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Investigation on picosecond laser ablation of dental material using FIB/SEM techniques

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Abstract

Results of laser ablation for human tooth are reported based on detected images from Focused Ion Beam/Scanning Electron Microscope (FIB/SEM) instruments. The ablation was carried out by exposing a 1 mm thickness of human tooth to 1J/cm^2 fluence of Nd: YAG laser operating at 532 nm with pulse duration of 40 ps. The capabilities of the FIB technique involving polishing, etching, drilling, milling in addition to imaging were investigated. The results showed that picosecond pulse duration was not enough to accomplish the ablation process unless the laser energy density is above the ablation threshold. The high potentiality of the technique used was observed for the analysis of FIB images.

Keywords: Laser ablation, ablation threshold, human tooth, FIB technique.

Introduction

Pulsed laser ablation (LA) could be defined as a layered removal of material by short high power laser pulses followed and controlled by an expansion of ablated species into surrounding area (Gorbunov & Konov, 1992). Although, laser ablation is a pure physical process, but several scientific groups directed their researches to the analysis and interpretation of the photo-ablation phenomenon in different fields (scientific, technological and medical) (Miller, 1994; Neey *et al.*,1996; Di Beraardo *et al.*, 2003; Rode *et al.*, 2003; Frankline *et al.*, 2005; Alti & Khare, 2006; Wieger *et al.*, 2006; Bashir, 2007). The production of nanotubes and nanoparticles, micro-machining, writing and prototyping were included in laser ablation for industrial applications (Becker *et al.*, 1998; Cai *et al.*, 1998; Scott *et al.*, 2001; Kokai *et al.*, 2005). Deeper insight into the ablation process and the development of diagnostics are justified taking into count the wide range of applications. The clear advantages for solid sample micro-analysis turned the attention of a large number of users to laser ablation (Bleioner & Bogaets, 2006). The new short-pulse high intensity regime which is available thanks to the introduction of chirped pulse amplification (CPA) technique. Recent laser technologies offer new regimes to the investigation. While, lasers can be used for producing ablation, ablation itself can be used as a potential diagnostics in realizing the laser beam characteristics (e.g. measuring laser intensity profile with shots at low-enough laser energy) (Bussoli *et al.*, 2007).

Dental material is known to be inhomogeneous and birefringent. The coronal portion of the tooth can be regarded as a biomechanical complex of two major tissues, enamel and dentin (Zip & Ten Busch, 1993). The amount of birefringence in enamel depends on the relative orientation between the enamel prisms and the incident probe light. While, light propagation in human dentin exhibits a strong directional dependence, it shows optical magnification property due to the divergence of the dentin tubules. Focusing Ion Beams (FIBs) techniques are now widely used in the analysis of a range of materials.

Recently, FIB that utilizes a liquid metal (typically gallium) source to produce a probe of small diameter, close to 10 nm (Vanes *et al.*, 2004) are being used in large number of applications such as metrology, inspection, cross sectioning, failure analysis, mask-less micromachining and preparation of thin foils for TEM. FIB preparation of human dentin was first reported (Hoshi *et al.*, 2001) where energy-filtered TEM analysis of FIB-prepared samples was used to study the organic-inorganic interface of apatite crystals, on the other hand, the FIB preparation of human tooth enamel was earlier (Giannuzzi *et al.*, 1999). The studies were concentrated on the microstructural characterization of the enamel and dentin samples, using FIB as a sample preparation technique in lieu of microtomy. The advantage of such a technique over microtomy is that focused ion beam preparation eliminates the need for time-consuming, multi-stage chemical fixation and also mechanical sectioning with a diamond knife, which potentially could introduce artificial damage. In addition, very precise site specificity when making specimens could be possible using dual beam FIB systems (Nalla *et al.*, 2005).

The interaction of laser beam with dental material (human tooth) was an interest for research groups especially in the last twenty years (Neev *et al.*, 1996; Serafetios *et al.*, 1996; Makropoulou *et al.*, 2005). However, the mechanism of interaction due to the use of ultra short pulses (ps, fs) is completely different (i.e. high intensities of lasers, the initial absorbed part induces multiphoton absorption MPA then multiphoton ionization (MPI). The description of the interaction of Nd: YAG laser in the picosecond time domain with human tooth was not sufficiently covered in the literature in comparison to the nanosecond domain.

In the present work, the focusing ion beam technique was utilized for the analysis of ablation in laser irradiated

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targets in the ps regime. Dental material (human tooth) was exposed to a second harmonic Nd: YAG laser (532nm), 40 ps, single shot and 1J/cm^2 energy density. The main purpose was to investigate the effect of picosecond laser on the surface morphology of human tooth during the ablation process using the potential capabilities of FIB technique for the analysis of the obtained results.

Materials and methods

A human tooth (18 year old) was prepared for the experiment. Non-caries premolars were transversally sectioned by a slow speed diamond disk (model 150, MTI corporation Richmond, CA). Water was used as a coolant during the slicing of the tooth to prevent the damage due to the friction. The thickness of the tooth was about 1 mm. It was stored in distilled water until the time of the experiment, so that the structure of the tooth was not disturbed.

An active passive mode-locked Nd: YAG laser system (Model SYL) P_2, Quanta system Sr1-Solbiate, Italy) was employed. To convert the laser emission to second harmonic (532 nm), a non-linear crystal (KD*P) was used. A pulse duration of 40 ps was obtained using saturable absorber and an acousto-optic modulator. The laser was operated with a pulse repetition rate of 1 Hz at 532 nm. The emitted mode was quasi-TEMoo. The human tooth was exposed to normal incidence laser beam. It was focused using 10 cm focal length of glass lens. The irradiation process was carried out in atmospheric pressure and standard relative humidity.

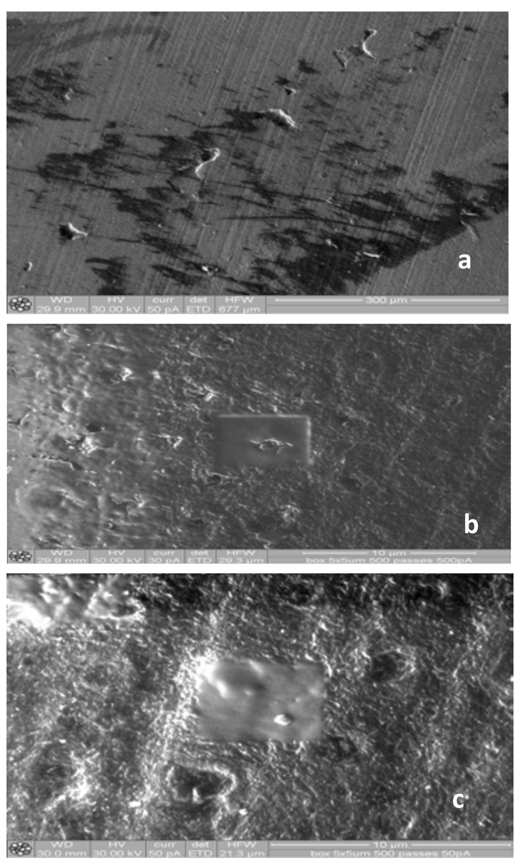
The FIB /SEM techniques

An electron column and an ion column embedded in the same specimen chamber of the scanning microscope, both beams are aiming at the same point on the specimen surface. In a FIB/SEM instrument, a finely focused ion beam mills away a precise amount of material from the sample and SEM images either the sidewall or the underlying layer exposed during the milling process. The sidewall was a precise cross-section of the material sample.

The FIB, generated by a Ga liquid metal ion source (LMIS) impacts the sample normal to the surface and could be focused to a spot as small as few nanometers. The interaction of the ion beam with the sample results in the ejection of atoms from the surface (sputtering), and the production of secondary electrons and ions, which are collected to obtain images. The plane of observation in situ could be changed depending on the ongoing collection of information. Imaging lower than 15 nm is possible. The obtained image shows both topographic information and material contrast, and could be used for navigating around the sample. This process could be used to form a cross-sectional view when the sample is tilted and imaged. The presence of an electron column that provides high resolution electron images is a further advantage of the FIB/SEM, that is, when coupled to the ion generated images, it can provide detailed information not only on surface, subsurface and internal morphology, but also on electrical and crystallographic properties.

A FIB/SEM Quanta 200 3D Dual Beam (FEI) was used in this work for the analysis of the irradiated sample. The aforementioned system incorporates a FIB and a SEM column tilted to each other at an angle of 52.The FIB current was adjusted from pA to nA at 30 kV. Furthermore, a VEGA TS 5136XM TESCAN system with Energy dispersive analysis (EDAX) was used. An Agar Auto Carbon Coater System was utilized to deposit about 1 nm thick of graphite on the tooth sample before the SEM imaging.

Fig. 1. Superficial FIB images for human tooth, 1 mm thickness,(A) before processing (B) with etching of 5 × 5 µm (C) magnified etching area



Results and discussion

The capabilities of the FIB/SEM technique namely drilling etching, polishing, milling (cutting) and imaging (the main function) were invested in closest to the real analysis.

A cross-section of healthy human tooth, 1 mm in thickness, was prepared to the irradiation process. Observations of the target material before, during and after the laser ablation were done. Fig. 1a reveals that the surface of the tooth was relatively rough due to the transverse sectioning process. That roughness could be eliminated using low current ion beam to etch a thin layer from the tooth superficially after the determination of the area to be processed as shown in Fig. 1b,c. In this regard, the roughness plays an important role to decrease the reflectivity of the surface for the laser beam, so that the absorption increase resulted in the enhancement of the ablation efficiency. However, that effect was effective in longer wavelength, long pulse duration and low level energy density while in the ultra-fast pulse duration the reflectivity of the surface could be neglected.

FIB images obtained for laser-irradiated human tooth sample are shown in Fig. 2a,b. The sample was exposed to a single shot Nd: YAG laser operating at 532 nm wavelength, the pulse duration (FWHM) = 40ps. Relatively low fluence of 1 J/cm^2 was used. The target was irradiated by focusing the laser beam using a lens of 10 cm focal length. To get large exposed area, about 1.8 mm in diameter, the sample was located in a defocusing position. The damage was easily created at the given fluence on both enamel and dentin surfaces (Fig. 2a). In this regard, the findings of Serafetios *et al*. (1999) was in a good agreement with our results that showed morphology changes in the fundamental and the second harmonic Nd: YAG laser. The most prominent modification was recorded for 532 nm. Clean edges of the damaged area are well observed in the ion beam image of Fig. 2b. The energy confinement in the ablative zone and hence the producing of sharp edges were due to three factors: low thermal conductivity, low thermal diffusivity for dentin (1.39 x 10^{-3} cal / sec cm c, 1.87x10^{-3} cm^2/sec respectively) (Brown, 1970) and ultra short pulse duration (40 ps). The morphology of the ablation zone was highly rough resulting from the re-solidification of the molten materials which were drawn to the central region. Hydrodynamic structures and splashed melt at the periphery of the ablation are not visible because the fluence used is not high enough to remove the material. The collagen fibers are distinguished in the magnified ion image of the edges for the exposing region to the laser beam as shown in Fig. 2b.

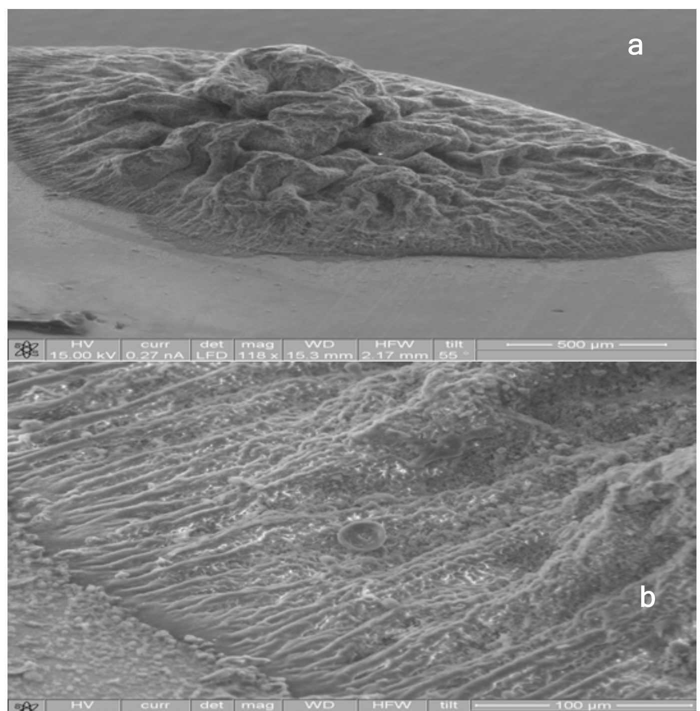
The interaction of 40 ps laser pulse duration with the human tooth could be described based on FIB analysis. The deposited energy on the sample have been absorbed within the duration of the pulse, the superficial layer was heated and reaching to the melting point. The absorbed energy was concentrated underneath the outer layer resulting in volume stress due the stretching of the heated material, but the fluence was not enough to eject the molten particles out and generating a crater. It is clear that the molten matter was drawn from the periphery to the central region which represents the evidence of the effect of internal volumetric pressure on the centre of ablation area. According to Niemz (2002), results of a series of experiments indicated that the incident energy density threshold in the ps range was given by $E_{th} \sim (\tau)^{0.5}$, where τ is the pulse duration in picosecond. For τ =40ps, it means that E_{th} (J/cm^2) ≈ 6J/cm^2 . Fig. 2a the energy density on the target was1J/cm^2. Hence, it is close to the threshold, confirming the analysis of the obtained FIB image for the irradiated target.

In general, the incident laser energy is partially absorbed during the laser interaction with the tooth surface. If the fluence was not so high, the initially absorbed part cannot induce multiphoton absorption (MPA) and multiphoton ionization (MPA). That means, free electrons could not be generated by means of MPA, i.e. additive energy cannot be absorbed from the laser to initial ionization avalanche and producing an exponential growth of free electron (Mirdan *et al*., 2009). The energy distribution across the laser beam and the temporal behavior of the laser pulse play an important role in the shape pf the ablation area and the generated crater.

A hole was drilled by FIB in order to investigate the shape and the composition of the material deeply. Fig. 3a represents three holes that were drilled using energetic ion beam for different depth (1, 2, and 4 µm). It was clear that an accurate hole could be made without the accumulation of molten particles on the periphery because the material was removed to the principle of sputtering. Two other holes were drilled, 4 µm in depth to select the best one as shown in Fig. 3b. The hole in the center of the image was magnified (vide Fig. 3c).

The crater generated was surrounded by destroying collagen fibers and the morphology structure varied in the center of the hole. For precise analysis of the profile of the created crater regarding its shape and structure, it should be cut via FIB. The processed area was covered by a thin layer of platinum to avoid damages during the

Fig 2. Nd:YAG laser induced morphology changes of human tooth sample. (a) ablated area of 1.8 mm in diameter (b) magnified ablation edge revealing the collagen fibers

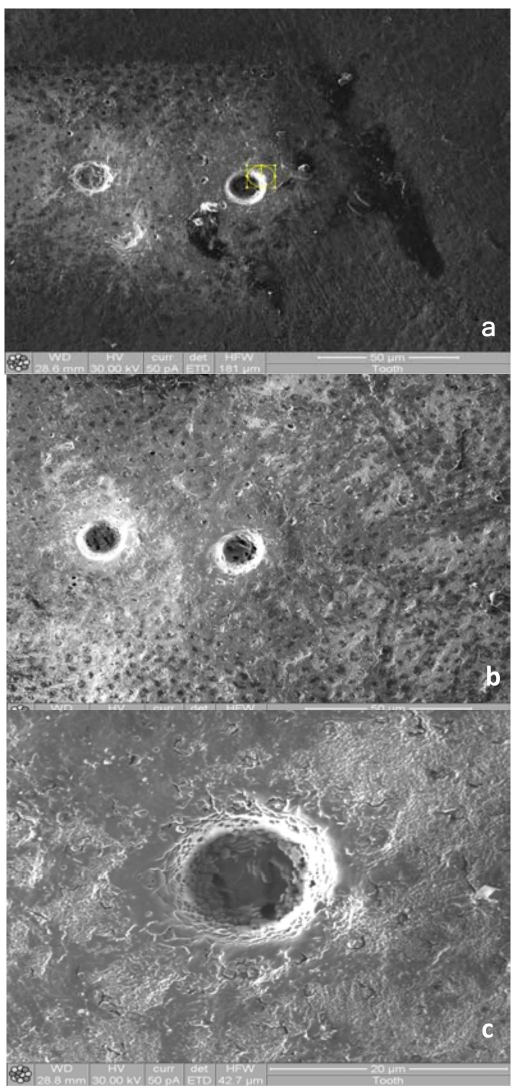


Analysis was carried out by focused ion beam. Laser parameters: wavelength= 532 nm, pulse duration= 40 ps, energy density= 1J/cm^2-singleshot

cutting process from one side to another then two ends of the profile was polished with low current ion beam to get more clear profile. The tooth sample was coated by a thin layer of graphite before the SEM imaging because the dental material is non conducting. The cutting sample was presented in Fig. 4a, different morphologies were observed inside and around the cutting area.

EDAX analysis for those areas was recorded to note the variation in the composition of the tooth material. Fig. 4b 1 illustrates the composition of the material inside the cutting area (no.4 in Fig. 4a), it is clear that no variation in the tooth material except the appearance of Ga due to the use of ion bean while the normal composition was detected in Fig. b2, b3 (no.5, 6 in Fig. 4a) which represents the surrounding area. The bright area in Fig. 4a is the region of Platinum coating which was recorded clearly in Fig. 4b4 in addition to the presence of Ga. To get an obvious imagination for the shape of the created crater, FIB image for the cutting region was carried out, as shown in Fig. 4c. Different morphology was observed, the processing area looks-like conical shape due to the focusing beam, also the surrounding area was clearly damaged because the energetic ions destroy the collagen fibers.

Fig. 3. FIB induced hole drilling for human tooth (a)Three holes with different depth (1, 2, 4 µm) (b)The best two holes done of 44 µm in depth; (c)The magnified image of the hole in the center of the image.



Conclusions

In the present work the extracted results for the picosecond laser ablation of human tooth revealed that the energy deposited (laser energy density) plays an important role even if ultra short pulse duration (40 ps) is used. FIB images showed that the morphology of the laser exposed area before the ablation threshold gave a clear impression for the stages of material removal. The high capability of using FIB for the analysis of ablation results was observed. In addition to the imaging, FIB proved the high potentiality of drilling, polishing, etching and milling of the human tooth.

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Fig. 4. SEM image for tooth processed via FIB technique. a) Four distinguished areas were observed numbered as 4, 5,6,7. b1), b2), b3, b4) are EDAX spectra according to the above numbers 4, 5, 6, 7 respectively. c) FIB image for the cutting area showing the shape of the hole drilled the internal structure of dental material