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THERMAL MANIFESTATIONS AND NANOINDENTATION OF BONE CEMENTS FOR ORTHOPAEDIC SURGERY

by

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Improving of bone cements properties is possible by research of variables influencing exothermal behaviour and mechanical properties. Paper deals with exothermal behaviour experimental evaluation of bone cements used for medical purposes. Specimens were prepared by a conventional manual mixing technique. The work addresses primary risk factor associated with application of bone cement to femoral canal. Different size samples of bone cement has been created with diameter d = 2; 5;12,5 mm fixed in dentacryl. As an experimental material, Palacos R+G high viscosity, radiopaque bone cement containing Gentamicin and Radiopaque bone cement Antibiotic Simplex with Tobramycin, was used. Thermal effect during exothermic polymerisation was measured with period 1 minute. Evaluated factors were mass and thickness of bone cement. Significant influence of bone cement mass on temperature has been found.

Key words: *bone cement, exothermic behaviour, nanoindentation, porosity, osteonecrosis*

Introduction

In the course of the last decades orthopaedic surgery has been developing rapidly. A significant qualitative change is being noted with the development of technical and biological sciences, [1-8]. New knowledge in the sphere of basic and applied research have formed preconditions and substantial possibilities in the case of conventional orthopaedic surgical operations, such as correction of uneven length and deformations of limbs, osteosynthesis, and particularly in possibilities of replacements of worn-out degenerative joints either on the developing, inflammatory, traumatogenic, metabolic grounds or in the postoperative states. Number of patients suffering from degenerative joint diseases (osteoarthritis) has been continually increasing and due to its seriousness, therapeutic complications and frequent patient/client (p/c) individualization, the disease represents inherent and significant medical, economic and social issue. The successful replacement of the destroyed joint means improvement of mobility, pain relief and returning back to accomplishing every day activities for a

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p/c. Many patients at working age re-enter into an employment relationship after successful surgery related to the total endoprosthesis. Achievement of implantation and reimplantation of the total hip joint replacement and long-term longevity of endoprosthesis depend on lot of factors involving, for instance, correct surgery indication, patient preparation, properly performed surgical operation. A surgical operation rests chiefly in selection and effective technique of the bone cutting which influence both quality and biological potential of the generated surface. Utilization of up-to-date techniques may cause frequent revision of surgical operations. An aseptic loosening of endoprosthesis represents the most common reason of subsequent implantation failure. A long-term success of the implanted endoprosthesis does not lie only in the used surgical techniques yet in the method of cutting and machining of the bones, their properties and technological procedures and used bone cement, [2, 9-12]. Currently on the market there is more than 60 types of such products [1]. A big disadvantage to these bone cements is that it heats up to quite a high temperature, [9-11, 13-17], while setting, potentially 82.5 °C, and because of this, thermal necrosis of neighbouring tissue might occur. A careful balance of initiators and monomers [10] is needed to reduce the rate of polymerisation, and thus the heat generated. Other problem is that bone cement is subjected to high mechanical stress in the body, [1, 18-19]. Therefore this must be continuously tested. This article presents review study on mechanical properties obtained by nano-indentation and exothermic behaviour. Results are important for rationalisation of surgical process and subsequently for nursing care after surgery. It is hypothesised bone cement selection has impact on vital capacity of bone surface in femoral channel e.g. osteonecrosis.

State of the art analysis and problematic issues

Application of bone cement during total hip (re)implantation of cemented femoral stems significantly affects the quality of surgical process in term long term survival. Bone cement creates the interface between bone and femoral stem, [15, 18, 20-21]. This interface is determined by type of bone cement and its physical and mechanical properties. Many authors have tested the mechanical properties and exothermic behaviour of bone cements and various factors that affect both important physical properties. However, the mechanical properties at the bone/bone cement interface under load bearing are not fully understood.

Many authors have examined the mechanical properties of bone cement and the various factors that affect its mechanical behaviour, [1-2, 12, 19-22] Great deal in that field has been done by Kuhn [1] who present in his study complete, detailed and comparative reference on all important physical and chemical properties of all commercially available bone cements. Sańczyk and Rietbergen reported in their study problems with the use of PMMA cement and osteonecrosis of surrounding bone due the high heat generation during polymerization and chemical necrosis due to unreacted monomer release, [14]. They found that the bone tissue, subjected to the highest temperatures, is also subjected to high leftover monomer concentration. Four-point bending static and fatigue test were carried by Ishihara et al., 2002 on bone-cement bonds. They use different bone cements, CMW and ZIMMER, were performed to compare the fatigue strengths for the both joints, [19]. Amirfeyz and Bannister investigated the relationship of bone porosity and bone-cement interface shear strength [22]. They found that greater porosity yielded stronger bone–cement interface shear strength under the identical cementation technique. The optimum porosity of cancellous bone is more than 90% which can be found by reaming or drilling to deeper bone in cemented acetabular fixation. Many authors have tried to understand the complex phenomena involving the cement flow and heat transfer during the hip

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replacement surgery using mathematical modelling. Authors [9, 23-24] proposed a mathematical model of the heat flow in surgical cement during implantation. Their model describes the temperature distribution in the bone during the surgery treatment. Kaorapapong et al. developed a mathematical model of heat transfer in the cement hip replacement to study heat transfer by conduction in the femur bone, [15]. They found that the initial temperature has significant effect on the heat transfer from the cement in the femoral canal to the surrounding bone. They also noted that implant material, used in model, has less effect on the heat transfer process. Many bioactive bone cements were developed for total hip replacement and found to bond with bone directly. However, the mechanical properties at the bone/bone cement interface under load bearing are still not fully understood [20]. Crucial factors affecting exothermic behaviour is thickness, mass, composition of bone cements.

Experimental set up

In these studies following commercial bone cements were reviewed: Palacos R+G® (ZIMMER) which has a broad spectrum covering gram-positive and gram-negative bacteria. As a pre-mixed formulation, Palacos R+G provide a homogenous cement/antibiotic mixture and saves valuable OR time and Antibiotic Simplex with Tobramycin®STRYKER, Howmedica Osteonics, Ireland). Bone cements were prepared by manual mixing in strict compliance with manufacturer's instructions. Samples are presented in Fig. 1. As a matrix material has been used polymethylmethacrylate technical dentacryl CH_2-$COOCH_3$-CCH_2 with drilled holes 3 mm. Diameters of holes are 2; 5 and 12,5 mm. During application of bone cements to dentacryl retainer temperature was measured. Temperature was measured by infrared thermometer UNI-T UT305C with single point laser with output < 1mV, wavelength 630 – 670 nm with period 1 minute. Total measurement time of temperature of samples was 40 minutes.
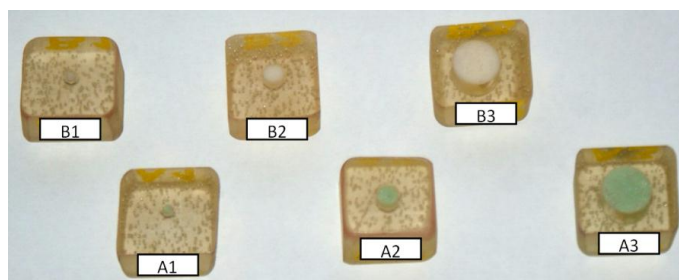

Fig. 1. Samples of bone cements in technical dentacryl after polishing

Mechanical properties determination

The samples sections for nanoindentation were metallographically polished down to roughness lower than 0.1 μm. All nanoindentation measurements were performed by nano-indentation tester TTX-NHT (by CSM Instruments, Switzerland). Berkovich pyramid diamond tip was used in simple monotonic mode with maximum loading of 10 mN. Loading and unloading rate was 20 mN/min., the hold time at the maximum was 10 s. The resulting load-penetration depth (*P-h*) curves were evaluated according to the analysis of Oliver and Pharr [25]. From the applied load and corresponding contact area, hardness values can be calculated very precisely. Besides that, the completely elastic character of the unloading part of a *P-h* curve enables to calculate also the modulus of elasticity for each indent according to formula Fischer-Cripps [26,27].

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$$E^* = \beta /\sqrt{A(hc)} \, dP/dh \mid_{h=hmax} \tag{1}$$

where β is the indenter shape factor, $A(h_c)$ is the contact area corresponding to the contact depth of penetration h_c, P is the applied load. E^* is the composite sample/indenter elasticity modulus, from which E for the sample is then determined. Up to 20 indentations were performed, values of hardness (*H*) and modulus of elasticity (*E*) were calculated and the obtained data were statistically evaluated. Mean values and standard deviation for both *H* and *E* are shown in Table 1.

Table 1 Young´s modulus and hardness of bone cements

Mechanical properties	Values	A1 Palacos R+G	A2 Palacos R+G	A3 Palacos R+G	B1 Antibiotic Simplex+T	B2 Antibiotic Simplex+T	B3 Antibiotic Simplex+T
HIT (O&P) [MPa]	**Mean**	340,594	278,029	322,720	321,395	282,938	366,495
	Std Dev	25,159	32,81	10,415	44,031	30,513	8,153
HV (O&P) [Vickers]	**Mean**	31,543	25,748	29,887	29,765	26,203	33,941
	Std Dev	2,330	3,039	0,965	4,078	2,826	0,755
EIT (O&P) [GPa]	**Mean**	4,534	4,352	4,948	4,614	4,475	5,099
	Std Dev	0,078	0,134	0,279	0,175	0,227	0,095

The resulting imprints were subsequently visualized using a confocal microscope Sensofar PLu Neox. Their morphology was measured and related to the results obtained from the nanoindenter. Significant differences in modulus of elasticity and hardness were derived by one-way ANOVA (Table 1).

Results and discussion

Graphs in Fig. 2 show exothermic peak of each reviewed bone cements. It is apparent the influence of bone cements mass on temperature. The temperature behaviour during exothermic reaction of bone cement is different. The high values of temperatures were found during measuring samples created from Palacos R+G. In case of sample with diameter 12,5 mm and width 3-4 mm (Sample A) fixed in dentacryl was found that temperature over-reach critical temperatures 47°C, [28], and 55°C when osteonecrosis appear, [23]. Maximal measured temperature was 64°C. When we compare thermal behaviour of Palacos R+G (fig. 2a) and Antibiotic Simplex with Tobramycin (fig. 3b), it has been found that temperature values are under critical values in the case of bone cement (Fig. 2b). Following figures and table show results after nano-indentation of samples. Fig. 3 shows examples of arrays of typical indents produced by the Berkovich pyramid. Differences in size (Fig. 4), corresponding to the measured hardness values is clearly visible.

The concave shape of the indent boundaries suggest that a large portion of the deformation is elastic and that it is after load removal to a large extent recovered which is in agreement with the shape of *P-h* curves (Fig. 5). Nano-hardness measurements have shown relatively small differences between the two used materials (with different antibiotic). No clear tendency caused by the type of antibiotic was found, the differences fall within typical errors of measurement. However, there were larger differences found between various states of the same material, as it is shown in Figures 6 and 7. In both types of bone cement the lowest values were found for the second states (prepared with d = 5mm). In all cases, the scatter of data, as suggested by the error bars, was relatively large due to local inhomogeities of microstructure.

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Microstructure evaluation

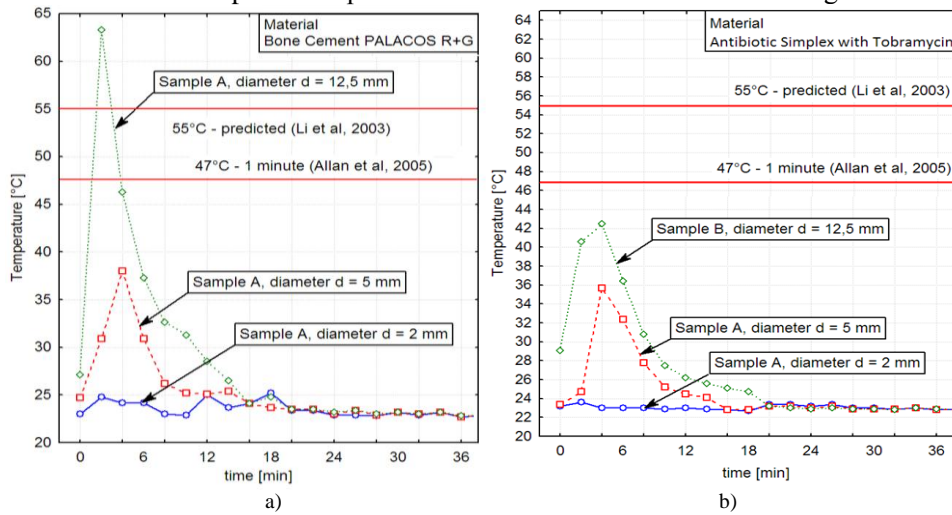Volume fraction of pores and pore sizes in terms of Feret's diameter are given in Table 2.

Fig. 2. Influence of material size on temperature during curing time, material Bone cement, a) Palacos R+G and b) Antibiotic Simplex with Tobramycin
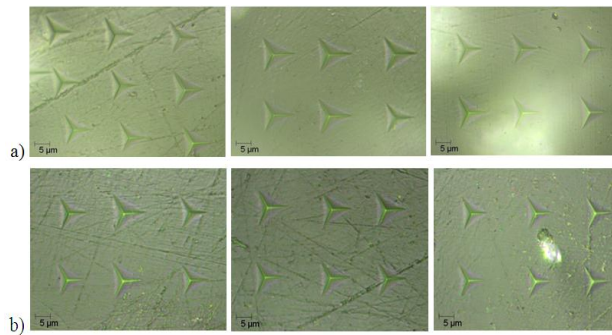

Fig 3. Typical arrays of indents made by Berkovich pyramid in individual samples a) Palacos R+G, b) Antibiotic Simplex with Tobramycin
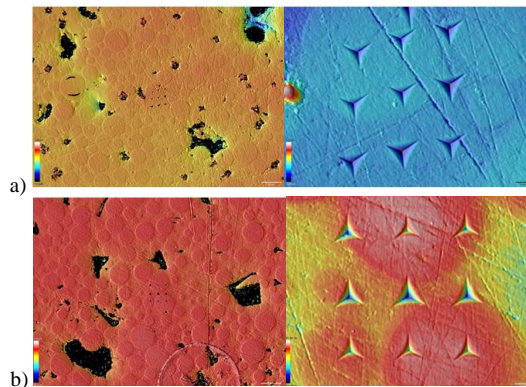

Fig. 4. Surface of indents in bone cement obtained using confocal microscopy, a) Palacos R+G, b) Antibiotic Simplex with Tobramycin

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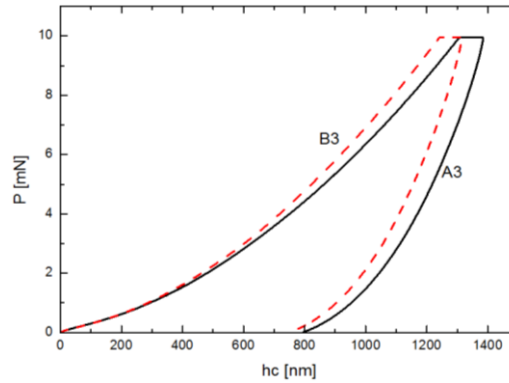THERMAL SCIENCE: Year 2014, Vol. 18, Suppl. 1, pp. S251-S258

Fig. 5. Comparison of typical indentation loading/unloading P-h curves for two (A3, B3) samples.
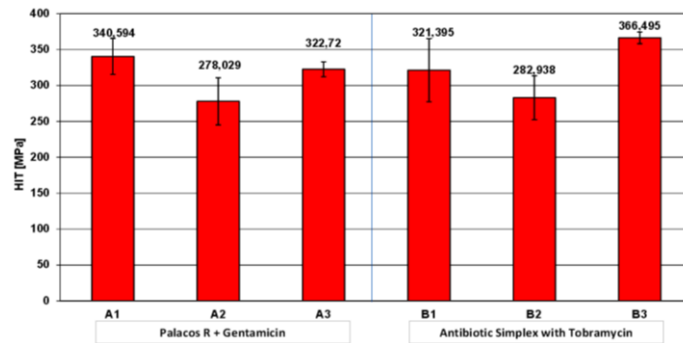

Fig.6. Comparison of Nano-hardness of surgical bone cements employed in the experiment

Table 2. Results of image analysis of micrographs of sample surfaces.

Sample	Number of counted pores	Volume fraction of pores, %	Feret's diameter, μm	Average pore cross section area, μm^2
A1	533	8.6	22	353
A2	855	9.4	24	532
A3	1311	10.9	26	767
B1	482	9.1	23	473
B2	646	10.4	26	667
B3	1063	11.9	32	1038
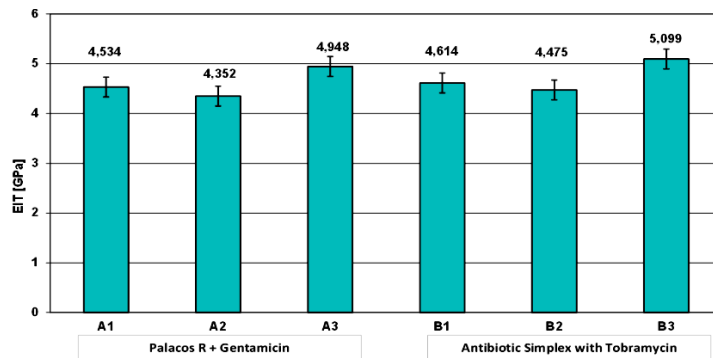
Fig. 7. Comparison of Young Modulus of Elasticity of surgical bone cements used in the experiment

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The samples were prepared by standard metallographic way with emphasis on preserving fine soft microstructure. It was polished by a series of sand papers and finished by diamond spray with grit size of 0.25 μm. The prepared samples were observed and photographed using an inverted metallographical microscope Olympus GX71 in reflected light. The obtained photographs were treated by image analysis. Software package ImageJ 1.46r was used to evaluate the porosity of the material. In each case at least 450 pores from at least 1 mm^2 area were taken into account. In both types of bone cements it was seen increasing average pore size and pore volume fraction with increasing size of the sample. More importantly it can be also seen that the sample B tended to form more and slightly larger pores than the sample A. This fact, however, did not lead to lower values of measured mechanical properties. Microphotographs (Fig.8) show porosity analysis. The increase in porosity is not significant. Because it is an artefact of the sample, the probability of existence of large pores increases with size of sample.
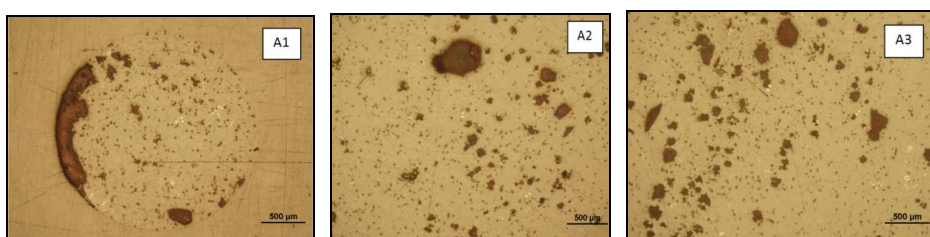

Fig. 8. Microphotographs showing evaluation of pores on samples.

Conclusions

This article presents a comprehensive survey on the reported mechanical properties of bone cement. Variables that influence mechanical properties, such as handling characteristics, strain rate, loading modes, additives, porosity, blood inclusion, in vivo environment and temperature, have also been investigated. Future research areas, important for fully characterizing the physical properties of PMMA, are also suggested. Nano-hardness of bone cements, porosity and exothermal behaviour expressed by temperature were evaluated, compared with other studies, where critical temperatures causing osteo-necrosis are estimated.

Acknowledgments

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