Effect of Time on Bond Strength in Indirect Bonding

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Abstract: The purpose of this in vitro investigation was to determine the influence of a reduced time interval before debonding on shear bond strength of stainless steel brackets bonded with a custom base indirect technique. A total of 135 bovine permanent mandibular incisors was randomly divided into nine groups of 15 specimens each. Three base composite-sealant combinations were investigated: (1) Phase II base composite, Custom I.Q. sealant, (2) Phase II base composite, Maximum Cure sealant, and (3) Transbond XT base composite, Sondhi Rapid Set sealant. Shear bond strength was measured for three different debonding time intervals: (1) time of transfer tray removal as recommended by the manufacturer, (2) 30 minutes after bonding of the sealant, and (3) 24 hours after bonding of the sealant. For groups bonded with Maximum Cure or Sondhi Rapid Set sealants, no influence of debonding time on shear bond strength was found. The Custom I.Q. sealant groups showed significantly lower bond strength measurements when debonded at the recommended tray removal time, and the Weibull analysis indicated a higher risk of bond failure at clinically relevant levels of stress. All base composite-sealant combinations showed acceptable bond strength at 30 minutes and 24 hours after bonding of the sealant. (*Angle Orthod* 2004;74:245–250.)

Key Words: Debonding time; Transfer tray removal; Indirect bonding; Bond strength; In vitro

INTRODUCTION

About 30 years ago, Silverman et al[1] introduced the indirect bonding technique. Most current indirect bonding techniques are based on a method described by Thomas,[2] ie, brackets with attached composite are bonded to the teeth with a chemically cured sealant: the unfilled catalyst resin is applied to the tray and the universal resin is painted on the etched enamel. The sealant is cured when the two components are brought in contact with each other on seating the tray in the patient's mouth. One of the criticisms of this method has been that curing of the sealant might be incomplete. Therefore, the "modified Thomas technique" was developed and uses a sealant that is mixed before placing the transfer tray on the tooth, thus ensuring complete mixing of the two components of the sealant.

Although bond strength measurements have been found to compare favorably with those of direct bonding,[3–5] difficulty in achieving consistent and predictable adhesion to the teeth and accidental removal of brackets with the tray might still present a problem in indirect bonding.[6,7] It has to be taken into consideration that in vitro bond strength testing is commonly performed 24 hours after the bonding procedure.[8] At this time, polymerization will be completed, and only minor changes in bond strength can be expected.[9,10] However, in clinical practice using indirect bonding techniques, the bond has to be able to withstand considerable forces during removal of the transfer tray only minutes after bonding, indicating that adequate bond strength shortly after sealant application is mandatory.

A number of indirect bonding studies have used bonding materials originally developed for direct bonding or for dental restorative purposes.[2–7,11–14] Chemically cured sealants were introduced recently for indirect bonding techniques.[15–18] Although bond strengths 24 hours after sealant bonding with these materials have been reported recently,[19] there is limited information available on bond strengths at the time of tray removal. Therefore, the aim of this in vitro investigation was to analyze the influence of a reduced time interval between bonding and debonding procedures on bond strength of indirect bonding resins.

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TABLE 1.   Description of Experimental Groups

Group Name	Sealant	Base Composite	Debonding Time	Number of Specimens (*n*)
C1	Custom I.Q.	Phase II	5 min	15
C2	Custom I.Q.	Phase II	30 min	15
C3	Custom I.Q.	Phase II	24 h	15
M1	Maximum Cure	Phase II	7 min	15
M2	Maximum Cure	Phase II	30 min	15
M3	Maximum Cure	Phase II	24 h	15
S1	Sondhi Rapid Set	Transbond XT	2.5 min	15
S2	Sondhi Rapid Set	Transbond XT	30 min	15
S3	Sondhi Rapid Set	Transbond XT	24 h	15

MATERIALS AND METHODS

Bonding procedure

A total of 135 extracted bovine permanent mandibular incisors was obtained from a local slaughterhouse and stored in 0.5% chloramine solution before the experiment. It has been shown that bovine enamel and human enamel have similar properties, and the adhesive strength of bovine enamel has been found to be equal or slightly lower compared with human enamel.[20–23] Teeth were randomly assigned to nine groups of 15 specimens. After cleaning the teeth with a brush and pumice-water slurry at slow speed, they were embedded in chemically cured dental acrylic (Palavit G, Heraeus Kulzer, Wehrheim, Germany) in plastic cylinders to allow standardized and secure placement during testing.

Maxillary central incisor 0.018-inch slot stainless steel mesh base brackets (Mini Mono, order no 0711-0103, Forestadent, Pforzheim, Germany) were used throughout the study. The average surface area of the bracket base was 13.5 mm². The indirect bonding technique was performed in the following manner: an alginate impression was obtained of each specimen and poured in orthodontic stone. On the dry model, the teeth were painted with diluted separating medium and allowed to dry for 24 hours. The bracket base was cleaned with alcohol. For a description of the experimental groups, see Table 1. In groups C1-C3 and M1-M3, chemically cured Phase II (Reliance Orthodontic Products, Itasca, Ill) adhesive was applied to the bracket to form the custom composite base. In groups S1-S3, Transbond XT adhesive (3M-Unitek, Monrovia, Calif) was applied to the bracket base before placement on the cast and was cured with a halogen curing light (Polylux II, Kavo, Biberach, Germany) for two minutes. This extended curing interval was chosen to achieve complete polymerization of the adhesive on the model. Transfer trays were made from vinyl polysiloxane impression material (Silagum AV-Putty soft, DMG, Hamburg, Germany). After the transfer tray material had set, the specimens were soaked in warm water for 30 minutes. The transfer trays were removed from the models. The composite adhesive on the custom bracket base was cleaned by sandblasting with 50 μm aluminum oxide for three seconds.

The teeth were etched with 37% phosphoric acid gel (Ormco, Orange, Calif) for 30 seconds, then rinsed thoroughly with water and air-water spray, and dried with compressed air for 20 seconds. For groups C1-C3, Custom I.Q. (Reliance Orthodontic Products) sealant was used. Groups M1-M3 were bonded with Maximum Cure sealant. Groups S1-S3 were bonded with Sondhi Rapid Set sealant (3M-Unitek). The sealants were used according to the manufacturers' recommendations. In groups C1-C3 and M1-M3, Plastic Conditioner (Reliance Orthodontic Products) was applied to the bracket base before bonding as suggested by the manufacturer.

After bonding was completed, the transfer trays were removed. Specimens in groups C2, C3, M2, M3, S2, and S3 were stored in distilled water. Specimens in groups C1, M1, and S1 were debonded immediately after transfer tray removal.

Debonding procedure

In groups C1, M1, and S1, debonding was performed at the time of tray removal as recommended by the manufacturer, ie, after 2.5 minutes for Sondhi Rapid Set, five minutes for Custom I.Q., and seven minutes for Maximum Cure (see Table 1). In these groups, specimens were placed in the testing apparatus immediately after tray removal. In groups C2, M2 and S2, brackets were debonded 30 minutes and in groups C3, M3, and S3, 24 hours after bonding of the sealant. The brackets were debonded with a Zwicki Z2.5 universal testing machine (Zwick, Ulm, Germany) at a crosshead speed of five mm/minutes. The plastic cylinders with the embedded teeth and the brackets were mounted on a joint and were aligned in the testing apparatus to ensure consistency in the point of force application and direction of the debonding force for all specimens. A stainless steel wire loop (0.020-inch diameter) was engaged under the occlusal bracket wings to produce a shear-peel force parallel to the bracket base in an occlusogingival direction. The load at failure was recorded.

TABLE 2. Shear Bond Strength (Mean, Standard Deviation) and Weibull Parameters

| | | | | Weibull Analysis | | | | |
Group	Mean (MPa)[a]	SD (MPa)	Group Differences*	Weibull Modulus	Correlation Coefficient	Characteristic Bond Strength (MPa)	Shear Stress at 5% Probability of Failure (MPa)	Shear Stress at 10% Probability of Failure (MPa)
C1	9.29	3.66	A	2.10	0.970	13.48	3.27	4.61
C2	13.90	3.56	B	2.98	0.966	18.40	6.80	8.65
C3	13.32	3.03	B	3.88	0.981	16.60	7.72	9.29
M1	15.10	2.00	A	7.05	0.981	17.15	11.25	12.46
M2	16.10	2.63	A	5.68	0.889	18.84	11.17	12.67
M3	14.70	1.70	A	7.63	0.927	16.56	11.22	12.33
S1	14.18	3.24	A	3.03	0.912	18.81	7.06	8.95
S2	15.34	2.66	A	4.81	0.966	18.42	9.93	11.53
S3	14.31	3.86	A	3.45	0.966	18.25	7.71	9.50

[a] MPa indicates megapascals; SD, standard deviation.

* Groups with the same letters are not significantly different from each other (Tukey, $P < .05$, based on one-factor ANOVA for groups C1-C3, M1-M3, S1-S3).
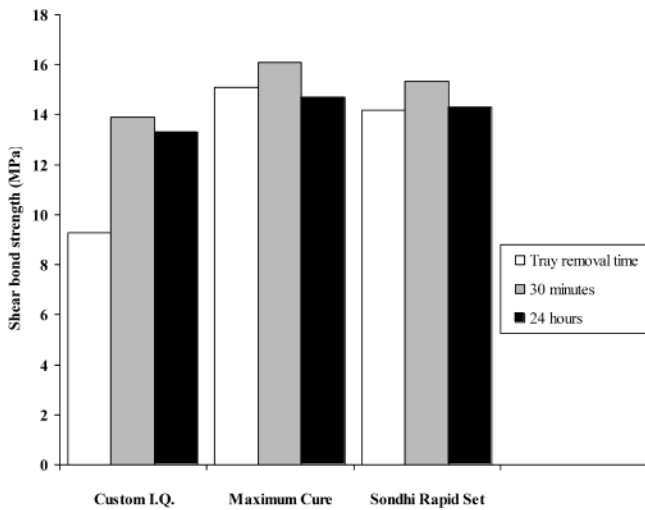

FIGURE 1. Means of shear bond strengths for the three sealants at the different debonding times.

FIGURE 2. Weibull distribution plots; groups C1-C3 (Phase II custom base composite, Custom I.Q. sealant).

For each specimen, the substrate surface was examined with an optical stereomicroscope (magnification 10×) and an adhesive remnant index (ARI) was determined.[24] ARI: 0, no adhesive left on the tooth; 1, less than half of the adhesive left on the tooth; 2, more than half of the adhesive left on the tooth; 3, all adhesive left on the tooth, with distinct impression of the bracket mesh. ARI scores were assessed by the same operator.

Statistical analysis

To calculate shear bond strength, the debonding forces (N) were converted into stress values (MPa) by taking into account the surface area of the bracket base. Bond strengths of the different groups were compared by two-way analysis of variance (ANOVA, $P < .05$) with the factors time of debonding and type of adhesive. A Weibull analysis was performed, and the Weibull modulus, characteristic bond strength, correlation coefficient, and the stress levels at 5%
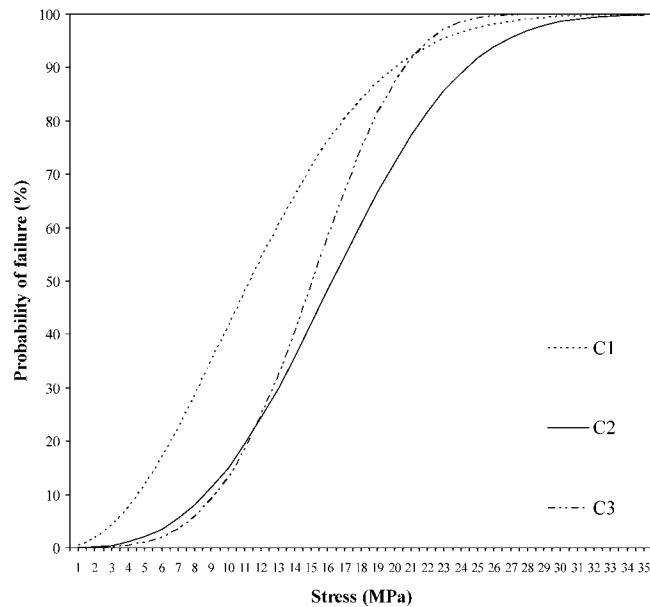
and 10% probability of failure were calculated. Kruskal-Wallis and Mann-Whitney nonparametric tests were used to determine whether there were any significant differences in the ordinal ARI values ($P < .05$).[25,26]

RESULTS

Mean shear bond strengths, standard deviations, and the parameters of the Weibull analysis (modulus, correlation coefficient, characteristic bond strength, and stress at 5% and 10% probability of failure) are given in Table 2. Figure 1 illustrates the mean shear bond strength values of the experimental groups. Figures 2, 3, and 4 show the Weibull distribution plots of the probability of failure at a certain shear stress level for groups C1-C3, M1-M3, and S1-S3, respectively.
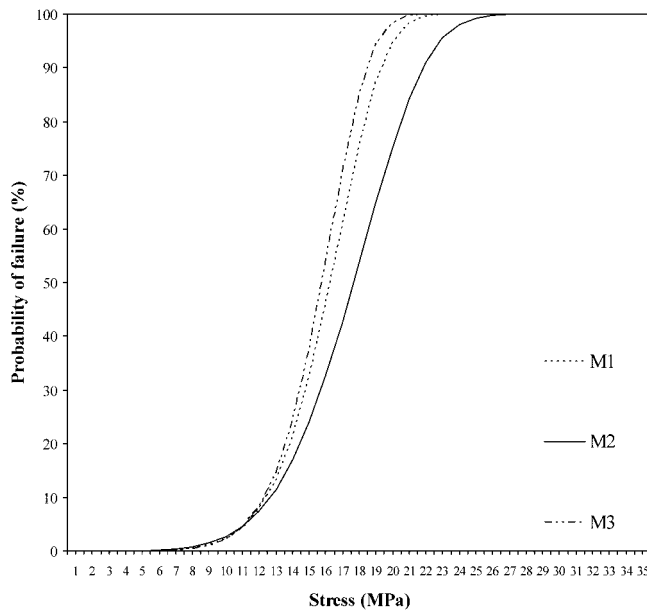
FIGURE 3. Weibull distribution plots; groups M1-M3 (Phase II custom base composite, Maximum Cure sealant).

FIGURE 4. Weibull distribution plots; groups S1-S3 (Transbond XT custom base composite, Sondhi Rapid Set sealant).

The two-way ANOVA indicated that there were significant interaction effects of the two factors, debonding time and adhesive type (F = 2.805, P = .029). Therefore, further ANOVA was carried out separately for the three adhesive types. The one-way ANOVA showed that there was no significant influence of debonding time on bond strength for groups M1-M3 and groups S1-S3. However, significant differences were present among groups C1-C3 (F = 8.023, P = .001). The post hoc Tukey test showed that bond strength for group C1 (9.29 $\pm$ 3.66 MPa) was significantly lower than for groups C2 (13.90 $\pm$ 3.56 MPa) and C3 (13.32 $\pm$ 3.03 MPa).

Means, standard deviations, and ranges of the ARI scores are given in Table 3. The Kruskal-Wallis test indicated that there were no significant differences among the groups (χ^2 = 3.853 and P = .146 for comparison of groups C1-C3, χ^2 = 3.802 and P = .149 for comparison of groups M1-M3, χ^2 = 0.696 and P = .706 for comparison of groups S1-S3).

DISCUSSION

In the present study, bond strength of three base composite-sealant combinations at different debonding time intervals was investigated. Two of the three sealants (Custom I.Q., Sondhi Rapid Set) were used according to the "original" Thomas technique, where the two components of the chemically cured sealant polymerize when they are brought in contact during placement of the tray in the patient's mouth. One sealant (Maximum Cure) was mixed before placement of the tray: this method is commonly referred to as the "modified" Thomas technique. It has been demonstrated in a previous study that both methods can result in
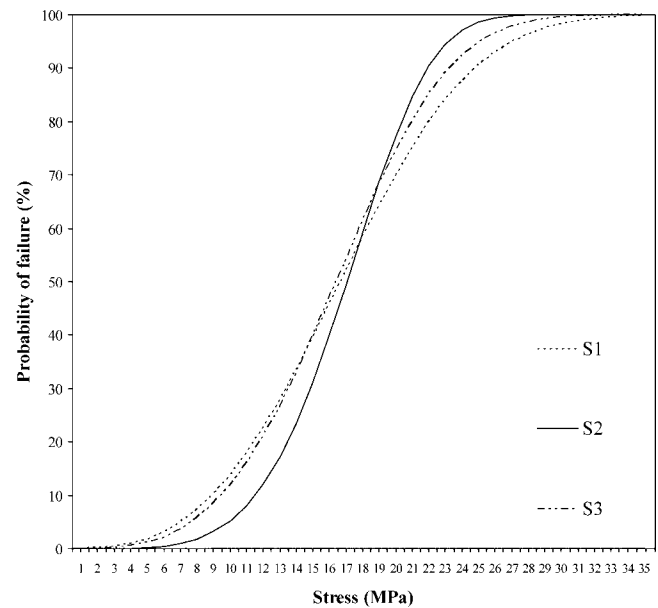
sufficient bond strength 24 hours after bonding.[19] The present investigation showed that composite-sealant combinations for both the original Thomas technique in groups S1-S3 and the modified Thomas technique in groups M1-M3 are able to obtain acceptable bond strength shortly after initiation of sealant polymerization.

Bond strength of a setting composite increases with time because of continued polymerization of the resin under the bracket base.[10,27,28] Previous studies indicate that lower strength may be present initially in chemically cured composite adhesives. When investigating composite adhesives in restorative dentistry, Braem et al[29] found a faster stiffness increase in light-cured composites compared with chemically cured composite adhesives during the first minutes after initiation of polymerization. Young's modulus was much lower at 10 minutes for most chemically cured composites. These authors concluded that composite resin fillings are vulnerable to distortion for at least 10 to 15 minutes after placement. Chamda and Stein[9] compared bond strength of light- and chemically cured composites and found higher bond strengths with light-cured resin two and five minutes after curing. For longer time intervals after bonding (10 minutes, 60 minutes, 24 hours), no difference in bond strengths between the two types of composite was found. Although all three sealants in the present study were chemically cured, bond strength measurements for Maximum Cure and Sondhi Rapid Set sealants only minutes after sealant polymerization were comparable with those obtained 30 minutes and 24 hours after bonding of the sealant. This indicates that the use of a chemically cured sealant for indirect bonding purposes in orthodontics does not preclude sufficient initial bond strength with this technique.

TABLE 3. Frequency Distribution of Adhesive Remnant Index (ARI) Scores

	ARI Scores[a]							
Group	0	1	2	3	Median	Mean	SD	Range
C1	—	13	2	—	1.00	1.13	0.35	1–2
C2	—	10	5	—	1.00	1.33	0.49	1–2
C3	—	8	7	—	1.00	1.47	0.52	1–2
M1	—	7	8	—	2.00	1.53	0.52	1–2
M2	—	8	7	—	1.00	1.47	0.52	1–2
M3	—	3	12	—	2.00	1.80	0.41	1–2
S1	—	8	7	—	1.00	1.47	0.52	1–2
S2	—	8	7	—	1.00	1.47	0.52	1–2
S3	—	6	9	—	2.00	1.60	0.51	1–2

[a] ARI: 0 indicates no adhesive left on the tooth; 1, less than half of the adhesive left on the tooth; 2, more than half of the adhesive left on the tooth; and 3, all adhesive left on the tooth, with distinct impression of the bracket mesh.

Bond strength testing so far has been performed predominantly at 30 minutes and at 24 hours after bonding.[8] Thirty minutes after bonding may be considered a typical time for stress induced on the bond when tying in the initial archwire. Bond strength at 24 hours after bonding may be important for the long-term survival of the bond and has been suggested to be the time when the composite reaches its maximum strength. However, many brackets are put to the test in vivo within a few minutes of bonding.[8] This is of particular importance in indirect bonding, where forces on the bond are applied during removal of the transfer tray shortly after polymerization of the sealant.

For the sealants used in the present study, removal of the transfer tray after initiation of sealant polymerization, 2.5 minutes for Sondhi Rapid Set, five minutes for Custom I.Q., and seven minutes for Maximum Cure, is recommended by the manufacturers. Bond failure at this stage results in a delay and extends chair time considerably. Therefore, ideally the clinician would like the sealant fully polymerized at this time to minimize the risk of bond failure. In the present study, at the time recommended by the manufacturer for removal of the transfer tray, the sealants Maximum Cure and Sondhi Rapid Set showed initial bond strengths comparable with those at 30 minutes and at 24 hours. However, for Custom I.Q., sealant bond strength at the recommended tray removal time was significantly lower and only approximately 67% of that 30 minutes after bonding.

In addition to the interpretation of mean bond strength values, the Weibull analysis gives the clinician an indication of how the material is likely to perform in the clinical situation.[8] Even for materials with a high mean bond strength value, there is a definitive measurable probability of failure occurring at relatively low stress levels.[30] Therefore, it is useful to apply a function that relates probability of bond failure to the level of stress. It has been implied that the force required to cause 5% bond failure is the type of information that has the most clinical relevance.[31] Littlewood

et al[32] suggested that the bond strength of a material with a 5% chance of failure should be at least 5.4 MPa. This recommended level of stress may also be useful in evaluating the risk of failure when the transfer tray is removed. In group C1, the shear stress for a 5% probability of bond failure was only 3.3 MPa (see Table 2) and hence markedly lower than the required shear stress level of 5.4 MPa, as suggested by Littlewood et al.[27] All the other experimental groups showed shear stress levels higher than 5.4 MPa at this probability of failure. It needs to be pointed out that in the present study, no bond failures on tray removal were noted in any of the specimens. Although it can be argued that this indicates a low risk of bond failure, it should be emphasized that the experimental setup using single-tooth trays for each specimen allows for very cautious removal of the tray. This might be different from the clinical situation where multitooth trays are typically used and result in higher forces on the bond on tray removal. Therefore, when using the Custom I.Q. sealant, special attention should be given to careful removal of the transfer tray.

It has been recommended that in vitro bond strength testing should be performed using a crosshead speed of 0.1 or 0.5 mm minute^{-1}.[8,33] This is done to ensure consistency and may facilitate interstudy comparison of results. Nevertheless, this crosshead speed lacks correspondence to clinical conditions.[33] In the present study, we attempted to simulate more closely the process of transfer tray removal, which is carried out faster. Therefore, a crosshead speed of 5 mm minute^{-1} was chosen. Although this higher speed may be more clinically relevant for the specific question addressed in our study, a possible influence of crosshead speed on bond strength needs to be taken into account when comparing the results with those of other investigations.[34]

ARI measurements of the experimental groups were not found to be significantly different from each other. No ARI scores of 0 or 3 were found in any of the specimens. This indicates that cohesive bond failures prevailed. The absence of ARI scores 0 and 3 may also be related to the fact that this was an in vitro investigation that allowed for adequate moisture control and therefore reduces the risk of failure at the enamel-adhesive interface. This might be different in a clinical environment. In addition, careful custom base preparation in the laboratory is likely to enhance adhesion of the composite to the bracket base, reducing the risk of failure between adhesive and bracket base.

CONCLUSIONS

The following conclusions can be drawn within the limitations of this in vitro study:

- Bond strength at 30 minutes after bonding of the sealant was comparable with that at 24 hours for all composite base–sealant combinations investigated.
- No significant differences in shear bond strengths were found for the bracket base composite-sealant combina-

tions, Phase II–Maximum Cure and Transbond XT–Sondhi Rapid Set, measured at the different debonding time intervals.
- Bond strength at the recommended time of transfer tray removal was significantly lower for Custom I.Q. sealant used in combination with Phase II composite. Care should be taken to limit forces on the bond during the first minutes after sealant polymerization when using this sealant.

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