Compositional change in human enamel irradiated with MIR free electron laser

LIU Nianqing[1], LI Yonggui[1], ZHU Junbiao[1], ZHANG Liwen[1], WANG Mingkai[1], WU Gang[1], YANG Xueping[1], Li Guangcheng[1], Huang Yuying[1], DONG Yanmei[2] & GAO Xuejun

1. Institute of High Energy Physics, Chinese Academy of Sciences, Beijing 100039, China;
2. Stomatological College, Peking University, Beijing 100081, China
Correspondence should be addressed to Liu Nianqing (e-mail: liunq@ihep.ac.cn)

Abstract **The purpose of this study was to investigate compositional changes in human enamel irradiated with the free electron laser (FEL). The exposure on dental enamel at the wavelength of 9.64 μm was observed with the Beijing free electron laser. The distribution of elements in the irradiated or non-irradiated enamel was measured by scanning electron microscope (SEM) with energy-dispersive spectroscopy and synchrotron radiation X-ray fluorescence (SRXRF) in Beijing Synchrotron Radiation Facility (BSRF). The results showed that the P/Ca ratio in the ablation region of enamel at the maximum wavelength of infrared absorption was obviously smaller than that at the non-maximum wavelength. In the ablation region the ratios of P/Ca and Ca/Sr were smaller than those in the non-ablation region. The distributions of P, Ca and Sr in the ablation region were heterogeneous due to the element change caused by FEL irradiation.**

Keywords: human enamel, Beijing free electron laser, synchrotron radiation X-ray fluorescence, ablation, element distribution.

The conventional lasers in dentistry applications, such as Er:YAG, CO2, Ho:YAG, possess thermal side effects within the vicinity of the irradiated area, due to their inherent characteristics of monochrome and continuousness of the long pulse width. The longer laser pulse causes the rupture of hydroxyapatite structure due to the sudden explosive gasification of the water, deep slits up 3 mm and melting phenomena in enamel and dentin[1−6]. Comparing with medium infrared FEL, MIR FEL has the characteristics of wide wavelength tunability, ultrashort pulse (ps) and high peak power. If the FEL is tuned to a characteristic value at an absorption maximum of the major component of the irradiated material, the material will be ionized. High ablation efficiencies reduce the required energy per pulse and the temperature increases for short pulse irradiation as well. The damage of soft or hard tissue within vicinity of the irradiated area was limited[7]. Therefore, FEL will offer a distinct combination of high ablation efficiency, low thermal side effects and the pain reduction as a potential benefit of light source. Ogino reported that the irradiated FEL induced desorption of ion from tooth dentine, the resonant multi-photon vibrational excitation of molecules and the change of the ratio of Ca to P[8−10]. In order to study the effects of FEL irradiation on dental composition, the distribution of elements in enamel irradiated and non-irradiated by FEL was investigated by SRXRF and SEM.

1 Materials and methods

(ⅰ) Samples. Experimental samples were prepared in Stomatological College, Peking University. The extracted human teeth were cut into the slices with the thickness of 0.5—1 mm along their longitudinal direction, and were polished to produce a flat surface, named sample 1, 2 and 3 respectively. Three slices were irradiated by FEL, then sample 1 was tested by SRXRF and samples 2 and 3 by SEM.

Meanwhile, the fragments of enamel of extracted human teeth were ground by a mortar into powder and pressed into the slices about 0.5 mm thick, as control samples for the measurement of SRXRF.

(ⅱ) MIR FEL. Beijing FEL was an S-band rf linac-based FEL with its tunable wavelength region of 7—18 μm whose double pulse time structure can be seen in fig. 1[11]. Within each macro-pulse of 2—4 μs pulse width and 3 Hz repetition rate there were more than 10000 micro-pulses with the width of 2—4 ps pulses and the repetition rate of 2856 MHz. About 1 μJ energy per micropulse corresponds to about 5 mJ laser photon energy per macropulse. The spot of FEL beam was about 100×300 μm^2 depending on the slanting degree of the beam on the sample surface.

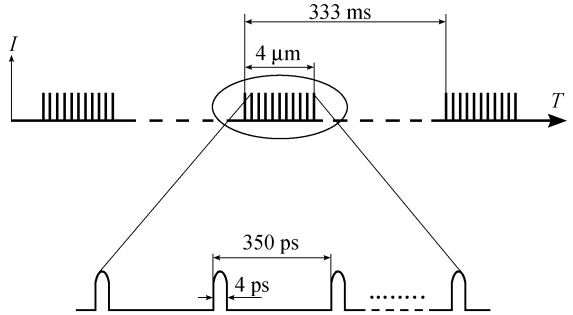

Fig. 1. Time structure of double laser pulses of the S-band rf-linac-FEL at BFEL.

Sample 1 and samples 2 and 3 were respectively irradiated for 15 and 10 min (samples 2 and 3) with FEL of 9.65, 9.46 and 10.6 μm respectively.

(ⅲ) SRXRF. The SRXRF experiment for the measurement of sample 1 was performed at the BSRF[12]. The spot size of the white light was about 40 μm×40 μm. Within the FEL irradiation region, sample 1 was scanned at 11 and 17 points on the cross lines from its center shown as *X* and *Y* in figs. 2 and 3 respectively. The control sample was scanned at 3 points. An AXIL program performed the processing of the X-ray spectra. The area of

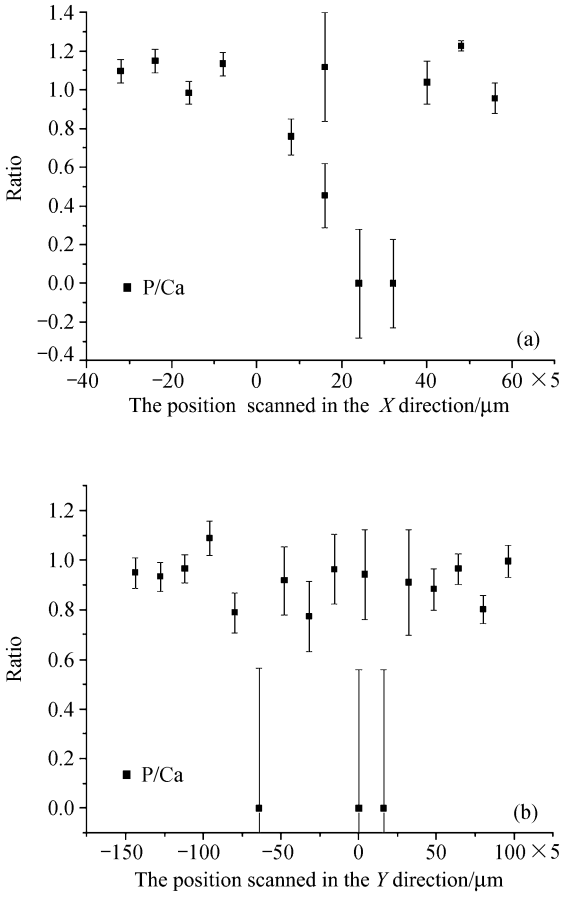

Fig. 2. Ratio of P/Ca changes in ablation enamel to that in the normal enamel along the *X* and *Y* directions.

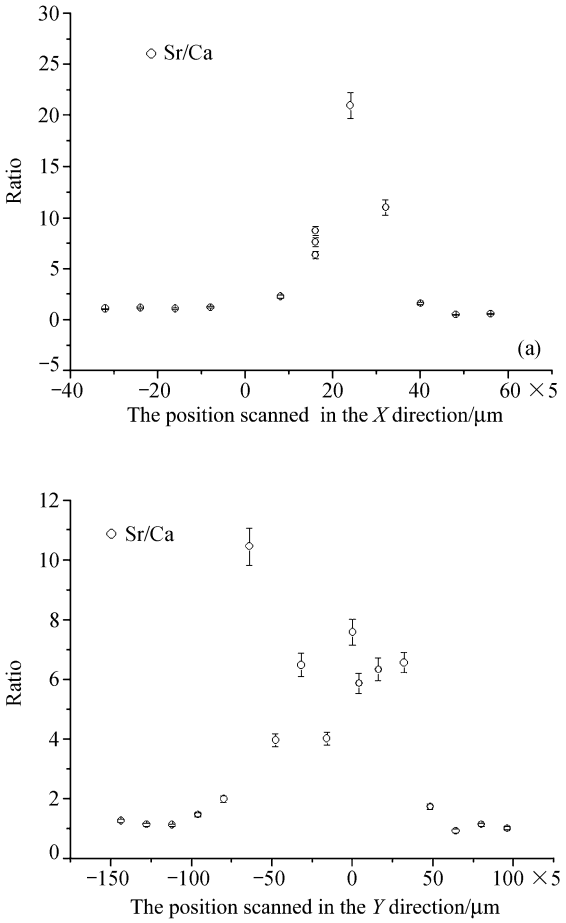

Fig. 3. Ratio of Sr/Ca changes in the ablation enamel to that in the normal enamel along *X* and *Y* directions.

the peak in X-ray spectrum was proportional to the content of the corresponding element. When the net area of the element peak in X-ray spectrum was 3 times more than the background underlying peak, it was taken as the limit of minimum detection (MDL).

(iv) SEM. A SEM (KYKY2800 with operation voltage of 15 KV and filament current of 75 μA) evaluation of the enamel samples 2 and 3 was performed. The beam spot about 1 μm was scanned within the irradiation and the non-irradiation areas of samples 2 and 3. The scanning area of 1.5 mm×0.5 mm was chosen to scan over the equivalent areas of sample 2. The P/Ca variation of the irradiated samples 2 and 3 and that of non-irradiated samples were obtained.

2 Results and discussions

Fig. 4 shows the IR absorption spectra of the powder sample of human enamel and dentine. The enamel is the hard tissue in human body, which contains 95% hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$), 4% water and other 1% organic matrix. Dentine consists of 70% hydroxyapatite,

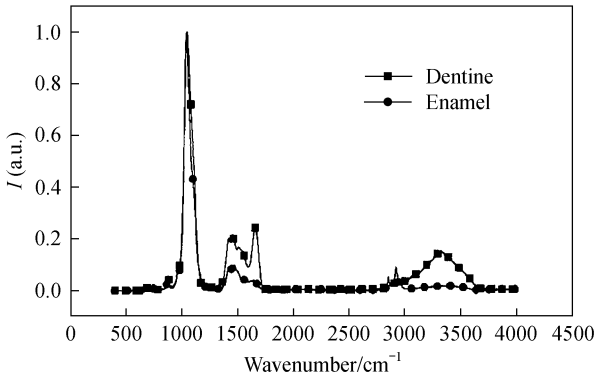

Fig. 4. FTIR absorption spectra of enamel and dentine.

10% water and 20% organic matrix (mainly collagen and elastin). The wavelength of FEL irradiation was chosen according to the IR absorption spectra. The maximum absorption wavelength is located at 9.66 μm, namely, characteristic absorption wavelength of hydroxyapatite.

During the FEL irradiation on the samples, the visible light emission and the smell of the burning protein

were observed at the very beginning, then they decreased gradually. Finally, under an optical microscope, an ellipse crater, i.e. an ablation region was found. These results suggested that there was a reaction of the organic matrix and the mineral of the enamel during FEL exposure. No more cracks were observed.

The SRXRF results of sample 1 showed that there were higher peaks of P, Ca and Sr, as well as lower peaks of Zn and Fe in the X-ray spectra of the enamel samples. The ratios of P/Ca and Sr/Ca in the irradiated area of sample 1 in the *X* and *Y* directions to these of non-irradiated area were changed, as shown in fig. 2 and 3, respectively. In addition, the lower energy X-ray, such as P X-ray, was easily absorbed by the air and Be window of Si(Li) detector. If the net area of P peak was less than MDL, the value of P/Ca was zero (fig. 2). The distribution of P, Ca and Sr in the ablation area was non-homogeneous due to the element change caused by FEL irradiation. The reduction of the element content in the center of the ablation area was more than that in the margin. The reduction of various elements was P>Ca>Sr. These results implied that the light atom in enamel was easily removed and ionized from crystal lattice during FEL irradiation at the resonance absorption band. According to the changes of the element content, the ablation region of the enamel sample was about 600 μm×200 μm.

The results of SEM are in the following: the values of P/Ca of the irradiated and non-irradiated enamels are listed in table 1. The reduction of P in the enamel irradiated with characteristic absorption wavelength of hydroxyapatite 9.64 μm was more than that of the non-characteristic absorption wavelength of 10.6 μm. The distribution of elements within the ablation area was consistent with that by SRXRF. The cause of the P reduction in the enamel irradiated with 10.6 μm was perhaps the comburent of the organic matter in the enamel.

Table 1 The P/Ca values of the irradiated and non-irradiated enamels in X-ray fluorescence spectra measured by SEM

Wavelength of FEL	Non-irradiated		9.64 μm		10.6 μm
Scanning area	1 μm^2	1.5 mm ×0.5 mm	1 μm^2	1.5 mm ×0.5 mm	1 μm^2
	0.706		0.646		0.650
Ratio of P/Ca	0.682		0.654		0.612
			0.437		0.646
			0.233		0.639
			0.477		0.663
			0.639		
			0.609		
Mean	0.694 ±0.012	0.682	0.528± 0.156	0.504	0.642± 0.019

3 Conclusion

When the enamel was exposed to the characteristic absorption wavelength of hydroxyapatite, P, Ca and Sr in the enamel were reduced effectively; meanwhile, the light and the ellipse ablation region could be observed. The content change region of the elements was very small (about 600 μm×200 μm). In the ablation region, the change of the elements is denoted by P>Ca>Sr. The measurement of SEM proved that the distributions of P and Ca in the ablation region were heterogeneous. The FEL might be a potential light source in the near future.

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