Ceramics-Silikáty 65 (1), 24-29 (2021)
www.ceramics-silikaty.cz doi: 10.13168/cs.2020.0038

EVALUATION OF SURFACE ROUGHNESS AND ELASTIC MODULUS IN CERAMIC AND RESIN COMPOSITE CAD/CAM MATERIALS DURING AGEING

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Submitted August 28, 2020; accepted October 5, 2020

Keywords: Nano-composite resin, Hybrid ceramic, Surface roughness, Elastic modulus, Thermal aging

Background and objectives: To evaluate and compare the surface roughness and elastic modulus in ceramic and resin composite CAD/CAM materials during thermal ageing. Materials and Methods: Four ceramic and resin composite CAD/CAM materials (Lava Ultimate HT and LT, VITA ENAMIC HT and LT) were selected for this study. The specimens were treated with the aim of measuring the surface roughness and mechanical properties. Before and after the accelerated ageing protocol (5 000 cycles, 5 °C and 55 °C), the specimens were subjected to nanoindentation and a 3D profilometer. Results: The surface roughness of the Ultimate HT before and after ageing ranged from 118.48 ± 12.87 to 123.42 ± 10.68 nm without any statistically significant differences ($p < 0.05$). The surface roughness of the Ultimate LT before and after ageing ranged from 90.18 ± 10.57 to 96.91 ± 6.28 nm without any statistically significant differences ($p < 0.05$). The Ra of the ENAMIC HT before and after ageing ranged from 87.84 ± 6.67 to 167.65 ± 21.80 nm with statistically significant differences ($p < 0.05$). The Ra of the ENAMIC LT before and after ageing ranged from 72.96 ± 6.61 to 97.75 ± 10.23 nm with statistically significant differences ($p < 0.05$). The ENAMIC HT and LT had an elastic modulus nearly twice as much as the Ultimate HT and LT, respectively. Conclusions: The thermal ageing did not affect the elastic modulus. The thermal ageing increased the Ra in the ENAMIC HT and LT Group, but did not affect the Ra in the Ultimate HT and LT Group.

INTRODUCTION

In the past decade, CAD/CAM dental materials have been widely used for aesthetic restorations manufactured in a digital workflow. More recently, resin composites and hybrid materials have become a new alternative. The strength, toughness and wear resistance are attributed to the ceramic parts, while the polymer parts are responsible for the flexibility [1, 2]. VITA ENAMIC is a kind of representative commercial Polymer-infiltrated-ceramic-network composites (PICNs), which was introduced by VITA in 2013. Lava Ultimate is essentially a filled composite, launched by 3M ESPE in 2012 [1, 3].

Idealistic restorative materials have to highly simulate a natural tooth in terms of both their structure and properties. The oral environment is quite complex, so dental restorative materials are subjected to chemical, biological, mechanical, and thermal changes. The surface roughness and mechanical properties might be affected by multiple factors [4]. A rough surface of a dental restoration will not only bring more bacterial adhesion and result in more abrasive damage to antagonist enamel [5-7]. A smooth surface with a long-lasting polish and a thermal ageing resistance are two important considerations in clinical practice.

EXPERIMENTAL

Four different CAD/CAM resin-ceramic restorative materials were used in the present study. The manufacturers, shades, types and chemical compositions of the tested materials are presented in Table 1. HT and LT represented the high and low translucency, respectively.

Table 1. Characteristics of the four materials used in the study.

Material	Shade	Code	Manufacturers	Composition	Type
Lava Ultimate	A2-HT	LH	3M ESPE, USA	80 wt. % nanoceramic particles embedded in a 20 wt. % highly cross-linked resin matrix	Nano-composite resin
	A2-LT	LL			
Vita Enamic	2M2-HT	EH	Vita Zahnfabrik, Germany	Porous structure-sintered ceramic matrix infiltrated with a polymer material (86 wt. % ceramic network and 14 wt. % polymer network)	Hybrid ceramic
	2M2-LT	EL			

Specimen preparation and thermocycling ageing

A total of four CAD/CAM dental materials were investigated, two hybrid ceramics, Vita Enamic (Vita Zahnfabrik, Germany), and two nano-composite resins, Lava Ultimate (3M ESPE, St. Paul, MN, USA). The materials are described in detail in Table 1. Discs were cut into thicknesses of 1 mm by a precision saw. All the tested surfaces were ground with #600, #800 and #1200 SiC papers, followed by polishing with a Sof-Lex kit (3M ESPE, USA) and an ENAMIC Polishing Set (Vita Zahnfabrik, Germany), respectively. The polishing process strictly followed the manufacturers' instructions. The four CAD/CAM materials were treated by pre-polishing (30 s) and high-gloss polishing (30 s). The practitioner kept moving the polishers around on the surfaces and did not stay in one place too long in order to avoid creating grooves or pits. Although machine polishing results in a significantly higher surface gloss than manual polishing, we chose manual polishing to simulate clinical conditions. To simulate the hydrothermal cycle and ageing of the teeth in an oral environment, all the samples were subjected to thermocycling (Proto-tech, USA) for 5 000 cycles at temperatures alternating between 5 and 55 °C with an immersion time of 30 s.

Surface roughness measurements

All the samples were tested using a profilometer device (VK-X200, Keyence, Japan). The surface roughness was expressed in terms of the roughness average (Ra), which is usually measured in nanometres (nm). Ten discs per group were prepared for the surface roughness evaluation. For each disc, the central area was selected. The mean values were calculated before and after the thermocycling ageing. The most representative 3D profile for each of the four materials are displayed.

Nano-indentation tests

The results of the nano-indentation tests were used to calculate the elastic modulus. Three discs per group for the nano-indentation were prepared. For each specimen, two points on the surface were examined with a nano-indenter (XP, Keysight Technologies, USA). The maximum depth of the indentation was 2000 nm. The force and length of the resultant diagonal were recorded simultaneously during each holding interval. The in-progress stress-strain characteristics were recorded and used to calculate the elastic modulus.

SEM observation

The material's surfaces were examined by using a scanning electron microscope (SEM) (Hitachi SU 8040) at ×2000 magnification. The measured voltage was set at 5 kV.

Statistical analysis

The average Ra values and the mean elastic modulus were recorded. Microsoft Excel 2013 software was used to input the data. A Mann–Whitney U test was conducted to detect any significant difference. In all the statistical tests, p-values < 0.05 were considered statistically significant when using SPSS (Version 22.0; Chicago, IL, USA).

RESULTS AND DISCUSSION

The mean surface roughness and standard deviation before and after thermal ageing for the various Ceramic and Resin Composite CAD/CAM Materials are shown in Figure 1. The surface roughness values ranged from 72.96 nm to 118.48 nm after polishing and from 90.18 nm to 167.65 nm after thermal ageing. The highest surface roughness in all the materials tested was observed in the Enamic HT Ageing Group, while the lowest surface roughness was observed in the Enamic LT Initial Group. After the thermal ageing, a significant increase ($p < 0.05$) occurred for both the Enamic HT and LT Group. No significant differences existed between the initial and ageing states in both the Ultimate HT and LT Group ($p > 0.05$). The Ra in the Ultimate HT and LT Initial Groups showed significantly higher differences than those in Enamic HT and LT Groups, respectively ($p < 0.05$). The Ra of the Ultimate HT and Enamic HT Initial Groups showed significantly higher differences than the LT ones, respectively ($p < 0.05$).

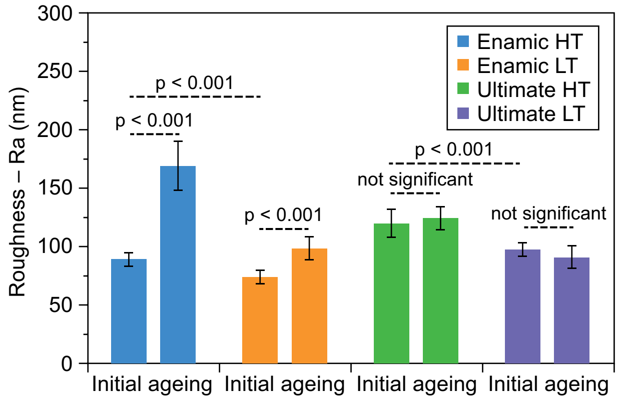


Figure 1. Surface roughness (Ra) values, by the material tested. All the data are presented as a mean ± standard deviation (HT – high translucency; LT – low translucency).

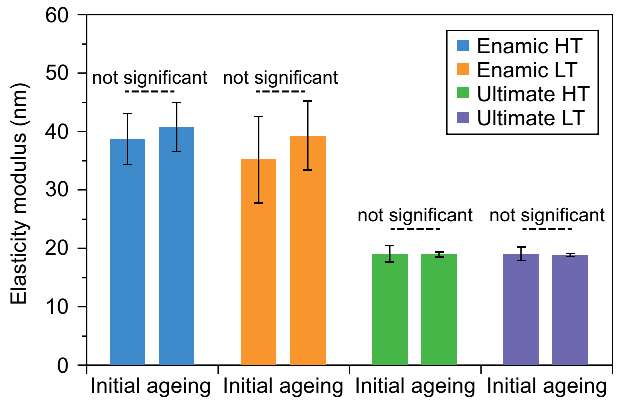


Figure 2. Elastic modulus values, by the material tested. All the data are presented as a mean ± standard deviation. (HT – high translucency; LT – low translucency).

The elastic moduli of the enamel, dentin, Enamic HT/LT and Ultimate HT/LT are illustrated in Table 2. The mean elastic modulus and the standard deviation before and after the thermal ageing for the various Ceramic and Resin Composite CAD/CAM Materials are shown in Figure 2. No significant differences between the initial and ageing states in all the tested materials ($p > 0.05$). No significant differences existed between the HT and LT in all the tested material ($p > 0.05$). The ENAMIC HT and LT had an elastic modulus nearly twice as large as the Ultimate HT and LT ones, respectively. The elastic moduli of the Lava Ultimate were closer to those of natural dentin. Our findings were similar to those that Hae-Yong Jeong et al. reported [16].

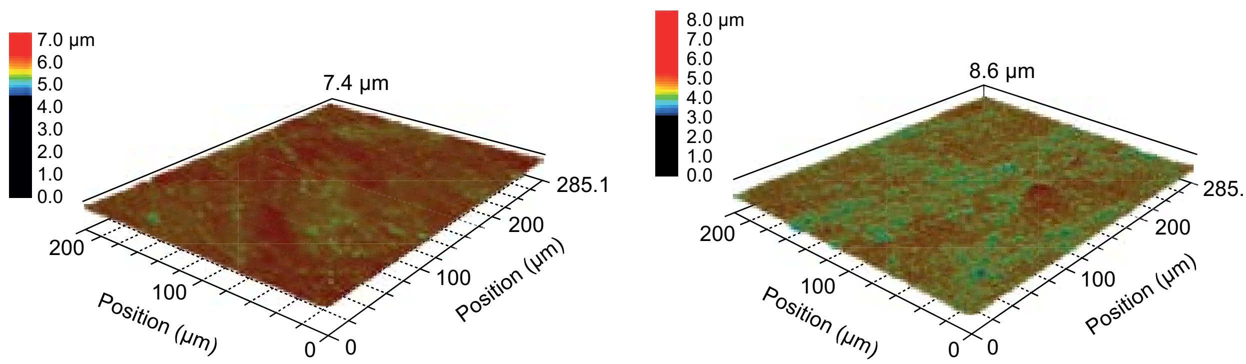
The representative 3D profilometry images of the four tested materials before and after the thermal ageing are shown in Figure 3. The red areas represent the part of the surface with the highest height (the peaks), while the blue areas represent the part of the surface with the lowest height (the valleys). The VITA ENAMIC HT and LT after polishing showed a much smoother surface than those after the thermal ageing. The Lava Ultimate HT and LT could keep a smooth surface after the thermal ageing. Furthermore, the SEM images for the four samples shown in Figure 4 present the surface microstructure.

This study evaluated and compared the surface roughness and elastic modulus in ceramic and resin composite CAD/CAM materials during thermal ageing. This study showed that thermocycling significantly increased the surface roughness of the ENAMIC HT and LT, while thermocycling did not affect the elastic modulus of the four CAD/CAM materials. The surface roughness and elastic modulus of the tested CAD/CAM materials may not be affected by thermocycling.

Table 2. Comparison of the elastic modulus with the commercial Ceramic and Resin Composite CAD/CAM materials and a natural tooth.

	Enamel	Dentin	Enamic HT	Enamic LT	Ultimate HT	Ultimate LT
Elastic modulus (GPa)	48-115[8-12]	8.7-25[8, 12-15]	38.67-40.63*	35.15-39.17*	18.97-19.02*	18.87-19.00*

** tested before and after the thermal ageing (HT, high translucency; LT, low translucency)*



a) Enamic HT – Initial

b) Enamic HT – Ageing

Figure 3. 3D profilometry of all the tested materials before and after the thermal ageing. *(Continue on next page)*

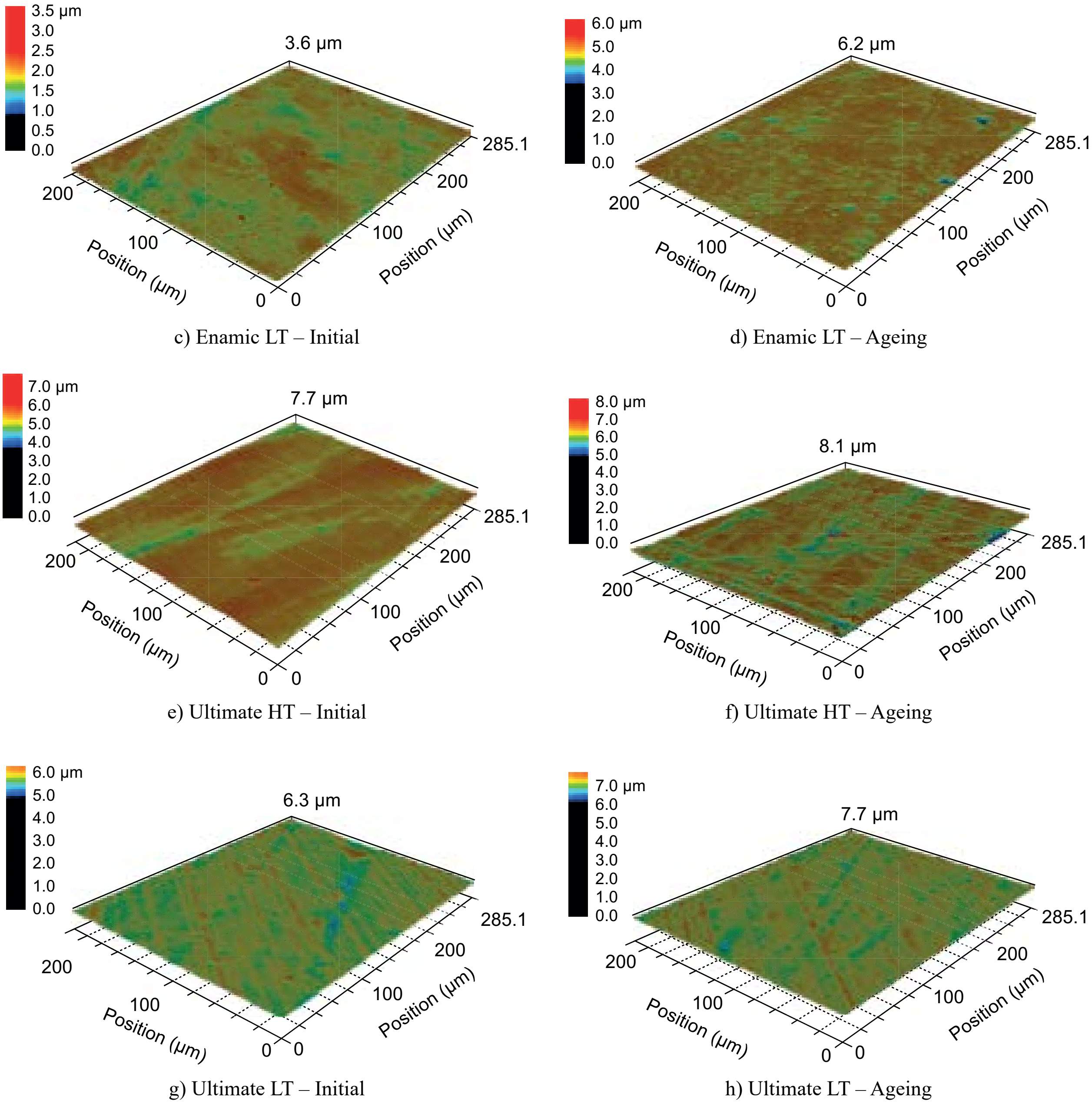
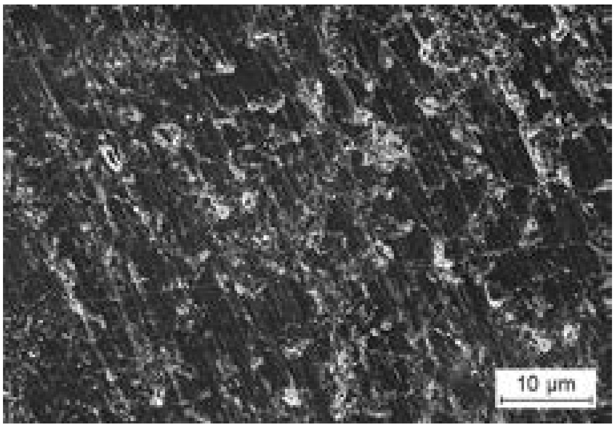


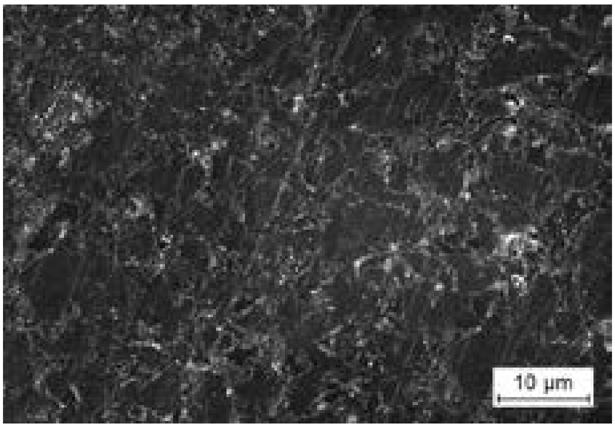
Figure 3. 3D profilometry of all the tested materials before and after the thermal ageing.

In addition, the elastic modulus of two CAD/CAM materials with different translucency ranges had no significant difference. The surface roughness of the Ultimate HT Group and ENAMIC HT Group showed significantly higher differences than the LT ones after polishing.

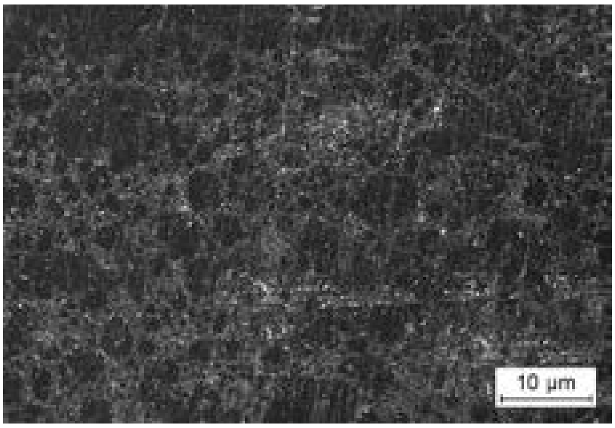
The results of this study showed that the thermal aging did not affect the elastic modulus irrespective of the resin-ceramic CAD/CAM material used. Our results are consistent with previous reports that indicated that the elastic modulus did not change significantly after thermocycling [16, 17]. However, it was reported that thermocycling affected the fracture toughness and biaxial flexural strengths of hybrid CAD/CAM materials, whereas leucite and lithium disilicate ceramics showed stability [16, 18]. Vita ENAMIC was classified as a polymer infiltrated-ceramic-network (PICN) and could be indicated for crowns, onlays/inlays and veneers. VITA ENAMIC had a higher elastic modulus compared to the composite resin-based materials (Lava Ultimate), which only has an indication for onlays/inlays and veneers. The ageing protocols employed in the present study were concerned with thermal ageing while subjecting the specimens to 5 000 thermocycles of 5/55 °C. It was estimated that it would represent approximately half a year of the dental function [19], so the thermal cycling treatment could represent a short time exposure to the oral environment.



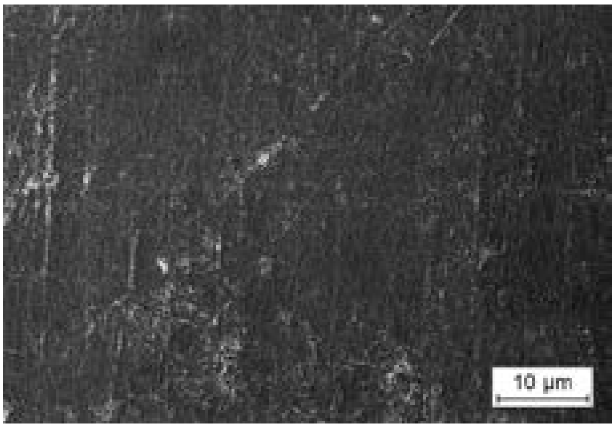
a) Enamic HT



b) Enamic LT



c) Ultimate HT



d) Ultimate LT

Figure 4. SEM images of all the CAD/CAM material surfaces (HT – high translucency; LT – low translucency).

The ENAMIC HT and LT Groups significantly increased the roughness (Ra) after the thermal ageing. Hae-Yong Jeong et al. reported similar findings [16]. In his study, the surface of the Lava Ultimate and Vita ENAMIC showed much smoother surface roughness with an Ra of 6 ~ 9 nm because of the machine polishing. Although machine polishing results in a significantly higher surface gloss than manual polishing, we chose manual polishing to simulate clinical conditions. Many studies reported that a higher roughness leads to increased bacterial adhesion or plaque formation and the critical Ra for increased bacterial adhesion is 0.2 µm [20-23]. The larger deposition of a bacterial biofilm on the tooth's surface could increase the probability of dental caries and periodontitis. All the roughness values were between 72.96 and 167.65 nm, which is lower than 0.2 µm, although the Ra in the ENAMIC HT Group after the thermal ageing was close to 0.2 µm. Hence, it could be considered to be clinically acceptable. A higher surface roughness indicates an increase in the surface irregularities which results from more wear in the dental restorations [24-26]. The 3D profilometry images showed the structures of the four CAD/CAM material changes after the thermal ageing. It might be speculated that the polymer part of the ceramic matrix was ageing. So, it is very important that regular check-ups and intraoral polishing should be carried out in dental practice.

The limitations of this study are that it is difficult to simulate various factors in the oral cavity, such as the chewing force and saliva. Furthermore, the limitation of the present study is that the applied ageing periods might be considered too short of a period. Additional studies are needed to clarify the influence of longer ageing periods on the mechanical properties and roughness of resin-ceramic composite CAD/CAM materials.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

- The thermal ageing process did not affect the elastic modulus irrespective of the resin-ceramic CAD/CAM material.
- A significant increase in roughness occurred for both the ENAMIC HT and LT Groups, while no significant differences were found in the Ultimate HT and LT Groups after the thermal ageing.

- The surface roughness in the Ultimate HT and LT Groups showed significantly higher than those in the ENAMIC HT and LT Group after the polishing treatment.

Acknowledgments

This research was supported by Research Foundation of Peking University School and Hospital of Stomatology (PKUSS20180111, PKUSS20200111) and Peking University School and Hospital of Stomatology Postdoctoral Fund YS0203.

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