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Poster Presentation

## Microstructural Investigation of ZrO<sub>2</sub> Modified Dental Porcelain Ceramics

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### Abstract

Dental porcelain ceramic is one of the promising dental materials, especially for restorative components due to increasing demands for better esthetics, biocompatibility and mechanical properties to withstand the conditions in the oval cavity [1-2]. However, apart from general problems of high degree of chemical heterogeneity and associated low mechanical properties, the stress induced by leucite phases commonly crystallized after high temperature treatment can create cracking in bulk porcelain ceramics [2,3]. To overcome these problems, several techniques have been introduced, such as employing ultrafine powders, carrying out appropriated firing conditions and using additives. Amongst all the issue reported so far, most attention has been concentrated on the use of alumina and titania particles whereas investigation on the nanocomposite structure induced via zirconia additive has not been widely carried out [2,3]. Thus, in this study, our attention has been paid on phase formation, densification and microstructural evolution of ZrO<sub>2</sub>-modified porcelain ceramics. The samples were prepared by vibro-milling commercially available dental porcelain powders with and without 20wt% of ZrO<sub>2</sub> for 30 min, slip-casted with polyvinyl alcohol into bar-shape samples and sintered in a vacuum furnace at 1040 °C for 5 min. The phase formation, densification and microstructures of the sintered products were characterized via X-ray diffraction (Fig. 1), Archimedes measurement, and scanning electron microscopy (Fig. 2). Grain size of the leucite phase was determined from SEM micrographs using an image analysis program (Image-Pro Plus v.6). In general, the results obtained in this work demonstrated that it was possible to obtain rather dense porcelain-based ceramics reinforced with nanocrystallite leucite particles by using zirconia additive approach. The strongest reflections in the majorities of diffraction peaks clearly distinguish the leucite phase and the zirconia additive as shown in Fig. 1. Representative microstructures for pure and ZrO<sub>2</sub> modified porcelain ceramics are given in Fig. 2. It can be seen that by comparison with 20wt% ZrO<sub>2</sub>-added samples, almost clean microstructure is generally found in pure dental porcelain ceramic. This microstructure is typical of a glassy matrix porcelain-based ceramics. For the case of zirconia additive, it is believed that leucite phase (spherical nanosized particles, Ø ~ 110-230 nm) with random agglomerations could be formed by the incongruent melting of potash feldspar. This is a mechanism by which a material melts to form a liquid accompanying with different crystalline phases.

### Acknowledgment

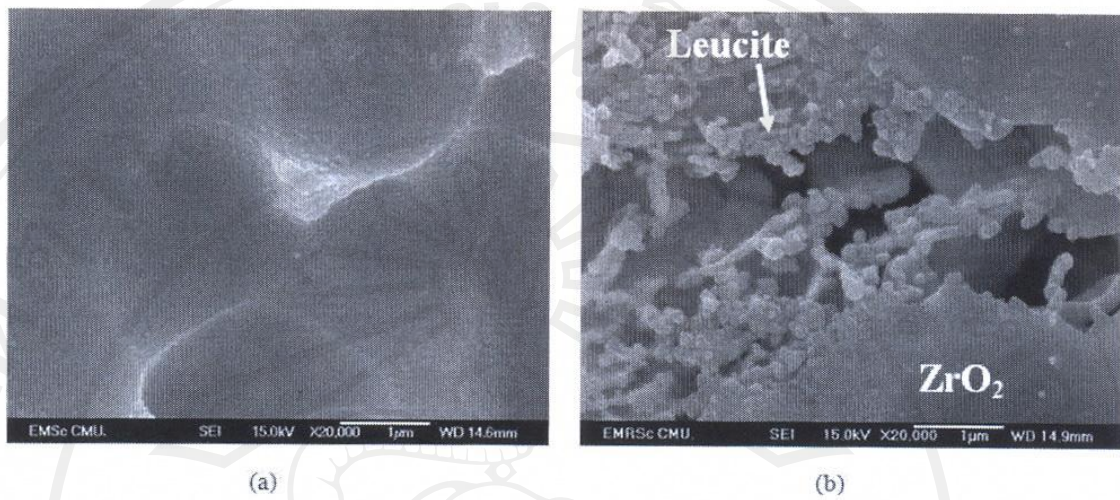
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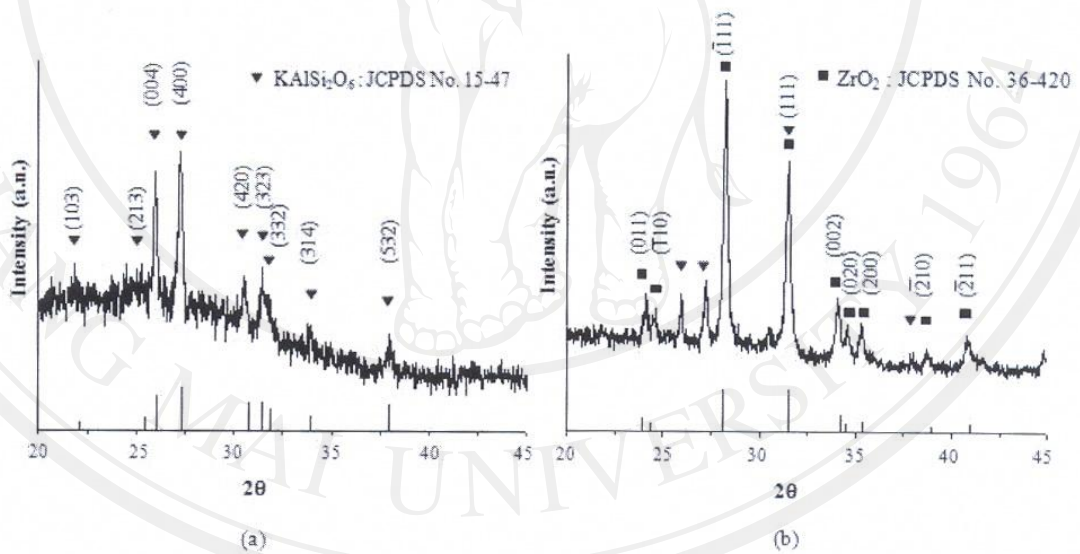
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**Figure 1** XRD patterns of sintered porcelain-based ceramics (a) without and (b) with 20 wt%  $ZrO_2$ -additive.



**Figure 2** SEM micrographs of sintered porcelain-based ceramics (a) without and (b) with 20 wt%  $ZrO_2$ -additive.



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 Contributed Paper

## Effects of Tempering Time on Phase Formation and Microstructural Evolution of Zirconia Modified-Dental Porcelain Ceramics

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### ABSTRACT

Dental porcelain ceramics reinforced with leucite nanocrystallines were successfully fabricated by using the two-step sintering technique together with some  $ZrO_2$  additive. Influence of firing conditions on leucite formation, densification and microstructural development of the sintered samples was investigated. It has been found that the choice of tempering time is one of the key factors controlling leucite crystallization on the zirconia surface in dental porcelain ceramics. The potentiality of a two-step sintering technique and Zr additive as a simple ceramic fabrication method to obtain highly dense dental porcelain ceramic-nanocomposites was demonstrated. After a two-step sintering, it was found that the microstructure was mainly composed of a dispersed dendritic leucite crystalline and a glassy matrix.

**Keywords:** composite; dental porcelain ceramics; leucite; tempering time.

### 1. INTRODUCTION

Porcelain-based ceramics have been widely investigated for the use of restorative materials in dental applications because of their esthetics, low thermal conductivity and excellent biocompatibility. However, similar to other glass-ceramics, a major impediment to dentistry of porcelains is their brittleness, which often restricts their utilization in fields where high strength and toughness are required [1-3]. Some attempts have been made to solve these issues by reinforcing the glassy matrix with some high strength crystalline phases such as alumina ( $Al_2O_3$ ), zirconia ( $ZrO_2$ ) or leucite ( $KAlSi_2O_6$ ) [2-5]. A significant milestone in this

respect was established in the development of dental ceramics containing leucite crystalline phase because their excellent thermal compatibility when bonding to metals and also a reinforcing agent in all ceramic restorations [6-9]. It has been reported that leucite glass-ceramics can be fabricated by controlled crystallization of a glass via leucite additive, heat treatment and etc. [10-12]. In general, commercial dental porcelain ceramics contain some amount of leucite crystals sized  $\sim 5$ - $10 \mu m$  causing extensive microcracking around these non-uniformed leucite crystals [10]. To overcome these problems, several efforts have



been made to tailor the leucite morphology, volume fraction and distribution which is thought to affect the mechanical properties of these porcelain ceramics via heat treatment [11,12] or heat pressing [7].

In recent years, there is a great deal of interest in the fabrication and characterization of ceramic-nanocomposites due to their excellent mechanical properties [2,3]. In connection with this, an effective method called "two-step sintering" has been proposed by Chen & Wang [13] for the preparation of  $Y_2O_3$  ceramics with nano-sized grains. In this method, the first step is conducted by firing materials with constant heating rate in order to achieve an initial high density, and then a second firing step is held at a lower temperature (i.e. tempering) by isothermal sintering aiming to increase the density without obvious grain growth [14]. So far there is only work of Cattell et al. [12] who successfully produced porcelain-based ceramics with uniform distribution of fine leucite crystals in glassy matrix by tailoring the tempering time via the two-step sintering method. However they could not reach the nanocomposites structure. Although the study on the formation of leucite crystalline in porcelains has been reported by several researchers [9-12], attentions paid on Zr-modified porcelain-based ceramics are very few. Moreover, to our knowledge a detail study considering the potential of two-step sintering for the production of nanocomposites in dental porcelain ceramics has not yet been widely reported. Thus, the aim of this work was to explore the potential of the two-step sintering technique in the fabrication of 20 wt%  $ZrO_2$ -modified porcelain ceramic-nanocomposites. In addition, the effects of tempering time on phase formation, densification and microstructure evolution in these porcelain ceramics were investigated. This would extend an understanding on the processing-composition-microstructure

relationships in dental porcelain ceramics.

## 2. MATERIALS AND METHODS

The as-received commercial dental porcelain powders are used as starting materials (Vita-VMK95, Vita Zahnfabrik, Bad Säckingen, Germany), which had particle size range of  $\sim 1.5$ - $2.5 \mu m$ . Porcelain powders were mixed with 20 wt% of zirconia powders (Sigma-Aldrich, purity > 99%) by using a rapid vibro-milling for 30 min [2,3]. The method of mixing, drying, grinding and sieving of the products were similar to those employed in the preparation of the alumina-modified dental porcelain powders as described previously [2,3]. Green samples were obtained by mixing powders with polyvinyl alcohol binder (PVA) via a slip-casting technique as recommended by the manufacturer [15], and then poured into a standard stainless steel mould with a normal-sized cavity of  $30 \text{ mm} \times 6 \text{ mm} \times 2 \text{ mm}$ , reproducing the desired dimensions and shapes [2,3]. After moulding, the ceramics were fabricated by employing different firing schemes with heating rate of  $25^\circ\text{C}/\text{min}$  in a vacuum furnace (Multimat Touch & Press, Germany), as detail demonstrated in Table 1. During heating, the temperature was maintained at  $500^\circ\text{C}$  for 1 h to burn out the PVA binder. The dental porcelain was sintered at  $980^\circ\text{C}$  for 5 min as recommended by the manufacturer. Some samples of dental porcelain ceramics modified with 20 wt%  $ZrO_2$  additive, i.e. the control group (CG), were singly sintered at  $1,040^\circ\text{C}$  for 5 min [4]. Another group of samples (coded G1-G8) were sintered by employing the two-step sintering process and then quenched into room temperature. Densities of the final sintered products were determined by using the Archimedes principle. Phase formation and microstructure were examined by room temperature X-ray diffraction analysis (XRD; X'pert MPD,



Philips Corp, Japan) using Ni-filtered Cu K $\alpha$  radiation and field emission scanning electron microscopy (SEM; JSM-840A 6335 F, Jeol, Japan), respectively. The mean crystallite size was determined using the diffraction peak (400) of the leucite pattern by using Scherrer equation [16]. Lattice parameters of the leucite phase were determined from the  $d$  spacings for the (400) and (004) peaks for the tetragonal phase [11,12]. The grain size and morphologies of leucite phase in the sintered samples were determined from SEM micrographs. The chemical composition of the phase formed was also elucidated by an energy dispersive X-ray (EDX) analyzer with an ultra-thin window. EDX spectra were quantified with the virtual standard peaks supplied with the Oxford Instruments eXL software.

### 3. RESULTS AND DISCUSSION

The densification of the Zr-modified porcelain ceramics at various firing schemes is investigated. The change in density versus the tempering time (0-90 min) is shown in Table 1 (G5-G8). In the tempering time range of 0-30 min, the density increases with increasing tempering time. Further increase in the tempering time to 90 min leads to the decrease of the density. This feature creates a maximum density value of about 2.76 g/cm<sup>3</sup> which is comparable to the values reported by Pisitanusorn *et al.* (~2.74 g/cm<sup>3</sup> [3]) where whisker-type TiO<sub>2</sub> was used as additive. The increasing density with rising tempering time up to 30 min may be explained by the enhanced densification related to the effect of ZrO<sub>2</sub> and the more reactive two-step sintering used [4,5,14]. Further increase in the tempering time causes a decrease in density values. This may be attributed to the suppression of atomic diffusion at probably too long tempering times resulting in an incomplete densification [16]. Similar behavior is also reported by Wang *et al.* [14] for fine alumina-zirconia ceramics.

As is well known, the difference in thermal expansion between the tetragonal leucite crystals and the glassy matrix also provides tangential compressive stresses around the crystals that are thought responsible for significant strengthening in dental porcelain [11,12]. The crystallization of a high expansion tetragonal leucite phase into a base glass is advantageous to increase its expansion coefficient to a level sufficient to allow efficient bonding to metals [17]. More recently, leucite has been used in all-ceramic materials, not for thermal compatibility, but as a reinforcing material for all-ceramic restorations [11,12].

To study the crystalline phase development with different firing schemes in each porcelain-based samples, they were heat treated at various conditions (Table 1), followed by phase analysis using XRD technique. The X-ray diffraction patterns of both unfired and single (one-step) sintered dental porcelain ceramics in this study are given in Figure 1(a and b), indicating a background of amorphous glassy phase and evidence of the formation of only leucite crystalline phase, which could be matched with JCPDS file no. 15-47, in agreement with other work [17,18]. To a first approximation, this crystalline phase has a tetragonal leucite-type structure in space group  $I4_1/a$  (no. 88) with cell parameters  $a = 1,306$  pm and  $c = 1,375$  pm [19].

As demonstrated in Figure 1(c-j), it can be seen that all samples showed almost identical XRD patterns. The strongest reflection in the majority of XRD traces derived from all 20 wt% ZrO<sub>2</sub>-modified porcelain groups indicated a combination of monoclinic-zirconia which could be match with JCPDS file no. 36-420 [20] and tetragonal-leucite phase. In addition, it should be noted that XRD peaks of leucite (323) and zirconia ( $\bar{1}11$ ) at  $2\theta \sim 28.5^\circ$  are superimposed. In this study, no phase transformation of leucite can be detected at 1,040°C and the XRD patterns

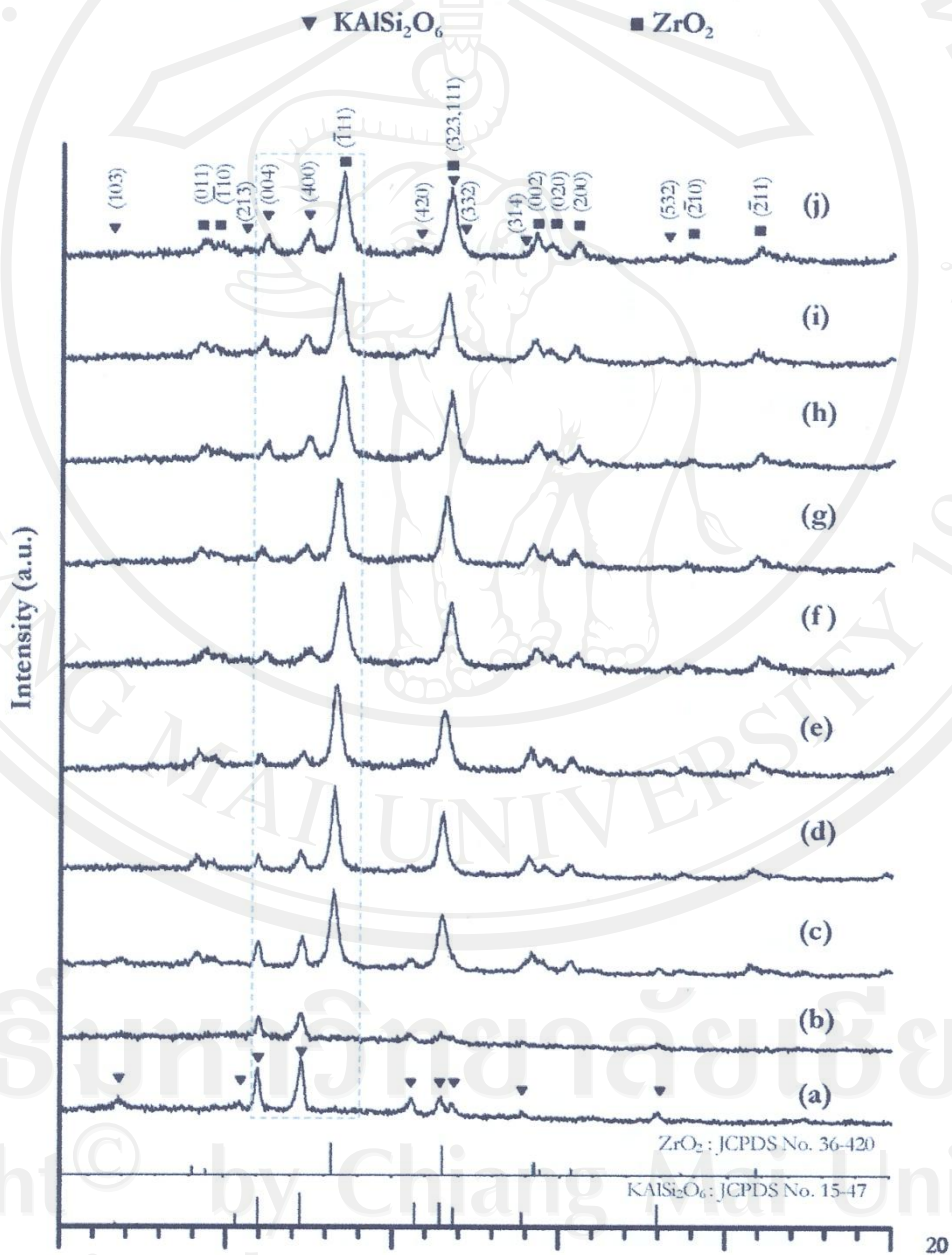
Table 1. The firing schemes employed for the production of samples and their physical properties.

Materials	Firing condition			Density (g/dm <sup>3</sup> )	Leucite content (wt%)	Leucite crystallite size (± 2 nm)	Leucite particle size range (± 30 nm)
	T <sub>1</sub> /Dwell time (°C/min)	T <sub>2</sub> /Dwell time (°C/min)	Quenched				
Dental porcelain	980/5	-	-	2.43	-	14.31	200-500
CG	1,040/5	-	-	2.73	26.51	12.53	80-220
G1	1,040/0	-	✓	2.72	23.00	9.40	30-93
G2	1,040/1	-	✓	2.71	23.80	9.86	37-110
G3	1,040/3	-	✓	2.73	24.47	10.04	40-130
G4	1,040/5	-	✓	2.72	25.90	10.83	60-130
G5	1,040/5	940/0	✓	2.73	25.81	11.93	60-170
G6	1,040/5	940/30	✓	2.76	28.40	14.32	60-170
G7	1,040/5	940/60	✓	2.71	27.63	16.11	70-180
G8	1,040/5	940/90	✓	2.71	30.00	19.90	70-190



of the samples subjected to the two-step sintering process for various tempering time from 0 to 90 min did not reveal the formation of any additional crystalline phases, in agree

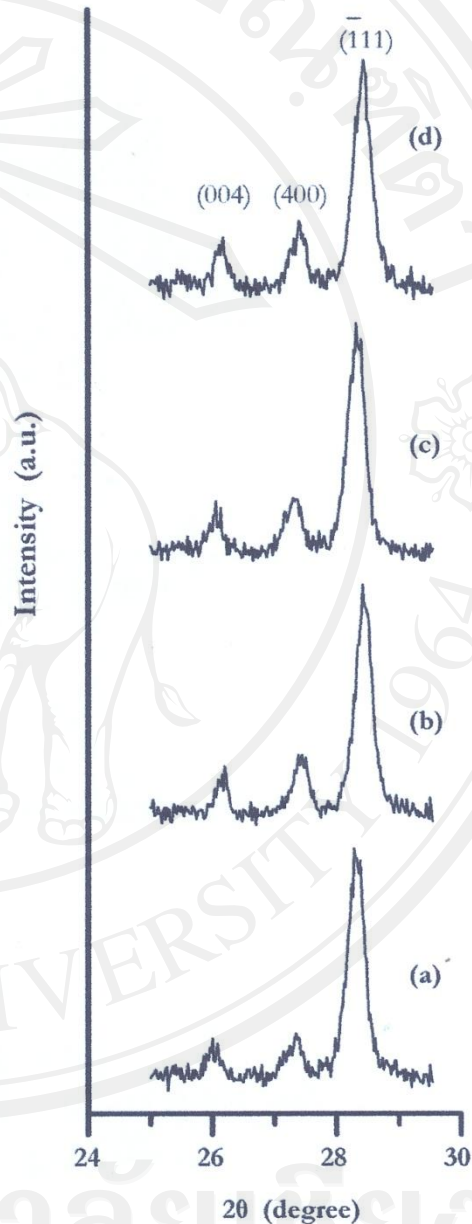
with previous studies [11,12]. This is probably indicating the effectiveness of Zr as the leucite stabilizer, in analogous with those found for other similar system [17].



**Figure 1.** X-ray diffraction patterns of non-sintered (a) and sintered (b) dental ceramics, non-sintered (c) and sintered (d) CG, two-step sintered (e) G3, (f) G4, (g) G5, (h) G6, (i) G7 and (j) G8 samples.

In order to evaluate the relative amounts of tetragonal leucite phase in each group, the leucite peak at the (004) and (400) reflections and the zirconia peak at the  $(\bar{1}11)$  reflection were the peaks of interest (Figure 2). As suggested by Ong *et al.* [18], powder X-ray diffraction method can be used to approximate the amount of leucite phase in porcelain ceramics. From Table 1, it can be seen that some relationship was found between the tempering time at 940°C and the concentration of leucite phase in the samples (G5-G8). In this study, the amount of leucite phase in the ZrO<sub>2</sub>-doped porcelain specimens subjected to the two-step sintering was found to slightly increase as a function of tempering time, consistent with those reported by Cattell *et al.* [11,12] for porcelain-based samples. It has been observed that with increasing tempering time, some diffraction lines gradually sharpen e.g. (004) and (400) peaks, an indication of a continuous increase in crystalline size and of the reduction of lattice strain. These observations point out that the prolonged tempering treatment affects the leucite crystallite size.

Additionally, the crystallite size of leucite phase was estimated from these XRD patterns as also given in Table 1. The calculated crystallite size value was also found to increase with increasing tempering time. Though, the relative intensities of the Bragg peaks exhibit independent of tempering time, it is well documented that, as Scherer's analysis provides only a measurement of the extension of the coherently diffracting domains, the crystallite sizes determined by this method can be significantly underestimated [16]. In addition to strain, factors such as defects, homogeneity of materials, the complex nature of the background due to amorphous matrix, processing variables and instrument effect can attribute to peak shape, making it almost impossible to extract a reliable crystallite size

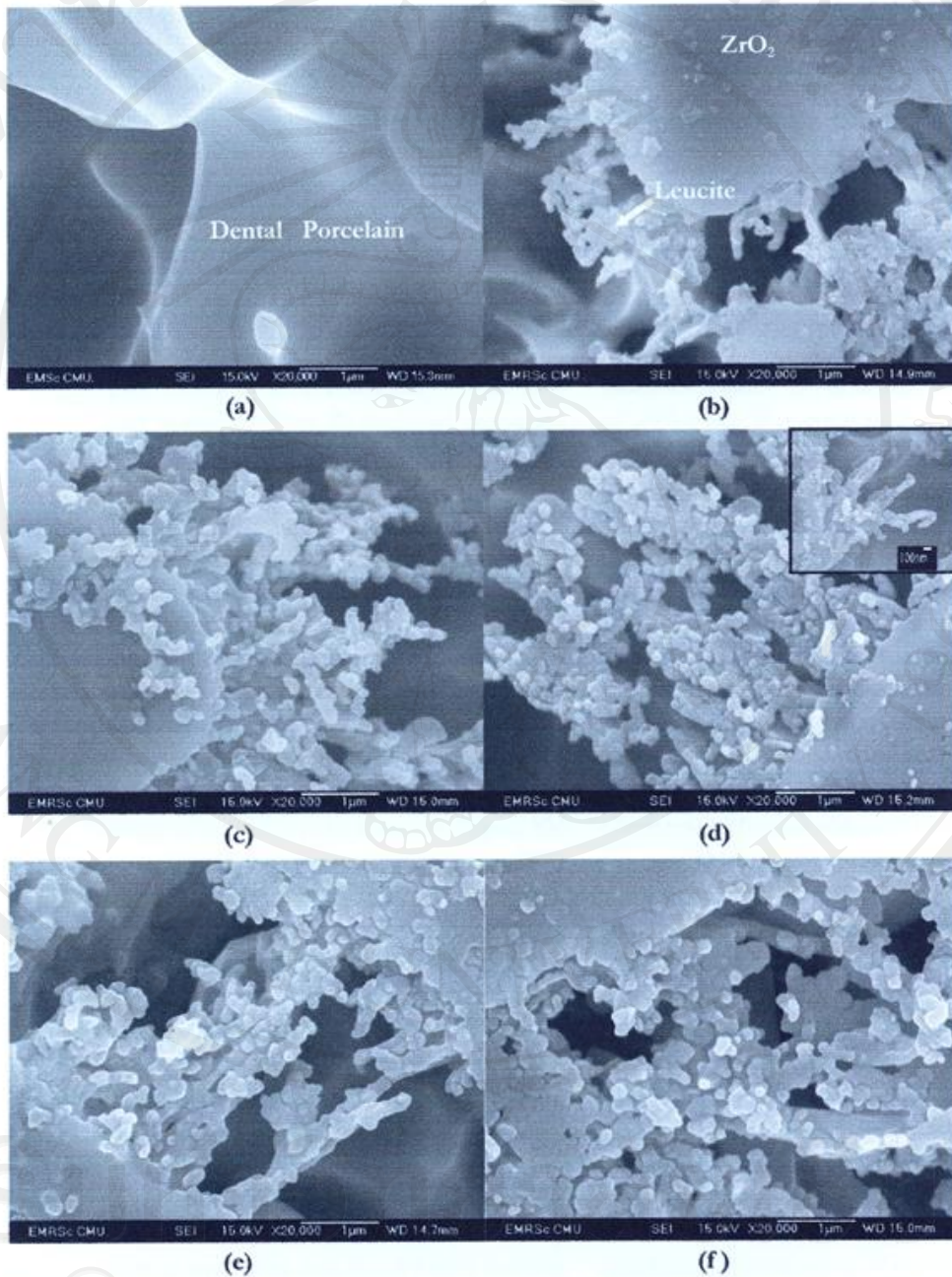


**Figure 2.** Enlarged X-ray diffraction patterns of (a) G5, (b) G6, (c) G7 and (d) G8 samples.

solely from XRD [16]. In this connection, SEM was also employed for grain size measurement (Table 1 and Figure 3).

Microstructural features of dental porcelain ceramics sintered at 1,040°C for 5





**Figure 3.** SEM micrographs of (a) dental porcelain, (b) CG, (c) G5, (d) G6, (e) G7 and (f) G8 samples.

min (CG) are shown in Fig. 3(a). It is seen that a smooth surface of typical porcelain glass-ceramics is observed, consistent with those

reported earlier [11,21,22]. As shown in Figure 3(b-f) for the case of all 20wt% Zr modified porcelain ceramics, it can be



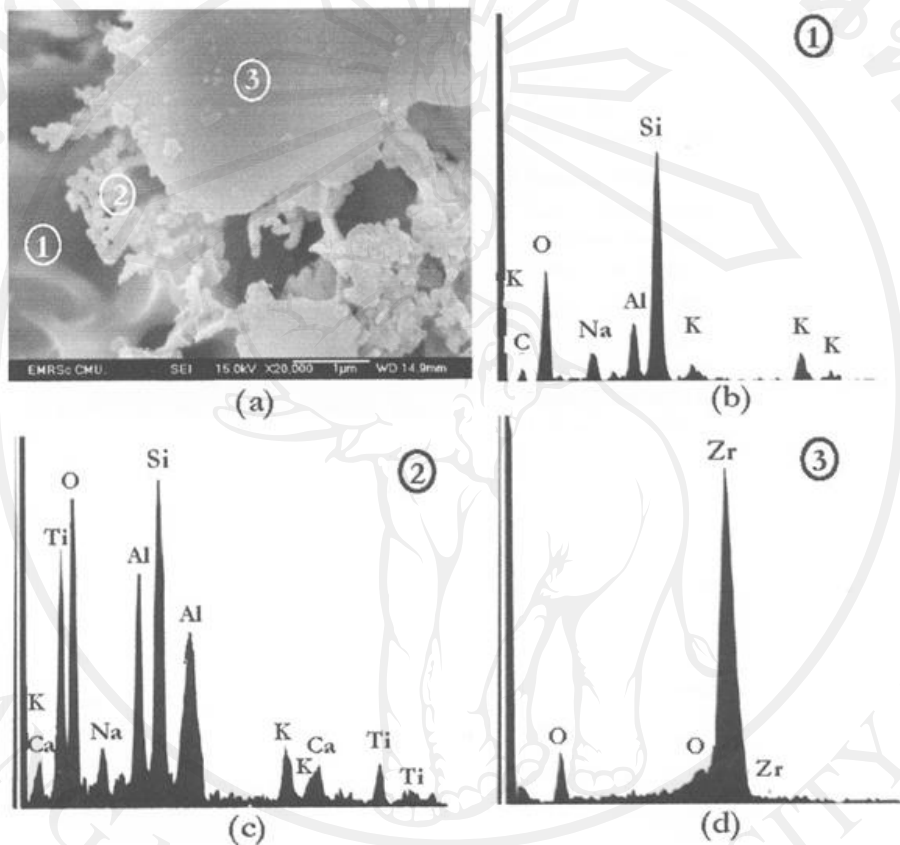
seen that some leucite particle sizes in the range ~100-300 nm (closer observation of Figure 3(d) as inserted) were initiated from the surface of zirconia grains. It is interesting to note that dendritic leucite morphology previously reported by several researchers [23,24] are also found here. These observations may be attributed to the influence of  $ZrO_2$  additives as nucleating agents for leucite crystallization behavior, similar to those found in other similar glass-ceramic systems [24]. The microstructural evolution for the two-step sintering of the Zr-modified porcelains as a function of tempering time was also revealed, as illustrated in the SEM micrographs (Figure 3(c-f)). All sintered samples showed two (or more) distinct phase structures with a glassy matrix phase reinforcing crystalline phase dispersed in the glassy matrix. In general, they have a very fine microstructure with agglomerates. SEM-EDX analysis of the dental porcelain ceramics marked as “(1)” reveals the strong presence of silica and oxygen indicating the composition of glass matrix (Figure 4(b)). As shown in Figure 4(a) and (c), EDX spectra obtained from area “(2)” confirms the existence of all key elements related to the composition of leucite ( $KAlSi_2O_6$ ) [19]. Whilst EDX analysis of the large grain marked as “3” show only zirconia rich phase together with spectra of oxygen, indicating the existence of  $ZrO_2$  additive. The results of SEM-EDX measurement supported the XRD observation discussed earlier (Figures 1 and 2).

Figure 3(c) is a SEM micrograph of the samples experienced the two-step sintering with no tempering time (i.e. at 0 min) showing leucite particulates which vary between 100 and 300 nm in size and are clustered together along the glassy grains. On the basis of this, the governing mechanism for the appearance of leucite phase in the glassy matrix is surface crystallization [11,25]. Furthermore, there is

no evidence of crack formation in the matrix or within the leucite crystals. A similar microstructure was evident for the tempering times 30, 60 and 90 min samples, but with increased leucite growth and signs of Zr growth, as shown in Figure 3(d-f). In both G7 and G8 samples, the additives are observable in two forms: the agglomerates as also found in G4 and G5, and the nanocomposite formations in reticulate sheets (~100 nm thickness). Such a difference in morphology may be described on the basis of difference in crystallization and growth mechanisms. The early stages of bulk leucite growth have been observed as dendrites growing in their preferred crystallographic directions. A diffusion controlled growth process that evolved at the smooth atomic-scale faceted crystal-glass interface was suggested. With longer tempering times applied, a significant change in dendrite shape due to the growth of secondary and tertiary fibrils and their ripening resulted in a highly organized tetragonal leucite structure, consistent with those observed in other similar systems [2,25,26]. Signs of leucite particle coalescence were also visible in all Zr-modified porcelain samples which may have been driven by a reduction in interfacial energy when larger particles grow at the expense of smaller ones (Ostwald ripening) [27]. In this study, it is believed that the prolonged tempering times may have allowed sufficient time for small particles below a critical size to dissolve and feed larger particles via a diffusion down the concentration gradient.

Thus, two aspects of this study are significant: (i) production of dental porcelain-based ceramic-micro/nanocomposites can be achieved by employing a combination of the two-step sintering and the Zr additive techniques and (ii) the tempering time is one of the key factors controlling leucite crystallization behavior. For better under-





**Figure 4.** Representative (a) SEM micrograph of CG samples and their corresponding EDX analysis, indicating the chemical compositions of (b) glassy matrix, (c) leucite and (d)  $ZrO_2$  phases, respectively.

standing and verifying the attractiveness of this nanocomposite further, a systematic study on the effect of variety content of zirconia on microstructure and amount of leucite-nanocomposites is required. Moreover, according to Chen and Wang [13], the success of the two-step sintering strongly depends on the choices of both firing temperatures ( $T_1$  and  $T_2$ ). Thus, further work with attention paid on the influences of the designed two-step sintering schemes on phase formation, microstructure and properties of these nanocomposites should also be very helpful.

#### 4. CONCLUSIONS

It has been shown that the processing method adopted for producing dental porcelain-based ceramics greatly influences the composition and microstructure of the products. Using commercially available dental porcelain powders as the starting materials, it has been demonstrated that the two-step sintering technique together with 20wt%  $ZrO_2$  additive has considerable potential for the production of leucite/porcelain ceramic-nanocomposites. It was also found that the tempering time is a key factor to controlling

dendritic morphology of leucite crystallization behavior and microstructural arrangement in Zr-modified dental porcelain ceramics.

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