

## **SHEAR BOND STRENGTH OF CERAMIC ORTHODONTIC BRACKETS TO CAD/CAM PROVISIONAL MATERIALS: INFLUENCE OF SURFACE TREATMENTS AND A NOVEL ADHESIVE SYSTEM**

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### **ABSTRACT**

**Objective:** To evaluate the effect of surface treatments and flash-free adhesive on the shear bond strength of ceramic orthodontic brackets to CAD/CAM provisional materials.

**Materials and Methods:** Specimens (n=160) from each provisional material (CAD-Temp and C-Temp) were categorized into four groups according to the surface treatment methods: C (no surface treatment), HP (37% H<sub>3</sub>PO<sub>4</sub>), DB (mechanical roughening by diamond bur) and SB (mechanical roughening by sandblasting). Half of the specimens in each group were bonded to one of the maxillary central incisor ceramic brackets according to the used adhesive system: (APC PLUS or APC Flash-free). All specimens were 5000-times thermocycled before the shear bond strength testing (SBS). Data were analyzed using three-way ANOVA, Tukey's multiple comparison tests. The adhesive remnant index (ARI) was also evaluated. The level of significance was set at 5% for all statistical tests.

**Results:** C-Temp significantly recorded higher SBS than CAD-Temp ( $p < .001$ ). DB and SB groups utilizing flash-free adhesive significantly recorded higher SBS ( $P < .05$ ) compared to other groups in the tested materials. Higher ARI scores were recorded in CAD-Temp and flash-free adhesive.

**Conclusions:** Bonding of orthodontic brackets to provisional restorations is a challenge for orthodontists in adult comprehensive cases that could be improved by an appropriate material, surface treatments, and adhesive system. Mechanical surface treatments and flash-free adhesive would enhance SBS of ceramic orthodontic brackets to CAD/CAM provisional materials. The higher ARI scores reported with CAD-Temp and flash-free adhesive reduces chair time for excess removal.

**KEYWORDS:** CAD/CAM Provisional material; Flash-free adhesive; Shear bond strength; Surface treatments

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## INTRODUCTION

Provisional restoration is an important element in fixed prothodontics, which protect dental surfaces from various oral environmental hazards until delivering the definitive restoration.<sup>1</sup> In addition, it could be used for long-term cases such as; oral implantation treatment, periodontal therapy, and orthodontic therapy or in situations involving comprehensive occlusal reconstructions.<sup>2</sup> Consequently, the challenge of effective orthodontic brackets bonding to provisional restorations may encounter the orthodontists in adult comprehensive cases<sup>3,4</sup>

Different types of provisional materials are available in the market. CAD/CAM is of a great interest to fabricate provisional restorations and to improve the material properties compared to conventional polymerization.<sup>5</sup> The provisional material type<sup>6,7</sup>, thermocycling<sup>4,8</sup>, surface treatments<sup>4,9</sup> and adhesive type<sup>10,11</sup> are among the aspects that could influence the bond strength to orthodontic brackets. A weak bond between orthodontic brackets and provisional restorations will lead to the high failure rate with adverse concerns on the cost and the patient comfort.<sup>4,9</sup> However, simple and appropriate means for pre-treating provisional restorations would be clinically encouraging to avoid de-bonding.<sup>12</sup>

Two bonding system is being utilized when directly placing orthodontic brackets; either by manual application or by a pre-coated bracket system in which the orthodontic adhesive applied to the bracket base. In both systems, flash removal step is needed to prevent the formation of rough surface and plaque accumulation that could consequently interfere with effective bonding.<sup>13,14</sup> Thus, 3M Unitek has developed a novel adhesive coated appliance system (APC Flash-free) to minimize flash amounts, to improve the bond strength and reducing the microleakage.<sup>15-17</sup> It is composed of a low viscosity resin applied to

a non-woven polypropylene mesh that attached to the orthodontic bracket base.<sup>17</sup> The bond strength of flash-free adhesive to CAD/CAM provisional material has not been investigated previously.

Therefore, this study aimed to evaluate the effect of surface treatments and flash-free adhesive on the shear bond strength of ceramic orthodontic brackets to CAD/CAM provisional materials. In addition, the adhesive remnant index (ARI) was evaluated. The null hypotheses tested were (1) the type of surface treatment (2) the type of CAD/CAM provisional material and (3) the type of adhesive does not affect shear bond strength.

## MATERIALS AND METHODS

Two types of CAD/CAM provisional materials (VITA CAD-Temp and C-Temp) as well as two types of maxillary central incisor pre-coated orthodontic ceramic brackets (APC PLUS and APC Flash-free) were used in the study (table 1). A sample size of 20 specimens in each group was required to give a 0.95 power using 0.05 level of significance according to the conducted power analysis (size effect=2.34,  $\alpha$ -two tailed=.05).

### Specimen Preparation and Grouping

One hundred sixty specimens (10x10x3mm) were cut from each type of CAD/CAM provisional material with an ISOMET (Techcut4, Allied, USA). Digital caliper (Mitutoyo Corporation, Tokyo, Japan) was used to ensure a uniform specimen thickness. Different grit sizes (600 to 1200 grits) of silicone carbide papers were used to finish the bonded surfaces of specimens under copious water-cooling followed by a 3 min ultrasonic cleaning with distilled water. The specimens were embedded in acrylic resin blocks exposing one surface for surface treatment methods and bonding. Specimens were categorized into four groups (n=40) according to the surface treatment methods performed on the provisional material surface as follows: C; no

TABLE (1) Materials used in the study

Product	Composition/ Manufacture	Indication	Lot. No.
CAD-Temp	- 83–86 wt. % PMMA, 14 wt. % microfiller (silica), Pigments (<0.1%). - VITA Zahnfabrik	Multi-unit, fully or partially anatomical long-term temporary bridges with up to 2 pontics	<b>38590</b>
Everest C-Temp	-Fibreglass-reinforced polymer. - KaVo, Biberach, Germany	Long-term temporary restoration up to 6 units	<b>6946</b>
APC PLUS adhesive coated orthodontic ceramic brackets	- Carboxylated methacrylate, fluoroaluminum silicate, Bis-GMA -3M Unitek (Monrovia, California, USA)	Orthodontic treatment brackets	<b>HW9AF</b>
APC™ Flash free adhesive coated orthodontic ceramic brackets	- A unique low viscosity methacrylate based resin with compressible nonwoven polypropylene fibers -3M Unitek (Monrovia, California, USA)	Orthodontic treatment brackets	<b>HU5ZX</b>

treatment (control), HP; surfaces were etched for 1 min with 37 % H<sub>3</sub>PO<sub>4</sub> gel (3M ESPE, St Paul, Minn, USA) then rinsed for 1 min, DB; surfaces were ground with a diamond bur under water cooling (medium grit, Komet Dental, GmbH & Co, KG, Germany) rotated at 45,000 rpm for 8 s<sup>18</sup> and SB; surfaces were air abraded with 50 µm aluminium oxide (LEMAT NT4, Wassermann, Germany) for 10 s at a distance of 10 mm with a pressure of 0.55 MPa then air-dried for 20 s.<sup>4</sup> Trans bond Plus self-etching primer (3M Unitek; Monrovia, California, USA) was applied to the treated surfaces according to the manufacturers' instructions.

### Bracket Bonding Procedure

Twenty specimens in each group were bonded to one of the ceramic brackets according to the used adhesive system : (APC PLUS or APC Flash-free). Half kg customized metallic tool was applied to the bracket top surface as standardized constant pressure to attain a uniform adhesive thickness. An explorer was used to remove the adhesive resin excess, only

in APC PLUS adhesive pre-coated bracket group. Ortholux Luminous curing Light (3M Unitek; light output: 1600 mW/cm<sup>2</sup>) was used to polymerize all adhesive resin for 12 s from two directions (6 s for each one) according to the manufacturers' instructions. To allow complete polymerization of the bonding material, specimens were kept in distilled water at 37°C for 24 h. Then all the groups were 5000-times thermocycled (SD Mechatronik Thermocycler, FT200, GmbH, Germany) between 5 and 55 °C with a 30 s dwell time before shear bond strength testing.

### Shear Bond Strength (SBS) Test

SBS test was conducted using a universal testing machine (Lloyd Instruments; Fareham, UK) at 0.5 mm/min crosshead speed until failure. After de-bonding, the residual adhesive on the provisional restoration surfaces were assessed by examining the fractured specimen using an optical stereomicroscope (Olympus SZ61, Tokyo, Japan) at 8 x magnification. The assessment was determined

using the modified ARI as described by Bishara and Trulove<sup>19</sup> and graded on a scale between 1 and 5 (1 all adhesive left on the provisional material surface with a distinct impression of the bracket mesh; 2 more than 90% of the adhesive left; 3 more than 10% of the adhesive left but less than 90%; 4 less than 10% of the adhesive left; 5 no adhesive left). The ARI were used to determine the bond failure sites between the provisional materials, the adhesive resin, and the bracket base.

### Scanning Electron Microscopy Evaluation

Three additional specimens from each group were produced in the same manner as in SBS test and cleaned with 96% ethanol in an ultrasonic bath for two minutes, then air-dried. Specimens were mounted on metallic stubs, gold sputter-coated, and evaluated under an SEM (Jeol-JSM-6510, Tokyo, Japan) with original magnification 500 x to detect topography of the treated surfaces.

### Statistical analysis

Data was first checked by the Shapiro–Wilk test for the normal distribution and was then analyzed by utilizing three-way ANOVA test considering three factors (the type of material, the surface treatments and the type of adhesive) to detect the interaction between the independent variables. One-

way ANOVA and Tukey's multiple comparison test were used to detect the significant difference in SBS regarding surface treatments. The overall SBS between the two adhesives was compared by independent sample t-test. The level of significance was set at 5% for all statistical tests. The Chi-square ( $\chi^2$ ) and Monte Carlo test were used to determine significant differences in the ARI scores at the 5 % level of significance.

### RESULTS

Independent variables (Material type, surface treatment methods and adhesive type) and their interactions were significantly affecting SBS as shown by the three-way ANOVA table (table 2). The mean and standard deviations of SBS values (MPa) are presented in (table 3). DB and SB groups significantly recorded higher SBS ( $P=.000$ ) compared to other groups for both types of materials. Flash-free adhesive showed higher significant SBS values only in mechanical roughening methods in comparison to APC PLUS adhesive (group DB,  $P=.045$  in CAD-Temp and group SB in C-Temp,  $P=.000$ ). In addition, C-Temp significantly revealed higher SBS in comparison with CAD-Temp for all groups (table 3).

ARI scores were significantly affected by the

TABLE (2) Three-way ANOVA for the material type, the surface treatment methods, the adhesive type and the interaction terms, according to the shear bond strength data (MPa)

Source of variations	Sum of squares	df	Mean squares	F	P value
Type of material	3775.705	1	3775.705	942.225	.000
Type of surface treatment	3965.259	3	1321.753	329.843	.000
Type of adhesive	114.691	1	114.691	28.621	.000
Type of material x type of surface treatment	70.610	3	23.537	5.874	.001
Type of material X type of adhesive	32.834	1	32.834	8.194	0.004
Type of surface treatment x type of adhesive	116.615	3	38.872	9.700	0.000
Type of material x type of surface treatment x type of adhesive	205.384	3	68.461	17.084	.000
Total	65077.586	320			

type of material ( $\chi^2=28.8$ ,  $P<.001$ ), the surface treatments (Monte Carlo test,  $P<.001$ ) and the adhesive type (Monte Carlo test,  $P<.001$ ) (tables 4 and 5). A closer look at the data in tables 4 and 5, C-Temp revealed a higher incidence of scores 1 and 2 more than CAD-Temp (41.9% and 23.8% respectively). However, CAD-Temp recorded high incidence of scores 4 and 5 more than C-Temp (48.7% and 34.4 % respectively).

Regarding surface treatment methods, DB and SB groups showed a high incidence of scores 1 and 2 (58.7% and 71.3%, respectively) than C and HP groups (0.0 % and 1.2% respectively). However, the highest incidence of scores 1 and 2 in (DB and SB groups) was more pronounced with C-Temp (63.5%) than CAD-Temp (36.5%). In addition, there

was a greater incidence of ARI scores 1 and 2 within APC PLUS more than Flash-free adhesive (37.5% and 28.1 %, respectively). Flash-free adhesive also showed a greater incidence of ARI scores 4 and 5 more than APC PLUS adhesive (61.3% and 36.2%).

The treated surfaces of CAD-Temp and C-Temp under SEM showed variations in their surface microstructures (fig. 1). Specimens treated with phosphoric acid showed random surface erosions (figs. 1 b and f). Roughening with a bur showed the uniform erosive appearance with undercuts (figs. 1 c and g). Sandblasted group showed well-defined micro-sized elevated and depressed areas (figs. 1 d and h). The effect of mechanical roughening including bur and sandblast were more homogenous, uniform and well oriented with C-Temp.

TABLE (3) Collected shear bond strength (MPa) data (Mean  $\pm$ SD) in all groups

Groups	Provisional materials		Independent t-test (P value)
	CAD Temp	C-Temp	
<b>APC PLUS adhesive</b>			
C	5.77 $\pm$ 1.51 <sup>cd</sup>	13.18 $\pm$ 2.43 <sup>bc</sup>	$P<.001$
HP	6.35 $\pm$ 1.56 <sup>c</sup>	13.98 $\pm$ 2.18 <sup>b</sup>	$P<.001$
DB	<u>10.01<math>\pm</math>1.53</u> <sup>b</sup>	17.77 $\pm$ 2.58 <sup>a</sup>	$P<.001$
SB	15.74 $\pm$ 1.79 <sup>a</sup>	<u>17.85<math>\pm</math>2.07</u> <sup>a</sup>	$P= .04$
<b>APC Flash-free adhesive</b>			
C	6.68 $\pm$ 1.9 <sup>cd</sup>	13.25 $\pm$ 2.07 <sup>cd</sup>	$P<.001$
HP	5.99 $\pm$ 1.66 <sup>c</sup>	14.47 $\pm$ 2.11 <sup>c</sup>	$P<.001$
DB	<u>11.78<math>\pm</math>2.31</u> <sup>b</sup>	18.04 $\pm$ 2.25 <sup>b</sup>	$P<.001$
SB	15.63 $\pm$ 1.93 <sup>a</sup>	<u>24.37<math>\pm</math>2.52</u> <sup>a</sup>	$P<.001$

\* Mean values represented with different superscript lowercase letter for each type of adhesive in each material are significantly different according to Tukey test ( $P<0.05$ )

\*\* Mean values represented with underline for comparing the two types of adhesive in each material are significantly different according to independent samples t-test ( $P<0.05$ )

TABLE (4) Collected ARI scores in all groups

Groups	Provisional materials									
	CAD Temp					C-Temp				
	1	2	3	4	5	1	2	3	4	5
<b>APC PLUS adhesive</b>										
C	0	0	6	13	1	0	0	10	10	0
HP	0	0	5	15	0	0	0	6	12	1
DB	0	11	6	3	0	6	11	3	0	0
SB	0	15	3	2	0	6	11	3	0	0
<b>APC Flash-free adhesive</b>										
C	0	0	3	14	3	0	0	5	13	2
HP	0	0	0	14	6	0	1	3	11	5
DB	0	5	10	5	0	3	11	6	0	0
SB	0	7	11	2	0	9	9	2	0	0

Table (5) The overall ARI scores (occurrence and percentages) according to the material type, the adhesive type and the surface treatment methods

ARI scores	Type of material		Type of adhesive		Surface treatment groups			
	CAD-Temp	C-Temp	APC PLUS	APC Flash-free	C	HP	DB	SB
1	0(0.0%)	24(15.0%)	12(7.5%)	12(7.5%)	0(0.0%)	0(0.0%)	9(11.2%)	15(18.8%)
2	38(23.8%)	43(26.9%)	48(30.0%)	33(20.6%)	0(0.0%)	1(1.2%)	38(47.5)	42(52.5%)
3	44(27.5%)	38(23.8%)	42(26.5%)	40(25%)	24(30.0%)	14(17.5%)	25(31.2)	19(23.8%)
4	68(42.5%)	47(29.4%)	56(35%)	59(51.3)	50(62.5%)	53(66.2%)	8(10.0%)	4(5.0%)
5	10(6.2%)	8(5.0%)	2(1.2%)	16(10.0%)	6(7.5%)	12(15.0%)	0(0.0%)	0(0.0%)
$\chi^2=28.8 P<.001$			MC test $P<.001$		MC test $P<.001$			

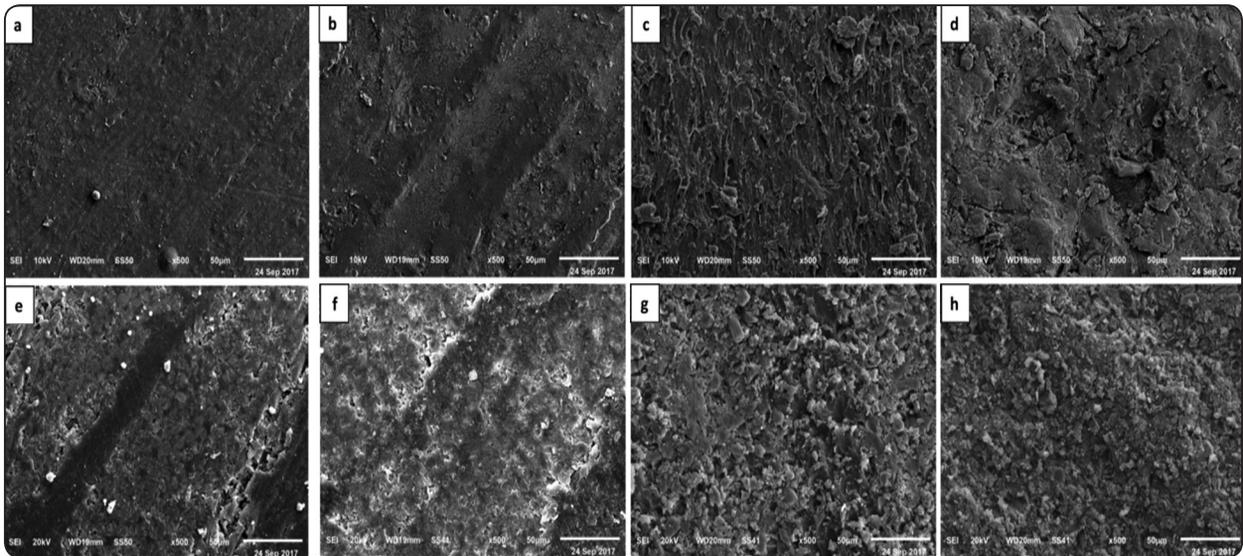


Fig. (1) SEM micrographs (500 x) of CAD-Temp (a-d) and C-Temp (e-h) provisional materials after different surface treatments; (a, e): C; (b, f): HP; (c, g): DB and (d, h): SB.

## DISCUSSION

To be clinically successful, the shear bond strength of orthodontic brackets to provisional materials should be adequately strong to prevent bracket de-bonding during the treatment. Accordingly, the aims of this study were to evaluate the bond strength of ceramic orthodontic brackets bonded to two different categories of CAD/CAM provisional materials based on the most reliable surface treatment methods and the adhesive system for optimal bonding. In addition, adhesive remnant index (ARI) was evaluated.

Surface treatments have been reported to enhance the bond strength to provisional materials.<sup>2</sup> Micromechanical retention can be provided through mechanical roughening with (diamond bur, sandblasting), or acid etching.<sup>2,9,20</sup> The specimens were 5000-times thermocycled simulating six months in clinical service in wet environmental conditions under standardized hydrothermal stresses.<sup>21</sup> Shear testing is considered the most common laboratory methods evaluating the shear bond strength of brackets.<sup>15, 22</sup> For standardization, two types of pre-coated ceramic orthodontic

brackets were used in this study: one with a novel adhesive system (APC Flash-free) that does not need removal of resin flash and one with traditional adhesive (APC plus) that needs flash removal.<sup>15-17</sup>

It has been reported that, 6-8 MPa is the optimal bracket bond strength.<sup>23</sup> In the present study, mechanically surface roughened groups (DB and SB groups) in both materials showed bond strength values above 6 MPa and subsequently could provide a clinical acceptable application. Mechanical surface treatments provide beneficial mechanical interlocking with pronounced effect for SB group as they provide small valleys and protruding peaks for additional bonding.<sup>4</sup> Roughening with a diamond bur creates deep grooves with macro- and micro retentive areas.<sup>24</sup> However, it was not surprising that the HP group significantly recorded lower SBS values compared to (DB and SB groups) as it has been reported as an ineffective approach for improving the micromechanical retention.<sup>25</sup> Therefore, the first null hypothesis was rejected.

Adhesive conditioning without mechanically roughened surface treatment did not give acceptable SBS values in CAD-Temp. This could be attributed to

the insufficient residual monomer in the industrially polymerized material to permit co-polymerization with the adhesive.<sup>18,26</sup> On the other hand, fiberglass reinforced polymer (C-Temp) revealed the higher shear bond strength than CAD-Temp and this finding is in agreement with Wiegand et al study.<sup>18</sup> This could be attributed to the penetration of the adhesive into the surface irregularities, which are created by fiber glass as shown by SEM micrograph and thus improving retention. Therefore, the second null hypothesis was rejected.

APC Flash-free adhesive recorded higher SBS values with mechanical surface treated groups. The resin utilized in APC Flash-free adhesive is unique among the orthodontic adhesives. It is a low viscosity adhesive resin and has a surface tension designed to wet and penetrate surface readily and consequently improving wettability and adhesion.<sup>17</sup> Therefore, the third null hypothesis was also rejected.

The modified ARI is a five-scaled scoring method that is used to quantify the amount adhesive left on the surface. It is one of the most frequently used indices in orthodontic adhesive testing. It has to be mentioned that higher ARI scores (more adhesive left on the brackets) appear to be favorable if chair time has to be reduced.<sup>19</sup> The lower ARI scores (more adhesive left on the provisional material) ensure few episodes of brackets dislodgement during orthodontic treatment.<sup>9</sup> The majority of bracket failures in CAD-Temp material utilizing flash free adhesive occurred within scores 4 and 5 which revealed adhesive failure between provisional material and adhesive in the pre-coated ceramic brackets. The adhesive failures are more favorable to avoid fracture of provisional materials during de-bonding. These findings are in agreement with the previous studies<sup>15,16</sup> that showed higher ARI scores with flash free adhesives. The higher ARI scores could be attributed to the slightly compressible non-woven polypropylene fiber positioned on the bracket base to hold back the excess adhesive, which is squeezed out during bracket application.<sup>17</sup>

Mechanically surface treated specimens in C-Temp showed lower ARI scores, which require further handling to remove the adhesive remnant from the provisional material surface. As noted to be mentioned, if SBS between the restoration and the adhesive resin is higher than 13 MPa, fracture will occur during de-bonding.<sup>27</sup> In this study, DB and SB groups in C-Temp and SB group in CAD-Temp recorded values higher than 13 MPa. Although no damage was observed to the de-bonded specimen in CAD-Temp, damage was observed in C-Temp (SB group-flash-free adhesive). This may be due to its higher SBS value.

The present study suggests that, using mechanical surface treatments and flash-free adhesive would enhance the bond strength of ceramic orthodontic brackets to CAD-Temp without liability of fracture during de-bonding. The recorded SBS is considered sufficient for orthodontic procedures. In addition, the higher ARI scores would reduce the chair time for excess removal. Regarding C-Temp, it is better not to perform mechanical surface treatments. The untreated surface gives sufficient and acceptable results for orthodontic treatment procedures. Although the mechanical surface treatments increased bond strength than CAD-Temp, the liability of fracture during de-bonding could occur in the sandblasted group and the lower ARI scores require more chair time for excess removal.

One of the limitations of this study is the visual inspections of the residual adhesive flash. We tried to assess and quantify the definite amount of adhesive flash remained around the bracket base with 30 x scanning electron microscope, but the adhesive margins could not be envisioned to obtain reliable measurements. Some other limitations do also exist, such as other oral environmental factors that could influence the bond strength; saliva components and differences in pH levels. Furthermore, the clinical performance assessment is required to provide reliable recommendations for orthodontists.

## CONCLUSION

- SBS of ceramic orthodontic brackets to CAD/CAM provisional materials depend on the type of material, the surface treatments and the type of adhesive.
- C-Temp provisional material significantly provided higher SBS than CAD-Temp in all groups.
- Mechanical surface treatments would enhance the bond strength of ceramic orthodontic brackets to CAD/CAM provisional material that is sufficient for orthodontic procedures.
- APC Flash-free adhesive showed higher SBS than APC PLUS adhesive in the mechanical roughening methods.
- APC Flash-free adhesive showed higher ARI scores than APC PLUS adhesive, which require less chair time for excess removal.

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