NONDESTRUCTIVE EVALUATION OF SOME MEDICAL PROSTHESIS COMPONENTS MADE FROM ZIRCONIA AND ALUMINA USING RESONANT ULTRASOUND SPECTROSCOPY

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ABSTRACT

This paper presents a possibility to use Resonant Ultrasound Spectroscopy as nondestructive evaluation method for ceramic elements of prosthesis such as ceramic femoral heads. With this method can be emphasized the incorrect sintering of these elements as well as eventually cracks. If the number of resonance peaks is big enough, the elements of elasticity matrix can be determined, demonstrated by effective measurements on zirconia with different stabilizers cylindrical samples taken into study.

Key words: medical prosthesis, zirconia, alumina, Resonant Ultrasound Spectroscopy.

1. Introduction

Over the last decades, total hip arthroplasty (THA) has become one of the most successful surgical operations. Symptoms such as pain and loss of functions indicate THA as the solution. These symptoms can appear in different forms, as rigidity, deformity, limb shortening or movement reduction. THA is the solution preferred also in case of osteoarthritis, rheumatoid arthritis, psoriatic arthritis, avascular necrosis, trauma and tumors [1], [2].

The femoral head has a spherical shape, slightly flattened both anteriorly and posteriorly. The femoral head is covered with articular cartilage with the exception of the fovea. On the acetabular face, fovea covers a horseshoe shaped zone and it is set in the central region. The fovea is thick, not articulated and it is filled with the some fat, the sinusoidal membrane and the ligamentum rotondum [3]. The articular capsule is inserted on the acetabular edge proximally and on the femur distally. The dense fibrous tissue on the capsule is reinforced anteriorly and inferiorly, and only slightly in the posterior aspect. A single thick layer called orbicularis zone surrounds the femoral head, increasing the stabilizing function of the fibrous cartilaginous head ring.

Today, a large variety of solutions are available: from the use of ultra-high molecular weight polyethylene for the hemispherical socket, which is fixed onto the acetabulum, to the ceramic one and metallic or ceramic head. Most of the implant is nowadays modular (Figure 1a). Modularity assures the possibility of adapting the geometry of the prosthesis to the joint
morphology of the patient. This solution provides more flexibility during primary surgery and simplified revision procedures [4].

![Typical components of a modular hip prosthesis: a) principles; b) photo](image)

Fig. 1. Typical components of a modular hip prosthesis: a) principles; b) photo

Usually, this type of prosthesis features a modular head to be inserted into a neck, which is on its turn fixed to a stem. The stem is inserted in the medullary canal of the femur. Both socket and stem can be fixed using methyl-methacrylate cement or by press-fit and direct bone ingrowth into metallic surface.

During the last three decades, oxide based ceramics are being increasingly introduced in orthopedic surgery as replacement of metallic parts as the hard, bio-inert component. Generally, in total hip-implants, the ceramic femoral heads are prepared with a tapered bore in which the projected male counter face of the metallic stem is fitted to fix the head with the stem (Figure 1b).

Oxide based ceramics are formed by close packed crystals of oxides of aluminum or zirconium metals. The arrangements of the crystals and the presence of impurities determine the characteristics of the resulting material. The medical grade ceramics are the most chemically and biologically inert of all materials. The ceramic is very resistant to scratches from the tiny particles that occasionally land between the artificial joint surfaces, be it particles of bone, cement or metal. The main disadvantage of medical grade ceramic materials is their fragility. The ceramic materials cannot deform under the stress, as plastics and metals can do. When the stress acting on medical grade ceramics exceeds a certain limit, ceramic material bursts, formally explodes in many splinters.

Alumina ceramic is composed of very small crystals of aluminium oxide; the impurities make less than 0.5% of the material’s volume. The obtaining of femoral heads from alumina is made by Hot Isostatic Pressure (HIPing) procedure.

Zirconia ceramic is one of the highest-strength ceramic suitable for medical use. It is two to three times stronger material than alumina ceramic. Thus one can make the femoral out of this material that is smaller (22mm diameter) than the femoral heads made out of alumina ceramic (28-32mm). The surface of the zirconia ball can be made smoother than the surface of the femoral heads made from alumina ceramic; the wear produced by the zirconia head coupled with polyethylene cup is only half as large as the wear produced by alumina ceramic ball in identical coupling. Whereas the high strength and low wear made the zirconia ceramic is so attractive for constructors of total hips, the instability of zirconia is a big and not well-understood problem. Ceramic made out of the strong tetragonal crystals may spontaneously transform in other crystalloid forms. The ceramic consisting of these other crystalloid form is weak, rough, and fragile. Thus, zirconia ceramic must be "stabilized" by addition of oxide of another metal,
yttrium. The obtaining of femoral heads from zirconia is made by sintering at approximate 1430°C and 1250 Barr pressure.

The main physical-mechanical requirements imposed to medical grade alumina are standardized in [5] and those for medical grade zirconia in [6].

This paper proposes a method for nondestructive evaluation of physical-mechanical properties of ceramic femoral heads made from alumina and zirconia, based on resonant ultrasound spectroscopy (RUS). This method has the advantage of being rapid, has good accuracy and can be used in quality control of femoral heads.

2. Theoretical principles of resonant ultrasound spectroscopy

Let \( \nu \) be an isolated body, that is, one bounded by a closed stress free surface. Let \( C_{ijkl} \) be its elastic stiffness tensor and let \( \rho \) be its density; both quantities may vary with position in \( \nu \).

Let \( \omega \) be a non-negative real number, and \( \vec{u}(r) \) be a real valued function at position \( r \) in \( \nu \).

Then, the combination \( \{ \omega, \vec{u} \} \) is a free oscillation or resonance if the real-valued displacement field

\[
S(\vec{r}, t) = \Re \left( \vec{u}(\vec{r}) \exp(j\omega t) \right)
\]

where \( j = \sqrt{-1} \), satisfies the elastic equations of motion in \( \nu \) and the stress-free boundary condition on its surface.

The potential energy \( E_p \) associated with the displacement field \( \vec{u} \) is given by the strain energy [7]

\[
E_p = \frac{1}{2} \int_{\Omega} C_{ijkl} \partial_j u_i \partial_k u_l dV
\]

where \( u_i, i=1,2,3 \) are the Cartesian components of \( \vec{u} \) and we are using the summation convention for repeated indices. The corresponding kinetic energy \( E_k \) is given by

\[
E_k = \omega^2 k
\]

where

\[
k = \frac{1}{2} \int_{\nu} \rho u_i u_i dV
\]

The quantity

\[
I = \omega^2 k - E_p
\]

is stationary if and only if \( \omega \) and \( \vec{u} \) are a resonance of \( \nu \) [7].

By stationery it means that the value of \( I \) does not change if we replace \( \vec{u} \) by \( \vec{u} + \delta \vec{u} \), where \( \delta \vec{u} \) is any small displacement. Stationery, happily, turns out to be the key to a powerful approximation technique, called Rayleigh-Ritz method.

Let \( \{ \Phi_\lambda(\vec{r}), \lambda = 1,2,\ldots, N \} \) be a set of \( N \) specified functions over \( \nu \)

\[
u_i = a_{i,\lambda} \Phi_\lambda
\]

If we insert the equations (6), (4), (3) and (2) into (5), we obtain

\[
I = \omega^2 \alpha \cdot \vec{k} \cdot \alpha - \alpha \cdot \vec{E} \cdot \alpha
\]

where \( \alpha \) is a vector comprising the juxtaposed components of \( a_{i,\lambda} \). \( I \) is stationery if and only if

\[
\omega^2 \vec{k} \alpha = \vec{E} \alpha
\]

Eq. (8) is a standard form for the generalized symmetric eigenvalue problem. Both \( k \) and \( E \) are real and symmetric, and \( k \) is positive definite.

The essence of Rayleigh-Ritz method consists into a convenient way of writing the set of basis functions \( \Phi_\lambda \), the simplest choice for \( \Phi_\lambda \) is represented by the form
\[ \Phi_\lambda = x^{\eta(\lambda)} y^{\theta(\lambda)} z^{\xi(\lambda)} \]  
(9)

where \( \eta, \theta, \xi \) are positive integers.

Then

\[ K_{\lambda} = \frac{1}{2} \int \rho x^{\eta(\lambda)+\eta(\lambda)} y^{\theta(\lambda)+\theta(\lambda)} z^{\xi(\lambda)+\xi(\lambda)} dV \]  
(10)

There is an analogous but more complicated expression for \( E \).

In any region \( S \subseteq \nu \) in which a function \( f(x, y, z) \) can be written as a sum of monomial basis functions

\[ I_{\nu}(l, m, n) = \int_S x^l y^m z^n dV \]  
(11)

For simple and regulated geometrical shapes of region \( S \) (parallellepiped, cylinder, ellipsoid, etc.), the integral (11) can be analytically calculated and for complicated shapes, its evaluation can be made only numerically.

Thus, eigenvalues that correspond to resonance frequencies and eigenvectors that correspond to displacement vector can be evaluated.

3. Studied samples

In order to validate the resonant ultrasound spectroscopy (RUS) method and for study the way in which the cesium and yttria oxides stabilize the zirconia, cylindrical samples sinterized at 1500°C, approximate 6 hours, were taken into study, their properties being presented in Table 1.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Chemical composition</th>
<th>Density [kgm(^{-3})]</th>
<th>Elasticity modulus [GPa]</th>
<th>Shear modulus [GPa]</th>
<th>Poisson ratio</th>
<th>Molecular mass [GPa]</th>
<th>Crystallographic structure</th>
<th>Diameter [mm]</th>
<th>Height [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \pi 2 )</td>
<td>ZrO(_2)+10% (vol) CeO(_2)</td>
<td>5378.3</td>
<td>145.85</td>
<td>56.49</td>
<td>0.291</td>
<td>127.62</td>
<td>Monoclinic+tetragonal</td>
<td>10.73</td>
<td>9.9</td>
</tr>
<tr>
<td>( \pi 3 )</td>
<td>ZrO(_2)+15% (vol) CeO(_2)</td>
<td>5885.2</td>
<td>168.53</td>
<td>64.47</td>
<td>0.307</td>
<td>129.58</td>
<td>tetragonal</td>
<td>10.48</td>
<td>9.5</td>
</tr>
<tr>
<td>( \pi 4 )</td>
<td>ZrO(_2)+20% (vol) CeO(_2)</td>
<td>6263.0</td>
<td>193.43</td>
<td>73.16</td>
<td>0.322</td>
<td>131.53</td>
<td>tetragonal</td>
<td>10.38</td>
<td>9.09</td>
</tr>
<tr>
<td>( \pi 5 )</td>
<td>ZrO(_2)+5% (vol) CeO(_2)+5% (vol)Y(_2)O(_3)</td>
<td>5731</td>
<td>188.68</td>
<td>72.46</td>
<td>0.302</td>
<td>126.95</td>
<td>Tetragonal</td>
<td>10.76</td>
<td>9.1</td>
</tr>
<tr>
<td>( \pi 6 )</td>
<td>ZrO(_2)+4% (vol)Y(_2)O(_3)</td>
<td>5768</td>
<td>193.79</td>
<td>74.29</td>
<td>0.305</td>
<td>123.06</td>
<td>Tetragonal</td>
<td>10.69</td>
<td>9.44</td>
</tr>
<tr>
<td>( \pi 7 )</td>
<td>ZrO(_2)+8% (vol)Y(_2)O(_3)</td>
<td>5474.2</td>
<td>182.1</td>
<td>69.88</td>
<td>0.303</td>
<td>122.89</td>
<td>tetragonal</td>
<td>10.21</td>
<td>10.7</td>
</tr>
</tbody>
</table>

The density has been determined by standard procedures, the crystallographic structure being determined by neutrons diffraction at Joint Institute for Nuclear Research, Dubna, Russia and elasticity modulus, shear modulus and Poisson ratio were determined by measuring the longitudinal and transversal velocity of ultrasound in the studied samples, by pitch-catch method, the transducer being placed on the bases of cylinders.
Besides these, two types of femoral heads have been studied, one made from alumina medical grade and the other from zirconia stabilized with 6\%(vol) \text{Y}_2\text{O}_3. The femoral heads are presented in Figure 2, the properties being presented in Table 2.

![Fig. 2. Photos of the femoral heads: a) ZrO$_2$+6\%(vol)\text{Y}_2\text{O}_3; b) ZrO$_2$+6\%(vol)\text{Y}_2\text{O}_3$ with cracks; c) Alumina.](image)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Chemical composition</th>
<th>Density [kg/m$^3$]</th>
<th>Young modulus [GPa]</th>
<th>Shear modulus [GPa]</th>
<th>Poisson ratio</th>
<th>diameter [mm]</th>
<th>observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>$S_1$</td>
<td>ZrO$_2$+6%(vol)\text{Y}_2\text{O}_3</td>
<td>6060</td>
<td>210</td>
<td>82</td>
<td>0.28</td>
<td>28</td>
<td>Standard femoral head</td>
</tr>
<tr>
<td>$S_2$</td>
<td>ZrO$_2$+6%(vol)\text{Y}_2\text{O}_3</td>
<td>5950</td>
<td>185</td>
<td>72.3</td>
<td>0.28</td>
<td>28</td>
<td>Incorrect sinterized parameters</td>
</tr>
<tr>
<td>$S_3$</td>
<td>ZrO$_2$+6%(vol)\text{Y}_2\text{O}_3</td>
<td>6060</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>28</td>
<td>With cracks detected by penetrant liquid method</td>
</tr>
<tr>
<td>$S_4$</td>
<td>Alumina</td>
<td>3860</td>
<td>380</td>
<td>147.3</td>
<td>0.29</td>
<td>32</td>
<td>Standard femoral head</td>
</tr>
<tr>
<td>$S_5$</td>
<td>Alumina</td>
<td>3750</td>
<td>362</td>
<td>140.3</td>
<td>0.29</td>
<td>32</td>
<td>Incorrect sinterized parameters</td>
</tr>
</tbody>
</table>

4. Experimental set-up

The scheme of the measurement equipment is presented in Figure 3.

![Fig. 3. Experimental set-up](image)

A Network/Spectrum/Analyzer 4395A Agilent USA generates a sweep frequency between 80kHz and 250kHz in 1kHz step. The signal is amplified into a Power amplifier AC 1012 AG&TC Power Inc. USA and applied to an US emission transducer P111.O.06P3.1 type,
selected for the large bandwidth. The signal delivered by the reception transducer, identical with the emission one, is applied to the B port of the 4395A Agilent, the spectrum being acquired by a PC that is used also to program the functioning of the equipment with a numerical code developed in Matlab, via PCI-B interface. The command of the power amplifier is made with the same PC via RS232 interface.

In order to determinate the elasticity and shear moduli for the samples $\pi_2-\pi_7$, the propagation speed of longitudinal and transversal ultrasound wave were measured using transmission procedure. The emission transducer is applied on one base of the sample through a delay line from Perspex with 20mm length; the reception transducer is applied on the other base. For the measurement of the propagation speed of longitudinal wave, the transducer G5KB GE with central frequency of 5MHz was used, the coupling being assured by ZG-F Krautkramer gel and for the transversal waves, the transducer employed was MB4Y GE with central frequency of 4MHz, the coupling being made with honey.

The generation of emission impulses and the reception of the signals delivered by the reception transducer were made with PR 5073 Pulser Receiver Panametrics NDT USA. The digitizing of the signals and the measurements of the time of flight was made with the digital oscilloscope Wave Runner 64Xi Le Croy USA.

5. Results and discussions

The resonance spectra were traced for the zirconia cylindrical samples, noted $\pi_2-\pi_7$, presented in Table 1. Because the samples $\pi_2-\pi_7$ are axisymmetric, considered isotropic and homogeneous [7] we can conclude that every mode of such sample must fall into one of three classes

- Torsional axisymmetric pure shear motion consisting of rigid rotations of rings of material around the sample axis. The frequencies of these modes depends entirely upon the sample’s shear velocity
- Extensional axisymmetric mixtures of compression and shear motions
- Flexural modes through along paths that arte tilted with respect to the cylinder axis. The flexural modes occur in pairs named doublets, both members of which have the same resonance frequency.

In Figure 4 are presented the resonance spectra for the samples $\pi_2-\pi_7$ in the frequency range 80kHz-250kHz and in Figure 5 are presented the simulation of deformations for two extensional modes (Fig.5a and b) and three flexural modes (Fig.5c, d and e) for the sample $\pi_2$, made with the finite element method using SolidWorks 2011.

![Fig.4. Resonance spectrum for the cylindrical samples $\pi_2-\pi_7$](image)

The resonance frequencies obtained by simulations correspond very well to those experimentally obtained. In order to determinate, in the basis of experimentally measured resonance spectra, of
the main elastic properties of the sample, the inversion of data was used, implying conjugate
gradient method [7], minimizing the objective function

\[ F = \sum w_i \left( f_i^{(p)} - f_i^{(m)} \right)^2 \]  

where \( f_i^{(p)} \) are the computed frequencies, \( f_i^{(m)} \) are the measured frequencies, \( w_i \) are the weights, which characterize the confidence we have in the measurements.

This optimization problem was numerically solved using Matlab 2013a.

Due to the fact that the number of peaks and corresponding, the resonance frequencies, is relatively small, the inversion was applied only for determination of \( E \) and \( G \), and not for geometrical dimensions and respectively for the densities of the cylindrical samples made from zirconia.

In the same manner, the elastic properties determined by inversion from the measured resonance spectra differ with less than 6% than those from Table 2.

![Fig. 5. Aspects of few resonant modes for the sample π2; a) extensional mode f= 163kHz; b) extensional mode f= 236kHz; c) flexural mode f=185kHz; d) flexural mode f= 208kHz; e) flexural mode f= 214kHz](image)

Having finished the experimental and simulation methods as well as those of inversion, we had proceeded to the nondestructive evaluation of femoral heads from zirconia stabilized with \( Y_2O_3 \) and alumina using resonant ultrasound spectroscopy.

We had at disposition, three femoral heads from zirconia, from which one is perfectly corresponding to the standard [6], the second is incorrect sinterized having the density smaller than the prescribed one, 5959kg/m\(^3\) (this is leading to the modification of all elastic properties) and the third presented a crack, emphasized by the method of penetrant liquid.

The ultrasound spectrum of these three femoral heads is presented in Figure 6.

It can be observed that there are big difference between the resonance frequencies and also the shapes of the spectra, so that the resonant ultrasound spectroscopy can become, effectively, a nondestructive method for the quality control of ceramic femoral heads.

In the case of femoral heads from alumina, it can be also observed that there are some difference between the shape for the spectra and the resonance frequencies of femoral head correct sinterized, having the properties according to standard [5] and of the one incorrect sinterized, with measured density 3750kg/m\(^3\) instead of 3860kg/m\(^3\), how it is for correct sintering. The resonance spectrum is presented in Figure 7. The big difference between the two spectra made that this procedure could be applied also at the quality control of the alumina femoral heads.
6. Conclusions

The resonant ultrasound spectroscopy can be used for quality control of certain ceramic elements of hip prosthesis, such as femoral heads. If the element are incorrectly sinterized, with a density smaller than the prescribed value and the elastic and shear moduli smaller, important modifications appears in the shape of the spectrum and the resonance frequencies. For the case in which the geometrical shape of the ceramic object to be controlled is relatively simple and the number the resonance peaks is big enough, the elasticity matrix elements can be determined integrally, as well as the density, with good precision.

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6. References