OPTIMIZATION OF FIBER SHAPES IN BIOCOMPOSITES

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Abstract

by

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Fibers can be used to improve the mechanical properties of acrylic bone cement. However, debonding of the fibers from the matrix due to the poor fiber/matrix interface is a major failure mechanism for such reinforcements. Optimization of the fiber shape can improve load transfer between the fibers and the matrix, thereby providing improved overall mechanical performance. The goals of this study include: (1) Develop an analytical model to evaluate the effects of fiber end geometry on the pullout load and stress distribution; (2) Determine the optimal fiber morphology for maximum stress transfer in composites using optimization and finite element modeling; (3) Fabricate the fibers with optimal morphology determined by the previous step; (4) Manufacture composites reinforced with the optimized fibers and demonstrate improved mechanical properties experimentally.

Analytical solutions were derived to predict the effects of the enlarged end shape on the pullout load and stress distribution. It is shown that the shape of the enlarged end has a significant influence on the stress distribution of the short fiber. A procedure for structural shape optimization of short fibers was developed. The effects of the interfacial bond and fiber orientation were investigated to obtain the optimal fiber shape. The
general optimal fiber shape is a variable diameter fiber (VDF). Due to the mechanical interlock, the VDF can both bridge matrix cracks effectively and improve the composite mechanical properties. Ceramic VDFs were successfully fabricated. Static and fatigue tests were carried out on the VDF reinforced composites. Conventional straight fiber (CSF) reinforced bone cement was also tested for comparison purposes. Results demonstrated that both the stiffness and the fatigue life of VDF reinforced bone cement are significantly improved compared with the unreinforced cement. Also, the fatigue life of VDF reinforced bone cement was significantly longer than that of CSF reinforced cement.

This study shows the feasibility of a novel fiber (VDFs) technology for reinforced polymers. This fiber family significantly improves the fatigue life of bone cement at a very high level of reliability. VDFs could potentially avoid implant loosening due to the mantle fracture of bone cement and delay the need for revision surgery.
CONTENTS

FIGURES ........................................................................................................................... vi
TABLES ............................................................................................................................ xi
ACKNOWLEDGMENTS ................................................................................................ xii

CHAPTER 1 INTRODUCTION .............................................................................................1

1.1 Bone cement in orthopaedics ...................................................................................1
  1.1.1 Material properties of bone cement .................................................................2
  1.1.2 Need for improving bone cement .................................................................3
  1.1.3 Requirements of new techniques in orthopaedics ...........................................4

1.2 Composite reinforcement of bone cement ...............................................................7

1.3 Fiber morphology ...................................................................................................11

1.4 Research objectives and scope .............................................................................13

1.5 Outline ....................................................................................................................14

CHAPTER 2 FIBER-END DEFORMATION EFFECTS IN ENLARGED-END,
FIBER-REINFORCED COMPOSITES ...........................................................................15

2.1 Introduction ..........................................................................................................15

2.2 Methods .................................................................................................................18
  2.2.1 Statement of problem .....................................................................................18
  2.2.2 Analytical solution .........................................................................................20
  2.2.3 Numerical procedure ......................................................................................26

2.3 Results and discussion .........................................................................................28
  2.3.1 Calibration of Ke by finite element analysis .................................................28
  2.3.2 Effects of the fiber end shape on the tensile stress distribution in the
      fiber shaft and fiber end displacement .................................................................32
  2.3.3 Effects of the fiber embedded length .............................................................34
<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.4 Conclusions</td>
<td>35</td>
</tr>
<tr>
<td>CHAPTER 3 IMPROVEMENT OF MECHANICAL PROPERTIES OF BONE CEMENT BY OPTIMIZATION OF FIBER SHAPES</td>
<td>37</td>
</tr>
<tr>
<td>3.1 Introduction</td>
<td>37</td>
</tr>
<tr>
<td>3.2 Methods</td>
<td>39</td>
</tr>
<tr>
<td>3.2.1 Finite element model</td>
<td>39</td>
</tr>
<tr>
<td>3.2.2 Numerical optimization technique</td>
<td>41</td>
</tr>
<tr>
<td>3.2.3 Geometry modeling/design variable definition</td>
<td>44</td>
</tr>
<tr>
<td>3.2.4 Process execution</td>
<td>47</td>
</tr>
<tr>
<td>3.3 Results</td>
<td>49</td>
</tr>
<tr>
<td>3.4 Discussion</td>
<td>54</td>
</tr>
<tr>
<td>3.5 Conclusions</td>
<td>57</td>
</tr>
<tr>
<td>CHAPTER 4 SHAPE OPTIMIZATION OF RANDOMLY ORIENTED SHORT FIBERS FOR BONE CEMENT REINFORCEMENTS</td>
<td>58</td>
</tr>
<tr>
<td>4.1 Introduction</td>
<td>58</td>
</tr>
<tr>
<td>4.2 Methods</td>
<td>60</td>
</tr>
<tr>
<td>4.2.1 Finite element model and boundary conditions</td>
<td>60</td>
</tr>
<tr>
<td>4.2.2 Optimization strategy</td>
<td>62</td>
</tr>
<tr>
<td>4.3 Results and discussion</td>
<td>62</td>
</tr>
<tr>
<td>4.3.1 Effects of fiber orientation on the center unit cell boundary conditions</td>
<td>62</td>
</tr>
<tr>
<td>4.3.2 Interfacial effects</td>
<td>64</td>
</tr>
<tr>
<td>4.3.3 Effects of the orientation angle on the optimal fiber shape</td>
<td>65</td>
</tr>
<tr>
<td>4.4 Conclusions</td>
<td>70</td>
</tr>
<tr>
<td>CHAPTER 5 MANUFACTURE OF VARIABLE DIAMETER FIBERS</td>
<td>72</td>
</tr>
<tr>
<td>5.1 Introduction</td>
<td>72</td>
</tr>
<tr>
<td>5.2 Design of VDF synthesis and manufacture</td>
<td>74</td>
</tr>
<tr>
<td>5.2.1 Materials compatibility experiments with the spin mix and spin bath</td>
<td>74</td>
</tr>
<tr>
<td>5.2.2 VSSP process control</td>
<td>75</td>
</tr>
<tr>
<td>5.3 Fiber synthesis and manufacture</td>
<td>76</td>
</tr>
</tbody>
</table>
7.2.1 Fiber shape optimization................................................................. 127
7.2.2 VDF fabrication ........................................................................... 128
7.2.3 Analytical model in crack shielding .............................................. 130

BIBLIOGRAPHY ..................................................................................... 131
FIGURES

Figure 1.1 Scanning electron micrograph of carbon fiber-PMMA matrix interface zone(780X) (Pilliar, Blackwell et al. 1976). ...............................................................10

Figure 2.1 Sketches of ¼ model shapes of fibers: (a) blunt fiber end; (b) hemispherical fiber end (hemi-nose); (c) V-shaped fiber end; (d) ellipsoidal shape; (e) “dogbone” shape. ........................................................................ 16

Figure 2.2 Schematic drawing of the pullout model.......................................................... 19

Figure 2.3 Geometry of the enlarged fiber end................................................................ 21

Figure 2.4 Schematic of the axisymmetric model used in the finite element (FE) analysis and the dimension parameters associated with the enlarged-end fiber and the surrounding matrix. ........................................ 27

Figure 2.5 Calibration of Ke using the FEA pullout force. The pullout force was normalized with respect to the case when major to minor axis ratio $\rho = 1$.................. 29

Figure 2.6 The relationship between the spring coefficient (Ke) and the major to minor axis ratio ($\rho$) when $L/r_f = 20$ ($L$: fiber embedded length; $r_f$: fiber radius). Ke was normalized with the case $\rho = 1$ ................................................. 30

Figure 2.7 Comparison of the pullout force between the numerical results and the analytical predictions with the same values of Ke shown in Fig. 2.6. The pullout force was normalized with the case when major to minor axis ratio $\rho = 1$ and Ef = 340 GPa................................................................. 31

Figure 2.8 Effects of the fiber embedded length on the pullout force. The pullout force was normalized with the pullout force of the flat-end fiber when $L/r_f = 10$ ($L$: fiber embedded length; $r_f$: fiber radius). ................................................. 32

Figure 2.9 Normalized tensile stress distribution along the embedded fiber length. The tensile stress was normalized by the maximum tensile stress of the flat-end fiber. ................................................................. 33

Figure 2.10 Normalized fiber end displacements of different enlarged-end fibers under the same pullout force. The displacements were normalized by the fiber end displacement of straight fiber under the same pullout force........ 33
Figure 3.1 Representative volume element model of straight short fiber in a matrix. ..... 40

Figure 3.2 Stiffness based model with boundary conditions. ........................................... 43

Figure 3.3 Pullout energy based model with boundary conditions. ............................ 44

Figure 3.4 Perturbation vectors for shape optimization of the fiber. The vectors change the nodal coordinates of the fiber thereby changing is width, or diameter, in the model. ................................................................. 45

Figure 3.5 Schematic diagram of StudyWizard/ABAQUS shape optimization process. 47

Figure 3.6 Fiber shapes: (a) initial shape – CSS fiber; (b) optimal shape in case of weak bond interface – TES fiber; (c) optimal shape in case of perfect interfacial bond – HBS fiber. ................................................................. 50

Figure 3.7 von Mises stress in the stiffness models: (a) CSS with a weak interfacial bond; (b) TES with a weak interfacial bond; (c) CSS with a perfect interfacial bond; (d) HBS fiber with a perfect interfacial bond. Stress unit is N/m². ........................................................................................................... 51

Figure 3.8 Stiffness comparison for initial and final shapes of fiber with different fiber/matrix interface conditions. ................................................................. 51

Figure 3.9 von Mises stress contours of the pullout energy models: (a) BSS fiber with a weak interfacial bond; (b) TES fiber with a weak interfacial bond; (c) CSS with a perfect interfacial bond; (d) HBS fiber with a perfect interfacial bond. Stress unit is N/m². ........................................................................................................... 53

Figure 3.10 Pullout force comparison for different fiber shapes and fiber/matrix interface conditions. ................................................................. 53

Figure 4.1 The composite is regarded as an array of unit cells containing a tilting fiber embedded in the matrix, center unit cell was used for shape optimization. 61

Figure 4.2 Displacement distribution along the edges of the center unit cell. The displacements were normalized with the length of the unit cell. U1 and U2 are the x and y displacement, respectively. ................................................................. 63

Figure 4.3 Optimal fiber shapes for different fiber orientation angles. ........................... 67

Figure 4.4 Variable Diameter Fiber (VDF)-general optimal fiber shape created by superimposing the optimal geometries generated in the different fiber orientation angles. ................................................................. 68

Figure 4.5 Effective Young’s Modulus of PMMA matrix reinforced with straight short fibers, the optimal fibers at specific orientation angle and the VDFs. 69
Figure 5.1 Schematic diagram of the Viscous-Suspension-Spinning Process

Figure 5.2 Block diagram of system setup

Figure 5.3 Viscosity and pH of alumina slurries with varying dispersant concentration. (a) Viscosity of alumina slurry (70 wt.%) vs. dispersant concentration at different shear rates (b) pH value of alumina slurry (70 wt.%) vs. dispersant concentration

Figure 5.4 Viscosity and pH value control of zirconia slurry by changing the dispersant concentration (a) Viscosity of zirconia slurry (75 wt.%) vs. dispersant concentration at different shear rates (b) pH value of zirconia slurry (75 wt.%) vs. dispersant concentration

Figure 5.5 Schematic of a Variable Diameter Fiber (VDF)

Figure 5.6 Schematic diagram of a peristaltic pump

Figure 5.7 Optical micrographs of (a) green ceramic VDF formed by VSSP and (b) sintered ZrO2 VDF at 1555°C for 30 min

Figure 5.8 Microstructure of Al2O3 VDFs of the composition, 70 wt.% A16 alumina powder, 29.3 wt.% water and 0.7 wt.% dispersant (a) after firing at 1600°C for 15 minutes and (b) after firing at 1650°C for 15 minutes

Figure 5.9 Microstructure of Al2O3 VDFs after firing at 1650°C for 15 min (a) without MgO powder and (b) with 0.2 wt.% MgO powder

Figure 5.10 Microstructure of ZrO2 VDFs of the composition, 75 wt.% zirconia powder, 24.5 wt.% water and 0.5 wt.% dispersant, after firing at 1555°C for 30 min

Figure 5.11 Characterization of (a) zirconia and (b) alumina VDFs

Figure 5.12 Flow rate vs. the peristaltic pump speed for the spin mix

Figure 6.1 Dimensions of the tensile specimen used in this study, corresponding to ASTM D638-98, Specimen Type II. All units are in mm

Figure 6.2 Dimensions of the fatigue specimen used in this study, corresponding to ASTM F2118-01a. All units are in mm

Figure 6.3 The tensile specimen (A) was fixed by two grips, an extensometer (B) was attached to the tensile specimen to measure the strain

Figure 6.4 Fatigue specimen (A) was fixed by specially designed drill chuck (B) in the environment chamber (C)
Figure 6.5 X-ray radiograph of a zirconia fiber reinforced tensile specimens (A) and the control group specimen (B). ................................................................. 96

Figure 6.6 Typical stress-strain behavior of the control group, bone cement reinforced with 2% zirconia CSFs and VDFs, and bone cement reinforced with 5% zirconia CSFs and VDFs. ............................................................................. 97

Figure 6.7 Elastic modulus of unreinforced bone cement (control), bone cement reinforced with 2% and 5% zirconia CSFs and VDFs, respectively (Levels not connected by same letter are significantly different, p < 0.05). 98

Figure 6.8 Ultimate strength of unreinforced bone cement (control), bone cement reinforced with 2% and 5% zirconia CSFs and VDFs, respectively (Levels not connected by same letter are significantly different, p < 0.05). 99

Figure 6.9 Elastic modulus of unreinforced bone cement (control), bone cement reinforced with 2% and 10% alumina CSFs and VDFs, respectively (Levels not connected by same letter are significantly different, p < 0.05). 100

Figure 6.10 Ultimate strength of unreinforced bone cement (control), bone cement reinforced with 2% and 10% alumina CSFs and VDFs, respectively (Levels not connected by same letter are significantly different, p < 0.05). 101

Figure 6.11 Number of cycles to failure of unreinforced bone cement (control), bone cement reinforced with 2% and 10% zirconia CSFs and VDFs, respectively (Levels not connected by same letter are significantly different, p < 0.05). 102

Figure 6.12 SEM micrograph showing a typical fracture surface for a tensile specimen of control bone cement. ................................................................. 104

Figure 6.13 SEM micrograph showing a typical fracture surface for a tensile specimen of zirconia CSF reinforced bone cement. ................................. 105

Figure 6.14 SEM micrograph showing a typical fracture surface for a tensile specimen of zirconia VDF reinforced bone cement. ................................. 105

Figure 6.15 SEM micrographs showing the fiber debonding lengths of (a) CSFs and (b) VDFs. ................................................................................................. 107

Figure 6.16 SEM micrograph showing cement adhering to the fiber after fracture in a tensile test specimen of zirconia CSF reinforced bone cement. ............... 108

Figure 6.17 SEM micrograph showing a tensile test specimen of zirconia VDF reinforced bone cement: not that the fiber surface features were reproduced on the matrix after pull-out. ............................................... 108
Figure 6.18 SEM micrograph showing a typical fracture surface for the fatigue specimens of the unreinforced bone cement (control). ............................... 110

Figure 6.19 SEM micrograph showing a typical fracture surface for the fatigue specimens of the 2% zirconia CSF reinforced cement................................. 111

Figure 6.20 SEM micrograph showing a debonded CSF in front of a crack.................. 111

Figure 6.21 SEM micrograph showing a typical fracture surface for the fatigue specimens of the 2% zirconia VDF reinforced bone cement ......................... 112

Figure 6.22 SEM micrographs showing typical fracture surfaces for fatigue specimens of the 10% zirconia VDF reinforced bone cement ......................... 114

Figure 6.23 SEM micrographs showing typical fracture surfaces for the fatigue specimens of the 10% zirconia CSF reinforced bone cement......................... 116
TABLES

TABLE 2.1 THE RATIO OF MAJOR TO MINOR AXES OF AN ELLIPSOID...........23

TABLE 2.2 PROPERTIES FOR THE ISOTROPIC FIBER AND MATRIX MATERIALS USED IN THE FINITE ELEMENT MODEL ...................... 27

TABLE 3.1 PROPERTIES FOR THE ISOTROPIC FIBER AND MATRIX MATERIAL USED IN THE FINITE ELEMENT MODEL ........................ 40

TABLE 3.2 COMPARISON OF ANALYTICAL AND FEA RESULTS ....................... 54

TABLE 4.1 INTERFACIAL EFFECTS ON THE COMPOSITE EFFECTIVE STIFFNESS................................................................................................... 64

TABLE 5.1 PARAMETERS FOR PONNDORF HOSE PUMP PX 10 ...................... 82

TABLE 6.1 RATIOS OF ZIRCONIA FIBER (GMS), PMMA (GMS) AND MONOMER (CC) USED TO CREATE TENSILE SPECIMENS............ 90

TABLE 6.2 RATIOS OF ALUMINA FIBER (GMS), PMMA (GMS) AND MONOMER (CC) USED TO CREATE FATIGUE SPECIMENS............. 92

TABLE 6.3 RATIOS OF ZIRCONIA FIBER (GMS), PMMA (GMS) AND MONOMER (CC) USED TO CREATE FATIGUE SPECIMENS............. 92
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1.1 Bone cement in orthopaedics

In the mid of 1950s, Charnley first introduced a self-curing bone cement to orthopaedic surgery. He successfully fixed both the femoral and acetabular components in a total hip replacement surgery using bone cement (Charnley 1960). Since then, bone cement has emerged as one of the premier synthetic biomaterials in contemporary orthopaedics. Now approximately one-half million hip replacement surgeries are performed every year worldwide and 70% of the surgeries were performed using bone cement (Kumar 2000). In most total joint replacement surgeries, including hip, knee and ankle, bone cement is used as a means of fixation of the prosthesis to the bone (Charnley 1970). The main functions of the cement are to transfer body weight and service loads from the prosthesis to the bone and/or increase the load carrying capacity of the prosthesis-bone cement-bone system (Kenny and Buggy 2003). Note that the function of acrylic bone cement is strictly mechanical and no chemical bonding takes place at the prosthesis or bone surfaces. It only fills the space between the bone and prosthesis to transfer the load -- it does not “glue” to the surfaces. Addition of bone cement to the femur creates two new interfaces. One is between the bone cement and metal prosthesis, and the other is between the bone cement and bone. For ultimate success of the total joint
arthroplasty, both of these interfaces and the bone cement itself must withstand the loading of the hip.

1.1.1 Material properties of bone cement

Most commercial bone cements (e.g. Osteobond®, Zimmer, Inc.) consist of a solid and a liquid component. The solid part consists of pre-polymerized poly methylmethacrylate (PMMA) beads ranging in size from 10 to 150 µm. Free radicals of benzoyl peroxide (BPO) are present within the beads as remnants from the emulsion polymerization process by which the beads were manufactured. An additional amount of benzoyl peroxide is mixed with the solid to obtain 1%-2.5% by weight benzoyl peroxide. Noted that the additional benzoyl peroxide is not in the form of free radicals. In addition, bone cements generally contain 10%-15% by weight barium sulfate, zirconia or other additive to provide radiopacity. The liquid component consists mainly of methyl methacrylate (MMA) monomer, with 0.75-2.5% by volume n,n-dimethyl-para-touluidine (DMPT). The amine in the bone cement, DMPT, acts as an accelerator. The liquid component also consists of 50-100 ppm hydroquinone, which inhibits the polymerization reaction within the monomer and allows for storage of the liquid component. On mixing the liquid and solid components of the bone cement, the DMPT reacts with the BPO to generate free radicals, which in turn are used in the addition polymerization of the MMA monomers to form PMMA.

The polymerizing process has several stages, including the (a) sandy stage (b) stringy (low viscosity) stage (c) doughy (increasing viscosity) stage and, after setting, (d) rigid mass stage. In the doughy stage, the curing polymer does not stick to surgical gloves, and can be manipulated by hand, or inserted into a cement gun for injection into the femoral
canal. During the period of time between the onset of the dough period and setting of the bone cement, the temperature of the mixture is increasing, due to the liberation of heat from the breaking of bonds in the monomer. Both the quality of the cement and the temperature it reaches during the curing process are dependent on a number of variables such as the ambient temperature, the ratio of the powder to liquid, the time of placement into the patient, and the size, thickness and mass of the bone cement (Walenkamp and Murray 2001).

As a material, the mechanical properties of bone cement are sensitive to such factors as method of testing, test temperature, environment, method of specimen preparation, and strain or loading rate. Generally, the tensile strength of the final bone cement product is reported to be from 24 to 49 MPa and the elastic modulus is in the range of 1.6 to 4.1 GPa (Lewis 1997).

1.1.2 Need for improving bone cement

Although used universally for many years, PMMA bone cement is still beset with a number of drawbacks including thermal and chemical necrosis of bone tissue, the shrinkage of the cement during polymerization, and a large stiffness mismatch between the cement and the contiguous bone, etc. (Lewis 1997). The main drawback is the role that bone cement has been postulated to play in the aseptic loosening and, hence, clinical life of the arthroplasty. Aseptic loosening is the major cause of implant failure in total hip arthroplasty (THA) in human patients (Goldring, Schiller et al. 1983). The process of aseptic loosening is associated with the formation of a synovial-like membrane at the interface between the bone and the cement. Despite improvements in implant design and surgical techniques, 15% to 20% of all THAs, in humans, require a revision for failed
primary arthroplasties (El-Warrak, Olmstead et al. 2001). Of these, 73.2% are carried out for aseptic loosening of the prosthesis (Malchau, Herberts et al. 1993). Currently, bone cement fracture is regarded as a major factor in the mechanical failure of implant fixation and a primary cause of aseptic loosening (Hertzberg and Manson 1980; Jasty, Maloney et al. 1991; Spector 1992). It is directly related to the mechanical properties of the cement, especially the resistance to fracture of the cement in the mantle at the cement-prosthesis interface or the cement-bone interface.

Furthermore, many investigators concluded that the fatigue failure of PMMA bone cement is an important factor in initiating mechanical failure of the cement mantle (Topoleski, Ducheyne et al. 1990; James, Jasty et al. 1992). Fractographic analysis of retrieved, in-vivo cement obtained after revision surgery has shown failure of the bone cement to be the result of slow fatigue crack growth and rapid fracture near large pores (Topoleski, Ducheyne et al. 1990; Jasty, Maloney et al. 1991; James, Jasty et al. 1992). Thus, improvement of the mechanical properties of bone cement, especially increasing the resistance to fracture and fatigue, remains essential for increasing the longevity of cemented THA.

1.1.3 Requirements of new techniques in orthopaedics

Orthopaedics is a rapidly growing industry with continued growth projections as the world and national population ages. Over the past several decades, the use of structural reinforcement in the human body has become increasingly common. People will live longer and desire to maintain a standard of active living unequaled by previous generations. Consequently, substantially increasing demands are expected to be placed on
the orthopaedic industry to provide improved methods for repairing common fractures and common orthopaedic ailments associated with aging.

Currently, innovation in the orthopaedic industry is aimed in two directions: (i) increasing implant life and (ii) minimizing patient trauma. The primary and secondary modes of failure in most orthopaedic devices are wear and fatigue, respectively. Furthermore, device design has been fully optimized for the present materials. Therefore, significant improvements in implant life (i) will only be achieved through the development of new materials with improved wear and fatigue properties. To reduce the time and trauma associated with orthopaedic surgery (ii), a new technology, minimally invasive orthopaedic implants (MIOIs), is emerging in orthopaedics. By minimizing the extensive patient trauma in traditional, common procedures, MIOIs are changing the way many orthopaedic fractures and ailments are treated. For example, a traditional hip replacement surgery is performed through an incision that is usually about 5 to 8 inches in length. Beneath the incision, the muscle is separated, and the hip joint is exposed. The surgeon then removes the arthritic hip joint, and replaces this with a metal and plastic implant. An extensive level of damage is incurred by the patient during surgery. Complete recovery can take 6-12 months. For patients who undergo this surgery, many die within the first year following the operation, never walk on their own again, or end up in a care facility mostly because of the extensive surgical trauma. Using MIOI techniques, the same surgery (hip replacement) can be performed through two small incisions and with minimal muscle dissection. The surgeon does not look directly at the arthritic hip, rather, he or she uses x-ray guidance in the operating room to position the artificial hip. Both the prosthesis and bone replacement filler material are introduced into the fractured
femur through a small surgical hole that minimizes trauma to the patient. These types of approaches have the potential to dramatically change the treatment of hip maladies and have begun to receive attention in the news media because of the obvious benefit to the patient. A key feature is that the filler material must be injectable in order to be implanted through the small hole. In the future, knee replacement and disc repair techniques are also on the drawing board. However, in all these applications the polymer implant material clearly must support significantly higher loads than in traditional hip surgeries. Such loads could lead to implant failure. Consequently, significant improvement in the mechanical properties, e.g. fatigue life, of cured polymer cement is needed to implement this approach and improve how these orthopaedic surgeries are performed.

To be suitable for use in fracture or joint arthroplasties, optimum synthetic biomaterials must meet several performance criteria: (1) They must have appropriate mechanical properties; (2) they must be biocompatible; and (3) they must be functional, e.g., injectable and affordable. For an ideal bone replacement material, the mechanical properties ought to be similar to, or slightly higher than those of natural bone.

Metallic implants most commonly used today (as well as dense ceramics) have mechanical properties that are typically an order of magnitude greater than natural bone, which may result in a weakened bone and bone-implant interface (Huiskes, Weinans et al. 1989). Efforts to utilize porous ceramics or PMMA bone cement in place of stiffer materials have been limited by the low fracture toughness and fatigue strength of these materials, as well as other biological issues. Thermoplastic polymers offer many potential advantages, but alone are often too weak and compliant for load bearing applications.
Thus, the unique and highly optimized mechanical properties of bone tissue can only be matched by intelligent design of composite materials.

1.2 Composite reinforcement of bone cement

In fiber-reinforced plastics, fibers of higher mechanical strength or stiffness (e.g. ceramics) are used to “reinforce” a polymer matrix of lower strength or stiffness. The standard means to provide the greatest effect of strengthening include: (1) aligning continuous fibers, (2) increasing the volume fraction of fiber reinforcements, and (3) using chemical coupling agents and surface treatments to provide a chemical bond between fibers and matrix (Gibson 1994; Kim and Mai 1998). However, all of these approaches are limited for bone cement reinforcement. Aligned continuous fibers and high fiber volume fractions are not feasible due to requirements that the reinforced bone cement should be injectable in fracture or joint arthroplasties. Thus, reinforcement must be discontinuous (chopped or short fibers) and of a low enough volume fraction (less than 20 volume percentage) in order to facilitate injection through a small opening (≈ 2 cm) prior to curing in vivo. Under these requirements, short fibers are the first choice for bone cement reinforcement. Short-fiber composites have certain advantages. They often have improved strength and stiffness over the unreinforced matrix. In addition, they can be adapted to conventional manufacturing techniques, such as powder metallurgy, casting, molding, drawing, extruding, machining, and welding. As a result, the composite fabrication cost is relatively low, which is an important design criterion. Finally, short-fiber composites also can be made with relatively isotropic mechanical properties and can be easily molded into complex shapes, as required in some applications (Zhu and Beyerlein 2002), such as hip implant cementing.
Thus, many investigators have attempted to improve the fatigue properties and fracture toughness of PMMA using short fibers as a second phase, including polyethylene (Pourdeyhimi and Wagner 1989), hydroxyapatite (Harper, Behiri et al. 1995), PMMA (Gilbert, Net et al. 1995), Kevlar (Wright and Trent 1979; Pourdeyhimi, Wagner et al. 1986), carbon (Pilliar, Blackwell et al. 1976; Saha, Saatdjian et al. 1981; Saha 1983), titanium (Topoleski, Ducheyne et al. 1992) and steel (Fishbane and Pond 1977; Saha and Kraay 1979; Kotha, Li et al. 2004). Much of the early work was summarized by Saha and Pal (Saha and Pal 1984). In this review, comments were made on the quantitative results of these composites. The fibers were mostly in the range of 5 to 15 millimeters long and 5 to 15 micrometers in diameter. Fiber loadings were in the range of 1 to 8 percent. For polyethylene fibers reinforced PMMA cement, modest increases in fracture toughness and bending strength are noted, with a 50% reduction in the modulus (Pourdeyhimi and Wagner 1989). Robinson, etc., reported that 2 volume percent of 1.5 mm graphite fibers lead to a statistically insignificant increase in compression strength. Graphite reinforcement of plain cement produced a 32% increase in fracture toughness (Robinson, Wright et al. 1981). For Kevlar fiber, it was reported that 1% weight percent of 13 mm Kevlar fibers increase the tensile strength of 17% and fracture toughness of 23% (Wright and Trent 1979).

Generally, these techniques have resulted in improvements in mechanical properties including fracture toughness and fatigue resistance over unreinforced PMMA. However, the magnitude of the increase (approximately 30%) is disappointing compared to the large increases found in similar fiber reinforced engineering composites (Robinson, Wright et al. 1981). Research shows that the fracture resistance of a composite is strongly
dependent upon efficient matrix/fiber load transfer through the matrix/fiber interface. In brittle matrix composites, energy can be absorbed from an advancing crack by four basic mechanisms: (1) deviation of the crack from the self-similar direction (which includes the crack following the fiber/matrix interfaces), (2) strengthening the material in front of the crack, (3) pullout and fracture of the fibers both in front of and behind a growing crack and (4) stretching of bridging fibers behind a growing crack (Friis 1994).

For short fiber reinforced bone cement, scanning electron microscopy (SEM) revealed that a poor interfacial bond exists between the fibers and the PMMA matrix. The poor interfacial properties between the fibers and PMMA matrix result in debonding between them (Fig. 1.1). This is a major failure mechanism for fiber reinforced bone cement (Pilliar, Blackwell et al. 1976; Robinson, Wright et al. 1981). In such a composite, the crack may deviate from the self-similar direction to propagate through the weak fiber/matrix interface. This deviation results in an increase in energy absorption in comparison to cracking of the neat cement. However, the lack of an adequate bond between fiber and matrix allows easy fiber pullout, a process requiring much less energy than fiber fracture or more difficult fiber pullout. Failed fiber/matrix interfaces also limit the ability of bridged fibers to absorb energy and/or shield the crack tip stress. A weak fiber/matrix interface improves the fracture toughness modestly, but significantly decrease the strength. This could also explain the lack of a significant increase in ultimate compressive strength of the reinforced cements compared to the cements without fibers (Robinson, Wright et al. 1981; Saha and Pal 1984).

Without good interfacial properties, the advantages of high strength fibers are lost. Therefore, to maximize fracture toughness and decrease the rate of crack propagation, it
may be desirable to form an optimum fiber/matrix interface so that the energy is absorbed in interface fracture and fibers are allowed to slip or pullout. In a fully optimized fracture mechanism, energy is absorbed by the fiber pullout and deformation of the fiber bridging behind the crack, fraction of the fibers in front of the crack and deflection of the crack along the length of the fibers. All modes of energy dissipation are utilized (Friis 1994).

Figure 1.1 Scanning electron micrograph of carbon fiber-PMMA matrix interface zone(780X) (Pilliar, Blackwell et al. 1976).

More recently, chemical bonding between the matrix and fiber has been shown to be a viable way to overcome this limitation (Yang, Huang et al. 1997; Chawla 1998). In polymers reinforced by ceramic fibers, chemical bonding is often limited by differences in the bonding and molecular structure of polymers and ceramics. Thus, common thermoplastic and thermosetting polymers, such as polyethylene (PE) and polymethylmethacrylate (PMMA), will not adhere to ceramic fibers without chemical modifications. Therefore, a variety of surface treatments and/or fiber coatings have been developed to provide a chemical “linkage” between the polymer matrix and ceramic fibers. However, these chemical treatments can become costly, require extra processing steps, and often utilize chemicals or materials mired in regulatory issues, such as silica.
and silanes (Kim and Mai 1998; Vickers and Lieberman 2002). Further, some treatments do no perform well in aqueous environments such as the human body (Ohashi and Dauskardt 2000). In general, these improvements have been avoided in the orthopaedic community because of stringent FDA regulation.

1.3 Fiber morphology

Due to the injectability requirement, short fibers should be used for bone cement reinforcement. However, conventional straight fibers (CSFs) do not provide sufficient strengthening because of poor bonding, the low aspect ratios and the low volume fraction required for injection. In order to reinforce bone cement while permitting injectability prior to curing, new methods need to be developed. Recently, a new approach has been developed to improve load transfer between the fibers and the matrix. The approach involves modifying the geometry of short fibers instead of the chemistry of the fiber/matrix interface (Zhu and Beyerlein 2002). Advantages of this approach include: the use of some ceramics, such as zirconia, which are already accepted in the orthopaedics community, chemical treatments are not required, stable interlocking in an aqueous environment, and easier FDA approval. This investigation explores this strategy in more depth to see if the mechanical properties can be improved enough to warrant use in orthopaedic applications.

The performance of the composite is strongly dependent upon efficient fiber/matrix load transfer through the fiber/matrix interface. Research shows that the fiber/matrix load transfer mechanism can be greatly influenced by fiber geometries. (Phan-Thien 1981; Phan-Thien 1981; Sun, Lin et al. 1983; Cameron 1994; Zhao, Zhou et al. 1995; Shuster, Sherman et al. 1996; Zhu, Valdez et al. 1999; Goh, Mathias et al. 2000; Wetherhold and
Bos 2000; Zhu and Beyerlein 2002; Bagwell and Wetherhold 2003; Tsai, Patra et al. 2003; Zhou, Li et al. 2003; Zhou, Li et al. 2005). In short fiber reinforced composites, a strong interface is desired to effectively transfer load from matrix to fiber. However, with a strong interface, it is difficult to relieve fiber stress concentrations in front of an approaching crack. Such stress concentrations can result in fiber fracture. A weak interface may lead to a significant loss of the composite strength, due to complete interfacial debonding and fiber pullout. A compromise in the interfacial bond strength may lead to a significant loss of the composite strength and only a minimal improvement of the composite toughness. Such a compromise will also compromise the load-carrying potential of short fibers. Therefore, many interfacial problems are unavoidable in conventional short straight fiber reinforced composites, and cannot be completely resolved by modifying the fiber/matrix interfacial properties (Zhu and Beyerlein 2002).

The key to optimize short fiber reinforced composite properties is to obtain both a weak interface and a strong load transfer mechanism from the matrix to the fiber. This can be achieved by modifying the morphology or shape of short fibers instead of modifying the chemistry of the fiber/matrix interface. One interesting proposed fiber morphology has recently been proposed in the literature: bone-shape short (BSS) fibers (Zhu and Beyerlein 2002). The ends of the short fibers were enlarged to effectively transfer load from matrix to fiber by mechanical interlocking. These ends remain anchored in the matrix, supplying considerable crack bridging and load transfer between fiber and matrix (Zhu and Beyerlein 2002). The improved load transfer permits a more efficient utilization of fiber stiffness while still improving toughness. Similar work was done with ductile fibers and proved that fiber pullout energy could be increased by using fiber plasticity
(Wetherhold and Bos 2000). It was also found that the efficiency of ductile fibers in increasing fracture toughness greatly depends on the fiber shape. However, both of studies do not address the question of optimal fiber shape. The shape examined is a function of the fiber manufacturing technique. There should exist an optimum fiber shape to achieve better bonding and more efficient reinforcement by mechanical interlock while maintaining injectability.

Based on economics, processing feasibility and biocompatibility, ceramics, such as zirconia (ZrO$_2$), alumina (Al$_2$O$_3$), and hydroxyapatite (HA), comprise the most logical choices for the reinforcement phase due to a significantly higher stiffness, strength and hardness relative to polymers. Ceramics are also desired over metals due to their biocompatibility, causing significantly less adverse biological reactions in vivo. Ceramics such as ZrO$_2$ and Al$_2$O$_3$ are bioinert, and calcium phosphate ceramics such as HA are favorably bioactive due to their chemical similarity to human bone mineral (Park 1979).

1.4 Research objectives and scope

The goal of this research is to examine the feasibility of a new ceramic fiber technology for bone cement reinforcement. An innovative fiber morphology will be developed to provide enhanced stress transfer between the fiber and matrix and, therefore, enhanced composite strengthening. The effects of fiber morphology on the mechanical properties of the composite will be analyzed theoretically. Based on the theoretical model, a general shape optimization procedure will be developed to determine the optimal fiber shape using finite element method. Finally, the feasibility of both the reinforcement concept and refinement of the fiber manufacturing will be tested experimentally. The fibers with optimal morphology -- the result of shape optimization -- will be developed
and applied to reinforce bone cement. The mechanical performance of the resulting composite will be tested statically and in fatigue.

1.5 Outline

In Chapter Two of this dissertation, an analytical model is developed to evaluate the effects of the fiber end shape, especially a rigid enlarged end, on the reinforcement performance. In Chapter Three, a procedure for structural shape optimization using finite element analysis is presented and applied to improve the load transfer between short fibers and a PMMA matrix by optimization of the shape of the fibers. In Chapter Four, the effects of the fiber orientation and interfacial bond are investigated to obtain the optimal fiber shape for bone cement reinforced with randomly oriented fibers. It is shown that, as opposed to aligned fiber composites where the optimum shape is an enlarged-end fiber, the general optimal fiber shape for randomly oriented fibers is a variable diameter fiber (VDF). In Chapter Five, a process to develop variable diameter fibers (VDFs) is described in detail. The characteristics and properties of ceramic VDFs and their dependence on forming conditions are also studied. In Chapter Six, alumina and zirconia VDFs are incorporated into PMMA bone cement for reinforcement. Static tests and fatigue tests are carried out on short VDFs reinforced bone cement. The effects of the fiber reinforcement on bone cement is analyzed and discussed. Finally, conclusions and future directions for research are discussed in Chapter Seven.
CHAPTER 2

FIBER-END DEFORMATION EFFECTS IN ENLARGED-END, FIBER-REINFORCED COMPOSITES

2.1 Introduction

The performance and fracture resistance of short fiber composites is strongly dependent upon efficient fiber/matrix load transfer between the fibers and the matrix. Due to the role played by the fiber ends in the fiber/matrix stress transfer mechanism, an important feature of a short fiber is the fiber end geometry (Cox 1952; Shuster, Sherman et al. 1996). For example, improved load transfer from fiber to matrix through the enlarged end can lead to better crack tip bridging and an increase in fracture toughness of these materials. To obtain a better load transfer mechanism and better stress distribution, many different fiber end geometries, shown in Fig. 2.1, have been tested with and analyzed (Phan-Thien 1981; Sun, Lin et al. 1983; Cameron 1994; Zhao, Zhou et al. 1995; Shuster, Sherman et al. 1996; Zhu, Valdez et al. 1999; Goh, Mathias et al. 2000; Wetherhold and Bos 2000; Bagwell and Wetherhold 2003; Tsai, Patra et al. 2003; Zhou, Li et al. 2005). Sun, et al. (1983) investigated blunt, semi-circular, V-shape, and wedge-shaped fiber end shapes using finite element analysis (Fig. 2.1a, b, c). The stress concentration was observed to depend on the fiber end geometry. Cameron (1994) used elastic finite element analysis to analyze various fiber configurations. A streamlined
shape is determined to result in 10% lower fiber average tensile stress and 30% lower matrix maximum shear stress (Fig. 2.1d). Goh, et al. (1999, 2000) analyzed the stress distributions in a tapered reinforcing fiber embedded in a plastic matrix and verified by finite element simulation (Figure 2.1c). The effect of the taper was to lower peak stress at the fiber center and make the stress distribution throughout the fiber more even. Shuster, et al. (1996) evaluated the effect of the fiber end geometry on the stress distribution using photoelasticity and finite element analysis. A spherical enlarged fiber end (Fig. 2.1e) helped to improve load transfer ability and delay debonding. Zhu and Beyerlein (2002) evaluated the mechanical behavior of enlarged-end short fibers (Fig. 2.1e) by experimental pullout of aligned fibers from a polyester matrix. The enlarged end remained anchored in the matrix, supplying considerable crack bridging and load transfer between the fiber and the matrix (Zhu and Beyerlein 2002). The increased load transfer permits a more efficient utilization of fiber stiffness while still improving toughness.

Figure 2.1 Sketches of ¼ model shapes of fibers: (a) blunt fiber end; (b) hemispherical fiber end (hemi-nose); (c) V-shaped fiber end; (d) ellipsoidal shape; (e) “dogbone” shape.
Among these various fiber shapes, the most interested typical fiber end geometry is the one with an enlarged round end. These enlarged ends anchor the fiber in the matrix and lead to a significantly higher stress to pull out than that required for flat-end fibers. However, the mechanics of this additional anchorage is not clearly understood. Due to the geometric complexity of the fiber, most of the investigators evaluated the effect of fiber end geometries using numerical methods or experimental methods. There have been few researchers who have estimated the effects of fibers with enlarged end geometry analytically.

To assess the contribution of enlarged-end fibers, a theoretical study on the mechanical behavior of a model system consisting of a short fiber with enlarged ends was reported by Phan-Thien (Phan-Thien 1981; Phan-Thien 1981). In his geometrically simple model, the fiber shaft was treated as an effectively rigid, slender rod and the enlarged end was modeled as a rigid spherical bead. According to the model, substantial increase in the elastic and fracture toughness properties of enlarged-end fiber reinforced composites over that of composites reinforced by the same amount of flat-end fibers was predicted. Similar to Phan-Thien’s model, in Zhao et al.’s model (Zhao, Zhou et al. 1995), the elastic stress distribution of enlarged-end fiber and matrix was analyzed. Results showed that the stress in the fiber was more uniform as the radius of the enlarged end increased. However, in both of these models, the enlarged end was treated as a rigid sphere. Deformation of the enlarged end and the interaction between the fiber shaft and the enlarged end were ignored. In fact, the enlarged end was deformable and the boundary conditions for a sphere embedded inside the matrix alone, used by these authors in their analysis, were much different from a sphere embedded inside the matrix at the
end of a fiber rod. Moreover, shape effects of the fiber end geometry were not considered in their models. Only a special case of fiber end geometry, spherical ends, was analyzed. It is clear that a spherical end is not the ideal geometry for an enlarged-end fiber. Further, the applicability of these models to other shapes is not known. It is expected that the shape of fiber ends will significantly influence the stress distribution and the load transfer mechanisms in these materials. Some insight into the mechanics of more complex shapes is desirable.

In this chapter, an analytical model is developed to evaluate the mechanical behavior of a fiber with an enlarged ellipsoidal end. As a first attempt to better understand the sources of the mechanical anchorage resistance at the embedded end, the enlarged fiber end was treated as flexible and, as such, was represented by a rigid enlarged fiber end and a spring. The spring component is introduced to connect the embedded fiber rod end with the enlarged ellipsoid for simulating deformation of the enlarged end and for simulating the interaction between them (Sujivorakul, Waas et al. 2000). Furthermore, the effects of the enlarged end shape were evaluated with parametrically different ellipsoids. Based on these concepts and assumptions, the pullout resistance and the stress distribution in the fiber shaft with different fiber end shapes were calibrated to finite element model and analyzed.

2.2 Methods

2.2.1 Statement of problem

A pullout model of a fiber with an enlarged end embedded in the matrix is shown in Fig. 2.2. The embedded length of the fiber is L and its radius is \( r_f \), \( E \) is the Young’s modulus, \( \nu \) is Poisson’s ratio, and the subscripts \( f \) and \( m \) express fiber and matrix,
respectively. Differing from Phan-Thien’s model, where only a spherical end is modeled, the enlarged end is modeled by a rigid ellipsoid with two axes a and b that may be unequal. The ratio of major to minor axes $\rho = b/a$, defines the shape of the enlarged end; ends with $\rho = 1$ are spherical, $\rho > 1$ are ‘egg-like’ or prolate ellipsoids, and $\rho < 1$ are ‘mushroom-cap shaped’ or oblate ellipsoids.

Figure 2.2 Schematic drawing of the pullout model.
In Fig. 2.2, F is the pullout force at the tip of the fiber, where a displacement load, \( W(0) \), was applied. For sufficiently low F, there exists a perfect bonding between the fiber and the matrix, so that the no-slip and no-opening bonding holds over all the interfaces including the lateral surface and the bottom. With constant end displacements \( W(0) \), the problem is axisymmetric, so that z is the axis of symmetry and r is the radial distance for the axis.

To account for the deformation of the enlarged fiber end and its interaction with the fiber shaft, a spring component with the spring coefficient equal \( K_e \) is used to connect the fiber shaft end with the enlarged ellipsoid. Because of its role in the model, it is expected that the constitutive property of the spring \( (K_e) \) will depend on the fiber enlarged end shape defined by the ratio of major to minor axes \( (\rho = b/a) \), the ratio of Young’s modulus of the fiber to matrix \( (E_f/E_m) \) and the embedded length of the fiber \( (L) \).

2.2.2 Analytical solution

A. Pullout force experienced by the ellipsoid

To calculate the pullout force of the fiber with an enlarged end, the additional resistance contributed by the enlarged end must be considered. First, the force necessary to displace the center of an ellipsoid by a distance \( U \) in a direction normal to the boundary of a semi-infinite elastic matrix will be derived. If \( P \) is the force needed to effect a displacement \( U \) then the deformation field in the matrix will not differ much from that produced by a point force \( P \) acting at the origin of the ellipsoid (Phan-Thien 1981). The displacement of the ellipsoid surface points in the z direction is given by the Kelvin solution (Phan-Thien 1981; Zhao, Zhou et al. 1995).
\[ u_z = AP \left[ \frac{3 - 4\nu_m}{R} + \frac{z^2}{R^2} \right] \]  

(2.1)

where the Kelvin constant \( A = \frac{1}{16\pi G_m(1-\nu_m)} \), \( G_m \) is the shear modulus of the matrix, \( R \) is the distance of an ellipsoid surface point to the center of the ellipsoid.

If we assume there is no relative displacement between the ellipsoid and the matrix, or the enlarged end bonds perfectly with the matrix within \( \theta_0 \leq \theta \leq \pi \) (Fig. 2.3), the force necessary to displace the center of an ellipsoid with the major axes equal to \( b \) and the minor axes equal to \( a \) by a distance \( U \) in \( z \)-direction can be calculated as follows:

\[
\iint S A P \left[ \frac{3 - 4\nu_m}{R} + \frac{z^2}{R^2} \right] ds = \iint S U ds
\]

(2.2)

Figure 2.3 Geometry of the enlarged fiber end.

With these assumptions, each ellipsoid surface point should satisfy the following equations

\[
\left( \frac{R \cos \theta}{b} \right)^2 + \left( \frac{R \sin \theta}{a} \right)^2 = 1
\]

(2.3)
\[ z = R \cos \theta \]  
\[ ds = 2\pi R \sin \theta \, dl = 2\pi R \sin \theta \sqrt{R^2 + R'^2} \, d\theta \]  

Therefore the left hand side of equation (2.2) can be written as
\[
\int \int s \left( \frac{3 - 4V_m}{R} + \frac{z^2}{R^2} \right) ds = 2\pi A P \int_{\theta_0}^{\pi} (3 - 4V_m + \cos^2 \theta) \sin \theta \cdot \frac{ab \sqrt{a^4 \cos^2 \theta + b^4 \sin^2 \theta}}{\sqrt{(a \cos \theta)^2 + (b \sin \theta)^2}} \, d\theta
\]
and the right hand side of equation (2.2) can be written as
\[
\int \int U ds = 2\pi a^2 b^2 U \int_{\theta_0}^{\pi} \sqrt{a^4 \cos^2 \theta + b^4 \sin^2 \theta} \sin \theta \, d\theta
\]

Let \( t = \cos \theta, \, \theta \in [\theta_0, \pi] \), then \( t \in [\cos \theta_0, -1] \), Thus
\[
P = \frac{ab}{A} \frac{\int_{-1}^{\cos\theta_0} \sqrt{a^4 t^2 + b^4 (1-t^2)} \, dt}{\left( a^2 t^2 + b^2 (1-t^2) \right)^{3/2}} U
\]

Recalling \( \rho = \frac{b}{a} \) and inserting this into equation (2.8)
\[
P = \frac{bH}{A} U
\]

where
\[
H = \frac{\int_{-1}^{\cos\theta_0} \sqrt{t^2 + \rho^4 (1-t^2)} \, dt}{\left( t^2 + \rho^2 (1-t^2) \right)^{3/2}}
\]

It is noted that H is a parameter representing the ellipsoid shape effects, which depends on the ratio of major to minor axes \( \rho = b/a \) of the ellipsoid. When \( \rho = 1 \), the ellipsoid becomes a sphere. In this special case,
\[ H = \frac{1}{(3 - 4\nu_m) + 1/3} \]  

which coincides with the expression given by Phan-Thien (Phan-Thien 1981). Since it is difficult to find the explicit analytical expression for \( H \) in the general conditions, numerical integration (Adaptive Simpson Quadrature) was used to solve the problem.

To evaluate only the effects of fiber end shape, and not fiber end size, the volume of the ellipsoid and all the other parameters, such as material properties, fiber/matrix interface conditions and boundary conditions were kept same. The volume of an ellipsoid can be computed from the formula,

\[ V = \frac{4}{3} \pi a^2 b \tag{2.12} \]

By changing the ratio of major to minor axes (\( \rho = b/a \)) of the ellipsoid (Table 2.1), the pullout resistance exhibited by the ellipsoid can be calculated according to equation (2.9).

<table>
<thead>
<tr>
<th>Minor axes (a/r_f)</th>
<th>3.54</th>
<th>2.81</th>
<th>2.37</th>
<th>2.00</th>
</tr>
</thead>
<tbody>
<tr>
<td>Major axes (b/r_f)</td>
<td>1.06</td>
<td>1.69</td>
<td>2.37</td>
<td>3.33</td>
</tr>
<tr>
<td>Ratio (( \rho = b/a ))</td>
<td>0.3</td>
<td>0.6</td>
<td>1.0</td>
<td>1.7</td>
</tr>
</tbody>
</table>

B. Pullout force of the enlarged-end fiber

Pullout of a long high-modulus fiber embedded in a homogeneous elastic space subjected to a longitudinal extension has been considered extensively by many researchers (Muki and Sternberg 1970; Lawrence 1972; Hsueh 1988; Hsueh 1990; Kim, Baillie et al. 1991; Slaughter and Sanders 1991; Cherepanov and Esparragoza 1995). An efficient analytical solution to the problem of pullout given by Cherepanov and
Esparragoza (Cherepanov and Esparragoza 1995) was used in this study to model the fiber. According to Cherepanov’s model, the displacement and stress distribution along the embedded length can be expressed as the following equations:

\[ W = C_1 \sinh \frac{kz}{L} + C_2 \cosh \frac{kz}{L} \]  

\[ \sigma = \frac{kE_f}{L} \left( C_1 \cosh \frac{kz}{L} + C_2 \sinh \frac{kz}{L} \right) \]  

where \( k = \frac{\sqrt{E_m / E_f}}{\alpha r_f} \sqrt{\frac{1 + \nu_m}{L}} \left( \ln \left( \frac{L}{r_f} \right) \right) \)  

and \( E \) is the Young’s modulus, \( \nu \) is Poisson’s ratio, and the subscripts \( f \) and \( m \) express fiber and matrix, respectively. Parameters \( r_f \) is the radius of the fiber and \( \alpha \) is the fitting constant, respectively (Cherepanov and Esparragoza 1995).

\( C_1 \) and \( C_2 \) are arbitrary constants, which can be found from the natural boundary conditions. The first is the displacement at the free surface, opposite the enlarged end:

\[ W = W(0) \quad \text{when } z = 0 \]  

On the opposite end the effect of an enlarged-end fiber may be represented as a flat-end fiber, which is ideally bonded with the matrix, experiencing the tensile stress, \( \sigma_L \), at the embedded fiber end. This stress \( \sigma_L \) is related to the displacement of the enlarged end through a spring constant. Thus, the boundary condition at the connection between the flat-end fiber and the enlarged ellipsoid is as follows:

\[ \sigma_L A_f = P ; \quad P = K_e \left( W(L) - U \right) \quad \text{when } z = L \]  

where \( A_f \) is the cross-section area of the fiber; \( K_e \) is the spring coefficient.
Substituting equation (2.9) into the boundary conditions (2.17b) one obtains the following equation.

\[ \sigma_e = QW(L) \]  \hspace{1cm} (2.18)

where \[ Q = \frac{bH}{AA_f} \frac{K_e}{bH/A + K_e} \]  \hspace{1cm} (2.19)

Thus, the unknowns \( C_1 \) and \( C_2 \) can be obtained by using equations (2.16) and (2.18) in the following form:

\[ C_1 = -\frac{QLW(0)\cosh(k) + kE_fW(0)\sinh(k)}{kE_f\cosh(k) + QL\sinh(k)} \]  \hspace{1cm} (2.20)

\[ C_2 = W(0) \]  \hspace{1cm} (2.21)

Substituting \( C_1 \) and \( C_2 \) in Eqs. (2.13) and (2.14) by equations (2.20) and (2.21) one obtains:

\[ W = -W(0)\left( \frac{QL\cosh(k) + kE_f\sinh(k)}{kE_f\cosh(k) + QL\sinh(k)} \sinh \frac{kz}{L} - \cosh \frac{kz}{L} \right) \]  \hspace{1cm} (2.22)

\[ \sigma = \frac{kE_fW(0)}{L} \left( -\frac{QL\cosh(k) + kE_f\sinh(k)}{kE_f\cosh(k) + QL\sinh(k)} \cosh \frac{kz}{L} + \sinh \frac{kz}{L} \right) \]  \hspace{1cm} (2.23)

Then the pullout force of a fiber with an enlarged end can be calculated as:

\[ F = \sigma(0)A_f \]  \hspace{1cm} (2.24)

The stress \( \sigma(0) \) can be calculated from equation (2.23) when \( z = 0 \). It is noted for the assumed boundary conditions (2.17) that the embedded end of the plain fiber is not perfectly bonded to the enlarged ellipsoid. The displacement at the embedded end of the plain fiber is theoretically larger than the calculated displacement of the enlarged ellipsoid. Thus, the spring component is introduced at the connection, as shown in Fig.
2.1, to express the relationship at the embedded end of the fiber between the pullout force experienced by the ellipsoid and the different displacements [W(L) - U] in terms of the secant spring coefficient $K_e$. The spring constant, accounting for displacement of the enlarged end, is not included in equation (2.9). In that equation certain assumptions were made, namely that the enlarged end is not deformable and that the enlarged end displacement is well predicted by Kelvin’s solution. The end is, in fact, deformable and as it becomes more elongated perpendicular to the z axis it may bend. Furthermore, the Kelvin solution in no way accounts for the presence of the attached fiber.

The spring constant, $K_e$ defined by equation (2.17b), is a measure of the role of fiber end deformation and fiber-end/fiber-shaft interaction and can be found from a numerical or physical experiment. It is expected that $K_e$ depends on the shape of the fiber enlarged end. We will require that for different fiber enlarged end shapes the value of corresponding pullout force determined by equation (2.24) should coincide with the numerical value obtained from numerical, finite element solutions.

2.2.3 Numerical procedure

The problem of pulling a fiber with enlarged end was also analyzed by means of the finite element method. A typical two-dimensional axisymmetric model is shown in Fig. 2.4. To calibrate the analytical model with numerical results for different fiber end geometries, different ellipsoids with the different ratios of major to minor axes were created as the fiber ends based on Table 2.1.
Figure 2.4 Schematic of the axisymmetric model used in the finite element (FE) analysis and the dimension parameters associated with the enlarged-end fiber and the surrounding matrix.

The symmetrical model (about z-axis) was meshed with 6-node and 8-node quadratic elements (i.e. ABAQUS CAX6 and CAX8 elements). In all FE calculations, both the fiber and matrix materials were assumed to be isotropic and linear elastic. The fiber-matrix interface was assumed well-bonded. The properties of the fiber (Online MatWeb database, http://www.matweb.com) and matrix (Beyerlein, Zhu et al. 2001) used are listed in Table 2.2.

| TABLE 2.2 PROPERTIES FOR THE ISOTROPIC FIBER AND MATRIX |
| MATERIALS USED IN THE FINITE ELEMENT MODEL |
| Constituent | Material  | Young’s Modulus (GPa) | Poisson’s Ratio |
| Fiber        | Alumina   | 340.0                  | 0.22           |
| Matrix       | Polyester | 0.5                    | 0.47           |
Boundary conditions were applied to simulate the pullout test. The boundary condition at the top end of the matrix prevents displacement in the z-direction, i.e., \( u_z = 0 \) at \( z = 2L \). The model was loaded by applying a uniform longitudinal displacement \( W(0) \) to all nodes at the bottom end of the fiber in the negative direction of z-axis, i.e., \( u_z = -W(0) \) at \( z = 0 \) and \( r < r_f \).

The pullout forces produced by differently shaped fibers embedded in a relatively large matrix material \((2L/r_f = 40)\) were calculated from the finite element analyses.

2.3 Results and discussion

2.3.1 Calibration of \( K_e \) by finite element analysis

\( K_e \) is expected to be related to the shape of embedded enlarged end and can be found from the relationship between the fiber forces and the displacements with the different fiber end shapes. For a specific fiber end shape, the pullout force can be calculated from the FE numerical experiments. \( K_e \) can then be calibrated using equation (2.17b).

For the calibration of \( K_e \), the geometric parameters about the fiber enlarged end were taken as listed in Table 2.1, which keep the volume of the ellipsoid as constant. The properties of the fiber and matrix were taken as those listed in Table 2.2. Taking the embedded length, \( L = 20r_f \), \( K_e \) was calibrated by the pullout forces for different fiber enlarged end shapes. The calibration curve of the pullout force for different fiber end shapes is shown in Fig. 2.5 and the corresponding \( K_e \) for each enlarged end shape, which is defined by the ratio of major to minor axis of the ellipsoid \((\rho)\), is shown in Fig. 2.6. \( K_e \) greatly depends on the fiber end shape. As \( \rho \) increases, \( K_e \) decreases, indicating fiber end deformation and the more approximate nature of Kelvin’s solution for this situation. For \( \rho \) close to zero, the spring constant approaches infinity, indicating that deformation of the
fiber end is minimal and Kelvin’s solution is a more accurate approximation of the deformation.

Figure 2.5 Calibration of $K_e$ using the FEA pullout force. The pullout force was normalized with respect to the case when major to minor axis ratio $\rho = 1$. 
Figure 2.6 The relationship between the spring coefficient ($K_e$) and the major to minor axis ratio ($\rho$) when $L/r_f = 20$ (L: fiber embedded length; $r_f$: fiber radius). $K_e$ was normalized with the case $\rho = 1$.

The deformation of the fiber end and the approximate nature of Kelvin’s solution dominates the behavior for $\rho > 1$. Both $E_f/E_m$ and the embedded fiber length affect $K_e$ only slightly. As shown in Fig. 2.7, to estimate the effect of $E_f/E_m$ on $K_e$, the Young’s modulus of the fiber was changed to 170 GPa and 680 GPa. All the other parameters were kept unchanged with respect to the original model. The pullout forces were then calculated by the numerical method and the analytical method. The group of $K_e$ calibrated by $E_f = 340$ GPa is also applicable to other cases with different $E_f/E_m$, which means the effects of $E_f/E_m$, for large values of this ratio, on $K_e$ is very slight. Likewise, the effect of embedded fiber length on $K_e$ was evaluated by changing the aspect ratio ($L/r_f$) as 10, 20, 30, 40, 60 and keeping all the other parameters unchanged according to the original model. $K_e$ is calibrated by the aspect ratio, $L/r_f = 20$. Results show that analytical results fit the numerical results very well for the whole range of length ratios when $K_e$ is calibrated by
the aspect ratio, $L/r_f = 20$ (Fig. 2.8), which shows the embedded length of the fiber almost has no effect on $K_e$. The normalized pullout force of the flat-end fiber with different fiber embedded length is also shown for reference in the figure, which was used to evaluate the effects of the fiber embedded length in later discussion (Section 2.3.3). Clearly, for the range of parameters investigated here $K_e$ is only sensitive to the fiber end shape but not to the material properties and the fiber embedded length.

Figure 2.7 Comparison of the pullout force between the numerical results and the analytical predictions with the same values of $K_e$ shown in Fig. 2.6. The pullout force was normalized with the case when major to minor axis ratio $\rho = 1$ and $E_f = 340$ GPa.
Figure 2.8 Effects of the fiber embedded length on the pullout force. The pullout force was normalized with the pullout force of the flat-end fiber when \( L/r_f = 10 \) (\( L \): fiber embedded length; \( r_f \): fiber radius).

2.3.2 Effects of the fiber end shape on the tensile stress distribution in the fiber shaft and fiber end displacement

Fig. 2.9 shows that the enlarged fiber end can greatly improve the load transfer efficiency between the fiber and matrix due to the mechanical anchorage effect. The maximum tensile stress in the fiber shaft with an enlarged end (\( \rho = 0.3 \)) increases about 40\% compared to the flat-end fiber. More load is supported by the fiber, which helps to better utilize the potential stiffness or strength of the fiber in the composite. Furthermore, as shown in Fig. 2.9 and Fig. 2.10, the enlarged end shape significantly affects the stress and the fiber end displacement and therefore distribution in the fiber shaft. As \( \rho \) decreases, the axial stress, \( \sigma_f \), increases and the displacement of the fiber end decreases significantly. Clearly, an enlarged end is superior to a flat end.
Figure 2.9 Normalized tensile stress distribution along the embedded fiber length. The tensile stress was normalized by the maximum tensile stress of the flat-end fiber.

Figure 2.10 Normalized fiber end displacements of different enlarged-end fibers under the same pullout force. The displacements were normalized by the fiber end displacement of straight fiber under the same pullout force.
The shape of the enlarged end is important as well. For the same volume of the ellipsoid, the maximum tensile stress increases more than 10% as the ratio of major to minor axes decreases from 1.7 to 0.3, which means a fiber with a ‘mushroom-cap shaped’ or oblate ellipsoid fiber end can transfer load more efficiently than a fiber with a ‘egg-like’ or prolate ellipsoid fiber end. Corresponding to the increase of the tensile stress, the fiber end displacement decreases as \( \rho \) decreases. These results are consistent with the experiment results obtained by Wetherhold and Bos (Wetherhold and Bos 2000), who evaluated different fiber end effects on the pullout force and showed that the fiber with a hook end has better improvement than the fiber with an enlarged spherical end on the pullout force. When \( \rho \) becomes a small value, the shape of the ellipsoid will be similar to a “hook” or “T” shape.

From the view of increasing load transfer efficiency, the fiber should be manufactured with a ‘mushroom-shape’ fiber end to maximize the potential strength or stiffness of the fiber to increase of strength or stiffness of the composite. However, stress concentration will be a limiting factor. As the ratio of major to minor axes decreases, the stress concentration at the transition from fiber to enlarged end will increase (Beyerlein, Zhu et al. 2001). To find the optimum fiber shape; i.e., the shape which maximizes the strength increment per unit mass (or volume) of the fibers without causing premature failure in the matrix or fiber, all these factors should be taken into account.

2.3.3 Effects of the fiber embedded length

The fiber embedded length affects the pullout force and the anchorage effect of the enlarged fiber end. Taking \( b/a =1 \) (spherical fiber end), \( E_f = 340 \) GPa and \( L/r_f = 10, 20, 30, 40, 60 \), the effects of fiber embedded length on the pullout force and the anchorage
effect of the enlarged fiber end were shown in Fig. 2.8. As the embedded length increases, the pullout force increases for both the enlarged-end fiber and the flat-end fiber. However, the difference of the pullout force between the enlarged-end fiber and the flat-end fiber decreases. It is expected that the strengths of the enlarged-end fiber and the flat-end fiber reinforced composites should approach the strength of continuous fiber reinforced composites as \( L \) increases further. In that case, the load is transferred mainly by a shear mechanism through the lateral interfaces parallel to the load direction. For the assumption of a perfect bond, made here, the load transferred by the mechanical interlock at the enlarged end becomes negligible. There is no extra anchorage effect of the fiber with enlarged end over the flat-end fiber when the length of the fiber is long enough. However, for an imperfect bond, or a failed bond, the effects may remain significant.

2.4 Conclusions

In this chapter, a pullout model for the fiber with enlarged end was developed. A spring component was integrated in the new model to account for the effects deformation of the enlarged end. The spring coefficient, \( K_e \), was calibrated by the numerical analysis. The effects of the fiber enlarged end shape, the ratio of Young’s modulus of the fiber and matrix \( (E_f/E_m) \) and the embedded length of the fiber \( (L/r_f) \) on the constitutive property of the spring \( (K_e) \) were considered. It is found that the constitutive property of the spring \( (K_e) \), and therefore the contribution of the deformation of the end to the resistance to pullout, greatly depends on the fiber enlarged end shape. However, the ratio of Young’s modulus of the fiber and matrix \( (E_f/E_m) \) and the embedded length of the fiber \( (L) \) only exert a small influence for the range of values examined here. Thus, previous models
have only limited application to enlarged-end fiber shapes other than a sphere or oblate ellipsoid. This is unfortunate because these are the least effective reinforcement shapes.

From the model, it is seen that the enlarged end shape has a significant influence on the distribution of the axial stress in the fiber. The axial stress in the fiber will increase as the ratio of major to minor axes ($\rho = b/a$) decreases, indicating better load sharing between fiber and matrix. The stress concentration at the point of attachment of the fiber shaft to its enlarged end will limit the allowable increase in this stress.

Finally, it is seen that for perfect bonding between the fiber and matrix, as assumed here, the anchorage effect of the enlarged fiber end will decrease as the embedded length of fiber increases because more load is transferred by a shear mechanism through the lateral interfaces on the shaft instead of through the mechanical interlock at the enlarged end. For imperfect bonds, the effects of the end may remain significant. Enlarged ends, therefore, are expected to be useful for all fibers when bonding is weak and only for short fibers ($L_\rho/r_f \leq 20$) when bonding is strong.
CHAPTER 3
IMPROVEMENT OF MECHANICAL PROPERTIES OF BONE CEMENT BY
OPTIMIZATION OF FIBER SHAPES

3.1 Introduction

As mentioned earlier, orthopaedic bone cements are predominantly based on poly(methylmethacrylate) PMMA, which has been the widespread choice for decades. The main function of the cement is to transfer stress from the prosthesis to the bone (Deb 1999). However, bone cement can fail mechanically because of its inherently poor intrinsic mechanical properties (Saha and Pal 1984). To improve the performances of PMMA, numerous attempts have been made at using composite reinforcement as introduced in Chapter 1. These additions have resulted in improvements in mechanical properties including fracture toughness and fatigue resistance over unreinforced PMMA. However, when compared with other fiber-reinforced composites, the improvements in fracture toughness and fatigue resistance for the fiber reinforced PMMA is relatively small (Robinson, Wright et al. 1981; Saha and Pal 1984). Scanning electron microscopy reveals that the poor interfacial properties between the fibers and PMMA matrix resulting in lower improvement in performance (Pilliar, Blackwell et al. 1976; Krause and Mathis 1988).
One interesting strategy to overcome weak bonding has recently been proposed in the literature: bone shape short (BSS) fibers (Zhu and Beyerlein 2002). These fibers have a constant cross-section except at their ends, where the section “balloons” in diameter. These ends remain anchored in the matrix, supplying considerable crack bridging and load transfer between fiber and matrix (Zhu and Beyerlein 2002). The improved load transfer permits a more efficient utilization of fiber stiffness while still improving toughness. Similar work was done with ductile fibers and proved the concept of increasing fiber pullout energy by using fiber plasticity (Wetherhold and Bos 2000). The efficiency of ductile fibers in increasing fracture toughness greatly depended on the fiber shape. Tsai et al. (Tsai, Patra et al. 2003) investigated stress profiles induced during pullout of two chosen shaped head families using a finite element method based numerical scheme. However, neither of these studies addresses the question of optimal fiber shape.

There should exist an optimum fiber shape at which composites will have the largest improvement in mechanical properties. Such increases in performance should be accomplished without sacrificing other desirable characteristics such as the overall fiber-matrix mix ratio, which may incur a composite density or cost penalty (Cameron 1994).

There is previous work on fiber shape using finite element analysis (FEA) which modified the cross-sectional geometry of continuous fibers using circular, ellipsoidal, kidney and "X" shape cross-sections (Brown and Lee 1992). The study concluded that fiber dominated composite properties are not affected by fiber cross-sectional geometry and matrix dominated properties are only moderately affected (Brown and Lee 1992).
This work eliminated the variable of cross-sectional geometry for improving load transfer. Therefore, research should mainly focus on geometry modifications along the axis.

Realizing the limitations of the model presented in chapter 2, namely limited allowable geometries and the assumption of perfect bonding between fiber and matrix, this chapter presents a procedure to optimize fiber shape using finite element analysis method. The composite is modeled by a representative volume element (RVE) composed of a single short fiber embedded in the PMMA matrix with symmetry boundary conditions. Considering that only poor bonding exists between the fiber and PMMA matrix in a real cement composite, contact elements between the fibers and matrix were used to simulate poor bonding, i.e., the real situation in the composite. Residual stress, due to matrix cure shrinkage and/or thermal mismatch, was also included.

3.2 Methods

A modularized shape optimization software system was developed. The system combined Altair’s optimization modules STUDYWIZARD (Altair Engineering Inc. 2001) and the finite element (FE) software package ABAQUS (ABAQUS Inc. 2001). The execution was controlled by the main program of STUDYWIZARD which manages the entire optimization run. In what follows, descriptions of the various components of the optimization process are presented.

3.2.1 Finite element model

A two-dimensional, plane stress representative volume element was used to model the composite. The element consists of a single short fiber embedded in the PMMA matrix.
Since there is symmetry about the x-axis and the y-axis, only one quarter of the actual model was used in the finite element (FE) calculation, as shown in Fig. 3.1.

![Diagram of Fiber/Matrix interface](image)

**Figure 3.1** Representative volume element model of straight short fiber in a matrix.

The FE model was meshed with 4-node elements (i.e., ABAQUS CPS4 elements (AB AQUS Inc. 2001)). Totally, there are 32181 elements and 33024 nodes in the model. Both the fiber and matrix materials were assumed to be isotropic and linear elastic. The properties of the fiber (Online MatWeb database, http://www.matweb.com) and PMMA matrix (Lewis 1997) used are listed in Table 3.1.

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Material</th>
<th>Young’s Modulus (GPa)</th>
<th>Poisson’s Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber</td>
<td>Alumina</td>
<td>340.0</td>
<td>0.22</td>
</tr>
<tr>
<td>Matrix</td>
<td>PMMA</td>
<td>2.81</td>
<td>0.33</td>
</tr>
</tbody>
</table>

The fiber/matrix interfaces of the composite materials are represented by frictional contact between the fibers and PMMA matrix (Freund 1992; Povirk and Needleman 1993). In order to better analyze the micromechanical characteristics of the fiber/matrix interfaces, the contact-element method is introduced at the fiber/matrix interfaces. The
contact element is an imaginary element (ABAQUS Inc. 2001). The change of modulus of the contact element reflects different contact status, i.e., contact or no contact.

For the fiber/matrix interfaces of actual composite materials, there is some normal force at the interface because of a thermoelastic mismatch between the fiber and the matrix (Freund 1992). Such a mismatch strain can arise as a result of high temperature processing of the materials and subsequent cooling to room temperature (Li, Wang et al. 2004). In general, the interfacial normal residual stress due to these effects is non-zero, \( \sigma_{Ns} \neq 0 \) and the fiber/matrix interface is in compression contact before the fiber is subjected to loading. When the tensile stresses \( \sigma_N \) of the contact elements are larger than the residual stress \( \sigma_{Ns} \), i.e. \( \sigma_N > \sigma_{Ns} \), the fiber/matrix interface will lose contact or debond. The interface is assumed to be in Coulomb frictional contact after the debonding has taken place and the coefficient of friction is taken as \( \mu \), which depends on the material properties of the contact pair (ABAQUS Inc. 2001). For a fiber reinforced PMMA matrix, it is assumed that \( \sigma_{Ns} = 8.0 \) MPa (Li, Wang et al. 2004) and \( \mu = 0.1 \) (Mckellop, Clarke et al. 1981). The fiber, matrix and fiber/matrix interface are discrete as in Fig. 3.1. In these FE calculations, the fiber is embedded in the representative volume element and the volume fraction of fiber \( (V_f) \) is constrained to 10\% \( (V_f = 0.1) \).

3.2.2 Numerical optimization technique

A shape optimization problem can generally be formulated as a constrained minimization problem, as follows.

Minimize: \( F(X) \) \hspace{1cm} (3.1)

Subject to: \( g_j(X) \leq 0, \ j = 1, m \) \hspace{1cm} (3.2)
\[
X = \begin{bmatrix}
  x_1 \\
  \vdots \\
  x_n
\end{bmatrix}, \quad x_i^L \leq x_i \leq x_i^U, \quad i = 1, n \tag{3.3}
\]

where \( X \), in this case, represents the design variable vector, \( g \) is a vector of constraints.

Shape design variables can be nodes or control points, which directly or indirectly define the boundary shape of the structural component. The objective function \( F(X) \) reflects the optimization goal. In design optimization of the fiber morphology, the design goal could be defined at different levels. To evaluate the bonding ability between the fiber and PMMA matrix, the apparent stiffness of the composite can be used as the objective function. Under the same load conditions, higher stiffness means a greater effect of the fibers, which suggests more load transfer to the fiber by fiber/matrix interface, corresponding a better interface bonding. With regard to fracture toughness, fibers can bridge the fracture surface, providing a decrease in the net stress intensity factor or providing an increase in energy dissipation through bridging often with fiber pullout, fiber fracture or plasticity (Wetherhold and Bos 2000). Improving the crack bridging ability of the fibers can enhance the fracture toughness of the composite. If the shape of the fibers can be optimized in such a way that the short fiber would pull out with increased difficulty, much more energy would be consumed during crack propagation, which will significantly improve the toughness (Zhu, Valdez et al. 1998). In this case, the objective function would be the work to pullout fibers.

In this study, we examined two objective functions with two different load conditions. To evaluate the stiffness of the model, the composite Young’s Modulus was used as the objective function. In this case, boundary conditions are applied to simulate the stress state generated under remote uniform tension (Fig. 3.2). The boundary condition along
the y-axis, which is a line of symmetry, prevents displacement in the x-direction. While the boundary condition along the top and bottom side of the model prevent the displacement in the y-direction. The model was loaded by applying a constant longitudinal displacement $\Delta L$ to all nodes at the right end of the matrix in the x direction. The value of the displacement chosen was 0.01 times the model length L, i.e. $\Delta L=0.01L$. The composite modulus for the finite element model was calculated using the method of mechanics of materials as shown in equation (3.4)

$$F(X) = E_z = \frac{\sigma}{\varepsilon} = \frac{PL}{A(\Delta L)} \quad (3.4)$$

In equation (3.4), $A$ is cross-sectional area and $P$ is the total reaction force at the right end of the matrix in the x direction.

<table>
<thead>
<tr>
<th>Fiber/Matrix interface</th>
<th>Matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber</td>
<td></td>
</tr>
</tbody>
</table>

Figure 3.2 Stiffness based model with boundary conditions.

To evaluate the fracture toughness of the composite, the pullout energy was used to address crack initiation, stable crack growth and the onset of rapid fracture (Katoozian and Davy 2000). In this case, a pull out test was simulated to optimize the fracture toughness of the composite (Fig. 3.3). The value of the work done to pull out the fiber, i.e. the pull out energy at failure, can be calculated from this simulation by evaluating the area under the true stress-strain curve. Thus, the total pull out energy $U$ of the fiber materials was used as the objective function. Boundary conditions are applied to simulate
the pull out test. The boundary condition at the right end of the matrix prevents displacement in the x-direction. While the boundary conditions along the top and bottom side of the model prevent displacement in the y-direction. The model was loaded by applying a constant longitudinal displacement $\Delta L$ to all nodes at the left end of the fiber in the negative direction of x-axis. The value of the displacement chosen was 0.01 times the model length $L$, i.e. $\Delta L = 0.01L$. The pullout energy was calculated in equation (3.5)

$$ F(X) = U = \frac{1}{2} \Delta L \sum_{i=1}^{N} |f_i| $$  \hspace{1cm} (3.5)

where $f_i$ is the reaction force of each node at the left end of the fiber, $N$ is the nodes number at the left end of the fiber.

![Figure 3.3 Pullout energy based model with boundary conditions.](image)

To evaluate the impact of fiber/matrix interface on the object function, a similar model with perfect bond interface was also calculated for shape optimization using the same objective function in each case.

3.2.3 Geometry modeling/design variable definition

Shape optimization generally requires that alternative shapes for the structure be specified. For the FEA, these shapes may be represented by a set of displacement vectors, which are known as perturbation vectors and form the design space (Garcelon, Balabanov...
Domain elements, which are independent from the finite element elements, are used to define regions of shape change in the structure. Perturbation vectors are applied at the exterior nodes of the domain elements. In Fig. 3.4, one can see the 20 domain elements used here. The finite elements are not shown for clarity due to high element numbers. Similar to isoparametric finite elements, domain elements have shape functions that define a displacement field within the elements. The interior nodes are interpolated by the domain element’s perturbation based on the shape function. The relation between vector perturbations and the nodal movement are expressed as following.

\[
x^{(s)} = x^{(0)} + \sum p_j \frac{dx}{dp_j}
\]

where, \(x^{(s)}\) is the updated node coordinates; \(x^{(0)}\) is the initial node coordinates; \(p_j\) is the value of the \(j^{th}\) design variable and \(\frac{dx}{dp_j}\) is the shape perturbation vector. The design variables are the values that determine the amount of the perturbations in the design process. The optimizer finds a linear combination of these perturbation vectors that represents the optimal design (Schramm and Thomas 1998). Here, perturbation vectors were chosen as the width or diameter of the fiber at selected locations along the length of the fiber. The diameter between those locations was interpolated from the diameters at those locations.

Figure 3.4 Perturbation vectors for shape optimization of the fiber. The vectors change the nodal coordinates of the fiber thereby changing is width, or diameter, in the model.
In a preliminary study, several sensitivity analyses were performed for the objective function. Results of the sensitivity analysis were used to gain insight into the optimization process and to identify the important design region for the objective function. Then domain elements and perturbation vectors were defined based on the sensitivity analysis results. Sensitivity analysis results showed that the shape of the fiber end had the largest and most important effect on the objective function. Consequently, more domains were concentrated at the end of the fiber. A total of 20 quadrature domains were defined in the fiber (Fig. 3.4). To avoid gaps in the fiber/matrix interface, the same number domains were defined in the matrix. The perturbation vectors were applied at the corner nodes of each domain element. Linear shape functions were used to interpolate the interior nodes of the domain. Fig. 3.4 shows the domain elements and perturbation vectors for the finite element model. Corresponding to the perturbation vectors, there were 21 design variables in this problem, which are the magnitudes of the perturbation vectors.

To remove fiber volume fraction effects, the change of fiber volume fraction ($\Delta V_f$) was limited to 0.0, $\Delta V_f \leq 0.0$.

$$\Delta V_f = \frac{1}{2} \sum_{i=1}^{n} w_i (p_i + p_{i+1}) \leq 0.0$$

(3.7)

where $w_i$ is the width of the domain (Figure 3.4), $p_i$ is the value of the $i^{th}$ design variable, $n$ is the number of domains.

To keep the geometry of the fiber realistic, inequality constraints were applied to the design variable perturbation vectors. The allowable fiber diameter was constrained to be between 10% and 190% of the initial straight fiber diameter ($d_f$). The following inequality constraints were used:
\[ -0.9d_j \leq p_j \leq 1.9d_j, \quad j = 1,21 \]  
\[ (3.8) \]

3.2.4 Process execution

Shape optimization was performed in this study using ABAQUS and StudyWizard. StudyWizard is a general-purpose optimization software package. Fig. 3.5 shows the operational flow of the numerical optimization strategy (StudyWizard) which makes use of ABAQUS FEA results for shape optimization. The shaded region shows the tasks performed by StudyWizard.

Figure 3.5 Schematic diagram of StudyWizard/ABAQUS shape optimization process.

To calculate the objective function \( F(X) \), a polynomial \( \psi(X) \) of degree \( q \) is introduced, termed the response surface, such that:
\[ F(X) \approx \psi(X) \]  
\[ \psi(X) = a_{i0} + \sum_{j=1}^{n} a_{ij} X_j + \sum_{j=1}^{n} \sum_{k=1}^{n} a_{ijk} X_j X_k + K \]

where,

- \( i = 1, m+1 \)
- \( j, k = 1, n \)

with the number of constraints \( m \), the number of design variables \( n \), and the polynomial coefficients \( a_{i0}, a_{ij}, a_{ijk} \).

The sequential response surface methodology searches for the optimum design in a local region. The response surface is constructed locally and is used to determine the next design iteration. The process is repeated until the sequence of approximate optimization have converged. In other words, the models do not attempt to characterize the objective function in the entire solution space but rather concentrate in the local area that the search is currently exploring.

In shape optimization, StudyWizard retrieves ABAQUS results and calculates the constraint values and objectives. The polynomial coefficients are determined using a least squares fit of the response surface function on these analysis results. The optimization procedure uses the Sequential Response Surface method and is as follows (Schramm and Thomas 1998):

1. Analyze the initial design and \( n \) perturbed designs \((1+n)\).

2. A least squares technique is used to determine the polynomial coefficients for the objective and each of the constraint functions.
• If the number of designs analyzed is 1+n, the linear coefficients are determined, resulting in a linear response surface RS1.

• As each of the next n designs are analyzed, the quadratic coefficients, are determined.

• If the number of designs analyzed are 1+n+(n+1)/(n/2), the designs are weighed to calculate coefficients to give the quadratic response surfaces (RS2, RS3, RS4, etc.).

3. Solve for the approximate optimum design using mathematical programming.

4. Analyze the approximate optimum design.

5. If the designs have converged then stop.

6. If the designs have not converged, go to step 2.

Sequential response surface methodology searches the optimum designs in a localized way. The response surface is used to determine a search strategy for moving to the estimated gradient direction and the process is repeated. In other words, the models do not attempt to characterize the objective function in the entire solution space but rather concentrate in the local area that the search is currently exploring. In each iteration, StudyWizard updates design variable values and converts these values into new node coordinates [equation. (3.6)], i.e. a new fiber shape, by modifying the ABAQUS input file.

3.3 Results

Results were obtained using the two objective functions discussed previously. For both of them, the optimization process converged after about 150 iterations. The conventional straight short (CSS) fiber, shown in Fig. 3.6(a), was used as the starting
design. For the case of stiffness optimization with a weak fiber/matrix interface, the typical final geometry after optimization is shown in Fig. 3.6(b), which is a threaded end short (TES) fiber. When the perfect bond was assumed in the fiber/matrix interface, a hyperbolic short (HBS) fiber was obtained, shown in Fig. 3.6(c). The von Mises stress contours are shown in Fig. 3.7(a) to (d) for the initial and optimal shapes of the two different interface conditions, weak interfacial bond and perfect interfacial bond. Fig. 3.8 illustrates the stiffness of PMMA matrix reinforced with the CSS fiber (Bar A), TES fiber (Bar B) with a weak bond interface and CSS fiber (Bar C), HBS fiber (Bar D) with a perfect bond interface. The total reaction force was normalized by the total reaction force of CSS fiber reinforced PMMA matrix with a perfect bond interface. In the case of a weak interfacial bond, the objective function was improved by 61% and reached 91% of the system with a perfect interfacial bond. However, in the case of a perfect interfacial bond, the objective function was improved less than 7.4% when compared with the initial CSS fiber.

Figure 3.6 Fiber shapes: (a) initial shape – CSS fiber; (b) optimal shape in case of weak bond interface – TES fiber; (c) optimal shape in case of perfect interfacial bond – HBS fiber.
Figure 3.7 von Mises stress in the stiffness models: (a) CSS with a weak interfacial bond; (b) TES with a weak interfacial bond; (c) CSS with a perfect interfacial bond; (d) HBS fiber with a perfect interfacial bond. Stress unit is N/m².

Figure 3.8 Stiffness comparison for initial and final shapes of fiber with different fiber/matrix interface conditions.
For pullout energy optimization, similar optimal fiber shapes were reached as those in the stiffness based optimization. Since a CSS fiber with a weak interfacial bond exhibits almost zero resistance to fiber pull-out, a bone shape short fiber (Zhu and Beyerlein 2002) (BSS fiber with the same volume percent) reinforced model with a weak bond assumed at the interface was used to evaluate the effects of the fiber shape optimization. Fig. 3.9(a) to (d) show the von Mises stresses contours of the composite reinforced with the BSS fiber, TES fiber with a weak interfacial bond and CSS fiber, and HBS fiber with a perfect interfacial bond. Fig. 3.10 illustrates the pullout force of the fiber in a PMMA matrix for the CSS fiber (Bar A), TES fiber (Bar B), and BSS fiber (Bar C) with a weak interfacial bond and CSS fiber (Bar D), HBS fiber (Bar E), and BSS fiber (Bar F) with a perfect interfacial bond. The total pullout force was normalized with respect to the total pullout force of a CSS fiber in PMMA matrix with a perfect interfacial bond. In the case of a weak interfacial fiber/matrix bond, compared with the CSS fiber, the BSS fiber showed a large improvement in fiber pullout loads and reached about 56% of the pullout energy of a CSS fiber with a perfect interfacial bond. For the optimal fiber shaped TES fiber, the objective function is about 30% more than a BSS fiber and reached 86% of the CSS fiber with a perfect interfacial bond. However, in the case of a perfect interfacial bond, the objective function was changed less than 8% when compared with the initial CSS fiber. No significant difference of pullout energy exists among CSS fiber, BSS fiber and HBS fiber reinforced composite.
Figure 3.9 von Mises stress contours of the pullout energy models: (a) BSS fiber with a weak interfacial bond; (b) TES fiber with a weak interfacial bond; (c) CSS with a perfect interfacial bond; (d) HBS fiber with a perfect interfacial bond. Stress unit is N/m².

Figure 3.10 Pullout force comparison for different fiber shapes and fiber/matrix interface conditions.
In all cases, the fiber volume fraction after optimization was always less than or equal to the initial value. This indicates that the improvement in objective function is due to the change in fiber geometry rather than an increase in volume percent.

3.4 Discussion

In this study, the composite was modeled with a representative volume element, which is the same as the modified Cox model used by Hwang and Gibson (Hwang and Gibson 1987). Based on the assumption of equal stresses in each element, the modulus of the modified Cox model is

\[ E_c = \frac{E_{cm}E_m}{\nu_mE_{cm} + \nu_{cm}E_m} \]  \hspace{1cm} (3.11)

where \( E_{cm} \) is the modulus of Cox model, \( E_m \) is modulus of the matrix, \( \nu_{cm} \) is the volume fraction of the original Cox model and \( \nu_m \) is the matrix volume fraction (Hwang and Gibson 1987). The analytical result calculated by Equation (3.11) and the FEA result were listed in Table 3.2. Comparing the results, it is clear that the FEA model conforms to the classical solution for CSS fibers, which verified the validity of the FEA model used in this study.

<table>
<thead>
<tr>
<th>TABLE 3.2 COMPARISON OF ANALYTICAL AND FEA RESULTS</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Modified Cox model</strong></td>
</tr>
<tr>
<td>Modulus (GPa)</td>
</tr>
</tbody>
</table>

Two different design objectives, stiffness-based and pullout energy-based, have been used to optimize the shape of short fibers used to reinforce PMMA bone cement. Typically, the optimum fiber contour looks like a bone shape with many threads in the
enlarged end. These threads significantly enhance load transfer between the fiber and the PMMA matrix by mechanical interlock.

In case of stiffness-based optimization, the von Mises stresses generated in the TES fiber are much higher than those in the CSS fiber and are similar to the von Mises stresses generated in the CSS fiber with a perfect interfacial bond (Fig. 3.7). These results show that more load is transferred from the PMMA matrix to the TES fiber through the threads in the enlarged end. Also, two characteristics are observed for these threads. First, all the threads are concentrated at the embedded end of the fiber. Second, the amplitude of the threads decreases from the peak (about \(2d_f\) from the fiber end) at the inner edge of the threaded region to the minimum as one moves toward the end of the fiber. The reason for this outcome is that for discontinuous fiber reinforced composites with a weak interface, the total force needed to slide the fiber against frictional resistance is independent of the length of the fiber, and load transfer is actually localized at the end of the fiber (Freund 1992). To improve the efficiency of load transfer, most shape changes focus on the fiber end, which results in many threads at the end of the fiber. On the other hand, discontinuities provided by fiber ends can produce stress concentrations on nearby fibers and promote matrix microcracking at these ends (Zhu, Valdez et al. 1999). These stress concentrations can induce fiber failure and lower the stiffness of the composite. However, low amplitude, rounded threads at the fiber end may help to reduce the stress concentration.

In the pullout energy-based objective function, mechanical interlocking between the TES fiber and PMMA matrix helps to prevent the TES fiber from pullout and force the TES fiber to deform instead, which results in high strain energy density in the TES fiber.
Comparing the von Mises stress contours of the models reinforced with BSS fiber and TES fiber, Fig. 3.9(a) and (b), it can be seen that higher strain energy density is generated in the TES fiber than the BSS fiber. As a result, the TES fiber will carry more load, eventually deform plastically, work harden, and fail. These mechanisms significantly increase resistance to crack growth and the maximum fracture toughness of the composite (Zhu, Beyerlein et al. 2001). Also, due to mechanical interlocking, load transfer between the matrix and the TES fiber is more efficient and occurs more rapidly along the length of the TES fiber. Thus, the aspect ratio for maximum load transfer will be much less for TES fibers. For example, in bolts complete load transfer from nut to bolt is well known to occur in the first two or three threads (Bickford 1995). Here, it is possible to reduce the aspect ratio of the fiber, making it more injectable, without losing mechanical properties.

The influence of the fiber/matrix interface was also evaluated. Results indicate that the fiber/matrix interface has an important effect on the objective function. If a perfect interfacial bond was assumed, the objective function is only slightly affected by shape optimization of the fiber. The reason for this is that the assumption of a perfect interfacial bond predetermines the load transfer properties in the analysis (Pisanova, Zhandarov et al. 2001). However, the properties of interfaces largely govern the mechanical properties of short fiber composites (Zhu and Beyerlein 2002). To get a reliable optimization result, the effect of the fiber/matrix interface must be considered; the two extremes are the weak bond and perfect bond assumption used here. Studies with both a perfect bond and a weak bond are needed to define the limiting behavior for various bond strengths.
Obviously, the mechanism of load transfer between the fiber and matrix was changed for the TES fiber reinforced composites. The load transferred from the matrix to the fiber was not solely by friction force in the interface as in the CSS fiber but also by mechanical interlock between the fiber and the matrix. In this case, the composite mechanical properties are not governed by fiber/matrix interface but depend more on the mechanical properties of fiber and matrix materials themselves.

In interpreting the results, it should be noted that there are several limitations to this study. To change the fiber shape, the domain elements and perturbation vectors were defined along the fiber axis. Consequently, the frequency of the threads at the fiber end shown here is dependent on the domain elements. Also, stress concentration at the thread tip and root can be high. Certainly, these high stresses around the threads may lead to fracture or failure in the matrix or the fiber and material for fiber and matrix should account for this new failure mode. In spite of these limitations, we are confident that the work gives valid insights into the ability of shape optimization of short fibers to enhance the performance of bone cement.

3.5 Conclusions

This study presents a procedure for structural shape optimization using finite element analysis and applies it to improve the load transfer between short fibers and a PMMA matrix by optimization of the shape of the fibers. The results demonstrate that a TES fiber can significantly improve load transfer between the fibers and the matrix by the introduction of efficient mechanical interlock.
CHAPTER 4
SHAPE OPTIMIZATION OF RANDOMLY ORIENTED SHORT FIBERS FOR BONE CEMENT REINFORCEMENTS

4.1 Introduction

In the previous chapter, we developed a procedure for structural shape optimization using finite element analyses and applied it to the optimization of single short fiber morphology. It was shown that an enlarged-end short fiber with many threads is desirable. In most of the previous studies on fiber morphology (Phan-Thien 1981; Shuster, Sherman et al. 1996; Zhu and Beyerlein 2002; Tsai, Patra et al. 2003; Zhou, Li et al. 2005), the fibers are assumed to be perfectly parallel to the load direction and perfectly bonded to the matrix. The random orientation of the fibers and the interfacial bond were not considered. In real materials, short fibers are not perfectly parallel to the load and the behavior of the composite can be expected to vary with the degree of fiber misalignment. Takao et al. (Takao, Chou et al. 1982) showed that the fiber orientation angle $\alpha$ greater than $15-20^\circ$ had a great effect on the composite stiffness. Thus, the effects of fiber orientation on the mechanical behavior of the composite must be considered. It has also been confirmed that the interfacial bond significantly influences the tensile properties of short fiber reinforced composites (Kerans, Hay et al. 1989). Therefore, the effects of fiber/matrix interfacial bond must also be taken into account.
To investigate the mechanical response of a composite containing randomly oriented fibers, finite element methods have proven to be a powerful tool. Finite element methods can be used to estimate composite properties usually by the analysis of a representative volume element (RVE) corresponding to a periodic fiber packing sequence. The effective elastic moduli of the composite are determined by finite element analysis of the RVE. However, it is paramount in such analyses that the correct boundary conditions be imposed on the RVE such that they simulate the actual deformation within the composite (Sun and Vaidya 1996). Under longitudinal and transverse normal loading, a typical RVE with fibers perfectly parallel to the load direction can deform in such a way that the boundaries remain plane (no distortion). However, when the fibers are misaligned with the load direction, the deformed boundary of RVE does not remain plane any more (Sun and Vaidya 1996). In this case, the appropriate boundary conditions considering distortion effects for the corresponding RVE must be applied to the finite element model.

In this chapter, a procedure for structural shape optimization of short reinforcement fibers using finite element analyses was developed. The composite is regarded as an array of RVEs composed of a tilting fiber embedded in the matrix. This method provides proper boundary conditions on the center RVE, which was used for the shape optimization. The objective of the optimization is to maximize the stiffness of the reinforced bone cement. Considering that only poor bonding exists between the fiber and PMMA matrix, a thin interfacial layer was inserted into the model between the fiber and matrix to simulate the weak bond in the composite (Chang, Bell et al. 1987; Termonia 1990). The fiber orientation was varied to determine the optimal fiber shape for bone cement reinforced with randomly oriented fibers.
4.2 Methods

The modularized shape optimization software system developed in Chapter 3 was used here. Only changes to the finite element model were necessary; the geometry was changed, plane strain conditions were assumed and a more exact interface model was implemented.

4.2.1 Finite element model and boundary conditions

The fiber orientation is expected to have a significant influence on the boundary conditions of the RVE. The composite is regarded as a 3x3 array of RVEs composed of a tilting fiber embedded in the matrix. In this embedded cell model, the center unit cell is surrounded by other 8 similar unit cells (Fig. 4.1). This technique was able to provide the proper boundary conditions for the center unit cell, and the boundary conditions are more conveniently applied to the outer RVEs to simulate the actual deformation within the composite. During the shape optimization process, the surrounding 8 unit cells are kept identical to the center unit cell by tracking any morphology change of the center short fiber in real time. As shown in Fig. 4.1, the center unit cell has the initial dimensions L and D. The short fiber orientation is characterized by the angle $\Phi$ between the loading direction and the fiber axis. The aspect ratio (a) of the short fiber with length l and diameter d is defined as $a = l/d$ and its volume fraction $V_f$ is equal to $V_f = ld/LD$. In this investigation, the short fiber aspect ratio is set to 8 and $L/D$ is equal to 5/4. The volume fraction $V_f$ is equal to 10%. In order to consider the effect of interfacial bond on the composite mechanical behavior, a thin interfacial layer was inserted into the model to simulate the interfacial bond, as shown in Fig. 4.1. The thickness of the interfacial layer $t_i$ was 0.1d. The strength of the interfacial bond was represented by the mechanical
properties of the interfacial layer (Chang, Bell et al. 1987; Termonia 1990). The elastic modulus of the interfacial layer $E_i$ can be used to represent the interfacial bond strength (Chang, Bell et al. 1987; Termonia 1990). In the model with interface, as shown in Fig. 4.1, the interfacial bond was represented only by the mechanical properties of the interfacial layer. When $E_i$ was equal to matrix modulus $E_m$, the bond was considered to be perfect. When $E_i$ was less than $E_m$, the bond can be considered to be weak. This kind of interface model is called “soft interface” ($E_i < E_m$) (Chang, Bell et al. 1987). In this study, $E_i$ is defined to be equal to or smaller than $E_m$.

![Figure 4.1](image)

**Figure 4.1** The composite is regarded as an array of unit cells containing a tilting fiber embedded in the matrix, center unit cell was used for shape optimization.

Boundary conditions were applied to simulate the stress state generated under remote uniform tension. For the cases $\Phi = 0^\circ$ and $\Phi = 30^\circ$, the finite element model was loaded by uniaxial tension in the longitudinal direction, i.e., the boundary condition along the y-axis, which is a line of symmetry, prevents displacement in the x-direction. While the boundary condition along the top and bottom of the model prevents the displacement in
the y-direction. A uniform distributed load $P$ was applied to the right edge of the matrix in the x direction. The value of the distributed load $P$ chosen was 100 MPa. For the cases $\Phi = 60^\circ$ and $\Phi = 90^\circ$, the analyses were performed by subjecting the same cells as the cases $\Phi = 30^\circ$ and $\Phi = 0^\circ$ to uniaxial tension in the transverse direction, respectively.

The FE model was meshed with 3-node and 4-node elements, i.e. CPE3 and CPE4 elements in ABAQUS. Both the fiber and matrix materials were assumed to be isotropic and linear elastic. The properties of the fiber (Online MatWeb database, http://www.matweb.com) and PMMA matrix (Lewis 1997) used are listed in Table 3.1.

### 4.2.2 Optimization strategy

The same optimization strategy as that outlined in Chapter 3 was used here. Only the stiffness of the composite was optimized, however, since pullout is no longer relevant.

### 4.3 Results and discussion

#### 4.3.1 Effects of fiber orientation on the center unit cell boundary conditions

The displacement distribution along the edges of the center RVE are shown in Fig. 4.2. When the fibers are perfectly parallel to the load direction, i.e. $\Phi = 0^\circ$, the deformed boundary remains plain (Fig. 4.2a). However, as the orientation angle $\Phi$ changes, the displacement distribution along the edges of the center unit cell is distorted. All the distorted edges resemble a sine curve. More distortion is observed on the left and right edges compared to the top and bottom edges. For the left and right edges, the amplitude of the distortion is about 10% of the averaged nodal x-displacement when $\Phi = 30^\circ$ (Fig. 4.2b). For the top and bottom edges, the amplitude of the distortion is about 4% of the averaged nodal y-displacement. These results indicate that the fiber orientation has a
significant influence on the unit cell boundary, which can distort the unit cell. Therefore, the proper boundary conditions considering these distortion effects must be applied to the finite element model to obtain reliable results.

Figure 4.2 Displacement distribution along the edges of the center unit cell. The displacements were normalized with the length of the unit cell. U1 and U2 are the x and y displacement, respectively.

(a) $\Phi = 0^\circ$

(b) $\Phi = 30^\circ$
4.3.2 Interfacial effects

The elastic modulus of the interfacial layer $E_i$ can be used to represent the interfacial bond (Chang, Bell et al. 1987; Termonia 1990). In the model with interface, as shown in Fig. 4.1, the adhesion between interface and matrix or fiber was assumed to be perfect. The interfacial bond was represented only by the mechanical properties of the interfacial layer. When $E_i$ was equal to the matrix modulus $E_m$, the bond was considered to be perfect. When $E_i$ was less than $E_m$, the bond can be considered to be weak. This kind of interface model is called “soft interface” ($E_i < E_m$) (Chang, Bell et al. 1987). In this study, $E_i$ is defined to be equal to or smaller than $E_m$.

The effects of varying fiber/matrix interfacial strength on the composite stiffness are shown in Table 4.1. Results indicate that the increasing of $E_i$ increases the stiffness of the composite. This affirms that a stronger interfacial bond enhances stress transfer between matrix and fiber; a weak bond reduces stress transfer to the fiber.

<table>
<thead>
<tr>
<th>$E_i/E_m$</th>
<th>0.1</th>
<th>0.2</th>
<th>0.5</th>
<th>0.8</th>
<th>1.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Straight fiber (GPa)</td>
<td>5.18</td>
<td>5.97</td>
<td>6.75</td>
<td>7.02</td>
<td>7.12</td>
</tr>
<tr>
<td>Optimal fiber (GPa)</td>
<td>6.49</td>
<td>7.25</td>
<td>7.97</td>
<td>8.22</td>
<td>8.27</td>
</tr>
<tr>
<td>Improvement ratio (%)</td>
<td>25.4</td>
<td>21.3</td>
<td>18.0</td>
<td>17.2</td>
<td>16.1</td>
</tr>
</tbody>
</table>

The mechanical properties of the composite can be improved by modifying fiber shape with optimization design in spite of a weak bond. In the case of a weak fiber/matrix interface, the composite stiffness is 72.8% of the composite stiffness with a perfect
fiber/matrix interface before shape optimization. The optimized stiffness can reach to 91% of a perfect bond after shape optimization.

The interfacial bond also has an important influence on the shape optimization effect. In the case of a weak interfacial bond, the objective function was improved by 25.4% comparing with the initial straight short fiber. However, in the case of a perfect interfacial bond, the objective function was improved by only 16.1%. Since the magnitude of $E_i$ represents the state of the interfacial bond, the above-mentioned results show that when the interfacial bond is weak, the shape optimization effect on the composite modulus is more significant.

4.3.3 Effects of the orientation angle on the optimal fiber shape

To evaluate the orientation effects, the shape optimization process was performed under different orientation angles ($\Phi = 0, 30, 60, 90^\circ$). The conventional straight short fiber was used as the starting design. The typical final geometries after optimizations are shown in Fig. 4.3(a) – (d), which correspond to the different fiber orientation angles. When the angle equals to $0^\circ$, the optimal fiber geometry looks like a dumbbell shape (Fig. 4.3a). The fiber “balloons” in diameter at the ends. Due to the role played by the fiber ends in the fiber/matrix stress transfer mechanism, an important feature of a short fiber is the fiber end geometry (Cox 1952; Shuster, Sherman et al. 1996). Most loads are transferred through the fiber end for short fiber reinforced composites with fibers aligned along the loading direction. To enhance this effect, increasing the diameter of the short fiber ends helps to anchor the short fiber in the matrix, supplying considerable crack bridging and load transfer between fiber and matrix. Similar fiber geometry was first introduced by Zhou (Zhao, Zhou et al. 1995) and he found that the composites reinforced
with dumbbell-shaped steel wires have higher strength than those reinforced by straight wires. Since then, Zhu and co-workers (Zhu and Beyerlein 2002) have reported much progress in developing bone-shaped short fibers using different composites. However, this fiber shape was determined by inspiration from biology (Zhao, Zhou et al. 1995) and was a function of the fiber manufacturing technique. The question of optimal fiber shape was ignored. Here, the dumbbell-shape fiber is confirmed to be the optimal fiber geometry for providing the maximum composite stiffness when the fiber direction is parallel to the loading direction, i.e. $\Phi = 0^\circ$. 
Figure 4.3 Optimal fiber shapes for different fiber orientation angles. 

The effect of orientation angle on the composite modulus and the optimal fiber shape were also examined. As the orientation angle increases, more enlarged knots are generated along the middle of the short fiber span. When the orientation angle equals to 90°, most of the enlarged knots are concentrated at the middle of the fiber. These enlarged knots can help to anchor short fibers within the matrix and improve the load transfer efficiency between the fibers and matrix when aligned off the axis of loading.
To determine the optimal fiber geometry for randomly distributed short fibers in reinforced bone cement, the optimal geometries generated in the different orientation angles were superimposed to create the final geometry, as shown in Fig. 4.4. This final geometry is a fiber of varying diameter, i.e., Variable Diameter Fiber or VDF. A variable diameter fiber is defined as a fiber that changes diameter along its length, much like a threaded bolt, in order to provide greater mechanical interlocking between the fiber and polymer matrix. The advantages of such fibers as they are described here are multiple. Due to mechanical interlocking, VDFs do not rely solely on shear at the fiber/matrix interface to transfer load from matrix to fiber. Consequently, VDFs facilitate an increase in the amount of strengthening attainable for any given fiber volume fraction, length, or degree of orientation, relative to standard straight fibers of fixed diameter. Furthermore, increased strengthening is provided without chemical bonding between fiber and matrix. Therefore, virtually any fiber can be used to reinforce any matrix without the need for coupling agents or surface treatments.

Figure 4.4 Variable Diameter Fiber (VDF)-general optimal fiber shape created by superimposing the optimal geometries generated in the different fiber orientation angles.
Fig. 4.5 illustrates the stiffness of PMMA matrix reinforced with the straight short fibers, the optimal fibers at specific orientation angle and the VDFs. The improvement of the objective function depended on the fiber orientation angles. The maximum improvement occurred when the orientation angle equals to 0°. In this case, the objective function was improved by 25.5%. While the minimum improvement occurred at Φ = 90° where it was only 11.4%. Note that in each fiber orientation the VDF, shown in Fig. 4.4, is about as good as the optimal fiber, shown in Fig. 4.3, and is superior to the straight fiber.

![Graph showing Young's Modulus vs Orientation Angle](image)

**Figure 4.5** Effective Young’s Modulus of PMMA matrix reinforced with straight short fibers, the optimal fibers at specific orientation angle and the VDFs.

In interpreting the results, it should be noted that there are several limitations to this study. To accurately illustrate the overall behavior of the composite, a three-dimensional (3D) analysis is required. In the present paper, a 2D plain strain model of misaligned short fibers is analyzed. The plane strain assumption, which is made to reduce the computational requirement, means that the fibers are really continuous in the direction
perpendicular to the plane. Even with this limitation, the results can be used for qualitative predictions of the effects of fiber misalignment on the behavior of short fiber reinforce PMMA matrix composites, which is the topic here. To change the fiber shape, the domain elements and perturbation vectors were defined along the fiber axis. Consequently, the position of the knots at the fiber is dependent on the domain elements. Also, stress concentration at the knot tips and roots can be high. Certainly, these high stresses around the knot may lead to fracture or failure in the matrix or the fiber. In spite of these limitations, we are confident that the work gives valid insights into the ability of shape optimization of short fibers to enhance the performance of bone cement.

Finally, it is necessary that experiments be conducted to confirm the results obtained by finite element analysis here and in the previous chapter. This is especially important as there are some stress values that do increase and may change the failure mode from that which was assumed. Also, the real-world effects of volume fraction ratios and the stress interactions between closely packed fibers that arise should be assessed. Clearly, in order to do that a technique for manufacturing such fibers must be developed. The Viscous Suspension Spinning Process (VSSP) for fiber manufacture (French and Cass 1998; Zhu and Beyerlein 2002) offers a feasible manufacturing technique for producing ceramic fibers with a variable diameter and in future studies this technique will be exploited to produce optimally shaped fibers for experimental evaluation.

4.4 Conclusions

This chapter presents a procedure for structural shape optimization of short reinforcement fibers using finite element analyses. Fiber orientation resulted in distortion of the unit cell boundary. Therefore, boundary conditions considering these distortion
effects must be applied to the finite element model to obtain reliable results. Representing composite structures as an array of RVEs was shown to be an efficient and accurate method in simulating the actual deformation within randomly oriented fiber composites.

Shape optimization of the fiber shape can greatly improve load transfer efficiency between the fiber and PMMA matrix, and further, greatly improve the mechanical properties of the reinforced bone cement. The fiber/matrix interfacial bond has an important influence on the shape optimization effect. The weaker the interfacial bond, the stronger the shape optimization effect on the composite modulus.

The effects of fiber orientation were evaluated by performing the shape optimization process under different orientation angles. The optimal fiber geometry looks like a dumbbell shape when the fiber is perfectly aligned to the loading direction. As the orientation angle increases, more enlarged knots were generated along the short fiber toward the middle of the fiber span. Variable Diameter Fiber (VDF) -- the general optimal fiber shape for randomly distributed short fibers reinforced bone cement is achieved by superimposing the optimal geometries generated in the different orientation angles.

Due to the mechanical interlock between the VDFs and the matrix, it is expected that VDFs can both bridge matrix cracks effectively and improve the stiffness of the composite when fibers are randomly oriented.
5.1 Introduction

Advanced Cerametrics, Inc. (ACI), of Lambertville, NJ, has developed the Viscous Suspension Spinning Process (VSSP), a patented fiber processing technology (Cass 1991; Cass, Loh et al. 1998; French and Cass 1998). The process is capable of producing continuous ceramic fibers of virtually any ceramic material. For example, ACI has demonstrated the robustness of the process by application to a wide variety of ceramic materials, including alumina (Al$_2$O$_3$), yttria stabilized zirconia (Y-ZrO$_2$), lead zirconate titanate (PZT), and hydroxyapatite (HA). A schematic of the process is shown in Fig. 5.1 and a brief summary of the process follows.

![Figure 5.1 Schematic diagram of the Viscous-Suspension-Spinning Process.](image)
The VSSP begins with ball milling the ceramic powder in water to obtain a slurry. This slurry is then mixed with viscose, a cellulose xanthate dissolved in aqueous caustic soda, pressure filtered to remove particle agglomerates, and de-aired under vacuum to prevent filament porosity and breakage. The spin mix is then precision metered through a spinneret (an extrusion die with hundreds of microscopic holes), which is immersed in a spin bath, and the individual filaments are formed. The spin bath is a warm, aqueous acid solution with salts. The acid acts to coagulate the spin mix and regenerate the cellulose by releasing xanthate groups. At the same time, the salt acts to dehydrate the viscose in the precursor. This dehydration provides a diameter reduction without excessive stretching that could cause the ceramic particles to be pulled apart. A second salt in the spin bath also acts to retard the cellulose regeneration process in order to improve fiber strength. The fiber is stretched slightly in the regeneration bath where hot dilute acid completes the cellulose regeneration reaction. Thus, green fibers are formed containing ceramic particles within aligned cellulose molecules. The green fibers are washed to remove residual acids and salts. The green fibers are finally passed through a finish bath, which provides lubricity and prevents fraying in the tow, is then dried, and wound on a take-up reel for further textile processing.

The green fiber diameter is dependent upon the spinneret hole diameter, but it can be increased or decreased with a given spinneret hole diameter or by changing the pump pressure or the fiber spinning speed. Thus the filament diameter can be tailored to any application. The green fibers are ductile and have a large breaking strain. They can be woven into fabrics or wound onto mandrels suitable for composite manufacture. They can also be bundled, braided or chopped. Dense ceramic fibers are produced by burning
off the cellulose binder at 550°C, and sintering at high temperatures. The high temperature treatment also results in a thorough cleaning of the fibers so that chemical residues are removed. The final sintered fiber is clean, dense, and strong. Ceramic fibers of 8-250 µm are produced from green fibers of roughly twice that diameter.

In the manufacturing process developed by ACI, challenging, yet reasonable, modifications to the existing system control can facilitate a change from producing constant diameter fibers to VDFs.

5.2 Design of VDF synthesis and manufacture

For the purpose of making VDFs, experiments were performed on the VSSP process by the author during an internship at Advanced Cerametrics, Inc. Several methods for modifying the VSSP process were explored and challenges associated with them were addressed. In the end, it was found that controlled variation of the pump pressure could be successfully used to produce suitable VDFs for this study.

5.2.1 Materials compatibility experiments with the spin mix and spin bath.

Each of the candidate materials must be prepared in slurry form to determine particle size, pH, viscosity, and reactivity with the viscose. If reaction is observed modifications to each of the parameters are performed until a VSSP-compatible spin mix can be produced. Drops of the spin mix must be immersed in the spin bath and the rate of coagulation measured. If the rate is too fast or too slow, the spin bath and spin mix should be altered accordingly. The variables in the spin bath are temperature, pH, and salt content and fiber speed through the bath. In order to achieve proper regeneration of the cellulose and coagulation of the fiber each condition was properly controlled. Because of
agreements with Advanced Ceramtrics Inc., the exact parameters used for processing are withheld.

5.2.2 VSSP process control

In the VSSP, the spin mix is fed to a metering pump. As the driver rotates, a high inlet pressure forces the mixture through a spinnerette while an extensional force is simultaneously applied to the extrudate. As the extrudate thins down, it solidifies and transforms into a filament. Any variations in this pressure will lead to corresponding variations in the extrusion rate which, in turn, will result in non-uniform diameter along the fiber length.

![Figure 5.2 Block diagram of system setup.](image)

In order to manufacture variable diameter fibers (VDFs), a precise time variation of the pressure should be produced. In practice, it can be realized by very precisely controlling the pump speed. Since the pump is driven by electric motors, by varying the voltage supplied to the motors, variable motor speeds and pump flow rate may be obtained. A procedure can be developed to find the flow-pressure-voltage relationship of
the pump. Research shows that the flow can be modeled as linearly increasing with voltage and linearly decreasing with pressure (Wang and Schueller 1993). By controlling the pump speed and valve opening simultaneously, a desired time variation of the pressure and the flow rate independently was able to produce in a pulsatile pipe flow (Kurokawa, L. et al. 1991).

The block diagram of a data acquisition system setup for this process is shown in Fig. 5.2. The pack pressure is sensed using a strain gage or fiber optic pressure sensor. The output of the pressure sensor was then passed through an anti-aliasing filter to a data acquisition board.

Challenges in VDFs synthesis and manufacture would be - designing a control system to vary pump pressure or pump flow rate sensitively and accurately, which will be exacerbated by the inertia effect of the control system. The control system enables to fabricate fibers of varied-diameter in a controlled manner.

5.3 Fiber synthesis and manufacture

5.3.1 Slurry preparation

Two kinds of ceramic powders (alumina powder and zirconia powder) were used to develop Variable Diameter Fibers (VDFs). To determine the spin compatibility of each powder, slurries were first formulated. Acceptable slurries for VSSP should have low viscosity (<100 cP @ 4s\(^{-1}\) shear rate) and a pH range of 8-10 (French and Cass 1998).

For alumina powder, slurries with 70 wt.% as-received Alcoa A-16 Premium alumina powder were made, and then mixed with a commercially available dispersant, Duramax D-3005 (Rohm and Haas Company, Philadelphia, PA). The median particle size of the
powders was 0.4 µm. The dispersant concentration was varied from 0.6 to 1.0 wt.% in order to control the viscosity of the slurry. Fig. 5.3a shows the slurry’s viscosity variation with dispersant content at different shear rates and the pH value is shown in Fig. 5.3b. The amount of dispersant percentage had a significant influence on the viscosity of the slurry. At 0.7 wt.% dispersant, the slurry had a minimum viscosity of 47 cP, making it suitable for VSSP. Compared with the effects on the viscosity, the amount of dispersant had a slight effect on the pH of the slurry, which remained almost constant at about 9.6.
Figure 5.3 Viscosity and pH of alumina slurries with varying dispersant concentration. (a) Viscosity of alumina slurry (70 wt.%) vs. dispersant concentration at different shear rates (b) pH value of alumina slurry (70 wt.%) vs. dispersant concentration.
Figure 5.4 Viscosity and pH value control of zirconia slurry by changing the dispersant concentration (a) Viscosity of zirconia slurry (75 wt.%) vs. dispersant concentration at different shear rates (b) pH value of zirconia slurry (75 wt.%) vs. dispersant concentration.

Similar with the alumina powder, slurries with 75 wt.% as-received partially stablized zirconia powder (HSY-3W, Daiichi kigenso kagaku kogyo CO., LTD) were prepared and then mixed with Duramax D-3005. The dispersant concentration was varied from 0.5 to 0.7 wt.%. The slurry’s viscosity and pH value variation with dispersant percentage are
shown in Fig. 5.4. A 0.5 wt.% of the dispersant, the slurry has a minimum viscosity of 28 cP@4 s\(^{-1}\) shear rate and the pH was about 9, which are suitable for VSSP.

5.3.2 Spinning with the peristaltic pump

The shape of Variable Diameter Fibers (VDFs) is determined by two important parameters -- the wavelength of the diameter variation (\(\lambda\)) and the amplitude of the diameter variation (d) (Fig. 5.5). To manufacture VDFs, these two parameters should be controllable and adjustable. According to the principle of VSSP, generating a periodical change in the flow rate (or flow rate pulse) of spin mix is the key to fabricate the VDFs. Thus, the wavelength of the diameter variation (\(\lambda\)) and the amplitude of the diameter variation (d) depend on the flow rate pulse frequency and the flow rate perturbation respectively, e.g., the wavelength of the diameter variation (\(\lambda\)) can be decreased by increasing the flow rate pulse frequency and the amplitude of the diameter variation (d) can be adjusted by controlling the flow rate perturbation. To get a desirable VDF shape, these two key parameters, combined with the spinning speed control and flow control must be adjusted properly.

![Figure 5.5 Schematic of a Variable Diameter Fiber (VDF).](image)

In this study, to generate the flow rate pulse, a peristaltic pump was used to pump the spin mix. A peristaltic pump is a self-priming rotating positive displacement pump
without any valves (Fig. 5.6). It uses rotating rollers pressed against special flexible tubing periodically to create a pressurized flow. The flow obtained is a function of the tube inner diameter and the pump rotational speed. By adjusting the pump speed and changing the tube inner diameter, the flow rate pulse frequency and the flow rate perturbation can be controlled, e.g., the flow rate pulse frequency is increased by increasing the pump speed and the flow rate perturbation is controlled by changing the inner diameter of the tube.

Figure 5.6 Schematic diagram of a peristaltic pump.

Two peristaltic pumps were tested during fabrication of VDFs in this program. A Ponndorf hose pump PX 10 was used to generate the flow rate pulse at first. The main parameters for the Ponndorf pump were listed in Table 5.1. Using this pump, the powders were spun successfully through 450 and 900 µm hole diameter spinneret jets with a desirable fiber shape. However, when spun with a 90 µm hole diameter spinneret jet, the flow pulse frequency generated by the Ponndorf pump was not high enough to get a satisfactory fiber shape. The wavelength of the diameter variation (λ) was about 60 mm. The fiber appeared almost straight. To decrease the wavelength of the diameter variation, a MasterFlex L/S 15 Standard pump head (Model No. 07015-20) was used to generate the high frequency flow rate pulse. Driven by the MasterFlex pump controller (Model No.
7553-50), the pump can be operated at 600 rpm. With this pump, VDFs were successfully fabricated using the 90 and 450 µm hole diameter spinneret jets.

**TABLE 5.1 PARAMETERS FOR PONNDORF HOSE PUMP PX 10**

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum viscosity</td>
<td>CP</td>
<td>35,000</td>
</tr>
<tr>
<td>max. Capacity</td>
<td>l/h</td>
<td>150</td>
</tr>
<tr>
<td>max. pressure</td>
<td>kPa</td>
<td>200</td>
</tr>
<tr>
<td>max. speed</td>
<td>l/min</td>
<td>165</td>
</tr>
<tr>
<td>Inside dia. of hose</td>
<td>mm</td>
<td>10</td>
</tr>
<tr>
<td>outside dia. of connecting</td>
<td>mm</td>
<td>14</td>
</tr>
</tbody>
</table>

Throughout this program ceramic VDFs of several compositions were successfully spun, sintered and characterized. All ceramic powders that were spun were made in-house in 0.8-1.0 kg batches by the mixed oxide route. Several iterations of spinning experiments were necessary for each composition to determine the conditions that yielded good quality green fiber, i.e., solid, strong and with high enough green density to sinter to full density. Once the fibers were spun they were debound and sintered. Typical good quality green and sintered VDFs are shown in Fig. 5.7a and, Fig. 5.7b respectively.
5.3.3 Fiber sintering

Experiments were conducted to examine sintering and grain growth in VDFs. For the alumina fiber, the sintering was performed in a tube furnace at 1600°C and 1650°C for 15 min. The sintered fiber microstructure is shown in Fig. 5.8. It notices that the fiber sintered at 1600°C does not have a fully dense microstructure. Porosity exists among the grains. Fibers sintered at 1650°C are characteristically solid and fully dense. The grain morphology appears to be hexagon.

Figure 5.7 Optical micrographs of (a) green ceramic VDF formed by VSSP and (b) sintered ZrO₂ VDF at 1555°C for 30 min.
Figure 5.8 Microstructure of Al$_2$O$_3$ VDFs of the composition, 70 wt.% Al6 alumina powder, 29.3 wt.% water and 0.7 wt.% dispersant (a) after firing at 1600°C for 15 minutes and (b) after firing at 1650°C for 15 minutes.

Although the fiber appeared to have a fully dense microstructure after firing at 1650°C, it was not uniform. Areas with abnormal grain growth were observed, as shown in Fig. 5.9a. To control the grain size, 0.2 wt.% MgO powder was added to the slurry. The sintered fiber microstructure after firing with MgO powder resulted in a more uniform microstructure and more evenly distributed grain size (Fig. 5.9b). Thus, it is necessary to add small amount of MgO powder to control the grain size.

Figure 5.9 Microstructure of Al$_2$O$_3$ VDFs after firing at 1650°C for 15 min (a) without MgO powder and (b) with 0.2 wt.% MgO powder.
A typical microstructure of zirconia VDFs sintered for 30 min at 1555°C is shown in Fig. 5.10. The fibers were characteristically solid, fully dense, straight, and round in cross section. The grain size is nearly identical. The mean grain size was about 500 nm. Corresponding to its microstructure, the sintered zirconia fiber has good mechanical properties. Sintered fibers can be bent to a circle and spring back when released.

![Microstructure of ZrO$_2$ VDFs of the composition, 75 wt.% zirconia powder, 24.5 wt.% water and 0.5 wt.% dispersant, after firing at 1555°C for 30 min.](image)

**Figure 5.10** Microstructure of ZrO$_2$ VDFs of the composition, 75 wt.% zirconia powder, 24.5 wt.% water and 0.5 wt.% dispersant, after firing at 1555°C for 30 min.

5.3.4 Characterization of VDFs

Typical sintered zirconia and alumina VDFs spun with 450 μm hole diameter spinneret jets are shown in Fig 5.11. For zirconia VDFs, the wavelength of the diameter variation (λ) is about 530 μm. The diameter of the enlarged nodes is about 175 μm and the diameter of the narrow neck is about 115 μm. For alumina VDFs, the wavelength of the diameter variation (λ) is about 1648 μm. The diameter of the enlarged nodes is about 194 μm and the diameter of the narrow neck is about 115 μm.
5.4 Discussion

In order to further improve the quality of the VDFs, especially for decreased size, the control system (e.g., pump control, spinning speed control, flow control) needs to be upgraded. In the current system, a peristaltic pump was used to generate the flow rate pulse. According to the principle of a peristaltic pump, there exists a linear relationship between the pump speed and the flow rate. However, experiment results showed that the linear relationship does not exist for the spin mix due to its high viscosity (about 5-8k CPS) (Fig. 5.12). Pump speed had no considerable influence on the flow rate for the spin mix. Thus, pump speed cannot be used to control the flow rate of the spin mix accurately. New methods are needed to control the flow rate for VDF manufacturing.
Figure 5.12 Flow rate vs. the peristaltic pump speed for the spin mix.

5.5 Conclusions

High quality straight and variable diameter alumina and zirconia fibers were successfully fabricated using the VSSP process. Different sizes of alumina and zirconia VDFs of several compositions were successfully spun, sintered and characterized. Although there is room to improve this manufacturing method for the production of VDFs, it was decided that mechanical tests of composites made with these fibers should be performed to determine whether further improvements of the VSSP process were warranted by increased mechanical properties of the composites.
6.1 Introduction

Once VDFs had been successfully manufactured, the goals of this study were to develop novel composites made from VDFs incorporated in a PMMA matrix and to evaluate the mechanical properties. Since fatigue, in addition to static loading, is a predominant in vivo loading mode that leads to bone cement failure (Topoleski, Ducheyne et al. 1990), improvements PMMA bone cement must address both static and fatigue failure modes. Therefore, both the static mechanical properties and fatigue behavior of these composites were evaluated and compared with the properties of the control bone cement and CSF reinforced bone cement.

To evaluate the effects of fiber content, VDF reinforced composites were fabricated at different fiber volume fractions. For comparison, the same volume fraction of CSF reinforced bone cements were also fabricated. Finally, the failure mechanisms in the VDF reinforced bone cement were evaluated using scanning electron microscopy (SEM) and optical microscopy.
6.2 Materials and methods

6.2.1 Specimen preparation

Both alumina and zirconia fibers were examined. These fibers are more viable than metal or carbon fibers due to aesthetic (white color) as well as regulatory concerns (zirconia is already used as a radiopacifier in some commercially available cements). All alumina and zirconia fibers (CSFs and VDFs) were obtained from Advanced Cerametrics Inc. (Lambertville, NJ) and were characterized in Chapter 5. The average diameter of CSFs is 120 µm. The as-received continuous fibers were cut with a sharp roller blade into discontinuous filaments of the lengths of 0.25 inch (6.35 mm) resulting in a nominal aspect ratio of 50.

For zirconia fiber, five groups of tensile test specimens were prepared by varying the fiber morphology (i.e. CSF and VDF) and content (i.e. 2 and 5 vol.%) (Table 6.1) in order to evaluate the effects of fiber shape and fiber volume percentage on the strength and stiffness of the composite bone cement. For comparison, since the composites were made without barium sulfate, the control group specimens were made using a commercially available bone cement (Osteobond®, Zimmer, Inc.) without barium sulfate. Zirconia fibers are themselves radiopaque negating the need for barium sulfate. Recall that bone cement was described in detail in Chapter 1).

The amount of zirconia fibers (density - 5.84 gm/cc) to be added to the mixtures was calculated by assuming a complete conversion of monomer to polymer, and assuming that the specific gravity of the monomer (0.936 gm/cc) would be changed to that of the polymer (1.17 gm/cc) on polymerization. The volume fractions of the reinforcements that were tested as well as the ratios of the zirconia, polymer beads, and monomer are
The volume of the PMMA powders to the monomer was kept constant in all cases to facilitate mixing.

**TABLE 6.1 RATIOS OF ZIRCONIA FIBER (GMS), PMMA (GMS) AND MONOMER (CC) USED TO CREATE TENSILE SPECIMENS**

<table>
<thead>
<tr>
<th>Group</th>
<th>Fiber Type</th>
<th>Fiber volume percentage (%)</th>
<th>Zirconia (g)</th>
<th>PMMA (g)</th>
<th>Monomer (cc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>None</td>
<td>0</td>
<td>0</td>
<td>36</td>
<td>20</td>
</tr>
<tr>
<td>2</td>
<td>CSF</td>
<td>2</td>
<td>5.57</td>
<td>36</td>
<td>20</td>
</tr>
<tr>
<td>3</td>
<td>VDF</td>
<td>5</td>
<td>14.38</td>
<td>36</td>
<td>20</td>
</tr>
<tr>
<td>4</td>
<td>VDF</td>
<td>2</td>
<td>5.57</td>
<td>36</td>
<td>20</td>
</tr>
<tr>
<td>5</td>
<td>VDF</td>
<td>5</td>
<td>14.38</td>
<td>36</td>
<td>20</td>
</tr>
</tbody>
</table>

The existence of air voids inside the specimens greatly decreases the reinforcement effects of the VDFs. In order to show the advantages of VDFs, specimens without defects must be prepared. To remove the air from the PMMA matrix, centrifuging or vacuum mixing may be used in specimen preparation. Also, decreasing the rate of polymerization can help to increase mixing time and eliminate the air. In this investigation, vacuum mixing was used to minimize the effects of voids.

In preparing tensile specimens, the appropriate weight of short fibers was mixed by gentle stirring of the fibers with the powder phase of the bone cement in the mixing bowl. Care was taken to ensure that the fibers were thoroughly incorporated into the powder. The liquid methylmethacrylate (MMA) monomer was subsequently added to the powder phase of the bone cement. Mixing was performed for 60 seconds at 1 Hz at room temperature (23°C) using a Zimmer Quick-Vac Vacuum mixing bowl, under a vacuum of 20-22 mm Hg. After mixing was complete, the cement mass was transferred by gravity flow into a cement cartridge and injected into a polysulphone mold using a commercial
cement gun (Power Flo cement injection gun, Zimmer Inc., Warsaw, IN). Each group had 10 specimens. The mold produced tensile specimens, as illustrated in Fig. 6.1, corresponding to ASTM D638-98, Type II (ASTM Standard D638-98 1998). The viscosity of the cement increased with the volume fraction of the zircoina fibers. But, in all cases the cement flowed out of the gun smoothly without aggregation of the fibers. After injection molding, specimens were cured in an oven at 37°C for 1 h. Specimens were taken out of the oven and removed from the mold before the excess material was trimmed. After polishing with the sand paper, the samples were stored in a dry environment before testing.

Figure 6.1 Dimensions of the tensile specimen used in this study, corresponding to ASTM D638-98, Specimen Type II. All units are in mm.

Similar with the preparation of tensile specimens reinforced with zirconia fibers, five groups of tensile test specimens reinforced with alumina fibers were prepared by varying the fiber morphology (i.e. CSF and VDF) and content (i.e. 2 and 10 vol.%). The amount of alumina fibers (density - 3.95 gm/cc) to be added to the mixtures was calculated by assuming a complete conversion of monomer to polymer. The volume fractions of the reinforcements that were tested, as well as the ratios of the alumina fibers, polymer beads, and monomer are tabulated in Table 6.2. The volume of the PMMA powders to the monomer was kept constant in all cases to facilitate mixing.
TABLE 6.2 RATIOS OF ALUMINA FIBER (GMS), PMMA (GMS) AND MONOMER (CC) USED TO CREATE FATIGUE SPECIMENS

<table>
<thead>
<tr>
<th>Groups</th>
<th>Fiber Type</th>
<th>Fiber volume percentage (%)</th>
<th>Ingredient Proportions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Alumina (g)</td>
</tr>
<tr>
<td>1</td>
<td>None</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>CSF</td>
<td>2</td>
<td>3.77</td>
</tr>
<tr>
<td>3</td>
<td>CSF</td>
<td>10</td>
<td>20.53</td>
</tr>
<tr>
<td>4</td>
<td>VDF</td>
<td>2</td>
<td>3.77</td>
</tr>
<tr>
<td>5</td>
<td>VDF</td>
<td>10</td>
<td>20.53</td>
</tr>
</tbody>
</table>

The procedure for preparing fatigue specimens was identical to that for the tensile specimens. Five groups of fatigue specimens reinforced with zirconia fibers were prepared by varying the fiber morphology (i.e. CSF and VDF) and content (i.e. 2 and 10 vol.%) (Table 6.3). Also, a control group was prepared using the commercially available bone cement (Osteobond®, Zimmer, Inc.) without barium sulfate. The fatigue specimens were produced corresponding to ASTM F2118-01a (ASTM Standard F2118-01a 2001). The mold produced fatigue specimen is illustrated in Fig. 6.2. After machining, all specimens were polished and maintained in 37°C water before testing. Specimen surfaces were as uniform as possible to ensure that material characteristics, rather than preparation techniques, governed the fatigue failure process.

TABLE 6.3 RATIOS OF ZIRCONIA FIBER (GMS), PMMA (GMS) AND MONOMER (CC) USED TO CREATE FATIGUE SPECIMENS

<table>
<thead>
<tr>
<th>Groups</th>
<th>Fiber Type</th>
<th>Fiber volume percentage (%)</th>
<th>Ingredient Proportions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Zirconia (g)</td>
</tr>
<tr>
<td>1</td>
<td>None</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>CSF</td>
<td>2</td>
<td>5.57</td>
</tr>
<tr>
<td>3</td>
<td>CSF</td>
<td>10</td>
<td>30.35</td>
</tr>
<tr>
<td>4</td>
<td>VDF</td>
<td>2</td>
<td>5.57</td>
</tr>
<tr>
<td>5</td>
<td>VDF</td>
<td>10</td>
<td>30.35</td>
</tr>
</tbody>
</table>
Due to the low viscosity, 5 and 10% by volume of the fibers could be easily mixed into the cements, though this may not be possible for other formulations. There was an increase in the viscosity of the cements due to the addition of reinforcements. The increase in the viscosity was not significant enough to affect the delivery of the cements through a commercial cement gun (Power Flo cement injection gun, Zimmer Inc., Warsaw, IN). Therefore, the formulations investigated are suitable for clinical use with current instrumentation.

6.2.2 Static test

Tensile test were performed on an ATS screw-driven universal testing machine (Series 910) within 3 and 14 days of specimen preparation (Fig. 6.3). A crosshead speed of 2.54 mm/min (0.1 in/min), resulting in a strain rate of 7.43e-4 s\(^{-1}\) along the gage length, was used to load the samples to failure. A minimum of eight samples per group were tested. The stress at failure and modulus of elasticity were all measured for each test.
Figure 6.3 The tensile specimen (A) was fixed by two grips, an extensometer (B) was attached to the tensile specimen to measure the strain.

6.2.3 Fatigue test

Fatigue tests were performed on three MTS hydraulic test frames. Testing was performed in an aqueous environment of using a specially designed environmental chamber maintained at $37^\circ C \pm 1^\circ C$ (Fig. 6.4). Specimens were subjected to uniaxial constant-amplitude fully reversed tension-compression loading ($\pm 15$ MPa in a sinusoidal cyclic manner), at a frequency of 10 Hz, until fracture. The stress level followed the industry standard -- Harris protocol for fatigue of bone cements (Davies, O'connor et al. 1987). The number of stress cycles to failure, $N_f$, was recorded for each test.
Tensile and fatigue test results were statistically analyzed using analysis of variance (ANOVA) techniques and Tukey-Kramer HSD method to determine where statistically significant differences exist. Data were analyzed using JMP (JMP IN 5.1, SAS Institute Inc., Cary, NC) with a significance level of $p = 0.05$.

6.3 Results

6.3.1 Static test

X-ray radiographs of zirconia fiber reinforced tensile specimens show that the fibers were completely and uniformly incorporated into the bone cement during the mixing process (Fig. 6.5). Most fibers aligned somewhat along the flow direction of the cement.
The typical stress-strain curves for specimens reinforced with zirconia fibers are shown in Fig. 6.6. The control bone cement had the largest total strain to failure. The introduction of zirconia fibers increased the elastic modulus of the material, and the elastic modulus increased with increasing fiber volume fraction. However, the ultimate tensile strength was not considerably improved due to the significant decrease in the total strain to failure, especially for bone cement reinforced with CSFs. Compared with the bone cement reinforced with CSFs, the total strain to failure of the bone cement reinforced with VDFs were much higher (about two times) for both 2% and 5% groups. Outside of the control group, bone cement reinforced with VDFs had the best tensile properties with the higher elastic modulus and failure strain.
Figure 6.6 Typical stress-strain behavior of the control group, bone cement reinforced with 2% zirconia CSFs and VDFs, and bone cement reinforced with 5% zirconia CSFs and VDFs.

The results of tensile tests for zirconia fiber reinforced bone cement are shown in Fig. 6.7 and Fig. 6.8. There was a significantly greater (p < 0.05) modulus for 5% CSF, VDF reinforced cements compared with the 2% CSF, VDF reinforced cement (Fig. 6.7). At the same time, the elastic modulus of the 2% CSF, VDF reinforced cement was significantly greater (p < 0.05) than that of the control cement. The elastic modulus increased by 73% and 168% for cements reinforced with 2% and 5% CSF, and increased by 46% and 152% for cements reinforced with 2% and 5% VDF. However, the differences between the CSF reinforced cement and the VDF reinforced bone cement at either 2% or 5% by volume were not significant (p > 0.05).

The average ultimate strengths of all groups were shown in Fig. 6.8. The average ultimate strength was significantly greater (p < 0.05) for the cement reinforced with
VDFs compared to the cement reinforced with CSFs at 2% by volume. However, the difference between CSF and VDF reinforced cements at 5 vol.% was not statistically significant (p > 0.05). Overall, the ultimate strengths were not statistically different for the groups reinforced with 2% VDFs, 5% CSFs, 5% VDFs and the control group. The average ultimate strength of bone cement reinforced with 2% CSFs by volume was lower than the control cement, but the difference was not statistically significant different (p > 0.05).

Figure 6.7 Elastic modulus of unreinforced bone cement (control), bone cement reinforced with 2% and 5% zirconia CSFs and VDFs, respectively (Levels not connected by same letter are significantly different, p < 0.05).
Figure 6.8 Ultimate strength of unreinforced bone cement (control), bone cement reinforced with 2% and 5% zirconia CSFs and VDFs, respectively (Levels not connected by same letter are significantly different, p < 0.05).

The results of tensile tests for alumina fiber reinforced bone cement were similar with the results of zirconia fiber reinforced bone cement (Fig. 6.9 and Fig. 6.10). There was a significantly greater modulus (p < 0.05) for 10% CSF and VDF reinforced cements compared with the 2% CSF and VDF reinforced cement (Fig. 6.9). The elastic modulus of the 2% CSF and VDF reinforced cement was significantly greater (p < 0.05) than that of the control cement. No significant differences (p > 0.05) were found between the alumina CSF reinforced cement and the alumina VDF reinforced bone cement at either 2% or 10% by volume.

The average ultimate strengths of all groups were shown in Fig. 6.10. At 2% fiber volume fraction, the average ultimate strength was greater for the VDF reinforced cement compared to the CSF reinforced cement. However, the difference was not statistically
When the fiber volume percentage increased to 10%, the average ultimate strength was significantly greater ($p < 0.05$) for the cement reinforced with VDFs compared to the cement reinforced with CSFs, which is opposite the results of zirconia fiber reinforced bone cement.

Figure 6.9 Elastic modulus of unreinforced bone cement (control), bone cement reinforced with 2% and 10% alumina CSFs and VDFs, respectively (Levels not connected by same letter are significantly different, $p < 0.05$).
Figure 6.10 Ultimate strength of unreinforced bone cement (control), bone cement reinforced with 2% and 10% alumina CSFs and VDFs, respectively (Levels not connected by same letter are significantly different, p < 0.05).

6.3.2 Fatigue test

Statistical analysis of the fatigue results of the unreinforced bone cement (control), bone cement reinforced with 2% and 10% zirconia CSFs and VDFs, respectively, are shown in Fig. 6.11. In the statistical analysis, the two run-out specimens after four million cycles of bone cement reinforced with 10% VDFs by volume were not included, and thus the analysis underestimates the actual fatigue data of the bone cement reinforced with 10% VDFs. The fatigue life of the reinforced groups were much higher than control bone cement. The number of cycles to failure increased by 59% and 141% for cements reinforced with 2% CSF and 2% VDF, respectively. Although the fatigue life of bone cement reinforced with 2% VDF is higher than that of the cement reinforced with 2% CSF, there was not statistically significant (p > 0.05) due to the wide scatter in the fatigue.
results. The fatigue life was dramatically increased when the fiber volume percentage was increased to 10%. There was approximately an order of magnitude increase in fatigue life for the bone cement reinforced with CSFs over the control cement. Compared with the bone cement reinforce with CSFs, the fatigue life of bone cement reinforce with VDFs was about four times longer. The number of cycles to failure is significantly greater (p < 0.05) for the bone cement reinforced with 10% VDFs by volume compared to the cement reinforced with 10% CSFs by volume, which shows the advantage of VDFs.

Figure 6.11 Number of cycles to failure of unreinforced bone cement (control), bone cement reinforced with 2% and 10% zirconia CSFs and VDFs, respectively (Levels not connected by same letter are significantly different, p < 0.05).
6.4 Fractography

Fractographic analysis of the failed cement is the most useful method for observing failure patterns and establishing significant mechanical properties. For fractographic analysis, representative fracture surfaces from the specimens were analyzed using the scanning electron microscopy (SEM). Each specimen was cut approximately 0.5 cm below the fracture surface. Fracture surfaces were coated with gold by spectra deposition. Specimens were imaged using a field emission scanning electron microscope (Model 4500, Hitachi Field Emission microscope) operated at an accelerating voltage of 30 kV.

6.4.1 Tensile test specimens

6.4.1.1 Control bone cement

A typical fracture surface of the control cement is shown in Fig. 6.12. Generally, the surface was rough with an irregular topography, characterized by an overall pattern of chips or flake like features called hackles (Kusy 1978). Hackles represent abrupt tensile failure when the stress reached the tensile strength of the material.
6.4.1.2 VDF and CSF reinforced cements

Typical fracture surfaces for the tensile specimens of zirconia VDF reinforced bone cement and zirconia CSF reinforced bone cement are shown in Fig. 6.13 and Fig. 6.14, respectively. Similar to the fracture surface of the control bone cement, the fracture surfaces are rough and represent a rupture fracture. Generally, three typical fracture characteristics can be found: (1) Fibers were evenly distributed in the cement (no aggregation of the fibers was observed); (2) Most fibers were nearly perpendicular to the fracture surface; (3) Fiber fracture dominated the fracture characteristics instead of fiber pull-out. These characteristics indicate that the fiber strength was not sufficient to allow fiber pull-out.
Figure 6.13 SEM micrograph showing a typical fracture surface for a tensile specimen of zirconia CSF reinforced bone cement.

Figure 6.14 SEM micrograph showing a typical fracture surface for a tensile specimen of zirconia VDF reinforced bone cement.
Although, fiber fracture dominated the fracture characteristic, fiber/matrix interface debonding can be observed for both CSF and VDF composites under higher magnification. Fig. 6.15 (a, b) shows the debonding interface of CSFs and VDFs, respectively. Both the CSF and VDF were debonded from the matrix at the fiber/matrix interfaces. The fiber debonding length of VDFs was much shorter than that of the CSFs due to the mechanical interlock provided by the fiber morphology. Furthermore, Fig. 6.16 shows that portion of the matrix adhered to the fiber surface after fracture. This indicates that some bonding occurred between the fiber and the cement matrix. Such bonding may be related to the irregularities in the fiber surface, which cause micro mechanical interlock between the fiber and matrix. Fig. 6.17 shows the rough features of the fiber reproduced in the surface of the cement envelope that formed around the fiber.
Figure 6.15 SEM micrographs showing the fiber debonding lengths of (a) CSFs and (b) VDFs.
Figure 6.16 SEM micrograph showing cement adhering to the fiber after fracture in a
tensile test specimen of zirconia CSF reinforced bone cement.

Figure 6.17 SEM micrograph showing a tensile test specimen of zirconia VDF reinforced
bone cement: not that the fiber surface features were reproduced on the matrix after pull-
out.
6.4.2 Fatigue test specimens

6.4.2.1 Control bone cement

A typical fracture surface of the fatigue specimens of unreinforced bone cement (control) is shown in Fig. 6.18. The fracture surfaces of all the specimens showed general characteristics of fatigue fracture. The fracture surface appearance can be differentiated into smooth and rough regions. The smooth region represents an area of early, slower crack growth. The right of the fatigue region was rougher with a more irregular topography and was characterized by the overall pattern of hackles, which represents the abrupt fracture. The fatigue crack in Fig. 6.18 appears to have initiated at the void and propagated toward a transition to rapid fracture on the right. Once the crack initiated, the severity of the stress intensity at the crack tip may lead to rapid fatigue crack propagation and subsequent catastrophic fracture.
6.4.2.2 Two percent by volume fiber reinforced cement

Fig. 6.19 shows a typical fracture surface for a 2% zirconia CSF reinforced specimen. In contrast to the control bone cement, no abrupt fracture zones were observed. The entire fracture surface appears smooth. It also appears that the fracture occurred at different planes, resulting in river-like facets. The river pattern tributaries point to crack origin (Topoleski, Ducheyne et al. 1990; James, Jasty et al. 1992). Fig. 6.20 shows the zone where the fiber is completely debonded from the matrix in front of the crack, indicating, once again, that bonding between the fibers and the matrix was weak.
Figure 6.19 SEM micrograph showing a typical fracture surface for the fatigue specimens of the 2% zirconia CSF reinforced cement.

Figure 6.20 SEM micrograph showing a debonded CSF in front of a crack.
Fig. 6.21 shows a typical fracture surface for a 2% zirconia VDF reinforced specimen, which looks similar to the fracture surface of 2% zirconia CSF reinforced specimen. The fibers were evenly distributed inside the matrix; no fiber aggregation was apparent; most fibers were nearly perpendicular to the fracture surface. Again, the crack initiated at a void and grew to the left.

Figure 6.21 SEM micrograph showing a typical fracture surface for the fatigue specimens of the 2% zirconia VDF reinforced bone cement.

6.4.2.3 Ten percent by volume fiber reinforced cement

Fractographs of bone cement reinforced with 10% by volume zirconia VDF are shown in Fig. 6.22. The fibers are more closely spaced to each other but they appear to have been uniformly dispersed into the PMMA matrix. Compared with the control cement and two percent by volume fiber reinforced cement, the fracture surface of 10% by volume VDF reinforced cement was much rougher. The main crack propagated by
coalescing with many smaller cracks (Fig. 6.22a). These smaller cracks were often not on the same plane as the main crack, resulting in a very rough fracture surface. As evidenced by the river-marks in the fractograph, the fracture surfaces of the fatigue test specimens exhibited microbrittle characteristics. In other words, they appeared brittle at the microscopic level. As the crack propagated, a few VDFs bridging the crack were pulled out, which resulted in extensive matrix damage (Fig. 6.22b), consuming large amounts of energy.
Figure 6.22 SEM micrographs showing typical fracture surfaces for fatigue specimens of the 10% zirconia VDF reinforced bone cement.
As shown in Fig. 6.23a, the fracture surface of 10% zirconia CSF reinforced cement was smoother when compared with the fracture surface of VDF reinforced bone cement. Therefore, the crack was able to propagate with less resistance for a long distance, leaving behind flat fracture surface. Once the crack reached a critical size, it propagated through the entire cross-section of the specimen, causing fiber fracture or pullout, and leaving a relatively flat fracture surface with river marks, similar to the fracture surface of a brittle material. In contrast to VDF, the pullout of CSF did not result in significant matrix damage (Fig. 6.23b), which means less energy was consumed during the fracture process.
Figure 6.23 SEM micrographs showing typical fracture surfaces for the fatigue specimens of the 10% zirconia CSF reinforced bone cement.
6.5 Discussion

6.5.1 Tensile test

Tensile tests demonstrated that incorporation of both zirconia and alumina fibers significantly increased the elastic modulus of bone cement. The elastic modulus of fiber reinforced bone cement also increased with increased fiber content. However, the tensile strength of fiber-reinforced cement was not significantly improved when compared with the control bone cement and the total failure strain was greatly decreased for the fiber-reinforced cement, especially for CSF reinforced cement (Fig. 6.6). This is most likely due to the increased local stresses caused by fibers, which can be considered rigid inclusions in an elastic matrix (Eshelby 1957). The stress concentrations may have promoted crack initiation. Once the crack initiated, the stress intensity was so large that potential fiber reinforcing effects may have been masked (Topoleski, Ducheyne et al. 1995). This can also explain the reduction in strength of bone cement reinforced with 2% zirconia CSF, in contrast to that of the control group.

There were almost no significant differences exist between tensile tests for the zirconia CSF and the VDF reinforced bone cements at either 2% or 5% by volume, except the ultimate strength of the CSF and the VDF reinforced bone cement at 2% by volume. The lack of a difference was likely because the fiber itself was not strong enough. The fracture surfaces showed that the failure mechanism in tensile tests for both CSF and VDF reinforced cements was fiber fracture. Although fiber/matrix debonding was observed for CSF reinforced bone cement, most CSFs fractured before being pulled out from the matrix, which shows that CSFs are weak (Fig. 6.15a). The failure mechanism was the same in VDF reinforced bone cement, although the fiber/matrix debonding
distance was much shorter due to the mechanical interlock between the fiber and the matrix. Most VDFs also fractured before being pulled out of the matrix (Fig. 6.14).

VDFs are optimal for composites with a weak fiber/matrix interfacial bond, and where fiber pullout is the major failure mechanism. In this case, significant improvement is expected for VDF reinforced composites due to improved load transfer between the VDF and matrix, and the change in failure mechanism. This of course assumes the fiber is itself strong enough to resist failure. In the current study, the advantage of VDFs in static loading was masked by the weak fiber strength for both CSFs and VDFs. The fibers fractured before pull-out could contribute to strength. This also assumes that the bonding between fiber and matrix is weak. The surface of the CSFs is very rough, which can cause micromechanical interlock between the CSF and the matrix. This mechanism was observed by bone cement adhering to the CSF surface after fracture (Fig. 6.16). Bonding between the CSF and matrix also weakened the CSFs and VDFs by strengthening the bond between fiber and matrix.

The tensile tests results of alumina fiber reinforced bone cement were similar with those of zirconia fiber reinforced cement, showing that the reinforcement effect of VDFs was independent from the fiber material properties.

6.5.2 Fatigue test

Fatigue tests demonstrated that incorporation of zirconia fibers increased the fatigue life of bone cement. Significant increases in the fatigue life of bone cement were realized by increasing fiber content. For 2% fiber reinforced bone cement, although the fatigue life was much higher than that of the control cement, there was no significant increase due to the wide scatter in the results. There was also, no significant difference between
the VDF reinforced cement and CSF reinforced cement. SEM fractographs of fiber reinforced bone cement showed that the fracture occurred at different planes forming a pattern of river-like facets. Thus, fibers diverted crack propagation direction and absorbed more energy. However, due to the low fiber content, the crack was still able to propagate easily for a long distance before being inhibited by the fiber, leaving behind a flat fracture surface, which looked similar to the fracture surface of the control bone cement. Therefore, the fatigue life was not significantly improved when the fiber content was relatively low.

As the fiber content was increased to 10% by volume, there was approximately an order of magnitude or more increase in fatigue life for CSF reinforced cement. Also, significant improvement in fatigue life was found for VDF reinforced cement compared with CSF reinforce cement leading to a 100 fold increase in life from the control cement to the VDF cement. Compared with the control cement and 2% fiber reinforced cement, the fracture surface of 10% VDF reinforced cement was much rougher. The main crack propagated by coalescing with many smaller cracks. The smaller cracks were often not on the same plane as the main crack, resulting in a very rough fracture surface. Although SEM fractographs showed that the fiber/matrix interfacial bond was weak (Fig. 6.22a), most VDFs were kept anchored inside the matrix due to the more efficient mechanical interlock, which caused the diversion of the crack out of the original crack plane. In contrast to VDFs, more CSFs were pulled out due to the poor interfacial bond between the fiber and matrix and lack of sufficient mechanical interlock, which provided less resistance to crack propagation and poor bridging ability.
Pullout of VDFs resulted in much more extensive matrix damage than that of the pullout of CSFs (Fig. 6.22b and 6.23b). This means that more energy was consumed for VDFs than CSFs during the pullout process. The results, along with the fracture morphology, clearly show that the VDF reinforced cement is significantly more resistant to fatigue, with a much greater energy dissipating capacity than CSF reinforced cement.

Fiber reinforcement affects the fatigue crack propagation phase of failure in bone cement, enhancing the fatigue crack propagation resistance. In general, the increased fatigue life seen in the VDF reinforced cement was the result of several deformation mechanisms operating in these specimens. These include increased load transfer at the fiber/matrix interface, and diversion of the crack out of the original crack plane by fiber splitting and fiber/matrix interface failure, both of which divert the crack from its fracture plane and increase the energy required for crack propagation. These modes of failure were seen in all of the VDF reinforced cement tested in this study.

In this study, the main failure mechanism was fiber fracture. This shows that the energy absorption capacity of fiber-reinforced cement could be increased by inducing more fiber pullout, which could be realized by improving fiber strength.

6.6 Summary and conclusions

In summary, VDF reinforced bone cement was developed. Both the static and fatigue mechanical properties were evaluated and compared with the properties of the control bone cement and CSF reinforced bone cement. The experimental results and accompanying statistical analysis lead to the following conclusions.

In tensile tests, bone cement reinforced with 2% and 5% zirconia VDFs resulted in a significantly increased elastic modulus over unreinforced bone cement (control).
However, no significant improvement was found for the ultimate strength at each fiber volume fraction. Increasing the fiber content from 2% to 5% produced a corresponding increase in mechanical properties for both VDF and CSF reinforced bone cement. No significant difference was found between VDF and CSF reinforced bone cement except the ultimate strength at 2% fiber fraction by volume. The tensile tests results of alumina fiber reinforced bone cement were similar with those for zirconia fiber reinforced cement, which shows that the effects of VDFs were independent from the fiber material properties.

Fatigue tests showed greater fatigue life for both VDF and CSF reinforced cement when compared with unreinforced bone cement (control). At 2% fiber volume fraction, no significant difference was found between the CSF and VDF reinforced cement. At 10% fiber volume percentage, the VDF reinforced cement has significantly greater fatigue life than that of CSF reinforced cement and the control cement.

The fractographic analysis of the fracture specimens revealed several important phenomena related to the reinforcing mechanisms of zirconia VDF and CSF reinforced bone cement.

1. In tensile tests, fiber fracture, instead of fiber pullout, dominated the fracture surface for both CSF and VDF reinforced cement. CSFs and VDFs debonded from the PMMA matrix during fracture, but had a negligible effect on the properties. Micromechanical interlock existed between CSFs and the PMMA matrix due to the irregularities in the fiber surface and may have contributed to the fiber failure.

2. In the fatigue tests, most VDFs were kept anchored inside the matrix due to efficient mechanical interlock. In contrast to the VDF, more CSFs were pulled out due to the poor interfacial bond between the fiber and matrix. Pullout of VDFs results in much
more extensive matrix damage than that of the pullout of CSFs increasing the resistance to fatigue.

3. The fracture surface of VDF reinforced bone cement was rougher than CSF reinforce bone cement at 10% percent fiber volume fraction. Therefore, the VDF reinforced cement was significantly tougher, having a greater energy dissipation capacity than CSF reinforced cement.

This study showed the feasibility of a novel fiber (VDF) technology for reinforced polymers. This fiber family significantly improved the fatigue life of bone cement at a very high level of reliability. VDFs could potentially inhibit implant loosening due to the mantle fracture of bone cement and delay the need for revision surgery.
CHAPTER 7
CONCLUSIONS AND SUGGESTED FUTURE WORK

7.1 Conclusions

The feasibility of a new ceramic fiber technology for bone cement reinforcement was examined in this research. An innovative fiber morphology was developed to provide enhanced stress transfer between the fiber and matrix and, therefore, enhanced composite strengthening. The effects of fiber morphology on mechanical properties of the composite were analyzed theoretically. Based on the theoretical model, a general shape optimization procedure was developed to determine the optimal fiber shape using finite element method. Finally, the feasibility of both the reinforcement concept and refinement of the fiber manufacturing was tested experimentally. Variable diameter Fiber (VDF) - the result of shape optimization - was developed and applied to reinforce bone cement. The mechanical performance of the resulting composite was tested statically and in fatigue.

7.1.1 Fiber-end deformation effects in enlarged-end, fiber-reinforced composites

A pullout model for the fiber with enlarged ends was developed. A spring component was integrated in the new model to account for the effects of deformation in the enlarged end. The spring coefficient, $K_e$, was calibrated by the numerical analysis. The constitutive property of the spring ($K_e$), and therefore the contribution of the deformation of the end to the resistance to pullout, greatly depended on the fiber enlarged end shape. A fiber
with a ‘mushroom-cap shaped’ or oblate ellipsoid fiber end was shown to transfer load more efficiently than a fiber with a ‘egg-like’ or prolate ellipsoid fiber end. Previous models have only limited application to enlarged-end fiber shapes other than a sphere or oblate ellipsoid. This is unfortunate because these are the least effective reinforcement shapes.

As another conclusion drawn from the present model, the enlarged end shape had a significant influence on the distribution of the axial stress in the fiber. The axial stress in the fiber increased as the ratio of major to minor axes ($\rho = b/a$) decreased, indicating better load sharing between fiber and matrix. However, the stress concentration at the point of attachment of the fiber shaft to its enlarged end will limit the allowable increase in this stress.

Finally, for perfect bonding between assumed the fiber and matrix, the anchorage effect of the enlarged fiber end decreased as the embedded length of fiber increased because more load is transferred by a shear mechanism through the lateral interfaces on the shaft instead of through the mechanical interlock at the enlarged end. For imperfect bonds, the effects of the end may remain significant. Enlarged ends, therefore, are useful for all fibers when bonding is weak and only for short fibers ($L_f/r_f \leq 20$) when bonding is strong.

7.1.2 Improvement of bone cement mechanical properties by optimizing fiber shapes

In Chapter Three and Chapter Four, a procedure for structural shape optimization of short fibers using finite element analyses was presented. Optimization of the fiber shape greatly improved the load transfer efficiency between the fiber and PMMA matrix and the mechanical properties of the reinforced bone cement.
The fiber/matrix interfacial bond had an important influence on the shape optimization effect. The weaker the interfacial bond, the stronger the shape optimization effect on the composite modulus. For aligned fiber composites, the results demonstrate that an enlarged-end short fiber with many threads can significantly improve the bonding ability between the fibers and the matrix by the introduction of efficient mechanical interlock. However, in real materials, short fibers are not perfectly parallel to the load and the behavior of the composite can be expected to vary with the degree of fiber misalignment. To obtain the optimal fiber shape for bone cement reinforced with randomly oriented fibers, the composite structures were represented as array of RVEs. The effects of fiber orientation were evaluated by performing the shape optimization process under different orientation angles. The optimal fiber geometry was a dumbbell shape when the fiber was perfectly aligned to the loading direction. As the orientation angle increased, more enlarged knots were generated along the short fiber toward the middle of the fiber span. The general optimal fiber shape was determined to be variable diameter fiber (VDF) for randomly distributed short fibers reinforced bone cement, which is achieved by superimposing the optimal geometries generated in the different orientation angles. Due to the mechanical interlock between the VDFs and the matrix, it is expected that VDFs can both bridge matrix cracks effectively and improve the stiffness of the composite when fibers are randomly oriented.

7.1.3 VDF development

High quality straight and variable diameter alumina and zirconia fibers were successfully fabricated using the VSSP process. Different sizes of alumina and zirconia VDFs of several compositions were successfully spun, sintered and characterized.
Although there was room to improve this manufacturing method for the production of VDFS, it was decided that mechanical tests of composites made with these fibers should be performed to determine whether further improvements of the VSSP process were warranted by increased mechanical properties of the composites.

7.1.4 Static and fatigue characterization of fiber reinforced bone cement

VDF reinforced bone cement was developed. Both the static mechanical properties and fatigue behaviour of these composites were evaluated and compared with the properties of the control bone cement and CSF reinforced bone cement.

In tensile tests, bone cement reinforced with 2% and 5% zirconia VDFs resulted in a significantly increased in elastic modulus over the unreinforced bone cement (control). However, no significant improvement was found for the ultimate strength. Increasing the fiber content from 2% to 5% increased mechanical properties for both VDF and CSF reinforced bone cement. No significant difference was found between zirconia VDF and CSF reinforced bone cement except the ultimate strength at 2% by volume fiber fraction. The tensile tests results of alumina fiber reinforced bone cement are similar with those of zirconia fiber reinforced cement, which showed that the reinforce effects of VDFs was independent from the fiber material properties.

Fatigue tests showed greater fatigue life for both VDF and CSF reinforced cement when compared with unreinforced bone cement (control). At 2% fiber volume fraction, no significant difference was found between the CSF and VDF reinforced cement. At 10% fiber volume percentage, the VDF reinforced cement has significantly greater fatigue life than that of CSF reinforced cement and the control cement.
The fractographic analysis of the fracture specimens revealed several important phenomena related to the reinforcing mechanisms of zirconia VDF and CSF reinforced bone cement. In the fatigue tests, most VDFs were kept anchored inside the matrix due to efficient mechanical interlock. In contrast to the VDF, more CSFs were pulled out due to the poor interfacial bond between the fiber and matrix. Pullout of VDFs results in much more extensive matrix damage than the pullout of CSFs. Furthermore, the fracture surface of VDF reinforced bone cement was rougher fracture surface than CSF reinforced bone cement at 10% percent fiber volume fraction. Therefore, the VDF reinforced cement was significantly tougher, having a greater energy dissipation capacity than CSF reinforced cement.

This study showed the feasibility of a novel fiber (VDF) technology for reinforced polymers. This fiber family significantly improved the fatigue life of bone cement at a very high level of reliability. VDFs could potentially inhibit implant loosening due to the mantle fracture of bone cement and delay the need for revision surgery.

Beyond the immediate application in orthopaedic biomaterials, opportunities to utilize the advantages of VDFs reinforcements in fiber-reinforced plastics could be plentiful in everything from aerospace structures and sporting goods to lightweight automotive parts and construction products. Ultimately, VDFs can provide significant benefits to both high- and low-tech products which comprise structural, reinforced plastics.

7.2 Suggested future work

7.2.1 Fiber shape optimization

In the current study, a 2D plane strain model was used for fiber shape optimization. The plane strain assumption, which was made to reduce the computational requirement,
means that the fibers are really continuous in the direction perpendicular to the plane. The results can be used for qualitative predictions on the behavior of short fiber reinforced composite. However, a short fiber reinforced composite is an inherently 3D problem. To more accurately illustrate the behavior of short fiber reinforced composite, a 3D model should be used for analysis.

Also, computational modeling at the macro-scale could be used to simulate the VDF reinforced composite fracture process and predict overall properties, such as static and dynamic strength, fracture toughness and fatigue behavior. Incorporation of micro-scale models and descriptions, such as fiber pullout, fiber bridging, matrix crack initiation and coalescence, statistical representations of fiber properties, orientation distributions and spatial distribution, and constitutive models, would make such modeling more representative and provide relationships between micro-scale features, such as fiber shape and size, and macroscopic response.

7.2.2 VDF fabrication

Fiber fracture was the major failure mechanism in the current study. Therefore, the energy absorption capacity of VDF reinforced bone cement could be increased by increasing fiber pullout, which could be realized by improving fiber strength. To further improve quality of the VDFs, especially for the small size VDFs, the control system (e.g., pump control, spinning speed control, flow control) needs to be upgraded. In the current system, a peristaltic pump was used to generate the flow rate pulse. According to the principle of a peristaltic pump, there exists a linear relationship between the pump speed and the flow rate. However, experiment result shows that the linear relationship does not exist for the spin mix due to its high viscosity (about 5-8k CPS). Pump speed has no
considerable influence on the flow rate for the spin mix. In this case, pump speed cannot be used to control the flow rate of the spin mix. New methods are needed to be setup to control the flow rate for VDF manufacturing. The following pump systems are considerable.

7.2.2.1 Electronic Metering Pump

An electronic metering pump can be used to dispense chemicals or fluids. This is achieved by an electromagnetic drive mechanism (solenoid), which is connected to a diaphragm. When the solenoid is pulsed by the control circuit it displaces the diaphragm, which, through the use of check valves, moves the fluid out the discharge under pressure. When the solenoid is de-energized it returns the diaphragm and pulls more fluid into the pump head and the cycle repeats. Capacity can be controlled by means of the stroke length and/or stroke rate. Stroke length can be controlled within 0 to 100% of the diaphragm displacement. The pump stroke rate is controlled by an internal circuit and is changed by turning the rate knob. Stroke frequency can be controlled from 10 to 100% (12 to 125 strokes per minute) by means of the electronic circuit. From the principle of the electronic metering pump, the flow rate perturbation and the flow rate pulse frequency can be controlled independently. Increasing the stroke length can increase the flow rate perturbation, which will increase the amplitude of the diameter variation (d). Stroke frequency can be used to control the wavelength of the diameter variation (λ). More information can be found at the website http://www.pulsa.com.

7.2.2.2 Multiple Channel Peristaltic Pump

Experimental results showed that the peristaltic pump speed cannot be used to control the flow rate for the spin mix due to its high viscosity. According to the principle of the
peristaltic pump, the flow obtained is a function of the tube inner diameter and the pump rotational speed. Thus, changing the tube inner diameter can be used to control the flow rate. With a multiple channel peristaltic pump, the flow rate can be adjusted by selecting different channel and the flow rate pulse frequency can be controlled by the pump rotational speed. In this way, the flow rate and the flow pulse frequency can also be controlled separately.

7.2.3 Analytical model in crack shielding

Micromechanical modeling of crack shielding in VDF reinforced bone cement could help to explain failure mechanism of VDF reinforced bone cement and find the implications of crack tip shielding to fatigue behavior. Due to its specific morphology, VDF might enhance crack tip shielding relative to CSF. Understanding these mechanisms could provide valuable information for composite design.


