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Wear behavior of additive manufactured zirconia

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Abstract

Complete consolidation of ceramic particles is feasible for SLA, but print directionality is well acknowledged to be a source of structural anisotropy. By varying build direction of SLA zirconia specimens, pin-on-disk tests were carried out to evaluate any differences in wear behavior and phase composition with respect to laminar architecture. All combinations of horizontal and vertical pin-disk combinations demonstrated equivalent wear coefficients and phase transformation from monoclinic to tetragonal zirconia but do exhibit a nominal trend in run-in wear. Given that microstructural consolidation of AM ceramics is highly dependent on debind and/or sintering rather than print parameters the absence of anisotropy introduce by print parameters could benefit the application and development around AM ceramics.

Keywords: Type your keywords here, separated by semicolons ;

1. Introduction

There are numerous opportunities for additive manufacturing (AM) in orthopaedics, but these are hindered by the lack of a good tribological material. Though a prerequisite for orthopedic applications, tribological performance of SLA ZrO_2 has not be characterized with respect to laminar orientation that invariably results with AM. Accordingly, coupons were produced with varying print directions to establish any volumetric wear and resultant wear coefficient dependence on build direction in the sintered microstructure for horizontal and vertically built parts.

Nomenclature

AM	additive manufacturing
R_a	surface roughness, arithmetic average
SLA	stereolithography
ZrO_2	zirconia

1.1. Impact of SLA print direction on wear resistance

Anisotropy is often unavoidable in additive manufactured parts. Layer-by-layer material deposition is known to induce directionality in mechanical, physical and electrical properties for different approaches. For example, anisotropic effects were reported for rectangular prisms built with selective laser sintering of 15 μm alumina particles coated in PMMA polymer [1]. Variation in the green strength was posited to be related to part orientation and energy density, as orientation resulted in a difference in scan time from layer to layer. Material extrusion

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is another additive approach that routinely results in heterogeneity due to vector-based deposition and the shear-effects imparted on particles by the extruder nozzle. Extruded silicon nitride bend test specimens were shown to have anisotropic shrinkage, but the dimensional adjustments were determined to be predictable in the initial design [2]. Laminated object manufactured (LOM) alumina specimens similarly demonstrated differences in linear shrinkage in the z-direction and x-y plane, and laminar defects incurred by additive manufacturing were mitigated by bulk sintering, which homogenized the microstructure. However, build direction was seen to have a measurable effect on resultant properties, as parts tested in three-point bending had higher strength when the load was orthogonal to the build layer [3]. As with LOM, SLA requires relatively little energy to fuse layers, requiring parts to be bulk fired in a high temperature furnace to achieve densification. Consequently, the technology has a lower susceptibility to anisotropy, but studies continue to report directional differences in as-built surface quality as the result of scan strategies [4].

Ceramics are often subject to final grinding and polishing prior to use in wear applications, but these are some of the most expensive and time-consuming operations in the manufacturing supply chain. Establishment of base line wear of as-received AM ceramics is sufficient to establish the degree at which additive manufactured surface texture influences tribology. Certainly, smooth surfaces will wear in, so that steady state wear coefficients should be insensitive to initial roughness. A previous study of traditionally manufactured (pressed and sintered) ceramics found that the measured wear volume of spherical alumina bearings scaled linearly with increasing roughness (from 0.01 to 0.60 μm) with a less pronounced effect for the counterface alumina disk [5]. This phenomenon was prescribed to a higher volume of asperities which interacted with the counterface body, raising stress concentrations by increasing radial cracking [6]. Similarly, by establishing how ceramics behave long-term when loaded orthogonal or parallel to the build layer, valuable insight in design of wear elements for SLA can be obtained.

1.2. Phase transformation of zirconia in wear applications

High strength and improved toughness consistently give zirconia an advantage over other orthopedic ceramics, but zirconia (ZrO_2) as a material presents trade-offs [7]. Polycrystalline ZrO_2 can exist in three allotropes: monoclinic (m-ZrO_2), tetragonal (t-ZrO_2), and cubic (c-ZrO_2). Transformation between the crystal structures can be initiated by temperature changes during manufacturing. When heated from room temperature, the stable monoclinic compositions shift first to tetragonal and then to cubic at very high temperatures. Upon cooling, a 100% tetragonal ceramic can be stabilized at room-temperature with the addition of an oxide such as yttria, magnesia, or ceria. These stabilizing oxides concentrate at grain boundaries and apply compressive stresses to prevent any uncontrolled t-ZrO_2 to m-ZrO_2 transformation. The t-ZrO_2 is characterized as metastable at room temperature as the result of t-ZrO_2 possessing a lower surface energy than m-ZrO_2. The desired toughness of ZrO_2 is attributed to tetragonal crystals as the shear strain generated by the wake of the crack front introduces stress into the lattice and imparts sufficient energy to induce a phase transformation. Since the phase shift is accompanied by a 3-4% volume expansion of the lattice, the larger monoclinic grains generate a compressive stress at the crack front, thereby increasing the energy required for crack propagation [8]. Long-term however, a t-m transition is detrimental to the overall integrity of the material bulk. Though the phase transformation toughening increases fracture toughness of a material, the t-m transition is also accompanied by the generation of small microcracks that can compromise the strength of the entire part.

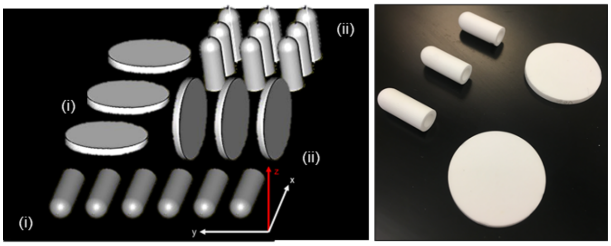
Sliding during wear has also been shown to induce a phase change for yttria-stabilized ZrO_2. A wear study of 2 mol% yttria ZrO_2 die used in wire drawing demonstrated high transformability with the primary wear mechanism being intergranular fracture [9]. Increased volume fraction of monoclinic ZrO_2 is not recommended as transformed ceramics exhibit reduced hardness and elastic modulus to depths of 1.6 μm [10]. Further still, since monoclinic ZrO_2 is shown to be rougher than tetragonal ZrO_2, a large percentage of monoclinic crystals may negatively affect wear resistance by increasing the friction force and presenting a more abrasive surface [11–13]. Studies have shown that lubricated and dry grinding environments can change the crystal composition of yttria stabilized ZrO_2, but careful polishing can remove transformed grains at the surface and does not induce additional stress or scratches [14,15]. Fatigue wear is the dominant wear mechanism frequently observed in ceramics, characterized by sub-surface cracks that propagate towards the surface and can lead to pull out or spalling [9].

Since laminar manufacturing can introduce anisotropic properties, wear behavior can be subsequently affected by the build orientation of parts. To investigate the effect on layer orientation on multiphase SLA zirconia, part orientation was varied such that loading was applied either orthogonal or parallel to the layer direction.

2. Methods

2.1. Mechanical coupons

Yttria (3 mol%) stabilized zirconia specimens were procured from 3DCERAM (Limoges, France). Hemispherical pin geometries were held at Ø3/8” [9.53 mm] by 1” [25.4 mm]. Given the small size and axisymmetic nature of the parts, no observable distortion existed. Pin orientation was controlled such that six pins were built horizontally, with the long axis parallel to the *x-y* plane, and six pins were built vertically, with the long axis perpendicular the X-Y plane (in the build direction) (Fig. 1a). However, to ensure proper debinding and sintering of the 3DCERAM coupons, the pins were hollowed to ensure a wall thickness of 4mm (Fig. 1b). General process inputs and set print parameters are reported in Table 1. Specific laser paths were set by the suppliers in order to produce the parts economically. All wear pins were slightly oversized parts to account for shrinkage and grinding allowance; the specimens were machined to meet final dimensions. Counterface inlay disks were also procured, which were built in the horizontal and vertical directions. Pressed and sintered, material-matched control specimens of the same geometry from CoorsTek (Golden, Colorado) were used for comparison.



(a) (b)

Fig. 1. (a) Schematic of wear coupons (a) printed in the (i) horizontal and (ii) vertical direction. (b) All 3DCERAM pins were hollowed out to ensure complete debinding/sintering.

Table 1. Wear coupon process parameters

Zirconia		CoorsTek[a]	3DCERAM		
			x	*y*	*z*
Particle Size (d_{50})	(µm)	<1		0.5	
Solids Loading	(%)	*n.r.*		50	
Sintering	*(°C)*	*n.r.*		1450	
Layer thickness	(µm)	*N.A.*			25
Young's modulus	(GPa)	330.0	212.2	211.9	221.0
Poisson's Ratio	*N.A.*	0.22		0.31	

[a]Reported for Technox ZrO_2, d_{50} = size of particles that represent 50% of the population. *n.r.* = not reported, *N.A.* = not applicable.

2.2. Material characterization

Surface roughness (R_a) was assessed with a stylus profilometer which mapped a 50 µm x 50 µm area on a location in the plane of wear (DektakXT Bruker, Tuscon, AZ). Relative bulk density and volumetric porosity were measured with the standard Archimedes approach (ASTM C373). Grinding and polishing was applied to all counterface disks prior to wear testing (mean R_a = 0.22-0.36µm), microhardness testing, and grain size analysis to reflect industry practice and achieve desired low abrasive wear. Microhardness was evaluated from the average of ten measurements on three independent specimens at 1 kgf (9.8 N) for 15 sec (Leco M-400, St. Joseph, Michigan). Grain size analysis on carbon-sputter coated wear scars was performed following thermal etching in a furnace for 15 min at 1400°C. The volume fraction of the monoclinic phase for the horizontally built, vertically built, and control wear pins (*n*=3 per group) both as-received and after wear testing was determined with X-ray diffraction (XRD), using a diffractometer with CuKα source (40 kV and 40 mA). Scans were taken between a 2θ range of 27° and 35°, step size of 0.01°, and a time per step of 1 second (D8 Advance, Bruker). The pin flat was used to measure the as-received surface for clarity. The fraction of monoclinic crystals, V_m, was calculated using Eq. 1 [16].

$$V_m = \frac{1.311 * (I_m^{(\bar{1}11)} + I_m^{(111)})}{1.311 * (I_m^{(\bar{1}11)} + I_m^{(111)}) + I_t^{(111)}} \qquad (1)$$

where I_m and I_t are the intensities of the diffraction peaks at the appropriate 2θ value for monoclinic and tetragonal zirconia respectively.

Accelerated low-temperature degradation on horizontally-built and vertically-built polished zirconia disks was induced by placing the parts (*n*=2 per group) into an autoclave (SSR-2A-MC Mark V CSS, Boston, MA) held at 2 bar and 131±1°C for 24 hours. Of note, this is 20°C less than the recommended temperature for Mg-stabilized zirconia (ASTM F2393). Diffraction patterns of the wear disks were assessed before and after aging.

2.3. Tribological testing

Pin and disk components were paired to evaluate all combination of build orientations. Wear testing occurred between machined pins and polished disks. In test conditions identical to earlier investigations of water-lubricated AM ceramics [17]. Dynamic loading was scaled to the Paul's walking curve and applied along 10 mm square path. Cycles occurred at a frequency of 1.6 Hz where the max load was 40 N and the average load across the stance phase was 21N. Calculation of the initial Hertzian contact of a Ø3/8" [9.53 mm] pin was 2.8 GPa for the horizontal pins and 2.9 GPa for the vertical pins, which are relevant to orthopaedic applications. The small difference was the result of orientation-dependent mechanical properties reported by 3DCERAM (Table 1). Six initial timepoints of 0.165 million cycles (Mc) were carried out to 0.99 Mc, after which an additional three timepoints at intervals of 0.33 Mc were taken, equating to ~80 km. The six earliest timepoints were performed at tighter intervals to better capture run-in wear, defined as 0-1 Mc. Steady-state wear was determined to be the remaining 1-2Mc. The rates (mm^3/Mc) for both run-in and steady-state wear were calculated by applying a least squares linear regression for each cycle range.

Wear scars were examined with optical microscopy at 50-100X magnification and quantified with Image J. Wear scars were used to calculate the volumetric mass loss according to ASTM G99-17 and wear coefficients were found using the Archard Wear Law [18]. Following testing, a select number of wear pins were sectioned perpendicular to the wear scar, sputter-coated, and imaged with scanning electron microscopy.

2.4. *Statistical analysis*

After assessing normality of the data sets, an ANOVA was used to determine if layer direction (build orientation) of either the pin or disk component had a significant impact on the wear of the pin ($\alpha<0.05$). Two-tailed unpaired *t*-tests were used to compare the monoclinic phase measured on the as-received surface compared to the worn surface for horizontally-built, vertically-built and control pins. A second unpaired *t*-test was used to compare the final XRD calculation for m-ZrO_2 fraction present in thermally aged AM zirconia disks ($\alpha<0.05$).

3. Results

Density, volumetric porosity, wear plane surface roughness before and after polishing, microhardness of the polished surface, and grain size were determined for a sampling of the pin and disk coupons (at least three independent specimens for all measurements) and compared to metrics measured or hot-pressed (control) ZrO_2 (Table 2). Bulk density, apparent porosity and hardness were similar across all groups, although variance is relatively high. Grain size was higher for CoorsTek compared to the SLA parts, while the as-received SLA R_a exceed that of as-received/polished CoorsTek. Following standard grinding and polishing, the average surface roughness AM counterface disks was reduced. However, despite identical processing, vertically-built disks still observed greater surface roughness compared to the horizontally-built disks. This can be attributed to the surface topography of the as-built surface, where the vertical plane exhibits the peaks and troughs associated with the 25 µm layers (Fig. 2b). The horizontal plane (Fig. 2a) does show curved steps associated with the build orientation.

Table 2. Measured properties of anisotropic wear coupons. Averages and standard deviations (in parentheses).

Property		CoorsTek Control		3DCeram Horizontal		3DCeram Vertical	
Bulk density	g/cm³	5.97	(0.04)	5.92	(2.63)	5.92	(0.09)
Porosity	%	1.25	(0.47)	1.38	(0.43)	1.22	(0.41)
Pin R_a as-received	µm	---	---	0.48	(0.14)	0.44	(0.11)
Disk R_a polished	µm	0.19	(0.01)	0.22	(0.05)	0.36	(0.03)
Hardness	GPa	12.4	(0.06)	12.9	(0.13)	12.8	(0.28)
Grain Size	µm	0.54	(0.04)	0.31	(0.02)	0.30	(0.02)

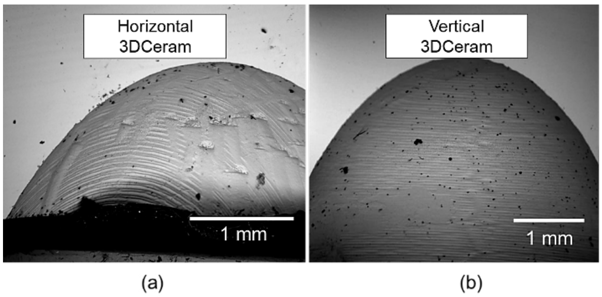


Fig. 2. Representative SEM images of as-built (a) horizontal and (b) vertical surface of interior hollowed cavity of each pin.

The scar for horizontal builds had a fringed appearance on the circumference that corresponded with the 25 µm layers. In contrast, the wear scars of the pins oriented in the $+z$ direction exhibited non-circular scars that generally follow the outline of the layer (Fig. 3b). At the zero-time point, the calculated Hertzian contact was between 2.8-2.9 GPa. For all subsequent time points, the pressure fell to a range of 0.1-2MPa (Fig. 4). A change in contact stress over time is a recognized characteristic of pin-on-flat wear with spherically tipped pins. The most notable decrease in average contact pressure was seen for the horizontal pin-on-vertical disk group. This corresponded to an increase in volumetric wear between 0.825-0.99 Mc. Figure 4 also shows a large variance in wear results, which is common with brittle materials. There are two points for H on V that have noticeable higher wear than the remainder of the data. This is not unusual in wear testing, and regardless the wear for all such cases is very low. As can be seen in Fig. 5, the wear coefficients were lower than 10^{-7} mm³/Nm for all pins, and comparable to self-mated pressed and sintered CoorsTek ZrO_2.

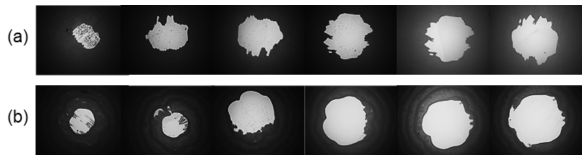


Fig. 3. Wear scar progression for (a) horizontally and (b) vertically built ZrO_2 pins, starting at 0.16 million cycles at the left and extending to 1,98 million cycles at the right.

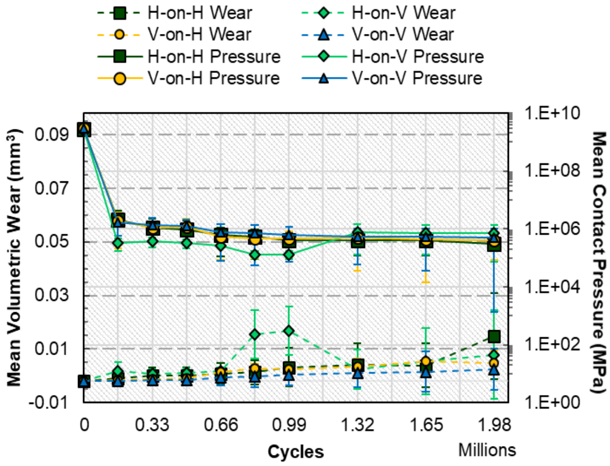


Fig. 4. Mean wear and contact pressure for the different build orientation pairings. horizontal (H) pin on vertical (V) disk. Error bars show the standard deviation for an $n = 3$ for each condition.

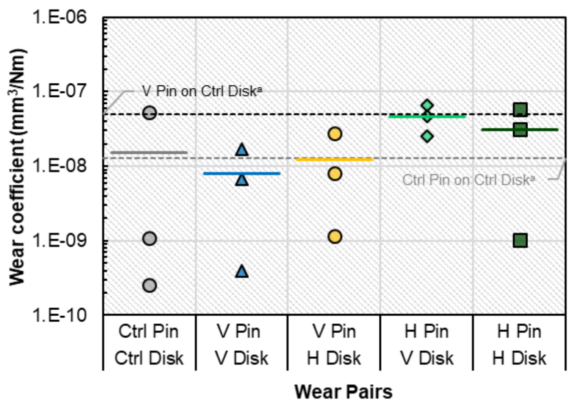


Fig. 5. Individual wear coefficients for each orientation combination plotted alongside the group average. [a]Reference lines are mean wear coefficients for the same materials of earlier work [17].

A Shapiro-Wilkes test revealed that the wear coefficient data had a non-Gaussian distribution (p<0.05). A logarithm transformation rectified normality (p=0.38) and permitted the use of a two-way Analysis of Variance (ANOVA) for only the 3DCERAM specimens. The results showed that neither component location (p=0.88) nor build orientation (p=0.38) had a statistically significant effect on wear-behavior. 3DCERAM pin data was therefore aggregated and used to evaluate build direction relative to control CoorsTek pins. No significant difference was resolved between non-HIP material-matched controls, horizontally-built, and vertically-built ZrO_2 wear pins (p=0.35) (Table 3).

The aggregated data set was also used to evaluate the run-in and steady-state wear rate. Using a least squares linear regression for 0-1Mc for run-in and 1-2Mc for steady-state wear the wear rates were plotted with their 10% confidence interval (Fig. 6a). For all three groups, the initial run-in was nominally higher than the steady state rate (Fig. 6b). Of note, though not significant, vertically-built pins depict lower run-in wear relative to conventional controls and horizontally-built specimens.

Table 3. ANOVA tables summarizing the effect of build orientation on SLA zirconia wear.

Two-way Source	df	log(Wear coefficient) log [mm³/Nm] Sum of Squares (SS)	Mean Square (MS)	F Value	p Value	Power
Component Location[a]	1	0.014	0.014	0.022	**0.88**	0.05
Build Orientation[b]	2	1.32	0.662	1.01	**0.38**	0.21
Interaction	2	0.742	0.371	0.567	**0.57**	0.24
Model	2	2.08	0.416	0.636	**0.67**	
Error	30	19.6	0.655			
Corrected Total	35	21.7				

[a]pin or disk, [b]horizontal, vertical.

One-way Source	df	log(Wear coefficient) log [mm³/Nm] Sum of Squares (SS)	Mean Square (MS)	F Value	p Value	Power
Manufg. Approach[c]	2	1.32	0.662	1.071	**0.35**	0.22
Error	33	20.4	0.618			
Corrected Total	35	21.7				

[c]horizontal, vertical, or control

Following wear testing, three pins per build condition (horizontal, vertical, control) were cross sectioned at the wear scar and imaged with SEM. Two of horizontally-built AM pins revealed penny crack-shaped areas, immediately below the wear scar (white arrow). The porosity was significantly reduced in these areas as indicated by the black arrows, highlighting a localized region where the microstructure shifted from tetragonal to monoclinic (Fig. 7). Areas of reduced porosity were not observed for the control or vertically-built pins for the chosen cross-sections.

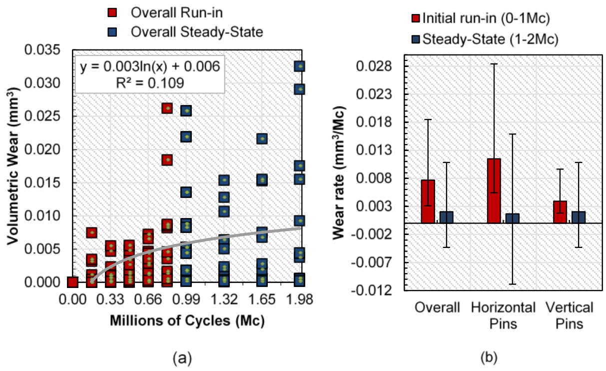


Fig. 6. (a) Least squares linear regression using aggregated wear pin data and (b) the calculated slope and 95% confidence interval for the run-in and steady state wear.

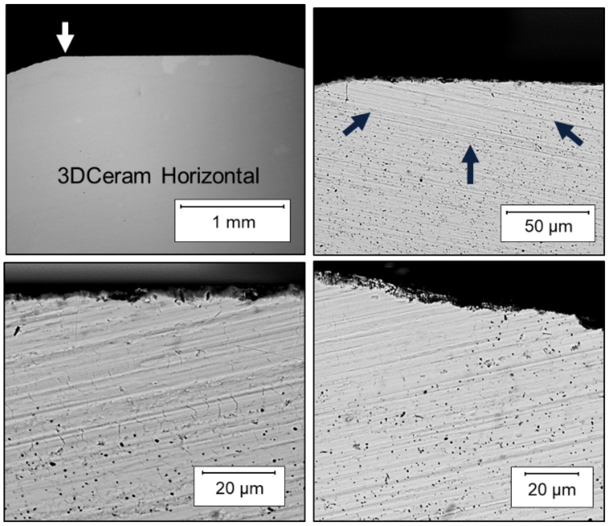


Fig. 7. Transformed area directly beneath the wear scar (white arrow) monoclinic grains are evident by the absence of porosity. Porosity can be seen as plack pores adjacent to the monoclinic grains.

To globally quantify the monoclinic volume fraction, x-ray diffraction was used to measure ZrO_2 specimens as-received and following wear testing. CoorsTek pins were shown to have higher starting monoclinic composition compared to 3DCERAM with a nominal drop following wear testing, but the final difference was not significant. The starting and ending percentage of monoclinic phase was similar for both vertical and horizontal prints, with the worn surfaces having a 10% higher measurement (Fig. 8).

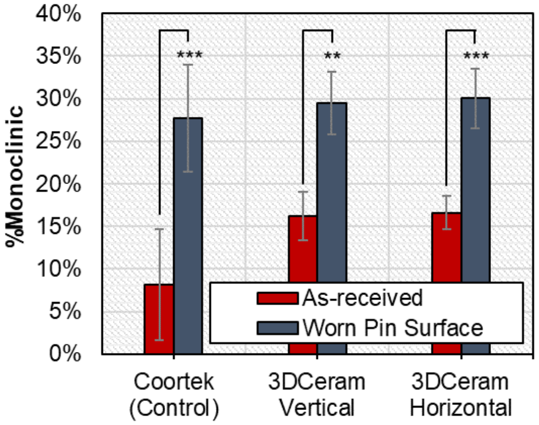


Fig. 8. Percent monoclinic on as-received and worn zirconia wear pin surfaces.

4. Discussion

The few works that have investigated wear performance of ceramic AM parts rarely consider the effect of build orientation. The present study controlled the build orientation of SLA ZrO_2 coupons, such that compressive and sliding loads were either parallel or perpendicular to the layers. The analysis demonstrated no clear anisotropy of wear behavior with respect to build direction.

Mechanical properties revealed the only notable difference between the horizontal and vertical specimens was the surface roughness despite identical polishing conditions. This can be attributed to the lower resolution of the printed disks in the *z* direction relative to the *x-y* plane, where the surface topography of the as-built surface of the vertical plane exhibits the peaks and troughs associated with the 25µm layers (Fig. 2)

However, since a significant difference in wear was not observed in the wear response, initial surface roughness does not appear to have a significant effect. This is mostly likely the result of the mirror-finish that develops on the wear surface. Following the first time point, a wear scar was observed for all groups (Fig. 9). This supports that testing occurred primarily between interfaces that were highly polished. This is supported by an earlier investigation of DMP ZrO_2 which concluded that anisotropic surface features were eliminated through careful polishing [4]. An additional consideration is the lack of a sizable sample population to make concrete conclusions. The statistical power calculated for an aggregated sample size of 36 (n=6 per group) was 0.2, well under the accepted 0.8, suggesting a greater number of tests would require definitive comparisons (Table 3). However, the same earlier work still supports the findings here, as they concluded that no clear evidence of directionality of layers were found to influence four-point bend strength or fracture toughness [4]. AM ceramics therefore could present an advantage over AM metals in tribological applications by eliminating print direction as factor in wear behavior. This can greatly simplify the use of AM parts for tribological applications, as they can be oriented in any direction.

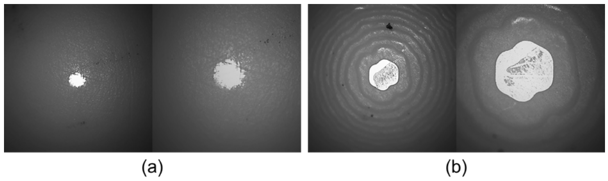


Fig. 9. Wear scars following the first timepoint, 0.165 Mc, for (a) a horizontal pin and (b) vertical pin at 50x and 100x mag.

Diffraction patterns of the worn hemispherical surface were noisy. However, the significant increase in the amount of m-ZrO_2 suggests that either mechanical or environmental exposure of the affected vertical and horizontal pins was similar. The aged ceramic coupons had lower monoclinic phase compared to the worn pins. This could be due to the noise in XRD pattern and the lower temperature used for aging, but ultimately the wear test could have introduced more strain in the lattice compared to simulated LTD. Furthermore, the aged horizontal disks observed a significantly lower level of transformation compared to the vertical disks. Since the surface roughness of the horizontal disks was much lower than the vertical printed disks, the measurement suggests that the surface topography in the build direction (25 µm layers) was not completely eliminated. Previous work on phase transformation of zirconia demonstrated that monoclinic nucleation occurs preferentially around scratches and surface features with valleys [11]. Therefore care, should be taken to eliminate all surface variation left by the deposition of layers. A coupled theta scan was utilized for the study with an X-ray penetration that correlates to a depth of ~5-6 µm, which was considered appropriate since transformed regions were approximately 20 µm deep [19]. However, a better measurement would be to obtain the monoclinic volume fraction at varying depths from the surface. Measurements just below the ZrO_2 surface have been successful using a low angle of incidence 1-2° XRD setting or the use of Raman spectroscopy [19,20].

It should be noted that the accelerated wear tests presented here correlate to the loadings and speeds encountered by a ceramic in a total hip replacement. The wear mode is dominated by adhesion and abrasion; fatigue wear is possible, but only after decades of use. Thus, the results are applicable to orthopaedic applications.

5. Conclusions

A common concern with AM parts is the property anisotropy inherent to a layered microstructure. Consolidation of SLA parts however, requires much lower energies than what are seen for powder bed fusion, and coupled with the use of resins, which can optimally incorporate submicron and high solids loading, to accomplish sintering at extreme temperatures, isotropic behavior is feasible for SLA zirconia. Thus, the wear behavior of self-mated SLA yttria stabilized zirconia was investigated and the following conclusions were made:

- The volumetric wear and calculated wear coefficients of SLA zirconia tested in physiological conditions displayed no dependence on part orientation.

- The location of a horizontally versus vertically wear component did not have an effect on the wear response.
- AM zirconia is transformable under sliding loads.

AM zirconia has demonstrated and superior wear resistance which remains important to establishing predictability around AM ceramics for orthopedics and additional benefit will come from continuing to characterize the long-term performance of AM zirconia to the strict reliability standards (ISO 13-356).

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