Effect of fiber patterns on the fracture of implant/cement interfaces

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Abstract

Electrospinning is a process by which fibers with micron to nanometer diameters can be obtained from an electrostatically driven jet of polymer solution. These fibers have a high surface area to volume ratio, which have numerous applications in biomedical implants such as total hip implant, dental implant. The present study is based on the hypothesis that the differences of the surface properties at titanium/cement interface due to incorporation of micro and sub-micron diameters fiber directions may have influence on the quality of titanium/cement union. The objectives of this research were to design and construct electrospinning unit for the fabrication uni-and bi-directions polycaprolactone (PCL) fiber on titanium and to measure the effect of fiber directions on the interface fracture strengths of sandwiched titanium (Ti) and poly methyl methacrylate (PMMA) cement samples with (uni-and bi-directions) and without fibers. Two groups of single edge sandwiched Ti/PMMA specimens were prepared. First group of specimen consists of Ti/PMMA sandwiched specimen without PCL fiber. Second group of specimen consists of Ti/PMMA sandwiched specimen with uni-and bi-direction PCL fibers. PCL fibers were ejected from the syringe via charged needle and deposited on two different grounded collectors to coat uni-and bi-directions PCL fibers on Ti plates. PMMA cement was poured and cured on the Ti plates with and without PCL fibers in a custom made mold to prepare Ti/PMMA samples with uni-and bi-directions fibers. Shear tests were conducted on each group of Ti/PMMA samples using Evex tensile test stage. Interface fracture toughness was calculated to determine the effect of fiber patterns on Ti/PMMA samples. This study successfully produced an electrospun unit that can produce uni-and bi-direction PCL fibers. Diameters of produced fibers were found to be in the range of 919 nm ~1.25 μm. This study found that the values of $K_{IC}$ of Ti/PMMA with uni-direction fiber were significantly higher when compared to the values of $K_{IC}$ of the Ti/PMMA without fiber ($p<0.05$), although the values of $K_{IC}$ of Ti/PMMA with bi-direction fiber were significantly lower when compared to the values of $K_{IC}$ of the Ti/PMMA without fiber. Results indicated that the addition of the fiber to Ti improved the quality of Ti/PMMA union and fiber directions have significant effect on the strength of the Ti-PMMA union.

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Keywords

Titanium; bone cement; PMMA; Polycaprolactone; Electrospin; interface fracture toughness; orthopedics

1. Introduction

Clinical follow-up studies of joint replacement show that loosening of the implant from the bone cement is the first mechanical event of loosening. Loosening can occur due to unsustainable interface stresses, usually initiated from defects along the interface [1].

Artificial total hip joint surgery has become an efficient and cost effective procedure in the treatment of patients with painful end-stage arthritis. The surgery allows relieving pain and restoring the daily activity. If the mechanical stress is properly transferred to the bone present around the implants, they can support the joint function without loosening and without causing periprosthetic osteolysis, which guarantees a longer and better survivorship. However, loosening and osteolysis of implanted total hip joints occur even if the insertion of the prosthesis is technically well performed [2].

Debris from implants has been considered as a critical factor inducing local host response. It seems to be influenced by multiple factors, i.e., type of material and fixation, design of prosthetic implants, bone quality, and activity and disease status of the patients. Extensive research had been done for bone remodeling to improve interaction with the implant. Development of wear resistant material and effort on maintaining better bone quality are also important [3]. The mechanical characteristic of the fiber induced implant can be obtained by sandwiching the micron or nanosize fiber in between the implant and cement. The effect of the fiber patterning on the strength of implant/cement interfaces has not yet been studied.

Electrospinning is the process of producing fibers in the range of micro to nano scale using polymer solution. The electrospinning process has been attracted greatly over a past decade due to the simple and versatile technique for the production of micro to nano sizes fibers. A liquid electrostatic polymer is ejected through a syringe, which is held at high voltage, causing the polymer to form electrically charged droplets at the tip of the syringe. At a certain critical voltage, the charged jets undergo an elongation and thinning to form one-dimensional fibers. These micro to nano fibers from the electrospinning has many applications in a diverse range of fields, such as biomedical engineering, filtration, electrical engineering, and optics [4]. Many techniques have been achieved over the past few years, to form uniform fibers of a wide variety of materials that includes polymers, ceramics, metals, and hybrid materials [5].

The fracture toughness of implant/cement interfaces is a mechanical property that measures the resistance to propagation of a crack originated at the interface. Our previous research [6] reported that the addition of the micron and nano sized MgO particles to PMMA improved the fracture toughness of bone/cement union due to increased surface energy by addition of MgO particles to PMMA. The deposition of the micron to nano sizes polymer fibers to implant surface may improve the fracture toughness of implant/cement union due to the
same reason. To the best of the present authors’ knowledge, however, no studies of this aspect have published. In this study, experiments were performed to analyze the differences of the surface properties at titanium/cement interface due to incorporation of micro and sub-micron diameters electrospun fibers. This study hypothesized that fiber pattern may have significant influence on the quality of titanium/cement union. The objectives of this study were to coat titanium plates by micro or nano level aligned fibers in uni-and bi-directions and to test the effect of fiber direction on the interface fracture toughness of Ti/PMMA samples with (uni-and bi-directions) and without fibers.

2. MATERIALS AND METHODS

2.1. Materials
Ti bars (6Al-4V ELI, ASTM B 348 standard, grade 23, biocompatible) of dimension (22×12×2) mm were purchased from Titanium Metal Supply, Inc., Poway, CA. Cobalt™ HV bone cement (Biomet Inc., Warsaw, IN) was used as the PMMA cement. PCL was purchased from Sigma Aldrich. PCL was selected as fiber material since it is biocompatibility, biodegradable and electrostatic. PCL solution was prepared by ultrasonic (Sonics & Materials, Inc., Vibra-cell VCV 130) mixing of 7.69 wt% of PCL beads with acetone. The sonication process was carried out at approximately 80°C for an hour. The solution was poured into a glass syringe in an infusion pump (Harvard Ins.).

2.2. Design and manufacture of the electrospun unit for uni-and bi-direction fibers
To produce unidirectional aligned fiber, vertical drum extraction method was used as shown in Fig 1 (a). A DC motor with the drum was mounted on a precision linear stage (Newport Corporation., model#426). The motion of stage was controlled by a linear actuator (Newport Corporation., model #LTA-HS). PCL fiber solution were ejected from the infusion pump glass syringe (Harvard Apparatus, mode # PHD ULTRA) via charged needle (23G blunt needle, aluminum hub, 1” length, model # BX 25). The fibers were deposited on a grounded custom made drum collector. The needle was charged by high voltage power source (Gamma High Voltage Research, Inc., model # ES 30 series). Six Ti plates and a carbon tape were attached on the drum using double sided tape as shown in Fig 1 (b). Carbon tape (Fig 1(c)) was used for the visualization and dimensioning of fabricated fibers using Nikon SMZ stereomicroscope and Hitachi TM 3000 scanning electron microscope, respectively.

For the fabrication of the bidirectional method, vertical electrospinning unit (Fig 2(a)) was used as well. Two sets of parallel plates were used as the ground collector instead of drum collector. The plates were mounted on an acrylic as shown in Fig. 2(b). Ti plates were placed in between the parallel plates. During the experiment, four plates were charged in such ways that two parallel plates were charged for certain time keeping the other two plates uncharged. The polarities on the parallel plate sets were changed using a timer controlled switch which changes the direction of the collected fiber on Ti.

2.3. Preparation and shear tests on Ti/PMMA samples
To determine the effect of fiber pattern on the interface fracture toughness of Ti/PMMA samples, unidirectional and bidirectional fibers were deposited on top of the titanium plates.
according to the method described earlier. According to Biomet\textsuperscript{HV} PMMA cement preparation protocol, PMMA cement was prepared by hand mixing 2.2 grams of PMMA powder with 1.1 ml of methyl methacrylate (MMA) monomer using powder: monomer ratio of 2:1. PMMA was poured on top of the different titanium samples during doughy phase in a custom made mold (Fig 3(a)). The base plate contains (22×12×25) mm curing chamber, which has front, back and top openings. Two ABS plastic blocks were used to cover the front and back sides of the chamber. A custom made clamp was used to restrict the side blocks movement. The top plate can slide freely to the curing chambers using 4 round rods. The top plate has (22×12×23) mm extruded block at the center that can close the top side of the curing chamber and apply pressure during curing. A set of weights were placed at the top plate to provide 60 KPa pressure \[7\]. A plastic sheet was accessed through one of the side blocks in the mold to create pre-crack in the Ti/PMMA samples. The edge crack prepared for Ti/PMMA specimens with and without fibers was in the range of 7 mm ~10 mm. The plastic sheet was removed manually after curing. Ti/PMMA samples were carefully glued with two ABS plastic holders (made using Dimension 3D printer) by cyanoacrylate adhesive. Shear tests were conducted on the Ti/PMMA samples with and without fibers at room temperature and loading rate 0.01 mm/sec using Evex tensile stage (Evex Analytical Instruments Inc., Princeton, NJ). Fig 3(b) shows the fabricated shear test setup with a Ti/PMMA sample. The load and displacement during the fracture tests were continuously recorded until the failure of the specimens.

3. Equations

The $K_{IC}$ values of bone-cement samples were calculated according to Wang and Agrawal \[8\] for a 90° loading angle using:

$$K_{IC} = \frac{P_C^{\gamma}Y \sqrt{\pi a}}{B W}, \quad (1)$$

where $K_{IC}$ is the mode I fracture toughness, $P_C$ is the critical load that breaks the interface, and $\lambda$ is a scale factor determined using:

$$\lambda = \sqrt{\frac{1 - \alpha}{1 - \beta^2}}. \quad (2)$$

The values of $\alpha$ and $\beta$ are Dundurs parameters, which estimate the elastic mismatch across the bi-materials interface \[9\], given by:

$$\alpha = \frac{E_1 (1-v_1^2) - E_2 (1-v_2^2)}{E_1 (1-v_1^2) + E_2 (1-v_2^2)} \quad (3)$$

$$\beta = \frac{E_1 (1-v_2^2) - 2E_2 (1-v_2^2)}{2E_1 (1-v_1^2) + 2E_2 (1-v_2^2)}$$

where $E_1$, $E_2$, and $v_1$, $v_2$ are elastic moduli and Poisson’s ratios of the cement and bone, respectively. In Eq.(1), $\psi$ is a correction factor determined using \[8\]:

$$\psi = e^{4.092(h/W)^{0.616}}, \quad (4)$$
and $Y$ is a shape function determined using [8]:

$$Y = \frac{1}{1 - \rho} \sqrt{\frac{-0.23 + 1.40 \frac{\rho}{1 - \rho}}{1 - 0.67 \frac{\rho}{1 - \rho} + 2.08 \left(\frac{\rho}{1 - \rho}\right)^2}}$$  \hspace{1cm} (5)$$

where $\rho = a/W$. Initial crack length, thickness and width of the specimen are given by $a$, $B$, and $W$, respectively. The fracture toughness of the Ti/PMMA specimen without and with uni-direction fibers as well as without and with bidirection fibers were compared by student’s t-test with significance being determined at the $P < 0.05$ level. The data was analyzed using Microsoft Excel 2010 $t$ test statistical function.

4. Results and Discussion

4.1. Fabrication of Unidirectional and bidirectional fiber

Uni-directional fibers were successfully fabricated using the electrospinning unit having horizontal drum collector. The fiber collections were viewed in stereomicroscope (Fig 4(a)) and scanning electron microscope (Fig 4(b)). Diameter of produced fibers was found to be in the range of 919 nm – 1.25 μm. Bi-directional fibers were successfully fabricated using the electrospinning unit with parallel ground plate collector. The fiber collections were viewed in stereomicroscope (Fig 5). The figure shows the bi-direction pattern of fiber on a carbon tape.

4.2. Interface fracture toughness of titanium/cement samples

Table 1 revealed that the mean value of $K_C$ of Ti/PMMA samples with uni-direction fibers was higher compare to the mean value of $K_C$ of Ti/PMMA samples without fibers. The study also found that the mean value of $K_C$ of Ti/PMMA specimens without and with uni-direction fibers was higher than those samples with bi-direction fibers. Statistical analysis ($t$-tests) indicated that uni-and bi-direction fibers had significantly increase and decrease the $K_C$ values of Ti/PMMA samples compare to the $K_C$ values of Ti/PMMA samples without fibers ($P<0.05$), respectively. Results indicated that the addition of the micron to nano size PCL fibers on Ti improved the quality of Ti/PMMA union, though bi-direction fibers negatively impact the Ti/PMMA union.

5. Conclusion

Both unidirectional and bidirectional fibers were successfully prepared using the fabricated electrospin unit. Several Ti/cement were prepared to measure the effect of fiber and fiber pattern on interface fracture toughness of Ti/PMMA samples. The data shows that fiber has significant influence on the interface fracture toughness of Ti/PMMA samples. Ti/PMMA samples with uni-directional fiber have higher interface fracture toughness compare to samples without fibers and with bi-directional fiber.

Acknowledgements

This publication was made possible by Grant Number P2PRR016478 from the National Center for Research Resources (NCRR), a component of the National Institutes of Health (NIH) and CURE-STEM faculty scholar.
award support from University of Central Oklahoma (UCO). Its contents are solely the responsibility of the authors and do not necessarily represent the official views of NCRR or NIH or UCO.

**Nomenclature**

- $K_{IC}$: Mode I fracture toughness (MPa.m$^{1/2}$)
- $P_C$: Critical load (N)
- $\lambda$: Scale factor (unitless)
- $\psi$: Correction factor (unitless)
- $B$: Thickness of specimen (mm)
- $W$: Width of specimen (mm)
- $a$: Initial crack length (mm)
- $\alpha, \beta$: Dundurs parameters (unitless)
- $E_1$: Elastic moduli of cement (MPa)
- $v_1$: Poisson's ratio of cement (unitless)
- $E_2$: Elastic moduli of Ti (MPa)
- $v_2$: Poisson's ratio of Ti (unitless)
- $h$: Thickness of Ti / cement in bilayer specimen (mm)

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Fig. 1.
(a) Vertical electrospinning unit, (b) collected fibers on Ti plates, and (c) collected fibers on carbon tape.
Fig 2.
(a) Experimental setup for bidirectional fiber. (b) Four parallel charge plates for bidirectional fiber production.
Fig 3.
(a) Fabricated mold used for the preparation of Ti/cement specimens. (b) Evex tensile test stage with Titanium-PMMA cement sample.
Fig. 4.
(a) Fabricated unidirectional fiber viewed under stereomicroscope at 8X and (b) SEM at 1000X magnification.
Fig. 5.
Fabricated bidirectional fibers viewed under stereomicroscope at 8X magnification.
Table 1
Descriptive statistics of the experimental single edge sandwiched Ti/PMMA specimens' data.

| Description              | without fiber | With uni-direction fiber | With bi-direction fiber |
|--------------------------|---------------|--------------------------|-------------------------|
| No of specimen           | 3             | 3                        | 2                       |
| Average width, W (mm)    | 22.76 ± 0.41  | 22.75 ± 0.37             | 22.76 ± 0.44            |
| Average thickness, B (mm)| 12.1 ± 0.03   | 11.93 ± 0.31             | 12.08 ± 0.08            |
| Average crack length, a (mm) | 10.22 ± 0.28  | 9.95 ± 0.26             | 7.92 ± 0.45             |
| Interface fracture toughness | 14.71 ± 1.03  | 20.09 ± 1.26             | 7.48 ± 0.47             |