Shear bond strength of Scotchbond 2/Silux to dentin

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Abstract: The shear bond strengths of the Scotchbond 2/Silux restorative system to dentin were determined on specimens assembled with two different procedures. In the initial assembly apparatus the platform was elevated with a rotational action to ensure that the dentin surface made firm contact with the lower border of a split teflon mold, while in the modified apparatus this was achieved by releasing a spring. Significantly higher shear bond strengths were obtained with the modified apparatus. Shear bond strengths were also determined on the approximal and occlusal dentin surfaces. The shear bond strengths were not significantly different. The effects of the components of the Scotchbond 2 dentinal bonding system on dentin were examined by SEM. An acid-resistant layer was formed on the dentin surface. Examination of the dentin aspects of fractured test specimens revealed that a variable fracture pattern was obtained. Examination of fracture sites which were classified as occurring at the dentin/resin interface indicated that the evaluation at low magnification was incorrect. At higher magnification these fracture sites revealed remnants of the restorative system adhered to dentin and it was resistant to phosphoric acid attack. The results of this study clearly indicate the dependence of shear bond strengths on the method of specimen asssembly and in the application of the shear load. An appeal is made for the standardization of the test methodology for shear bond strength tests.

Key words: Dentin; restorative dentistry; Scotchbond 2.

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INTRODUCTION

The lack of adhesion of dental restorative materials to tooth structure is a major problem in clinical dentistry. The application of orthophosphoric acid (H_3PO_4) to enamel, a concept which was first introduced by Buonocore,[1] has resulted in a significant increase in the bond strength of polymeric restorative resins to etched enamel,[2,3] and has practically eliminated microleakage at the restoration/enamel interface.[4,5] Although the application of H_3PO_4 to dentin effectively removes the smear layer and opens the dentinal tubules into which the curing resin flows, acid etching of dentin does not enhance the bond strength of composite restorative resins to dentin.[6,7] The direct application of H_3PO_4 to dentin is not advocated because of the pulpal responses elicited by the acidic etching agents.[8,9]

During the past 30 years, extensive research efforts have been directed towards the development of dentin bonding agents. Several dentin bonding agents have recently become commercially available. The development of these systems in the USA,[10] Germany,[11] and Japan[12] were reviewed. The dentin bonding agents are difunctional molecules containing reactive groups which interact with the dentin surface and with the monomer of the restorative resin.

This study determined the shear bond strength of Scotchbond 2[a]/Silux[a] to dentin by means of different test methods, and evaluated the effects of the components of the dentin bonding system on dentin. In addition, the dentin aspects of fractured test specimens were examined by scanning electron microscopy.

MATERIALS AND METHODS

Shear bond strength tests

Scotchbond 2[a] bonding system consists of Scotchprep[a] dentin primer (Batch #7E7P), an aqueous solution of maleic acid and hydroxyethyl methacrylate (HEMA) and Scotchbond 2[a] light cure dental adhesive (Batch #7C6P), a mixture of Bis-GMA and HEMA. The shear bond strength of Silux[a] (Batch #7R1R), a microfill light cure composite resin to treated dentin was determined.

Tooth preparation

One hundred-fifty extracted non-carious human mandibular permanent molars which were stored in 70% ethyl alcohol were used. This storage medium was selected because it was shown that storage of teeth in 70% ethyl alcohol does not affect the physical and chemical properties of teeth.[13] The roots were removed with a diamond disk and the mesial approximal surfaces ground wet on a polishing machine[b] on 160 grit silicon carbide paper. The crowns of the teeth were embedded in brass tooth cups with cold cure acrylic resin[c] so that the ground surfaces projected well above the lips of the cups. The teeth were stored in physiological saline. Immediately prior to the preparation of a test specimen, the exposed tooth surface was ground wet in a polishing block on 600 grit silicon carbide paper to expose the underlying dentin. The embedded tooth was washed in running tap water and dried with oil-free compressed air. The Scotchprep[a] dentin primer was applied to the dentin surface with a disposable brush and coverage maintained for 60 seconds by continual agitation with the brush. The primed dentin surface was dried to a dull surface with compressed air for 15 seconds. The Scotchbond 2[a] light cure dental adhesive was applied to the dentin surface, thinned with a gentle stream of compressed air, and cured by exposure to visible light[d] for 30 seconds.

Fig. 1. Initial apparatus for the assembly of test specimens. A) Split teflon mold; B) Knob which was rotated to elevate and lower the elevating platform; C) Platform; D) Tooth embedded in brass tooth cup.

Specimen preparation

Two test systems were used to prepare the test specimens for the determination of the shear bond strength. In the first system the tooth cup with the embedded tooth was mounted in the assembly apparatus (Fig. 1). A split teflon mold with a circular hole, 3.5 mm in diameter and 5 mm in depth, was positioned in the device (Fig. 1A) and the knob (Fig. 1B) rotated clock-wise to elevate the platform (Fig. 1C) so that the embedded tooth (Fig. 1D) made firm contact with the lower surface of the teflon mold. The Silux[a] composite resin was transferred to the opening in the teflon mold, compressed firmly, and exposed to visible light for 30 seconds. Two further increments of Silux[a] were transferred and cured in a similar manner. A test specimen was removed from the assembly apparatus after 15 minutes by turning the knob (Fig. 1B) anticlockwise to lower the elevating platform. The split teflon mold was removed to expose the composite cylinder bonded to the dentin surface.

A test specimen was allocated to one of five test variables.

Procedure A: The shear bond strength was determined 15 minutes after cure.
Procedure B: The test specimens were stored in physiological saline at 37°C for 24 hours prior to testing.
Procedure C: Same as in *Procedure B* but the test specimens were subjected to 250 temperature cycles in a cycling machine[e] between 8°C and 50°C with a dwell time of 15 seconds.
Procedure D: The test specimens were stored in physiological saline at 37°C for 4 weeks prior to testing.
Procedure E: Same as in *Procedure D* and temperature cycled as in *Procedure C*.

The tooth cup with the bonded composite cylinder was then placed in a special device which was positioned on the compression load cell of an Instron[f] testing machine. A shear load was applied to the bonded cylinder with a 1.8 mm width rectangular based rod (Fig. 2A) at a crosshead speed of 0.02 inch.min^{-1}. The shear bond strengths were calculated and expressed in MN.m^{-2}.

Approximately one third of the test specimens failed during removal from the initial assembly apparatus. On closer examination it was evident that rotational forces were introduced to the tooth cup (Fig. 1D) when the knob (Fig. 1B) was rotated anticlockwise to lower the elevating platform (Fig. 1C) to release the test specimens. Following the suggestion of one of our graduate students, Dr. Stanley Wendt, the assembly apparatus was redesigned (Fig. 3). Instead of using a rotational force to elevate and subsequently lower the platform (Fig. 3A) a spring (Fig. 3B) was incorporated in the device. During the assembly of the test specimens, the spring was disengaged from a locking device to firmly press the elevating platform against the tooth cup with the embedded tooth (Fig. 3C). To remove the test specimen after cure, the spring was pulled back and locked. The

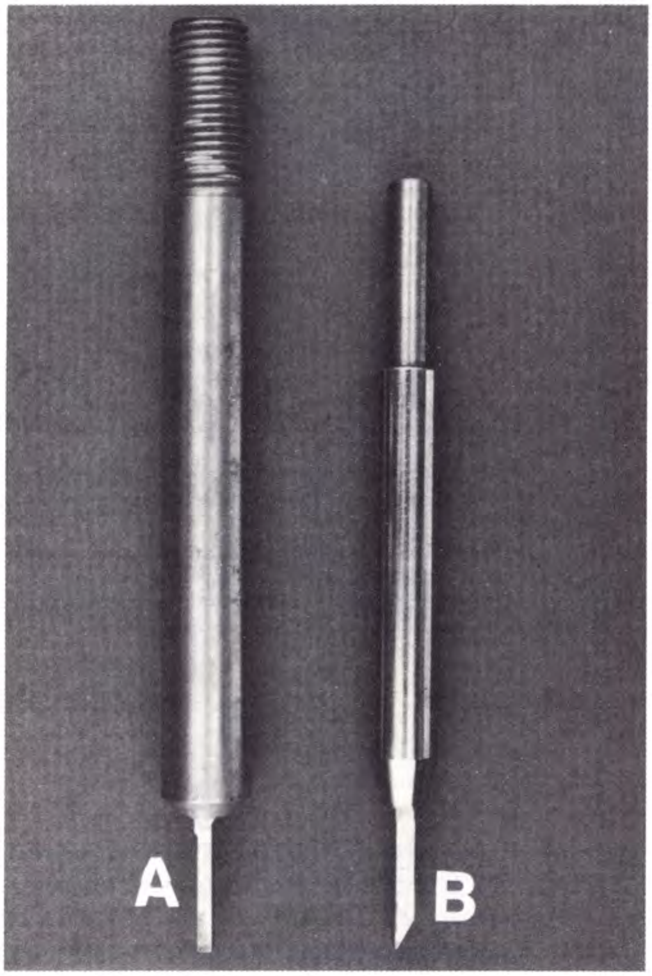

Fig. 2. Rods used to apply a shear load to the bonded composite cylinders. A) Rectangular base, 1.8 mm in width; B) Knife-edge base, 0.5 mm in width.

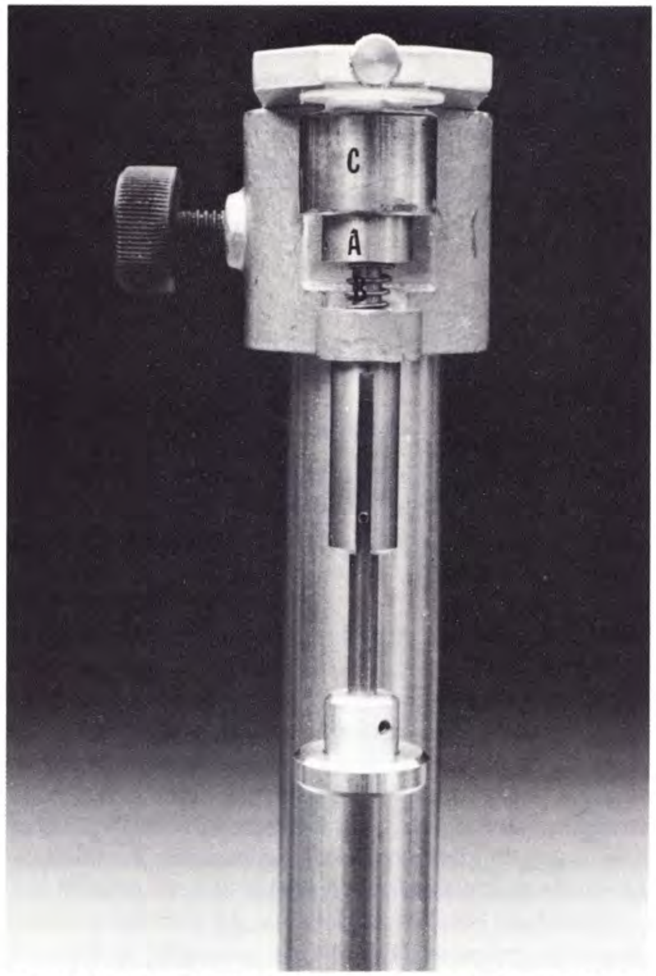

Fig. 3. Modified apparatus for the assembly of test specimens. A) Elevating platform; B) Spring; C) Embedded tooth in brass cup.

experiment was repeated using the same test variables as previously employed (Procedures F, G, H, I, J). None of the test specimens failed during disassembly.

The procedure was further modified by applying the shear load with a knife-edged rod with a width of 0.05 mm (Fig. 2B). Fifteen test specimens were prepared, removed from the assembly apparatus 15 minutes after cure, and the shear bond strength determined after storage in physiological saline at 37°C for 24 hours (Procedure K).

A further modification was introduced by curing the dentin on the occlusal aspects of the extracted molar teeth as opposed to the dentin on the approximal surfaces. Fifteen test specimens were prepared, removed from the modified assembly apparatus after 15 minutes, stored in physiological saline at 37°C for 24 hours, and a shear load applied with the knife-edged rod (Procedure L).

Scanning electron microscopy

The effects of the components of the dentinal bonding system on dentin were examined by scanning electron microscopy (SEM). Two specimens of the following surfaces were examined.

A. Dentin surface ground on 600 grit silicon carbide paper.
B. Ground dentin surface treated with Scotchprep[a] dentin primer for 60 seconds.
C. Ground, primed dentin surface treated with Scotchbond 2[a] dental adhesive and light-cured for 30 seconds.
D. Ground, primed dentin surface treated with Scotchbond 2[a] light cure dental adhesive and etched with 37% H_3PO_4 gel for 30 seconds.

Fractured test specimens which exhibited various interfacial fracture patterns were selected for examination in the SEM. Some of these test specimens were etched with 37% H_3PO_4 gel for 30 seconds. The specimens were coated with gold/palladium in a HUS-4[g] high vacuum evaporator and viewed in an ISI-100B[h] scanning electron microscope operated at 20 kV.

Statistical analysis

The shear bond strength data was analyzed by a one-way analysis of variance,[14] Duncan's multiple range test,[15] and the Student's *t*-test.[16] The dentin aspects of the fractured test specimens were examined microscopically at x50 magnification and the percentage of the bonding

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Table 1. Shear bond strengths ($MN.m^{-2}$) of the Scotchbond 2/Silux restorative system to dentin. Initial assembly apparatus.

Procedure	Number of specimens	Shear bond strengths $-MN.m^{-2}$ Mean	SD	Range	Coefficient of variation %
A: 15 Min	15	4.1	1.1	2.4- 6.2	28
B: 24 Hours	15	6.6	3.0	3.0-13.4	45
C: 24 Hours Temp. Cycle	15	6.8	3.4	3.6-14.2	50
D: 4 Weeks	15	3.3	1.4	1.8- 6.9	42
E: 4 Weeks Temp. Cycle	15	3.3	1.5	1.7- 7.2	47

area which failed within the resin estimated. The correlations between shear bond strength and percentage failure were established.[16] All computations were done by the SAS system.[17]

RESULTS

Shear bond strength tests

The shear bond strengths obtained with the initial assembly apparatus (Fig. 1) are presented in Table 1. Analysis of variance revealed that the shear bond strengths were significantly different (P=0.0001). These differences in shear bond strengths were identified by Duncan's multiple range test (Table 2).

The shear bond strengths obtained with the modified assembly apparatus (Fig. 3) are presented in Table 3. Analysis of variance revealed that the shear bond strengths were not significantly different (P=0.1082).

The shear bond strengths obtained on the approximal and occlusal dentin surfaces with the modified assembly apparatus and with the shear load applied with a knife-edged rod are depicted in Table 4. Analysis of variance revealed that the shear bond strengths were not significantly different (P=0.2115).

The data were analyzed by the Student's *t*-test and the results are in Table 5. The results showed that except for the shear bond strengths obtained 24 hours after specimen preparation and subjected to temperature cycling (Procedure C *vs* Procedure H), the shear bond strengths obtained with the modified assembly apparatus were significantly greater than with the initial assembly apparatus. It further showed that the shear bond strengths obtained 24 hours after specimen preparation when the shear load was applied with a knife-edged rod (Procedure K) were significantly greater than when the shear load was applied with the blunt-edged rod (Procedure G)(P=0.0006). The shear bond strengths obtained on the approximal dentin surfaces (Procedure K) were not significantly different than the shear bond strengths recorded on the occlusal dentin surfaces (Procedure L)(P=0.1606).

The correlations among shear bond strengths and percentage failure in the resin obtained on test specimens prepared with the initial (P=0.1396) and the modified assembly apparatus (P=0.1093) were not significant.

Table 2. Analysis of the shear bond strength data by Duncan's multiple range test. The shear bond strengths are listed in decreasing order of magnitude.

Procedure		Mean shear bond strength $-MN.m^{-2}$
C	*	6.8
B		6.6
A	*	4.1
D		3.3
E		3.3

*Means linked by vertical lines were not significantly different.

Scanning electron microscopy

The effects of the various components of the dentinal bonding system on dentin are shown in Fig. 4. A dentin surface ground on 600 grit silicon carbide paper indicated the grooves produced on the dentin surface by the abrasive particles (Fig. 4A). The application of the Scotchprep[a] dentin primer to a ground dentin surface for 60 seconds removed the smear layer but left the dentinal tubules partially occluded with debris (Fig. 4B). An even deposit was observed on a ground, primed dentin surface to which the Scotchbond 2[a] light cure dental adhesive was applied and cured for 30 seconds (Fig. 4C). Etching such a surface with 37% H_3PO_4 for 30 seconds showed that the deposit was resistant to acid attack (Fig. 4D).

Examination of the dentin aspect of fractured test specimens showed a variable fracture pattern. A specimen with 0% fracture within the resin (100% at the interface) is shown in Fig. 5A; with 10% failure in the resin in Fig. 5B; with 30% failure within the resin seen in Fig. 5C; with 60% failure within the resin in Fig. 5D; with 90% failure within the resin in Fig. 5E. Twenty one of the test specimens failed within dentin (Fig. 5E).

The dentin aspects of some of the fractured test specimens were examined by the SEM. A fractured test specimen which failed at 9.3 $MN.m^{-2}$ showed a surface deposit which suggested that failure did not occur at the

Table 3. Shear bond strengths ($MN.m^{-2}$) of the Scotchbond 2/Silux restorative system to dentin. Modified assembly apparatus.

Procedure	Number of specimens	Shear bond strengths -$MN.m^{-2}$ Mean	SD	Range	Coefficient of variation %
F: 15 Min	15	6.9	2.3	4.9-13.9	34
G: 24 Hours	15	8.5	1.6	7.1-13.5	19
H: 24 Hours Temp. Cycle	15	8.4	1.7	6.3-12.1	19
I: 4 Weeks	15	8.3	2.0	5.7-12.1	23
J: 4 Weeks Temp. Cycle	15	8.1	1.2	6.5-10.7	15

Table 4. Shear bond strengths ($MN.m^{-2}$) of the Scotchbond 2/Silux restorative system on the approximal and occlusal dentin surfaces. Modified assembly apparatus, shear load applied with a knife-edge rod.

Procedure	Number of specimens	Shear bond strengths -$MN.m^{-2}$ Mean	SD	Range	Coefficient of variation %
K: 24 Hours Approximal	15	13.6	4.8	4.3-19.3	36
L: 24 Hours Occlusal	15	11.3	3.8	5.5-16.7	34

bonding system/dentin interface (Fig. 6A). Open dentinal tubules were observed in another test specimen which failed at 13.5 $MN.m^{-2}$ (Fig. 6B). The surfaces of two test specimens which failed at 6.1 $MN.m^{-2}$ and 13.5 $MN.m^{-2}$ and which were etched with 37% H_3PO_4 for 30 seconds, are depicted in Figs. 6C and 6D, respectively. These scanning electron micrographs indicated that an acid-resistant material was present at the interface and suggested that failure occurred within the Scotchbond 2[a] adhesive system and not at the restorative/dentin interface.

DISCUSSION

The shear bond strengths of the Scotchbond 2[a]/Silux[a] restorative system to dentin which were obtained by applying the shear load via a rectangular based rod, 1.8 mm wide, and with the initial assembly apparatus in which the specimens were released by applying a rotational force (Table 1) were significantly lower than the shear bond strengths obtained with the modified assembly apparatus in which the specimens were released by lowering the elevating platform with the spring device (Table 3).

Table 5. Analysis of the shear bond strength data by the Student's *t*-test.

Procedures	P-value
A *versus* F	0.0003
B *versus* G	0.0408
C *versus* H	0.1050
D *versus* I	0.0001
E *versus* J	0.0001
G *versus* K	0.0006
K *versus* L	0.1606

The shear bond strengths of Scotchbond 2[a]/Silux[a] restorative system to dentin 24 hours after specimen preparation; which were obtained with the modified assembly apparatus and in which the shear load was applied with a rod with a 1.8 mm width rectangular based rod (Procedure G; 8.5 $MN.m^{-2}$) were significantly lower than the shear bond strengths recorded when the shear load was applied with the 0.5 mm width knife-edged rod (Procedure K; 13.6 $MN.m^{-2}$) ($P=0.0006$). With the rectangular based rod a cantilever loading is applied while with the knife-edged rod a wedge-opening load is applied. The moment of force (M) is measured by the product of the force (F) and the perpendicular distance (d) from the line of action of the force to the axis.

$$M = F.d$$

It can be assumed that the perpendicular distance from the line of action of the force to the axis will be greater when the rectangular based rod was employed to apply the shear load than when the knife-edged rod was used. It is therefore not surprising that the shear bond strengths were significantly lower when the rectangular based rod was used to apply the shear load than when the knife-edged rod was used.

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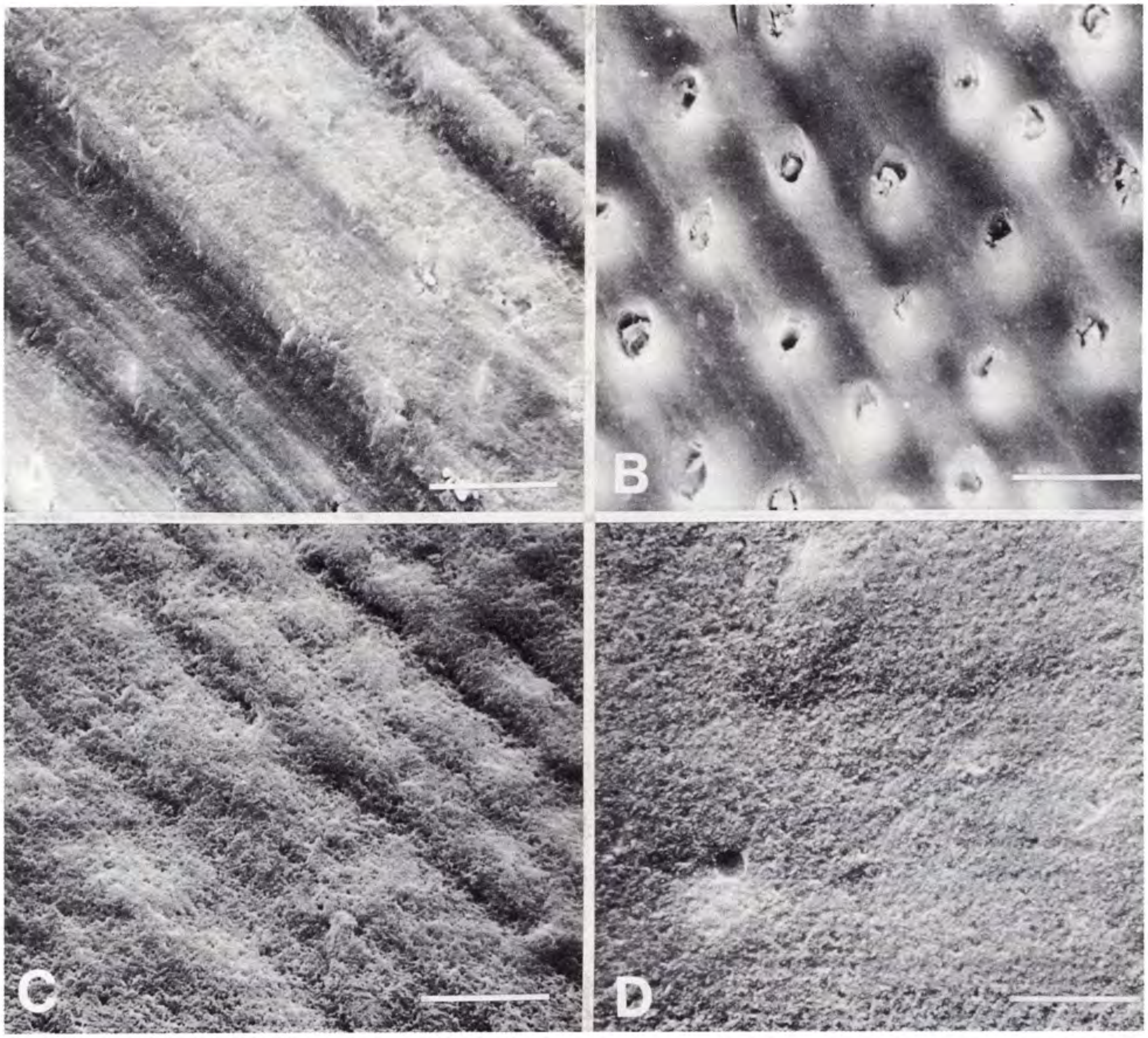

Fig. 4. Effects of the components of the Scotchbond 2 dentinal bonding system on ground dentin. A) Dentin surface ground on 600 grit SiC. SEM x2000; Application of the Scotchprep dentin primer to ground dentin for 60 seconds. SEM x 2000; C) Application of the Scotchbond 2 light cure dental adhesive to ground, primed dentin. SEM x2000; D) Same as C but etched with 37% H_3PO_4 for 30 seconds. SEM x2000. Bar = 10 μm.

The shear bond strengths of the Scotchbond 2[a]/Silux[a] restorative system to approximal dentin 24 hours after specimen preparation recorded with the modified system and with the application of the shear load with the knife-edged rod (Procedure K; 13.6 $MN.m^{-2}$), were not significantly different than the shear bond strengths to occlusal dentin using the same test system (Procedure L; 11.3 $MN.m^{-2}$) (P=0.1606).

The results of the present study clearly demonstrate the dependence of shear bond strengths on the test methodology used for the assembly of the test specimens and for the application of the shear load. An appeal is made for the standardization of shear bond strength tests.

The scanning electron micrographs of ground, primed dentin surfaces treated with Scotchbond 2[a] light cure dental adhesive clearly demonstrated that an acid-resistant surface deposit was formed on the dentin surfaces (Fig. 4). Examination of the dentin aspects of fractured test specimens revealed a great variation in the fracture pattern (Fig. 5). Twenty-one of the 180 test specimens failed in dentin. It is interesting to note that the mean shear bond strength at which fracture occurred within dentin was 13.1 $\pm$ 4.6 $MN.m^{-2}$. Eight of the 21 specimens failed in dentin at a lower shear bond strength while seven specimens failed at higher shear bond strengths without fracture in dentin. The extracted human mandibular permanent molars used in this study were stored in 70% ethyl alcohol for at least six months. Recent studies conducted in our laboratory indicate that dentin fractures are less likely to occur in freshly extracted teeth.

The dentin aspects of some fractured test specimens were examined at x50 magnification and the percentage

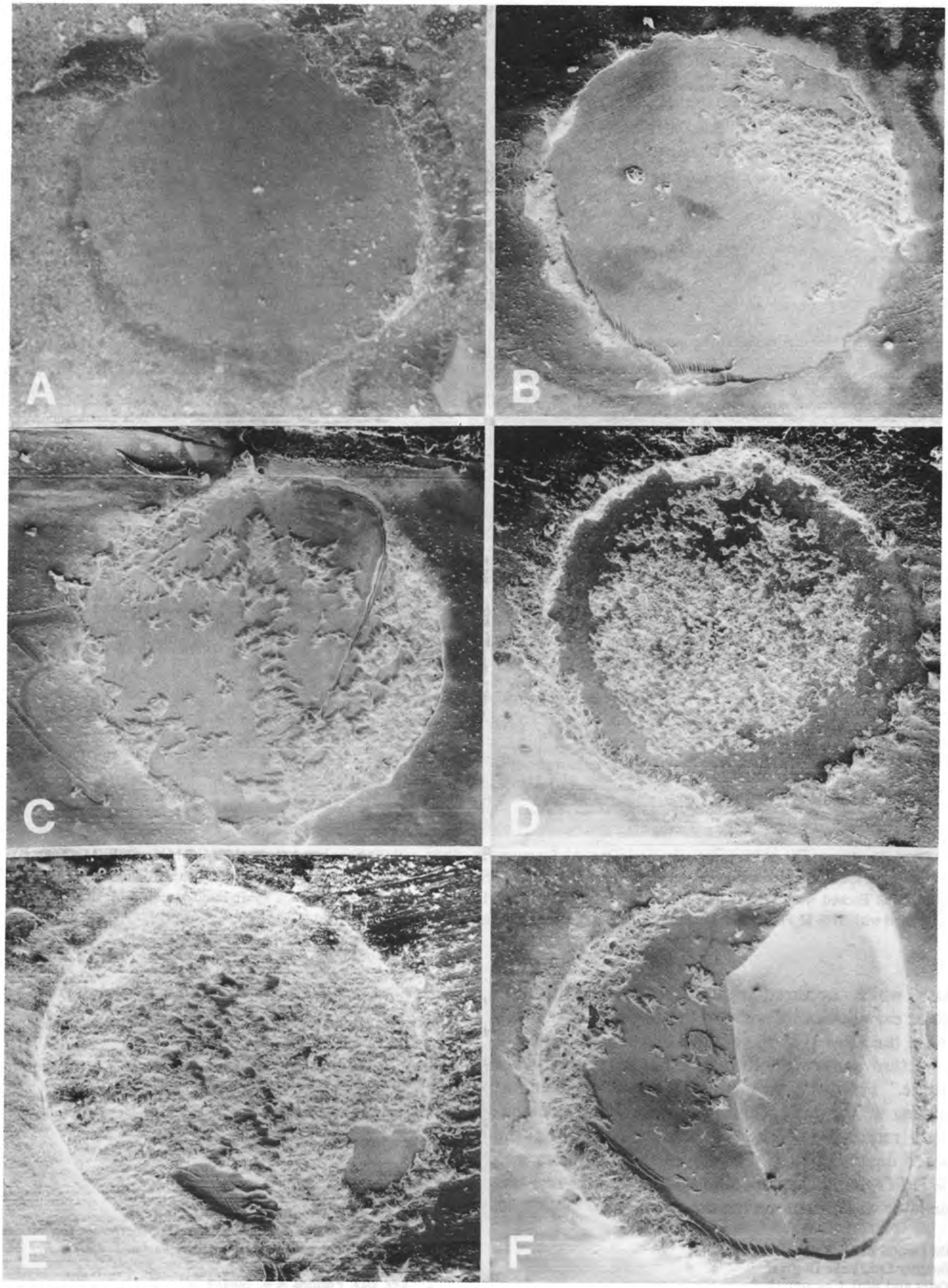

Fig. 5. Dentin aspects of fractured test specimens within the resin. A) 0% failure; B) 10% failure; C) 30% failure; D) 60% failure; E) 90% failure; F) Fracture within dentin. SEM x15.

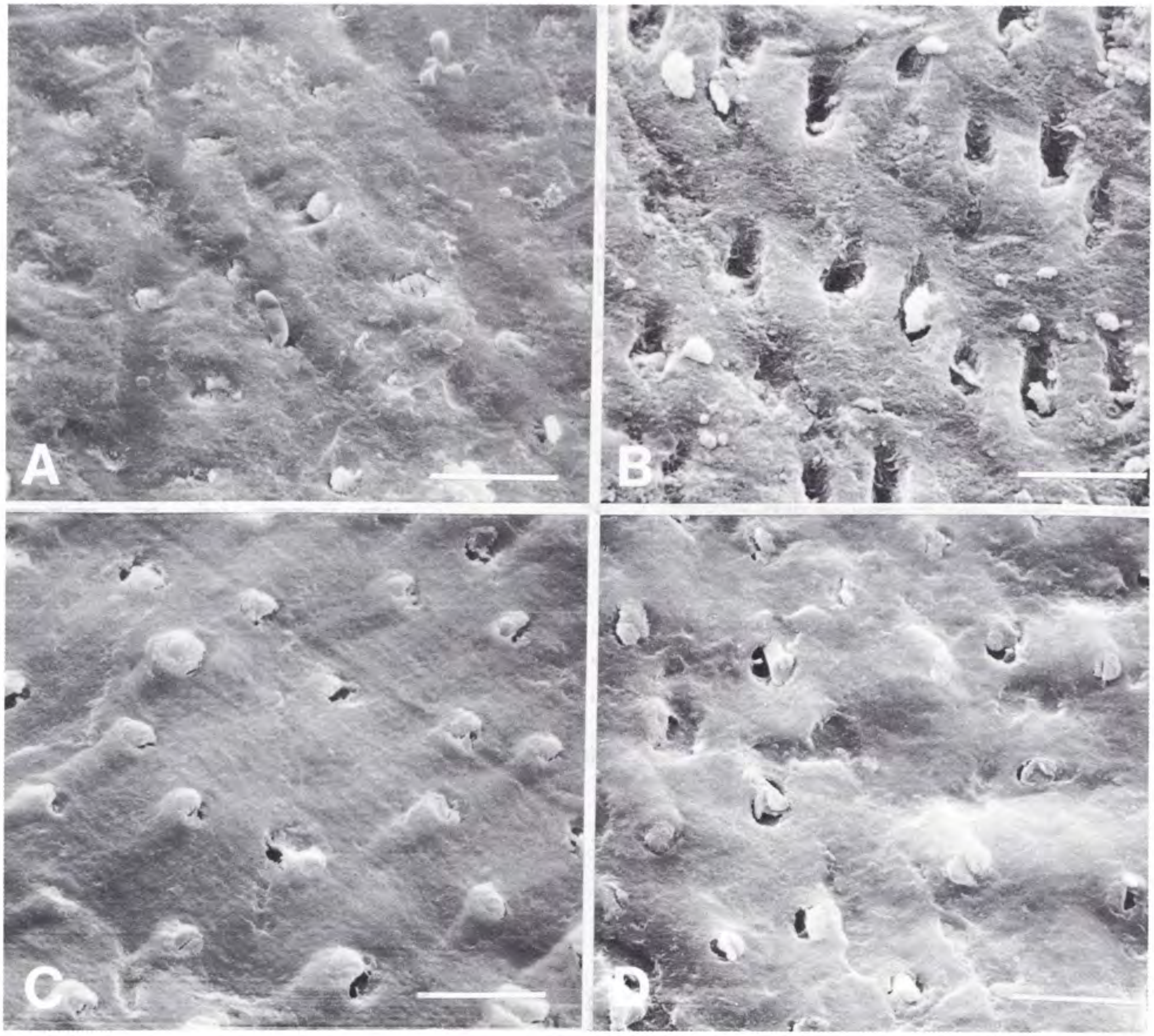

Fig. 6. Dentin aspects of fractured test specimens. A) Fractured specimen with a shear bond strength of 9.3 $MN.m^{-2}$. SEM x2000; B) Fractured specimen with a shear bond strength of 13.5 $MN.m^{-2}$. SEM x2000; C) Fractured specimen with a shear bond strength of 6.1 $MN.m^{-2}$. Etched with 37% H_3PO_4 for 30 seconds. SEM x2000; D) Fractured specimen with a shear bond strength of 13.5 $MN.m^{-2}$. Etched with 37% H_3PO_4 for 30 seconds. SEM x2000. Bar = 10 μm.

failure which occurred within the resin estimated. Further examination of some of the fractured test specimens in the SEM (Fig. 6) revealed that the evaluation of the fracture pattern at x50 magnification (Fig. 5) is not accurate. Fracture sites which were classified as occurring at the resin/dentin interface clearly demonstrated remnants of the restorative system on the exposed dentin surfaces. Etching of these surfaces with 37% H_3PO_4 for 30 seconds showed that an acid-resistant resin layer was present on the dentin surface.

a. 3M Dental Products, St. Paul, MN
b. Buehler Ltd., Lake Bluff, IL
c. Yates and Bird, Chicago, IL
d. Coe Laboratories Inc., Chicago, IL
e. Trans-Temp, W.B. O'Neal, Birmingham, AL
f. Instron Corp., Canton, MA
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