

# Defectoscopy of Denture Polymers

MEDA LAVINIA NEGRUTIU<sup>1</sup>, COSMIN SINESCU<sup>1\*</sup>, FLORIN IONEL TOPALA<sup>1\*</sup>, LUCIANA GOGUTA<sup>1</sup>, CORINA MARCAUTEANU<sup>1</sup>, ALEXANDRU OGODESCU<sup>1</sup>, MIHAI ROMINU<sup>1</sup>, ADRIAN GH. PODOLEANU<sup>2</sup>

<sup>1</sup> University of Medicine and Pharmacy "Victor Babeş" Timișoara, Faculty of Dentistry, 9 Revolutiei 1989 Blv., 300070, Timisoara, Romania

<sup>2</sup> University of Kent, Applied Optics Group, School of Physical Sciences, Canterbury, Kent, UK

*When making a denture, a removable partial prosthesis or a removable orthodontic appliance, long term preservation of structure and function require that potential irritating effects are kept at minimum. Polymer manufacturing technology with optimal properties and the material type are essential for a successful treatment. Increasing the biomechanical comportment of polymeric materials implies fiber reinforcing. The different fibers reinforcing products made very difficult the evaluation of their performances and biomechanical properties analysis. The aim of this study was to assess the quality of various polymeric materials used in dental technology and to validate the en face OCT defectoscopic evaluation of polymeric dental prostheses by using scanning electron microscopy (SEM). SEM investigations evidenced the nonlinear aspect of the interface between the polymeric material and the fiber reinforcement and materials defects in some samples. The advantages of the OCT method consist in non-invasiveness and high resolution. In addition, en face OCT investigations permit visualization of the more complex stratified structure at the interface between the polymeric material and the fiber reinforcement.*

*Keywords: polymeric materials, defectoscopy, en face optical coherence tomography, scanning electron microscopy*

When making a denture, a removable partial prosthesis or a removable orthodontic appliance, long term preservation of structure and function require that potential irritating effects are kept at minimum. The polymer manufacturing technologies with optimal properties, as well as the type of material used are very important for the treatment success [1, 2].

In order to avoid deficiencies of dental prostheses, several alternative systems and procedures were imagined, directly related to the material[3, 4] used and also to the manufacturing technology[5]. So, to enhance the mechanical strength, complete denture bases are reinforced with metallic or unmetallic nets or fibers[6]. These different reinforcing products made very difficult the evaluation of their performances and biomechanical properties analysis[7]. The reinforcing effect increased with the increase of the number of glass fibers in the case of thick specimens [8].

Fiber reinforcements placed on the tensile side resulted in considerably higher flexural strength and flexural modulus values compared with same quantity of fibers placed on the compression side[9].

In case of metallic reinforcement, the polymerization shrinkage level has a significant influence on the residual stress at the resin-metal interface. Enhancement of the bond strength on the interface can reduce the failure probability at a resin-metal joint[10].

The main problems are the material defects at the interface between the reinforcing materials (metal or fibers) and the polymer, that could decrease the strength of the prostheses. Several methods are utilized in the practice of dental prostheses investigation [11-14] The major disadvantage of the most of them consists in the stiffness of general evaluation of big, complex and anfractuous defects. The invasive characteristic of some of this methods implies the desert of the investigated prosthesis and lead to sample destruction[15].

OCT is an emerging technology, representing a paradigm shift over conventional light microscopy. It is a tomographic imaging technology capable of producing high-resolution cross-sectional images of the internal architecture of materials in a non-invasive manner. OCT images provide microstructural details that cannot be obtained with any other imaging modalities[16]. Advances in OCT technology, on the optimal use of galvanometer scanners for lateral scanning in OCT [17, 18] have made it possible to apply OCT in a wide variety of applications, but medical applications are still dominating. Specific advantages of OCT are its high depth and transversal resolution, the fact, that its depth resolution is decoupled from transverse resolution, high probing depth in scattering media, contact-free and non-invasive operation, and the possibility to create various function dependent image contrasting methods.

In dentistry, OCT was successfully used for acquiring images of incipient and advanced carious lesions[19-21], for the evaluation of the microleakage of dental restorations[22, 23] and endodontic fillings [24-28], the dental implant status[29], the integrity of complete dentures [30-33] and various dental prosthesis, their quality and their marginal fitting[34, 35], for early characterization of occlusal overloaded teeth and dental abfractions[36-38] or for investigation of the temporo-mandibular joint disc[39].

The aim of this study was to assess the quality of various polymeric materials used in dental technology and to validate the *en face* OCT defectoscopic evaluation of polymeric dental prostheses by using scanning electron microscopy (SEM).

## Experimental part

### Methodology

Several complete dentures and removable orthodontic appliances were made of various materials using the

\* email: minosinescu@yahoo.com; Tel.: 0040 722280132

florin.topala@gmail.com,

conventional pressure-pack method or injecting procedures, with reinforced bases with polymer preimpregnated glass fiber, polyethylene fibers, polyethylene and glass fiber net and golden metal net. Prior to the investigations, the samples were stored for a week in tap water at 37° C.

First, time domain optical coherence tomography working in *en face* mode (TDefOCT) was employed to evaluate the samples. Due to the capability of the *en-face* OCT to generate views in orthogonal planes (B- and C-scans) in real time, such an imaging method seems better suited to deliver tomography information [40-44].

The system operated at 1300 nm, with a working distance of 2 to 3 cm and depth resolution of 18 to 20 μm (in air), supplementary equipped with a confocal channel operating at 970 nm. Confocal microscopy is here used to guide the OCT investigation.

In order to validate the *ef*OCT defectoscopic evaluation, the samples were investigated by SEM, using the Inspect S device (FEI WORLDWIDE CORPORATE HEADQUARTERS North America NanoPort, Hillsboro, Oregon – USA). The samples surfaces were scanned with a high-energy beam of electrons, which interact with the atoms that make up the samples producing signals that contain information about the sample's surface.

High vacuum mode (10<sup>-5</sup>mbar) is used for imagistic and micro analysis of conventional prepared probes. Low vacuum mode (<270 Pa) is used for imagistic and micro analysis of not prepared probes. The system owns a wolfram filament in the electronic cone and works with 200V at 30kV. The current fascicle is >2 μA.

The resolution is 3.0 nm on standard specimen with gold particles separated by carbon particles, in both operational modes high and low-vacuum. The focusing domain is 3-99mm and the magnitude is from 6x to >1,000,000x on standard LCD display. The view field is the same in high and low-vacuum (18mm at the largest working distance).

Micro-computed tomography was also employed to evaluate the samples. The device (Infinix, Model RTP12303J-G9E, Toshiba Medical Systems Corp., Tustin, CA) was working at 35 μm pixel, resolution >10 lp/mm, with a field of view (FOV) of 3.6 cm. The x-ray exposure parameters were: 40 kVp, 1 mA and 300 ms exposure per frame.

The samples were placed onto the rotary stage at a magnification between 2 and 1.1, depending on the sample size and scanned using one degree step increments. After

projection acquisition they were reconstructed using a (512)<sup>3</sup> volume with a 45 μm<sup>3</sup> per voxel. The scanning was performed from occlusal towards apical zone of the tooth.

## Results and discussions

In all of the investigated samples, especially in the samples made using the conventional pressure-pack procedure were found defects which are totally included in the prostheses material and may cause their fracture. The polymeric material is very porous. The classical method involves liquid powder mixing resin and conventional pressure-pack procedure. During this procedure, multiple air inclusions can be captured inside the resin. The areas depicted present several small canals in the base that can be colonized in time with bacteria (fig. 1).

For another sample, armed with metallic net, the materials defects were identified at aproximative 627 micron (in air) in depth (fig. 2). The defects were considerable bigger compared with those in samples armed with fibers. The volume of these defects is around 0.032 mm<sup>3</sup>. The defect resulted in the samples armed with fibers is around 0.00015 mm<sup>3</sup>. This mean that the defects in the samples armed with fibers are aprox. 0.4 % from the defects in the samples armed with metallic net. This is one of the many causes of fractures in the second type of dental prostheses.

For a sample, reinforced with fibers, a small material defect was spotted between the fibers and the polymer resin. The first fibers stock was identified at aprox. 145 microns inside (fig. 3). At this depth the material defect could easily lead to a fracture into the resin material. The material defect identified between the fibers has a medium size. The problems with the materials defects are the possibility of interconnection during the loading and the possibility of fracture induction that affect all the defects situated in the same area of the sample.

In a sample reinforced with metallic net and made by the conventional pressure-pack technique, using a PMMA based material, we could identify defects at the interface metallic net – polymeric material at 525-527 microns (in air) in depth (fig. 4). The slices are separated by 0.021 mm.

SEM investigations evidenced the nonlinear aspect of the interface between the polymeric material and the fiber reinforcement and materials defects in some samples (fig. 5).

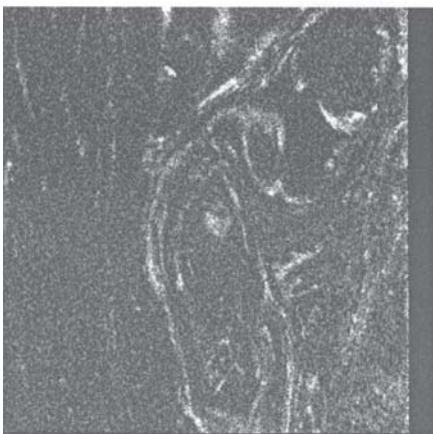


Fig. 1. Detection of areas with small canals in a removable orthodontic appliance's base produced using the classical method. *En-face* OCT proximal scanning, 5 mm lateral size, 0.28 mm depth inside

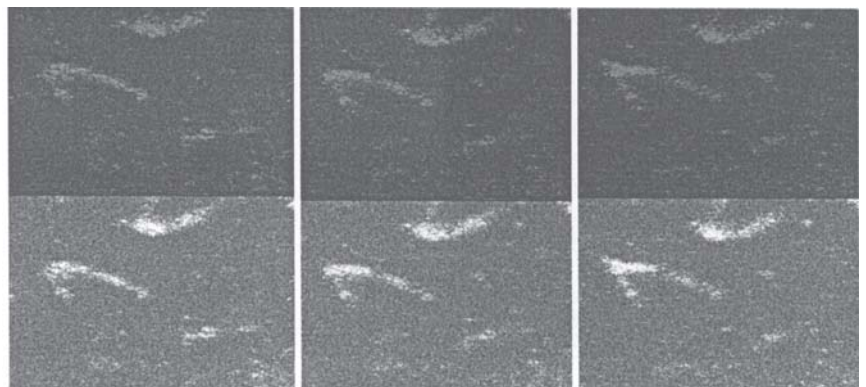


Fig. 2. Aspects from the *ef*OCT/CM investigation for sample 23 armed with metallic net – 0,627 mm in depth (in air). The volume of the defects is around 0.032 mm<sup>3</sup>.

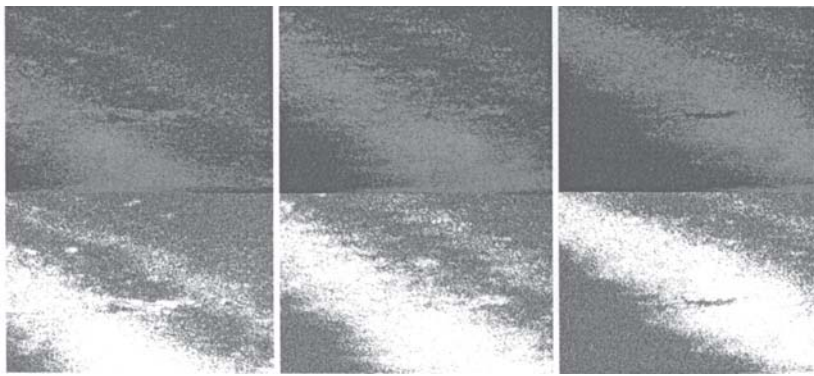


Fig. 3. Aspects from the *ef*OCT/CM investigation for a sample armed with fibers – 0.145 mm in depth (in air).



Fig. 4. Aspects from the *ef*OCT/CM investigation for a sample made by the conventional pressure-pack technique and reinforced with metallic net – 0.525-0.527 mm in depth (in air). The polymeric material is very porous.

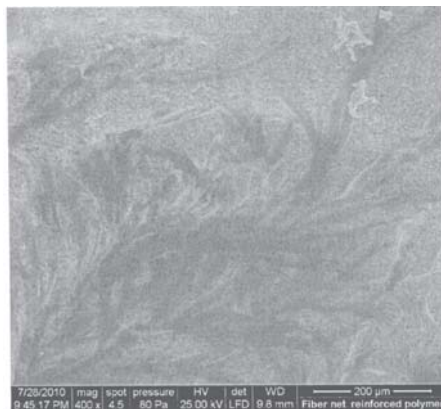


Fig. 5. SEM investigations evidenced the nonlinear aspect of the interface between the polymeric material and the fiber reinforcement.

## Conclusions

The defects identified above can lead to fractures of prostheses or/and to bacterial infiltration. These represent esthetic and functional failures of the treatment.

Dentures or removable orthodontic appliances with reinforced base present defects especially at the interface between the reinforcing material and the base polymer.

The samples made by injection methods are more compact. The number of pores in the injected polymer bases is reduced and therefore the risk of fractures is lower, compared to them, made by the conventional pressure-pack procedure.

The SEM evaluations allow observing the interested zones by surface mapping. The nonlinear aspect of the interface between the polymeric material and the fiber reinforcement and materials defects in some samples were evidenced.

In order to avoid future prosthetic failures, it is important to employ a noninvasive imaging method such as the OCT demonstrated here to assess the quality of dental prostheses before their insertion in the oral cavity.

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## References

1. PHOENIX RD, MANSUETO MA, ACKERMAN NA, JONES RE, "Evaluation of mechanical and thermal properties of commonly used denture base resins", *J Prosthodont.* Mar;13(1):17-27 (2004).
2. \*\*\* ISO 1567:1999(E), [Dentistry - Denture base polymers], Third Edition, 1999-02-15.
3. KUTSCH VK, WHITEHOUSE JW, SCHERMERHORN K, BOWERS B, "The evolution and advancement of dental thermoplastics", *DentalTown*, 4(2):52-56 (2003).
4. LOWE LG, "Flexible denture flanges for patients exhibiting undercut tuberosities and reduced width of the buccal vestibule: a clinical report", *J Prosthet Dent.*, 92(2):128-31 (2004).
5. PARVIZI A, Lindquist T, Schneider R, Williamson D, Boyer D, Dawson DV, "Comparison of the dimensional accuracy of injection-molded denture base materials to that of conventional pressure-pack acrylic resin", *J Prosthodont*, 13(2):83-9 (2004).
6. NARVA KK, VALLITTU PK, HELENIUS H, YLI-URPO A, "Clinical survey of acrylic resin removable denture repairs with glass-fiber reinforcement", *Int J Prosthodont*, 14: 219-224 (2001).
7. ÇÖKELILER D, ERKUT S, ZEMEK J, BIEDERMAN H, MUTLU M, "Modification of glass fibers to improve reinforcement: A plasma polymerization technique", *Dent Mater*, 23(3):335-42 (2007).
8. KANIE T, FUJII K., ARIKAWA H., INOUE K., "Flexural properties and impact strength of denture base polymer reinforced with woven glass fibers", *Dental Materials*, 16 (2): 150-158 (2000)
9. KATJA K. NARVA, LIPPO V. LASSILA AND PEKKA K. VALLITTU, "The static strength and modulus of fiber reinforced denture base polymer", *Dental Materials*, 21 (5): 421-428 (2005).
10. TOMOYUKI IKEDA, NORIYUKI WAKABAYASHI, MASAHIRO ONA, TAKASHI OHYAMA, "Effects of polymerization shrinkage on the interfacial stress at resin-metal joint in denture-base: A non-linear FE stress analysis", *Dental Materials*, 22 (5):, 413-419 (2006).
11. HSIN-YI LIN, BONNIE BOWERS, JOHN T. WOLAN, ZHUO CAI, JOEL D. BUMGARDNER, "Metallurgical, surface, and corrosion analysis of Ni-Cr dental casting alloys before and after porcelain firing", *Dental Materials*, 24 (3): 378-385 (2008).

12. OGODESCU, A ; SINESCU, C ; OGODESCU, E ; NEGRUTIU, M , "Engineering and Biomechanics in the Orthodontic Treatment of Periodontally Compromised Adult Patients", Proc.Wseas, Advances In Manufacturing Engineering, Quality And Production Systems, Vol I, Book Series: Mathematics and Computers in Science and Engineering, pg 194-196 (2009).
13. OGODESCU, AS; SINESCU, C; OGODESCU, EA; NEGRUTIU, M; ROMINU, R; BRATU, E, "Computer Science in the Orthodontic Treatment of Adult Patients", Proc.Wseas, Advances In Communications, Computers, Systems, Circuits And Devices, Book Series: European Conference of Systems, pg 15-18, (2010).
14. ROMINU M., LAKATOS S., FLORITA Z., NEGRUTIU M. -"Investigation of the mikroleakage at the interface between a Co-Cr based alloy and four polymeric veneering materials", Journal of Prosthetic Dentistry, 87, 6, pg.620-624, 2002.
15. LAKATOS S., ROMINU M., NEGRUTIU M., FLORITA Z. "The microleakage between alloy and polymeric materials in veneer crowns", Quintessence International, vol.34, nr.4, 2003, 295-300.
16. PODOLEANU A. GH., ROGERS J.A., JACKSON D.A. AND DUNNE S., "Three dimensional OCT images from retina and skin" Opt. Express, Vol. 7, No. 9, p. 292-298, (2000) <http://www.opticsexpress.org/framestocv7n9.htm>;
17. DUMA V. F., LEE K.-S., MEEMON P., ROLLAND J. P., Experimental investigations of the scanning functions of galvanometer-based scanners with applications in OCT, Applied Optics 50(29), 5735-5749 (2011)
18. DUMA V. F., Theoretical approach on optical choppers for top-hat light beam distributions, Journal of Optics A: Pure and Applied Optics, 10(6), 064008 (2008)
19. COLSTON, JR. B. W., SATHYAM U. S., DASILVA L. B., EVERETT M. J., "Dental OCT", Optic Express, 3(6):230-238 (1998).
20. AMAECHI B.T., PODOLEANU A., HIGHAM S.M., JACKSON D.A., "Correlation of quantitative light-induced fluorescence and optical coherence tomography applied for detection and quantification of early dental caries", Journal of Biomedical Optics 8 (4), 642-647 (2003)
21. SINESCU C., NEGRUTIU M. L., TODEA C., BALABUC C., FILIP L., ROMINU R., BRADU A., HUGHES M., PODOLEANU A., "Quality assessment of dental treatments using en-face optical coherence tomography", Journal of Biomedical Optics, Vol. 13(5), (2008).
22. ROMINU M., SINESCU C., NEGRUTIU M. L., ROMINU R. O., POP D. M., TOPALA F., STOIA A., PETRESCU E., BRADU A, DOBRE G., PODOLEANU A. G., "Adhesive improvement in optical coherence tomography combined with confocal microscopy for class V cavities investigations", Proc. SPIE 7626, 76260Y (2010)
23. NEGRUȚIU M. L., SINESCU C., TOPALA F., IONITA C., MARCAUTEANU C., PETRESCU E. L., PODOLEANU A. G., "Imagistic evaluation of direct dental restoration: en face OCT versus SEM and microCT", Proc. SPIE 8091, 80911T (2011)
24. FELDCHEIN F. I., GELIKONOV G. V., GELIKONOV V. M., IKSANOV R. R., KURANOV R. V., SERGEEV A. M., "In vivo OCT imaging of hard and soft tissue of the oral cavity", Optics Express 3(6), (1998).
25. NEGRUTIU M. L., SINESCU C., HUGHES M., BRADU A., TODEA C., BALABUC C. I., FILIP L. M., PODOLEANU A. GH., "Root canal filling evaluation using optical coherence tomography", Proc. SPIE 6991, 69911T (2008)
26. TODEA C., BALABUC C., SINESCU C., FILIP L., KEREZSI C., CALNICEANU M., NEGRUTIU M., BRADU A., HUGHES M., PODOLEANU A. GH., "En face optical coherence tomography investigation of apical microleakage after laser-assisted endodontic treatment", Lasers Med Sci, 25 (5):629-639 (2010).
27. NEGRUTIU M. L., SINESCU C., TOPALA F., NICA L., IONITA C., MARCAUTEANU C., GOGUTA L., BRADU A., DOBRE G., ROMINU M., PODOLEANU A. GH., "Root canal filling evaluation using optical coherence tomography", Proc. SPIE 7715, 77151T (2010)
28. NEGRUTIU M. L., NICA L., SINESCU C., TOPALA F., IONITA C., BRADU A., PETRESCU E. L., POP D. M., ROMINU M., PODOLEANU A. GH., "SEM and microCT validation for en face OCT imagistic evaluation of endodontically treated human teeth", Proc. SPIE 7961, 79614W (2011); doi:10.1117/12.878320
29. SINESCU C., HUGHES M., BRADU A., NEGRUTIU M., ANTONIE S., LAISSUE P., ROMINU M., PODOLEANU A. GH., "Implant bone interface investigated with a non-invasive method: optical coherence tomography", Biophotonics: Photonic Solutions for Better Health Care, Proc. SPIE, Vol. 6991, 69911L-1 – 69911L-9 (2008).
30. NEGRUTIU M. L., SINESCU C., TODEA C., PODOLEANU A. GH., "Complete denture analyzed by optical coherence tomography", Laser in Dentistry XIV, Proc. SPIE, Vol. 6843, 68430R-1 – 68430R-8 (2008).
31. NEGRUTIU M. L., SINESCU C., HUGHES M., BRADU A., GOGUTA L., ROMINU M., NEGRU R., PODOLEANU A. GH., "Fibres reinforced dentures investigated with en-face optical coherence tomography", Biophotonics: Photonic Solutions for Better Health Care, Proc. SPIE, Vol. 6991, 69911U-1 – 69911U-6 (2008).
32. NEGRUTIU M., SINESCU C., GOGUTA L., TOPALA F., ROMÎNU M., PODOLEANU A.G., "Different Types of Fiber Reinforced All Dentures Bases Evaluated by En-Face Optical Coherence Tomography and Numerical Simulation", International Journal of Medicine and Medical Sciences, 1(1): 50-55 (2010).
33. NEGRUȚIU M.L., SINESCU C., TOPALĂ F.I., IONIȚĂ C., GOGUȚĂ L., MĂRCĂUȚEANU C., ROMÎNU M., PODOLEANU A. GH., "Optical investigations of various polymeric materials used in dental technology", Optical Complex Systems: OCS11, Proc. of SPIE Vol. 8172, 817216-1 (2011)
34. SINESCU C., NEGRUTIU M., TODEA C., HUGHES M., TUDORACHE F., PODOLEANU A. GH., "Fixed partial denture investigated by optical coherence tomography", Coherence Domain Optical Methods and Optical Coherence Tomography in Biomedicine XII, Proc. SPIE, Vol. 6847, 684707-1 – 684707-10 (2008).
35. SINESCU C., NEGRUȚIU M., PETRESCU E., ROMINU M., MĂRCĂUȚEANU C., DEMJAN E., HUGHES M., BRADU A., DOBRE G., PODOLEANU A. GH., "Marginal adaptation of ceramic veneers investigated with en-face optical coherence tomography", Proc. SPIE, Vol. 7372, 73722C (2009).
36. MĂRCĂUȚEANU C., NEGRUTIU M., SINESCU C., DEMJAN E., HUGHES M., BRADU A., DOBRE G., PODOLEANU A. G., "Early detection of tooth wear by en-face optical coherence tomography", Proc. SPIE 7162, 716205 (2009)
37. MARCAUTEANU C., NEGRUTIU M., SINESCU C., STOICA E. T., IONITA C., FLORIN T., VASILE L., BRADU A., DOBRE G., PODOLEANU A. G., "Early characterization of occlusal overloaded cervical dental hard tissues by en face optical coherence tomography", Proc. SPIE 8091, 80911X (2011)
38. DEMJAN E., MĂRCĂUȚEANU C., BRATU D., SINESCU C., NEGRUȚIU M., IONITA C., TOPALĂ F., HUGHES M., BRADU A., DOBRE G., PODOLEANU A. GH., "Analysis of dental abfractions by optical coherence tomography", Proc. SPIE 7549, 754903 (2010)
39. MĂRCĂUȚEANU C., DEMJAN E., SINESCU C., NEGRUTIU M., MOTOC A., LIGHEZAN R., VASILE L., HUGHES M., BRADU A., DOBRE G., PODOLEANU A. G., "Preliminary optical coherence tomography investigation of the temporo-mandibular joint disc", Proc. SPIE 7554, 75542G (2010)
40. PODOLEANU A. GH., DOBRE G. M., WEBB D. J., JACKSON D. A., "Coherence imaging by use of a Newton rings sampling function", Optics Letters 21(21), 1789 (1996)
41. PODOLEANU A. GH., ROSEN R. B., "Combinations of techniques in imaging the retina with high resolution," Prog. Ret. Eye Res. 27(4), 464-499 (2008)
42. VAN DER JEUGHT S., BRADU A., PODOLEANU A. GH., "Real-time resampling in Fourier domain optical coherence tomography using a graphics processing unit", J. Biomed. Opt. 15, 030511 (Jun 03, 2010)
43. TRIFANOV I., NEAGU L., BRADU A., RIBEIRO A. L., PODOLEANU A. GH., "Characterization of a fibre optic swept laser source at 1 μm for optical coherence tomography imaging systems", Proc. SPIE 7889, 78892T (2011)
44. BRADU A., NEAGU L., ROGERS J., PODOLEANU A. GH., "Generating multiple depth en-face images in optical coherence tomography", Proc. SPIE 7889, 78891Y (2011)