Influence of pH Value of the Oral Cavity on Biaxial Flexural Strength of Layered Ceramics

Xiaofeng Qin¹, Jun Ou², Xiaohui Su³, Yumin Jiang⁴, Qing H. Qin⁵

¹²⁻College of Material Science & Engineering, Guilin University of Technology, Guilin 541004, China
²Key Laboratory of New Processing Technology for Nonferrous Materials, Ministry of Education, Guilin University of Technology, Guilin 541004, People’s Republic of China
¹Department of Prosthodontics, School of Stomatology, Guangxi Medical University, Nanning 530021, China
⁵Research School of Engineering, Australian National University, Canberra, ACT 2601, Australia

¹qin.dent@gmail.com; ²gloujun2012@yahoo.cn; ³pangfanghe@hotmail.com; ⁴jiangyumin2@yahoo.cn; ⁵qinghua.qin@anu.edu.au

Abstract

In this paper, effect of pH value of oral cavity on the bi-axial flexural strength of veneering porcelain containing zirconia is investigated by means of cyclic loading tests. In the experiments, standardized disc specimens (n=240, 16 mm × 1.6 mm) are pre-loaded with 600 N for 10,000 cycles and stored in a dry or wet (pH: 4, 7 and 8) environment prior to biaxial flexural strength testing (ISO 6872 standard). Hsueh’s simple solutions are used to calculate stress distribution along the direction of thickness of bilayered discs subjected to biaxial moment loading. The fracture mode and location of cracks are identified by optical microscopy. A Weibull statistical approach is used to evaluate the reliability of the failure strength data. It is found that the flexural strength of specimens tested under dry conditions is approximately 20 ~ 50 % stronger than that of specimens soaked in the solution. The flexural strength of specimens soaked in an acidic solution is approximately 30 % greater than that of specimens soaked in an alkaline solution. The Weibull modulus of specimens is 17.73, 17.88, 10.53 and 17.92 in pH 4, pH 7, pH 8 and dry environment respectively. Thus, the structural reliability of specimens used in this work are better when they were soaked in pH 4, pH 7 and was in dry environment than that in pH 8. The test results of bi-axial flexural strength in different storage media may provide a guide to the clinical application of all-ceramics dental crowns.

Keywords

Bi-axial Flexural Strength; Cyclic Loading; All-ceramics Dental Crown

Introduction

All-ceramic materials are increasingly becoming a preferred alternative to metal-ceramic restoration due to their excellent esthetics, chemical stability, and biocompatibility. The brittleness and low tensile strength of conventional glass-ceramics, however, restrains its long-term clinical application in restorations. Compared to other dental glass ceramic systems, zirconia-based ceramics is used clinically for restorations with up to four units. The problem is that current technologies can neither make zirconia frameworks as translucent as natural teeth nor provide internal shade characterization or facilitate customized shading (Luthy et al, 2005; Zarone et al, 2011). As a result, zirconia cores (or frameworks) must be veneered with porcelain to achieve acceptable esthetics (White et al, 2005). In clinical applications, the veneering porcelain has already proved to be the weakest link in such reconstructions (Fischer et al, 2008; Raigrodski et al, 2006; Sailer et al, 2007a;b). Chipping of the veneer during mastication under wet conditions is identified as the main reason for failure, at the rate of 15.2 % after a service time of 35.1 ± 13.8 months (Sailer et al, 2007a).

On one hand, all-ceramic restorations have less longevity than porcelain-fused-to-metal (PFM) fixed partial dentures (FPDs), as the bilayered ceramic materials have low flexural strength. On the other hand, the survival rates of all CAD/CAM ceramic single tooth restoration are equal to PFM-restorations while FPDs show higher complications rates (Land and Hopp, 2010). So, the study of mechanical properties of all-ceramic restorations is necessary.

In recent years many testing and modeling methodologies have been developed to characterize mechanical properties of dental restorative materials (Hsueh, 2006; Qin and Swain, 2004; Sailer et al, 2007b; Thompson, 2000; Zeng et al, 1998). Those tests did not contradict each other, but rather reflect the mechanical
properties of the dental restorative materials under different conditions (Chai et al, 2007). Testing specimens for bi-axial flexure were reported to be easier to prepare reproducibly, to match more closely the size of the clinical restoration, and to be less sensitive to edge defects than those for uniaxial flexure (Ban and Anusavice, 1990; Pagniano et al, 2005; Rosenstiel et al, 1993). All-ceramic dental crowns are usually fabricated into an esthetically pleasing multilayered structure. Because the flexural strength formulas of ISO 6872 (International Organization for standardization, 2008) and ASTM 1499 do not adapt to multilayered specimens (Zeng et al, 1998), Hsueh’s simple solution (Hsueh and Kelly, 2009) is employed in this study.

As stated by Zeng et al. (1996), mean flexural strength values are related not only to the test method but also to the test environment (dry, wet, and repetitive stress). To the authors’ knowledge, there are no available reports on the flexural strength of veneering ceramics for zirconia by way of cyclic loading test under different pH conditions. This is the motivation of the study whose objective is to evaluate the effect of humidity and pH value on the biaxial flexural strength of bilayered ceramics experiencing cyclic loading. The results may serve as a guide to clinical applications of all-ceramics dental crowns and to developing new-style all-ceramics dental crowns. The zirconia (t-ZrO₂) used in this study is obtained from Guang Xi Cercon Corporation and the content of ZrO₂ is 95%. A Weibull statistical methodology is utilized to identify both the difference in magnitude of the calculated failure stress and the distribution of the failure stress data.

Materials and Methods

The materials used in this work were obtained from Guang Xi Cercon Corporation and their properties were provided by the manufacturer (see Table 1).

As indicated in (Pinto et al, 2008), the normal pH of saliva varies from 6.8 to 7.2. However, when an individual takes foods or drinks into the mouth, those foods may produce acidic or alkaline environments, resulting in acidic or alkaline environment. As a response to this phenomenon, the saliva flux may increase in order to provide bicarbonate ions that will adjust the oral pH to the normal level again (within buffering capacity). But, when the buffering capacity is exceeded, the oral pH cannot be adjusted to the normal level by buffering capacity of saliva flux. Thus, the oral pH may vary even from 4 to 8. To assess the effect of extreme pH value of the oral cavity on the biaxial flexural strength of bilayered ceramic, this study considers three experimental groups (pH=4, pH=7, and pH=8) and one control group (dry) with 60 specimens each. The specimens in each group are subjected to 10,000 cyclic loadings.

<table>
<thead>
<tr>
<th>Material</th>
<th>Cercon base</th>
<th>Ceron Ceram S powder dentine</th>
<th>Ceron Ceram S powder liner</th>
</tr>
</thead>
<tbody>
<tr>
<td>Manufacturer</td>
<td>Degudent GmbH, Hanau-Wolfgang, Germany</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Batch</td>
<td>18002346</td>
<td>60668, 63481</td>
<td></td>
</tr>
<tr>
<td>Composition</td>
<td>ZrO₂ (H₂O)₂ : 95 (&lt;2 H₂O₂); Y₂O₃ : 5; Al₂O₃ + other oxides &lt; 1 (+SiO₂)</td>
<td>SiO₂ : 60.0 – 70.0; Al₂O₃ : 7.5 – 12.5; K₂O : 7.5 – 12.5; Na₂O : 7.5 – 12.5</td>
<td>SiO₂ : 60.0 – 70.0; Al₂O₃ : 7.5 – 12.5; K₂O : 7.5 – 12.5; Na₂O : 7.5 – 12.5</td>
</tr>
<tr>
<td>Modulus / GPa</td>
<td>210</td>
<td>60–70</td>
<td>60–70</td>
</tr>
<tr>
<td>Flexural strength / MPa</td>
<td>900</td>
<td>80–90</td>
<td>80–90</td>
</tr>
<tr>
<td>CTE /10⁻⁶K⁻¹</td>
<td>10.5</td>
<td>9.5</td>
<td>9.5</td>
</tr>
</tbody>
</table>

Note: Thermal expansion coefficient is in 10⁻⁶K⁻¹ and valid between 25 and 500°C

Preparation of Zirconia Core

In the preparation of specimens, discs (16 mm diameter and 0.8 mm thickness) are cut from cercon zirconia blocks by an electrical high-precision saw (Labcut1010, EXTEC, USA), and then polished by grinding and polishing machine (Labopol-6, Struers, Danmark), 320 and 600 grit silicon carbide paper prior to sintering. This is followed by a process of sintering to full density according to the manufacturer’s instructions (1350 °C for 6 h) by Cercon heat (DeguDent GmbH, Germany). It should be mentioned that the specimens are cut oversized because approximately 20 % shrinkage will occur during the process of sintering. Disc dimensions are measured with a high-precision micrometer caliper with an accuracy of ± 0.01 mm. The total time of the whole process of zirconia core preparation, including heating, sustaining and cooling, is approximately 8 hours. Nominally identical disc shaped specimens containing Y-TZP core and with the dimension of 16 mm diameter and 0.8 mm thickness are then produced.
Finally, the specimens of 0.8 mm thickness are treated like fixed partial dentures.

**Preparation of Core Veneer Specimen**

Regarding the core veneer specimens, the core is sandblasted with 110 μm Al₂O₃ particles for 30 s at 2.5 bar pressure (Rocatec-Pre, Espe, Seefeld, Germany) after sintering. Before veneering, all core specimens are cleaned with ultrasonic cleaner and air-dried. According to the procedure of manufacturers (Cercon, DeguDent GmbH, Germany), the veneering porcelain is built up to the final dimensions (1.6 mm × 16 mm) in a metal mold by Programat furnaces (P200, Ivoclar Vivadent Inc., Liechtenstein).

**Specimen Soaking**

The solutions used in specimen soaking consist of the following ingredients: 0.78 g Na₂HPO₄•2H₂O; 0.4 g KCl; 0.4 g NaCl; 0.795 g CaCl₂•2H₂O; 0.005 g Na₂S•2H₂O and 1 g Urea. Three solutions are produced with pH of 4, 7 and 8. The pH value of the solution is adjusted to 4 by adding HCl and to 8 by adding NaOH. The pH values of all storage media are measured using a pH meter (WPH2, DWYER, USA). The 180 specimens used in the test are equally divided into the three groups and each group is immersed in one solution for 2 weeks at 37 ºC. And the other 60 specimens used as control group are not soaked in the solutions. In order to simulate laminated composite of all-ceramic crown, specimens are cemented to polyethylene fiber resin posts by epoxy resin after soaking. The polyethylene fiber resin posts, having an elastic modulus similar to that of dentin, are all incubated in water for more than 2 weeks before cementation to allow for hydroscopic expansion. After soaking, all of the specimens are used for fatigue test and flexural strength test in order to evaluate the influence of pH on fatigue damage and biaxial flexural strength of bilayered ceramics.

**Fatigue Tests**

The cyclic loading applied on the top surface (veneering porcelain) of the bilayered discs is delivered with a spherical tungsten carbide indenter (r = 3.18 mm) by a mechanical cycling machine (AG-20I, Shimadzu Corp, Japan) that can generate mechanical forces similar to those occurring in the chewing cycle. The specimens are tested with a controlled stroke profile: maximum load (biting force) of Pₘ =600 N for 10,000 times (Pittayachawan et al, 2009); loading and unloading rates being 1000 N/sec; and a chewing frequency of 1/6 Hz. The steps of each load cycle consist of the indenter coming into contact with the specimen, loading to a maximum, holding for 0.2 sec, unloading, and lifting off (0.5 mm) from the specimen surface, then loading to a minimum load of 50 N. Fatigue tests are stopped after a pre-set number of loading cycles. It is estimated 65% specimens fractured during fatigue testing and the equivalent stress on zirconia was 900MPa during fatigue testing. We noticed that while the core (zirconia) can survive at 900MPa cycling load, the top surface material (porcelain veneer) might fail at a load level below 900MPa. It should be mentioned that the zirconia plays an important role as support materials in improving failure behaviour of brittle layer structures. The specimen is monitored for surface damage with an optical microscope (LV150, Nikon, Japan). All specimens are selected for evaluating the extent of subsurface damage.

**Biaxial Flexure Strength Measurement**

After fatigue testing, flexural strength tests are carried out for those un-fractured specimens using the piston-on-three-balls method according to ISO standard 6872 (International Organization for standardization, 2008) in a universal testing machine (AG-20I, Shimadzu Corp, Japan). The biaxial strength tests conducted under the same environment condition (in air). The disk containing specimens is supported by three balls. The balls have a diameter of 4 mm each and are positioned at an angle of 120° to each other, forming a circum-circle 12 mm in diameter. The piston (diameter 1.5 mm) acts on the center of the specimen at a speed of 1 mm/min until the specimen is broken. A closed-form solution in the guidelines of Hsueh (2009) is used to calculate the biaxial flexural strength of different regions of specimens. To investigate the failure mode, the fracture fragments of each bilayered specimen are reassembled and inspected by an optical microscope (LV150, Nikon, Japan).

**Statistical Analysis**

Biaxial flexural strength data corresponding to each group are analyzed using a one-way analysis of variance (ANOVA) and comparison is made by way of Tukey’s post hoc test at a pre-set significance level of 5 %. In addition, in order to calculate the Weibull modulus, the biaxial flexural strength data of each group are listed in an ascend order. Then the Weibull modulus is calculated by the equation (Weibull, 1951):

\[ P_i = 1 - \exp \left[ -\left(\frac{\sigma}{\sigma_0}\right)^m \right] \]
where $\sigma_0$ is a constant, $m$ is the Weibull modulus, $\sigma$ is the biaxial flexural strength, $P_f$ is the fracture probability of the 60 specimens in each group. The variables $P_f$ and $\sigma$ are transformed to $\ln[1/(1-P_f)]$ and $\ln \sigma$, respectively. This is the double natural logarithm of $1/(1-P_f)$ and the natural logarithm of $\sigma$, which is usually used for Constructing plots with $\ln[1/(1-P_f)]$ as the ordinate, a corresponding $\ln \sigma$ as the abscissa, and the slope of the plot is equal to $m$ (International Organization for Standardization, 2008).

**Results**

**Fatigue Damage**

After fatigue test, the statistical analysis is performed using the testing results of subsurface damage. The extent of damage can be divided into several cases, which include conspicuous crack and inconspicuous crack. The results of fatigue tests confirm the influence of pH on fatigue damage of bilayered ceramics to be significant, especially in alkaline environment; where the conspicuous damage is visibly increasing by 50% in contrast to that in dry condition (see Fig. 1).

**Biaxial Flexural Strength**

Fig. 2 shows that discs have the highest value of flexural strength in the dry condition and the lowest value of flexural strength in pH 8. The flexural strength of the discs in the dry condition is approximately 7% higher than that in pH 7, 20% higher than that in pH 4 and 50% higher than that in pH 8. The strength values of the material at the bottom of the discs are 95% higher than those of the materials at the top of discs regardless of storage medium. While the flexural strength of specimens in acid solutions is approximately 30% stronger than those were soaked in alkaline solutions. The mean failure stresses at the interface 1 (the contact surface which is near to zirconia between zirconia and porcelain) and interface 2 (the contact surface which is near to porcelain between zirconia and porcelain) of discs are lower than those at the disc surface (top and bottom). Fig. 2 shows the mean value and standard deviation of biaxial stress at $r = 0$ (center of disc). The biaxial stress through the disc thickness is shown in Fig. 3. The positive and negative values represent the tensile and compressive strength, respectively. There are two stress zones, the compressive force zone (0 ~ 0.6 mm thickness) and the tensile force zone (0.6 ~ 1.6 mm thickness). Because of the different elastic properties between the two layers, the stress is discontinuous at the interface and the stress gradients are different between the two layers.

**Damage Maps Analysis**

Damage maps analysis is a characterization tool to detect the types of crack formed by cyclic loading.
Subcritical crack growth is notable for its extreme sensitivity to applied stresses (Esquivel-Upshaw et al., 2001). Fig. 4 shows the influence of cyclic loading on crack modes of bilayered structures in both wet and dry conditions. Fig. 4a shows contact damage on the surface after 10,000 cycles in dry air. Fig. 4b shows that radial cracks (RC) are generated outside Hertzian cone cracks; circumferential ring cracking (CRC) is formed outside the indentation and the subsequent secondary radial cracking (SRC) is formed outside the CRC after 10,000 cycles in pH 4. In Fig. 4c the sub-critical radial cracks extend to the edge of the disc after 10,000 cycles in pH 8. Fig. 4d shows contact damage on the surface after 10,000 cycles in pH 7. Fig. 4e displays a cross-section of the Hertzian cone cracks, which are under the contact region of the indenter. The Hertzian cone crack as observed in Fig 4e expands downward as a relatively high rate during fatigue testing or biaxial strength testing. The angle of the outer cone crack relative to the occlusal surface is about 22 ± 5° and that of the inner cone relative to the occlusal surface is about 55 ± 15°, which expands downward at a relatively high rate and a sharp angle. Note that secondary radial cracking starts from the top circumferential ring cracking. It is different from the bottom-initiated radial cracking in that it does not pass through the indentation (Wang and Darvell, 2007).

**Weibull Analysis**

Most ceramics are reported to have values of the Weibull modulus in the range of 5 ~ 15 (Johnson, 1981). In this study, the Weibull modulus of specimens is averagely 17.75, 17.88, 10.53 and 17.92 in pH 4, pH 7, pH 8 and dry environment respectively (Table 2). A higher Weibull modulus reflects smaller or fewer defects, thus greater structural reliability (Ritter, 1995).

**TABLE 2 THE WEIBULL MODULUS OF THE DIFFERENT PART OF THE SPECIMENS**

<table>
<thead>
<tr>
<th></th>
<th>pH4</th>
<th>pH7</th>
<th>pH8</th>
<th>Dry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Top</td>
<td>17.73</td>
<td>17.87</td>
<td>10.53</td>
<td>17.92</td>
</tr>
<tr>
<td>Interface 1</td>
<td>17.73</td>
<td>17.85</td>
<td>10.53</td>
<td>17.90</td>
</tr>
<tr>
<td>Interface 2</td>
<td>17.78</td>
<td>17.90</td>
<td>10.52</td>
<td>17.92</td>
</tr>
<tr>
<td>Bottom</td>
<td>17.74</td>
<td>17.90</td>
<td>10.54</td>
<td>17.92</td>
</tr>
<tr>
<td>Average</td>
<td>17.75</td>
<td>17.88</td>
<td>10.53</td>
<td>17.92</td>
</tr>
</tbody>
</table>

**Discussion**

It is found from the experimental results that the flexural strength of specimens tested under the dry condition is approximately 7 ~ 50% higher than that of specimens tested in a wet environment; the flexural strength of specimens under dry condition is approximately 7% higher than that of specimens in neutral solutions (pH=7); the flexural strength of specimens in acid solutions (pH=4) is approximately 30% higher than that of specimens in alkaline solutions (pH=8). These findings show that the changes in flexural strength under cyclic loading are closely associated with the humidity and pH value.

Firstly, the flexural strength of the material is impaired by water. It is known that none of the examined porcelains is chemically inert, even in a neutral aqueous environment. The causes may be chemical and physical actions which result in slow crack growth. On one hand, the reaction between water molecules and Si-O-Si bonds results in chemical destruction, which is one of the mechanisms for slow crack propagation. As explained in (Suputtamongkol, 2003), the process is described as follows: (1) formation of a hydrogen bond between a water molecule and Si-O-Si bonds at the crack tip; (2) interaction of the lone pair of electrons from the oxygen atom in water (O\textsubscript{w}) and the Si atom; (3) formation of two new bonds between O\textsubscript{w} and Si, and between the hydrogen and oxygen atoms from the silica molecule; (4) rupture of the bonds between hydrogen and O\textsubscript{w} leads to Si-O-H groups on fracture surfaces. These Si-O-H groups will not reform if there is no external energy being supplied.
Secondly, flexural strength degradation may be related to the dissolution of porcelain surfaces promoted by an alkaline or acidic storage medium. The composition of ceramic includes various oxides that can be divided into three categories (alkaline oxide, acidic oxide, neutral oxide). The contents of the various components in the bilayered ceramic discs of our experiment are as follows: SiO₂ 60.0 ~ 70.0%; Al₂O₃ 7.5 ~ 12.5%; K₂O 7.5 ~ 12.5%; Na₂O 7.5 ~ 12.5%. When the discs are soaked in acidic or alkaline solution, there will be some chemical reactions which will result in the decomposed erosion of the discs. The chemical reaction processes are as follows.

\[ \equiv \text{Si}-\text{O}-\text{Na} + \text{H}^+ \rightleftharpoons \equiv \text{SiOH} + \text{Na}^+ \]  
(1)

\[ \equiv \text{Si}-\text{O}-\text{K} + \text{H}^+ \rightleftharpoons \equiv \text{SiOH} + \text{K}^+ \]  
(2)

\[ \equiv \text{Si}-\text{O}-1/3\text{Al} + \text{H}^+ \rightleftharpoons \equiv \text{SiOH} + 1/3\text{Al}^{3+} \]  
(3)

\[ \equiv \text{SiOH} + \text{HO}-\equiv \equiv \text{Si}-\text{O}^- + \text{H}_2\text{O} \]  
(4)

\[ \equiv \text{Si}-\text{O}^- + \text{OH}^- \rightarrow \equiv \text{SiOH} + \equiv \text{SiO}^- \]  
(5)

The chemical reaction equations 1, 2, 3 and 4 are the major chemical reaction processes of the discs in acidic solution. The chemical reaction equation 5 is the major chemical reaction processes of the discs in alkaline solution. According to the chemical reaction equations 1, 2 and 3, the surface of the discs will form =SiOH layers in acidic solution. The insoluble protecting films =Si-O-Si= subsequently is formed through polymerization reaction of =SiOH in acidic solution (eq. 4). The insoluble protecting films =Si-O-Si= can prevent the decomposed erosion by acidic solution. However, both the rupture of =Si-O-Si= and the increase of =SiO^- result in the structural damage and continual dissolution of SiO₂ of the discs in alkaline solution (eq. 5). And the insoluble protecting films =Si-O-Si= cannot be formed in alkaline solution, too. All of those factors result in the increase of radial cracks in pH 8 (Fig. 3(c)) and finally in a decrease of strength than that in pH 4 (Fig.2 and Fig. 3(b)).

All of these cracks grow slowly in cyclic loading with time, but the inner cone is made larger in pH 8 than that in both pH 4 and pH 7 or under the dry condition by hydraulic pumping and chemical etching. At the same time, the number and length of radial cracks increase with the increase of inner cone cracks. The strength of the disc decreases with the increase in radial cracks.

It should be pointed out that adjustments should be made when using the results of the present study for predicting clinical behavior of porcelains, because the oral environment presents differences from the storage media used here. In order to obtain authentic data, further study is needed to simulate the real and complex oral environment.

**Conclusion**

The main influence of storage medium on bi-axial flexural strength of layered ceramics is investigated in this study. The study found that the flexural strength of specimens tested under dry conditions is approximately 7 ~ 50% stronger than that of specimens soaked in the solution; and the flexural strength of specimens soaked in an acid solution (pH=4) is approximately 30% greater than that of specimens soaked in an alkaline solution (pH=8). The Weibull modulus of specimens is approximately 17.73, 17.88, 10.53 and 17.92 in pH 4, pH 7, pH 8 and dry environment respectively. Thus, the specimens have greater structural reliability in pH 4, pH 7 and dry environment than that in pH 8. The chemical etching of aqueous solution is one of the main influencing factors on the bi-axial flexural strength of layered ceramics in water, especially in alkaline water. So how to avoid chemical etching of aqueous solution is one of the challenges in applications of dental layered ceramics.

**ACKNOWLEDGMENT**

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Born in Guilin city, China. **Professor Jun Ou** is working as a professor in the College of Material Science & Engineering at University and Guilin University, China. He received his Bachelor of Engineering degree in Chemical metallurgy from Chengdu University of Science and Technology, China, in 1986, his Master of Engineering in inorganic chemistry from Guangxi Normal University in 2002, and obtained his PhD in Biomedical Engineering from University of Sichuan in 2007. He taught on the faculty of the College of Material Science & Engineering at Guilin University of Technology for a total of twenty years. He also leads a biomedical research laboratory shared jointly by between Guangxi Medical University and Guilin University of Technology. His research focuses on Chemical modification, surface treatment and characterization of nanocrystalline-based and biomacromolecule-based systems for and their biomedical applications, including tissue engineering scaffolding and systems for controlled delivery of drug.

**Qing H. Qin** received his Bachelor of Engineering degree in mechanical engineering from Chang An University, China, in 1982, and his Master of Science and PhD degrees from Huazhong University of Science and Technology (HUST), China, in 1984 and 1990, respectively. Both MS and PhD are in applied mechanics. He joined the HUST Department of Mechanics as an associate lecturer in 1984, and was promoted to lecturer of mechanics in 1987 during his PhD candidature period. After spending ten years on lecturing at HUST, he was awarded a DAAD/K.C. Wong research fellowship in 1994, which enabled him to work at the University of Stuttgart in Germany for nine months. In 1995 he left HUST to take up a postdoctoral research fellowship at Tsinghua University, China, where he worked until 1997. He was awarded a Queen Elizabeth II fellowship in 1997 and a Professorial fellowship in 2002 at University of Sydney and stayed there till December 2003, both by the Australian Research Council. He is currently working as a professor in the Research School of Engineering at the Australian National University, Australia. He was appointed as a guest professor at HUST in 2000 and was a recipient of the J.G. Russell Award from the Australian Academy of Science. He has published over 200 journal papers and six monographs.