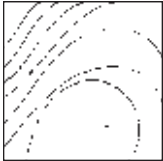




## Do Adhesive Systems Leave Resin Coats on the Surfaces of the Metal Matrix Bands? An Adhesive Remnant Characterization



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*Reestablishing proximal contacts with composite resins may prove challenging since the applied adhesives may lead to resin coating that produces additional thickness. The aim of this study was to investigate the surface of metal matrix bands after application of adhesive systems and blowing or wiping off the adhesive before polymerization. Seventeen groups of matrix bands were prepared. The remnant particles were characterized by energy dispersive spectrum and scanning electron microscopy. Total etch and two-step self-etch adhesives did not leave any resin residues by wiping and blowing off. All-in-one adhesive revealed resin residues despite wiping off. Prime and Bond NT did not leave any remnant with compomer. Clinicians must be made aware of the consequences of possible adhesive remnants on matrix bands that may lead to a defective definitive restoration. The adhesive resin used for Class II restorations may leave resin coats on metal matrix bands after polymerization, resulting in additional thickness on the metal matrix bands and poor quality of the proximal surface of the definitive restoration when the adhesive system is incorporated in the restoration. (Int J Periodontics Restorative Dent 2013;33:e43–e50. doi: 10.11607/prd.1129)*

Smooth and firm interproximal contacts between adjacent teeth play an important physiologic role in maintaining oral health.<sup>1</sup> Generally, it is assumed that an optimal contour and proximal contact strength are important characteristics to prevent food impaction, tooth migration, periodontal complications, and caries.<sup>1</sup> Nevertheless, reestablishing optimum physiologic contact between adjacent proximal surfaces is a fundamental prerequisite for achieving interdental integrity and preserving oral health.<sup>2</sup> However, restoration of proper proximal contact and contour with direct restorative materials presents several challenges for the clinician.<sup>2</sup> This is partly inherent to the polymerization shrinkage of the composite resin and partly to the fact that composite resins cannot be condensed like amalgam due to their viscoelasticity.<sup>3</sup>

Numerous techniques and materials can be used to reduce these drawbacks.<sup>4,5</sup> Several precontoured matrix and wedge systems,<sup>6</sup> teflon or titanium-covered special instruments,<sup>7</sup> composite resin/glass-ceramic inserts,<sup>8,9</sup> and light tips have been introduced for this purpose.

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**Table 1** Research outline of the experimental groups

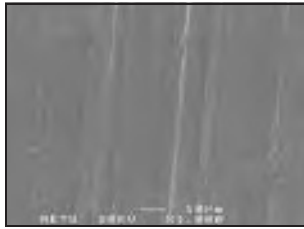
Group	Application procedures										
	Intact	Acid etching (AE)	Single bond (SB)	Clearfil SE primer (SEP)	Clearfil bond (CB)	S3	Prime and Bond NT (PB)	Blowing off	Wiping off	Filtek (F)	Dyract extra (DE)
1		•									
2		•	•								
3		•	•							•	
4		•	•					•		•	
5		•	•						•	•	
6				•							
7				•	•						
8				•	•					•	
9				•	•			•		•	
10				•	•				•	•	
11						•					
12						•				•	
13						•			•	•	
14							•				
15							•				•
16							•		•		•
17	•										

In addition, high viscous composites and packable or condensable composite resins were developed to condense composite resins. Other proposed approaches have included the use of a wooden wedge kept in the interdental space during preparation,<sup>10,11</sup> orthodontic separators,<sup>12</sup> and withdrawal of the matrix during the final moments of

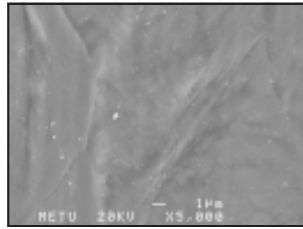
the restoration.<sup>13</sup> The general conclusion from these studies is that the key factor for producing a tight proximal contact is the attainment of interdental separation during restoration placement.<sup>6,14–17</sup>

The placement of the matrix system is usually carried out prior to adhesive application, because of its crucial role in contamination

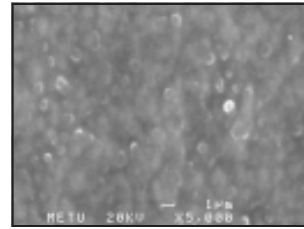
control.<sup>18</sup> To obtain a tight proximal contact with Class II composite resin restorations, the clinical procedure has to compensate for the thickness of the matrix as well as the polymerization shrinkage of the composite resin. Besides, adhesive resin systems may leave resin residues which may add additional thickness. This additional



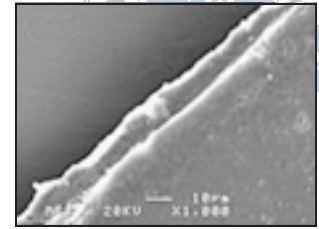
**Fig 1a** Intact metal matrix band (original magnification  $\times 1,000$ ).



**Fig 1b** Metal matrix band surface after AE (original magnification  $\times 5,000$ ).



**Fig 2** Metal matrix band surface after AE and SB application (original magnification  $\times 5,000$ ).



**Fig 3** Metal matrix band surface after AE + SB + F applications (original magnification  $\times 1,000$ ).

thickness might cause a challenge for the clinician in obtaining a healthy proximal contact, which is already difficult to obtain. To avoid this, the clinician may either gently blow off the excess resin with an air syringe or wipe the surface before polymerization. Thus, the aim of this study was to investigate the surface of the metal matrix bands after the application of different adhesive systems and after blowing or wiping off the adhesive before polymerization. The null hypothesis tested was that different adhesive resin systems would not leave any resin residues on the metal matrix bands.

## Method and materials

Seventeen groups (five samples per group) of metal bands (DML) were prepared, and the adhesives were applied. Each group received the following application techniques by the same operator (Table 1):

1. Acid etching (AE) (37% orthophosphoric acid; 3M ESPE)
2. AE + single bond (SB) (3M ESPE)
3. AE + SB + Filtek Z250 (F) (3M ESPE)
4. AE + SB + gentle blowing off + F
5. AE + SB + wiping off + F
6. Clearfil SE Primer (SEP) (Kuraray)
7. SEP + Clearfil Bond (CB) (Kuraray)
8. SEP + CB + F
9. SEP + CB + gentle blowing off + F
10. SEP + CB + wiping off + F
11. Clearfil S3 Bond (S3) (Kuraray)
12. S3 + F
13. S3 + wiping off + F
14. Prime and Bond NT (PB) (Dentsply DeTrey)
15. PB + Dyract Extra (DE) (Dentsply)
16. PB + wiping off + DE
17. Intact metal band

Wiping off was carried out with a microbrush to remove excess resin material from the metal matrix band, while blowing off the excess resin was done gently. A special cylindrical mold was placed over the metal matrix bands, and the restorative materials were placed and removed. Specimens were chemically dried using hexamethyldisilazane (HMDS, Electron Microscopy Sciences), mounted on aluminum stubs, sputter-coated with gold-palladium (Balzers-SCD 050 sputter coater), and observed under scanning electron microscopy (SEM) (JSM-6400 V, JEOL) at 20 kV of accelerating voltage. The

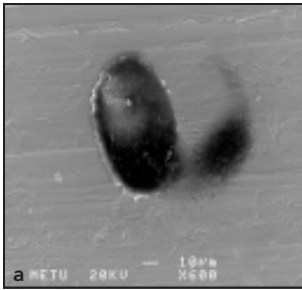
remnant particles were characterized using the energy dispersive spectrum (EDS).

## Results

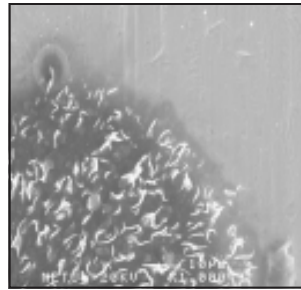
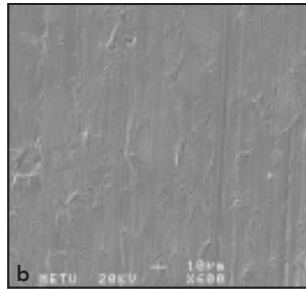
Figures 1a and 1b show the surface of the metal matrix before and after AE application. Figure 2 shows the representative SEM of the surface after AE + SB application. It is natural to note that the surface is completely coated with resin after AE + SB application.

Figure 3 represents the surface after AE + SB + F application; the composite resin is almost completely detached and absent from the metal band except at one point. However, excess resin remains at the margins of the mold that may be a result of the adhesive application.

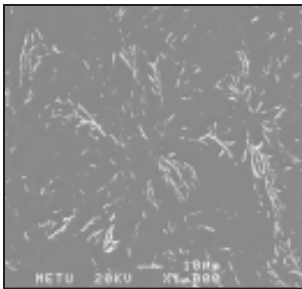
Figure 4a represents the surface after AE + SB + blowing off + F. Only a small amount of resin is captured by SEM. Similarly, Fig 4b displays a surface without any trace of resin after wiping off (AE + SB + wiping off + F). Figure 5 shows the metal matrix bands after SEP application. It is interesting to note that the primer is dried with stains.



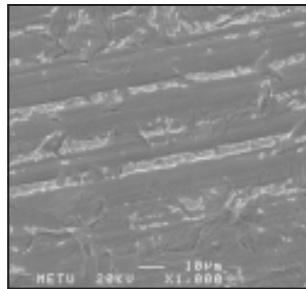
**Fig 4** (a) SEM of metal matrix band surface after AE + SB + blowing off + F application (original magnification  $\times 600$ ). (b) SEM of metal matrix band surface after AE + SB + wiping off + F application (original magnification  $\times 600$ ).



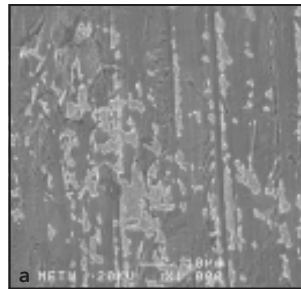
**Fig 5** SEM of metal matrix band surface after SEP application (original magnification  $\times 1,000$ ).



**Fig 6** SEM of metal matrix band surface after SEP + bond application (original magnification  $\times 1,000$ ).



**Fig 7** SEM of metal matrix band surface after SEP + bond + F application (original magnification  $\times 1,000$ ).



**Fig 8** (a) SEM of metal matrix band surface after SEP + bond + blowing off + F application (original magnification  $\times 1,000$ ). (b) SEM of metal matrix band surface after SEP + bond + wiping off + F application (original magnification  $\times 600$ ).

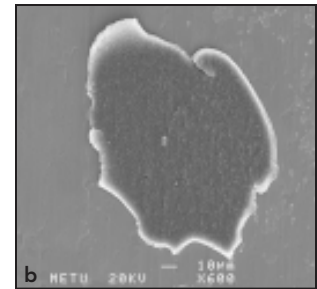


Figure 6 shows the surface after SEP + bond. It is not surprising to see the surface covered with resin.

The SEM image of the metal matrix band surface after SEP + bond + F application is demonstrated in Fig 7. Resin remnants appear to be on the surface but EDS analysis could not capture any resin elements.

Figure 8a shows an SEM figure of the metal matrix band surface after SEP + bond + blowing off + F application. The spectral elemental analysis also showed no resin residues on the surface. Figure 8b shows the surface of the metal matrix band after SEP + bond + wiping off + F application. Unlike

blowing off, the wiping technique demonstrated more resin residues on the metal matrix bands using the EDS.

SEM analysis of the metal matrix band surface after S3 application is shown in Fig 9a; note that the surface is inevitably covered by resin material. Figure 9b shows the SEM figures of the metal matrix bands after S3 + F application; note that the surface is somewhat coated with resin material and EDS spectrum also verifies the existence of resin material on the surface.

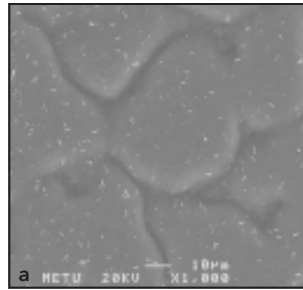
The SEM figure of the metal matrix band surface after S3 + wiping off + F is shown in Fig 10; note the small amount of resin residue

on the surface despite wiping off before polymerization.

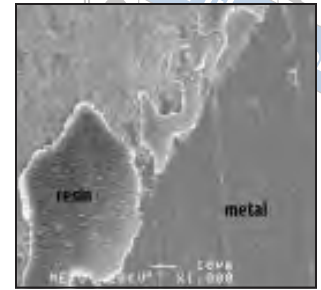
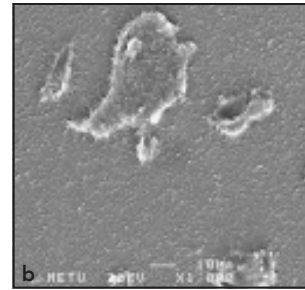
Figure 11 demonstrates the metal matrix band surface after PB application. The surface is thoroughly covered by resin material. Figures 12a and 12b show the same surface after PB + DE application alone and with wiping off. In both cases, the surfaces are completely free of resin residue.

## Discussion

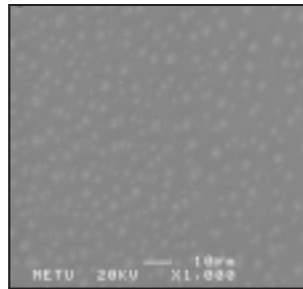
Establishing clinically acceptable proximal contacts when placing composite resin in Class II preparations always poses a challenge



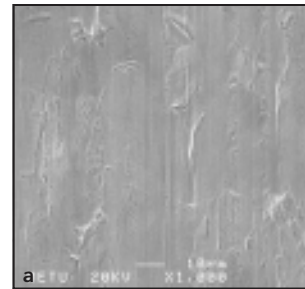
**Fig 9** (a) SEM of metal matrix band surface after S3 application (original magnification  $\times 1,000$ ). (b) SEM of metal matrix band surface after S3 + F application (original magnification  $\times 1,000$ ).



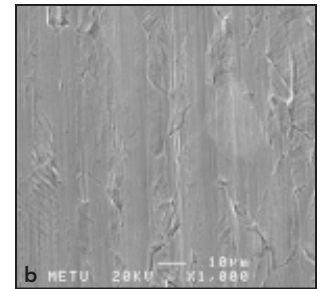
**Fig 10** SEM of metal matrix band surface after S3 + wiping off + F application (original magnification  $\times 1,000$ ).



**Fig 11** SEM of metal matrix band surface after PB application (original magnification  $\times 1,000$ ).



**Fig 12** (a) SEM of metal matrix band surface after PB + DE application (original magnification  $\times 1,000$ ). (b) SEM of metal matrix band surface after PB + wiping off + DE application (original magnification  $\times 1,000$ ).



to the clinician since the proximal contour of the restored tooth should ideally resemble that of an intact natural tooth.<sup>19</sup>

Indeed, the proximal contact plays an important role in protecting periodontium against damage of food impaction.<sup>20</sup> In addition, the lateral components of occlusal forces are transmitted to the adjacent teeth and the surrounding periodontal ligament with the aid of proximal contacts.<sup>21</sup> Furthermore, loss of contact allows for the tilting of teeth that disturbs occlusal relations with antagonistic teeth and facilitates food and plaque retention.<sup>21</sup> When contact is too tight, tooth migration or trauma of

the periodontal tissues may result when excessive force is used to pass dental floss through the proximal contact.<sup>22</sup>

There has been extensive research to improve the proximal contact quality to reestablish physiologic contacts, including different resin systems, placement techniques, matrix systems, wedges, and special hand instruments. However, to the authors' knowledge, there is a lack of research concerning the adhesive resin residue coats on metal matrix bands. Thus, no comparison can be accomplished.

The results of the SEM analysis demonstrated that when matrix is

removed from the restoration, most of the adhesive resin leaves the surface of the metal matrix band. Thus, the null hypothesis was rejected. However, there remains a varying amount of adhesive resin residue on the metal matrix bands with different types of adhesive systems.

This finding may lead readers to another hypothesis that the cured adhesive resin on the metal matrix band may incorporate at the surface of the restoration itself after removal of the matrix band, complicating the prognosis of the proximal contact. As a result, the approximal surface of the definitive restoration may be of low quality with unfilled or poorly filled porous

irregularities and defects. Thus, the resulting surface will be less resistant to wear due to tribologic proximal attrition and can enhance biofilm accumulation and cause localized inflammation of marginal periodontium. In fact, a 6-month clinical study by Loomans et al<sup>23</sup> indicated that increased proximal contact tightness tends to loosen in 6 months time. This issue may be attributed to proximal wear as, after 6 months, the mean proximal wear of a highly filled composite was  $\pm 50 \mu\text{m}$ , whereas the enamel lost was  $\pm 5 \mu\text{m}$ .<sup>24</sup> However, the insight derived from this prediction must be quantitatively studied in future research.

Some supplementary factors such as the surface geometry of the cavity walls may cause the adhesive resin to precipitate along the margins of the metal matrix band and the cavity walls, leading to a thick adhesive layer. On smooth, convex surfaces, the adhesive layer can be 60 to 80  $\mu\text{m}$ , while in concave regions, this thickness might be greater than 200 to 300  $\mu\text{m}$ .<sup>25–29</sup> Even when long air drying was employed, there was evidence of ineffective drying and pooling of the adhesive when applied on narrow Class I caries lesions.<sup>30</sup>

There may be other material-dependent factors such as the viscosity, filler ratio of the adhesive resin, and solvent evaporation from the adhesive system. All the adhesive systems used in this study contain different ratios of monomers/co-monomers and/or inorganic filler constituents (Table 2). Thus, it will be more difficult to eliminate more

viscous un-polymerized resin remnants from the metal matrix bands.

Water, ethanol, and acetone are the main solvents in commercial formulations.<sup>31,32</sup> A combination of some physical and chemical factors, namely, water pressure, molar fractions, molar weight, and solubility, is considered to determine the evaporation capacity of different solvents.<sup>33–36</sup> Solvent evaporation after application to the class box is extremely important because failure to remove excess solvent by gentle air drying may contribute to the degradation of the adhesive.<sup>33,37</sup>

The samples in which the metal matrix bands were wiped before polymerization revealed the best SEM results with etch-and-rinse adhesive systems (see Fig 5). Wiping off the excess etch-and-rinse adhesive resin before polymerization may diminish excess material on the surface, and no adhesive resin remained on the metal matrix bands. However, there may be some technical errors while wiping off the surface with brushes and cotton pellets since cellulose fibers may be incorporated into the restoration. On the contrary, the results where the self-etch adhesive systems were used demonstrated that blowing off the excess resin produced the cleanest metal matrix band surface. PB also demonstrated the cleanest surface when used with DE. In those ways, clinicians may be able to minimize the space occupied by the metal matrix bands, and there will be no additional thickness with the adhesive systems. However, it is still interest-

ing to see that all-in-one adhesives left so much resin residue on the metal matrix bands.

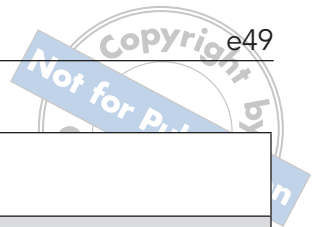
A limitation of this study was that the quantification and qualification of the amount of remnant resin thickness, which was insufficient to conclude that these remnants produce additional thickness on the surfaces of the metal matrix bands. Nonetheless, the findings of this research may provide an indication for more qualified quantitative research in the future.

## Conclusions

Etch-and-rinse and two-step self-etch adhesives did not leave any resin residue on the metal matrix with air thinning or wiping. All-in-one adhesive revealed resin residues on the surface despite wiping off. PB did not leave any remnant with compomer. However, the absence of the adhesive remnants on the matrix band does not necessarily mean that the final resin surface is free of adhesives since the polymerized adhesive resins present on the metal matrix bands may be incorporated into the definitive restoration's surface. Thus, the clinician must be cautious not to spread the adhesive resins on the metal matrix band before polymerization.

## Acknowledgment

The authors reported no conflicts of interest related to this study.


**Table 2** Material descriptions and manufacturers of the materials used

Adhesive system	Material	Chemical composition	Manufacturer
Etch-and-rinse	Adper Single Bond Plus	Ethyl alcohol (25% to 35%) Silane-treated silica (nanofiller) (10% to 20%) bis-GMA (10% to 20%) HEMA (5% to 15%) Glycerol 1,3-dimethacrylate (5% to 10%) Copolymer of acrylic and itaconic acids (5% to 10%) Water (< 5%) Diurethane dimethacrylate (1% to 5%)	3M ESPE
Self-etch (two step)	Clearfil SE Bond	bis-GMA (25% to 45%) HEMA (20% to 40%) 10-methacryloyloxydecyl dihydrogen phosphate Hydrophobic aliphatic dimethacrylate Colloidal silica DI-Camphorquinone Initiators Accelerators Others	Kuraray
Self-etch (all-in-one)	Clearfil Tri-S	bis-GMA (15% to 35%) HEMA (15% to 35%) Ethanol (< 20%) 10-methacryloyloxydecyl dihydrogen phosphate Colloidal silica DI-Camphorquinone Water Initiators Accelerators Others	Kuraray
Self-priming (one step)	Prime and Bond NT	Di- and trimethacrylate resins PENTA (dipentaerythritol penta acrylate monophosphate) Nanofillers—amorphous silicon dioxide Photoinitiators Stabilizers Cetylamine hydrofluoride Acetone	Dentsply

bis-GMA, bisphenol glycidyl; HEMA, hydroxyethyl methacrylate.

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