waveguide antenna using high-impedance ground plane, IEEE Antennas Wireless Propag Lett 2 (2003), 86-88.
5. G.H. Zhang and N.C. Yuan, Radation characteristics improvement in waveguide-fed slot antenna with a high-impedance ground plane (HIGP), Microwave Opt Tech Lett 45 (2005), 176-179.
6. F. Yang and Y.R. Samii, Refection phase characterizations of the EBG ground plane for low profile wire antenna applications, IEEE Trans Antennas Propag 51 (2003), 2691-2703.

BIOCOMPATIBILITY STUDY OF HYDROXYAPATITE-CHITOSAN COMPOSITE FOR MEDICAL APPLICATIONS AT MICROWAVE FREQUENCIES

Robin Augustine, Ullas G. Kalappura, and K. T. Mathew
Microwave Tomography and Materials Research Laboratory, Department of Electronics, Cochin University of Science and Technology, Cochin 682022, India; Corresponding author: ktm@cusat.ac.in

Received 12 March 2008

ABSTRACT: *Hydroxyapatite (HAp, $Ca10(PO_4)_6(OH)_2$) bioceramic and chitosan (poly [(β-1-4) D-glucosamine]) biopolymer show good biocompatibility in vivo. They have biological origin and show excellent interactions with microwave. Microwave study of HAp made using different drying techniques and their composites with chitosan in the ISM band is presented. Pastes are made using HAp and chitosan with different ratios of mixing. The dielectric properties of this composites match with that of human fat, collagen tissues. Some of the compositions exhibit dielectric property close to that of natural bone. This makes them more biocompatible and better substitutes for natural bone. Thus composite bioceramics can be considered as phantom model constituents for imaging purposes. Their dielectric properties prove that they are biocompatible.* © 2008 Wiley Periodicals, Inc. Microwave Opt Technol Lett 50: 2931–2934, 2008; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/mop.23806

Key words: *bioceramics; hydroxyapatite (HAp); chitosan; dielectric properties; osteoconductivity*

1. INTRODUCTION

Biomaterials such as hydroxyapatite (HAp) and chitosan are being used in bone replacement procedures, e.g., alveolar ridge reconstruction, periodontal bone filling, treatment of osteomytitis, maxillofacial and orthognathic implantation. HAp is produced by hydrothermal precipitation and successive drying, whereas chitosan is obtained from chitin after deacetylation procedure. Pastes are made using HAp and chitosan with different ratios of mixing. These pastes are used as fillers in bone fissures. It is found that their dielectric properties are more close to natural bone when compared to other bioceramic compounds. This makes them more biocompatible and better substitutes for natural bone.

Chitosan (poly [β-1-4]) D-glucosamine) biopolymer has proved to be effective in diverse fields such as clarification, purification, chromatography, paper and textiles photography, food, nutrition, agriculture, pharmaceutical and medical implants [1]. Chitosan mixed with HAps ($Ca_{10}(PO_4)_6(OH)_2$) and other bioceramic materials facilitates better collagen growth in implants [2].

2. THEORY

For the development of any material for a particular application, proper knowledge about the dielectric and mechanical properties of a material is essential.

Biological materials are very much influenced by microwaves. Therefore all biomaterials used for or deposited in living system will interact with microwaves in the same way as that of biological system.

The cavity perturbation technique [3] is employed for the study of dielectric properties of chitosan. A closed section of waveguide constitutes the waveguide cavity resonator. The cavity resonator can be of transmission or reflection type. Electromagnetic energy is coupled through the cavity using coupling irises at the ends of the cavity. A nonradiating slot is provided at the broad wall of the cavity for the introduction of the sample. The cavity resonates at different frequencies, depending on its dimensions. The basic principle involved in the technique is that the field within the cavity resonator is perturbed by the introduction of the dielectric sample through the nonradiating slot. The resonant frequency and the quality factor of the cavity are shifted due to perturbation. The determination of the complex permittivity and conductivity is based on the theory of perturbation. When a dielectric material is introduced in a cavity resonator at the position of maximum electric field, the contribution of magnetic field for the perturbation is minimum. The field perturbation due to the introduction of dielectric sample at the position of maximum electric field is related as [3]

$$\varepsilon_r' - 1 = \frac{f_c - f_s}{2f_s}\left(\frac{V_c}{V_s}\right), \tag{1}$$

$$\varepsilon_r'' = \frac{V_c}{4V_s}\left(\frac{Q_c - Q_s}{Q_c Q_s}\right). \tag{2}$$

The real part, ε_r', of the complex permittivity is usually known as dielectric constant. The imaginary part, ε_r'', of the complex permittivity is associated with dielectric loss of the material. The effective conductivity, σ_e, is given as

$$\sigma_e = \omega\varepsilon'' = 2\pi f\varepsilon_0\varepsilon_r'', \tag{3}$$

where $\tan\delta$ is the loss tangent, given as $\tan\delta = \varepsilon_r''/\varepsilon_r'$.

The relation of skin depth [3] with resonant frequency and conductivity is given by

$$\delta_s = \sqrt{\frac{2}{\omega\mu\sigma}}, \tag{4}$$

where ω is radian frequency, $\mu = 4\pi \times 10^{-7}$ H/m the permeability and σ the conductivity. Practically all applications of polymers in electrical and electronic engineering require materials with a low $\tan\delta$. However, one application that takes advantage of a high value of loss tangent is high-frequency dielectric heating. In this application, the efficiency of heating is usually compared [3] by means of microwave heating coefficient (J), which is defined as

$$J = \frac{1}{\varepsilon_r \tan\delta}. \tag{5}$$

Higher the J value, poorer will be the polymer for dielectric heating purposes. Of course, the heat generated in the polymeric material comes from the loss tangent, but the loss does not come

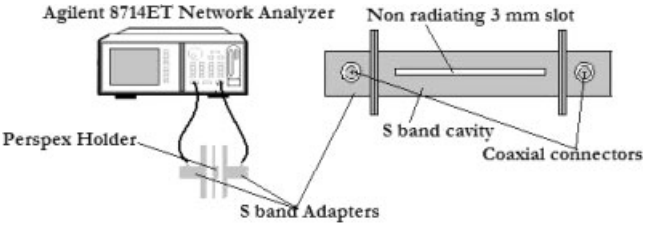

Figure 1 Experimental setup

entirely from the relaxation loss. Rather conductivity of the polymeric material may also contribute to the tanδ. This situation may be compared with ohmic heating in metals; the charge carriers are electrons, whereas those in dielectric polymeric materials may be ions.

Using the vector network analyzer, the *S*-parameters S_{11} and S_{21} are measured. Reflection coefficient R and transmission coefficient T are given as $R = S_{11}^2$ and $T = S_{21}^2$. The absorption coefficient A can be obtained from the simple relation [4]: $A + R + T = 1$.

3. METHODS OF PREPARATION AND EXPERIMENTAL TECHNIQUES

The HAP was prepared by a hydrothermal precipitation [5, 6] route involving ammoniated calcium nitrate tetrahydrate ($Ca(NO_3)_2{\cdot}4H_2O$) and dihydrogen ammonium phosphate ($NH_4H_2(PO_4)$) at 200°C. The precipitate was aged in mother liquor for 24 h. Then it was filtered and washed thoroughly to get a filter cake which is dried using either freeze drying or furnace drying methods. Both powders are free flowing. The freshly obtained powders, both freeze dried and furnace dried ones, are pressed into pellets of 5 mm radius and 2.5 mm thickness using a cylindrical die, by applying pressures of 200 kg/in.^{2} and 1 tone/in.^{2}. HAps prepared are then separately calcined at different temperatures 500, 1000, and 1200°C for 4 h with a heating rate of 5°C/min, both in the form of powders and pellets. X-ray diffraction patterns (Figs. 2 and 3) of unsintered and sintered freeze-dried powder were taken to study the polycrystalline structure of the ceramic compositions. The chitosan powder is prepared from crustaceans such as shrimp and prawn. They are dried and powdered. Protein separation is done by subsequent treatment with alkali such as sodium hydroxide. It is followed by demineralization by treating it with hydrochloric acid. Then the sample is washed and dried. The resultant material is chitin, which is again deacetylated (treated with sodium hydroxide) to obtain chitosan [7]. The chitosan and HAp thus obtained are mixed in different ratios (HAp:chitosan – 1:19, 1:9, 2:1, 4:1, etc.) and ground well to form paste. This paste is pressed into pellets of 1 cm diameter and 0.3 cm thickness, which are subjected to successive sintering at 300°C. The cavity resonator is excited in $TE_{10}p$ mode initially, and the resonant frequency f_0 and the corresponding quality factor Q_0 of each resonant peaks of empty cavity are determined. These pellets are inserted into cavity and the dielectric parameters are determined one by one using cavity perturbation. For absorption measurements a holder is designed with a dimension of 35 mm × 70 mm, i.e., the inner dimension of the S-band waveguide is used, and having a filling thickness of 2.5 mm. The holder filled with different compositions of HAp:chitosan is kept between two coaxial-to-waveguide adapters and tightened, one at a time. Using the vector network analyzer, the *S*-parameters S_{11} and S_{21} are measured. Scattering parameter measurements were determined for unsintered HAP powder and those sintered at 900, 1000, and 1100°C, respectively. Using the *S*-parameters, S_{11} and S_{21}, the microwave absorption coefficient was deduced. Experimental setup for the determination of dielectric properties consists of a transmission type rectangular S-band cavity resonator and HP 8714 ET network analyzer as shown in Figure 1.

4. RESULTS AND DISCUSSION

The dielectric properties of HAp pellets are measured in ISM Frequency (2–4 GHz) band. The dielectric parameters of the pellets are tabulated in Table 1. The dielectric properties have no significant variation with frequency. They are calculated at a central frequency of 2685 MHz. In pellets, the thickly packed powder under pressure increases the dielectric constant, but above 1-ton pressure there will not be any substantial variation of ε_r'. The highly packed molecules in the pellet experiences high frictional strain causing low loss factor. Pellets subjected to low palletizing pressure exhibit increase in the dielectric constant ε_r'' as the sintering temperature is increased from 1000 to 1200°C. However, this variation is not observed for pellets prepared at 1 ton. ε_r'' is almost the same for those sintered at 1000 and 1200°C. However, slight variation in ε_r'' was observed when the temperature increased. Skin depth is a measure of depth of penetration of electromagnetic wave through the material and is inversely proportional to the conductivity. Skin depth of HAp is found to be in the same range as that of natural bone free of water and collagen.

In the case of HAp-chitosan composite bioceramics, the dielectric constant ε_r' at the central frequency is found to be 6.19, 5.59, 4.64, and 3.83 for HAp-chitosan combinations 1:19, 1:9, 2:1, and 4:1, respectively (Table 2). As the HAp chitosan is a polymer composite, the microwave heating coefficient J was included in this study. As J is less, the dielectric heating will be lower. The dielectric constants of HAp-chitosan composites are low because of their lower polarizability. The reason for this is the nominal water content.

The dielectric parameters of HAp-chitosan composite are the same as that of certain biological constituents of human body. A comparative study of dielectric parameters of human organs and their phantoms are given in Table 3. The ratio of composite can be

TABLE 1 Dielectric Parameters of HAp Pellets Measured at Center Frequency of 2684 MHz

Method of Preparation	Pressure (kg/in.^{2})	Sintering Temperature (°C)	ε_r'	ε_r''	Conductivity σ (S/m)	Skin Depth δ_s (m)
Freeze dried pellet	200	1000	2.67	0.014	0.002	0.197
		1200	3.19	0.073	0.0110	0.093
	1000	1000	3.89	0.063	0.0090	0.100
		1200	3.37	0.020	0.0030	0.178
Furnace dried pellet	200	1000	2.58	0.013	0.0020	0.222
		1200	3.33	0.012	0.0018	0.244
	1000	1000	3.76	0.008	0.0011	0.900
		1200	3.48	0.086	0.0013	0.086

TABLE 2 Dielectric Parameters of HAp-Chitosan Composite

Ratio of Composition of HAp and Chitosan (HAp:Ch)	Frequency (MHz)	ε_r'	ε_r''	Conductivity σ (S/m)	Skin Depth δ_s (m)	Microwave Heating Coefficient J
1:19	2430	6.48	0.318	0.00429	0.00493	3.16
	2680	6.19	0.522	0.00778	0.00349	1.92
	2970	5.93	0.489	0.00806	0.00326	2.05
1:9	2430	5.78	0.134	0.00181	0.00758	7.47
	2680	5.59	0.223	0.00332	0.00534	4.49
	2970	5.47	0.374	0.00617	0.00372	2.67
2:1	2430	4.77	0.323	0.00437	0.00488	3.10
	2680	4.64	0.397	0.00591	0.00400	2.52
	2970	4.53	0.347	0.00572	0.00387	2.88
4:1	2430	3.93	0.231	0.00313	0.00577	4.33
	2680	3.83	0.228	.00340	.00527	4.38
	2970	3.74	0.320	.00528	.00402	3.12

appropriately selected according to the required properties of the human sample. The dielectric parameters of HAp-chitosan composite at ratios 1:19, 1:9, 2:1, 4:1, and collagen, bone marrow, human abdominal wall fat, and human chest fat, respectively, falls in the same range. Because human abdominal wall fat falls in the range of bone marrow, it can be utilized by using 1:9 ratio. HAp-chitosan derivatives are known as biomaterials and essentially should be biocompatible. Their biocompatibility can be confirmed by the close similarity in their dielectric properties with that of human fat tissue, collagen, and bone marrow. They will not be rejected when implanted in human body and are capable of becoming part of the body itself eventually. Hence HAp-chitosan composites can be used for biological implant application such as in bioceramics for tissue regeneration. Because the dielectric properties of HAp-chitosan composites match with that of body parts they can be used as phantom materials for imaging studies. An environment with all the dielectric properties of bone marrow, fat, collagen, and breast can be simulated using these composites. It is seen that a composition of 1:19 is nearer to the permittivity of natural bone and it can be considered as a more compatible bone implant. But a trade off has to be attained when the strength of the implant is considered, and so, 1:9 (HAp:chitosan composition) will be more appropriate.

The XRD pattern of the unsintered powder (Fig. 2) coincides with standard HAP XRD pattern [6], whereas the XRD pattern of sintered sample at a higher temperature of 1000°C (Fig. 3) shows that the peaks are sharp and the sample is well crystallized. An increase in crystal perfection with temperature is observed.

Absorption characteristics are measured for pure HAp powder and HAp-chitosan composite. The measurement is conducted for powders sintered at different temperatures and particular thickness. The experiment is repeated for other thicknesses also. The absorption is minimum at higher temperature and lower frequencies. From the scattering parameter measurements it is inferred that HAP has nonuniform absorption characteristics with frequency for particular sintering temperature and 5 mm thickness (see Fig. 4).

The variation in microwave absorption coefficient of HAp: chitosan composites of thickness 2.5 mm with frequency is shown in Figure 5. This is due to the higher HAp content. The absorption coefficient is found to be highly frequency dependent. Being biological materials they show good absorption of microwave and

TABLE 3 Dielectric Constants of Human Organs and Their Phantoms

Human Organ at 37°C [8]	Dielectric Constant at 3 GHz	Equivalent Phantoms in Terms of % Composition of HAp:Chitosan
Collagen in vitro	5.5–6.5	1:19
Bone marrow in vitro	4.2–5.8	1:9
Human abdominal wall fat	4.92	2:1
Human chest fat	3.94	4:1

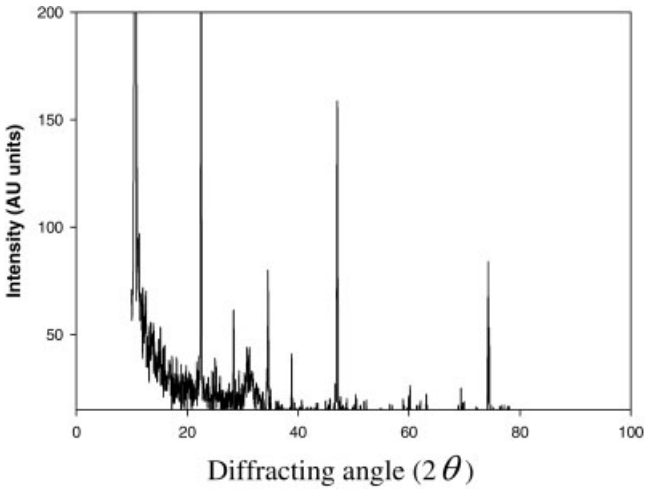

Figure 2 XRD of unsintered HAp

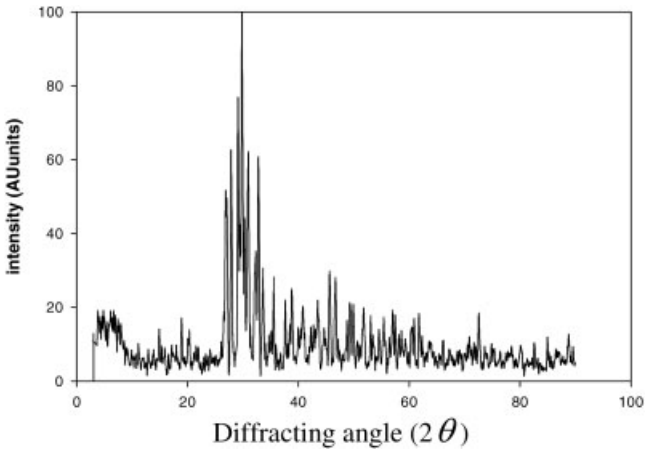

Figure 3 XRD of sintered HAp

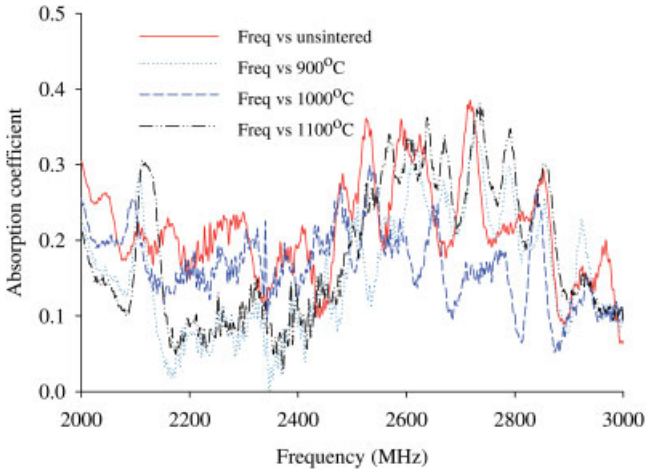

Figure 4 Variation of absorption coefficient with frequency of HAp powder at different sintering temperatures. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com]

can be used as microwave absorbing material in microwave tomographic chamber used for imaging. Almost similar microwave absorption is shown by different compositions.

But a slight absorption variation is obvious in the frequency range of 2000–2500 MHz. The 2:1 and 4:1 ratios are found to have absorbing power when compared with other compositions. These materials can be easily prepared, degradable, and biocompatible, and is expected to be an ideal candidate for microwave medical tomographic applications.

5. CONCLUSION

HAp was prepared using different drying techniques. The dielectric properties of HAp composites with chitosan are studied. The variations in absorption characteristics are discussed. The dielectric properties of HAp-chitosan composites are similar to that of fat, collagen, bone marrow, and these composites are biodegradable, nontoxic, and biocompatible. These composites can be easily prepared and used as phantom materials for microwave medical tomographic applications.

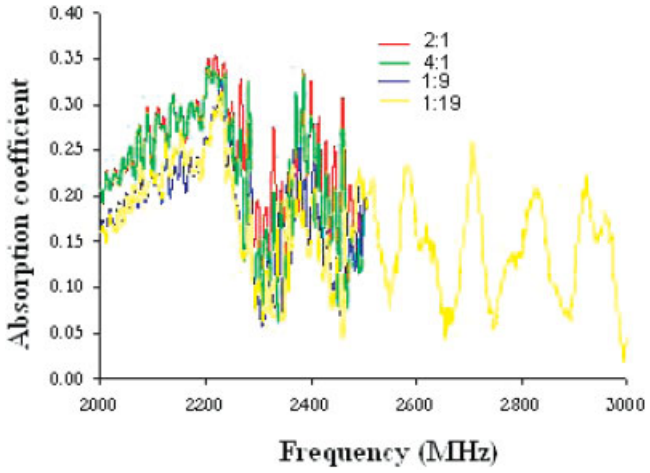

Figure 5 Variation of absorption coefficient with frequency of HAp-chitosan composite. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com]

ACKNOWLEDGMENT

The authors acknowledge the financial help received from the University Grants Commission, India, as Research Fellowship in Science for Meritorious Students (to R.A. and U.G.K.) and Emeritus fellowship (to K.T.M.).

REFERENCES

1. S.H. Pangburn, P.V. Trescony, and J. Heller, Partially deacetylated chitin: Its use in self-regulated drug delivery systems, In: Proceedings of the joint U.S.-Japan seminar on advances in chitin chitosan and related enzymes, University of Delaware, Newark, Delaware, April 24–27, 1984, pp. 3–19.
2. R.M. Finisie, A. Josue, T.V. Favere, and M.C.M. Laranjeira, Synthesis of calcium-phosphate and chitosan bioceramics for bone regeneration, Anais da Academiabrasileira de Ciencias 73 (2001), 1–11.
3. K.T. Mathew, S.B. Kumar, A. Lonappan, J. Jacob, T. Kurian, J. Samuel, and T. Xavier, Microwave studies on ZnS-HSR ionomers, Mater Chem Phys 79 (2003), 187–190.
4. C.Y. Lee et al., Electromagnetic interference shielding by using conductive polypyrrol and metal compound coated on fabrics, Polym Adv Technol 13 (2002), 577–583.
5. Harikrishna Varma and Rajagopalan shivakumar, Preparation and characterisation of free flowing hydroxy apatite powders, Phophorous Res Bull 6 (1996), 35–38.
6. J.S. Earl, D.J. Wood, and S.J. Milne, Hydrothermal synthesis of hydroxy apatite, J Phys: Conf Ser 26 (2006), 268–271.
7. J.C. Brine, Introduction chitin: Accomplishments and perspectives, In: Proceedings of the Joint U.S.-Japan Seminar on Advances in Chitin Chitosan and Related Enzymes, University of Delaware, Newark, DE, April 24–27, 1984, pp. xvii-xxiv.
8. S. Gabriel, R.W. Lau, and C. Gabriel, The dielectric properties of biological tissues. II. Measurements in frequency range 10Hz to 20GHz, Phys Med Biol 41 (1996), 2251–2269.

CMOS BANDPASS FILTERS FOR 77 GHz AUTOMOTIVE RADAR SYSTEMS

Lan Nan,[1,2] Koen Mouthaan,[1] Yong-Zhong Xiong,[2] Jinglin Shi,[2] Subhash Chander Rustagi,[2] James Brinkhoff,[2] and Ban-Leong Ooi[1]

[1] Department of Electrical and Computer Engineering, National University of Singapore, Singapore 117576; Corresponding author: nanlan@nus.edu.sg

[2] Institute of Microelectronics (IME) Agency for Science, Technology and Research, 11 Science Park Road, Science Park II, Singapore 117685

Received 18 March 2008

ABSTRACT: *This article presents a systematic design and measurement of narrow and wide bandpass filters at 77 GHz in a 0.18-μm CMOS technology. Various bandwidths from 10 to 50% are realized using a parallel coupled line topology in a thin film microstrip configuration. The relationship between the insertion loss and the 3-dB bandwidth of the filters is experimentally determined for this technology. The result is also compared with theoretical prediction.* © 2008 Wiley Periodicals, Inc. Microwave Opt Technol Lett 50: 2934–2937, 2008; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/mop.23854

Key words: *automotive radar system; bandpass filter; CMOS technology; thin film microstrip (TFMS)*