ISSN: 0975-833X

Available online at http://www.journalcra.com

International Journal of Current Research
Vol. 9, Issue, 08, pp.56562-56567, August, 2017

INTERNATIONAL JOURNAL OF CURRENT RESEARCH

RESEARCH ARTICLE

EFFECT OF AGING ON Y-Zr ON PHASE STABILITY AND SURFACE CHARACTERISTICS – AN IN VITRO STUDY

*Dr. Gouri V Anehosur, Dr. Pragnya Medappa, Dr. Salian Bhavya Keshav and Dr. Lekha K

Department of Prosthodontics, SDM College of Dental Sciences and Hospital Dharwad 580009, Karnataka, India

ARTICLE INFO

Article History:
Received 24th May, 2017
Received in revised form
18th June, 2017
Accepted 23rd July, 2017
Published online 31st August, 2017

Key words:

Yttria- Stabilized Tetragonal Zirconia ceramic, Low temperature Aging, X ray Diffraction, Scanning Electron Microscopy, Phase transformation, Surface Characteristics.

ABSTRACT

Objective: The purpose of this study was to evaluate the phase stability and surface characteristics of Yttrium stabilised zirconia ceramics after various low temperature aging using X-ray diffraction analysis and Scanning Electron Microscopy respectively.

Materials and Methods: Our study included discs of Yttrium stabilized tetragonal zirconia from two manufacturers (n=15) Sample A AIDITE (Qinhuangdao Technology Co., Ltd, Hebei, China) and Sample B UPCERA (Shenzhen Upcera Dental Technology Co., Ltd, Guangdong, China), for each brand which were machined and sintered and glazed according to manufacturer's specifications to mimic a final polished crown that is ready for cementation. Specimens were artificially aged in distilled water by heat treatment at temperatures of 100, 150 and 200°C for 10 hours each in order to induce phase transformation. These specimens after aging were individually evaluated for the phase transformation using X- ray diffraction (JDX 8P, JEOL machine)and surface characteristics using scanning electron microscopy (6380 LA, JEOL machine).

Results: The results from x-ray diffraction analysis of both the specimens with and without low temperature aging revealed only the tetragonal phase. On scanning electron microscopy, the surface of the specimens in as received condition appeared to be smooth with a few blebs here and there. As aging was carried out, the surface irregularities became more pronounced.

Conclusion: The invitro tests conducted reveal that the two materials Aidite and Upcera did not show significant results on x-ray diffraction analysis and scanning electron microscopy.

Citation: Dr. Gouri V. Anehosur, Dr. Pragnya Medappa, Dr. Salian Bhavya Keshav and Dr. Lekha K, 2017. "Effect of aging on Y-Zr on phase stability and surface characteristics – An in vitro study", *International Journal of Current Research*, 9, (08), 56562-56567.

INTRODUCTION

Zirconia, emerging a supreme material with great value of strength and esthetics is being widely used in the field of dentistry. The array of applications of zirconia in dentistry include fabrication of posts, crowns, bridges, implants, implant abutments and esthetic orthodontic brackets (Manicone *et al.,* 2007). Zirconia exists in three crystal phases at different temperatures namely cubic, monoclinic and tetragonal. Zirconia is in the monoclinic phase at room temperature and transforms into tetragonal phase above 1070°C. This tetragonal phase of zirconia is stable between a temperature of 1170°C and 2370°C. The cubic phase is stable only at very high temperatures (Kim *et al.,* 2009). Clinically, the tetragonal phase of zirconia is important as it is hard, has good fracture toughness and is corrosion resistant. In order to maintain this clinically significant tetragonal phase at room temperature, stabilisers such as yttria, magnesia, ceria etc are added.

***Corresponding author: Dr. Gouri V Anehosur,**
Department of Prosthodontics, SDM College of Dental Sciences and Hospital Dharwad 580009, Karnataka, India.

Occlusal and lateral forces along with added influence of saliva bring about a transformation from the tetragonal to monoclinic phase which would be undesirable normally. Interestingly, during clinical use this transformation is beneficial as this exhibits a volume expansion of 3-4% (Subbarao *et al.,* 1974). This expansion inhibits the crack propagation that is initiated by various stresses. This transformation toughening phenomenon brings about high strength and fracture toughness of zirconia in vivo. Though zirconia has excellent mechanical properties, various phenomenon that decrease the life time of zirconia have been reported. Kobayashi et al were the first to observe the degradation after aging process at 150-400°C (Kobayashi *et al.,* 1981). Since then, various studies have been carried out on low temperature aging phenomenon affecting the mechanical properties (Chevalier *et al.,* 1999). To test the clinical performance of Yttrium stabilised tetragonal zirconia (Y-TZP) in vitro, a process known as low temperature aging is carried out. Low temperature aging of zirconia is tetragonal to monoclinic transformation under simulated clinical conditions. This mimics the clinical conditions in the oral cavity and tests the performance of Y-TZP over a period of time. For example, aging at 134°C for 1hour is theoretically similar to 3-4 years in vivo (Kim *et al.,* 2009; Chevalier *et al.,* 1999; Cales, 2000; Deville *et al.,* 2005).

MATERIALS AND METHODS

Discs of Y-TZP from two manufacturers (n=15) Group A-AIDITE(Qinhuangdao Technology Co. Ltd, Hebei, China) and Group B UPCERA(Shenzhen Upcera Dental Technology Co. Ltd, Guangdong, China), for each brand were machined and sintered and glazed according to manufacturer's specifications to mimic a final polished crown that is ready for cementation (Fig-1a and 1b). Control specimens (n=10) for each brand were evaluated in as-received condition.

Fig 1a. Y-TZP disc- Group A:-AIDITE

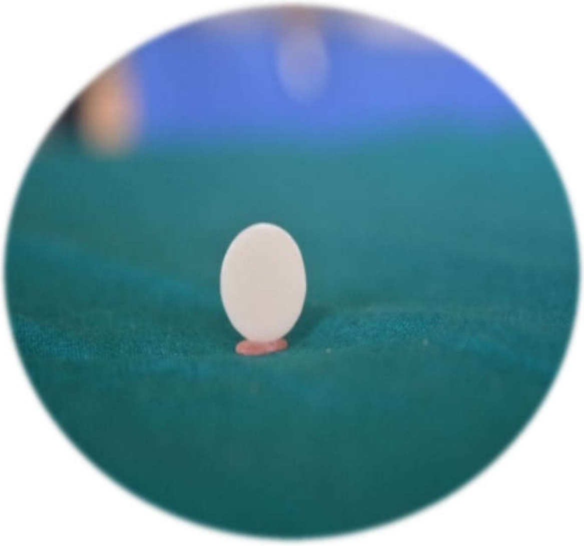

Fig. 1b. Y-TZP disc- Group B-UPCERA

Low temperature aging

Specimens were artificially aged in distilled water by heat treatment at temperatures of 100, 150 and 200°C for 10 hours each in order to induce phase transformation (Fig-2). Further they were individually evaluated for phase transformation using x-ray diffraction (XRD) and surface characteristics using scanning electron microscopy (SEM).

X ray diffraction analysis

Diffraction pattern is characteristic for a particular element. This pattern can be compared with the library data to understand the composition of the material. In our study, XRD was carried out using a diffract meter. JDX 8P,JEOL machine using Copper-potassium α radiation with a wavelength of λ= 1.54056Å, diffraction angle of 25 to 40°, step size of 0.02° and scan speed of 1° per minute was used (Fig-3a, 3b and 3c). Peaks from the XRD output were compared with the library data, which is the x-ray diffraction standards file (17-0923) on zirconium oxide (Kim *et al.,* 2009).

Figure 2a.

Figure 2b.

Fig. 2a and Fig 2b. Low temperature aging

Scanning electron microscopy

Scanning electron microscopy (SEM) is a procedure wherein the surface characteristics of the said specimen is scanned by a beam of electrons are reflected to form an image. Gold sputtering was carried out using a JFC 1600 Autofine Coater, JEOL machine wherein the specimens were coated with a 15nm gold layer in order to make their surface conductive (Fig-4a, 4b and 4c). The surface grains were analysed and photographs were taken at magnifications of 5.0k and 15.0k using a 6380 LA, JEOL machine (Fig-5).

RESULTS

XRD: The XRD results of both the specimen prior to aging revealed tetragonal phase. The highest phase of transformation was seen at a peak that ranged from 29.18° to 29.76° in Group A and 29.1° to 29.56° in Group B (Fig-6a and 6b). After aging only tetragonal phase was visible (Fig-7a and7b). No other significant changes were observed.

Figure 3a.

Figure 3b.

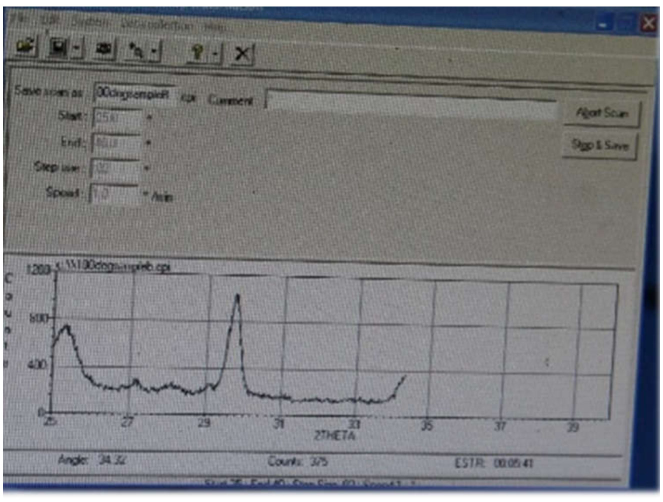

Figure 3c.

Fig. 3a, Fig-3b and Fig-3c. XRD using diffractometer JDX 8P, JEOL machine

Figure 4a.

Figure 4a.

Figure 4a.

Fig. 4a, Fig. 4b. and Fig. 4c. Gold sputtering using JFC 1600 Autofine Coater

Fig. 5. SEM JEOL machine

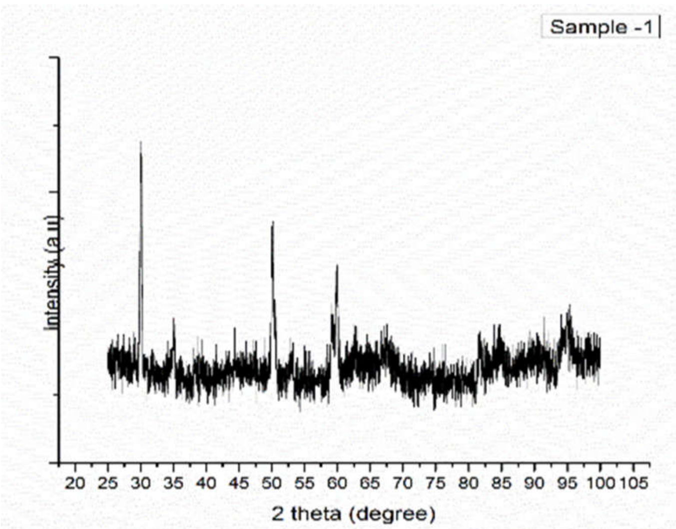

Fig. 6a. XRD of Group A before aging

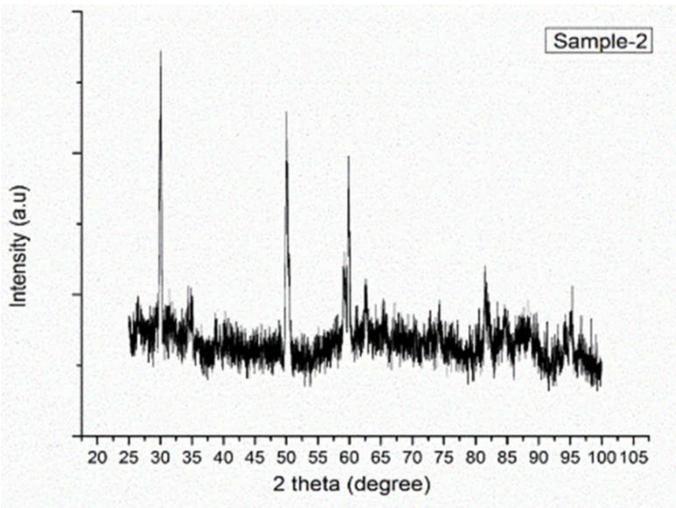

Fig. 6b. XRD of Group B before aging

SEM

The surface of the specimen in as-received condition appeared to be smooth except for a few irregularities. With low temperature aging, the surface irregularities became more pronounced in Group B as compared to Group A (Fig-8). These irregularities represented initiation of crack propagation.

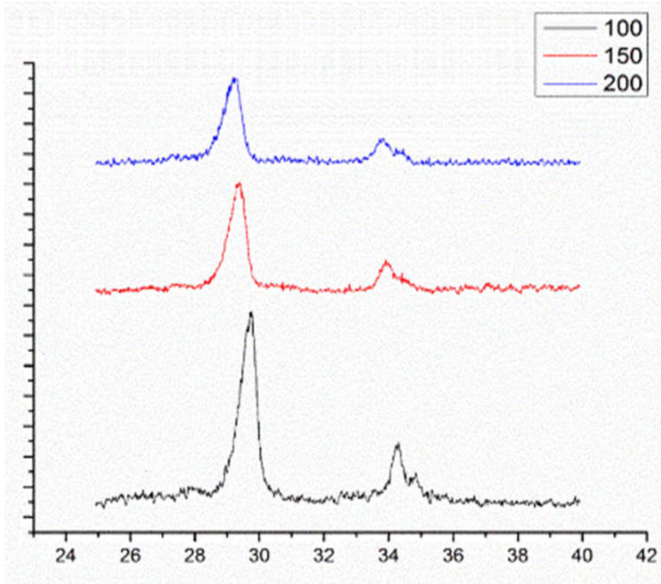

Fig. 7a. XRD of Group A after aging

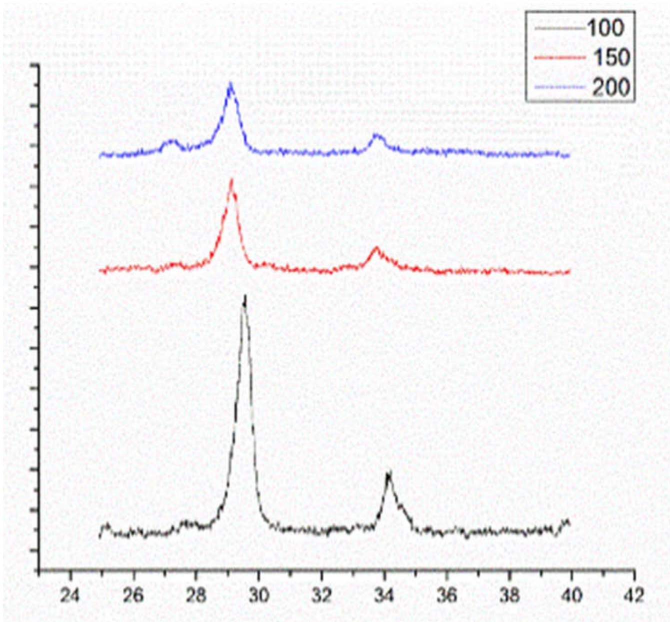

Fig. 7b. XRD of Group B after aging

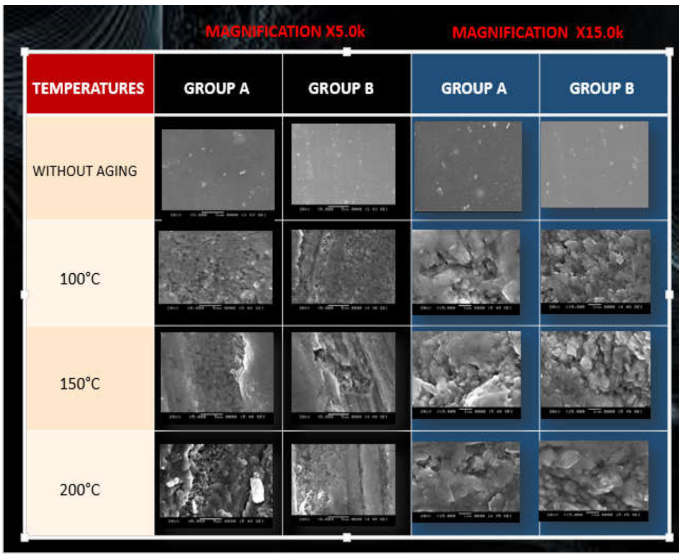

Fig. 8. Images from SEM before and after aging at magnifications of 5k and 15k

DISCUSSION

The phase transformation from tetragonal to monoclinic (up to 12%) is favourable in clinical situations as it does not allow crack to propagate. There is an increase in the flexural strength hence demonstrating an improved clinical performance of the material. When the percentage of monoclinic crystal increases beyond this i.e. 12-54%, there is a decrease in the flexural strength. In vitro phase transformation from tetragonal to monoclinic starts taking place from 100°C onwards. There is an increase in the monoclinic phase as the temperature increases. Therefore, to test the clinical performance of the material over a period of time, temperatures of 100°C, 150°C and 200°C were used. Also, the three temperatures used in the study correspond to the duration of the material in the oral cavity which signifies the longevity of the material. For instance, aging at 134°C for 1 hour is theoretically similar to 3-4 years in vivo. Hence aging at 100°C, 150°C and 200°C for 10 hours corresponds to approximately 8-10, 10-15 and 15-20 years in vivo respectively. XRD provides the percentage of particular phase of a material. According to a study conducted by Kim *et al.,* 2009 XRD indicated that the flexural strength increased upto 12% of monoclinic concentration. Monoclinic concentration increased sharply from 12 to 75% as the temperature was increased to 125°C and above. The peak of the monoclinic phase transformation is seen at the 2 theta angle of roughly about 28° and second peak for tetragonal phase transformation is seen roughly at an angle of 30°. As opposed to this, our study showed only the tetragonal peak at the aforementioned temperatures during XRD. This implies that the monoclinic phase transformation is not very evident in the materials in Group A and Group B when aging is carried out at a temperature above 100°C. In the study conducted by Kim *et al.,* 2009 SEM revealed an orange peel texture. It was found that, with an increase in temperature, there was an increase in the monoclinic phase giving it the orange peel appearance. As opposed to this, our study showed an increase in surface irregularities which became more pronounced as the temperature increased, thereby representing initiation of crack propagation. This implies that there has not been any volume expansion at the crack tip that inhibits crack propagation, suggesting the absence of monoclinic crystals in the two materials tested with aging.

Conclusion

The tetragonal to monoclinic transformation was caused by low temperature aging at temperatures of 100°C, 150°C and 200°C. After aging treatment up to 100°C, monoclinic phase started to appear, representing a clinically satisfactory performance of 8 -10 years. But the percentage of monoclinic crystals decreased with the higher temperatures i.e. 150°C and 200°C. This suggests that these two materials cannot be expected to perform clinically beyond 10 years.

Clinical implications of the study

The in vitro tests reveal that the two materials Aidite and Upcera did not show significant results on XRD and SEM which implies that the flexural strength of these two materials may be compromised after 10 years in vivo.

Scope of the study

- The need to study flexural strength of the two materials in vitro.
- The need to study the percentage of monoclinic crystals.
- An in vivo simulation of lateral and vertical forces after low temperature aging.

Acknowledgement

We would like to thank Rajiv Gandhi University of Health Sciences (RGUHS) for funding this study with reference id RGU: Adv. Res: Proposal-D-40:2015-16.

REFERENCES

Aboushelib, M.N., De Jager, N, Kleverlaan, C.J., Feilzer, A.J. 2005. Microtensile bond strength of different components of core veneered all-ceramic restorations. *Dent Mater.,* 21:984-91.

Ardlin, B.I. 2002. Transformation-toughened zirconia for dental inlays, crowns and bridges: chemical stability and effect of low-temperature aging on flexural strength and surface structure. *Dent Mater.,* 18:590-5.

Cales, B. 2000. Zirconia as a sliding material: histologic, laboratory, and clinical data. *Clin Orthop Relat Res.,* 379:94-112.

Cales, B., Stefani, Y., Lilley, E. 1994. Long-term in vivo and in vitro aging of a zirconia ceramic used in orthopaedy. *J Biomed Mater Res.,* 28:619-24.

Cattani–Lorente, M. et al. 2011. Low temperature degradation of a Y-TZP dental ceramic. *Acta Biomaterialia,* 7:858-65.

Chevalier, J., Cales, B., Drouin, J.M. 1999. Low-temperature aging of Y-TZP ceramics. *J Am Ceram Soc.,* 82:2150-204.

Chevalier, J., Deville, S., Mu ¨nch, E., Jullian, R., Lair, F. 2004. Critical effect of cubic phase on aging in 3mol% yttria-stabilized zirconia ceramics for hip replacement prosthesis. *Biomaterials*, 25:5539-45.

Deville, S., Chevalier, J., Fantozzi, G., Bartolome, J.F., Requena, J., Moya, J.S. 2003. Low-temperature ageing of zirconia-toughened alumina ceramics and its implication in biomedical implants. *J Eur Ceram Soc.,* 23:2975-82.

Deville, S., Gremillard, L., Chevalier, J., Fantozzi, G. 2005. A critical comparison of methods for the determination of the aging sensitivity in biomedical grade yttria-stabilized zirconia. *J Biomed Mater Res B Appl Biomater,* 72:239-45.

Emsley, J. 1991. The Elements (2nd ed.). Oxford University Press, Oxford, UK.

Garvie, R.C., Nicholson, P.S. 1972. Phase analysis in zirconia systems. *J Am Ceram Soc.,* 55:303-5.

Guazzato, M., Quach, L., Albakry, M., Swain, M.V. 2005. Influence of surface and heat treatments on the flexural strength of Y-TZP dental ceramic. *J Dent.,* 33:9-18.

Kim, H.T., Han, J.S., Yang, J.H., Lee, J.B., Kim, S.H. 2009. The effect of low temperature aging on the mechanical property and phase stability of Y-TZP ceramics. *J Adv Prosthodont,* 1:113-7.

Kobayashi, K., Kuwajima, H., Masaki, T. 1981. Phase change and mechanical properties of ZrO2-Y2O3 solid electrolyte after ageing. *Solid State Ionics,* 3/4:489-93.

Kosmac, T., Oblak, C., Jevnikar, P., Funduk, N., Marion, L. 1999. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. *Dent Mater.,* 15:426-33.

Lawson, S. 1995. Environmental degradation of zirconia ceramics. *J Eur Ceram Soc.,* 15:485-502.

Lilley, E. 1990. Review of low temperature degradation in Y-TZPs. In: Tressler RE, McNallan M, editors. Ceramic transaction: corrosion and corrosive degradation of ceramics. Westerville: American Ceramics Society, 387-407.

Lube, T., Manner M, Danzer R. The miniaturization of the 4point bend test. Fatigue Fract. Engng Mater Struct 1997;20:160516.

Manicone, P.F., Lommetti, P.R., Raffaelli, L. 2007. An overview of zirconia ceramics:Basic properties and clinical implications. *Journal of Dentistry Nov,* 35(11):819-26.

Oblak, C., Jevnikar, P., Kosmac, T., Funduk, N., Marion, L. 2004. Fracture resistance and reliability of new zirconia posts. *J Prosthet Dent,* 91:342-8.

Piconi, C., Maccauro, G. 1999. Zirconia as a ceramic biomaterial. *Biomaterials*, 20:1-25.

Pittayachawan, P., McDonald, A., Petrie, A., Knowles, J.C. 2007. The biaxial flexural strength and fatigue property of Lava Y-TZP dental ceramic. *Dent Mater,* 23:1018-29.

Roebben, G., Basu, B., Vleugels, J., Van der Biest, O. 2003. Transformation induced damping behaviour of Y-TZP zirconia ceramics. *J Eur Ceram Soc.,* 23:481-9.

Shimizu, K., Oka, M., Kumar, P., Kotoura, Y., Yamamuro, T., Makinouchi, K., Nakamura, T. 1993. Time-dependent changes in the mechanical properties of zirconia ceramic. *J Biomed Mater Res.,* 27:729-34.

Suarez, M.J., Lozano, J.F., Paz Salido, M., Mart, nez, F. 2004. Three-year clinical evaluation of In-Ceram Zirconia posterior FPDs. *Int J Prosthodont.,* 17:35-8.

Subbarao, E.C., Maiti, H.S., Srivastava, K.K. 1974. Martensitic transformation in zirconia. *Phys Status Solidi Series A,* 21:9-40.

Wen, M.Y., Mueller, H.J., Chai, J., Wozniak, W.T. 1999. Comparative mechanical property characterization of 3 all-ceramic core materials. *Int J Prosthodont.,* 12:534-41.

Yoshimura, M. 1988. Phase stability of zirconia. *Am Ceram Sot Bull,* 67:1950-5.

****