Degree of conversion and surface hardness of bulk-fill composite versus incremental-fill composite

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Abstract

Objective: The aim of this study was to evaluate the degree of conversion and surface hardness of two bulk-fill composites and one incremental-fill composite.

Methods: Bulk-fill composites (x-tra fil, Voco; QuiXfil, Dentsply) and incremental-fill composite (Grandio, Voco) were used. Twenty five cylindrical specimens (5 × 4 mm) were made from each material in Teflon molds. Mold was filled in one increment for the bulk-fill composites and in two increments for the incremental-fill composite. Specimens were stored dry in dark at room temperature for 24 h before testing. Degree of conversion (DC) was determined using Fourier transform infrared spectroscopy (FTIR). A microhardness tester was used to measure the Vickers hardness number (VHN) on top and bottom surfaces of each specimen. Data for DC and VHN were analyzed by ANOVA and pair-wise Newman–Keuls test.

Results: X-tra fill recorded significantly the highest DC, while no significant difference was noted between the other two composites. The VHN mean values of all composites tested were significantly different from each other (P < 0.0001), either in top or bottom surface, with Grandio showed the highest mean value and QuiXfil showed the lowest mean value. Only QuiXfil recorded no significant VHN difference between its top and bottom surfaces. There was no significant difference in bottom/top hardness ratio among materials. Non significant Correlation between VHN and DC was noted.

Conclusions: X-tra fil showed the most DC performance. Incremental-fill composite showed higher VHN than bulk-fill composites. Differences in DC and VHN values among materials proved to be a material dependent.

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Keywords: Bulk-fill composite; X-tra fil; Grandio; QuiXfil; Degree of conversion; Surface microhardness

1. Introduction

Resin-based composites have been successfully used in dentistry for many years and widely replaced amalgam as a posterior restorations [1]. Dental composites are expected to have mechanical properties comparable to those of tooth enamel and dentin and...
provide a long life of service [2,3]. However, several factors limit the performance of composites, especially depth of cure and degree of conversion (DC) [2,4–6].

In spite of great advances in resin based composite technologies, an insufficient depth of cure is one of its major disadvantages [7]. Due to insufficient depth of cure, incremental placement technique, with a maximum 2 mm thickness, was used for large composite restorations, especially class II restorations [8]. However, the use of dental composite in an incremental placement technique, and light curing each increment individually is time consuming for the patient and the operator [9]. There is also an increase possibility of air bubble inclusion or moisture contamination between individual increments of resin composite restorations [10].

Recently, a new class of resin-based composite, the so-called “bulk-fill” composites have been introduced into the dental market with the purpose of time and thus cost savings [9]. The unique advantage of this new material class is stated that it can be placed in a 4 mm thickness bulks to be cured in one step instead of the current incremental placement technique, without adverse effect on polymerization shrinkage, cavity adaptation, or degree of conversion. Furthermore, the manufacturers stated that the polymerization shrinkage of those materials is even less than that of commonly used flowable and conventional resin-based composites [11]. Consequently, problems arise from polymerization shrinkage could be reduced [12]. This new material class includes flowable and high viscosity (paste) material types.

Adequate polymerization transforms the monomers into a complex polymer structure. Monomer conversion into polymers does not attain 100%, but results in monomers that remain unreacted. Resin composites start the polymerization process by absorbing light in a specific range of wavelength around 400–500 nm; once activated, react with the aliphatic amine to produce free radicals. The number of double carbon links (C=C) present in the monomers, which are converted into single links (C–C) to form the polymer chains during the polymerization process, is called degree of conversion [13,14].

For achieving long-term durability of dental composites, it is important that most of their monomers converted into polymers during polymerization reaction. Unfortunately, the dimethacrylate polymer exhibit considerable unsaturated monomers in the final product [15–18].

There are several contributing factors that can influence the DC such as light source used [18], power density [6], wavelength [19], irradiation time [4], light-tip size [6], photo-activation method [20], chemistry of organic matrix formulations [15–17], distribution and quantity of inorganic fillers [21], the type and quantity of the photo-initiator [22], and color of the composite resins [9].

The Physical and mechanical properties of dental composites are directly influenced by the degree of conversion achieved during polymerization [23]. Lower degree of conversion provides composites with an inferior mechanical properties and greater discoloration and degradation [24] and as a result, restorations with poor wear resistance and poor color stability [25].

In the study of Tiba et al. [26], multiple bulk-fill (flowable and high viscosity) and incremental-fill resin composites were evaluated regarding depth of cure to be acceptable according to international standard 4049 [27]. Three of the high viscosity bulk-fill resin composites (SonicFill, Kerr; Tetric EVO Ceram Bulk Fill, Ivoclar-Vivadent; Alert Condensable Composite, Pentron), one flowable bulk-fill composite (Filtek Bulk Fill Flowable Restorative, 3M ESPE), and one incremental-fill composite (HelioMolar HB, Ivoclar-Vivadent) did not achieve adequate depth of cure according to the standard. However all other materials tested either high viscosity bulk-fill resin composites (QuiXfil and x-tra fill) or flowable bulk-fill composites (SureFill SDR flow, Dentsply; Venus Bulk Fill, Heraeus–Kulzer; x-tra base, Voco) and incremental-fill composite (Filtek Supreme Ultra Universal Restorative, 3M ESPE) attained the depth of cure claimed by the manufacturers and accepted by the standard. In another study, the DC of Tetric EvoCeram Bulk Fill (high viscosity bulk-fill composite) and x-tra base (flowable bulk-fill composite) were evaluated by FTIR spectrometer and the mentioned DC values of these materials were 41.4% and 43.8%, respectively [28]. In another work, the DC of nine of the available bulk-fill (flowable and high viscosity) composites were measured and it was found a great diversity in the results with the DC ranged from the lowest, 43.6%, for Filtek Bulk Fill (flowable) to the highest, 76.5% for SonicFill (high viscosity) [29].

Surface hardness is one of the most important properties used to compare restorative materials, and is defined as the resistance to permanent indentation or penetration [30]. It is a mechanical property of the restorations that should always be taken into account, especially when they are faced with large areas of masticatory force [5,6]. Substantial surface microhardness of the restoration is one of the main requirements especially in posterior stress-bearing areas [23].

One of the most important factors that affect dental restoration is that it undergoes wear during function or
whereas being cleaned [30]. As wear is due to abrasion, surface hardness is an essential property. It is the mechanical property most frequently used to characterize the wear resistance of materials. A material that has a higher surface hardness, in general, considered to be more wear resistant [31].

In one study, based on hardness results of bulk-fill resin composites, as a material class, the authors classify this material as between the flowable resin composites and the hybrid resin composites. The results of a previous study on hardness of bulk-fill materials (Tetric EvoCeram Bulk Fill and x-tra base) confirmed that both materials enable at least 4 mm thick increments to be cured in one step [28]. In the same study, it was mentioned that x-tra base, although being a low viscosity bulk-fill material, showed a higher VHN than that of Tetric EvoCeram Bulk Fill, a high viscosity bulk-fill material. Tiba et al. [26] assessed the Knoop hardness ratio of several bulk-fill versus incremental-fill resin composites and stated that all bulk-fill composites tested, except one (Alert Condensable Composite) exhibited adequate hardness ratio that is comparable to that of the conventional incremental-fill composites. However, in other investigation, it was reported that the surface hardness of some of bulk-fill composite materials was significantly decreased after ethanol storage, which raises distress about long-term stability of these materials [29].

Many studies have reported a good correlation between the hardness and degree of conversion [32-34]. On the contrary, no correlation was found between DC and microhardness of several resin composites [35,36]. In addition, one study mentioned that polymers with the same DC displayed different hardness numbers [37].

In the dental literature, there are few studies about the DC and surface hardness of these new bulk-fill resin composites; in most of them, these properties were measured for the flowable bulk-fill, and very few for the high viscosity (paste) bulk-fill composites. The aim of this study was to evaluate the DC and surface microhardness of two high viscosity bulk-fill composites in comparison to one another and to one regular incremental-fill composite.

2. Materials and methods

Two types of composite resin indicated for bulk-fill (X-tra fil³ and QuiXfil⁴) and one composite resin indicated for incremental-fill (Grandio³) were used in
this study. The list of composites, types, shades, compositions, and manufacturers are given in Table 1.

Twenty five cylindrical specimens (5 mm diameter × 4 mm thickness) of each tested material were prepared in a Teflon split mold. A mylar strip was placed on a 10 mm thick glass plate and the Teflon mold was placed over it. For each of the bulk-filled composites, the material was packed in bulk inside the mold until it was slightly overfilled. A second mylar strip was placed over the composite resin and another glass slide, 1 mm thickness, was slightly compressed to extrude excess material. Photoactivation was performed by positioning the light guide tip to be in contact with the glass slide on the top surface of the specimen. Each specimen was irradiated according to the manufacturer instructions for 10 s at 850 mw/cm² light intensity with LED curing unit (blue phase C). The output power of the curing unit was verified regularly every five exposures using a light radiometer. Standardization of the distance between light source and specimen was obtained by the thickness of the glass slide and mylar strips which gave smooth surfaces for the specimens.

The specimens for the incremental-filled composite were prepared with the same method except that the material was packed inside the mold cavity in two increments (2 mm each) and each increment was cured from the top surface for 20 s, according to the manufacturer instructions. The shade used for x-tra fil and QuiXfil (bulk-filled composites) was universal shade as those brands have no other shades, while that used for Grandio (incremental-filled composite) was B2 as it has no universal shade. B2 shade was selected as it is one of the most commonly used shades clinically.

After photo-activation, the specimens were stored dry in dark container at room temperature for 24 h before testing. Five specimens of each composite material were used for conducting the DC test (n = 5) and the remaining twenty specimens were used for measuring surface microhardness (n = 20).

2.1. Degree of conversion test

Fourier transform infrared spectroscopy [FTIR] was used to evaluate the degree of conversion. Each of the polymerized specimens (n=5) of each composite was milled into a fine powder with a mortar and pestle. Fifty micrograms of the powder was mixed with 5 mg of potassium bromide powder and pressed to produce a thin disc, which placed in a specimen holder and transferred to the spectrophotometer. The absorbance peaks were recorded using the diffuse-reflection mode of FTIR under the following conditions: 32 scans, over a wave length of 400–4000 cm⁻¹ and a resolution of 4 cm⁻¹.

Unpolymerized specimens (n=5) of each composite resin were smeared onto thin potassium bromide discs, placed into a cell holder in spectrophotometer, and then a spectrum was obtained with the same parameters as for the polymerized specimens.

Degree of conversion was determined by estimating the changes in peak height ratio of the absorbance intensities of aliphatic C=C peak at 1638 cm⁻¹ and that of an internal standard peak of aromatic C=C at 1608 cm⁻¹ during polymerization, in relation to the uncured material. DC % for each specimen was calculated using the following equation:

\[
\text{DC\%} = \left(1 - \frac{(1638 \text{ cm}^{-1}/1608 \text{ cm}^{-1}) \text{ cured}}{(1638 \text{ cm}^{-1}/1608 \text{ cm}^{-1}) \text{ uncured}}\right) \times 100\%
\]

2.2. Vickers hardness test

The Vickers hardness number (VHN) was determined on the top and the bottom surfaces for each specimen using a microhardness testing machine (Micro Hardness Tester) equipped with a diamond pyramidal microindenter to apply a load of 50 g for 30 s at room temperature. Each surface of the specimen (top and bottom) was divided into 4 equal quadrants; one indentation took place for each quadrant. The VHN for each surface was recorded as the average of the four readings. Vickers hardness ratio (VHR) for each material was calculated according to the following equation:

\[
\text{VHR} = \left(\frac{\text{bottom VHN mean value}}{\text{top VHN mean value}}\right) \times 100\%
\]

| Composite       | DC % (SD) | ANOVA (P value) |
|-----------------|-----------|-----------------|
| x-tra fil       | 67.74 (2.65)^A | 0.0027^*        |
| QuiXfil         | 48.68 (5.30)^B |                |
| Grandio         | 56.76 (6.30)^B |                |

Means with the same superscript letters were not significantly different at P < 0.05. *Significant (P < 0.05).

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5 Polydentia, Switzerland.
6 Ivoclar Vivadent.
7 Certified Dental Supply LLC, Nutley, NJ, USA.
8 (JASCO, FT/IR-4100, JAPAN).
9 Model HV-1000, China.
10 Campina Grande, Paraiba state, Brazil.

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2.3. Statistical analyses

The data for DC and VHN were statistically analyzed with one-way ANOVA. Multiple comparisons were made by using pair-wise Newman-keuls test.

Correlation between VHN and DC were indicated by regression analysis. Statistical analysis was performed using Assistat 7.6 statistics software for windows. \(^{10} \) P value \(< 0.05\) was considered to be statistically significant in all tests.

3. Results

3.1. Degree of conversion (DC)

DC mean values and standard deviations (SD) for all composite materials investigated are presented in Table 2.

One-way ANOVA revealed a statistically significant difference in DC among the materials tested \((P < 0.05)\). x-tra fil, bulk-fill composit, recorded the highest significant DC in comparison to the other two composites \((P < 0.05)\). Although QuiXfil, bulk-fill composite, had the lowest DC, but it was not significantly different from that of Grandio, incremental-fill composite, \((p > 0.05)\).

3.2. Vickers microhardness

Table 3 shows the VHN mean values for the top and bottom surfaces of all materials tested. VHRs were also presented in the same table.

The significant differences in VHN mean values among materials either in top or bottom surfaces were identical. One-way ANOVA followed by multiple comparison tests indicated that the VHN mean values of the three composites were significantly different from each other \((P < 0.0001)\) with Grandio showed the highest mean values and QuiXfill showed the lowest mean values.

3.3. Top vs. bottom

The VHN mean values of the top surfaces for x-tra fil and Grandio materials were found significantly greater \((P < 0.05)\) than those of their bottom surfaces. However, no significant difference in VHN mean values \((p > 0.05)\) was noted between top and bottom surfaces of QuiXfil material.

3.4. Vickers hardness ratio (VHR)

The highest VHR was found for QuiXfil \((99.2\%)\), followed by x-tra fil \((90.6\%)\), and the lowest value was recorded for Grandio \((86.5\%)\). No significant difference in VHR was noted among materials \((p > 0.05)\).

3.5. Correlation between VHN and DC

No significant correlation was presented between DC and VHN results as indicated by regression analysis \((r = 0.1376, r^2 = 0.0189, p = 0.06249)\). This correlation is also shown in Fig. 1.

![Fig. 1. Linear chart presenting correlation between VHN and DC.](image-url)
4. Discussion

Currently, there is a growing trend among practitioners to use bulk-fill resin based composite materials because of their more simplified procedures [28]. Manufacturers mentioned that the main advancement of bulk-fill composite materials, namely increased depth of cure, which probably results from higher translucency [11], and low polymerization shrinkage stress are related to modifications in the filler content and/or organic matrix [29] with the help of advanced technology.

Adequate polymerization all over composite resin restorations is one of the main important factors influencing their clinical success. The degree of conversion is an important tool to estimate the physical, mechanical and biological properties of composite resin restorations [6,19]. Higher degree of polymerization is an essential factor for obtaining superior physical and mechanical properties [19,38].

Inadequate polymerization might lead to marginal microleakage [3], discoloration [39], and decreased bonding strength [2] of resin composite restorations. A lower degree of conversion might, also cause increase in the amount of released unreacted monomer, leading to less biocompatible restorations [40,41]. In addition, unevolved functional groups can act as plasticizers, producing restorations with inferior mechanical properties [22,42]. Furthermore, oxidation and hydrolytic degradation caused by monomer trapped in the restoration might result in discoloration and accelerated wear [43].

In this study, DC of the investigated composites were assessed using FTIR spectroscopy because it has been widely used as an appropriate and reliable method as it detects the C=O stretching vibrations directly before and after curing of the composite resins [6,13,20]. Because the polymerized specimens need to be pulverized this method is time consuming and can’t determine the depth of cure [17,44].

According to many previous studies, several Bis-GMA based resin composite materials exhibited considerable unreacted monomers in the final restorations, with a degree of conversion were in the range of 52−75% with most of the materials were in the range of 55−60%, under conventional irradiation methods [6,15,16,18]. The minimum DC% for clinically acceptable restoration has not yet been exactly recognized [6]. Soares et al. [45] reported that, for occlusal restorative layers, DC values should be at least 55%, which conceded to the results of our study concerning x-tra fil and Grandio. However, QuiXfil failed to fulfill the accepted minimum value of DC in Soares et al.’s study. In a previous study [28], although the DC of Tetric EvoCeram Bulk Fill was measured with similar parameters like those in this study, it recorded a lower DC than that of both bulk-fill composites in our study and this may be attributed to the differences in materials composition (chemistry of organic matrix formulations and filler characteristics). In another work [29], the DC values recorded for x-tra fil and Grandio were 62.1% and 62.8%, respectively, which are different than those obtained for the same materials in the present study, where they were 67.74% and 56.76%, respectively. This difference could be related to the variations in method of measurement, specimens thickness and irradiation intensity, where in that study DC was measured using a Raman Spectrometer on the upper sample surface of 2 mm thickness that was light-activated by four 40 s overlapping irradiations.

The main two characteristics of a monomer that influence the DC are the initial monomer viscosity and flexibility of its chemical structure [46]. In the present study, x-tra fil (bulk-fill composite) showed significant higher DC than Grandio (incremental-filled composite), although they both have almost the same weight% of organic matrix. This might be attributed to the different chemistry of their organic matrix. The organic matrix of x-tra fil consists of Bis-GMA, UDMA, and TEGDMA, while that of Grandio consists of Bis-GMA and TEGDMA. It was reported that the ultimate degree of conversion of different monomer systems increases in the following order: Bis-GMA < Bis-EMA < UDMA < TEGDMA [47]. Bis-GMA is considered the most viscous and least flexible monomer due to the strong intramolecular hydrogen bonding of its hydroxyl groups (-OH) on the backbone and the presence of rigid aromatic nuclei interactions given by aromatic rings in its structure. UDMA is also a viscous monomer due to the hydrogen bond intramolecular interaction between its Amine (−NH−) and carbonyl groups (−C=O). However, the viscosity of UDMA is much lower and flexibility is higher in relation to Bis-GMA because of the weak hydrogen bond of its Amine group compared to that of hydroxyl groups [48]. Also, the presence of Amine groups in the urethane structure of UDMA monomer is responsible for the characteristic chain transfer reactions that provide an alternative path for the continuation of polymerization. These reactions result in increased mobility of radical sites on the network and consequently enhanced polymerization and monomer conversion [47]. This explains the increase reactivity and higher DC of UDMA.
containing organic matrix of x-tra fill when compared to that of Bis-GMA containing organic matrix of Grandio.

Although QuiXfil is UDMA based Composite, its DC was significantly lower than that of x-tra fill and insignificantly less than that of Grandio composite resins. This is probably because polymerization characteristics are influenced by the variety of organic matrix chemistry [49] and by the concentration of each monomer in the matrix [20]. TEGDMA has the lowest viscosity and the highest DC among the different monomer systems as mentioned earlier. When Bis-GMA is diluted with the low viscosity TEGDMA monomer, a synergistic effect on the rate of polymerization and DC has been observed [47]. This might explain the significant high DC of x-tra fill than that of QuiXfil since it may contain a higher concentration of TEGDMA monomer. Sgarbi et al. [20] showed that resin composites that have Bis-GMA and TEGDMA as organic matrix in their composition can contain larger quantity of TEGDMA which was considered the main contributor to polymerization after irradiation.

Another contributing factor that might affect the DC of resin composite is the filler particles size. DC decreased in composites whose filler particles size closer to the wavelength of the activating light. This is due to the scattering effect of fillers of this size which reduces the amount of light transmitted through the resin composite [50]. Therefore, the light scattering produced by nanofillers may adversely affect the physical properties of nanofilled composites [51]. The larger particles of hybrid composites have a greater depth of cure as they less affected by light scattering [52]. It was, also reported that fillers with sizes approaching half of that wavelength used for irradiation showed increased scattering [53–55]. The bigger filler size of x-tra fill, compared to that of Grandio, decreases the total filler surface and, consequently, the filler matrix interface. Thus, light scattering at the filler–matrix interface is reduced allowing more light to penetrate the material and to better cure the composite in depth. This finding was, also noted in a previous study [28] and this is might be another reason that could explain, to some extent, the significant higher DC of x-tra fill in comparison to that of Grandio in the present study. However, this is not the case for QuiXfil despite of its bigger filler size, where its DC not significantly different from that of Grandio, which may be contributed to the involvement of other parameters, such as type of organic matrix monomers and ratios (as mentioned before), and filler particles morphology and distribution, which vary greatly between products [50,57,58].

Surface hardness of the tested materials in this study was assessed using Vicker's hardness test as it is easy to apply and the data obtained are reliable [6]. The diamond indenter used in the procedure does not deform over time and is reportedly suitable for measurement the hardness of fragile brittle materials [59].

In this study, significant high differences were noted in VHN mean values among all tested resin composite materials, either in top or bottom surfaces, with the highest VHN value was obtained for the incremental, nanohybrid composite (Grandio) in comparison to that of both bulk-filled composites (x-tra fill and QuiXfil). At the same time, there was a significant difference between the two bulk-filled composites. These results are supported with previous findings of Leprince et al. [29]. In that study, it was found that Grandio had a significant higher VHN value than that of several high viscosity bulk-filled composites, namely x-tra fill, Sonic Fill, Tetric EVOceram Bulk Fill, and Xenius (GC, Europe). In the same study, there were significant differences in VHN values among all bulk-filled materials. All these findings are in agreement to a great extent with our results. Several factors related to composition were reported to affect the surface hardness of resin composite restorative material [60]. It was reported that, mass fractions [34,56,61], size and distribution of filler particles have a significant effect on some physical and mechanical properties, including surface hardness [62–64]. It was also mentioned that other parameters such as, particle shape and density, monomer type and ratio, degree of polymers cross-linking, and photoinitiators seem to have a significant influence on surface hardness [56,64,65]. This could explain the different VHN values recorded among the materials tested in this study, in spite of they all have almost equal fractions of filler loading. The increased VHN value of the incremental-bulk filled composite compared to that of both bulk-filled composites, either for top or bottom surface, might also be related to the more total energy delivered to the incrementally filled composites [66].

Within each material, The VHN on bottom surfaces of x-tra fill and Grandio were significantly decreased, while that of QuiXfil not affected compared to that of their top surfaces. For any resin-based composite increment, the VHN on the top surface would be expected to differ markedly with VHN on the bottom surface as a result of monomer reactivity and filler/matrix refractive index mismatch [67]. As the bottom surface is more critically affected by the light intensity, it is considered as a better gauge of the effectiveness of cure of composite [20]. The results in one study...
indicated that bulk-fill resin composites were more translucent than conventional resin composites [63]. In other study, it was reported that a reason for an enhanced depth of cure of bulk-fill composites is considered to be an increased translucency, due to increased filler size [28]. This could explain why QuiXfil bottom VHN is not significantly different from its top since its filler particle size is larger than that of the other two materials. The insignificant difference of bottom VHN of QuiXfil from that of its top may, also be related to the use of Dynamic Curing Control (DCC) system, as stated by its manufacturer [68], which allows QuiXfil to achieve the perfect balance of a maximum depth of cure in 10 s and a prolonged working time under surgical lighting to 100 s (no more information are available so far about the DCC system).

According to previous studies [69,70] the curing efficiency or depth of cure could be measured by the ratio of bottom to top surface hardness values and a ratio of 80% has often been used as a minimum clinically acceptable value. In agreement with the results of preceding investigation [27] the hardness ratio of all materials tested in this study succeeded to fulfill this minimum value, without any significant differences among the ratios.

In the present study, there was no correlation between the DC and microhardness, which agreed with other investigations [35,71]. Other studies showed a negative correlation between the DC and hardness [31–33]. In another study, though surface hardness showed a good correlation with the DC of a specific resin composite, hardness values can’t be used to predict the DC when different resin composites are compared [72].

5. Conclusion

Among tested composite materials, x-tra fil showed the most DC performance. However, Incremental-fill composite (Grandio) showed the highest VHN than both bulk-fill composites (x-tra fil and QuiXfil). No significant correlation was detected between DC and surface hardness and all materials had sufficient VHR. The reported curing depth of 4 mm for the bulk-filled composite can be verified from the VHN data analysis. Variation of organic matrix chemistry formulations and filler features (mass fraction, density, and particle size, shape, and distribution) contribute to significant differences in DC and VHN values among materials tested, so the susceptibility to differences in these properties proved to be a material dependent.

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