
Material	Biaxial	Coefficient of	Weibull
	Strength (SD)	Variation %	Modulus
Empress 1 Pressed Repressed Empress 2 Pressed Repressed	148 (18) 149 (35) 	$     \begin{array}{r}                                     $	$     \frac{-}{8}     4.7     \frac{-}{9}     5.8 $

was repeatedly observed in both small and large leucite crystals (Fig 1E). The repressed material demonstrated similar microstructural features to those of the pressed material (Fig 2A). Both materials also demonstrated a similar distribution of crystals within the glass matrix, which indicates that a better crystal distribution was not achieved following second pressing (Figs 2B and C). However, the presence of microcracking damage, associated with scratching that was not completely eliminated by polishing, was noticed within a glass matrix in both pressed and repressed materials (Fig 1F).

Scanning electron microscopic observations of E2 also showed similar microstructural features in the pressed and repressed materials (Figs 2D and E). Numerous elongated lithium disilicate crystals were present within a glass matrix after the first pressing, measuring approximately 3 to 5  $\mu$ m long (Fig 2D). However, the lithium disilicate crystals of the repressed material appeared larger than those of the pressed material; these were in the range of approximately 7.5 to 8.5  $\mu$ m in length (Fig 2E).

# Discussion

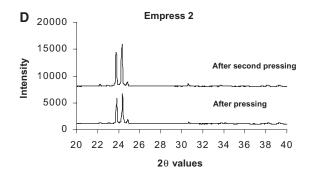
Microstructural investigations revealed a continuous glassy matrix, which did not appear to change following repressing, in E1 materials. The size and shape of the tetragonal leucite crystals were also very similar in both materials, pressed and repressed. Microcracking was present within the glass matrix and more prominent surrounding larger leucite crystals. Such damage has been linked to the significant difference in the coefficient of thermal expansion between leucite crystals and the glass matrix. This creates internal tension within the crystals and compensating compressive stresses within the glass matrix upon cooling. Furthermore, cracks are deflected around the leucite crystals as a result of these radial tensile and compensating hoop stresses.<sup>25,33</sup> The effect of larger leucite crystals on the degree of microcracking has been addressed previously by Mackert et al. (2001).<sup>34</sup> These investigators reported that the microcracking in leucite containing porcelain could be minimized by decreasing the mean leucite crystals. Shareef et al. (1994) noted that smaller particle size enhanced a homogenous distribution of the leucite crystals



**Figure 1.** (A) Illustration of the Weibull plots of E1 pressed and repressed material. (B) Illustration of the Weibull plots of E2 pressed and repressed material. (C) X-ray diffraction traces of E1, showing similar patterns for the pressed and repressed samples, with the main leucite peaks occurring at approximately the same positions and heights. (D) X-ray diffraction traces of E2, showing similar patterns for the pressed and repressed samples, where the main lithium disilicate peaks occurred at approximately the same positions and heights. (E) SEM photomicrograph of E1 after pressing, showing various shapes and sizes of dispersed leucite crystals in a glass matrix, and twinned crystals. (F) SEM photomicrograph of E1 after pressing, showing areas of accumulated cracks surrounding the leucite crystals.

within the glass matrix, and consequently there was less evidence of glass matrix microcracking.<sup>35</sup>

Twinning of leucite crystals was observed in both pressed and repressed specimens. This was attributed to the shear deformation associated with relief of the shear strains during the transformation from the cubic to tetragonal phase at  $625^{\circ}$ C.<sup>5</sup> No leucite crystal agglomerates were





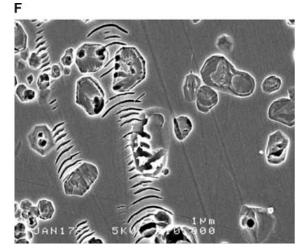
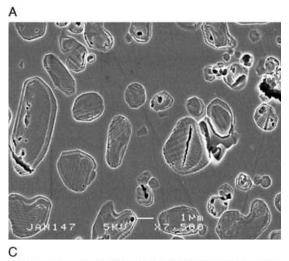


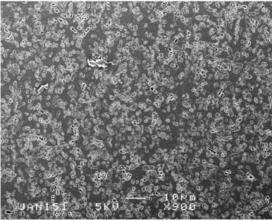
Figure 1. continued

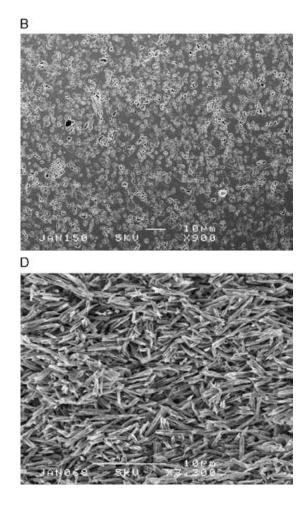
noticed, and similar crystal dispersion within the glass matrix can be seen. These microstructural features may signify that repressing did not noticeably affect the microstructure of the repressed samples.

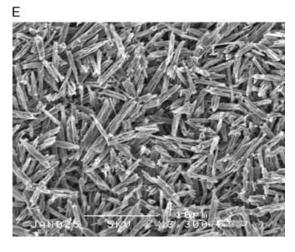
The main crystalline phase of E2 material, pressed and repressed samples, is lithium disilicate. These elongated crystals were present in the glass matrix, and appeared to form an interlocking pattern in some sites; however, the lithium disilicate crystals in the repressed material were seen to be larger than those of the pressed samples. This behavior is called Ostwald ripening<sup>36</sup> and is common for all precipitated materials. It takes place when the microstructure coarsens and liberates surface energy excess due to the solubility of small particles. As a consequence, larger grains are expected to grow at the expense of those small particles. The present results are similar to previously reported findings by Oh et al. (2000),<sup>37</sup> who noticed that the lithium disilicate crystals, after pressing, were approximately double the size of those before pressing. No further homogenous crystal distribution was noticed after repressing; however, crystal alignment can be observed in some sites. The orientation of lithium disilicate crystals of E2 material, as a result of viscous deformation of the glass matrix phase, occurred during sprue extrusion as previously reported.<sup>21</sup> This preferred crystal orientation was less prominent in the repressed materials, which showed a more interlocking pattern than the pressed material. Another crystalline phase, lithium orthophosphate, could not be identified in the SEM images, perhaps due to its greater solubility upon acidic etching.

The strength value of E1 obtained in this study was not significantly affected by repressing the material. This result supports the insignificance of changes found in the microstructures of these materials. It is also comparable to results achieved in previous studies;<sup>7-9</sup> however, some areas showed severe glass cracking and damage (Fig 1F). Such damage is thought to result from sliding deformation and damage of individual particles of the SiC grit paper with E1 surface. This creates relatively small partial Hertzian cone cracks, which became more visible with the aid of acidic etching, behind the sliding grit contacts.<sup>38</sup> The significant residual stress accrual in E1 material during cooling, due to the prominent thermal expansion mismatch between leucite crystals and the glass matrix, can also cause localized damage within the glassy matrix surrounding these crystals. For larger leucite crystals, these defective sites may become detrimental to the strength and could be sites of catastrophic failure, especially when they are in areas of high tensile stresses.









**Figure 2.** (A) SEM photomicrograph of E1 after repressing, showing similar microstructure features to that of E1 after pressing, where glass areas, leucite crystals, and twinning can be seen. (B) SEM of E1 at lower magnification, showing dispersed leucite crystals in a glass matrix. (C) SEM photomicrograph of E1 after repressing, showing similar crystals distribution to that of E1 after pressing. (D) SEM of E2 after pressing, showing many elongated lithium disilicate crystals protruded from a glass matrix. (E) SEM of E2 after repressing, showing larger lithium disilicate crystals than that of after pressing, taken at the same magnification of E2 after pressing.

The improved mechanical properties of E2 material over that of E1 are attributed to the unique interlocking microstructure of densely packed high content of lithium disilicate crystals, 70%.<sup>24</sup> A recent study reported higher crystalline content of E2 material, reaching 90%.<sup>23</sup> It is well known that higher crystalline content tends to improve the mechanical properties of ceramic materials;<sup>39</sup> however, the behavior of the crystals during either heat treatment or external forces can also play a major role in the mechanical properties. In a previous study by the present investigators, it was shown that a preferred orientation of these crystals after pressing may occur and cause fracture toughness anisotropy. This alignment may also result in overestimation of the fracture toughness and strength if stresses, during testing, are applied parallel to these aligned elongated crystals.<sup>21</sup>

X-ray diffraction was used in this study to evaluate the effect of second heat pressing on the crystalline phases of both materials. The results for both E1 and E2 showed similar X-ray diffraction traces for the pressed and repressed samples of each material. These results support other findings, namely mechanical testing and the microstructure features, which suggest that repressing did not cause a significant change in the studied materials, E1 and E2.

Flaws and microcracks may develop during the processing of brittle materials or may occur as a result of residual stresses within the microstructure during heat treatment. The influence of these flaws and defects on strength measurements can cause large variations in the strength data. Weibull modulus is used to describe the variation of the strength results. The lower the value of Weibull modulus, the greater the variability of the strength data which, in turn, points to more flaws and defects of the material, and unreliability.<sup>32</sup> In fact, Weibull modulus values are also affected by the method adopted to finish specimens and test environment, because of possible influences on residual flaw sizes and subcritical crack growth. In this study, both materials demonstrated lower Weibull modulus values after repressing. During repressing, flaws might have developed as a result of occasional porosity entrapment between the remnant buttons used for repressing. This may create large pores or cracks that significantly degrade strength; however, since the mean strength value was not significantly affected, this indicated that only a few specimens, among those tested, were affected.

## Conclusion

Within the limitations of the present study, it may be concluded that second pressing of both materials did not affect their mechanical strength; however, greater strength scatter of the repressed samples of both materials may indicate less reliability of these materials after repressing. Further studies and/or testing of other mechanical properties, such as Elastic modulus or fracture toughness, may provide a better understanding of the effect of repressing on the mechanical properties of the pressable materials, E1 and E2.

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