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1．研究テーマ

破折歯の接着強度に関する研究
(Bond Strengths of Adhesively Reattached Root Fracture)

2．本年度の研究業績

(1) 学会・研究会等における発表 (有)・無 (学会名・演題)

1、第37回日本歯科理工学会、演題A-16：破折歯の接着強度に関する研究（第3報）：4-META/MMA-TBB系接着性レジンによる歯根象牙質の接着強さに及ぼす間隙、形状の

2、第38回日本歯科理工学会、演題P-4：破折歯の接着強度に関する研究（第4報）：4-META/MMA-TBB系接着性レジンによる歯根象牙質の接着強さに与えるサーマルサイクルの影響

(2) 学会誌等に発表した論文 有・(無) (雑誌名・論文名)

3．今後の研究計画

今回、『破折歯の接着強度に関する研究』を博士論文試験のテーマとし、三年間の研究実験を進め、よい結果を得た。臨床では成功例が多く発表されている破折歯の接着技法について、歯科理工学の立場から、研究データーおよび治療根拠を提供することができた。微小引張試験法を破折歯研究に用いることにより、成功な臨床治療法を確信され、さらに健康な歯質と接着修復した破折歯の接着強度を比較できることが認められた。

歯科医学は理工材料学よって発展し、これからも理工学によって支えられていく』とよく言われている。歯科研究者と歯科医師に対して、基礎知識が歯科材料の使用や治療方針の決定などにより非常に重要な物である。今後さらに本研究の結果を生かし、微小面積分析の手法を用い、歯根部だけではなく、歯冠部破折の症例にも試み、各種歯科臨床接着治療方法の理論的根拠を明らかにしたい。

4．指導責任者の意見

劉佳君は,1998 年 4 月に専修科生として本学に入学し,翌年より本学大学院博士課程に進学し,３年次を終了するに至っております．この間に,「歯科用銀合金の疲労特性についての研究」「支台歯とされる歯牙の破折と再接着に関する研究」を行い、学会発表５回，原著論文 1 編の報告を行っております．貴協会の助成金により，在学中の経済的困難が軽減され、研究に専念できたものと考えます．

現在，博士論文として「歯牙の破折と再接着に関する研究」を完成させたいと実験をほぼ終了し，データ整理と博士論文の作成に取り組んでおります。

また、学生教育にも積極的に協力し、学生実習の指導に当たっております。現在はその着実で勤勉な態度、真摯な研究態度と勉学意欲からして、十分な成果を期待できるものと確信しております。

指導責任者氏名 小田 豊 ㊞

5．研 究 報 告 書

別紙報告書作成要領により、添付の用紙で研究報告書を作成して下さい。

研究発表中または研究中の本人のスナップ写真を添付して下さい。

※研究成果を発表する場合は、発表原稿・抄録集等も添付して下さい。

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—日中医学協会助成事業—

破折歯の接着強度に関する研究

(Bond Strengths of Adhesively Reattached Root Fracture)

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ABSTACT: An investigation of the bond strength of bovine root dentin was conducted with purpose to provide mechanical evidence for the successive treatment of adhesively reattached vertical root fracture. The microtensile dumbbell specimens were fractured as a vertical root fracture with a tensile force and bonded by 4-META/MMA-TBB adhesive resin. It was found that the better bonding (above 10MPa) to the parallel tubules on the bonding interface of butt joint was due to the compacted gap space and the positive effect of etching treatment. The adhesive treatment in a wedge joint, with oblique tubules on the bonding interface, was revealed that it contributed to the strongest bond strength (about 19MPa) and good durability (after 3000 thermocycling). The test method in this study was proved that it allowed of comparing the fracture and re-fracture resistance of root dentin by reusing the same specimen.

Key Words: Vertical root fracture, Microtensile bond strength, Adhesive resin, Root dentin

INTRODUCTION

Vertical root fracture (VRF) in endodontically treated teeth occurs frequently. As we all known, the selection of treatment often depends on the extent of the fracture lines. The conservation of VRF, a vertical fracture down to the root, is particularly difficult. Tooth extraction is necessary to end treatment at last. Several conventional techniques have been used to repair fractured teeth by tightened wire on the grooves around the tooth crown or by glass ionomer cement. Over the years, due to the advances of dental adhesive systems, the new clinical operation have been challenged for bonding fractured teeth, including the use of cyanoacrylate cement, composite resins, and even fusion of the fracture line by laser. Especially, significant advances have led to a relatively high bonding strength to dentin. The conservational treatment of VRF by adhesively repairing method with 4-META/MMA-TBB resin was reported with a long-term clinical succession[1].

4-META/MMA-TBB resin has a long research and clinical history. Nakabayashi[2] addressed its use on extracted bovine dentin substrates, and introduced firstly the concept of hybrid layer from formation by 4-META/MMA-TBB resin in the demineralized dentin of extracted bovine teeth[3]. It was investigated in vivo research[4] that process good biocompatibility and little cytotoxicity to the periodontal tissue. But no evaluation on the micro-bond strength of repaired VRF was reported. In addition, as for the root dentin, Fogel et al.[5] showed that the permeability of root dentin is much lower than that of coronal dentin.

In order to reveal adhesion mechanism of root dentin and make a mechanical evidence for the bonding treatment of VRF, the purpose of this study was to investigate the effect of surface treatment (polishing and etching), gap space, and the shape of bonding joints (butt joint and wedge joint) on the microtensile bond strengths of 4-META/MMA-TBB adhesive resin to the vertically fractured bovine root dentin, and the durability of adhesion was also evaluated on effect of thermal cycling.

MATERIALS & METHODS

Twelve freshly extracted bovine lower central incisors in frozen storing were used within 6 months following extraction.

Specimen preparation of with parallel tubules on the fractured surface

The roots were sectioned longitudinally in bucco-lingual direction with a low-speed diamond saw (No. 11-4254, Buehler Ltd., USA) under copious water. Two pieces of dentin-slices (distal and mesial) in 1.0mm thickness were obtained from each root. Within two dentin-slices of each root, 10 pieces of dentin specimens could be obtained. (Fig. 1)

The span between the plastic jig was decided on 3.0mm. The rectangular cross-section (1.0×1.5mm) of micro-tensile dumbbell specimen was carefully shaped and trimmed with a diamond bur (Diabur, SF-12, Mani Inc., Japan) in a dental handpiece under copious air-water spray. Specimens were subjected to a tensile load at a crosshead speed of 1.0 mm/min until failure to obtain the ultimate tensile force, and then the microtensile strength was calculated by dividing by the area of rectangular cross-section of dumbbell specimens. The fractured specimens were stored in 0.9% normal saline solution at 37°C for 24hrs prior to reattachment.

Reattachment and measurement of bond strength

Followed by thorough rinsing of the fractured surfaces for 30 seconds with distilled water, the fractured specimens were randomly divided into 10 groups (n=12×10), and the fractured surfaces were conditioned according to different criteria such as polishing treatment, etching treatment, gap spaces, and joint types (Tab. 1). 4-META/MMA-TBB adhesive resin (Super-Bond, Sun Medical Ltd., Japan) was used.

The bonded specimens were stored in 0.9% normal saline solution at 37°C for 24hrs prior to micro-tensile bond test. The half of specimens (n=6×10) were fractured under tensile force (RTC-1150, Orientec, Japan) at a crosshead speed of 1.0mm/min, and the microtensile bond strength (MTBS: MPa) was recorded.

Evaluation on the durability of adhesion

The other half of specimens (n=6×10) was use for investigating the durability of bond strengths in the test of thermal cycling. The bonded specimens were also stored in 37° C 0.9% saline solution for 24 hours prior to loading on the thermal cycling (TC: 4-60° C for 3000cycles, dwell time 20 s). The microtensile bond strengths were also measured.

Statistical analysis

One-way analysis of variance (ANOVA) was used to assess the effect of surface treatments and thermal cycling on microtensile bond strength of root dentin. Data differences between each group were analyzed with the Fisher PLSD (protected the least significant difference).

SEM observations (the bonding interfaces)

The interfaces of various conditioned specimens were ground with a series of increasingly finer silicon carbide abrasive papers up to 1200-grit, and polished with diamond pastes down to a 0.03 μm particle size (Buehler). And then, the specimens were immersed into 6 mol/L HCl solution (30s) and 1% NaClO solution (10min) with rinsing interval, in order to partially remove the inorganic and organic parts of the dentin. The specimens were rinsed thoroughly with the distilled water, air-dried slightly, and stored in silica gel for 24hrs prior to the gold sputter-coated for SEM observation (JEOL, JSM-6340F, Tokyo, Japan).

RESULTS

Bond strength measurement.

According to the result within the same butt joint before the employment of thermal cycling: 1) Bond strengths of both non-treated groups (N, NT) and etched groups (E, ET) were about 10MPa. There were no statistically significant differences, regardless of a wide or narrow gap-space. 2) Bonding of the polished specimens (P, PT and PW) showed significant weak in a result of pre-fracture, without data, before setting up to the test machine. 3) The significant improvement ($p<0.01$) of bond strength (about 5-6MPa) was found due to the additional etching treatment after the polishing (PE, PET). 4) As for the effect of gap space (50 μm and 500 μm) on butt joint specimens, the significant differences were not obtained ($p>0.05$). 5) Between the butt joint and wedge joint, the specimens of wedge joint (PEW) showed the highest value (about 17MPa) with statistically significant difference ($p<0.01$). (Fig. 2)

Due to the influence of 3000 thermal cycles, the bond strength of a wide gap-space (500 μm) in butt joint decreased significantly ($p<0.01$) within non-treated group (about 6MPa), etched group (about 5MPa),

and polished-etched group (about 2MPa), respectively. The specimens with a narrow gap-space (50 μm) of butt joint had no significance exchanges ($p>0.05$). On the contrary, the wedge joint (PEW) was still on stable bond strength with the highest level (about 19MPa).

SEM investigation on a side view of the bonding interface

An image of closed mechanical connection between the resin and dentin surface is showed, even though there is an absence of the hybrid layer (Fig. 4: N and E); the combination seemed not be closely and the extending of resin is not actively because of the absence of resin tags. The absence of connection between resin and dentin was remarkable, regardless of tubule orientation (Fig. 4: P and PW); most of the smear layer and smear-plugs in tubule were removed. The superficial layer of dentin was shown a demineralized image; the layer of resin-infiltrated seems alike the texture named as the hybrid layer (Fig. 4: PE); the hybrid layer occurred in oblique to the tubule orientation. It seems be more regular, entirely than that of PE specimens bonded parallel to the tubules. And much more resin tags and their lateral branches were appeared. Observations of this bonding interface with the oblique tubules seem give a show of well bonding. (Fig. 4: PEW)

There is no any evidently different appearance of bonding situation for each pair of conditioned groups by comparing before-TC and after-TC respectively.

DICUSSION

In our previous study[6], it was indicated that multiple specimens (about 8-10 specimens) were be prepared from one root with the nearly similar tensile strength (36MPa) and parallel tubules on the segment as the fractured surface of VRF. In addition, it was not found the significant influence of dentin locations from the cervical to the apical root on the tensile strength. It was contributed to imitating practically the fractured surface of VRF, and standardizing correctly the original conditions of specimens. On the other way, it makes a possible to measure the stress on different detailed locations that is different with the conventional study by a tapered tip tool inserted into the whole root canal [7,8]. It is better suitable for imitating local situation of a reattached VRF. In addition, this method allows the presence of obtaining a percentage data to compare the fracture resistance (about 36MPa[6]) of intact root dentin and the bond strength of a bonded VRF by the same one specimen at one testing load. In order to determine the correlation of the bond strength with tubule orientations and other surface treatments, bovine dentin was used in this study, although it have a lower resistance compared to human dentin[9].

In this study, we obtained an interesting result in the group of non-treatment (N/NT), a just-fractured situation after tensile force. The bond strength was not showed significant difference to the etched groups (E/ET), and showing an image of closed mechanical connection at side view of the bonded interface even without the appearance of the hybrid layer. In this case of non-treated group, there was no the collapse of the superficial layer of collagen at where the minute networks of collagen fibers was shown on the bonding surface, (Fig. 3). This bonding surface had higher surface energy and surface roughness for the easier spreading of adhesive materials and better interfacial contact. It was also proposed that it is possible to contribute the well adhesion by micromechanical interlocking between the freshly fractured surfaces. And it is considerable for refitting the fractured segment accurately because of less deformation of the fractured surface. Since the good reattachment of fractured segments is also a critical point for successful treatment of teeth fracture.

In this study, the bond strengths retained with the smear layer (P/PT and PW) were very weak, which is contrary with the report of Watanabe[10]. The smear layer has been defined as any debris calcific in nature produced by reduction or instrumentation of dentin, enamel or cementum[11]. The smear layer is composed of hydroxyapatite and altered collagen with an external surface formed by gel-like denatured collagen. As the SEM picture in this study, the smear layer revealed a uniform layer of cutting debris that covered the dentin and obscured the tubules orifices. Because of their inherent weakness, the existing of smear layer can interfere with good dentin adhesion.

The stronger bond strength was improved in the etched group (E/ET and PEW) with a comparing to the polished group (P/PT and PW). The dentin impregnation by applied adhesive monomers was realized only

after removing the weak smear layer. Etching method is commonly used as this actor. The acidic agents remove the smear layer, demineralized the dentin surface, open the dentin tubules, and increase the micro porosity of the intertubular dentin[12]. In this study, the 4-META/MMA-TBB adhesive resin was used due to its good biocompatibility and little cytotoxicity[4] and its successive clinics of the repaired VRF[1]. The 4-META/MMA-TBB resin was unable to adhere to dentine until the development of 10%critic acid and 3%ferric chloride solution to remove the smear layer. It was found that 4-META promoted diffusion of MMA into the dentin and Fe^{2+} in 10-3 solution was also effective in increasing the permeability of demineralized dentin. The 10-3 solution, a kind of shallow etching, tends to prevent collapse of the collagen fibril network and may provide a stiffer interface due to non of over-collapse of collagen fibrils[13]. The achievement of the bond between adhesive resin and dentin depends on the penetration of the primer and adhesive resin into the conditioned dentine surface in order to create micromechanical interlocking between the dentin collagen and resin.

But, the depths and quality of hybrid layer and infiltration of resin tag into the conditioned dentin surfaces were showed different for the perpendicular and parallel tubules[14]. As for the bond strength of adhesion, it was revealed that the value of wedge joint group (PEW: about 13° to tubules) was significantly higher than that of butt joint groups (PE: 0°). The exchangers of bonding areas in two kinds of bond joints can be abbreviated ($\cos 13° \approx 0.97$). The cause of different bond strength was thought that it was the different influence of tubule orientation on the bonding surfaces between both bonding joint. At a right angle to dentinal tubules, relative to parallel tubule, the rate of etched dentin has been shown to be higher when the acids can diffuse down dentinal tubules[15]. On the other way, it was described by Griffiths and Watson[16] that the lateral diffusion can only occur in areas where open dentinal tubules are available for a fast penetration of the acids and following dissolution of the peritubular dentin lining the tubule walls. Resin penetration into tubules can effectively seal the tubules and contribute to bond strength if the resin bonds to the tubules wall[17]. The oblique orientation between the tubules and bonding surface in the case of PEW specimen of this study make it possible to represent.

As for the durability of adhesion, in this study, we invested the effect of 3000 thermal cycles on the root dentin with a thermal change of 4-60° C. Loyd et al.[18] suggested that several thousand thermal cycles might occur *in vivo* in several years. The storage period was 15-30 seconds in each dwelling bath. Noguchi et al.[19] concluded that the desirable thermal cycling temperatures were between 5° C and 60° C that could simulate *in vivo* conditions closely. The situation of root around in the periodontal tissue is different with that of crown, at where it is thought there is little frequent thermal change. But the mechanical stresses can be occurred directly by thermal change through bonded interfaces. Therefore, it is permitted to accelerate a long resting periods by a simulation of the mechanical stresses *in vivo*.

The significant difference of bond strength was not found after 24hrs ether in the groups of 50 μm or 500 μm. But the significant decreasing of bond strength was revealed in the groups of 500 μm after 3000 thermal cycling with temperature exchange from 4° C to 60° C, regardless of various etching or polishing surface treatments. The decreasing of bond strength in the 500 μm groups was thought the mixture action of two factors. One is larger linear thermal expansion of PMMA resin in the 500 μm of gap space than that of 50 μm space. Another reason is the larger water absorption of the mass resin in the 500 μm gap space. At a combined result, the adhesive material between a wide gap spaces is easier to be influenced by the thermal stress.

The results were shown that the durability of the wedge joint (PEW) was stable with the mean value of bond strength about 19MPa, which was different to the specimens with parallel tubules on the bonding surface of butt joint. The force of about 19MPa of the reattached specimens in the wedge joint of this study, which is about 50% of the fractured strength of intact root dentin[6], seems be enough to resist the re-fracture force. Due to the report of Friedman S et al.[7] that the force required re-fracturing the bonded roots was at the most 20% of what it was for intact roots. This percentage was lower than 50% calculated when crowns of teeth were horizontally fractured. In addition, such reinforcement in the case of repaired VRF may have to include an intracanal resistance with adhesive resin used as a root canal filling. The actions of composite resin as root canal fillings or as core materials are being taken

a large regard not only in the fractured root but also as a endodontically treatment of integral root canal to prevent the occurrence of VRF.

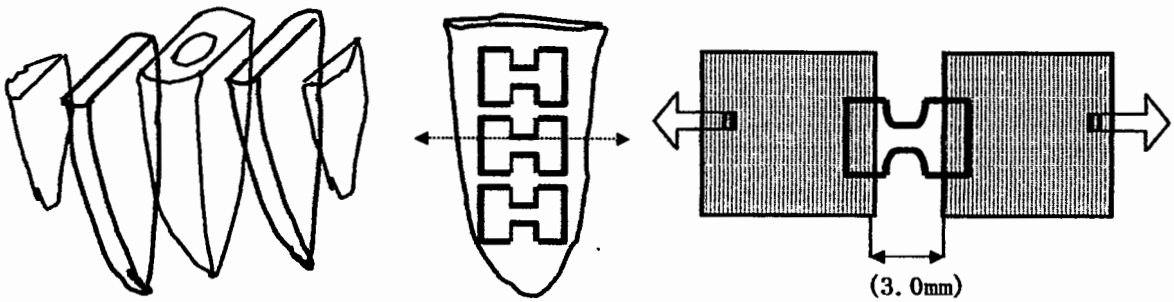

Fig. 1: Specimen preparing

Tab. 1: Assignment and abbreviations of different groups

Joint Types (Gap Space: μm) / Surface Treatments	Butt Joint (50)	Butt Joint (500)	Wedge Joint (50-500)
Non-treated	N	NT	
Etched (10-3 Solution)	E	ET	
Polished	P	PT	PW
Polished and Etched	PE	PET	PEW

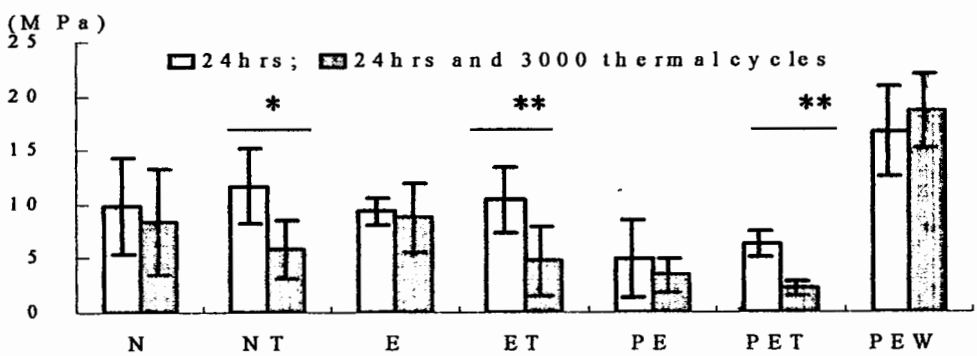

Fig. 2: Bond strength of reattached root dentin before and after thermal cycles. (The bonding of some specimens in the 3 groups of P, PT and PW were too weak to become failures at a pre-fracture before the finishing of thermal cycling test. *: $p<0.05$; **: $p<0.01$)

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