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Bond strength of implant to the bone tissue and the stress–strain state of "bone–implant" system by the finite element method

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Two types of new composite implant materials are investigated. Their mechanical characteristics and biocompatibility are determined. The first type of the biomaterials is based on silicate glass (SG) and hydroxyapatite. Both the natural (NHAp) and a synthetic (HAp) hydroxyapatites were used. The second type of the biomaterials was made of an ultrahigh-molecular polyethylene (UHMPE) and the NHAp. Composite materials of both the types were implanted into the rabbit femur. The bond strength between the bone tissue and the implants was determined in 2, 4, 10, and 25 weeks. The stress–strain state of bone–implant system was determined by the finite element method (FEM).

1. Introduction

The creation of artificial organs and substitutes for biological tissue and systems is one of the most vital problems of biomechanics. Various biomaterials, such as alumina, hydroxyapatite, titanium, Co–Cr–Mo and Ti–Al–V alloys, methylmethacrylate, polyethylene, composites based on porous nano-hydroxyapatite, collagen and alginate as well as many others, play an important role in the creation of artificial materials for replacing the bone tissue [1]–[6]. In this case, the problem of designing a material close to the natural bone tissue in its mechanical characteristics and biocompatibility is of key importance. Such materials include UHMPE and SG reinforced with hydroxyapatite [6]–[7].

In the present study, a procedure is described for obtaining natural (NHAp) and artificial (HAp) hydroxyapatites as well as composite materials based on them, namely SG–NHAp, SG–HAp and UHMPE–NHAp. The structure of bone tissue before and after deproteinization and the structure of composite materials based on UHMPE and NHAp (with different percentage) were investigated by the method of

scanning electron microscopy. Some mechanical characteristics of SG–NHAp, SG–HAp and UHMPE–NHAp biocomposite materials and the bond strength between a live bone tissue of rabbits and SG(60)–HAp(40), SG(60)–NHAp(40), UHMPE(70)–NHAp(30), and UHMPE(50)-NHAp(50) composites were determined. The stress–strain state of the bone–implant system was determined by the FEM.

2. Procedure

The NHAp was obtained from the bone tissues of cattle. Before deproteinization, the bone was freed of the soft tissue and fat and cut into 2-mm-thick layers. Then, the bone specimen was placed in a furnace and heat-treated in a suspended state in a stream of air at a temperature gradually increasing from room temperature to 300–320 °C for 6 h, after which it was kept at a constant temperature for 37–40 h.

The results of experiments, performed by the methods of thermogravimetry and infrared spectroscopy, show that the protein was removed from the heat-treated specimens of bone tissue practically completely.

Then, the deproteinized bone tissue (NHAp) was crushed to the particles whose size ranged from 10 to 50 μm. Such particles retain the four highest levels of the natural structure of bone tissue [8]. As a binder, we used an UHMPE. The NHAp and UHMPE particles were mixed for 10 min at T = 180–200 °C. The resulting mass was molded into plates under a pressure of 75 MPa.

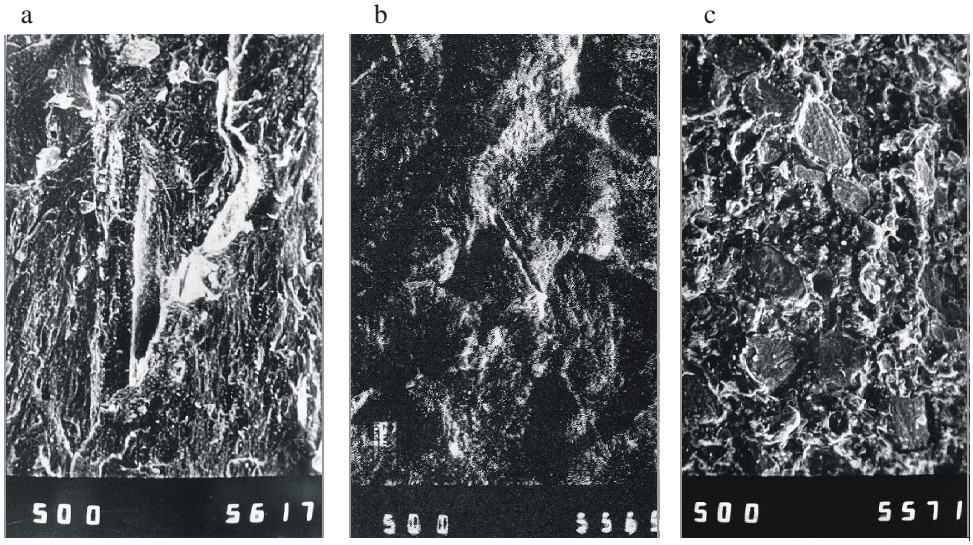

Fig. 1. Micrographs of bone tissue before (a) and after (b) deproteinization and of a UHMPE(60)–NHAp(40) composite material (c). Magnification 500×

The structure of bone tissue before and after deproteinization as well as the structure of compositions based on UHMPE and NHAp (with different percentage) were investigated by the method of scanning electron microscopy (figure 1).

The characteristics of mechanical properties of the materials were determined on flat dumbbell specimens. The working part of the specimens was 40 mm long, 3.3±0.1 mm wide, and 1.3±0.05 mm thick. The curves $\sigma_1 - \varepsilon_1$ had a pronounced yield point (figure 2); therefore, up to and beyond the yield point σ_1^Y, they were approximated in different ways:

$$\varepsilon_1 = a_1\sigma_1 \text{ at } \sigma_1 \le \sigma_1^Y \quad \text{and} \quad \varepsilon_1 = a_0 + a_2\sigma_1 \text{ at } \sigma_1 > \sigma_1^Y .$$

Based on the experimental results, we determined the initial elastic modulus $E = 1/a_1$, the breaking stress σ_1^*, and the strain at break ε_1^*.

Table 1. Mechanical characteristics of composite materials based on UHMPE and NHAp

Material, wt. %	σ_1^*, MPa	E, MPa	ε_1^*, %
UHMPE(70)–NHAp(30)	50.6±2.1	327±27	229±10
UHMPE(60)–NHAp(40)	35.2±3.7	368±45	161±25
UHMPE(50)–NHAp(50)	29.5±1.9	385±123	9±4

The experiments in uniaxial tension were carried out with an IMP 0.5 automatic testing machine controlled by an MTS testing system (USA). The mechanical characteristics of the materials are given in table 1 and figure 2.

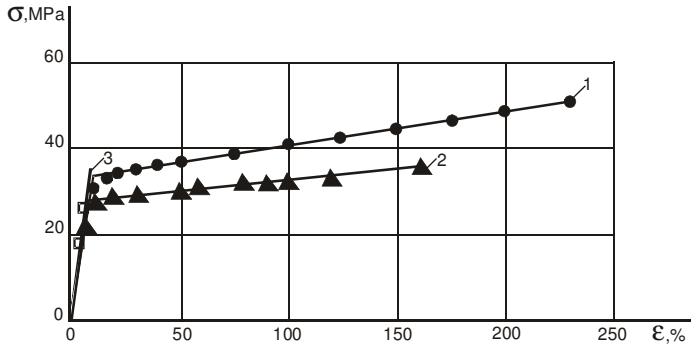

Fig. 2. Stress-strain relationship σ–ε for various composite materials:
UHMPE(70)–NHAp(30) (1); UHMPE(60)–NHAp(40) (2); UHMPE(50)–NHAp(50) (3)

The HAp was synthesized in laboratory conditions from $Ca(OH)_2$ and H_3PO_4 as a result of the reaction $10\ Ca(OH)_2 + 6\ H_3PO_4 \rightarrow Ca_{10}(PO_4)_6(OH)_2 + 18\ H_2O$.

The composite biomaterials were manufactured from silicate glass [73 SiO_2, 9 CaO, 10 Na_2O, 0.8 Al_2O_3, 3.2 MgO, 3 K_2O], which is a composite itself, and hydroxyapatites of two types, i.e., NHAp and HAp. The SG–HAp and SG–NHAp powders were mixed in propanol in a ball mill. The powder particles were 10–50 μm in size. The SG–HAp and SG–NHAp gels were dried up in air for 96 h. Paraffin (10 wt.%) was added to the dry mass obtained. The mixed powder blends were pressed into specimens under 85 MPa and then heat-treated at different temperatures for 1 h (table 2). The temperatures were raised up to the temperature of synthesis. The specimens had the form of a parallelepiped 14.0±0.5 mm long, 4.0±0.05 mm wide, and 4.0±0.05 mm thick.

The compression tests were carried out with an Instron-4301 testing machine (GB). The tests were continued up to failure of the specimens. The results of mechanical tests of these biomaterials in uniaxial compression are presented in table 2.

Table 2. Mechanical characteristics of composite materials based on SG and hydroxyapatite

Material, wt. %	σ_1^*, MPa	E, MPa	ρ, g/cm^3	Manufacturing temperature of materials, °C
SG–100	197.6±78.3	7501±1944	2.081	800
Hap–100	24.37±3.07	5053±1565	2.017	1100
NHAp–100	41.94±14.85	4461±510	2.043	1100
SG(80)–HAp(20)	42.05±10.48	3282±701	1.146	800
SG(60)–HAp(40)	263.7±73.3	6929±987	2.110	800
SG(40)–HAp(60)	95.49±29.9	4516±429	1.830	800
SG(80)–NHAp(20)	35.98±12.79	3321±755	1.199	750
SG(60)–NHAp(40)	179.6±92.8	5967±1150	2.043	850
SG(40)–NHAp(60)	97.57±7.91	4059±437	1.680	850

During the experiment it was found that the mechanical properties greatly depended on the material density, which, in turn, was determined by the temperature at which they were manufactured. Taking into account the change in the density of the composite materials because of the porosity appearing during in their production, the elastic modulus of the materials can be calculated theoretically from the formula

$$E_{c.m} = (E_m + (E_f - E_m)V_f) \times k_\rho , \tag{1}$$

where E_m and E_f are the elastic moduli of the matrix and filler; V_f is the volume content of filler; k_ρ is the coefficient determining the relation between the true ρ and theoretical ρ_t density of the material:

$$k_\rho = \frac{\rho}{\rho_t}, \quad \text{where} \quad \rho_t = \frac{\rho_m V_m + \rho_f V_f}{V_m + V_f}.$$

Here ρ_m and ρ_f are the densities of the matrix and filler, and V_m is the volume content of the matrix. The test results and the values calculated from formula (1) are given in table 3. From the data in this table it is seen that the difference between the experimental and calculated values of elastic modulus is about 10% on an average.

Table 3. Comparison between the experimental and calculated by equation (1) elastic moduli (MPa) of composite materials based on SG and hydroxyapatite

Material, wt %	Experiment	Calculation by eq. (1)	Difference, %
SG–100	7501±1944	7501	0
Hap–100	5053±1565	5053	0
NHAp–100	4461±510	4461	0
SG(80)–HAp(20)	3282±701	3877	15.35
SG(60)–HAp(40)	6929±987	6677	3.64
SG(40)–HAp(60)	4516±429	5388	16.18
SG(80)–NHAp(20)	3321±755	3979	16.54
SG(60)–NHAp(40)	5967±1150	6202	3.79
SG(40)–NHAp(60)	4059±437	4621	12.16

The biocompatibility and bond strength between the artificial biomaterials and a live bone tissue were tested on adult male rabbits, whose weight ranged from 3.0 to 3.7 kg. Holes, 3.1 mm in diameter, were made in the rabbit femur by a surgical drill for implanting the following biocomposites: SG(60)–HAp(40), SG(60)–NHAp(40), UHMPE(70)–NHAp(30), and UHMPE(50)–NHAp(50). The specimens were cylinders, 3 mm in diameter and 3 mm in height. The biocomposites were implanted in 48 rabbits. The experiment was completed by giving the rabbits a lethal doze of pentobarbital solution in 2, 4, 10, and 25 weeks. The implants together with the surrounding bone tissue were cut out in blocks from the femur and prepared for experiments on punching out the implant from the bone tissue. Before the experiment, the specimens were kept in the saline at room temperature. Each experiment was carried out within 6 h after extraction of the material from the live tissue of a rabbit. The bond strength between the bone tissue and the implant was determined at a punching rate of 0.5 mm/min.

The stress–strain state of the bone–implant system was determined by the finite element program ANSYS. The specimen of artificial material (implant) during the experiments on punching out from a bone tissue had a cylindrical shape. Therefore to determine the stress–strain state of reconstruction system of bone–implant, ¾ of cylinder and bone tissue were removed. This problem was solved by the use of the diagram shown in figure 3.

Deciding this theoretical problem, the values of characteristics of mechanical properties such as elastic modulus, Poisson's ratio were taken from literature [6], [8], [9] and tests. For cortex bone tissue an elastic modulus was taken from the range of 7–20 GPa, and Poisson's ratio (μ) was taken from the range of 0.3–0.4. In the case of

implantation, material elastic modulus was taken 0.385 GPa and 6.0 GPa, and Poisson's ratio was taken 0.35.

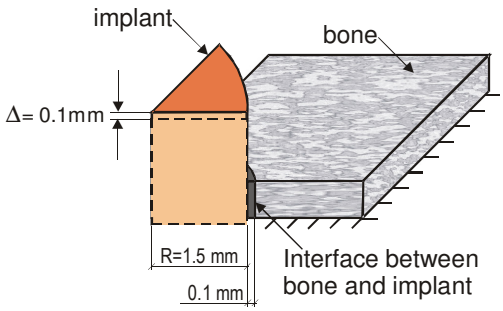

Fig. 3. Schematic illustration of bone tissue, implant and interface between them in FEM

The callus was located between bone tissue and implant material in FEM model. It was as interface between bone tissue and implant material. The values of elastic modulus and Poisson's ratio for callus were taken from the range of 0.000005–7 GPa, and from 0.25 to 0.4, respectively. Displacement value was taken 0.1 mm. This problem was decided few times with using different values of elastic modulus, Poisson's ratio and displacements.

3. Results

All the specimens implanted were punched out from the bone tissue in 2, 4, 10, and 25 weeks after implantation. We should note that some cracks occurred on the interface between the bone and implants. Table 4 presents the results of the bond strength between the biocomposites and bone tissue.

Table 4. Bond strength between the implant materials and bone tissue

Time after implantation (weeks)	Bond strength, kPa			
	SG(60)–HAp(40)	SG(60)–NHAp(40)	UHMPE(50)–NHAp(50)	UHMPE(70)–NHAp(30)
2	5.4±2.6	6.3±1.7	3.1±2.0	0
4	527.0±97.3	571.6±109.3	280.6±120.8	0
10	1211.0±183.6	981.7±217.6	760.0±277.2	70.2±19.0
25	1496.0±147.9	1342.8±203.3	973.7±321.3	189.9±70.7

The data in table 4 show that the bond strength between the UHMPE(50)–NHAp(50), SG(60)–HAp(40), and SG(60)–NHAp(40) composite biomaterials and the surface of

the cortical bone increases considerably in four weeks after the implantation, and reaches almost its maximum in 10 weeks.

The SG(60)–HAp(40), SG(60)–NHAp(40), and UHMPE(50)–NHAp(50) composites, having sufficiently good mechanical properties and a good bond strength with the cortical bone tissue, can be recommended as implant materials for covering bone defects in order to create a new bone structure after orthopaedic interventions.

The new method for removing protein from bone tissues by heat treatment at a temperature below 400 °C allows one to perfectly preserve the mineral structure of bones with the purpose of its further use as a filler for biocomposite materials.

A comparison between the composites based on UHMPE and NHAp has shown that a change in the matrix to filler ratio changes the mechanical characteristics of these materials. A greater percentage of filler increases the elastic modulus and decreases both the breaking strength and the strain at break.

As can be seen from the data in table 2, the mechanical characteristics of composite materials based on SG–HAp and SG–NHAp, such as the breaking stress in compression and the elastic modulus, depend on the density of the materials.

The results obtained show that the bond strength of composite biomaterials based on SG–HAp and SG–NHAp with bone tissue is greater than that of the materials based on UHMPE–NHAp. When an implant is applied to the bone surface, the bond strength between the bone tissue and the implant is one of the most important factors indicative of the reliability of fixing the implant to the bone tissue.

Figure 4 shows theoretical results of stress–strain state of the reconstruction system of bone–implant by using $E_{\text{bone}} = 14$ GPa, $\mu_{\text{bone}} = 0.3$, $E_{\text{implant}} = 6$ GPa, $\mu_{\text{implant}} = 0.35$, $E_{\text{callus}} = 5$ GPa, $\mu_{\text{callus}} = 0.49$.

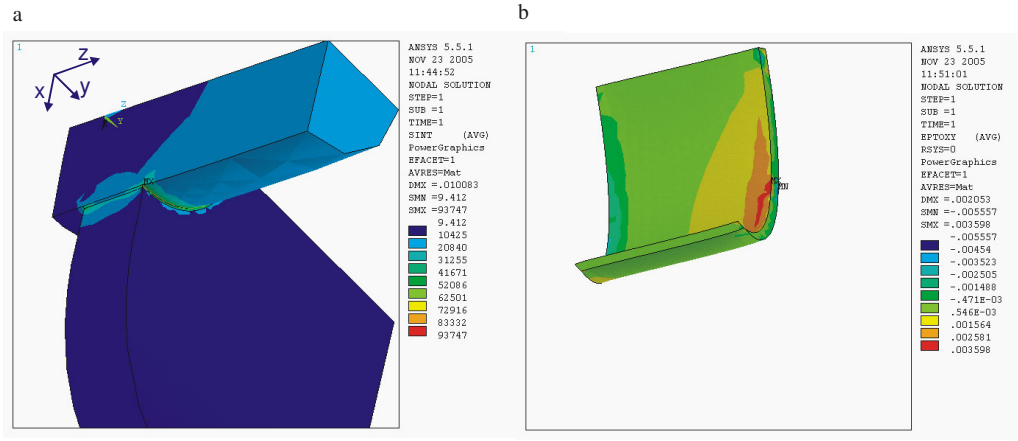

Fig. 4. The stress–strain of the bone–implant system:
total stress (a); average strain in *XY* plane (b)

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