



METHODOLOGY OF FATIGUE TESTS FOR GLUED DENTAL SAMPLES

Tomasz Topoliński, Mateusz Wirwicki

University of Technology and Live Sciences in Bydgoszcz
ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland
tel.: +48 52 3408497, fax: +48 52 3408245
e-mail: wirwicki@utp.edu.pl

Abstract

In human teeth, depending on the age at which it is located, there are two types of teeth: milk and solid. During the consumption of various food groups of teeth meet their unique tasks. During Eating teeth are exposed to cyclic loading resulting in microcracks in the structure of the tooth. Using special preparations of dental enamel can be broken by bind to the tooth. The article presents the strength tests for determination of different bonding characteristics of the tooth. Also presents a literature review was divided into groups: the test material and methods of researchers. Of all disguised literature describes and compares the two close to each endurance test methods for tissue tooth blade in tempered. Methods of Testing was conducted under conditions of low and high cyklowych. Summarizes the methods shown and described as would appear the author's own research.

Keywords: *strength tests, human dentition, molar teeth, dentin*

1. Introduction

Human dentition is divided into two categories depending on the stage of life: primary teeth and permanent teeth. Primary dentition appears in children at the age between 2 and 6. Altogether, there are 20 teeth, 10 in the lower jaw and 10 in the upper jaw. Permanent teeth replace the primary ones in children aged between 6 to 14. There are 32 permanent teeth in total and they are categorised into several groups: incisor teeth, canine teeth, premolar teeth and molar teeth [pic. 1]. During consumption of various foods, these groups play different and vital functions. The basic role of incisors is to chop bits of food, facilitate proper pronunciation and construct the external mouth profile. Next type of teeth, canine teeth, support lip muscles, chop and smash food and protect premolar teeth from chewing pressure. Then, premolar and molar teeth, together called molars, are necessary for grinding and chewing of food to make it easy for further assimilation in the body [23]. Picture [2] presents anatomical construction of a tooth. Four main tissues can be distinguished. Enamel, dentine, root cement and pulp. Mineralised elements include dentine and cement. The pulp is vascularised and innervated. The solid tissue located on the surface protects it from external factors [23].

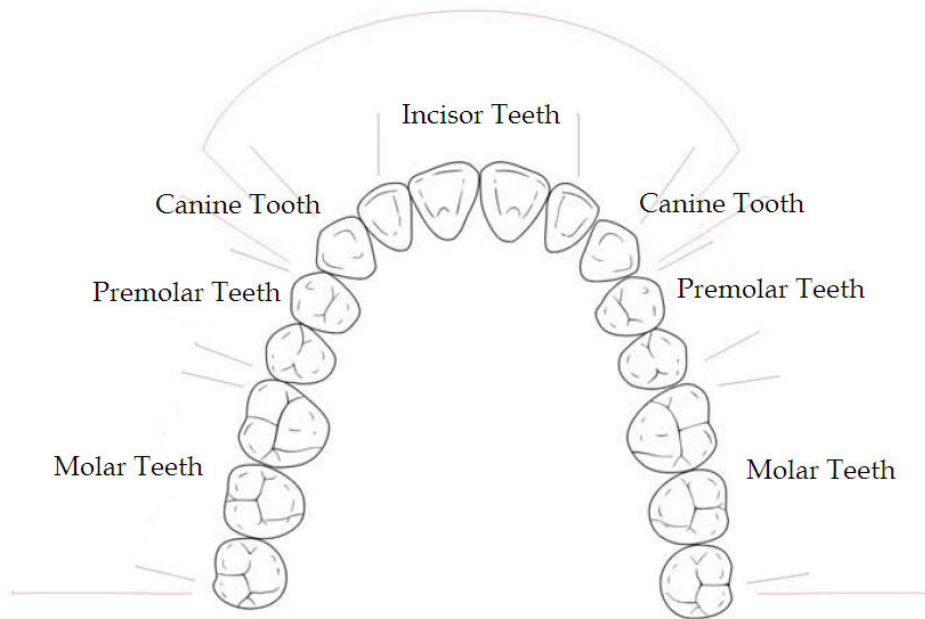


Fig. 1 Permanent teeth - occlusion view [23]

During food consumption, teeth can be affected by cyclical load which causes micro cracks in the tooth structure. Deepening of cracks in the tooth tissue may lead to damage or partial chipping of the material. Tooth construction analysis confirms that the damage occurs mainly at the dentine – enamel junction, enamel as such gets fractured more rarely. In some instances, the pulp cavity gets open and tooth nerves become destroyed.

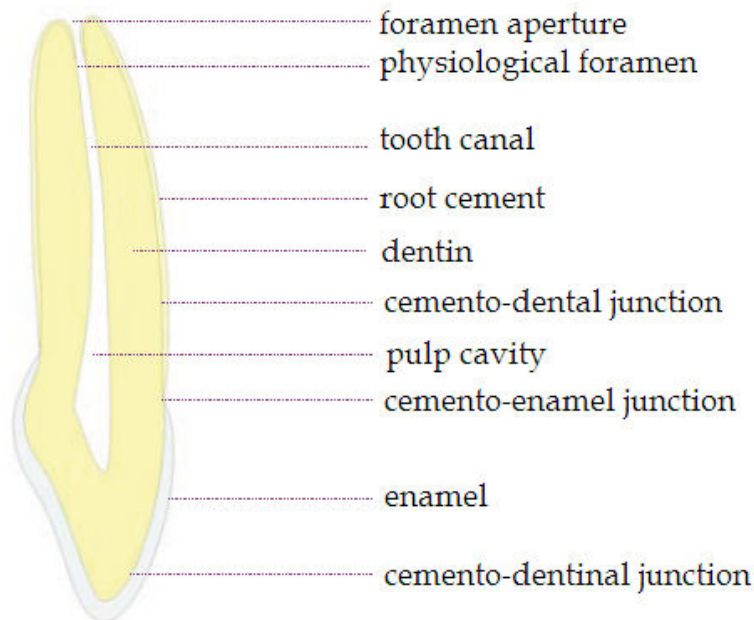


Fig. 2 Tooth anatomy layout [23]

Fatigue – endurance tests are classified on the basis of the analysed material or on the basis of the analysis method and are grouped in two categories. The first group includes the analysis of:

- natural teeth, especially human, e.g.: [1], [2], [3], [4], [6], [7], [9], [10], [12], [14], [16], [17], [18], [19], [20], [28], which is further divided into the analysis of incisor teeth, e.g.: [12], [14], [16], premolar teeth: [1], [6] and molar teeth: [2], [10]
- false teeth, e.g.: [13], [15], [31]
- bovine teeth, e.g.: [5], [28],
- dental materials, e.g.: polymer resin [21], [22], [30], cement: [21], junction technologies [7], [21].

The second category comprises various analysis methods. It can be divided into:

- static tests, e.g.: [2], [5], [15], [18], [19], [21], [26], [30], [31],
- dynamic tests, e.g.: [1], [2], [4], [6], [7], [9], [10], [12], [13], [14], [15], [16], [17], [21], [22], [30], [31], high-cycle: [1], [10], [13], [16], [17], [22], and low-cycle: [1], [3], [4], [12], [14], [17], [20], [28],

Static testing helps to determine pressure and deformations which the material undergoes due to high, higher than average, load. In article [18] authors analyse the endurance of metacrylan dental glues exposed to wet and dry conditions.

Test were conducted for cylinder shaped samples (beams), bent at 3 points. Article [26] presents fatigue tests of a stretched bovine sample. After trimming, the samples were stored in salt solution and at the end of the test their structure was scanned. In article [19], authors selected carefully researched material and attempted to test by damaging samples statically at the angle of 45°. Damaged points were analysed thoroughly. The article concludes that teeth which were additionally filled up with composite glue demonstrated better endurance than those which underwent tests right after gluing. In article [5] authors used as test material molar bovine teeth. They compared 3 composite types of glue hardened by light. Their results indicate that there is a huge range for a tooth fracture resistance, which can reach even up to 60%.

Dynamic testing constitutes the second category of analysis method. It can be divided into high-cycle and low-cycle. Paper [28] belongs to the former group. Authors present a new view on fatigue testing for glued junctions during teeth mineralisation. They also mention the analysis environment – wet and dry conditions. Samples are tested in 40.000 cycles at the frequency of 0.25Hz. Article [3] describes tests in which 4 “all in one” glues were used in 5×10^4 cycles at the frequency of 0.2Hz. Article [4] presents two methods of sample preparation for tests in a material testing machine. In the first method, similar to the above mentioned, the analysed element is trimmed to match the size of testing machine jaws. In the second method, the sample is fixed in metal attachment point (2mm in height) which is fixed to the material testing machine. Article [20] presents low-cycle dynamic tests. By the selection of modern glues and modified resin, samples are destroyed in 10^4 cycles. Concluding from authors work [20], “all in one glues” and self etching glues do not provide any substantial resistance to fatigue.

This paper will focus on low-cycle tests. Similar analysis samples were user for the two methods described below.

2. Materials and methods

2.1. Dynamic and static bond-strength [24]

Tests were conducted on healthy and selected molar teeth. Before the analysis, samples were kept in water and chloramine 0,5% solution at the temperature of 4°C. Teeth were fixed in a gypsum block and trimmed by a saw ISOMET 1000. The cut surface was checked under a microscope for any remaining residue or enamel.

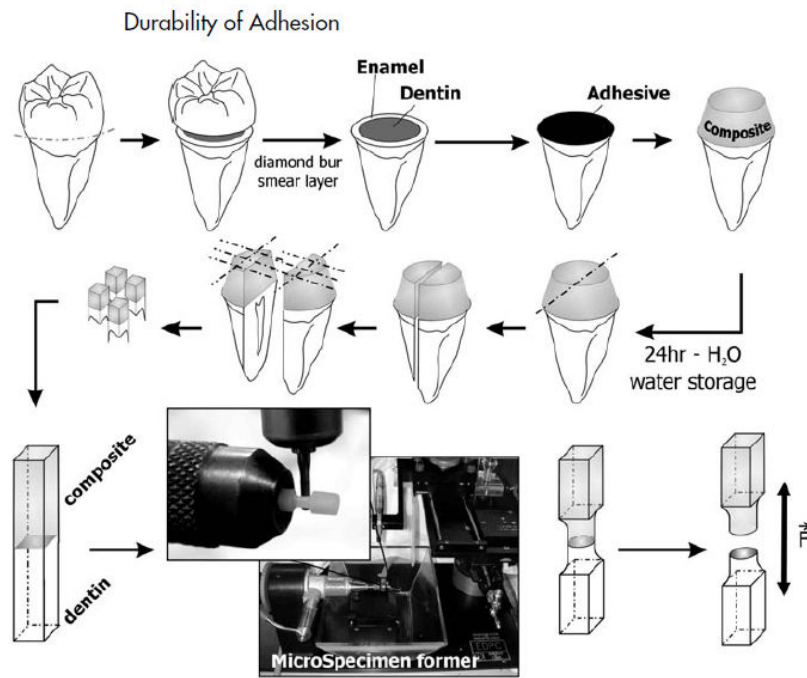


Fig 3. Layout of sample preparation and the conduction of endurance tests [8]

The samples were analysed for glue 3-E & Ra: OptiBond FL. The producer's procedure was applied by gluing. After gluing, surfaces were complemented with composite resin Z100. Each of the applied resins had to be hardened by the light from Optilux 500 machine for 40s. After 7 days the samples were trimmed down to 6mm in height, which enables static stretch and fatigue analysis. Materials were placed in a testing machine INSTRON 5848 and stretched with 500N where the speed of relocation for upper jaw equalled 1mm/min. μ TBS index – static stretching – was introduced in the analysis. Dynamic – cyclical load was indexed as μ TFR for samples which were tested at the frequency of 2Hz and for which 10Hz frequency caused destruction or when cycles reached the value of 10^4 , which equalled 3 months of constant food chewing in the oral cavity. Samples were divided into 3 groups: samples analysed directly after gluing, samples stored in the solution for one week and samples stored in the solution for one month.

2.2. Fatigue of dentin-composite [27]

Samples for analysis were extracted from human molar teeth which were sterilised with gamma radiation [4]. The samples were trimmed to the dimension 1.1mm x 1.1mm x 6mm. The surface was polished with 600 thick sand paper. Gluing point was located in the middle of the dental crown height. The junction of composite with the remaining part of the tooth was created of 3 layers. Firstly, the glued surface had to be prepared. Etching liquid (SE Bond Primer) was used to this purpose and spread on the glued surface. Then, resin glue (SE Bond), previously hardened by light for 20sec., was placed. Finally, the composite was applied. It was formed to the shape of the analysed beam. After initial polymerisation, samples were placed in a water container for 24 hours.

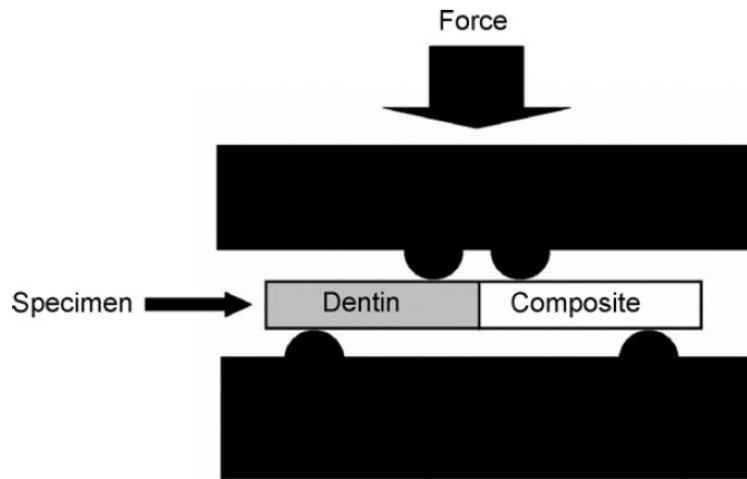


Fig. 4. Four points of sample bent layout[27]

In the first stage of the experiment, a test of static bent of elements until destruction was carried out. Upper jaw relocation grew linearly every 0.001mm/sec. The second group of samples was put to cyclical load test until destruction point or 10^6 cycles.

3. Results

3.1. Method 1 [2]

The results of static and dynamic load for the samples glued with the OptiBond FL glue are presented in table 1. It is noticeable that highest scores are found in tests for μ TFR at 2Hz for samples stored 1 week. Analysing the chart presented in picture 5, which demonstrates loads for the sample glued at various cycles and loads, one can see that at low pressure values (up to 21MPa) the tested material does not become destroyed. It was only the sample which underwent cyclical loads of 2Hz and was previously stored for 3 months in the solution that was damaged quickest. The sample showed material fatigue already at 19.7Mpa. Picture 6 presents the percentage of surface fracture for different fatigue tests (glue OptiBond FL).

Tabele 1. Static and dynamic stretching divided according to storage period[24]

| Glue | μ TBS [MPa] | μ TFR 10 Hz after first week [MPa] | μ TFR 2 Hz po after first week [MPa] | μ TFR 2 Hz after 3 months |
|----------------------|-----------------|----------------------------------------|------------------------------------------|-------------------------------|
| -E & Ra: OptiBond FL | 51. \pm 17.6 | 20.5 \pm 16 | 21.3 \pm 17.4 | 19.7 \pm 16.6 |

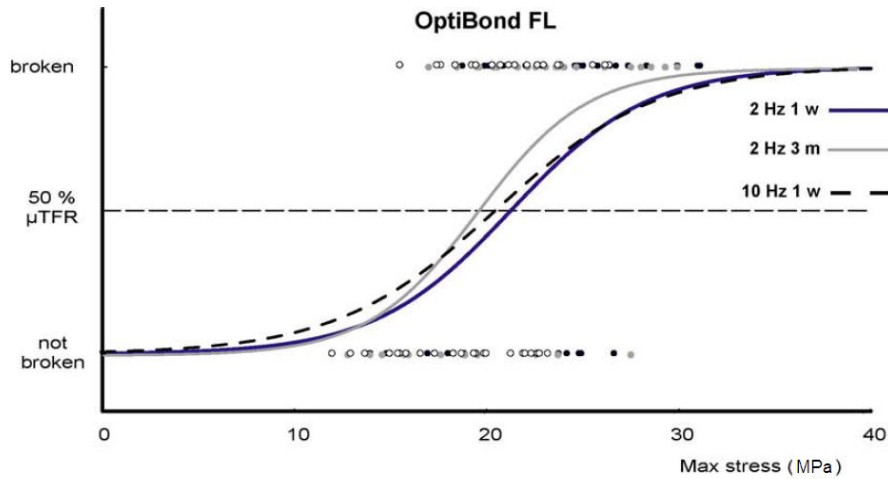


Fig 5. Chart for cyclical load of a sample glued with OptiBond FL glue, including 3 storage periods [24]

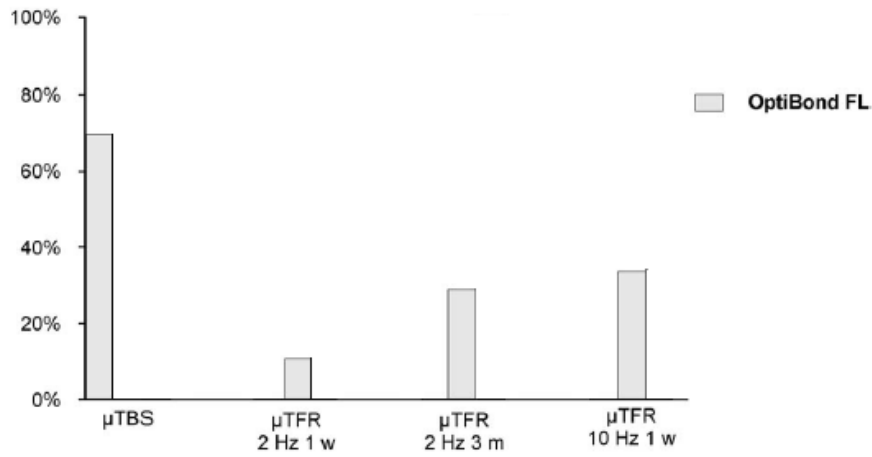


Fig 6. Percentage of surface destruction for particular test method[24]

The highest percentage of fractures occurs in static load of the tested element. The results reach even up to 70%. Cyclical fatigue tests for 2Hz and 10Hz display relatively little damage of the analysed teeth enamel surface. The range falls between 17% to 40%.

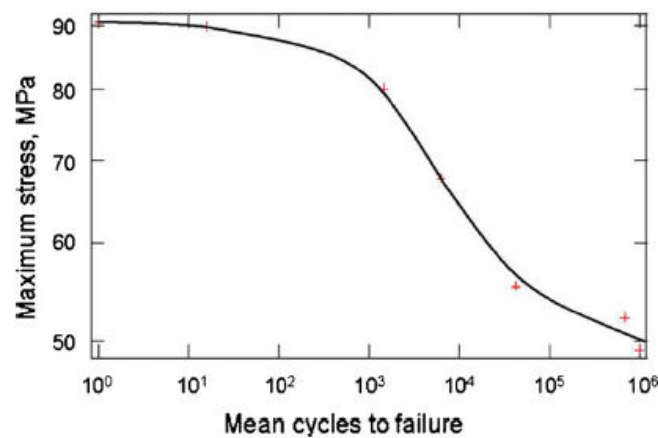
3.2. Method 2 [27]

Results for samples which were bent statically are very similar and they amount to: 164.4 ±9.1 and 164±2.4MPa (table 1).

Tabela 2. Dental samples endurance to four-point bending [27]

| Type of sample | Mean fracture stress in MPa | Statistical grouping |
|-----------------|-----------------------------|----------------------|
| Solid dentin | 164,4 ± 9,1 | A |
| Solid composite | 164,6 ± 2,4 | A |
| Bonded beam | 90,6 ± 2,5 | B |

Among samples that underwent cyclical load, all samples endured the maximum number of cycles which equalled 10^6 at the maximum pressure – 49.2MPa. Picture 7 indicates that along the increase of cycle number, the maximum pressure for the sample decreases.



Pic. 7. Scheme representing an average number of cycles for the maximum pressure [27]

4. Summary

The purpose of this paper was to present the endurance and fatigue tests methodology for human and bovine dental samples. The article describes two methods which help to determine properties of analysed glue. The first testing method for glued junctions in molar teeth is much more time consuming as it includes methods of static stretching of the analysed sample and cyclical – dynamic stretching at the frequency of 2Hz and 10Hz. The obtained results confirm there is a difference in the range of endurance depending on the method and period of sample storage in the solution. Table 2 lists results of analysis methodology for the two above described methods. In this article only one glue type is mentioned as the aim of the paper was to present the method for properties determination of glued tooth – composite junctions. Similarly to the above mentioned, method 1 will be used by the author for own analysis. A difference will be introduced by altering the mode of storage – right after extraction, dental samples will be frozen down to -32°C and later cleansed of any remaining soft tissue. Samples will be disinfected with gamma rays just as in method no 2. The ISOMET 4000 machine will be used for the analysis. It will be applied for trimming the samples into beams into the dimension: 1.5mm x 1.5mm x 15mm. Samples will undergo tests in a material testing machine of a load 500N.

Table 2. List of methods

| Methods | 1 | 2 | 3[3] |
|-----------------------------|-------------------------------------------------------------------|----------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------|
| Load | dynamic | dynamic and static | dynamic |
| Frequency | 2 Hz and 10 Hz | 10^6 cycles | 0,25Hz 4×10^4 cycles |
| Storage period | 0,5% chloroamine in 4°C | Sterilisation with γ rays Hanks salt solution (HBSS) in 25°C | 24h in distilled water at 37 °C |
| Range of testing (pressure) | 500 N; 0 – 40 MPa | 165 N; 0 – 90 MPa | 24 – 54 MPa |
| Problem type | Measurement of dynamic and static tensions for results comparison | Determination of junction properties for tooth- composite junction during static and dynamic tests | Shear fatigue limit testing coupled with shear bond strength measurements |

5. References

- [1] Attia A., Abdelaziz K.M., Freitag S., Kern M.: Fracture load of composite resin and feldspathic all-ceramic CAD/CAM crowns, *J Prosthet Dent*, 2006, Vol. 95, No. 2, ss. 117 – 123
- [2] Bajaj D., Nazari A., Eidelman N., Arola D.D.: A comparison of fatigue crack growth in human enamel and hydroxyapatite, *Biomaterials*, 2008, Vol. 29, ss. 4847 – 4854
- [3] Barkmeier WW, Erickson RL, Latta MA. : Fatigue limits of enamel bonds with moist and dry techniques, *Dental materials journal* Vol 25(12) ss. 1527 – 1531
- [4] Belli S, Ozçopur B, Yeşilyurt C, Bulut G, Ding X, Dorsman G. : The effect of loading on microTBS of four all-in-one adhesives on bonding to dentin, *J Biomed Mater Res B Appl Biomater*, Vol 91(2) ss. 948 – 956
- [5] Bijelic J, Garoushi S, Vallittu PK, Lassila LV. : Fracture load of tooth restored with fiber post and experimental short fiber composite, *The open dentistry journal*, Vol 5 ss. 58 – 65
- [6] Bolhuis P., de Gee A., Feilzer A.: Influence of fatigue loading on four post-and-core systems in maxillary premolars, *Quintessence Int.*, 2004, Vol. 35, No. 8, ss. 657 – 667
- [7] Braem M.: Microshear Fatigue Testing of Tooth/Adhesive Interfaces, *J Adhes Dent.*, 2007, Vol. 9, Supplement 2, ss. 249 – 253
- [8] De Munck J., Van Landuyt K., Peumans M., Poitevin A., Lambrechts P., Braem M. and Van Meerbeek B.: A Critical Review of the Durability of Adhesion to Tooth Tissue: Methods and Results, *Journal of Dental Research* 2005, Vol. 84 ss. 118 - 133
- [9] Drummond J.L.: Degradation, fatigue and failure of resin dental composite materials, *J Dent Res.*, 2008, Vol. 87, No. 8, ss. 710-719
- [10] El-Mowafy O., Rubo M.: Retention of a Posterior Resin-Bonded Fixed Partial Denture with a Modified Design: An In Vitro Study, *The International Journal of Prosthodontics*, 2000, Vol. 13, No. 9, ss. 425-431
- [11] Garcia Eugenio J., Gomes Osnara M.M., Gomes Joao C.: In vitro analysis of bond strength of self – etching adhesives applied on superficial and deep dentin, *Acta Odontol*, Vol. 22 ss. 57-62
- [12] Garoushi S.K., Lassila L.V.J., Vallittu P.K.: Fatigue Strength of Fragmented Incisal Edges Restored with a Fiber Reinforced Restorative Material, *The Journal of Contemporary Dental Practice*, 2007, Vol. 8, No. 2, ss. 1-10
- [13] Gateau P., Sabek M., Dailey B.: Fatigue testing and microscopic evaluation of post and core restorations under artificial crowns, *J Prosthet Dent.*, 1999, Vol. 82, No. 3, ss. 341-347
- [14] Goto Y., Nicholls J.I., Phillips K.M., Junge T.: Fatigue resistance of endodontically treated teeth restored with three dowel-and-core systems, *J Prosthet Dent.*, 2005, Vol. 93, No. 1, ss. 45-50
- [15] Herion T., Ferracane J.L., Covell D.A.Jr.: Three Cements Used for Orthodontic Banding of Porcelain Molars, *Angle Orthod.*, 2007, Vol. 77, No.1, ss. 94-99

- [16] Heydecke G., Butz F., Hussein A., Strub J.R.: Fracture strength after dynamic loading endodontically treated teeth restored with different post- and-core systems, *J Prosthet Dent*, 2002, Vol. 87, No. 4, ss. 438-445
- [17] Hsu Y.B., Nicholls J.I., Phillips K.M., Libman W.J.: Effect of Core Bonding on Fatigue Failure of Compromised Teeth, *The International Journal of Prosthodontics*, 2002, Vol. 15, No. 2, ss. 175-178
- [18] Huang H.H., Lin M.C., Lin C.C., Hsu C.C., Chen F.L., Lee S.Y., Hung C.C.: Effects of welding pulse energy and fluoride ion on the cracking susceptibility and fatigue behavior of Nd: YAG laser-welded cast titanium joints, *Dent Mater J.*, 2006, Vol. 25, No. 3, ss. 632-640
- [19] Inoue T, Nishimura F, Debari K, Kou K, Miyazaki T. : Fatigue and tensile properties of radicular dentin substrate, *Journal of Biomechanics* 2011, Vol 44(4) ss. 586 – 592
- [20] Li BH, Zhao X, Bao Y, Ai HJ. : Resistance to cyclic fatigue of pulpless teeth with flared root canals restored with three kinds of post-and-cores, *Shanghai Kou Qiang Yi Xue* Vol. 18 ss. 69 - 72
- [21] Loher H., Behr M., Hintereder M., Rosentritt M., Handel G.: The impact of cement mixing and storage errors on the risk of failure of glass-ceramic crowns, *Clin Oral Invest*, 2009, Vol. 13, No. 2, ss. 217-222
- [22] Minami H., Suzuki Sh., Minesaki Y., Kurashige H., Tanaka T.: In Vitro Evaluation of the Effect of Thermal and Mechanical Fatigues on the Bonding of an Autopolymerizing Soft Denture Liner to Denture Base Materials Using Different Primers, *Journal of Prosthodontics*, 2008, Vol. 17, ss. 392-400
- [23] Piątowska D., Jarosław Cynkier, Małgorzata Paul-Stalmaszczyk: *Stomatologia Zachowawcza, współczesne metody opracowania i wypełniania ubytków próchnicowych. Bestom DENTOnet.pl, Łódź 2009*
- [24] Poitevin A., Jan De Munck, Marcio Vivan Cardoso, Atsushi Mine, Marleen Peumans, Paul Lambrechts, Bart Van Meerbeek :Dynamic versus static bond-strength testing of adhesive interfaces, *Dental Matherials* 2010 Vol. 26, ss. 1068–1076
- [25] Shono Y., Ogawa T., Terashita M., Carvalho R.M., Pashley E.L. and Pashley D.H.: regional measurement of resin – dentin bonding as an array, *Journal of Dental Research*, Vol. 78, ss. 699 – 705
- [26] Singh V, Misra A, Marangos O, Park J, Ye Q, Kieweg SL, Spencer P. : Viscoelastic and fatigue properties of model methacrylate-based dentin adhesives, *Journal of Biomedical materials research* 2010, Vol 95(2), ss. 283 – 290
- [27] Staninec M., Kim P., Marshall G. W., Ritchie R.O., Marshall S. J.: Fatigue of dentin–composite interfaces with four-point bend, *Dental Materials* 2008, Vol. 24, ss. 799 - 803
- [28] Stellini E, Stomaci D, Zuccon A, Bressan E, Ferro R, Petrone N, Favero L, Mazzoleni S. : Tooth fragment reattachment through the use of a nanofilled composite resin, *European journal of paediatric dentistry*, Vol 11(2), ss. 77 – 81
- [29] Topoliński T.: *Problemy wytrzymałości i trwałości zmęczeniowej w materiałach i konstrukcjach inżynierii biomedycznej, WNITiE, Bydgoszcz 2009*

[30] Yamamoto M., Takahashi H.: Tensile Fatigue Strength of Light Cure Composite Resins for Posterior Teeth, Dental Materials Journal, 1995, Vol. 14, No. 2, ss.175-184

[31] Zahran M., El-Mowafy O., Tam L., Watson P.A., Finer Y.: Fracture Strength and Fatigue Resistance of All-Ceramic Molar Crowns Manufactured with CAD/ CAM Technology, Journal of Prosthodontics, 2008, Vol. 17, ss. 370-377