Available online at www.sciencedirect.com

ScienceDirect

Ceramics International 42 (2016) 2306–2316

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Effect of hydrothermal treatment on tribological properties of alumina and zirconia based bioceramics

Ajoy Kumar Pandey[a,*], Koushik Biswas[b]

[a]Department of Metallurgical and Materials Engineering, National Institute of Technology Warangal, Warangal 506004, Telangana, India
[b]Department of Metallurgical and Materials Engineering, Indian Institute of Technology, Kharagpur 721302, West Bengal, India

Received 21 July 2015; received in revised form 4 October 2015; accepted 5 October 2015
Available online 17 October 2015

Abstract

Four types of ceramic composites; ceria stabilized zirconia (CSZ), yttria stabilized zirconia (YSZ), ceria stabilized zirconia toughened alumina (CSZ-TA) and yttria stabilized zirconia toughened alumina (YSZ-TA) were developed through powder metallurgy route using conventional pressure less resistance heating. Hydrothermal treatment was carried out to estimate the ageing stability of the developed materials. Fretting wear using ball on flat geometry were carried out before and after hydrothermal treatment to understand the effect of hydrothermal treatment on wear properties of developed materials. Variation of wear properties like wear depth, wear volume and coefficient of friction with duration of hydrothermal treatment was correlated with the microstructure and phase developed before and after hydrothermal treatment. It was found that presence of ceria decreases wear damage after hydrothermal treatment. For CSZ, wear volume decreased to 800×10^5 μm^3 from 1488×10^5 μm^3 while for YSZ it increased to 220×10^5 μm^3 from 28×10^5 μm^3 at 20 N load. Similar trend was found in CSZ-TA and YSZ-TA specimens also. It was understood that during hydrothermal treatment, a part of tetragonal phase of CSZ transforms to orthorhombic zirconia which in turn improves the wear properties.

Keywords: B: Composites; C: Wear resistance; D: Al_2O_3; D: ZrO_2

1. Introduction

Zirconia and alumina based bioceramics are intensively used ceramic material for load bearing orthopedics and dental field. For such applications, a material needs to be good corrosion and wear resistant and should possess low coefficient of friction. The wear resistivity of materials mainly depends upon the composition, hardness, fracture toughness, grain size, porosity, phase distribution, and experimental parameters such as applied load, sliding speed, frequency, humidity present and lubricating conditions, etc. [1–6]. The coefficient of friction mainly depends upon the surface topography and the adhesive properties of the tribo-system. The zirconia–alumina composites possess controversy regarding phase stability, toughness, design flexibility and wear resistive property. Alumina possess high hardness as well as wear resistive property but have poor toughness. So under stressed condition fracture and chipping may cause brittle failure of alumina [7]. On the other hand, zirconia exhibits higher toughness but low wear resistive property compared to alumina and also suffers from low temperature degradation (ageing) in long run [8–12].

The effect of aqueous environment on wear of alumina as well as zirconia has been studied by different researchers. It was found that alumina can form a tribo-chemical layer on the surface in presence of water which act as a lubricating "interface" during wear [8]. This layer effectively reduces wear rate. On the other hand, in wet condition zirconia undergoes some phase transformation which effectively degrades the mechanical and also wear properties [8,13]. The wear of alumina is affected by the pH of the aqueous solution [14,15]. It was also found that the wear rate of alumina under concentrated contact pressure is higher than that of zirconia in bovine serum solution. The probable reason was assumed to be the micro-cracking of brittle alumina under concentrated stress [16]. Hence, from the above

---

*Corresponding author.
E-mail address: ajoy100@gmail.com (A.K. Pandey).

http://dx.doi.org/10.1016/j.ceramint.2015.10.026

discussion it is obvious that wear resistance of alumina–zirconia composite depends upon several parameters and to decide particular materials superiority in a typical condition, comparative analysis is highly required. Study of fretting wear is recommended for orthopedic implants [17] and few study on alumina, PSZ and TZP are already carried out without analyzing the effect of ageing on wear properties [18–21]. Hence, a comparative study with the ageing/degradation of the material is required.

In this study, fretting wear response for four kinds of developed bioceramics, i.e., ceria stabilized zirconia (CSZ), yttria stabilized zirconia (YSZ), ceria stabilized zirconia toughened alumina (CSZ-TA) and yttria stabilized zirconia toughened alumina (YSZ-TA) were compared. Effect of ageing on wear properties were estimated by conducting wear study on hydrothermally treated (accelerated ageing) specimens. Hydrothermal treatment was carried out for a total duration of 25 h inside an autoclave in the presence of simulated body fluid (SBF) at 134 °C/0.02 MPa. It is reported that one hour ageing at such a condition can be extrapolated to 3–4 year *in vivo* [10,22–24].

2. Materials and methods

2.1. Material development

Four types of ceramic composites powders (CSZ, YSZ, CSZ-TA and YSZ-TA) were synthesized by co-precipitation techniques. Nitrate salts in proportionate quantities were used to synthesize the powder. In this study, 14 mol% ceria stabilized zirconia (14CSZ), 8 mol% yttria stabilized zirconia (8YSZ), 15 wt% zirconia (stabilized with 14 mol% ceria) toughened alumina (15CSZ-TA) and 15 wt% zirconia (stabilized with 8 mol-% yttria) toughened alumina (15YSZ-TA) powder were used. To synthesize powders, proportionate quantities of nitrate salts were dissolved in distilled water and later ammonium solution was added drop wise to start precipitation. pH of the solution was raised around 9–10 for complete precipitation. Precipitated powders were filtered, washed and dried and calcined at 1000 °C for 2 h. Calcined powders were compacted uniaxially at 600 MPa and sintered using a two step sintering process (optimum sintering schedule is mentioned in Table 1) in a resistance heating furnace. Detailed synthesizing process and optimization of composition, calcination, compaction and sintering of powders were discussed elsewhere [25–27].

Sintered specimens were observed under a Scanning Electron Microscope (SEM) and grain size was estimated through the following formula [23]:

$$d_i = 1.56 \times 2 \times \sqrt{\frac{A_i}{\pi}} \tag{1}$$

where, A_i is the area of each grain, and d_i is the equivalent diameter of the grain (assuming spherical grain). The constant 1.56 was adopted to represent the 3D grain size from observed 2D intersecting plane of the same [28]. During sintering, uniformly distributed grains with almost 100% theoretical density was ensured.

2.2. Hydrothermal treatment (ageing)

Sintered specimens were rough and diamond polished (polishing using 25 μm sized diamond paste on a velvet cloth) to surface finish of R_a (arithmetic average of the absolute values) = 0.03 μm. To know the ageing response in an accelerated condition, polished and ultrasonically cleaned samples were hydrothermally treated inside an autoclave in the presence of SBF prepared according to Tadakama et al. [29]. The hydrothermal treatment was carried out for a total duration of 25 h at 134 °C/ 0.02 MPa. After hydrothermal treatment the samples were characterized by X-ray diffraction (XRD) and scanning electron microscope (SEM) and further fretting wear were carried out.

2.3. Fretting wear test

Fretting wear was carried out on diamond polished ($R_a \sim 0.03$ μm) as well as hydrothermally treated samples. The test was carried out in ball (counterpart) on flat (specimen) geometry at 20 N and 50 N load (approx. hertzian pressures calculated to be around 2.0 and 2.7 GPa, respectively) using fretting wear tester (TR-283M-M4, Ducom, India), for a total duration of 10^5 cycles with a stroke length of 1 mm and 10 Hz frequency of oscillation. In this study, tungsten carbide (WC) ball was used as counterbody.

2.3.1. Characterization of wear

During experiment, total wear depth and frictional force were recorded continuously using two different sensors attached with the equipment. In this case, the total measured wear depth comprises of four different contributions: (i) wear

Table 1
Sintering schedule grain size and mechanical properties of developed materials.

Samples	Optimum sintering schedule	Sintered density (g cm^{-3})	Hardness (HV20[a])	Avg. grain size (μm)
14CSZ	1500 °C for 1 h and 1400 °C for 2 h	6.1	965 ± 17	2.86 ± 0.92
8YSZ	1450 °C for 30 min and 1250 °C for 14 h	5.9	1240 ± 08	0.54 ± 0.12
15CSZ-TA	1550 °C for 1 h and 1450 °C for 2 h	4.32	1730 ± 06	Zirconia:1.13 ± 0.29 Alumina: 1.26 ± 0.23
15YSZ-TA	1500 °C for 1 h and 1400 °C for 2 h	4.20	1808 ± 09	Zirconia:1.00 ± 0.40 Alumina: 0.76 ± 0.25

[a] Vickers hardness were measured using 20 kgf load and 10 s dual time.