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Multi-Response Evaluation of Zirconia Toughened Alumina Dental Ceramic Composite using Desirability Optimization Methodology

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Abstract*—Zirconia Toughened Alumina (ZTA) composites possess excellent mechanical and tribological properties, which make it relevant to both industries and biomedical practice. Failure of monolithic alumina and zirconia in dental and orthopedic restorations has made researchers to explore innovative applications of ZTA as excellent biomechanical and biocompatible prostheses. Significant technological and scientific attention has focused on biomaterials that are of great interest to patients and care-givers seeking for optimal solution, hence the study. The study involves Design of Experiment (DoE) that encompasses modified conventional slurry method of specimen production, Mechanical testing with micro hardness tester FM-7, and scanning electron microscopy, optical microscope, using America Society for Testing and Material protocols. The MINITAB 18-based Response Surface Methodology was used in Central composite Inscribed Design (CCID) design of experiment involving two factors: sintering temperature and composition of range 1500-1650℃ and 5-15wt% respectively on hardness and fracture toughness .The test results of experimentation were analysed using RSM while Desirability Function Analysis (DFA) was applied in optimization. The result indicated on surface plot that increase in sintering temperature (Ts) increases hardness and fracture toughness. Similarly, increase in composition of ZrO2 decreased hardness but increased fracture toughness. At optimal setting of 1681.01 ℃ and 9.357wt%, the Vickers hardness and fracture toughness of 15.2963GPa and 4.9172 MPa$m^{1/2}$ and composite desirability (D) of 0.6875 of was achieved in ZTA composite. The ZTA has improved fracture toughness of 19.93% and 9.27% and a compensatory reduction in hardness of 6.5% and 4.0% on monolithic alumina and zirconia respectively. The excellent mechanical properties can complement aesthetics, long- term biocompatibility and superior biomechanical properties to define the future of dental prostheses.

Keywords*— *Desirability optimization methodology, hardness, sintering, fracture toughness, zirconia toughened alumina, composite desirability

I. INTRODUCTION

Zirconia toughened alumina are polycrystalline ceramic composite composed of alumina matrix and secondary phase of meta-stable 0-50wt% tetragonal zirconia particles either unstabilized or stabilized [1]. The secondary phase addition results in an enhancement process [2] that builds an infrastructure for excellent mechanical –properties [3], optimal tribological properties and long-term biocompatibility [4] and superior biomechanical characteristics. With excellent properties, high hardness, high strength, high fracture toughness, good mechanical strength at high temperature, good thermal shock resistance, [3] wear resistance [5], ZTA have led a broad indication spectrum for long-span prostheses in dentistry and industries. In this frame, ZTA composite, has ameliorated the concern for cytotoxicity and allergy in alumina and long term hydrothermal stability of Zirconia [6]. The later was responsible for critical event of 2001, where femoral head prostheses fractured due to accelerated low temperature degradation [7, 32]. The combined benefits of excellent mechanical properties, biocompatibility and high biomechanical properties are essential in dental applications where the prostheses remain in direct contact with body fluids and susceptible to phase transformation due to stress and LTD.

In a Al2O3-ZrO2 system, alumina provides high strength and hardness while tetragonal zirconia exerts a toughening effect, by controlling transformation into the monoclinic phase [8-9]. More important in composite engineering is the distribution of the zirconia particles in alumina matrix. Zirconia aggregates can lead to localized ageing phenomenon while alumina can become preferential sites for crack propagation [10-11]. Then, homogenization becomes imperative to produce transformation-toughened

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composites, provided that zirconia particles are sufficiently small sized, matrix does not react with particles and has high elastic modulus to maintain a low martensitic transformation (Ms) temperature [5]. The presence of dispersed tetragonal zirconia particle in matrix affect the strength and toughness of the material through martensitic transformation and transformation taking place near the surface of the macroscopic piece [5]. [12] reported that increasing the uniformity in the zirconia particle size and homogeneity of distribution improved the mechanical properties of ZTA. Some of the homogenization technologies include: grinding and mixing (slurry method), wet chemical method or electrochemical repulsion technique [13]. Similarly, increased volume of fraction of zirconia affects the stability of the tetragonal phase. An optimum amount of zirconia is necessary to retain favourable amount of tetragonal content that enhance thermal and mechanical properties of ZTA without grain growth during sintering [5]. Most researchers concluded that an optimum amount of zirconia should exist between 8-15 vol% in ZTA, considering a trade-off between improvements in fracture toughness, decrease in grain size and lower hardness [14-16].

Sintering is another process parameter that influences the dispersion of tetragonal zirconia particles in the matrix. It is suggestive that the particles are mobile in nature, likely to separate from each other and grow during sintering, and move within the grain boundary of the matrix [5]. Sintering cycle is also important to create a full dense components and microstructure with high performance. Densification of ZTA eliminates voids in the microstructure and improves mechanical properties. Often, magnesia (MgO) (<1wt%) are needed for needed for good densification and to limit abnormal grain growth during sintering [1,15,16,17,18,19]. Densification and strength of sintered zirconia are dependent on sintering temperature and temperature gradient. In the work of Wu *et al*.[20], the sintering temperature of alumina/ zirconia system was varied at 1500, 1550,1600 and 1650℃, and the holding time of 1hr using muffle furnace.

Conventional ceramic shaping methods such as dry pressing, isostatic pressing, slip casting, tape casting [21-24] , injection molding [25] and stereolithograph-based additive manufacturing (3D) [3], have been used to fabricate ZTA ceramics. Understanding each and every stage of the process is required to develop microstructure that possesses the value required for biomedical grade ZTA for oral and maxillofacial restoration/reconstructions. The mechanical properties of the system are greatly influenced by the factors identified. In a multi-response optimization (MO) problem as this, it is essential to represent the relationship of all responses to be optimized. Only by doing so, one can achieve the ideal balance among the desired response levels. Understanding these properties and working conditions are absolutely essential when engineered systems are set.

The present work deals with determination of optimum responses of different compositions of ZTA composites produced with modified slurry method at different sintering temperatures.

II. EXPERIMENTAL DESCRIPTION

A. Sample Preparation

As–received alumina powder (α- Al2O3, d_{50}=150nm,BET= 12.8m^2/g, Inframat® Advanced materials™ USA) and zirconia powder (HWYA-N-IS, d_{50}= 150nm, BET= 12m^2/g, Guangdong Orient Zirconic Ind. Sci. Tech.co;China) were mixed together by varying the compositions of zirconia between 8-15wt%. Pure Al2O3 and ZrO2 samples were prepared as control samples. The mixture was partially homogenized with ultrasound and mechanical milling, in a planetary ball mill, was performed in distilled water with 65wt% of solid content for 12hrs using zirconia balls. The slurry of the powder mixtures were dried in an oven for 24h at 100℃ and the dried lumps were crushed and passed through a 100mesh screen. In all composition, the Al2O3 powder was doped with 25ppm of MgO (Guangdong Orient Zirconic Ind. Sci. Tech.co; China) powder to inhibit the grain growth during sintering.

B. Preparation of the Ceramics

Magnesia (Zchimner & Schartz, Lahnstein, Germany), <1wt%, was mixed as a binder with powder green sample with strength for handling. The compaction was conducted by using hardened steel die of 16mm diameter. 4.5g of the powder were poured into the die cavity and uniaxially pressed at a pressure of 60MPa (beyond pressure for crack-free sample) using Elvec hydraulic press at a constant strain rate; to obtain a green sample of Ø16mm and 5mm thickness.

C. Post Processing of ZTA green body

Further processes were performed on ZTA green body to improve its performance:

i. Debinding. A 2-step debinding profile consisting of a vacuum step followed by air pyrolysis debinding was adopted to remove binders in the green body. The samples were heated at 600℃ for 3h in vacuum with heating rate of 1℃/min. after, the samples were heated for 1000℃ for 30mins in air at 5℃ /min.

ii. Sintering. The samples were finally sintered (pressureless sintering) in a box furnace between 1500- 1650℃ at 1hr dwell time. The furnace was shut off after the heat treatment to cool. The sintered samples were ground with 0.25µm diamond paste and subsequently polished.

D. Characterization

The Vicker's hardness was tested on polished sintered samples by using a micro hardness tester FM-7. Indentations were made on surfaces with a load of 98N held for 10 seconds and in accordance with ASTM E384. Ten indentation tests were performed for each sample at randomly chosen spots and average hardness determined.

Toughness was calculated directly from crack lengths produced by Vickers indentation at 98N(10kg) load. The corresponding indentations sizes and crack lengths were determined using optical microscope (Olympus PMG 3). The fracture toughness (K_{ic}) was computed using a model equation given by Niihara *et. al*, [16] for palmqvist cracks;

$$K_{ic}(MPa) = 9.052\ x\ 10^{-3} H^{\frac{3}{5}} E^{\frac{2}{5}} dc^{\frac{1}{2}} \qquad (1)$$

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Where H, Hardness; E, Young modulus (assumed 210GPa for the samples); d, average diagonal line length of the indentation, c, length of palmqvist crack. The valid measurements satisfied requirements of the JIS R 1607[17] on acceptable indentation crack. The microstructure of the sintered composite was characterized using scanning electron microscopy (SEM, Nova NanoSEM 430, the Netherland).

III. RESULTS AND DISCUSSIONS

A. Microstructure

The microstructure of the composite reveals a fairly fine distributed, dense ZTA, with zirconia particles evenly dispersed long the grain boundaries of the matrix. It has a limited number of pores at boundaries as shown in Fig 1.The zirconia grain size is small enough to exhibit toughening phenomenon of zirconia in ZTA system. This is in agreement with [26] that small grain size for ceramics is the target for increased resistance to crack propagation, to provide maximum hindrance on the path of crack. The ZrO_2 particles in Al_2O_3 grain boundaries suppress the grain growth of the matrix and improve the mechanical properties. The microstructure was not completely homogenized even with ultrasonic and mechanical milling, which is in agreement with [12], that homogenization remains a challenge in many techniques applied. Similarly, the high sintering temperature of 1681℃, zirconia content of 10wt% and MgO binder <1% were not sufficient to guarantee high densification as indicated with the presence of voids (pores).

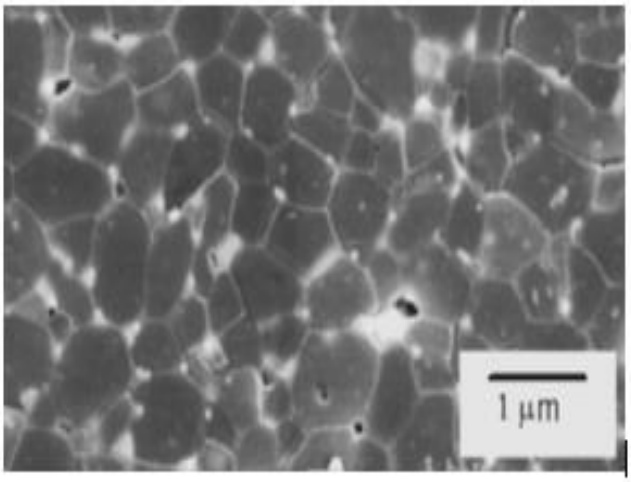

Fig .1. Microstructure of Al2O3-ZrO2 at 1681℃ and 10 wt% composition of Zirconia

B. Analysis of Mechanical Properties

Minitab 18 software was used in the Design of experiment, in planning, analyzing and interpreting data. Box Wilson's central composite inscribed design (CCID) of response surface methodology (RSM) and desirability function analysis (DFA) were used for designed experiment and optimization respectively.x1 and x2 represent sintering temperature of 1500-1650℃ and composition of 5-15wt% ZrO2 respectively. With RSM-Box Wilson's CCID, a design matrix of 1500, 1571, 1575, 1650, 1681 ℃ for sintering temperature (Ts) and 2.92, 5, 10, 15 and 17.1 wt% composition of Zirconia was generated for data collection.

Hardness

The surface plot shown in Figure 2 indicates a decrease in hardness as the sintering temperature increases till 1550℃ and then increased. According to [5][27], as higher sintering temperature offers better densification, hardness value increases as a whole. Sintering temperature and ZrO2 contents strongly affect flexural strength and hardness of sintered specimen [28]. [26] Stated that the higher alumina content, hardness would be higher and fracture toughness lower, [5] stated that hardness decreases with increase in ZrO2 content. The study is in agreement the various mentioned researchers. The amount of zirconia has adverse effect on the hardness due to coarsening effect that cause porosity [5]. The result of hardness is similar to hardness property of ZTA prepared through colloidal processing route [28, 34].

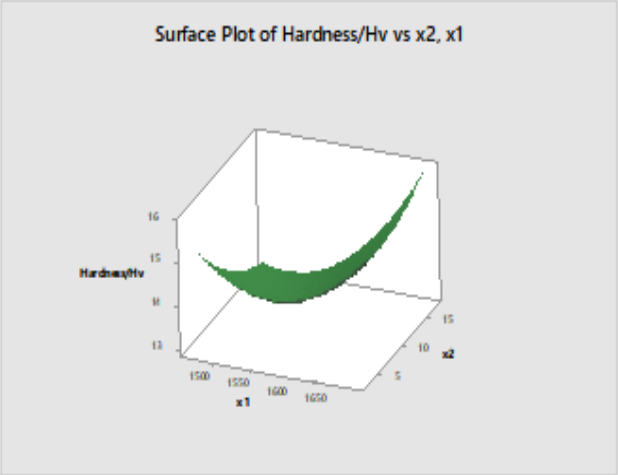

Fig. 2. Surface Plot of Vickers Hardness.

C. Fracture Toughness

The fracture toughness increases with sintering temperature and ZrO2 content as indicated in Figure 3. Sintering in practice is the control of both densification and grain growth [5]. Similarly, the fracture toughness increases with increased ZrO2 content. Higher zirconia content in ZTA results to higher fracture toughness and flexural strength but lower hardness [29]. Generally, homogeneity of alumina and zirconia improve fracture toughness in a composite material [26]. The increasing alumina content of the composite increases the hardness but decrease the fracture toughness.

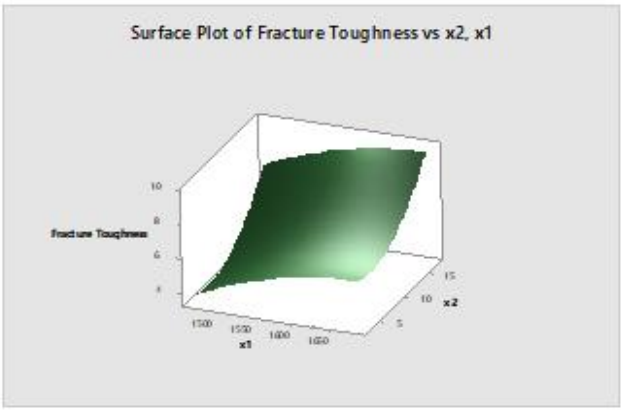

Fig 3. Surface Plot of Fracture Toughness

According to [5], m-ZrO2 phase increases with ZrO2 content. The retention of t-ZrO2 varies during fracture and t-

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ZrO_2 decrease with increasing ZrO_2 content. T-ZrO_2 retention readily triggers the transformation to monoclinic. The phenomenon yields transformation toughening, however, m-ZrO_2 retention improves micro-crack toughening. The work [30] stated that toughness determination based on direct crack length measurements have recently suffered harsh criticism. The work [12] concluded that the direct crack measurements may be incorrect concerning the absolute toughness, they still show correct trend in toughness. The goal was to maximize the responses on x1 and x2. The optimized performance of Al_2O_3-x%volZrO_2 in terms of fracture toughness and Vickers hardness and individual desirability function(IDF) were 4.9172MPa$m^{1/2}$ (0.47267) and 15.2963GPa(1.0000) respectively as shown below in optimization plot, Figure 4. The fracture toughness value was unimpressive. The composite desirability (D) of 0.6875 was achieved at maximum setting of sintering temperature and composition of 1681℃ and 9.357 wt%ZrO2 indicated on

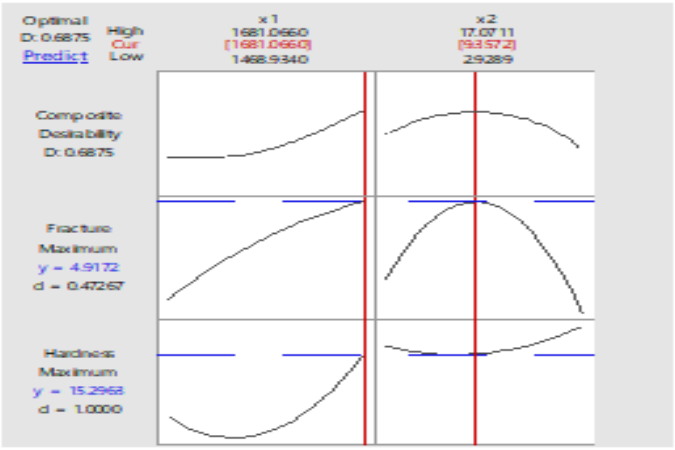

Fig.4. Optimization plot

Table 1: Comparative Analysis Table of Al2O3,- x% vol ZrO2 and Control Samples.

Table 2. Solution Computation

Solution	x_1	x_2	Fracture Toughness Fit	Hardness/ GPa Fit	Composite Desirability
1	1681.07	9.3572	4.91715	15.2963	0.687513

Table 3. Multiple Response Prediction for Response Optimization

Response	Fit	SE Fit	95% CI	95% PI
Fracture Toughness	4.917	0.615	(3.462, 6.372)	(2.580, 7.254)
Hardness/GPa	15.296	0.456	(14.218, 16.374)	(13.565, 17.028)

The optimal setting of zirconia content of 9.35718wt% falls within the range of 8-15 wt%, considered by [14-16] as appropriate in terms of tradeoff between improvements in fracture toughness, decrease in grain size and lower hardness. [12] Stated that mechanical properties are attractive for a ZTA with only 10vol% reinforcement approximate to values required for biomedical grade ZTA.

C. Comparative Analysis

The response values at optimal setting of sintering temperature and ZrO_2 content, could be the best basis for comparison between the composite and monolithic Alumina and zirconia.

S/N	Response	ZTA composite	Alumina	*Zirconia*
1	Vicker Hardness(GPa)	15.2963	16. 37(-6.5%)	15.94(-4.0%)
2	Fracture Toughness(MPa$m^{1/2}$)	4.9172	4.1(+19.93%)	4.5(+9.27%)

From Table 1, the ZTA has improved fracture toughness of 19.93% and 9.27% and a compensatory reduction in hardness of 6.5% and 4.0% on monolithic alumina and zirconia respectively. The reduction in hardness is in agreement with many researches. The composite possesses superior biomechanical properties and long- term biocompatibility in addition to excellent mechanical properties. Due to its biocompatibility, good aesthetics, and high mechanical properties, one of the most popular uses of zirconium dioxide is in dentistry, mainly in dental restorations for bridges, crowns, and feldspar porcelain veneers and dental prostheses [31], however, suffers LTD and consequently not sate for dental restoration. Alumina has good mechanical properties but has aesthetic and biocompatibility concern. The results in Table 1 agree with [32,33] that if most zirconia is retained in the tetragonal phase, the addition of zirconia to alumina yields a higher strength and fracture toughness with little reduction in hardness and elastic modulus compared to monolithic alumina ceramics. The work also stated that the excellent wear characteristics and low susceptibility to stress-assisted degradation of high performance alumina ceramics is also preserved in zirconia toughened alumina ceramics.

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IV. CONCLUSION

At optimal setting of 1681.01℃ and 9.357wt%, the Vickers hardness and fracture toughness of 15.2963GPa and 4.9172 MPa$m^{1/2}$ and composite desirability (D) of 0.6875 of was achieved in ZTA composite. The excellent mechanical properties can compliment aesthetics, long-term biocompatibility and superior biomechanical properties to define the future of dental prostheses. The result of fracture toughness yielded impressive result when compared with monolithic alumina and zirconia. For any process optimization of dental and orthopedic prostheses, optimized dispersion of the second phase and close control of the zirconia particle size are factors for biomedical- grade -ZTA mechanical properties irrespective of fabrication route. To avoid reintroduction of LTD, a magnesium-stabilized zirconia was used. ZTA blends the superior qualities of zirconia and alumina in addition to biocompatibility. But, a review in process parameters especially homogenization technique and sintering temperature gradient are essential.

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