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On the Possibility of Describing the Microstructure of Human Dentin as Soft Matrix Filled by Solid Particles

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Abstract. Experimental verification of the applicability of self-similar hierarchical mechanics model for calculation of the mechanical properties of human dentin was carried out. A comparison of the deformation behavior of human dentin with the several polymer based dental composites under compression and bending was performed. It was shown that the deformation behavior of some highly filled composites was close to human dentin in the elastic regime whereas they cannot be deformed plastically under compression. Low filled composites possess higher plasticity, but a lower Young's modulus than dentin under compression. Mechanical properties of a highly filled composite (~60%, by volume) with smaller size of fillers are closer to the properties of human dentin under bending whereas other tested dental materials have a lower Young's modulus and flexural strength in comparison with dentin. Therefore, we concluded that it was impossible to achieve complete combination of the mechanical properties of human dentin such as high elasticity and plasticity at high strength by means of varying the amount and size of solid filler in polymer matrix. Self-similar hierarchical mechanics model can be used for describing of the deformation behavior of human dentin in elastic regime and one level is sufficient for these calculations.

1. Introduction

Many biological tissues have exceptional strength properties. They can have both high elasticity as in resins and high strength as with stone. This combination of the mechanical properties is realized by their complicated hierarchical microstructures. Highly mineralized bio minerals consist of inorganic and organic components. The quantity of hierarchical levels can achieve seven for human bone [1-3]. Understanding the relationship between microstructure and the mechanical properties of such materials allows developing new types of composites, which have high strength at low weight and can be used for manufacturing of tissue-equivalent materials.

A self-similar hierarchical mechanics model was developed for calculating the mechanical properties of solid natural tissues, where the particles of solid fillers are enveloped in soft matrix in staggered that forms solid particle to the next structural level. The total amount of hierarchical levels in the model defined by the number of transitions (Fig. 1) [4,5]. The solid particles are much harder than the soft matrix, and the tensile zone in the soft matrix near the ends of solid particles is assumed to carry no mechanical load. Anisotropy of the mechanical properties of material is due to variations in the ratio of solid particles [5]. The strength of the hierarchical material increases with increasing aspect ratio of particles and continues to be constant with each added hierarchical level. Toughness first increases and then decreases with increasing number of hierarchical levels. There exists an optimal number of levels

(N = 4) when the toughness is maximal. It is assumed that each additional level of hierarchy increases the overall protein content. Hence, the elastic modulus decreases when the quantity of levels increases. Actually, the microstructure of biological tissues is much more complicated [6]. For example, there are three structural levels in human dentin. The first level is collagen fibers and calcium hydroxyapatite; the second is network-like structure of collagen fibers in intertubular dentin; the third is the dentin tubules surrounded by a highly mineralized cuff (peritubular dentin) [7-9]. There are 60% of inorganic components, 30% organic components and 10% water by volume in human dentin [10,11].

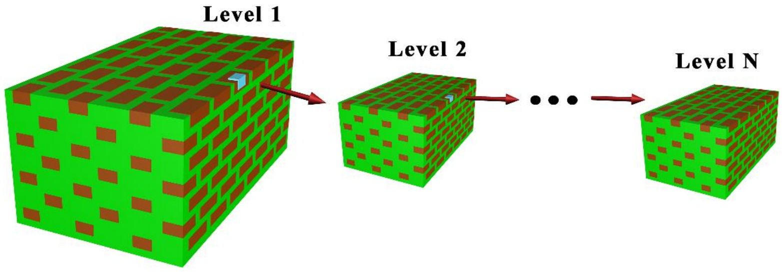

Figure 1. Hierarchical mechanics model of self-similar system.

Microstructure of polymer based dental restorative materials, where particles of solid filler are engaged in a polymer matrix is similar to self-similar hierarchical model. Hierarchy in these composites is achieved by means of the particles of filler possessing different sizes. Many types of commercial dental composite materials are distinguished by their consistency. In most cases, polymer matrix of such composites consist of bis-GMA, which is mixed with other dimethacrylates, such as TEGDMA, UDMA or other monomers. The composites could be divided on two groups, the highly filled materials and low filled ones in depending on the amount of fillers more than 50% and less than 50%, respectively. Additionally, restorative materials are classified according to the size of particles of filler: macrofill - a particle size greater than 10 microns, microfill- a particle size less than 100 nm and hybrid materials contain both macro and micro particles [12-15]. Also, manufacturers used extra classifications by dimensions of particles in dental composites, such as nanofill, midfill, nanohybrid and microhybrid [13]. Besides, the bonding within single polymer chain between molecules is much stronger than with the molecules in neighboring chains in the structure of polymers [16]. The similar situation takes place in the collagen fibers of intertubular dentin. In addition, manufacturers of materials try bringing their properties closer to the ones of dental hard tissues.

Sometimes calculation of the mechanical properties using the hierarchical self-similar models contradicts the data of mechanical tests of the hard natural materials such as bone, enamel and pearl [3]. This may be explained by either the mismatch of proposed model to deformation mechanism acted in biological tissue or the existing contradictions in understanding true mechanical properties of these materials, due to difficulties in conducting mechanical tests. Comparison of the deformation behavior of human dentin under compression and bending with the polymer based dental restorative materials allows estimating the possibility of application of self-similar hierarchical model to analysis of the mechanical properties of hard tissues. This approach will allow conducting comprehensive analysis of the model. Therefore, the aim of this work is experimental determining the possibility of using a self-similar hierarchical model to application to the mechanical properties of human dentin. Comparison of deformation behavior of human dentin under compression and bending with commercial dental polymer based composites with different amount and size of filler will be carried out for this purpose. The findings could be used for developing of the physical model of the deformation mechanisms of human dentin. In addition, the results of this study can be useful for elaboration and choice of restorative materials for dentistry.

2. Materials and Methods

Five commercial dental polymer based composites with different quantity and size of particles of filler were chosen as model materials (Table 1). Materials were selected based on their composition and amount of filler to cover the whole range of the filling of their polymer matrix used in dentistry. The amount of filler in the materials varied from 46% to 63% by volume. Model materials were cast in special forms for preparation samples for mechanical testing. Further, they were condensed to avoid the possibility of the pore nucleation in the samples [17]. Then they were cured by means of the halogen curing light (3M ESPE Elipar FreeLight 2, 1000 mW/cm^2) during 30 seconds. Beam of the light cover the whole sample. For comparison, twenty intact human molars and premolars were used in this work.

Table 1. The model materials (manufacturers' information)

Model material	Manufacturer	filler loading, % by volume
Filtek Ultimate	3M ESPE	63.3
Filtek P60	3M ESPE	61
SonicFill	Kerr	~55
Filtek Ultimate Flowable	3M ESPE	46
SDR	DENTSPLY	~50

The teeth were extracted from mature subjects (25-40 years old) according to the medical diagnoses and the Ethics Protocol of the Urals State Medical University at Yekaterinburg, Russia. They did not contain any visible defects and were stored in 2% NaCl water solution before testing. The teeth were cut off by means of the diamond saw under water irrigation according to the scheme described previously [18]. The surfaces of the samples of model materials and human dentin were abraded by the abrasive paper for removing the damaged layer on the back surfaces of the samples. Finally, the samples have the form of parallelepipeds with dimensions 2x2x1.3 mm^3 for compression and 12x2x0.8 mm^3 for bending. The deviation in the size does not exceed 5 μm. Each group consisted of 10 samples.
A Shimadzu AGX-50kN testing machine was used for uniaxial compression and three point bending tests with traverse rate of 0.1 mm/min. The distance between the support prisms was 8 mm. Value of plastic deformation under compression was calculated from the distinction between the thickness of the sample prior and after testing whereas elastic deformation was determined by means of subtracting plastic deformation from total deformation. Total deformation was obtained using of the testing machine. Under bending and under compression when the samples are separated after test, elastic and plastic deformations are determined as longitude of the strain axis under linear region of curve and longitude of the strain axis under non-linear region of curve, respectively. Young's modulus was calculated from the slope of deformation curves on the linear region. Compression strength was accepted as the maximal stress under testing. Standard deviation was determined for each experimental parameter by means of Trapezium – X ™ standard software for the Shimadzu facilities. Microstructure of the model materials were studied on the fracture surfaces by means of JSM-6390LV (20kV) scanning electron microscope (SEM).

3. Results

Drop of stress occurred on the deformation curves that corresponds to separation of the sample for the highly filled composites and appearance of cracks in the sample for the low filled composites and dentin samples as soon as compression testing was stopped. The model materials can be divided the two groups by deformation behavior in depending of the amount of filler. Deformation curves of the highly filled composites are linear whereas there are two linear regions with the different slope on deformation curves of the low filled composites (fig. 2). Deformation curve of the dentin sample can be divided the two parts (linear and non-linear). The slope of dentin curve on the first region is close to the slope of curve of the highly filled composites. The mechanical parameters of tested materials under compression are collected in Table 2. Compression strengths of Filtek Ultimate and Filtek P60 are higher in comparison with dentin, whereas the compression strength of dentin is similar to Filtek Ultimate Flowable and it higher than SonicFill and SDR. Young's modulus of the dentin is similar to Filtek Ultimate and Filtek P60 and it higher than SonicFill, Filtek Ultimate Flowable and SDR. Elastic deformation of all model materials is higher in comparison with dentin. Plastic deformation of SDR is higher that of dentin, while plastic deformations of SonicFill and Filtek Ultimate Flowable are similar to dentin. Filtek Ultimate and Filtek P60 are elastically deformed only.

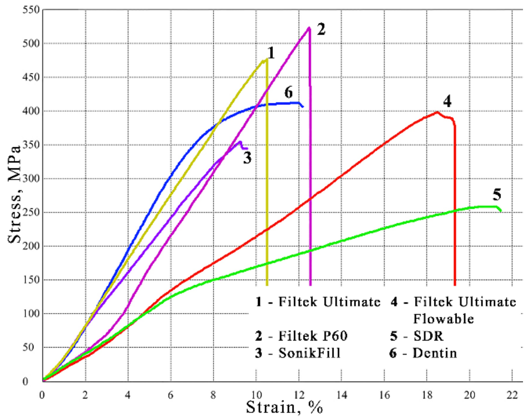

Figure 2. Stress-strain curves under compression.

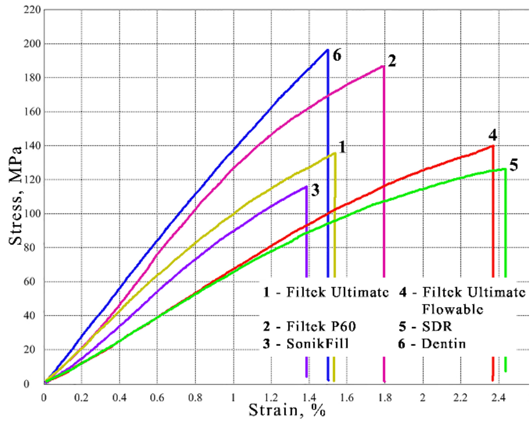

Figure 3. Stress-strain curves under bending.

Table 2. The mechanical properties with standard deviations of the model materials and human dentin under compression.

Model material	Young's modulus, *GPa*	Compressive strength, *MPa*	Elastic deformation, *%*	Plastic deformation, *%*	Total deformation, *%*
Filtek Ultimate	5.07±0.19	477±16	10.9±0.4	-	10.9±0.4
Filtek P60	4.98±0.04	525±24	12.7±0.4	-	12.7±0.4
SonicFill	4.02±0.17	351±29	7.5±1.1	2.3±1.2	9.8±0.9
Filtek Ultimate Flowable	2.31±0.11	403±18	12.9±0.9	5.5±0.8	18.4±0.7
SDR	2.06±0.17	261±17	10.1±1.3	11.6±1.8	21.7±0.5
Dentin	5.46±0.35	406±25	7.0±0.7	4.7±1.5	11.7±2.0

Table 3. The mechanical properties with standard deviations of the model materials and human dentin under bending.

Model material	Young's modulus, *GPa*	Flexural strength, *MPa*	Elastic deformation, *%*	Plastic deformation, *%*	Total deformation, *%*
Filtek Ultimate	10.39±1.10	137±13	0.8±0.1	0.7±0.2	1.5±0.2
Filtek P60	13.11±0.50	184±8	0.9±0.1	0.9±0.3	1.8±0.2
SonicFill	9.84±0.75	116±3	1.0±0.1	0.4±0.0	1.4±0.2
Filtek Ultimate Flowable	7.12±0.20	134±12	1.3±0.1	1.1±0.2	2.4±0.4
SDR	6.81±0.49	124±13	1.1±0.1	1.3±0.3	2.4±0.4
Dentin	14.46±2.49	195±38	1.0±0.1	0.5±0.1	1.5±0.1

The samples separate the two equal parts along the line of loading under bending. The trend of curves is similar for all materials (fig. 3). There are two regions (linear and non-linear) on the deformations curves. The mechanical properties of the tested materials under bending are shown in Table 3. Mechanical properties of Filtek P60 are closer to the properties of dentin among the model materials. Compression strength and the Young's modulus of other model materials are low in comparison with dentin. Elastic deformations are comparable for all materials, whereas plastic deformation of the dentin is comparable to highly filled composites and lower than the low filled composites (Table 3).
SEM observation of the fracture surfaces of the model materials has shown that Filtek Ultimate and Filtek P60 are microfill whereas SonicFill, Filtek Ultimate Flowable and SDR are hybrid materials and consist of two types of filler (macro and micro) differing from each other in size. (fig. 4-8). The size of filler particles in Filtek P60 is less than in Filtek Ultimate, ~1μm and ~2μm, respectively (fig. 4,5). The

sizes of both type of fillers (~3μm and ~10μm) in SonicFill are larger than size of particles in Filtek Ultimate and Filtek P60 (fig. 6). Amount of macro-particles in SDR much more than in Filtek Ultimate Flowable, but the size of macro-particles in Filtek Ultimate Flowable (from ~10μm to~100μm) larger than in the material from SDR (~10μm) (fig. 7,8). At that, the size of micro- particles in Filtek Ultimate Flowable (~2μm) is larger than in material from SDR (less than1μm).Moreover, no pore or flaws are observed on the fracture surfaces of the model materials. Deformed areas of intertubular dentin are observed on the fracture surface of the dentin sample after compression (fig. 9a). In addition, there are the trajectory of cracks between dentin tubulars (fig. 9b). On the contrary, the fracture surface of the dentin sample is macroscopically smooth and it did not contain cracks after the bending test (fig. 10).

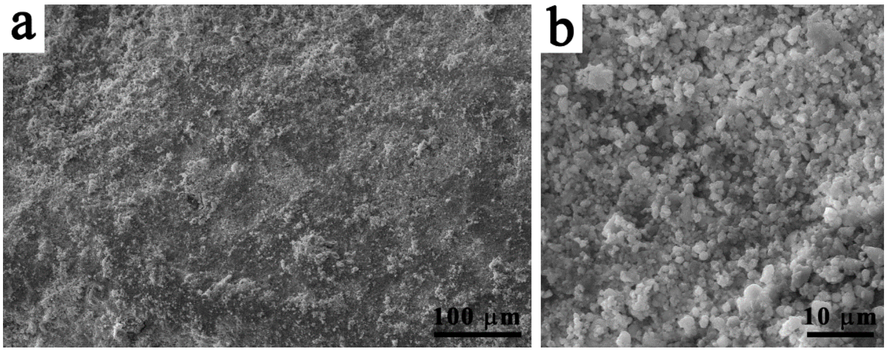

Figure 4. SEM images of microstructure of Filtek Ultimate: a – magnification x200; b – magnification x1500.

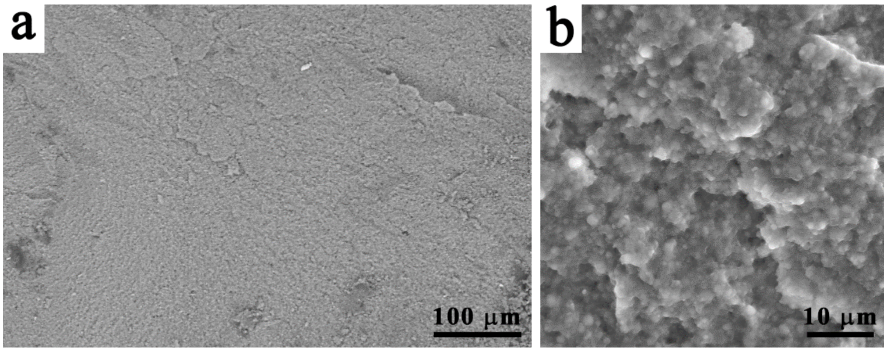

Figure 5. SEM images of microstructure of Filtek P60: a – magnification x200; b – magnification x1500.

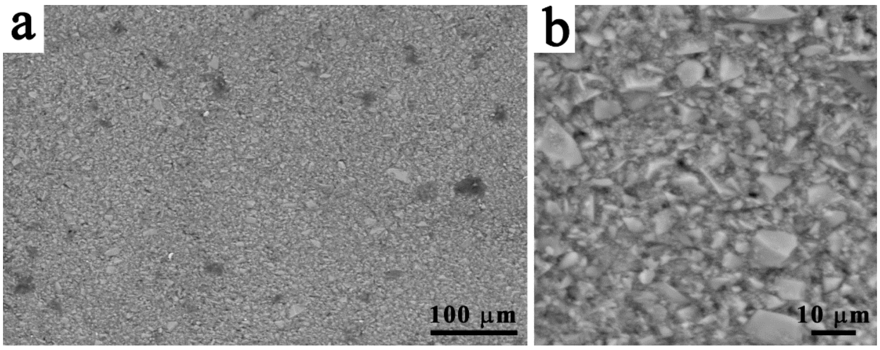

Figure 6. SEM images of microstructure of SonicFill: a – magnification x200; b – magnification x1000.

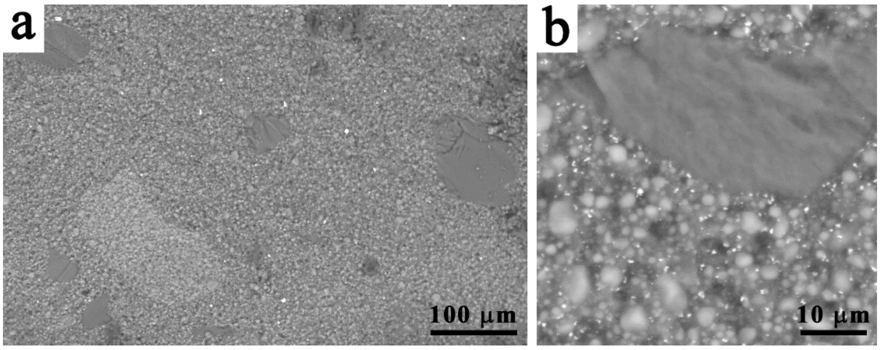

Figure 7. SEM images of microstructure of Filtek Ultimate Flowable: a – magnification x200; b – magnification x1500.

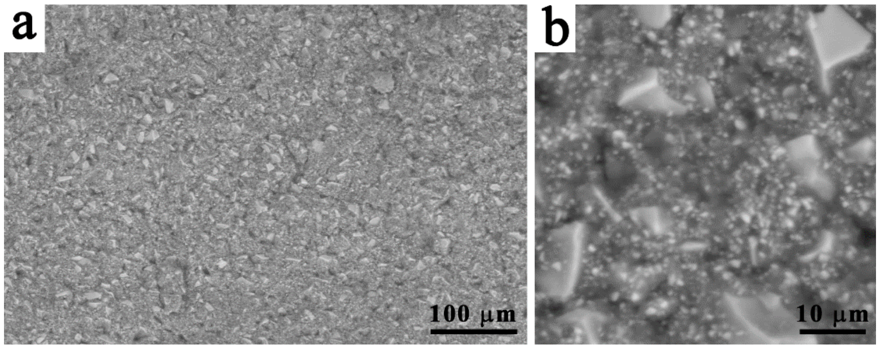

Figure 8. SEM images of microstructure of SDR: a – magnification x200; b – magnification x1500.

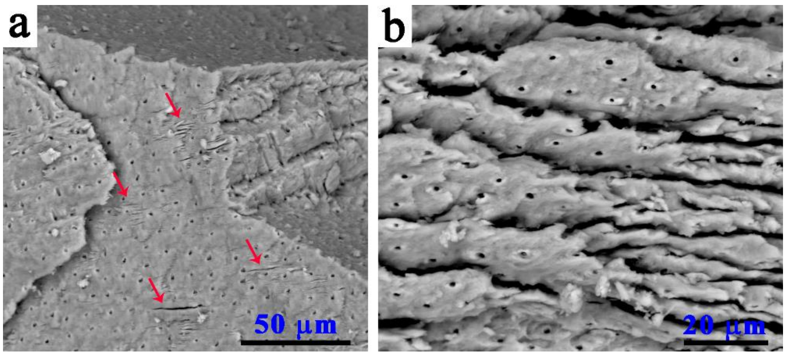

Figure 9. SEM images of fracture surfaces of dentin under compression: a – the area of compressed intertubular dentin, indicated by the red arrows; b – the cracks propagated between dentin tubular.

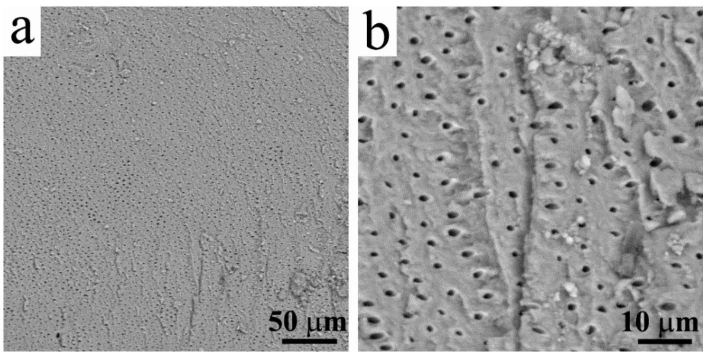

Figure 10. SEM images of fracture surfaces of dentin under bending: a – magnification x200; b – magnification x1000.

4. Discussion

Mechanical testing has shown that the deformation behavior of highly filled composite with smaller size of fillers (Filtek P60) in the elastic regime is similar to deformation behavior of human dentin under compression and bending. Besides, the deformation behavior of t Filtek P60 coincides with human dentin under bending in both elastic and plastic regions of the deformation curves (fig. 3). However, the highly filled composites with the higher volume of filler loading (Filtek Ultimate and Filtek P60) are not capable to plasticity under compression. The samples of SonicFill with intermediate volume of filler loading possess some plasticity under compression, but its value is low and its compression strength and the Young's modulus are lower in comparison with dentin. Low filled composites are capable to considerable plastic deformation, but they have lowest Young's modulus. It should be noted that the value of Young's modulus is ruled by the volume of filler loading, whereas the strength of material depends on amount and size of filler. The volume of filler loading in Filtek Ultimate and Filtek P60 is comparable (Table 1), but strength of Filtek P60 is higher due to smaller size of filler. This feature is also take place in self-similar hierarchical mechanics model, where the elastic modulus decreases with increasing quantity of protein or levels [3]. Increasing of the portion of macro-filler in SDR in comparison with Filtek Ultimate Flowable leads to a decrease of the strength, too. Elastic deformation does not depend on quantity and size of filler in diapason of filing from 46% to 63%, by volume and is ruled by polymer matrix. The plastic deformation does not depend on quantity and size of filler under bending except SonicFill. It is caused by the fact that the tensile stresses are maximal and interaction of the solid particles with each other are minimal. Under compression, when the portion of compression stresses is higher and interaction between the solid particles is more considerable, the portion of macro-fillers rules plasticity. Increasing the portion of macro-filler increases the plasticity in so much as there are areas with a lower filler content, where the plastic deformation is realized.

Hence, volume of filler loading ~60% of polymer matrix is optimal for material, where the Young's modulus is similar to human dentin. However, it is impossible to achieve the needed plasticity under compression (~5%) by varying of particle size of the filler at this volume of filler loading. Therefore, it is impossible to create polymer based dental composite with the mechanical properties coincided with properties of human dentin by means of varying of amount and size of fillers only. Indeed, it was shown that plasticity in human dentin is realized by means of two channels under compression (organic matrix and porosity), whereas under tension it is provided by organic matrix only [19,20]. Indeed, deformation and fracture are realized in intertubular dentin under compression whereas no defects is observed in microstructure of dentin under bending (fig. 9 and 10). Therefore, it is necessary to use another method of materials production of dental restorative materials. One of this method is the formation of pores inside material. Despite, restorative materials with volume of filler loading ~60% of polymer matrix can be effectively used in dentistry up to 350 MPa. Taking into account that stress in a mouth usually do not exceed 30MPa, can conclude that these materials may be using. Of course, this conclusion is based only the mechanical compatibility of human dentin with the restorative materials, whereas there are many other factors that determine the lifetime of the tooth after restoration.

Findings allow concluding that self-similar hierarchical mechanics model can be used for calculation of the mechanical properties of human dentin in elastic regime. At that, one level can be used for the calculations of the mechanical properties of dentin. The microstructure of human dentin can be considered as a soft matrix filled by solid particles having the similar size (~2μm for Filtek Ultimate and ~1μm for Filtek P60) because Filtek Ultimate and Filtek P60 are not hybrid (fig. 4, 5). Indeed, the intertubular dentin rules the deformation behavior of human dentin whereas dentin tubulars contribute on the fracture process only. Calcium hydroxyapatite have similar size (thickness of these crystallites approximately 5 nm and diameter ~50 nm). It can be considered as filler of organic matrix in dentin [21,22]. It should be noted that the deformation behavior of dentin in elastic regime is closer to the high filled model materials under compression in comparison with bending test. It caused with that deformation and fracture are realized in intertubular dentin under compression while it has been shown that the trajectory of crack in dentin including both intertubular and peritubular dentin under tensile stress [23,24]. The microstructure of intertubular dentin is similar to polymer based dental restorative materials and self-similar hierarchical mechanics model. Therefore, self-similar hierarchical mechanics model is more suitable for describing of compression than bending or tension of human dentin.

5. Conclusion

Deformation behavior of the highly filled composites (~60%, by volume) is closer to human dentin in comparison with the low filled composites. However, highly filled composites cannot be deformed plastically under compression. Low filled composites exhibit high elasticity and plasticity under compression and bending, but they have the Young's modulus lower than human dentin. Decreasing of the size particles of the filler increases the strength and the Young's modulus of polymer based dental composite. In spite of it is impossible to produce polymer based dental composite with the mechanical properties consisted with properties of human dentin by means of varying of amount and size of fillers only. Self-similar hierarchical mechanics model can be used for calculation of the mechanical properties of human dentin in elastic regime and one level is sufficient for these calculations.

Acknowledgements

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References

[1] Chen P Y, Lin A Y M, Stokes G A, Seki Y, Bodde S G, McKittrick J, Mayers M A 2008 JOM **60** 23
[2] Fratzl P, Weinkamer R 2007 Progr Mater Sci **52** 1263
[3] Bechtle S, Ang S F, Schneider G A 2010 Biomaterials **31** 6378
[4] Ji B, Gao H 2004 J. Mech. Phys. Solids. **52** 1963
[5] Zhang Z, Zhang Y-W, Gao H 2010 Proc. R. Soc. B; **278**, 519
[6] Meyers M A, Chen P Y, Lin A Y M, Seki Y 2008 Progr. Mater. Sci. **53** 1
[7] Pashley D H 1989 Scanning microscopy **3** 161
[8] Buehler M J 2006 PNAS **103**(33) 12285
[9] Habelitz S, Balooch M, Marshall S J, Balooch G, Marshall G W 2002 J. Struct. Biol. **138** 227
[10] Waters N E 1980 Symp. Soc. Exp. Biol. **34** 99
[11] Kinney J H, Marshall S J, Marshall G W 2003 Crit. Rev. Oral. Biol. Med. **14**(1) 13
[12] Leinfelder K F, Bayne S C, Swift E J 1999 J. Esthet. Dent. **11** 234
[13] Ferracane J L 2011 Dental Materials **27** 29
[14] Chen M-H 2010 J. Dent. Res. **89** 549
[15] Cramer N B, Stansbury J W, Bowman C N. 2011 J. Dent. Res . **90**(4) 402
[16] Riande E, Diaz-Calleja R, Prolongo M G, Masegosa R M, Saldm C. 2000 Marcel Dekker, Inc., New York
[17] Heymann H, Swift E, Ritter A. 2013 Elsevier, Mosby
[18] Zaytsev D, Ivashov A S, Mandra J V, Panfilov P 2014 Mat. Sc. Eng. C **41** 83
[19] Zaytsev D 2015 Mater. Sci. Eng. C **49** 101
[20] Zaytsev D, Panfilov P 2014 Mater. Sci. Eng. C **42** 48
[21] Gawda H, Sekowski L, Trebacz H 2004 Acta. Bioeng. Biomech. 6(1) 41
[22] Kinney J H, Pople J A, Marshall G W, Marshall S J 2001 Calcif. Tissue. Int. **69**(1) 31
[23] Nalla R K, Kinney J H, Rotchie R O 2003 Biomaterials **24** 3955
[24] Yahyazadehfar M, Ivanchik J, Majd H, An B, Zhang D, Arola D 2014 Appl. Mech. Rev. **66** 1