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Improving the wear performance of feldspathic veneering porcelain by ion-exchange strengthening

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ABSTRACT

Objectives: The present study examined the effects of $Na^+ \rightarrow K^+$ ion-exchange on the wear performance of feldspathic veneering porcelain.

Methods: Bar and disk specimens were prepared using IPS classic as the feldspathic veneering porcelain. After ion-exchange by immersion of the specimens in melted KNO_3 at two temperatures for different time-periods, the bars were tested for flexural strength and Vickers surface hardness. The disks were paired with zirconia antagonists and tested with a pin-on-disk tribometer with 10 N for 70×10^4 wear cycles in artificial saliva. Wear analysis of the porcelain and zirconia was performed using 3D profilometer and analysed with one-way analysis of variance and Tukey's post-hoc pairwise comparison procedures. Worn surfaces were examined with scanning electron microscopy.

Results: The feldspathic veneering porcelain exhibited strong time-dependent wear behaviour, with typical running-in and steady wear stages. Ion-exchange treatments at 380 °C and 440 °C both enhanced the mechanical properties, decreased the wear rates of running-in wear and steady wear. The wear performance of porcelain treated by ion-exchange at lower temperature (380 °C) was improved significantly, especially reducing the wear rate of the running-in stage.

Conclusion: A thicker ion-exchange layer with less stress relaxation may be obtained by ion-exchange at lower exchange temperature for a long processing time. Such a protocol improves the wear performance of the porcelain effectively.

Clinical significance: Restorations with veneering porcelain may fail prematurely due to excessive wear. It important to improve the wear performance of the porcelain. Ion-exchange has the potential to strengthen dental veneering porcelain. Understanding the effect of ion-exchange on the wear performance of porcelain provides insight improving the wear performance of these restorations.

1. Introduction

Veneering porcelains functional as stress-bearing components in metal-ceramic and all-ceramic assemblies [1]. The wear behaviour of the porcelain veneering layers is an important attribute in the characterization of metal-ceramic or all-ceramic restorations [2,3]. For long-term clinical success, the wear resistance of the veneering porcelain should ideally be similar to natural enamel [4–6]. However, veneering porcelain surfaces usually exhibit severe wear [7,8]. Excessive wear of dental ceramic results in restoration failure [9] because of fracture or chipping of the ceramic that is initiated from wear facets at the occlusal surface [10]. Accordingly, it is important to improve the wear performance of veneering porcelain.

As a tribological property, the wear behaviour of a material is closely related to its surface mechanical properties. Improving the wear behaviour of a material may be achieved by surface strengthening. To

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date, different methods have been deployed for strengthening glasses and ceramics. A frequently used protocol is the creation of a residual compressive stress layer on the surface of the material [11]. This may be achieved by thermal tempering or chemical strengthening. Thermal tempering improves the residual compressive stress on the surface of the bulk ceramic, enhancing its mechanical properties [12–15]. Nevertheless, the thickness of tempered glass or ceramic is generally limited [16] and the procedure is difficult to be performed in the dental clinic. Ion-exchange is a well-known method of chemically strengthening monolithic glasses that contain alkali ions [17]. Residual compressive stresses on the surfaces of glasses may be induced by exchanging smaller alkali ions in the glasses with larger ions derived from molten salts at temperatures below the glass transition temperatures (T_g) [18]. This chemical strengthening strategy may be employed for glass-ceramic systems because ion-exchange also occurs in glassy matrices [19]. Previous studies have shown that ion-exchange is capable of successfully enhancing the mechanical properties of dental feldspathic porcelain and leucite glass-ceramic systems [11,20–25].

Ion-exchange has the distinct advantage that glass or glass-ceramic products with almost any geometry and thickness may be effectively strengthened [26]. This is significant for different types of dental restorations with individualized geometries and thicknesses. In addition, the translucency of the glass/glass-ceramic is not affected after ion-exchange [20–25]. Veneering porcelain is an important surface layering material for metal-ceramic and all-ceramic dental restorations because of its excellent aesthetics [27]. Hence, ion-exchange strengthening may find use in improving the wear characteristics of dental veneering porcelain.

Surface strengthening of veneering porcelain systems by ion-exchange may be performed at temperatures substantially below T_g, which has implication as the final processing step of glass/glass-ceramic restorations after clinical occlusal adjustment. Ion-exchange may be subsequently performed to strengthen the prosthesis surface within minimal distortion because of the low processing temperature.

The objective of the present work was to investigate the feasibility of using ion-exchange for improving the wear performance of feldspathic veneering porcelain. Specifically, ion-exchange was applied to a feldspathic veneering porcelain at a temperature below its T_g. By analysing the $Na^+ \rightarrow K^+$ ion-exchanged surface and sub-surface, the effects of ion-exchange on the mechanical behaviour and wear performance of the veneering porcelain were examined. The null hypothesis tested was that the ion-exchange has no effect on the wear performance of feldspathic veneering porcelain.

2. Materials and methods

2.1. Specimen preparation

Dental feldspathic porcelain materials are classic veneering glass-ceramics that are widely used in prosthetic dentistry for porcelain-fused-to-metal (PFM) restorations. The porcelain possesses a microstructure consisting of a glassy matrix with a volume fraction of about 70% and a leucite crystalline phase [28]. Feldspathic veneer porcelain powder (IPS classic, Ivoclar Vivadent, Liechtenstein) was mixed with the proprietary liquid and vibrated into two cuboid-shaped moulds ($24 \times 4.5 \times 3\,mm^3$ for testing mechanical properties and $13 \times 13 \times 3\,mm^3$ for the wear test). Each mould was shaken to remove excess liquid to keep the cuboid-shaped ceramic in a condensed state. The feldspathic veneer porcelain was sintered at 920 °C. All specimens were subsequently polished with silicon carbide paper, up to 1200 grit, and finished with diamond suspensions. The polished specimens were cleaned ultrasonically with acetone. The final dimensions of the polished specimens were $22 \times 4 \times 2\,mm^3$ for the bars and $13 \times 13 \times 2\,mm^3$ for the plates.

The corresponding parent glass of the feldspathic veneering porcelain was also obtained by re-melting the powder in a alumina crucible at 1450 °C for 1 h and at 1500 °C for 10 min. The re-melted glass was cooled to room temperature and removed from the crucible to yield a glassy block. The latter was used as a reference source for the ion-exchange experiment.

2.2. Ion-exchange processing

The T_g of the feldspathic veneer porcelain was analysed by differential scanning calorimetry (DSC; LabsysTG-DTA/DSC, SETARAM Instrumentation, France). Glassy powder with a mass of 30 ± 1 mg was place in a high-purity alumina crucible for DSC measurement. Measurement was conducted at a heating rate of 40 K/min under a flow of purified argon. After that, a T_g of ~567 °C was subsequently obtained from the DSC curve.

Based on the DSC analysis, two temperatures were selected for ion-exchange: 440 °C and 380 °C, which were 127 °C and 187 °C lower than the T_g, respectively. Reagent grade KNO_3 (99% purity, Sinopharm, China) with a melting point of ~333.8 °C was used for the ion-exchange process [29], which was performed in a tubular furnace (BTF-1100C-S, BEQ™, China). The feldspathic porcelain and parent glass specimens were placed in a molybdenum wire mesh basket as separated pieces, lowered into the furnace and held over the molten salt bath for 20 min as a pre-heating step [17]. After immersion, the specimens were withdrawn from the bath and cooled down in air to ambient temperature to accurately control the treatment time. After the ion-exchange step, the specimens were cleaned in deionized water to wash off the excess salt on the surfaces of the specimens [30]. Ion-exchange for the mechanical test specimens were conducted at the two aforementioned temperatures for 8, 16, 32 or 64 h. The specimens were designated according to the different ion-exchange experimental parameters; for example, "440 °C/64 h" means that a specimen was subjected to ion-exchange at 440 °C for 64 h.

The distribution of K^+ was determined using energy dispersive X-ray spectroscopy (EDS; S-4800, Hitachi High-Technologies Corp., Tokyo, Japan) for evaluation of the depth of ion-exchange [31]. The Na^+ and K^+ gradient distribution profiles of the ion-exchange specimens were detected by line scan analysis. The depth of the ion-exchange layer was obtained from the line scan curve.

2.3. Mechanical testing

Flexural strength before and after ion-exchange ($N = 10$) was determined by three-point bending using the $22 \times 4 \times 2\,mm^3$ specimens, according to ISO 6872 guidelines [32]. Testing was performed with a screw-driven testing machine (CMT4204, SUNS, China) at a constant cross-head displacement rate of 0.5 mm/min. Flexural strength (σ_f) was calculated using the equation :

$$\sigma_f = \frac{3Pl}{2wt^2} \quad (1)$$

where P is the fracture load, l is the test span (15 mm), and w and t are the width and thickness of the specimens, respectively.

After flexural strength evaluation, the fracture segments were used for micro hardness (H) testing, which was performed using a Vickers hardness tester with a load of 0.5 N (to ensure no obvious crack formation around the indents) and a dwell time of 15 s. Three specimens were used for each of the processing profiles to determine hardness, and ten indentations were made on each specimen.

2.4. Wear testing

The wear performance of feldspathic porcelain specimens before and after ion-exchange processing was investigated using Pin-on-Disk tribometer (CSM Instruments, CH-2034 Peseux, Switzerland). Wear was conducted with a constant vertical load 10 N that was applied during cyclic rotation. All specimens were fixed in the lower station where

rotation was performed at a constant speed of 200 rpm, against a fixed upper zirconia antagonist, for 70×10^4 cycles in artificial saliva (pH = 7) at 37 °C. This wear testing method was recognized as an important tool to understand the wear process and surface properties [33]. The ion-exchange treatment condition was chosen based on the results of mechanical testing. All the wear specimens were embedded in a round stainless-steel mould using auto-polymerizing acrylic resin. The antagonist pins were made of yttria-stabilized zirconia ceramic (Zenostar Zr Translucent, Weiland, Germany) with Vickers hardness of 13,000 MPa. Each cylindrical pin was 3 mm in diameter and 8 mm long. The sliding surface of the antagonist pins were ground and mirror-polished. The surface roughness (S_a) value of the original sliding surface of all wear specimens were controlled at 0.362 ± 0.019 μm and 0.226 ± 0.028 μm for the disk and pin specimens, respectively.

The mean height loss during wear and worn S_a values of the feldspathic veneering porcelain and the zirconia antagonist were determined at each checkpoint by a non-contact 3D white-light profilometer (PS50, Nanovea, Irvine, CA, USA) with scanning step-sizes of 20 μm in the X and Y directions and a resolution of 2 nm in the Z direction. These parameters were reported to be appropriate for ceramic materials [34]. A silicone rubber impression technique [35,36] was employed to avoid inaccurate measurement caused by dismounting of the specimen between checkpoints. Impressions of the worn surfaces of specimens were obtained using a silicone impression material (Silagum, DMG, Germany). The scanning area of the impression surface was 10 mm × 10 mm (lower specimen) and 4 mm × 4 mm (upper specimen), which was large enough to cover the entire wear area. Worn area morphology of the specimens was observed by scanning electron microscopy (SEM) using a Hitachi S-4800 microscope (Hitachi High-Technologies Corp., Tokyo, Japan) to identify wear tracks and wear mechanisms in different cycles.

Wear curves of the tested specimens were generated after different checkpoints. The wear rate was calculated using equation:

$$V = \Delta H / \Delta N \quad (2)$$

where V is the wear rate, $\triangle H$ is the wear height loss between two adjacent checkpoints, and $\triangle N$ is the wear cycle difference between two adjacent checkpoints.

2.5. Statistical analysis

Statistical analysis of the data was conducted with Prism Graphpad version 8.0 (GraphPad Software, Inc., San Diego, CA, USA). For the results of mechanical property and wear height loss, the means and standard deviations were analysed using one-way analysis of variance (ANOVA) and post-hoc Tukey's test, to determine whether the ion-exchange process affected mechanical properties and wear performance. The level of significance was pre-set at $\alpha = 0.05$.

3. Results

3.1. Microstructure

For comparison, SEM images of the sub-surfaces of the parent glass before and after ion-exchange at 440 °C for 16 h are shown in Fig. 1a and b. The corresponding EDS profiles for Na^+ and K^+ along the lines perpendicular to the surfaces are superimposed on the images. The original Na^+ and K^+ profiles in the monolithic glass revealed homogeneous distributions (Fig. 1a). After ion-exchange, the K^+ concentration presented a maximum on the surface, and continuously deceased to the un-exchanged level. The Na^+ concentration exhibited the opposite trend. The information in Fig. 1a and b indicated that Na^+/K^+ exchange between the molten KNO_3 bath and the glassy phase occurred by a general diffusion process in the parent glass [37]. Based on the K^+ EDS profile (or Na^+ EDS profile), the depth of ion-exchange may be roughly estimated by determining the tangent point of the profile and the corresponding base line (i.e. the un-exchanged level line). The depth of ion-exchange in Fig. 1b was estimated to be ~125 μm.

Fig. 1c and d show SEM images and superimposed EDS profiles for Na^+ and K^+ in the sub-surfaces of feldspathic porcelain before and after the ion-exchange at 440 °C for 16 h. At high magnification (insets in Fig. 1c and d), the porcelain exhibited typical glass-ceramic microstructure with leucite dendrites distributed within the glassy matrix – a result of dendritic growth [38]. The original Na^+ and K^+ in the microstructure displayed fluctuating distributions due to chemical composition difference between the leucite crystallites and the glassy matrix [38]. After the ion-exchange in Fig. 1d, the fluctuating distributions of Na^+ and K^+ did not change. However, the K^+ concentration presented an overall decreasing tendency from the surface maximum, while the Na^+ concentration exhibited the opposite trend, similar to the parent glass. Because ion-exchange occurs primarily between the molten salt and the glassy phase [26,30], the inclusion of leucite crystallites in the microstructure of a glass-ceramic causes an impeding effect on the ion-exchange [37]. The depth of ion-exchange for the porcelain in Fig. 1d was ~85 μm, which was less than that of the parent glass (Fig. 1b).

In Fig. 1e and 1f, SEM images and superimposed EDS profiles for Na^+ and K^+ in the sub-surfaces of the feldspathic porcelain after ion-exchange at 380 °C for 16 h and 64 h were shown respectively. The depth of ion-exchange layer in Fig. 1e was ~56 μm. Compared with Fig. 1d, the depth of ion-exchange layer formed by the same exchange time at lower temperature was relatively thinner than that at higher temperature. In Fig. 1f, the depth of the exchange layer was ~119 μm, indicating the increased exchange layer depth by prolonging the exchange time at lower temperature.

The depths of ion-exchange for the porcelain processed at 440 °C and 380 °C were plotted respectively as functions of the square root of the ion-exchange time (Fig. 2), which were determined from their corresponding EDS profiles. Ion-exchange depth at each of the temperatures increased proportionally with the square root of the treatment time, which was consistent with the universal rule for ion-exchange according to the Fick's second law [30,39]. To obtain a certain depth of ion-exchange, longer time was needed at the lower temperature. This is indicative of temperature-dependence of the diffusion process, according to Arrhenius law [40].

3.2. Mechanical properties

Changes in flexural strength (σ_f) of the feldspathic veneering porcelain with increasing ion-exchange time at 380 °C and 440 °C are shown respectively in Fig. 3a. Significant differences in σ_f were observed before and after ion-exchange ($p < 0.05$). The σ_f value of the porcelain before ion-exchange was ~79 MPa. Strength of the veneering porcelain increased to ~130 MPa and ~ 121 MPa after 16 h of ion-exchange at 380 °C and 440 °C, respectively. The σ_f values appeared to plateau with ion-exchange time at 380 °C, but decreased with increasing ion-exchange time at 440 °C. The σ_f values were significantly higher ($p < 0.05$) at 380 °C than 440 °C after 16 h of ion-exchange.

The surface Vickers hardness of the porcelain exhibited a similar tendency (Fig. 3b). Hardness increased significantly with ion-exchange time up to 16 h at either temperatures. Porcelain hardness at 440 °C continued to decrease significantly when ion-exchange time increased to 64 h. In contrast, a relative plateau in hardness for the specimens that were treated at 380 °C. Hardness values at 380 °C at all time-checkpoints were significant higher than those at than 440 °C ($p < 0.05$).

3.3. Wear behaviour

The wear height loss of the feldspathic veneering porcelain disk and the opposing zirconia pin up to 70×10^4 cycles are shown in Fig. 4a, and the wear rates are summarised in Fig. 4b. The boundary between

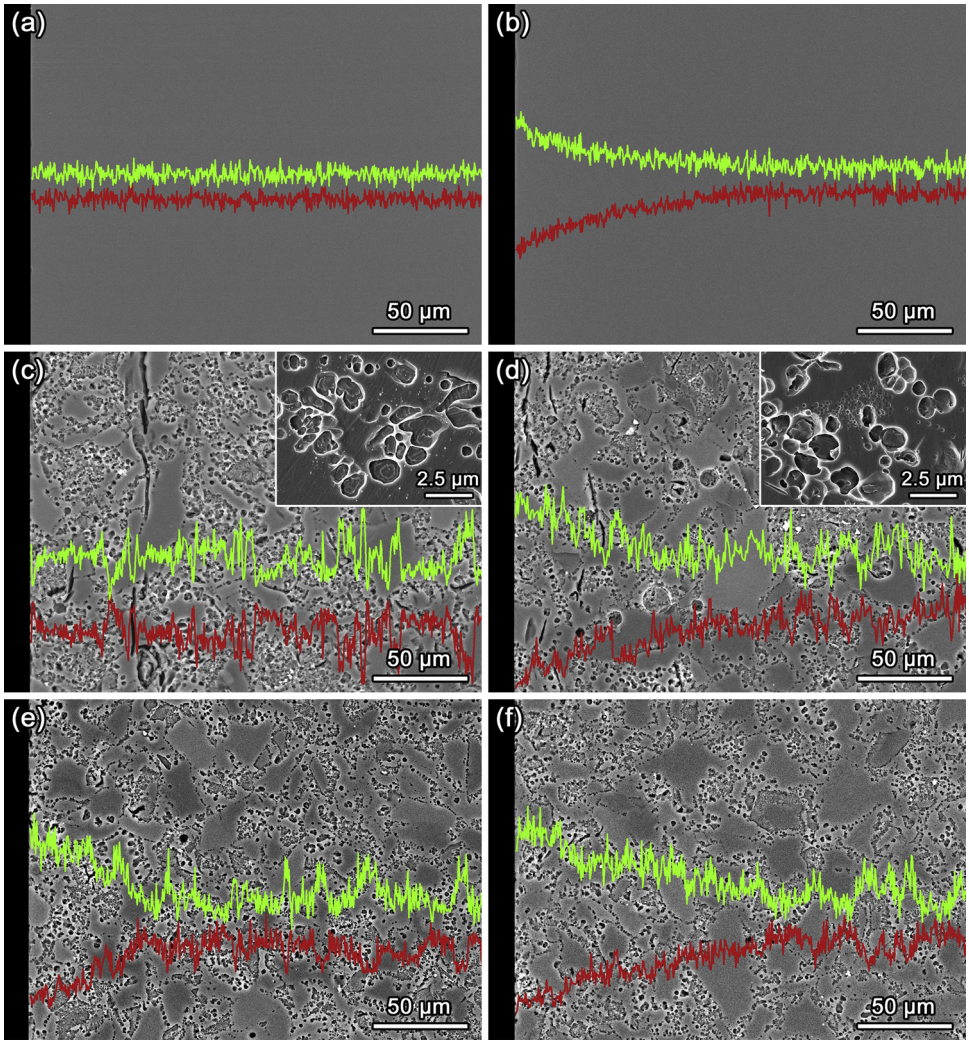

Fig. 1. Sub-surface SEM images. (a) and (b). Parent glass before and after ion-exchange at 440 °C for 16 h; (c) and (d). Feldspathic porcelain before and after ion-exchange at 440 °C for 16 h. Insets show enlarged microstructure; (e) and (f) the porcelain after the ion-exchange at 380 °C for 16 h and 64 h respectively. EDS profiles for Na^+ (red) and K^+ (green) are superimposed on the images.

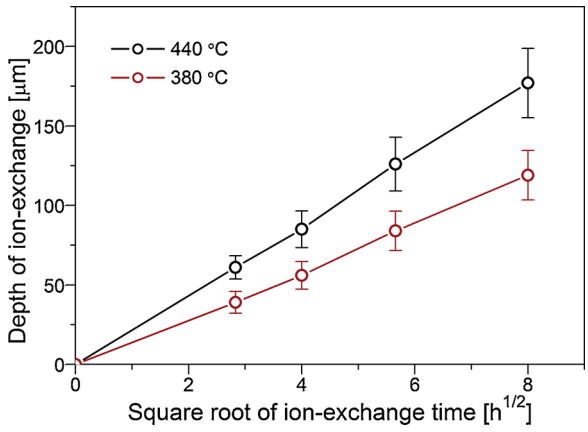

Fig. 2. Depths of ion-exchange as functions of the square root of the ion-exchange time for porcelain specimens processed at 440 °C and 380 °C, respectively.

the running-in wear stage and the steady wear stage of the porcelain disk was situated at approximately 18×10^4 cycles because the mean wear rate at 22×10^4 cycles (1.49 ± 0.34 μm/10^4 cycles) was significantly lower than that at 18×10^4 cycles (4.33 ± 0.62 μm/10^4 cycles) ($p < 0.05$), while no significant difference in wear rate was observed between 18×10^4 cycles and 70×10^4 cycles. Accordingly, the running-in wear stage of the feldspathic veneering porcelain ranged from 0 to 18×10^4 cycles and the steady wear stage ranged from 18×10^4 to 70×10^4 cycles.

The wear curves of the antagonist zirconia pin showed a similar tendency. The boundary between the running-in and steady wear stages was 18×10^4 cycles. After 18×10^4 cycles, the zirconia pin underwent a steady wear stage.

3.4. Effect of ion-exchange condition on wear performance

According to the results of the time-dependent wear behaviour, the average wear rate of the wear stages was calculated using Eq. (2). The wear heights (Δh) and wear rates (R_w) of porcelain disks and antagonist zirconia pins under different ion-exchange conditions are summarised in Tables 1 and 2, respectively. The mean wear rates in the running-in and steady wear stages are shown in Fig. 5. In the running-in stage, the average wear rates of all the ion-exchanged porcelain disks (Fig. 5a) were lower than those prior to ion-exchange ($p < 0.05$). Wear rate of the 380 °C/64 h group (0.89 ± 0.21 μm/10^4 cycles) was the lowest ($p < 0.05$). In the steady wear stage, the mean wear rates of the ion-exchanged porcelain were significantly lower ($p < 0.05$) than those prior to ion-exchange. Wear rate of the 380 °C/64 h group

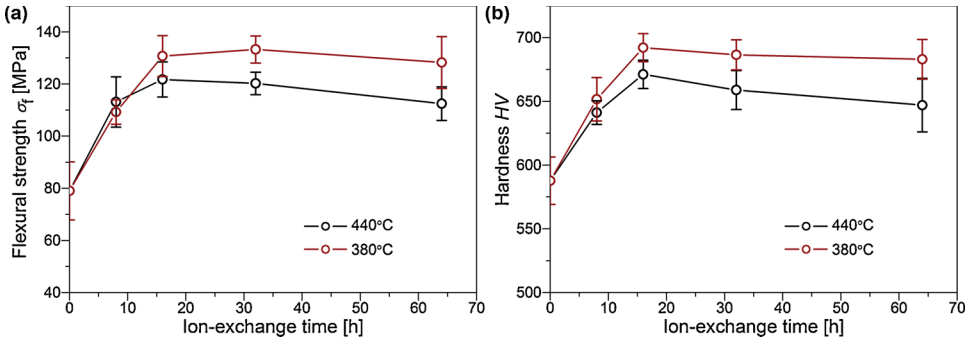

Fig. 3. Changes in flexural strength (a) and surface Vickers hardness (b) of the porcelain specimens with increasing ion-exchange time at 440 °C and 380 °C, respectively.

(0.11 ± 0.01 μm/10^4 cycles) was the lowest; however, there was no significant difference between the 380 °C/64 h group and the 440 °C/64 h group ($p > 0.05$).

The corresponding mean wear rates of the antagonist zirconia pins in running-in wear stage under different ion-exchange conditions (Fig. 5b) were lower than the value prior to ion-exchange ($p < 0.05$). There was no significant difference between the pre-in-exchange group and the 440 °C/16 h group. In the steady wear stage, the mean wear rates of the antagonist zirconia pins were significant lower than the value prior to ion-exchange ($p < 0.05$). There was no significant difference among the exchanged groups ($p > 0.05$). The wear rate of pins in the 380 °C/64 h group was the lowest.

3.5. Effect of ion-exchange condition on worn surface morphology

The worn surface morphology of the porcelain disks before ion-exchange and the porcelain disks which were processed at 440 °C/64 h and 380 °C/64 h are presented in Fig. 6. Running-in wear stage was represented by the results of 10×10^4 cycles. The boundary between running-in and steady wear stage was 18×10^4 cycles. Steady wear stage was represented by the results of 54×10^4 cycles. During the entire wear process, the disk surface before ion-exchange (Fig. 6a–c) was rougher and showed deeper, ploughed worn tracks than the disks that had been subjected to ion-exchange disks for the same number of test cycles. Porcelain disks in the 440 °C/64 h group (Fig. 6d–f) were rougher than those in the 380 °C/64 h group (Fig. 6g–i).

Fig. 7 shows the worn surface morphology of the antagonist zirconia pins before ion-exchange and after ion-exchange at 380 °C for 64 h. The pin surfaces in the 380 °C/64 h group were smoother than those without ion-exchange for the entire wear process. The grooves on the zirconia pins that were paired with porcelain disks that underwent ion-exchange at 380 °C for 64 h ion-exchanged disk were very shallow (Fig. 7d–f).

Worn surface roughness values (S_a) of untreated porcelain disks and those that had undergone ion-exchange at 440 °C for 64 h and 380 °C for 64 h are shown in Table 3. The S_a values of the 380 °C/64 h group at different cycles were significant lower ($p < 0.05$) than other treatment conditions at the same cycles. The antagonist zirconia pins surface S_a showed similar results (Table 3). The S_a values of antagonist zirconia pins used for wearing porcelain disks that underwent ion-exchange at 380 °C for 64 h were the lowest ($p < 0.05$) during the entire wear process.

4. Discussion

Wear performance of dental porcelains is an important attribute that is responsible for the longevity of metal-ceramic or all-ceramic restorations [35]. Ion-exchange is a well-known chemical strengthening strategy to fortify conventional monolithic glasses or dental glass-ceramics that contain alkali ions [11,17,20–25]. Previous investigations did not report the effects of ion-exchange on wear performance. Accordingly, the present study investigated the effects of $Na^+ \rightarrow K^+$ ion-exchange on the wear performance of feldspathic veneering porcelain. Because significant differences in wear performance were identified among the experimental groups, the null hypothesis that the ion-exchange has no effect on the wear performance of the feldspathic veneering porcelain has to be rejected.

Compared with the parent feldspathic glass, $Na^+ \rightarrow K^+$ ion-exchange occurs in the glassy matrix of feldspathic veneering porcelain under the condition. Because of the existence of leucite crystallites in the porcelain, they exert an impeding effect on the ion-exchange process [37] and reduce the depth of the ion-exchange layer under the same treatment conditions. And the depth of ion-exchange layer can be created deeper by prolonging the processing time at the same temperature (Fig. 1e, f). The relation between the depth and treatment time (Fig. 2) indicates that the depth of the ion-exchange layer created at higher temperature (440 °C) was deeper than created at lower temperature (380 °C) for the same processing time. Thus, it is necessary to prolong the processing time to obtain a thicker ion-exchange layer at lower

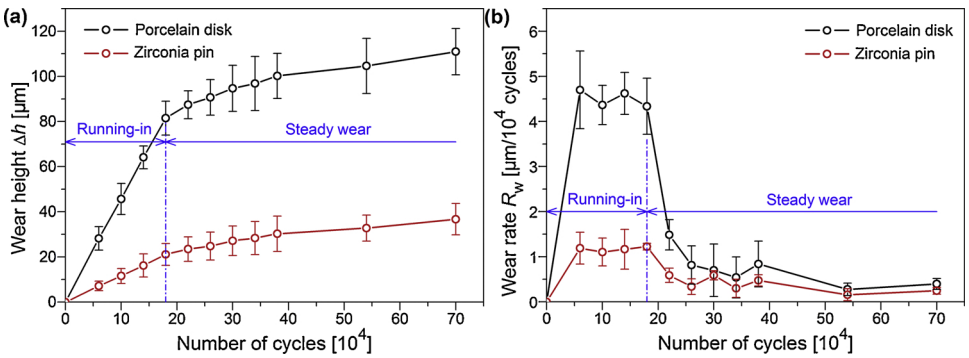

Fig. 4. (a) Wear curves of the porcelain disk before ion-exchange and the opposing zirconia pin over the cycle range up to 70×10^4 cycles. (b) The corresponding wear rate curves.

Table 1
Wear heights (Δh) and wear rates (R_w) in running-in and steady wear stages for the porcelain disks under different testing conditions.

State	Running-in stage (0–18 × 10^4 cycles)		Steady wear stage (18–70 × 10^4 cycles)	
	Δh (μm)	R_w (μm/10^4 cycles)	Δh (μm)	R_w (μm/10^4 cycles)
Before ion-exchange	81.47 ± 7.53 a	4.53 ± 0.42 a	29.48 ± 2.70 a	0.57 ± 0.05 a
440 °C/16 h	63.86 ± 5.50 b	3.55 ± 0.31 b	22.81 ± 2.39 b	0.44 ± 0.05 b
380 °C/16 h	33.23 ± 4.96 c	1.85 ± 0.28 c	22.64 ± 2.34 b	0.44 ± 0.05 b
440 °C/64 h	35.77 ± 6.02 c	1.99 ± 0.34 c	6.17 ± 1.98 c	0.12 ± 0.04 c
380 °C/64 h	16.06 ± 3.86 d	0.89 ± 0.21 d	5.65 ± 0.57 c	0.11 ± 0.01 c

Values are means ± standard deviations ($N = 6$).
For analysis of the wear rates for the porcelain disks in different states opposing zirconia pins, groups identified with the same letter are not significant different ($p > 0.05$).

processing temperature.

Surface strengthening of feldspathic veneering porcelain by $Na^+ \rightarrow K^+$ ion-exchange should be correlated with microstructural changes. When heated to temperature below T_g (380 or 440 °C), the molecular network within glassy matrix phase of the porcelain surface expands. This expansion is restricted by the interior glassy matrix phase, resulting in the generation of compressive stress within the surface layer [18] that increases the flexural strength of the porcelain after ion-exchange. The compressive stress built within the surface layer of an ion-exchanged glass may be progressively relaxed during ion-exchange by viscoelastic deformation due to the lowered surface T_g, and/or structural relaxation by reorganization of the glassy network [18,31]. Stress relaxation has been reported to be dependent on treatment time and temperature [41]. Accordingly, the flexural strength decreased with increasing ion-exchange time. Stress relaxation is significantly accelerated at ion-exchange higher temperature, as illustrated by the results in the present study; flexural strength decreased more at 440 °C. In addition, the maximum compressive stress generated after prolonged processing does not occur on the porcelain surface but occurs beneath the surface. This is because stress relaxation occurs most readily at the outermost layer, given the increased concentration of the invading K^+ ion [42].

Stress relaxation behaviour of the $Na^+ \rightarrow K^+$ ion-exchanged porcelain may be indirectly reflected by its surface hardness. Higher residual compressive stress in the surface results in higher deformation-resistance, hence higher hardness value [15]. The surface hardness of the ion-exchanged porcelain increased significantly after 16 h of ion-exchange, and then decreased with further increase in treatment time. The hardness was reduced more significantly at higher ion-exchange temperature. Based on these results, it may be deduced that compressive stress along the porcelain surface builds up rapidly during the first 16 h of ion-exchange, which is followed by gradual relaxation of the compressive stress with further processing.

The restoration surface finish plays an important role in the outcomes of clinical wear studies [43,44]. Hence, strengthening the porcelain surface may change the wear behaviour. The time-dependent wear behaviour of a ceramic restoration is a result of nonlinear wear relationships. The wear process of materials may be divided into a running-in wear stage, a steady wear stage and a severe wear stage [45]. Wear loss increases rapidly initially and the wear rate is higher during the running-in wear stage. Wear rate gradually decreases from as wear continues during the steady wear stage. In the present study, the time-dependent wear behaviour of the feldspathic veneering porcelain exhibited two stages during the 70 × 10^4 cycles of wear testing, with 18 × 10^4 cycles being the boundary between the running-in and the steady wear stages.

The antagonist pin was made of an yttria-stabilized zirconia ceramic with the aim of adequately evaluating the wear properties in a standard *in vitro* assessment; this method was widely used in many wear studies [35,46–49]. According to the wear curves of the zirconia pins that were paired with the porcelain disks prior to ion-exchange, the pins also exhibited two-stage wear. The time-dependent wear behaviour of the zirconia pins have the same tendency as the porcelain disks, with 18 × 10^4 cycles being the boundary between the two wear stages.

Based on these results, it appears that $Na^+ \rightarrow K^+$ ion-exchange can create a compressive stress layer that improves the hardness of the porcelain. Hardness is the ability to resist deformation and scratching. Hence, glass-ceramics with higher surface hardness should be more wear resistant [34]. The mean wear rates in the running-in and steady wear stages of the porcelain disks tested under different conditions demonstrate that the condition in which ion-exchange condition is performed significantly affects the wear performance of the treated porcelain. Such a phenomenon should be attributed to the compressive stress generated after ion-exchange. The compressive stress layer probably reduces the generation of deformation and scratch during the wear process. Accordingly, the worn surfaces of the ion-exchanged porcelain disks were smoother than those without ion-exchange when specimens were compared after the same number of cycles.

At a high ion-exchange temperature (e.g., 440 °C), a thicker ion-exchange layer can be created (see Fig. 2). With increasing processing time, the maximum compression stress is not at the surface because of structural relaxation in the outermost layer [42]. Stress relaxation can be significantly accelerated at higher temperatures [50]. This explains why the surface hardness of porcelain disks processed at 440 °C was lower than that of those processed at 380 °C, using the same processing time. Several studies have shown that wear of dental ceramics is aggravated with increase in surface roughness [51–53]. Hence the mean wear rate of porcelain disks processed at 380 °C for 64 h was the lowest

Table 2
Wear heights (Δh) and wear rates (R_w) in running-in and steady wear stages for the zirconia pins paired with the porcelain disks in different states.

State	Running-in stage (0–18 × 10^4 cycles)		Steady wear stage (18–70 × 10^4 cycles)	
	⊗h (μm)	R_w (μm/10^4 cycles)	Δh (μm)	R_w (μm/10^4 cycles)
Before ion-exchange	21.11 ± 4.83 a	1.17 ± 0.27 a	15.59 ± 2.13 a	0.30 ± 0.04 a
440 °C/16 h	16.11 ± 2.88 ab	0.90 ± 0.16 ab	12.04 ± 0.78 b	0.23 ± 0.02 b
380 °C/16 h	15.02 ± 2.80 b	0.83 ± 0.16 b	11.50 ± 0.88 b	0.22 ± 0.02 b
440 °C/64 h	13.43 ± 3.54 b	0.75 ± 0.20 b	10.88 ± 0.16 b	0.21 ± 0.002 b
380 °C/64 h	10.50 ± 2.39 b	0.58 ± 0.13 b	10.23 ± 0.26 b	0.20 ± 0.005 b

Values are means ± standard deviations ($N = 6$).

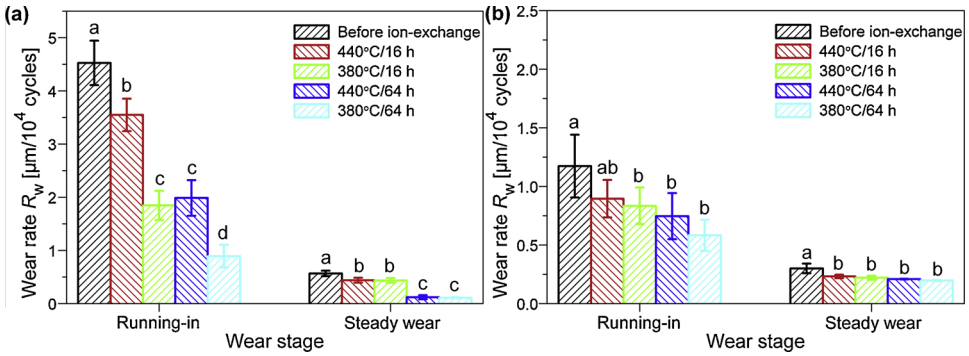

Fig. 5. (a) Average wear rates in the running-in and steady wear stages for the porcelain disks in different states. (b) The corresponding average wear rates of the antagonist zirconia pins. Groups with the same letter above each column are not significantly different ($p > 0.05$).

among the groups tested. With increase in wear cycles, all worn surfaces became smoother especially during the steady wear stage.

The depths of ion-exchange for the porcelain in different states and the corresponding total wear heights for 70×10^4 wear cycles are compared in Fig. 8. According to the figure, the corresponding wear heights were close to the depths of ion-exchange layer for 440 °C/16 h and 380 °C/16 h groups. It appears that the total wear height of 440 °C/16 h group was a little lager than the ion-exchange layer. For the longer exchange time groups, the exchange layers were much thicker than the total wear height. Although the ion-exchange layer of 440 °C/64 h group was thicker than that of 380 °C/64 h group, the total wear height of the later was significant less than that of the former. Therefore, the exchange layer after 380 °C/64 h ion-exchange processing can resist wear for more cycles. It appears that the ion-exchange condition of processing at 380 °C for 64 h is optimal for improving the wear resistance of the feldspathic veneer porcelain investigated in the present study.

Previous studies have demonstrated that the polished surface of dental materials can remarkably decrease the wear intensity of the opposing teeth [51,52]. In Table 3, the S_a values of the worn surface of the antagonist zirconia pins that run against porcelain surfaces processed at 380 °C for 64 h were significant lower than the other groups at the same number of wear cycles. This should be attributed to the generation of a thicker compressive stress layer on the porcelain surface

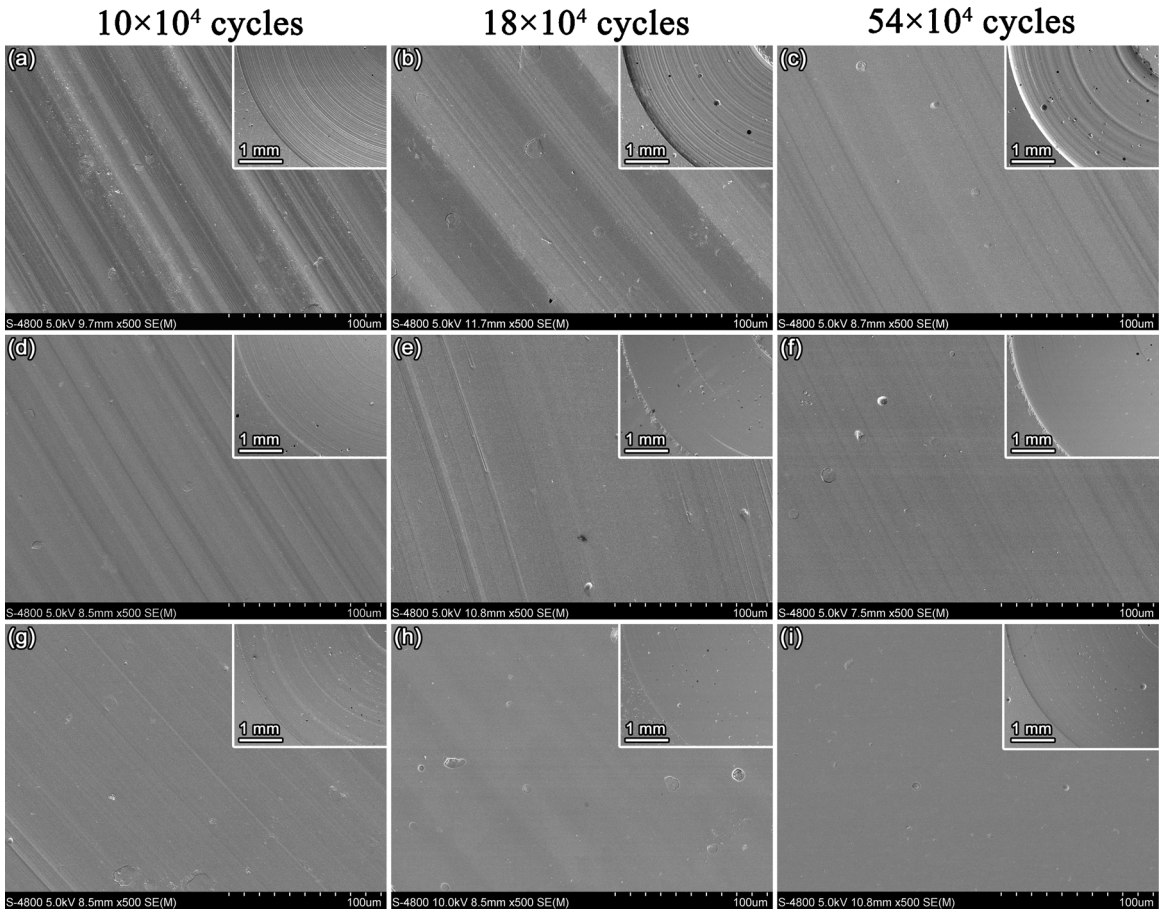

Fig. 6. Representative SEM images showing the worn surface morphology of the porcelain disks in different states opposing zirconia pins at different cycles: (a), (b) and (c) before ion-exchange; (d), (e) and (f) after 440 °C/64 h ion-exchange; (g), (h) and (i) after 380 °C/64 h ion-exchange. Insets show the corresponding partial wear tracks on the disks.

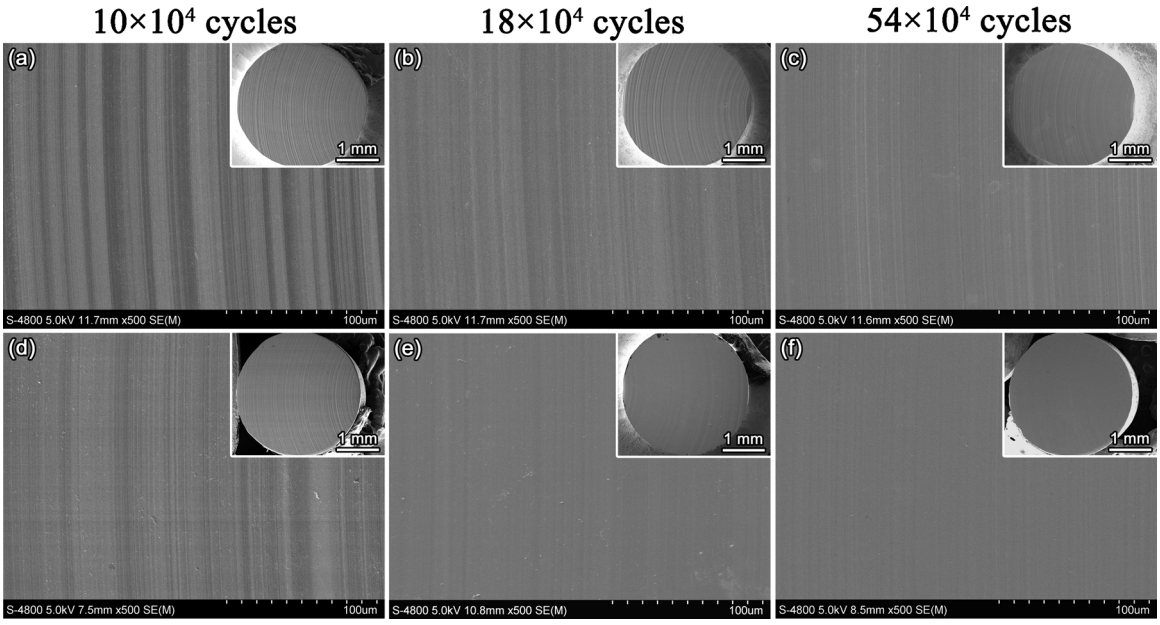

Fig. 7. Representative SEM images showing the worn surface morphology of the zirconia pins paired with the porcelain disks before and after 380 °C/64 h ion-exchange at different cycles: (a), (b) and (c) before ion-exchange; (d), (e) and (f) after 380 °C/64 h ion-exchange. Insets show the corresponding end faces of the pins.

Table 3
Worn surface roughness values (S_a) of the porcelain disks in different states and the corresponding zirconia pins at different cycles.

Wear cycle	S_a of disk (μm)			S_a of pin (μm)		
	Before ion-exchange	440 °C/64 h	380 °C/64 h	Before ion-exchange	440 °C/64 h	380 °C/64 h
0	0.37 ± 0.02	0.36 ± 0.02	0.36 ± 0.01	0.23 ± 0.03	0.23 ± 0.02	0.23 ± 0.02
10×10^4	1.47 ± 0.13 a	0.81 ± 0.06 b	0.53 ± 0.02 c	1.14 ± 0.03 A	0.56 ± 0.03 B	0.50 ± 0.04 C
18×10^4	1.09 ± 0.22 a	0.61 ± 0.04 b	0.39 ± 0.05 c	0.96 ± 0.03 A	0.51 ± 0.03 B	0.36 ± 0.02 C
54×10^4	0.69 ± 0.05 a	0.44 ± 0.03 b	0.32 ± 0.05 c	0.69 ± 0.05 A	0.40 ± 0.02 B	0.30 ± 0.01 C

Values are means ± standard deviations ($N = 6$).
For analysis of S_a of disk with different states at different cycles, groups identified with the same lower case letter are not significant different ($p > 0.05$).
For analysis of S_a of the corresponding zirconia pins at different cycles, groups identified with the same upper case letter are not significant different ($p > 0.05$).

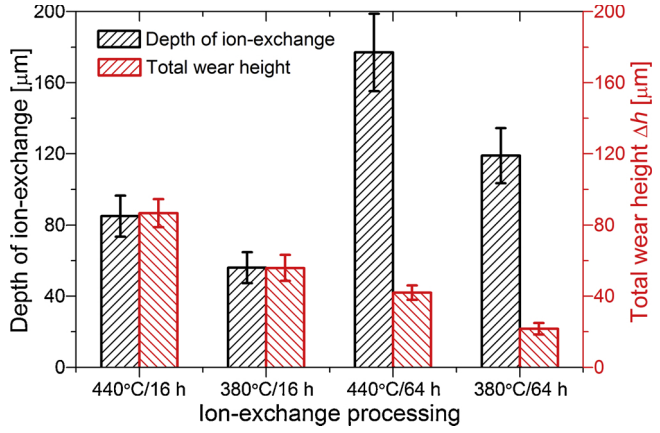

Fig. 8. Comparison of the depths of ion-exchange for the porcelain in different states and the corresponding total wear heights.

under this processing condition. This thicker exchange layer not only improves the wear performance of the feldspathic veneering porcelain, but also reduces the wear of the antagonist component.

5. Conclusions

Within the limits of the present study, it may be concluded that ion-exchange is an effective way to improve the wear performance of feldspathic veneering porcelain. The latter exhibits strong time-dependent wear behaviour, and ion-exchange decreases the wear rates during the running-in and steady wear stages. Creation of a thicker exchange layer with less stress relaxation, obtained by lower ion-exchange temperature and longer processing time, improves the wear performance of the feldspathic veneering porcelain especially during the running-in wear stage.

Declaration of Competing Interest

There is no conflict of interest.

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