Factors influencing shear strength of incrementally cured composite resins

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Factors influencing the shear strength of incremental curing of three different composite resins were examined. The first increment was cured under different surface conditions, in air, or under a coverglass. A separate experiment studied the effect of curing in nitrogen atmosphere. There was no difference in the shear strength of the specimens if the first increment was created in air or under a coverglass. One material (Heliomolar RO) showed higher shear strength when cured in increments and one when bulk-cured (Herculite XR). Curing increments in a nitrogen atmosphere increased the shear strength for two of the three materials tested (P50 and Heliomolar). The results showed that the bond strength between the increments of composites was influenced by the surface properties of the composites themselves after they had been cured under the different conditions; it was also affected by the inherent chemical and physical properties of the materials utilized. ☐ *Bond strength; dental bonding; oxygen inhibition*

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Light-cured composite materials are today frequently used for minor class-II restorations and, to an increasing extent, also for larger class-II fillings in molars. Posterior composite fillings are usually built up in two or more layers, in which the increments have generally been placed with uncovered surfaces and light-cured from the level of the occlusal surface. Some operators, however, use a round condenser point or similar instruments to put pressure on the composite and the matrix band while curing, to improve the contact with the adjacent tooth.

Improved contact can also be attained by using a transparent cone attached to the curing wand. For curing, the cone is pressed down into the composite material and the light from the wand is distributed hemispherically through the first layer of material. This technique has also been shown to achieve more efficient curing in the deep parts of the cavity and to reduce both the cervical gap and the cusp deflection in class-II restorations (1–3). This technique is widely used throughout Scandinavia.

It is important to attain a substantial and consistent bond strength between the layers in multi-layer fillings. When curing the first layer in immediate contact with metal, plastic, or other materials that prevent direct access to air oxygen, the conditions for bonding will be changed. Several authors have studied the influence of matrix bands or other coverings that might affect the oxygen-inhibited surface layer, which often is proposed to be essential for attaining sufficient bond strength. Boyer et al. (4) found no loss of bond strength when the first layer of a two-paste composite was cured in contact with a plastic matrix, as compared with curing in air, if the second layer was added immediately after the first layer had set. Lloyd et al. (5), using two-paste composites, and Boyer et al. (6) and Forsten (7), using light-cured composites, all found no significant differences between layered specimens and the cohesive strength of the same materials, even though the first layer had been cured against plastic surfaces followed by direct addition of the second layer. Chin et al. (8), however, measured higher bond strength in samples cured with access to air than in samples cured against a highly polished amalgam surface. Additionally, Eliades & Caputo (9) investigated the oxygen-inhibited zone and found it to be primarily composed of catalyst-free monomer. They also measured the bond strength under different conditions and found that when the inhibited zone was left untouched, the bond strength was significantly lower than the cohesive strength, but it increased considerably when the catalyst-free monomer was removed with acetone.

The previously published results are thus contradictory, and the purpose of this study was therefore to further investigate the role of air oxygen in determining bond strength between composite increments.

Materials and methods

Two hybrid composites, Herculite XR, A3, batch 405361 (Sybron/Kerr, Romulus, Mich., USA) and P50, A3, batch 9350U (3M, St Paul, Minn., USA), and one microfilled composite, Heliomolar radiopaque, A3, batch 614642 (Vivadent, FL-9494 Schaan, Liechtenstein), were used in the studies. A Luxor model 4000 (ICI Dental) curing lamp was used for curing procedures. The light intensity was checked regularly with a curing radiometer (CL-tester, Dendema AB, Stockholm, Sweden).

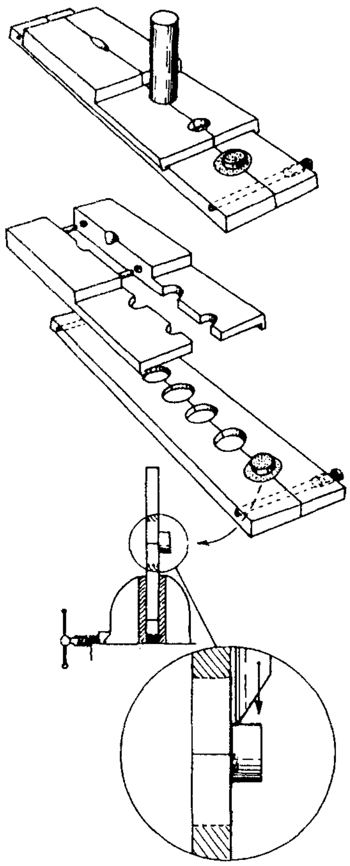

Fig. 1. Schematic illustration of the metallic molds for specimen preparations and testing.

The specimens were made in demountable brass molds, comprising separate parts for each of the two composite layers. The mould for the first layer held five samples, which were 4 mm in diameter and 3 mm high, whereas the second layer formed by the upper mold measured 2.75 mm in diameter and 2 mm in height (Fig. 1). The second mold could be locked in position over the sample in the basic mold. For reference samples cured in one piece, the basic mould had cut-outs 2.75 mm in diameter, conforming to the upper mould.

Specimen preparation

Five different groups of samples were prepared, with 50 samples being made for each of the materials in each group.

Group I. The first sample form was filled with composite paste to the level of the surface of the mold. A thin cover glass (0.15 mm) for microscopic use was placed over the composite. With the glass in place, the composite was cured for 30 sec. The cover glass was removed, and the second mold was placed over the cured composite, pressed down to a firm contact, and filled with composite. After curing for 30 sec the upper mold was separated, the next space in the basic mold was filled with composite, and the procedure was repeated.

Group II. The basic mold was filled and cured in the same manner as in group I, but without a cover glass. Instead, a washer, 0.15 mm thick and with a 4-mm cut-out in the center, was placed on the mold to support the curing-wand in the same position and distance as in group I.

Group III. The upper mold was placed on the basic mold with 2.75-mm-diameter form spaces, and the forms were aligned and filled with composite paste, which was then cured for 60 sec, resulting in an all-in-one cured control specimen.

Group IV. The samples were made as in group II but in a closed chamber (Safetech Ltd, Cleansphere, Calif., USA), where the working area was manipulated with airtight plastic gloves. The curing lamp was placed in the chamber, where a constant air flow was maintained.

Group V. The samples were made under the same conditions as in group IV, but instead of air, a flow of nitrogen was led into the chamber for 15 h before and during sample preparation. The composite materials were kept in a nitrogen atmosphere for 12 h before testing.

Owing to the illumination required for precision work in the chamber, the temperature tended to increase. For that reason the nitrogen or air entering the chamber was cooled, and the temperature close to the molds checked with an electronic thermometer (Schwille, Germany). The temperature was kept within 24 ± 4 °C, since closer control was not possible in the relatively small chamber (0.053 m^3) available.

All samples were immersed in deionized water at 37 °C for 24 h before the shear test was performed. The brass mold containing the samples was placed in a universal material testing machine (Fig. 1) (Alwetron 50T, Stockholm, Sweden). The outer parts of the samples were sheared off one by one with a blunt tool, operating at a constant distance from the basic mold and with a cross-head speed of 1 mm/min.

The mean values and the standard deviations for the different groups were found to be normally distributed and were analyzed for significant differences by using an unpaired Student's *t* test (two-tailed). $P < 0.05$ was used as the level of significant difference.

Results

The results are presented in Figs. 2 and 3. No significant difference was observed between the incrementally cured composite materials cured under free access to air oxygen (group II) and the samples cured with a coverglass over the first layer (group I). The bond strength of Herculite XR in groups I and II was significantly lower (−31% and −35.5%, respectively)

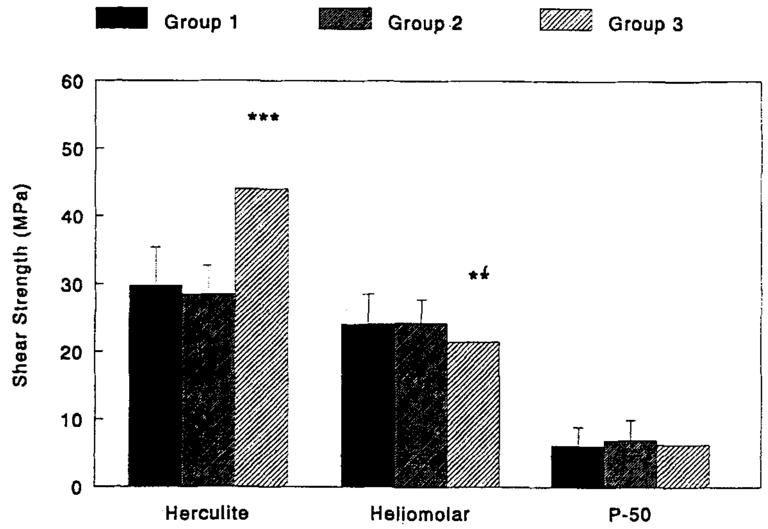

Fig. 2. Shear strength of specimens cured with (group I) and without (group II) glass cover on the first composite layer and bulk-cured controls (group III) (**$P < 0.01$; ***$P < 0.001$).

than the bond strength in group III (control group). In the case of Heliomolar, the bond strength of group III was significantly lower than those of groups I and II ($P < 0.05$) (+12% and +12.8%, respectively). For P50 no significant difference was found between the three groups.

Specimens cured in a nitrogen atmosphere (group V) had significantly higher bond strengths than the specimens cured in air (group IV) in the cases of Heliomolar and P50. For Herculite there was no significant difference between group IV and group V. The bond strength of P50 was consistently lower than that of Herculite and Heliomolar.

Specimens of Herculite cured in the chamber with a constant air flow (group IV) had a significantly higher bond strength than the specimens cured in free air (group II). For Heliomolar and P50 no significant difference between group IV and group II was found.

Discussion

The reactivity of the free radicals to oxygen is higher than their reactivity to the monomers of dental composite. Therefore, when curing in contact with oxygen, a thin uncured film is formed on the surface of cured dental composites. This film has been claimed to contribute to the bond strength of an added composite layer (8). Data presented by Eliades & Caputo (9), however, demonstrated that the inhibited layer consists

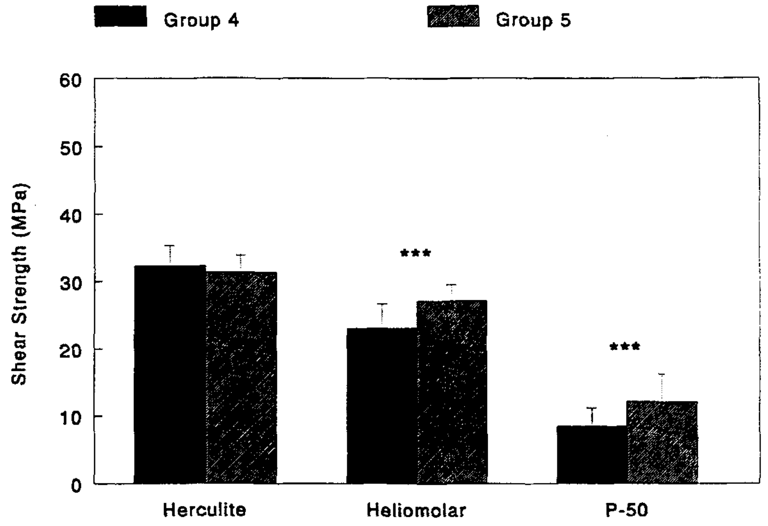

Fig. 3. Shear strength of specimens cured in a closed chamber under air (group IV) or nitrogen (group V) atmospheres (***$P < 0.001$).

of catalyst-free monomer and that the formation of a strong bond with a new composite layer either requires that the catalyst-containing resin of this composite penetrates the film to make contact with the solid surface below or that the film is removed by a method that does not destroy the remaining double bonds of the underlayer.

In agreement with several previous studies, there were no significant differences in bond strength between covered and uncovered samples (groups I and II). This does not, however, altogether exclude the influence of oxygen on the surface layer, since oxygen may to some extent be absorbed on the surfaces of the glass or plastic coverings. Additionally, it can be concluded that noteable differences in surface roughness of the uncovered samples compared with the glass-covered ones did not seem to enhance the bond strength. Other factors, such as the preservation of double bonds in the surface layer, appear to be of greater importance. Thus, it seems unlikely that the use of a device, such as the small plastic tip in contact with the composite material, will impair the bond of subsequent composite layers.

The purpose of studying curing under nitrogen was to further minimize the influence of air oxygen in the curing process. Since the presence of some oxygen in the composite and/or on the instrument surfaces cannot be excluded even after 15 h in nitrogen, and because no analyses of such oxygen remnants were made, the results are not definitely conclusive. However, it is interesting to note that the bond strengths of samples constructed in a nitrogen atmosphere were significantly higher for two of the materials than for corresponding samples made under otherwise identical conditions in air. One possible explanation could be that the polymer free radicals possess longer half-lives in nitrogen than in air. This might, in turn, lead to the formation of more covalent bonding between the increments, resulting in increased physical bond strength. However, this explanation may not be generally applicable, as one of the materials was not affected by the nitrogen environment. This material, Herculite XR, was also the only material to show significantly greater strength when cured in one piece (group III) than when cured in two increments (groups I and II). Heliomolar RO was significantly stronger when cured in increments, whereas for P50 there was no significant difference between the groups.

We noted a greater curing depth for Herculite than for Heliomolar and P50 during the testing. This is probably one of the reasons of the difference in behavior between the materials when bulk-cured. Since the fracture plane in the specimens was 2 mm from the illuminated surface, a curing depth of more than 2 mm was essential for achieving a strong bulk-cured specimen. In the case of Heliomolar and P50 a 2-mm curing depth was a critical limit, as recommended by the manufacturers. On the other hand, the difference in elasticity in fracture plane of Heliomolar specimens may contribute to the significantly lower bond strength of bulk-cured specimens than that of the incrementally cured specimens.

The significant difference between group II and group IV of Herculite was difficult to explain; it may be caused by the temperature increase, up to 28 °C, during the preparation of the group-IV specimens in the closed plastic chamber. Even though the air and nitrogen entering the chamber were cooled, it was not possible to keep a closer control of the temperature than 22–28 °C, with a tendency towards the higher level.

The ideal surface conditions for bonding between consequential layers of composite materials remain unknown and may vary for different materials. Specifications for posterior composite materials ought to include not only the curing depth but also the strength when cured in layers. Incremental build-up seems to be a useful and, in many cases, indispensable technique for posterior restorations and will contribute to good results, with or without direct access to air on curing, provided that the layers are of appropriate thickness and that the irradiation is adequate.

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