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Effect of Cyclic Loading on the Bond Strength of Class II Restorations with Different Composite Materials

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Clinical Relevance

New resin composite technologies, such as nano-filled-based systems, have been developed. The assessment of such materials in a simulated laboratory chewing condition may assist with the selection of composites in a clinical situation.

SUMMARY

This study evaluated the effect of cyclic loading on the bond strength of Class II restorations using different composite materials. Class II preparations with gingival margins located in dentin were performed on the mesial surface of 80 bovine incisors. The teeth were randomly allocated to eight groups (n=10) according to resin composite (Filtek Z250, Filtek Supreme, Tetric Ceram HB and Esthet-X) and use of cyclic loading. The restorations were bonded with the Single Bond adhesive system. Simulated aging groups were cyclic loaded for 200,000 cycles with 80N load (2Hz). The specimens were vertically sectioned (two slabs per restoration) and further trimmed into an hour-glass shape at the adhesive interface to obtain a final bonded area 1 mm². Samples were placed in an apparatus and tested under tension using a universal testing machine. The data were analyzed using two-way ANOVA and Tukey test with a 95% confidence level. Aged groups presented significantly lower means when compared to the groups that were not aged (*p*=0.03). However, significant differences among composite materials were not observed (*p*=0.17). Regardless of the restorative composite material used, it could be concluded that the bond strength of Class II restorations at the gingival wall was affected by simulated cyclic loading.

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INTRODUCTION

In the past, patients' demands for posterior composite restorations had significantly increased, and the longevity of these restorations has become a common concern among researchers and clinicians.[1-2] The durability of an adhesive restoration seems to be closely related to the integrity of the tooth-restorative material interface. Failures within the bonded interface may result in undesirable effects, such as post-operative sensitivity, marginal staining, recurrent caries and pulp pathology.[3] Several aspects can contribute to the poor performance of posterior restorations, including composite shrinkage stresses, thermal/mechanical fluctuations, light attenuation,[4-6] technique sensitivity and incorrect indication. In addition, posterior restorations with margins in dentin, such as Class II and Class V preparations, are more challenging, since dentin bonding is less predictable than enamel bonding.[7]

An ideal restorative material should be able to resist stresses that are present in the oral environment and protect the interface between the tooth and the restorative material. However, there is no consensus in the literature regarding the most appropriate composite material to be used in direct posterior restorations. Among the many kinds of restorative materials that are available for posterior restorations are universal composites, which can be used both in anterior and posterior teeth (microhybrid composites and monomodal composites) and composites specifically designed for the restoration of posterior teeth. Recently, nanofilled composites were introduced into the market and, according to their manufacturers, these resin-based materials have adequate properties that can be used in stress-bearing applications, such as posterior restorations.[8]

Long-term clinical trials of adhesive restorative materials are the most efficient methods of evaluating the durability of restorations.[9] However, due to the rapid development of resin-based materials, it is difficult to track the evolution of these materials and collect sufficient clinical data to evaluate their durability.[10] To overcome this limitation, laboratory methods that simulate some conditions of the oral environment were developed, such as cyclic loading.[5,11-13] These *in vitro* simulated tests could accelerate deterioration of the interface between dentin and the restoration, providing a better evaluation of these restorative materials.[5]

This study evaluated the effect of cyclic loading on bond strength at the gingival wall of Class II restorations using different composite materials (three universal composites [Filtek Z250, Filtek Supreme and Esthet-X] and one posterior composite [Tetric Ceram HB]). Two null hypotheses were tested: 1) no differences in bond strength at the gingival wall will be observed among resin composite materials and 2) cyclic loading will not affect dentin bond strength at the gingival wall of Class II restorations.

METHODS AND MATERIALS

Eighty extracted bovine incisors were collected, cleaned and stored in 0.1% thymol solution. The incisal surfaces were horizontally sectioned 5.0 mm above the cement-enamel junction (CEJ) using a double-faced diamond disc (KG Sorensen, Barueri, SP, Brazil), allowing for the configuration of a flat standard occlusal surface (Figures 1A and 1B). The teeth had part of their roots embedded in cold cure polystyrene resin (Cromex, Piracicaba, SP, Brazil)

Class II slot preparations were prepared on the mesial surface using a high-speed #245 carbide bur (KG Sorensen, Barueri, SP, Brazil) under constant water-

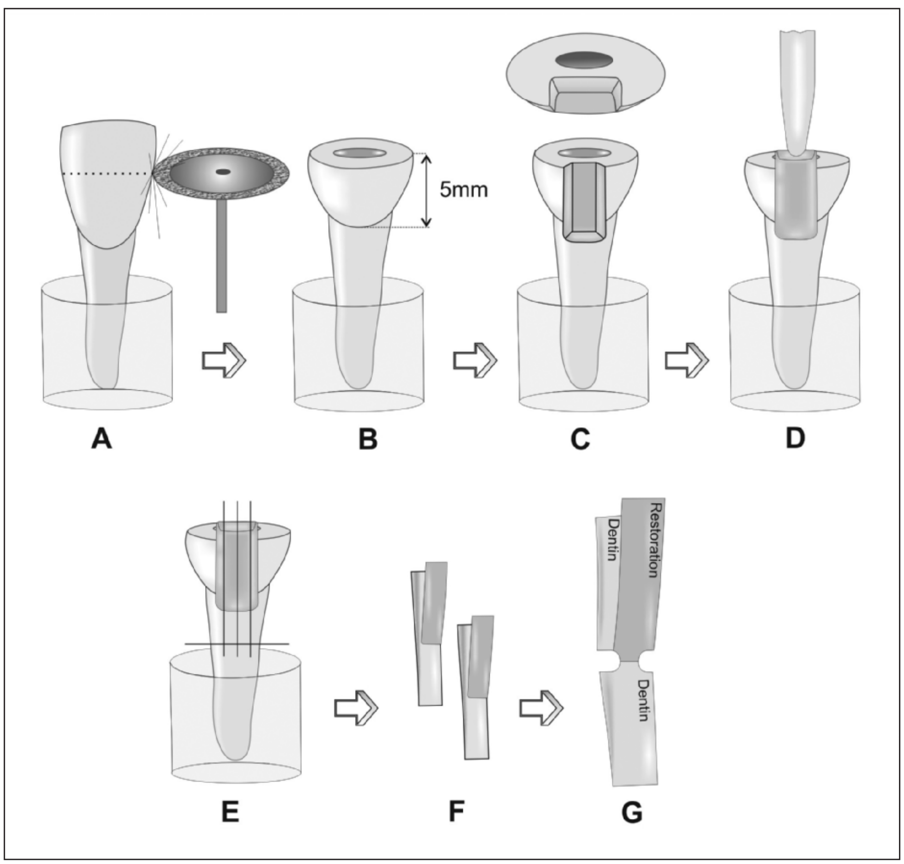

Figure 1: A) *Sectioning the tooth with a double-faced diamond disc; B) Flat surface 5.0 mm above the CEJ; C) Class II slot preparation in the proximal and occlusal-gingival directions; D) Loading device on top of the restoration; E) Restoration sectioning; F and G) slab configuration for microtensile bond strength test (F and G).*

Table 1: *Materials Used in This Study, Their Classification, Manufacturer and Composition*

Material/Classification	Manufacturer	Composition*
Filtek Z250/Universal composite	3M ESPE, St Paul, MN, USA	Bis-GMA, UDMA, Bis-EMA, TEGDMA, zirconia/ silica fillers.
Filtek Supreme/Universal nanocomposite	3M ESPE, St Paul, MN, USA	Bis-GMA, UDMA, Bis-EMA, TEGDMA, nanosilica filler.
Esthet-X/Universal composite	Dentsply DeTrey, Konstanz, Germany	Urethane modified Bis-GMA-adduct, Bis-EMA, TEGDMA, photo initiators, stabilizers, barium fluoro alumino boro silicate, highly dispersed silicon dioxide.
Tetric Ceram HB/Posterior composite	Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, urethane dimethacrylate, decandiol dimethacrylate, barium glass, barium alumino fluorosilicate glass, ytterbium trifluoride, silicon dioxide, speroid mixed oxide, additives, catalysts, stabilizers, pigments.

Abbreviations: Bis-GMA: Bisphenol A diglycidyl ether dimethacrylate; UDMA: urethane dimethacrylate; Bis-EMA: Bisphenol A polyethylene glycol diether dimethacrylate; TEGDMA: triethylene glycol dimethacrylate
**Provided by the manufacturer*

cooling. The cavity preparations were 4.0 mm wide, 6.0 mm high (1.0 mm below the CEJ) and 1.5 mm deep toward the pulp chamber (Figure 1C). The burs were replaced after every five preparations. The inner angles of the cavities were rounded and the margins were not beveled. Dentin morphology at the gingival wall resulted in bonding parallel to enamel rod and dentin tubule orientation, where lower bond strengths were expected when compared to using the ends of prisms/tubules.

The cavities were randomly allocated into eight distinct groups according to the composite material used and the occurrence of cyclic loading. The materials were tested in two different conditions (submitted or not submitted to the cyclic loading) and each material served as its own control. The experimental groups were as follows:

G1—Filtek Z250 without cyclic loading

G2—Filtek Supreme without cyclic loading

G3—Tetric Ceram HB without cyclic loading

G4—Esthet-X without cyclic loading

G5—Filtek Z250 + 200,000 load cycles

G6—Filtek Supreme + 200,000 load cycles

G7—Tetric + 200,000 load cycles

G8—Esthet-X + 200,000 load cycle

The preparations were bonded using the Single Bond (3M ESPE, St Paul, MN, USA) adhesive system, following manufacturers' instructions. The dentin surface was etched with 35% H_3PO_4 for 15 seconds, rinsed with water for 15 seconds and gently air dried for two seconds. Two consecutive coats of adhesive were applied, lightly air dried for two seconds and light-cured (Optilux 501, Sybron Kerr, Danbury, CT, USA) for 10 seconds.

The resin composite materials used in the current study are listed in Table 1. Class II preparations were restored using the respective composite material and three 2.0 mm horizontal increments. Each increment was light-cured for 20 seconds through the occlusal surface by maintaining the light tip in contact with the occlusal surface. A 1.0 mm overfill was left on the occlusal surface to enable cyclic loading over the restorative material only.[14] During all restorative procedures, light output of the light-curing unit was measured and found to be greater than 660mW/cm^2. Upon completion of the restorative procedures, the specimens were stored in distilled water at 37°C for 24 hours. They were then finished and polished with Al_2O_3 abrasive discs (Sof-Lex Pop-On, 3M ESPE).

Cyclic Loading

Cyclic loading was conducted in a Mechanical Loading Machine (Erios International, São Paulo, SP, Brazil) with a 15.0 mm cylindrical metallic tip attached to a steel bar placed in contact with the restoration (Figure 1D). The loading device delivered an intermittent axial force of 80 N at two cycles/seconds. The specimens were maintained in water at 37°C during the 200,000 load cycles.

Microtensile Bonding Test

The restorations were sectioned perpendicular to the cervical bonded interface of each tooth (Figure 1E) into 1.0 ± 0.2 mm thick slabs (n=2 per restoration) (Figure 1F) using a slow-speed diamond blade (Buehler, Lake Buff, IL, USA) and constant water coolant. The slabs were trimmed into an hour-glass shape at the adhesive interface using a #1093FF fine diamond bur (Injecta, Diadema, SP, Brazil) to obtain a final bonded area of 1 mm^2 (Figure 1G). The specimens were then mounted in a testing apparatus with cyanocrylate adhesive (Super Bonder; Henckel Loctite, Itapevi, SP, Brazil) and tested in tension using a universal testing machine (DL 500; EMIC Ltd, São José dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/minute until failure. The means and standard deviations were calculated and expressed in MPa. Statistical analysis was performed using two-way ANOVA and Tukey's test at a 95% confidence level.

Table 2: *Mean Values—Expressed in MPa—(standard deviations) on the Microtensile Bond Strength at the Gingival Wall of Class II Restorations*

Composite Material	Mechanical Loading		
	Control	200,000 Cycles	
Filtek Z250	25.6 (8.4)	23.9 (6.1)	a
Filtek Supreme	23.2 (7.7)	18.1 (6.3)	a
Esthet-X	22.7 (8.1)	17.3 (4.9)	a
Tetric Ceram HB	23.6 (4.9)	21.5 (6.9)	a
	A	B	

Same letters are not statistically different (two-way ANOVA/Tukey test, α=0.05). Upper case letters compare mechanical loading. Lower case letters compare composite materials. Coefficient of variation = 30%.

RESULTS

Table 2 shows the bond strength values and standard deviations. Two-way ANOVA did not show a significant interaction (p=0.77) between the two factors—restorative composite and cyclic loading. Cyclic loading resulted in a significant decrease in bond strength at the gingival wall of all restorative composites (p=0.03). Nevertheless, no significant differences among composites were observed (p=0.17). Even though bond strengths decreased after mechanical stress, the restorative materials continued presenting similar performance after loading.

DISCUSSION

Resin composite restorations are technique sensitive, particularly when placed in posterior teeth where access, visibility and moisture are difficult to control.[15] The current study was conducted to investigate whether different types of composite materials behave differently in posterior restorations submitted to mechanical fatigue. A nanofilled composite (Filtek Supreme), a composite indicated only for posterior restorations (Tetric Ceram HB) and two universal composites (Filtek Z250 and Esthet-X) were selected due to their different characteristics. Class II restorations were placed in bovine incisors, and dentin bonding was tested at the gingival wall of the proximal preparations. The use of bovine incisors to replace human teeth was intended to standardize the age of the teeth, sclerosis, wear and dimensions.[1]

Degradation of the adhesive interface due to mechanical loading is a very important issue in restorative dentistry, since the demand for aesthetic restorations in posterior teeth has significantly increased. Cyclic loading is based on the application of repeated load cycles that simulate some clinical chewing movements.[10] Several studies have used mechanical loading to artificially age composite restorations.[5,9-10,14,16-17] However, as a general rule, the findings of such studies cannot be compared because of discrepancies in their methodologies, such as the wide range of load applied (from 50N to 125N) and the number of cycles (from 4,000 to 500,000).[5,10,18-19] The current study was conducted following the same methodology and number of mechanical load cycles proposed by a previous study.[5] Mitsui and others[5] stated that 200,000 mechanical cycles can significantly affect the bond strength of the Single Bond system at the gingival wall of Class II preparations. Therefore, in order to investigate whether the restorative material would have a significant effect on bond strength at the gingival wall, the same number of mechanical cycles was used in the current study.

According to the current study, none of the four resin composites evaluated was able to prevent a decrease in bond strength at the gingival wall following cyclic loading. All the resin composites presented significantly lower bond strength after mechanical fatigue. A possible explanation for this finding is that the composites were not able to absorb stresses from cyclic loading. Therefore, this strain is completely transmitted to the bonding interface. Studies have suggested that posterior restorations should use an intermediary resin layer with a low elastic modulus to act as a stress absorbing layer and preserve the adhesive bond.[20-21] However, in a previous study,[13] the authors observed that the intermediary layer, which is more flexible, had no significant effect on bond strength to dentin, and groups with and without the intermediary layer presented the same performance after being submitted to thermal and mechanical stresses. Also, according to De Munck and others,[22] once an intimate bond that can withstand polymerization shrinkage is created, no extra elastic properties appear necessary to withstand the stress generated by chewing and clenching.

The second explanation for the lower bond strength observed after mechanical loading could be related to the adhesive system used in this study. Single Bond is an etch-and-rinse system that presents Bis-GMA (bisphenol-A-diglycidyl ether dimethacrylate) and HEMA (2-hydroxyethyl methacrylate) as its main chemical components. An outcome commonly related to etch-and-rinse systems is incomplete resin infiltration of hydroxyapatite-depleted collagen.[7] This suboptimal resin infiltration can lead to a porous zone within the hybrid layer, compromising durability of the bond.[7,23] In addition, it was stated that HEMA monomer reacts with the collagen fibrils in dentin to form either hydrogen bonding or a new bond to the ester group of

HEMA.[24] However, hydrogen bonding might be reduced by thermal and mechanical tensions, or by the presence of intrinsic water contained within the collagen fibrils in the moist bonding technique.[5,25] If demineralized dentin at the gingival location of Class II preparations is too wet and not fully hybridized, it can become vulnerable to hydrolytic breakdown, compromising the integrity and strength of the bonded interface.[26] In the current study, use of the wet bonding technique and cyclic loading under water may have resulted in a combined effect that was accountable for the lower bond strengths after simulated loading. In addition, since the restorations were loaded in the presence of water, degradation of the interface components could have played a fundamental role in decreasing the restorations' bond strength.

Although the composite materials investigated presented different resinous matrices and filler contents, they did not demonstrate a significant effect on dentin bonding. A previous study[27] observed significant differences in the dentin bond strengths of Class I restorations with eight commercially available composite materials (hybrid, microfilled and nanocomposite). The eight composite materials presented a highly significant correlation between shrinkage stress and bond strength to dentin.[27] According to the authors, the extent of stress that developed within the composite materials during polymerization, using the same light exposure and technique, depended on its inherent properties, such as chemical composition and fraction of the matrix, reinforcing material and silane, the rheology and viscosity of the paste, the flow behavior of the material during the pre-gel phase and duration of the pre-gel phase, the type and amount of the initiator systems, the inherent defects during sample preparation, temperature and humidity.[27]

Hence, stress developed during composite shrinkage and its effect on the quality of dentin bonding will also vary according to the geometry of the cavity (C-factor).[6] Although Class II preparations present a lower C-factor when compared to Class I preparations, polymerization shrinkage will be present and might impair dentin bonding at the gingival wall. In this study, despite the lack of differences among the restorative materials, it could be suggested that stress developed during polymerization shrinkage could have affected the dentin-composite interface, enabling a higher effect of cyclic loading.

According to the findings of this study, the first null hypothesis was accepted, since restorative materials did not present a significant effect on dentin bonding, while the second null hypothesis must be rejected, because bond strengths decreased in all groups after cyclic loading.

CONCLUSIONS

Within the limitations of this study, it can be concluded that dentin bond strengths at the gingival wall of Class II preparations were affected by simulated cyclic loading but not by the type of resin composite used.

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References

1. Spencer P, Wang Y & Bohaty B (2006) Interfacial chemistry of moisture-aged Class II composite restorations *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **77(2)** 234-240.
2. Raj V, Macedo GV, Ritter AV & Swift EJ Jr (2007) Longevity of posterior composite restorations *Journal of Esthetic and Restorative Dentistry* **19(1)** 3-5.
3. Van Meerbeek B, Perdigão J, Lambrechts P & Vanherle G (1998) The clinical performance of adhesives *Journal of Dentistry* **26(1)** 1-20.
4. Vandewalle KS, Ferracane JL, Hilton TJ, Erickson RL & Sakaguchi RL (2004) Effect of energy density on properties and marginal integrity of posterior resin composite restorations *Dental Materials* **20(1)** 96-106.
5. Mitsui FHO, Peris AR, Cavalcanti AN, Marchi GM & Pimenta LAF (2006) Influence of thermal and mechanical load cycling on microtensile bond strengths of total and self-etching adhesive systems *Operative Dentistry* **31(2)** 240-247.
6. Feilzer AJ, de Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.
7. Van Meerbeek B, Van Landuyt K, De Munck J, Hashimoto M, Peumans M, Lambrechts P, Yoshida Y, Inoue S & Suzuki K (2005) Technique-sensitivity of contemporary adhesives *Dental Materials Journal* **24(1)** 1-13.
8. Lopes GC & Oliveira GM (2006) Direct composite resin restorations in posterior teeth *Compendium of Continuing Education in Dentistry* **27(10)** 572-579; quiz 580-571.
9. Nikaido T, Kunzelmann KH, Chen H, Ogata M, Harada N, Yamaguchi S, Cox CF, Hickel R & Tagami J (2002) Evaluation of thermal cycling and mechanical loading on bond strength of a self-etching primer system to dentin *Dental Materials* **18(3)** 269-275.
10. Bedran-de-Castro AK, Pereira PN, Pimenta LA & Thompson JY (2004) Effect of thermal and mechanical load cycling on microtensile bond strength of a total-etch adhesive system *Operative Dentistry* **29(2)** 150-156.
11. Bouillaguet S, Ciucchi B, Jacoby T, Wataha JC & Pashley D (2001) Bonding characteristics to dentin walls of Class II cavities, *in vitro Dental Materials* **17(4)** 316-321.

12. De Munck J, Van Landuyt K, Coutinho E, Poitevin A, Peumans M, Lambrechts P & Van Meerbeek B (2005) Microtensile bond strength of adhesives bonded to Class-I cavity-bottom dentin after thermocycling *Dental Materials* **21(11)** 999-1007.

13. Cavalcanti AN, Mitsui FH, Ambrosano GM & Marchi GM (2007) Influence of adhesive systems and flowable composite lining on bond strength of Class II restorations submitted to thermal and mechanical stresses *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **80(1)** 52-58.

14. Bedran-De-Castro AK, Pereira PN & Pimenta LA (2004) Long-term bond strength of restorations subjected to thermo-mechanical stresses over time *American Journal of Dentistry* **17(5)** 337-341.

15. Baratieri LN, Ritter AV, Perdigão J & Felippe LA (1998) Direct posterior composite resin restorations: Current concepts for the technique *Practical Periodontics & Aesthetic Dentistry* **10(7)** 875-886; quiz 888.

16. Bedran-de-Castro AK, Pereira PN, Pimenta LA & Thompson JY (2004) Effect of thermal and mechanical load cycling on nanoleakage of Class II restorations *Journal of Adhesive Dentistry* **6(3)** 221-226.

17. Mitsui FH, Bedran-de-Castro AK, Ritter AV, Cardoso PE & Pimenta LA (2003) Influence of load cycling on marginal microleakage with two self-etching and two one-bottle dentin adhesive systems in dentin *Journal of Adhesive Dentistry* **5(3)** 209-216.

18. Abdalla AI & Davidson CL (1993) Comparison of the marginal integrity of *in vivo* and *in vitro* Class II composite restorations *Journal of Dentistry* **21(3)** 158-162.

19. da Cunha Mello FS, Feilzer AJ, de Gee AJ & Davidson CL (1997) Sealing ability of eight resin bonding systems in a Class II restoration after mechanical fatiguing *Dental Materials* **13(6)** 372-376.

20. Kemp-Scholte CM & Davidson CL (1990) Complete marginal seal of Class V resin composite restorations effected by increased flexibility *Journal of Dental Research* **69(6)** 1240-1243.

21. Montes MA, de Goes MF, da Cunha MR & Soares AB (2001) A morphological and tensile bond strength evaluation of an unfilled adhesive with low-viscosity composites and a filled adhesive in one and two coats *Journal of Dentistry* **29(6)** 435-441.

22. De Munck J, Van Landuyt KL, Coutinho E, Poitevin A, Peumans M, Lambrechts P, Braem M & Van Meerbeek B (2005) Fatigue resistance of dentin/composite interfaces with an additional intermediate elastic layer *European Journal of Oral Sciences* **113(1)** 77-82.

23. Phrukkanon S, Burrow MF & Tyas MJ (1999) The effect of dentine location and tubule orientation on the bond strengths between resin and dentine *Journal of Dentistry* **27(4)** 265-274.

24. Xu J, Stangel I, Butler IS & Gilson DF (1997) An FT-Raman spectroscopic investigation of dentin and collagen surfaces modified by 2-hydroxyethylmethacrylate *Journal of Dental Research* **76(1)** 596-601.

25. Hashimoto M, Ohno H, Kaga M, Endo K, Sano H & Oguchi H (2001) Fractographical analysis of resin-dentin bonds *American Journal of Dentistry* **14(6)** 355-360.

26. Purk JH, Dusevich V, Glaros A & Eick JD (2006) Adhesive analysis of voids in Class II composite resin restorations at the axial and gingival cavity walls restored under *in vivo* versus *in vitro* conditions *Dental Materials* **23(7)** 871-877.

27. Ilie N, Kunzelmann KH & Hickel R (2006) Evaluation of micro-tensile bond strengths of composite materials in comparison to their polymerization shrinkage *Dental Materials* **22(7)** 593-601.