

The logo of Chiang Mai University is a large, faint watermark in the background. It features a central elephant, a traditional Thai symbol, surrounded by a circular border containing the university's name in Thai and English. The Thai text at the top reads 'มหาวิทยาลัยเชียงใหม่' and the English text at the bottom reads 'CHIANG MAI UNIVERSITY 1964'.

VITA

Name Miss Nolapan Vudhivanich

Date of birth 5 September 1983

Education Doctor of Dental Surgery, Faculty of Dentistry,
Chiang Mai University, (2008).

Research Output:

International Conferences:

Effect of Zirconia Additives on Leucite Crystallization in Dental Porcelain Ceramics

“Proceedings of 38th Congress on Science and Technology of Thailand, Chiang Mai (2012)”

N. Vudhivanich, A. Pisitanusorn, S. Ananta

ลิขสิทธิ์มหาวิทยาลัยเชียงใหม่

Copyright© by Chiang Mai University
All rights reserved



EFFECT OF ZIRCONIA ADDITIVES ON LEUCITE CRYSTALLIZATION IN DENTAL PORCELAIN CERAMICS

Nolapan Vudhivanich,^{1,*} Attavit Pisitanusorn,¹ Supon Ananta²

¹ Department of Prosthodontics, Faculty of Dentistry, Chiang Mai University, Chiang Mai 50200, Thailand

² Department of Physics and Materials Science, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand

*e-mail: nolapan@yahoo.com

Abstract: In this work, influence of ZrO₂ additives on leucite formation in dental porcelain ceramics derived from a two-step sintering technique were investigated. It was found that the amount of ZrO₂ additive is one of the key factors controlling leucite crystallization on the zirconia surface in dental porcelain ceramics. The amount and size of leucite particles were found to decrease with increasing amount of zirconia additives.

Introduction: Dental porcelain ceramics are widely used in dentistry due to their excellent esthetics and biocompatibility. However their useful mechanical properties are limited by brittleness. Some attempts have been made to solve these issues by reinforcing the glassy matrix with some high strength crystalline phases such as alumina (Al₂O₃), zirconia (ZrO₂) or leucite (KAlSi₂O₆).¹ In general, commercial dental porcelain ceramics contain some amount of leucite crystals sized ~ 5-10 μm causing extensive microcracking around these non-uniformed leucite crystals.² To overcome this problem, the two-step sintering technique together with 20 wt% ZrO₂ additive was proposed earlier by our group,³ for the production of leucite nanoparticles. However, so far, no information on the effects of zirconia content on both amount and size of leucite crystals in dental porcelain ceramics is available in literature. Thus, in this work, leucite crystallization in the two-step sintered dental porcelain ceramics with various amount of ZrO₂ additives was investigated and discussed.

Methodology: Porcelain powders were mixed with various amount of ZrO₂ additives ranging from 15-25wt% (no ZrO₂ additive as a control group). Green samples were obtained by mixing powders with polyvinyl alcohol binder (PVA) via a slip-casting technique, and then poured into a standard stainless steel mould with a normal-sized cavity of 30 mm x 6 mm x 2 mm, reproducing the desired dimensions and shapes.¹ After moulding, the Zr-modified porcelains were fabricated by employing the two-step sintering process, sintered at 1040°C for 5 min then tempering at 940°C for 90 min, with heating rate of 25°C/min in a vacuum furnace³ and then quenched into room temperature, as detail demonstrated in Table 1. The control group was sintered at 980°C for 5 min as also recommended by the manufacturer.³ Information of leucite crystallization were extracted from results obtained via a room temperature X-ray diffraction (XRD; X'pert MPD, Philips Corp, Japan) technique operated by using Ni-filtered Cu K_α radiation. Morphological evaluation of leucite phase in the sintered samples were carefully examined by using field emission scanning electron microscopy (SEM; JSM-840A 6335 F, Jeol, Japan).

Results, Discussion and Conclusion: 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 (111) reflection were the peaks of interest (Fig. 1). As suggested earlier by Ong et al.,⁴ powder X-ray diffraction method can be used to approximate the amount of leucite crystalline phase in porcelain ceramics. From Table 1, it can be seen that some relationship was found between the amount of ZrO₂ additive and the concentration of leucite phase crystallized in the two-step sintered samples. In general, it has been observed that with



increasing amount of ZrO_2 additive, some major diffraction lines e.g. (004) and (400) peaks indicate a continuous decrease in amount of leucite content (Fig. 2), in agreement with earlier work reported by Sanitnapapong et al.³ Thus, it is believed that leucite crystallization in porcelain ceramics can be effectively suppressed by addition of ZrO_2 .

Table 1. The firing schemes employed for the production of dental porcelains with various amount of ZrO_2 additive and the resulting leucite crystallization

ZrO ₂ additive (wt%)	Firing condition			Leucite formation	
	Sintering temperature /Dwell time (°C/min)	Tempering temperature /Dwell time (°C/min)	Quenched	Content (wt%)	Size/Mean (± 30 nm)
0	980/5	-	-	39.86	180-500/256
15	1040/5	940/90	√	37.06	72-211/162
20	1040/5	940/90	√	32.41	61-212/164
25	1040/5	940/90	√	26.44	48-214/147
20 ³	1040/5	940/90	√	30.00	70-190

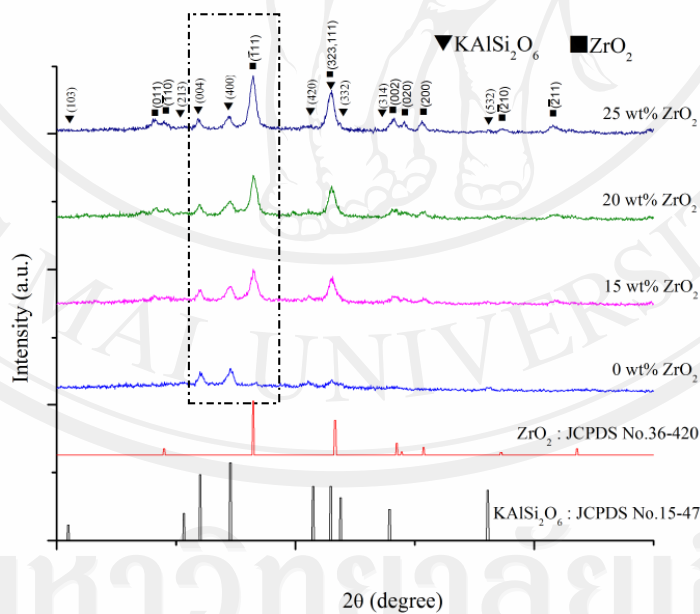


Figure 1. X-ray diffraction patterns of dental porcelain doped with various amount of ZrO_2 additive

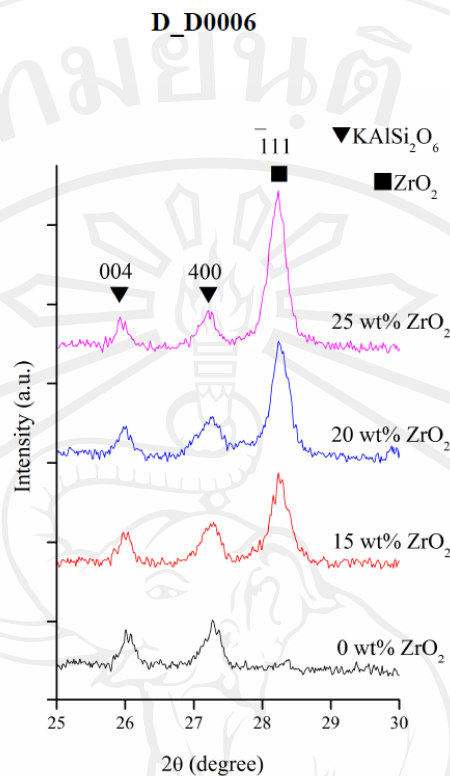


Figure 2. Enlarged X-ray diffraction patterns of dashed box in Fig. 1

From Fig. 3, leucite particle sizes, as data also given in Table 1, were found to decrease with increasing amount of ZrO_2 additive. According to earlier work of Apel et al.,⁵ they found that ZrO_2 additive can effectively suppress crystal growth of lithium disilicate in glass-ceramics. Similarly, in this study, it is believed that the ZrO_2 additives may inhibit the growth of small leucite particles below a critical size to dissolve and feed larger particles via a diffusion down the concentration gradient. Moreover, it is seen that all samples showed two (or more) distinct phase structures with a glassy matrix phase reinforcing crystalline phase dispersed in the glassy matrix. In general, the leucite particles are clustered together along the glassy grains. The governing mechanism for the appearance of leucite phase in the glassy matrix observed in this work should be related with the surface crystallization.⁶ Furthermore, there is no evidence of crack formation in the matrix or within the leucite crystals. Microstructural features of dental porcelain ceramics sintered at $980^\circ C$ for 5 min (control group) are shown in Fig. 3(A), it is seen that a smooth surface of typical porcelain glass ceramics and some leucite particle size range ~ 180 - 500 nm are observed, consistent with those reported earlier.⁶ As shown in Fig. 3(B-D) for the case of 15-25wt% Zr modified porcelain ceramics, it can be seen that large particle of ZrO_2 (~ 5 μm) and some leucite particles (~ 70 - 200 nm) were initiated from the surface of zirconia grains. These observations could be attributed to the influence of ZrO_2 additives acting as nucleating agents for leucite crystallization behavior, similar to those found in other similar glass-ceramic systems.^{7,8} However, it is of interest to point out that by increasing the amount of ZrO_2 additive further up to 25 wt%, both amount and size of leucite particles in the sintered porcelain ceramics was found to decrease, consistent with work of Apel.⁵ It is possible that the nucleating agent capability of ZrO_2 for the crystallization of leucite is suppressed above a critical amount of ZrO_2 additive. Thus, This work demonstrated that the ZrO_2 additive especially its content is one of the key factor controlling leucite crystallization behavior in dental porcelain ceramics.



Future works with attention paid on the extending investigation of the amount of ZrO_2 additives together with the powerful electron microscopy techniques such as TEM and HRTEM should be considered for better analysis and understanding of these materials.

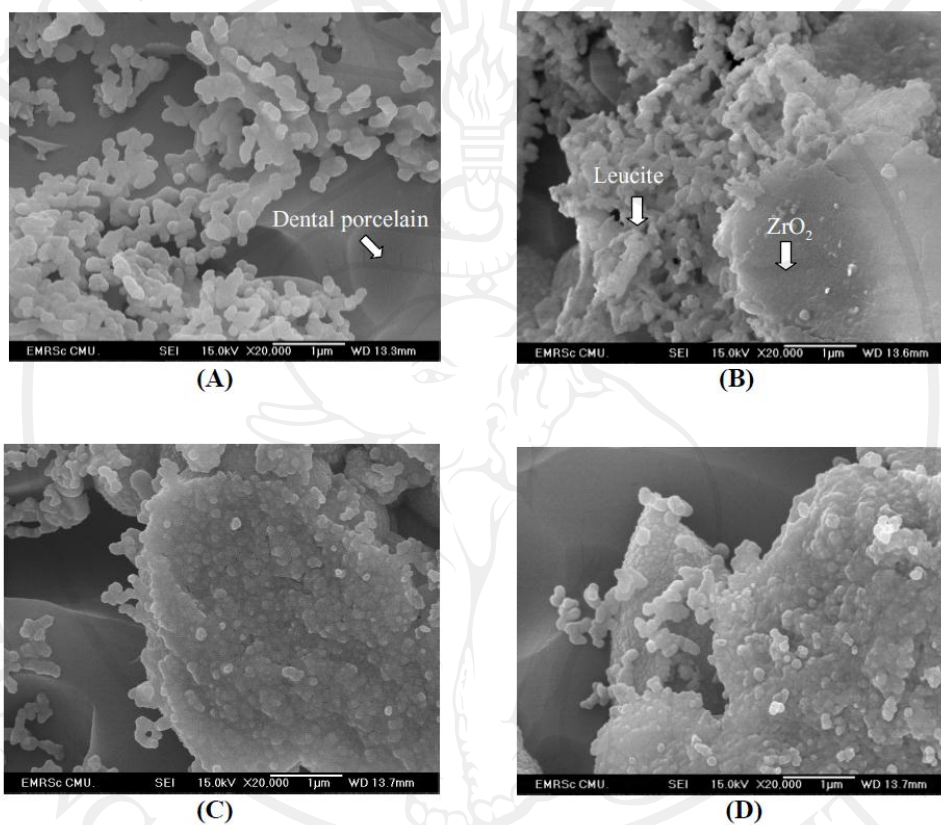


Figure 3. SEM micrographs of dental porcelain ceramics with (A) 0, (B) 15, (C) 20 and (D) 25 wt% ZrO_2 additives

References:

1. Pisitanusorn A, Thiansem S, Schulle W, Ananta S. *Interceram* 2006;55:250-253.
2. Mackert JR, Williams AL. *J Dent Res* 1996;75:1884-1890.
3. Sanitnapong C, Pisitanusorn A, Ananta S. *Chiang Mai J Sci* 2011;38:176-178.
4. Ong JL, Farley DW, Norling BK. *Dent Mater* 2000;16:20-25.
5. Apel E, Hoen CV, Rheinberger V, Holand W. *J Eur Ceram Soc* 2007;27:1571-1577.
6. Cattell MJ, Chadwick TC, Knowles JC, Clarke RL. *Dent Mater* 2005;21:811-822.
7. Barreiro MM, Riesgo O, Vincente EE. *Dent Mater* 1989;5:51-57.
8. Höland W, Rheinberger V, Schweiger M. *Phil Trans R Soc Lond A* 2003;361:575-589.

Acknowledgements: This work was supported by the Graduate School, Faculty of Science and Faculty of Dentistry, Chiang Mai University.

Keywords: dental porcelain ceramics, leucite crystallization, zirconia additive