The study of lifetime of polymer and composite bone joint screws under cyclical loads and *in vitro* conditions

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The "strain-life time" method has been adapted for life-time prediction of polymer and composite bone joint screws. Mechanical and fatigue properties of screws made of biostable (polysulfone), biosorbable (poly(lactide-co-glycolide) and short carbon fibre reinforced polymer composite materials have been examined in this study. The lifetime predictions under *in vitro* conditions were calculated for polymer and composite implants. The forecasting of joint screws stability under conditions close to natural body environment is shown to be feasible based on equations describing lifetime of the examined joint screws.

1. Introduction

Screws used in bone surgery are the types of implants which fulfill the function of joining elements during the process of bone fixation. Unfortunately, the most commonly used metal screws do not fully meet the requirements of biofunctionality and biocompatibility. Limited biocompatibility results from the effects of metals' corrosion in the environment of body fluids. The mechanical function is usually limited on one hand by the excessive stiffness of metal part as compared to that of bone tissue, and on the other by the unsatisfactory fatigue properties. The basic limitation in use of metals, which is related to their limited biocompatibility [1], is the necessity of the follow-up surgical intrusion aimed at removal of screws after the healing period.

The described deficiencies of metal screws are the reasons for extensive research work carried out in many countries in order to develop new materials which would better meet both the biological and/or mechanical challenges. Polymer based composites are some of these materials raising interest and hopes due to their high potential for use in surgical applications. Their design gives the possibility of adjusting their mechanical properties while allowing for development of materials fully biocompatible with life body tissues. Additionally, the design of implants with time-controllable properties appears also feasible.

In the case of composite screws made of biostable (inert) matrices, these implants will be of long-lasting character. The application of bioresorbable polymer matrices leads to situations where the biomechanical function will be fulfilled for a limited time only, which will be adjusted to bone fixation process. In this latter case the development may lead to designing of new materials which would not require the removal surgery, because they will biodegrade into the substances removable from the body on the basis of natural cell metabolism process.

The advantage of application of polymer composite screws as bone surgery implants may be fully taken of only on condition of assuring the appropriate set of these composites' mechanical properties which would be stable during the bone fixation period. The required mechanical properties and their time stability depend on the area and the type of potential bone fixation process. It becomes mandatory therefore that the complete definition of biomechanical requirements must be performed, together with full analysis of choice of joint material, its mechanical properties, durability, as well as the effects of real body environment on critical parameters during the use of the implant.

Most of available literature [2–4] on mechanical properties of polymer and composite implants relates only to preliminary data obtained during static examination. This obviously does not provide complete information relative to real performance of the implant under cyclical loading and immersed in the real body's chemical environment.

This study presents the analysis of mechanical fatigue properties of polymer and composite joint screws including their lifetime prediction under *in vitro* conditions, performed with the scope of their potential application in osteosynthesis.

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TABLE I Selected properties of examined polymeric materials

	Polisulfone—Aldrich Chemical Company Inc., USA	Poli(lactid-co-glycolid) (PLA:PGA—18:82)—Centre for Polymer Chemistry PAN, Poland
Molecular mass	$M_n = 26\,000$	$M_n = 85\,000$
Temperature of vitrification	187 (°C)	27 ÷ 52 (°C)
Melting point	345 (°C)	150 ÷ 190 (°C)

TABLE II Mechanical properties of polymer and composite joint screws

Material	Failure force (N)	Tensile strength* (MPa)	Young's modulus* (GPa)
PSU	587.4 ± 47.1	72.8 ± 1.2	2.48 ± 0.16
CF/PSU MD	784.5 ± 10.1	94.2 ± 6.5	5.44 ± 0.21
PLGA	441.4 ± 31.8	43.6 ± 5.4	3.20 ± 0.12
CF/PLGA	1102.9 ± 269.5	106.4 ± 9.5	7.63 ± 0.19

*Determined on oar shaped samples, test speed 2 mm/min.

2. Materials and methods

The fatigue behaviour of joint screws was examined using joining elements made of biostable polymer—polysulfone, and of bioresorbeable material—poly(lactide-co-glycolide). Table I shows basic characteristics of the examined materials.

The geometry shown in Fig. 1 is a modification of the steel bone screw, type OSTEO MW II, of the external thread diameter $\phi = 4.5$ mm. Changes of the shape of thread had for purpose the increase of its load-bearing capacity and consisted of change of the angle of inclination as well as the pitch [5]. Both polymer and composite screws were manufactured by injection molding, and this way were obtained the non-reinforced implants as well as the implants reinforced with 15 v/o of reinforcing phase of random fibre distribution (MD) within the volume of the material. The screws made of polysulfone (PSU) and of polysulfone reinforced with short carbon fibre (CF/PSU) were made at the injection molding temperature of 345 °C, while the screws made of poli(lactide-co-glycolide) (PLGA), as well as the screws made of the same material reinforced with short carbon fibres (CF/PLGA) were obtained at the injection molding temperature of 185 °C (Figs. 2 and 3).

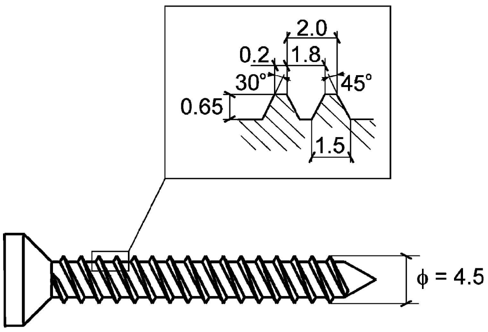

Figure 1 Geometry of joint screws.

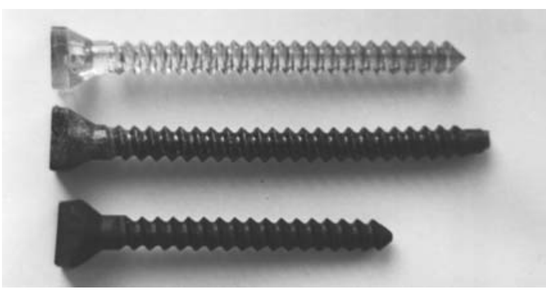

Figure 2 The PSU and CF/PSU screws.

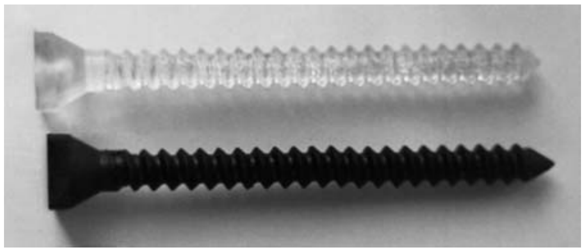

Figure 3 The PLGA and CF/PLGA screws.

The tensile strength and Young's modulus of the examined materials, and additionally the failure force of screws, were determined before the fatigue tests were performed. Table II shows the mechanical properties of screws studied. The fatigue tests of screws were performed in threaded grip under conditions of constant strain, varying the strain amplitude from zero to maximum strain $\varepsilon_{\max}$. In this case the average strain is greater than zero and is equal to $\varepsilon_{\max}/2$. The tests were performed within the subcritical strain range of each screw ($R = 0$), untill total fracture occurred, or up to million cycles (10^6). ZWICK 1435 testing machine was used and the experiments were carried out under "dry" conditions, as well as under conditions simulating the life body environment (Ringer Fluid, 37 °C). The S-N curves were analysed based on statistical distribution close to normal [6, 7].

3. Results and discussion

3.1. PSU and CF/PSU screws

Fig. 4 shows the results of experiments (in semi-logarithmic co-ordinates) for PSU screws examined under both "dry" and *in vitro* conditions. Slight reduction of the number of cycles to failure can be noticed for experiments carried under *in vitro* conditions, as compared to tests performed under "dry" conditions. This is confirmed by the calculated average numbers of cycles to failure $2N_f$, shown in Fig. 5 in semi-logarithmic co-ordinates, and forming the classical Wöhler's curve.

The lifetime prediction analysis was based on "strain-lifetime" (ε_{tot}) relationship, and the fitting was done using both power and logarithmic curves. The results and equations describing "strain-lifetime" relationships are shown in Figs. 6 and 7. For fitting with use of power curves the coefficients $R^2 = 0.8674$, and $R^2 = 0.9272$ were obtained for "dry" and for *in vitro* conditions, respectively. In the case of fitting with use of logarithmic curves, relatively higher values of R^2 coefficients were

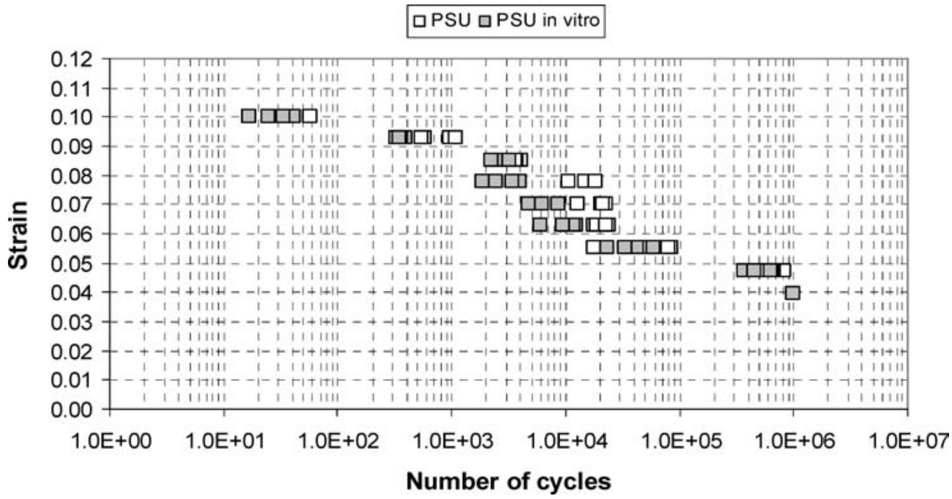

Figure 4 Measurement points obtained during fatigue tests of PSU screws under "dry" and *in vitro* conditions.

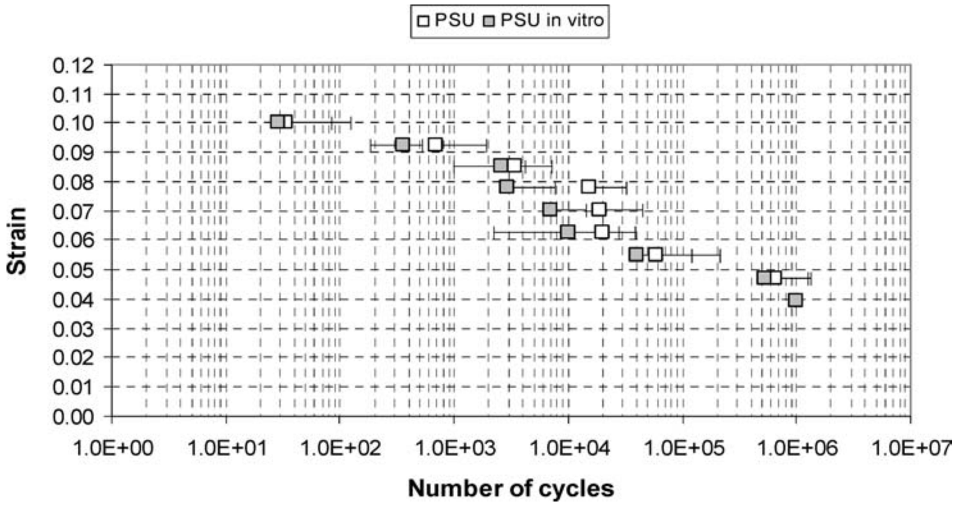

Figure 5 Strain vs. number of cycles to failure relationships determined for PSU screws on the basis of calculation of average values under "dry" and *in vitro* conditions.

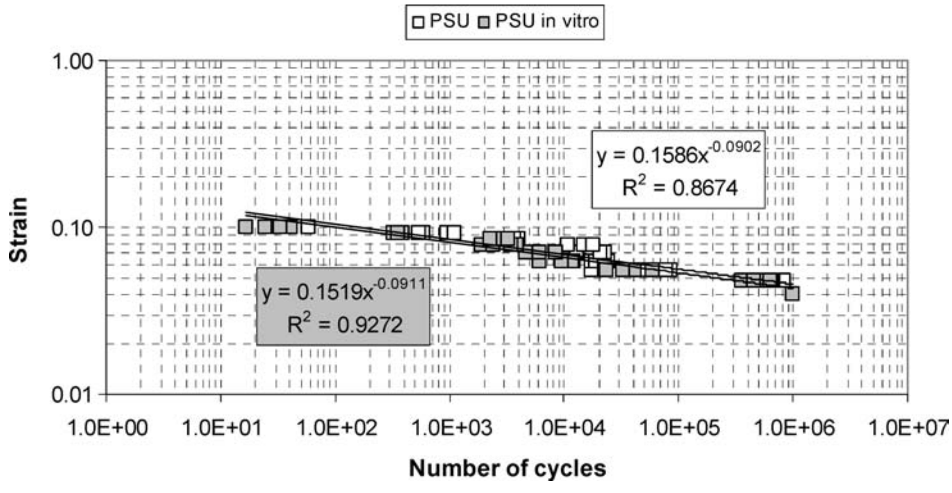

Figure 6 The "strain-lifetime" relationship (in log-log co-ordinates) for PSU screws with power fitting.

obtained: 0.9056 and 0.9385 for "dry" and for *in vitro* conditions, respectively.

The performed analysis allowed defining equations describing the lifetime of joint elements as a function of strain applied. The lifetime prediction under cyclic loading conditions was possible based on power and logarithmic fittings presented for "strain-lifetime" relationships. The comparison of lifetimes of joint screws under "dry" and *in vitro* conditions showed that logarithmic fitting is more accurate than power fitting. These observations are in agreement with the results described by Teoh [8], who used complex logarithmic relationship

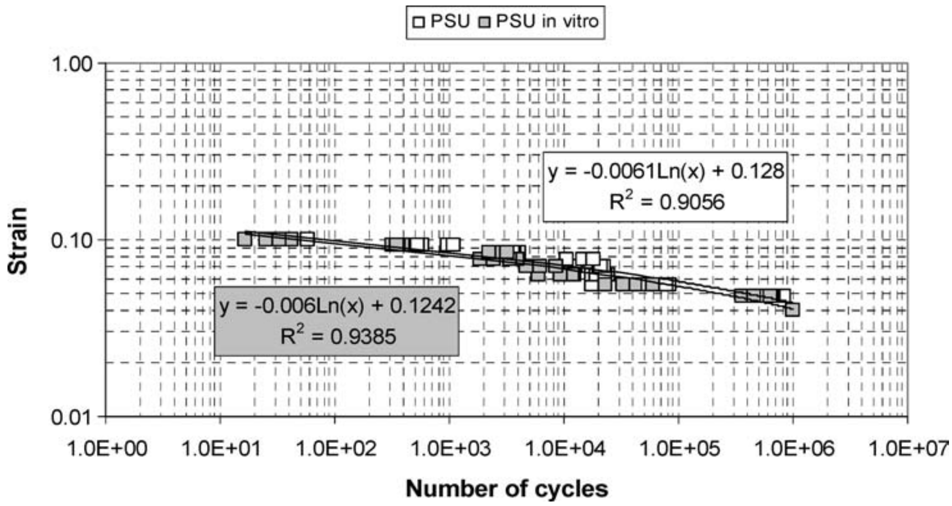

Figure 7 The "strain-lifetime" relationship (semi-log co-ordinates) for PSU screws with logarithmic fitting.

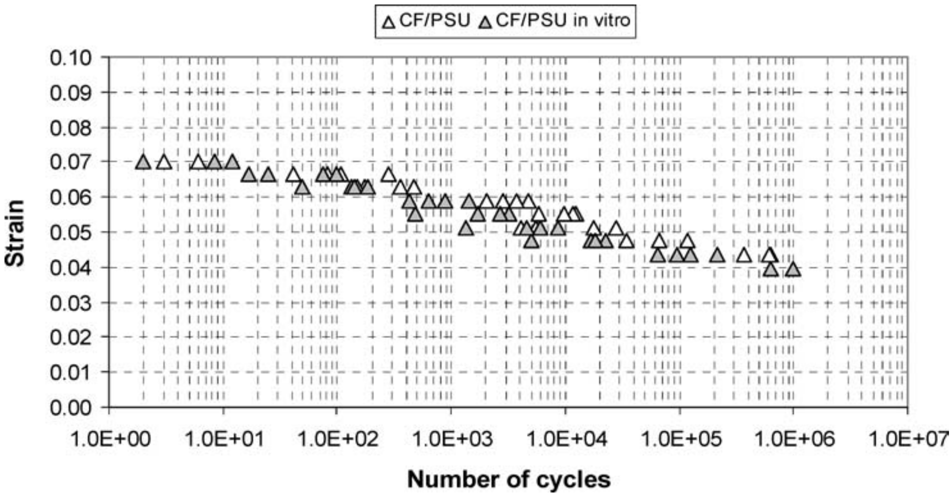

Figure 8 Measurement points obtained during fatigue tests of CF/PSU screws under "dry" and *in vitro* conditions.

for description of fatigue behaviour of polymer biomaterials. The CF/PSU screws were examined with use of identical tests as the PSU screws. Fig. 8 shows the experimental points obtained during testing of CF/PSU screws under "dry" and *in vitro* conditions, and set in semi-logarithmic co-ordinates.

Fig. 9 shows Wöhler's curves plotted in semi-logarithmic co-ordinates and determined on the basis of calculations of average values of number of cycles to failure for different strain values. There is a visible effect of *in vitro* conditions on the lifetime of composite joint screws at the given strain level. This is illustrated

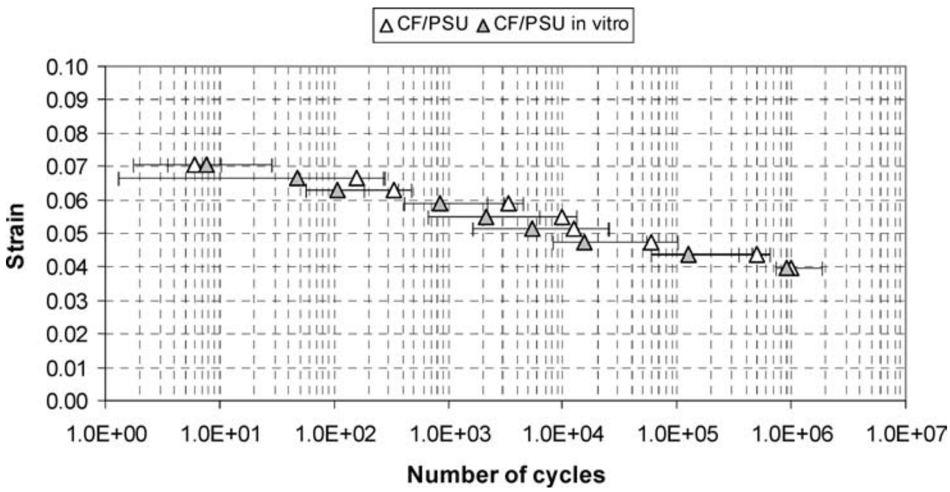

Figure 9 Strain-Number of Cycles to Failure relationship obtained for CF/PSU screws on the basis of average value calculations for "dry" and *in vitro* conditions.

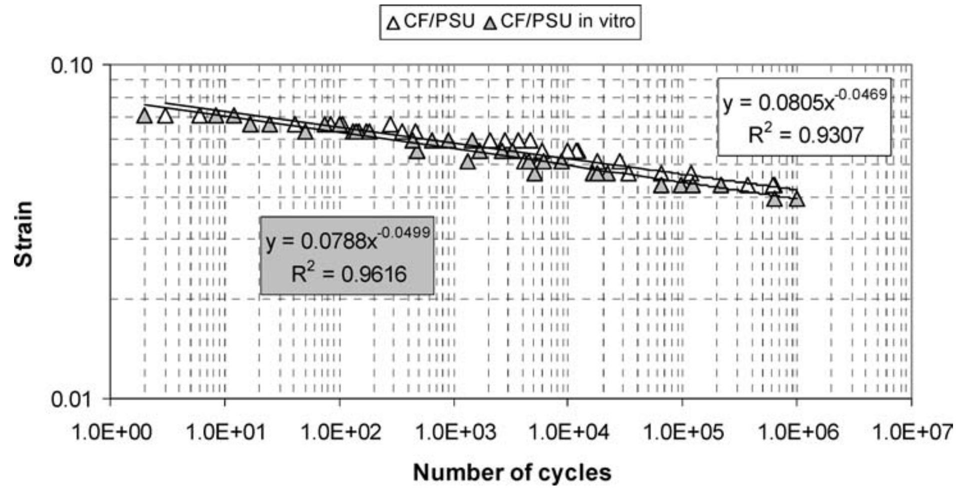

Figure 10 Strain-lifetime relationship (log-log co-ordinates) with power fitting, for CF/PSU screws.

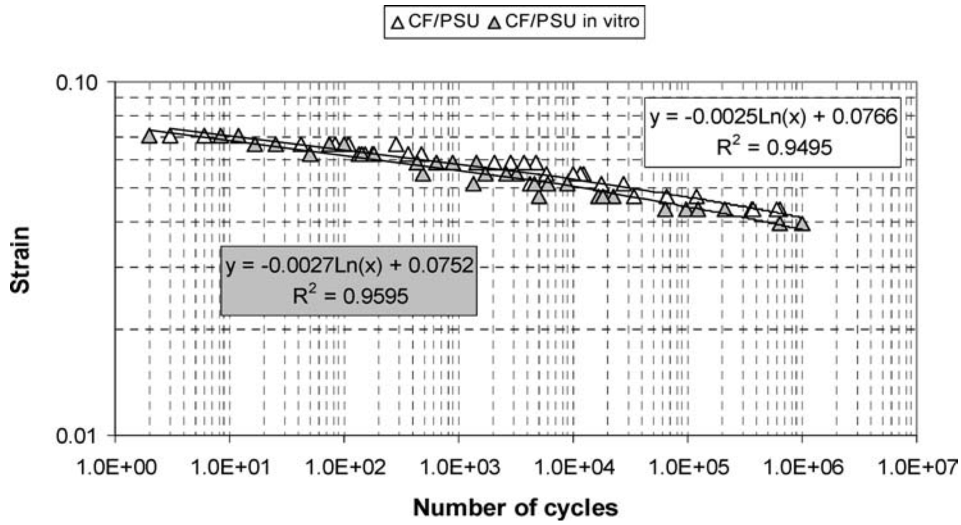

Figure 11 Strain-lifetime relationship (semi-log co-ordinates) with logarithmic fitting for CF/PSU screws.

by the decrease of the average lifetime, in particular within the high cycle range. The analysis of the lifetime was also performed by application of the "strain-lifetime" method, and the lifetime prediction was done based on total strain with power and logarithmic fitting. The relationships obtained are shown in Figs. 10 and 11. The coefficients $R^2 = 0.9307$ ("dry" conditions) and $R^2 = 0.9616$ (*in vitro*) were obtained for power fitting. The logarithmic fitting gives similar values of R^2 coefficients: 0.9495 for "dry" and 0.9595 for *in vitro* conditions. As in the case of PSU screws, the analysis performed allowed for CF/PSU material lifetime prediction under cyclic loading conditions. The comparison of lifetime of joint screws under "dry" and *in vitro* conditions (as determined on the basis of power and logarithmic equations), with the predicted values, indicates better correlation of experimental data with lifetimes predicted using logarithmic fitting. These results are in good agreement with the results obtained, among others, by Muca [9], Bonda and other researchers [10], in their works relative to fatigue behaviour and lifetime prediction of composite materials.

Figs. 12 and 13 show the fracture surfaces of screws CF/PSU after fatigue testing under "dry" and *in vitro* conditions, respectively. Well developed fracture surface proves that the process of rupture of composite screws is complex and different from purely brittle catastrophic failure. It is determined in the examined case by several phenomena occurring in the range of increased stresses. These phenomena are related to existence of reinforcing fibres within the examined

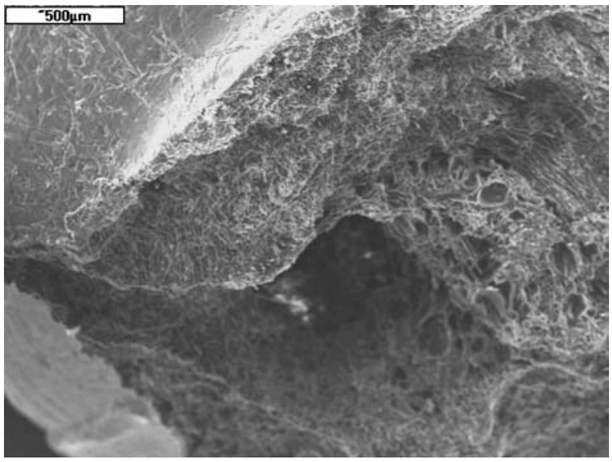

Figure 12 Fracture surface of CF/PSU screw after fatigue tests under "dry" conditions (mag. 50×).

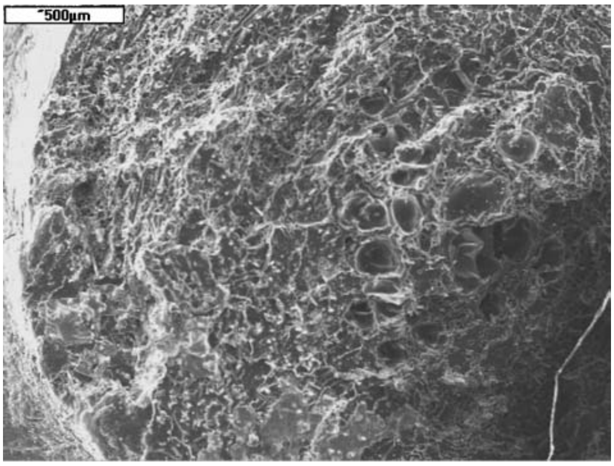

Figure 13 Fracture surface of CF/PSU screw after fatigue tests under *in vitro* conditions (mag. 50×).

material, and in particular to the properties of "fibre-matrix" interface.

Observations of fracture faces indicate significant development of their surfaces, what may be considered as a prove for deflection of crack's path as a principal feature affecting the failure. This indicates that the composite's fracture energy is much higher than that of pure polymer, seen the necessity of formation of excessive surfaces. It may be concluded that the "fibre-matrix" bond is responsible for failure of composite screws. This hypothesis is proved by lower values of composite screws' lifetime under *in vitro* conditions, as compared to screws tested under "dry" conditions.

This phenomenon may be explained by weakening of "fibre-matrix" interface due to penetration of fluids simulating the environment of a life body deep into the composite material. This element has to be taken into account during the assessment of the possibilities of use of polymer implants and composites in osteosynthesis. It may be concluded in summary that the PSU and CF/PSU screws can be applied as short-term implants exposed to relatively high loads, or long-term implants, provided they are subjected to relatively small loads.

3.2. The PLGA and CF/PLGA screws

Fig. 14 shows in semi-logarithmic co-ordinates the results of experiments on PLGA screws examined under "dry" and *in vitro* conditions. Fig. 15 presents the points forming the Wöhler's curve set in semi-logarithmic co-ordinates, determined on the basis of calculation of the

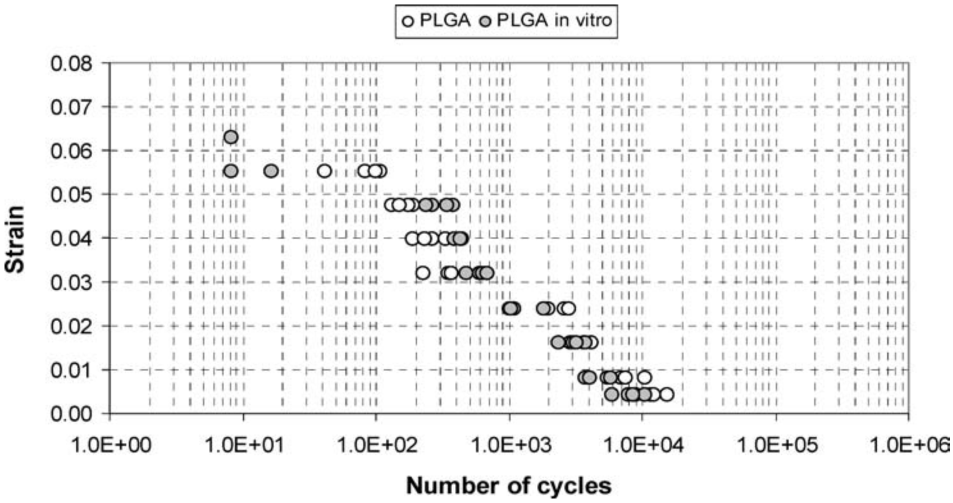

Figure 14 Measurement points obtained during fatigue tests of PLGA screws under "dry" and *in vitro* conditions.

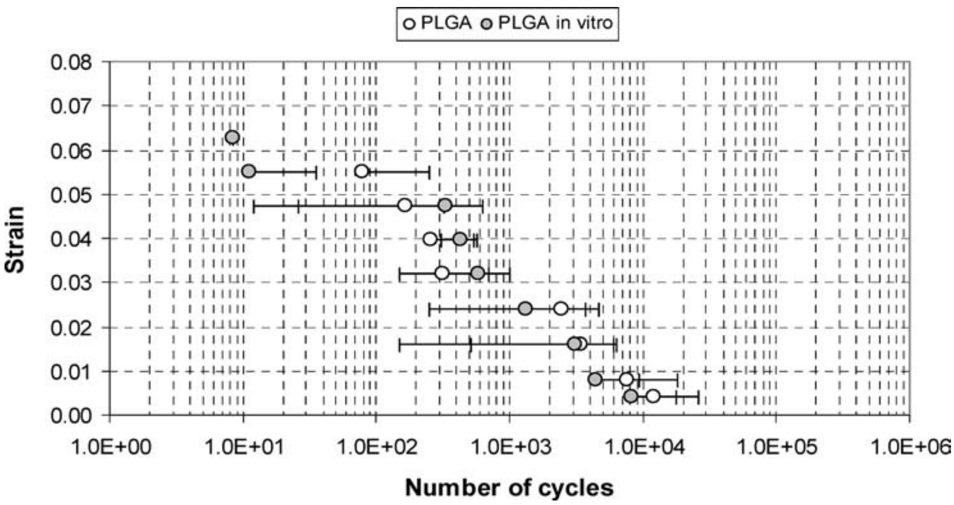

Figure 15 "Strain-Number of Cycles to Failure" relationship determined for PLGA screws on the basis of calculations of average values.

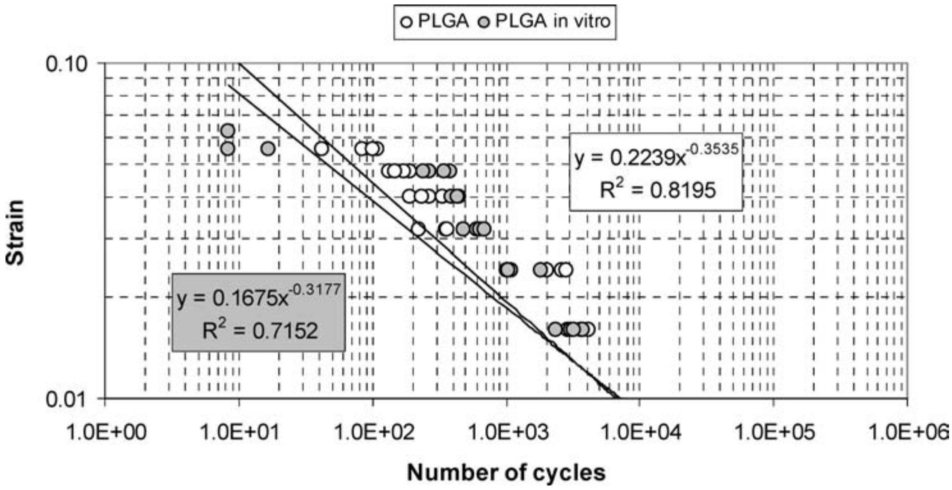

Figure 16 The "Strain-Lifetime" relationship (log-log, power fitting), for PLGA screws.

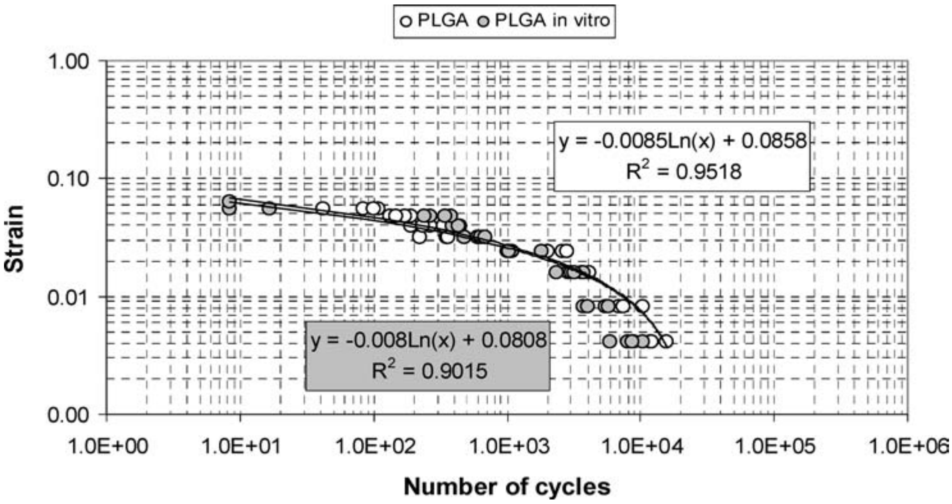

Figure 17 The "Strain-Lifetime" relationship (semi-log, logarithmic fitting), for PLGA screws.

value of average number of cycles to failure $2N_f$ for particular level of strain. Obtained results point at very limited durability of poly(lactide-co-glycolide) screws under cyclic loading conditions. The number of cycles to failure rarely exceeds 1000 cycles, which in fact is a very low value, even under limited load. There has been no significant effect of *in vitro* conditions on screw's lifetime found throughout the whole range of strains examined.

Similarly to the case of PSU screws, the lifetime predictions for PLGA screws were based on the relationship "strain-lifetime" (ε_{tot}). Both power and logarithmic curve fittings were applied, and their results are shown in Figs. 16 and 17, together with equations describing the "strain-lifetime" relationship during cyclic deformations. For power fitting, the values of R^2 coefficients equal to 0.8195, and 0.7152 were obtained for "dry" *in vitro* conditions, respectively. Low values of R^2 coefficients indicate limited power curve fitting of experimental data. Similarly to PSU screws, much better fitting was obtained using logarithmic curves. The relationships allowing for lifetime predictions of PLGA screws under cyclic loading were based on power and logarithmic curve fittings. The comparison of lifetimes determined during experiments and on the bases of equations describing fatigue behaviour indicates that lifetime predictions based on logarithmic equations are more accurate.

Fig. 18 shows experimental points ranged according to "strain-number of cycles to failure" relationship obtained during tests of CF/PLGA screws under "dry" and *in vitro* conditions. Fig. 19 shows the Wöhler's curves determined on the basis of calculations of the values of average number of cycles to failure for particular strain levels. Obtained results indicate the effect of *in vitro* conditions on lifetimes of the examined screws. This effect is more pronounced within the range of high-cycle fatigue, and is probably related to the influence of physiological fluids on the bond within "reinforcing fibre-matrix" interface. This effect is significantly stronger here than in the case of CF/PSU screws, which can be related to the biodegradable nature of polymer matrix itself.

Lifetime predictions of joint elements in form of CF/PLGA screws under conditions of cyclic loading became available based on power and logarithmic fittings (Figs. 20 and 21). In the particular case of CF/PLGA screws, both types of fittings allowing for lifetime predictions of composite elements gave similar results.

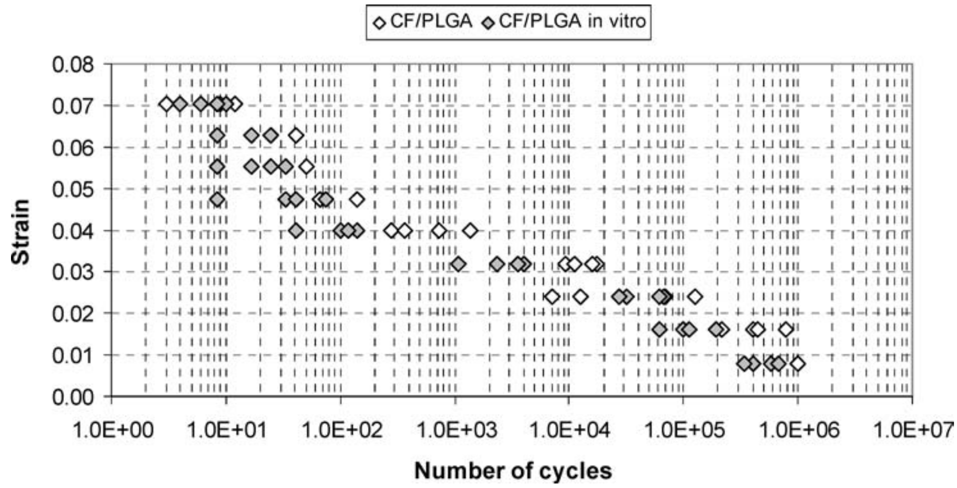

Figure 18 Measurement points obtained during fatigue tests of CF/PLGA screws under "dry" and *in vitro* conditions.

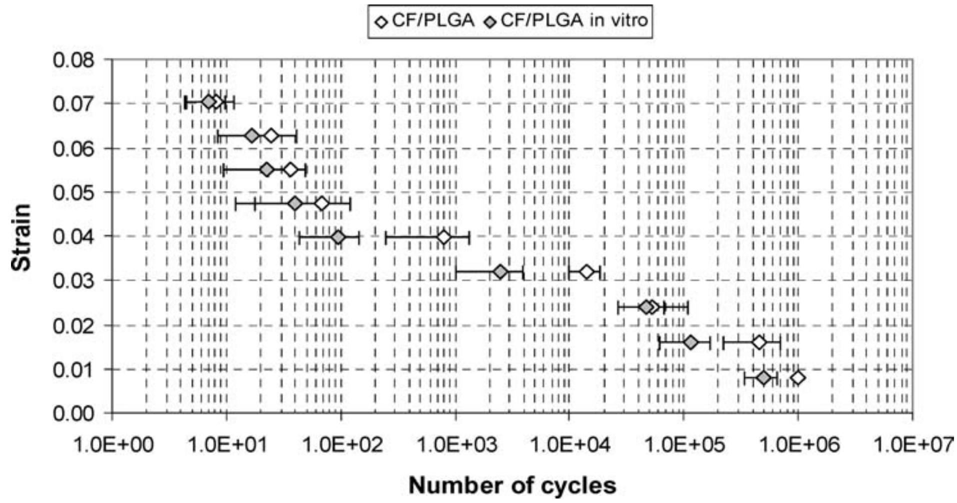

Figure 19 "Strain-Number of Cycles to Failure" relationship determined for CF/PLGA screws based on calculations of average values.

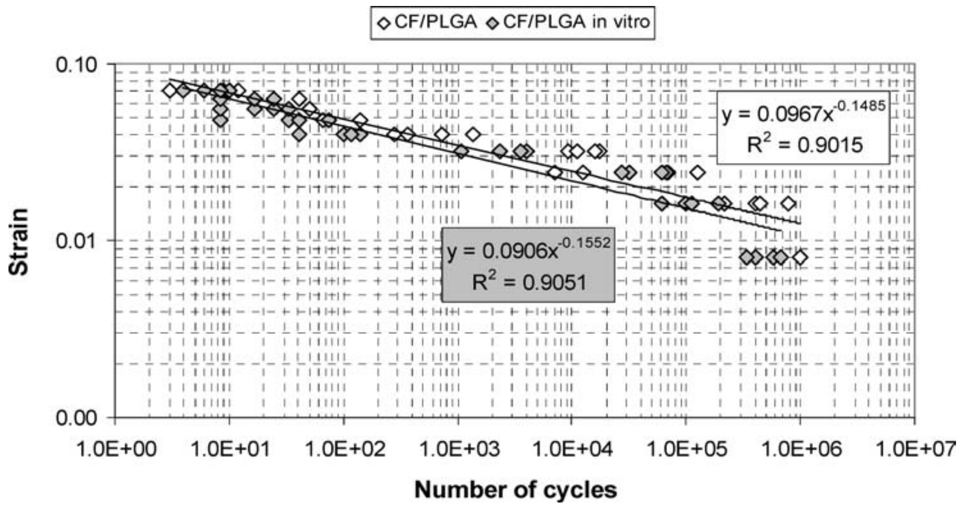

Figure 20 "Strain-Lifetime" relationship (log-log setting, power fitting) for CF/PLGA screws.

Figs. 22 and 23 show fracture surfaces of poly (lactide-co-glycolide) screws reinforced with short carbon fibres, after tests under "dry" and *in vitro* conditions. Highly complex fracture surfaces are visible, confirming that failure occurs due to loss of continuity at the fibre—matrix interface. It is particularly obvious in the case of screw shown in Fig. 23, where within the central part of fracture surface of the core, the deep hole can be seen formed due to "pull-out" of large fragment of the material of better consistency. The assessment of fatigue behaviour of PLGA and CF/PLGA screws indicates that despite promising mechanical properties determined during monotonic strength tests of screws made of poly(lactide-co-glycolide)

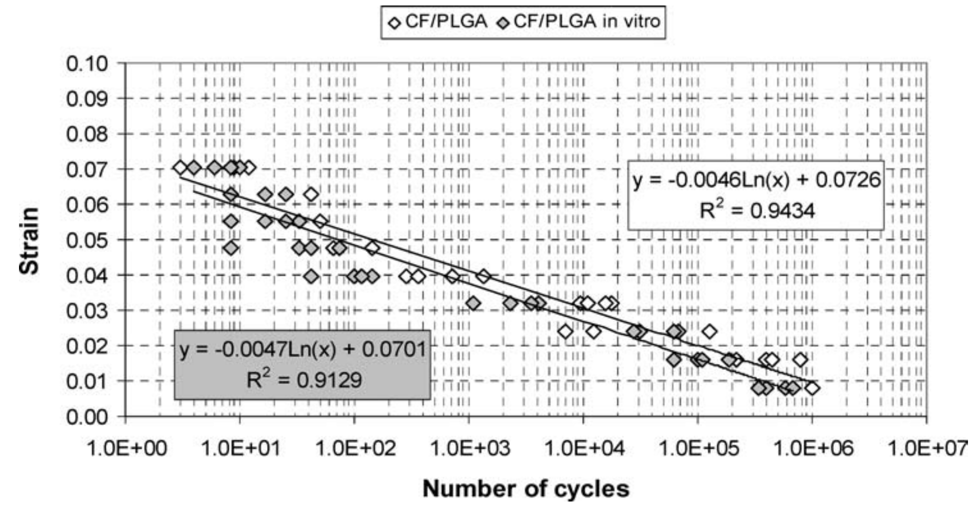

Figure 21 "Strain-Lifetime" relationship (semi-log setting, logarithmic fitting) for CF/PLGA screws.

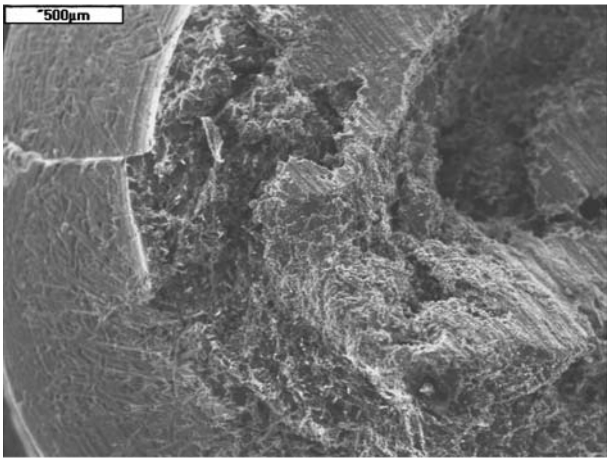

Figure 22 Fracture surface of CF/PLGA screw after "dry" fatigue tests (mag. 50×).

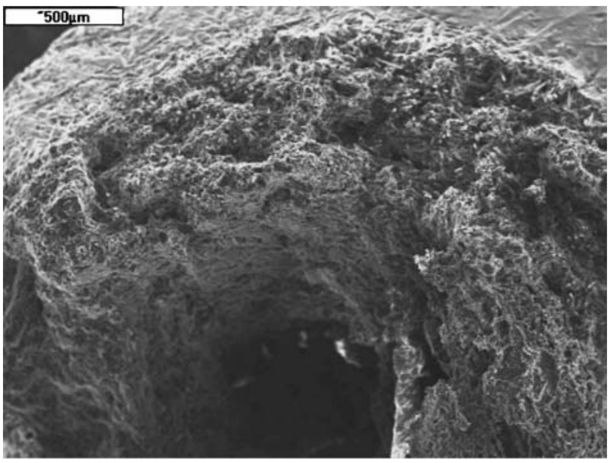

Figure 23 Fracture surface of CF/PLGA screw after *in vitro* fatigue tests (mag. 50×).

reinforced with nonresorbable carbon fibre, these materials have limited durability under cyclic loading conditions, particularly in the presence of humour fluids.

The properties of polymer matrix are responsible for this situation, showing the sensibility to the action of loads occurring in a cyclic mode. For this reason these screws can be applied in osteosynthesis as the long-term implants only under conditions of very limited strains, for instance in neutralizing osteosynthesis. However, the fact that poly(lactide-co-glycolide) is a resorbable material also has to be taken into account, meaning that it can fulfil its function only during the time limited by the time of resorption. For the cases of CF/PLGA screws subjected to the action of larger loads they may be considered as implants only for a limited period of time, which can be predicted on the basis of performed fatigue tests.

4. Conclusions

Fatigue resistance and accordingly long lifetime under cyclic loading are the most important criteria used for assessment of usefulness of screws in biomedical applications. These properties constitute so called fatigue behaviour of the material and determine the applicability of elements working during long enough periods of time under cyclic loading conditions.

For none of the examined cases the obvious limit of fatigue resistance could have been observed within the assumed period of time (10^6 cycles). This means in practical terms that there is no secure range of loads in which the examined joint materials could work. This conclusion applies only to the examined period of time and does not mean that beyond such time there is no limit of fatigue resistance.

However it may be concluded based on the obtained experimental results that screws PSU and CF/PSU have better fatigue properties. They may be recommended for screw osteosynthesis even in the areas exposed to relatively long lasting deformations. These may be for instance neutralizing joints and joints exposed to relatively low stresses of anatomic nature.

The situation is different in the case of poly(lactide-co-glycolide) screws, which show high sensitivity to the occurrence of loads of cyclic nature. For these reasons these screws can be applied only in limited number of types of osteosynthesis, where the state of cyclic loading does not occur, for instance in facial braincase.

It seems that in the cases of both PSU and PLGA screws, the properties of polymer matrix are responsible for fatigue behaviour. In composite, screws the presence of reinforcing phase in form of carbon fibre decreases the durability of the examined implants under *in vitro* conditions. This is related to the penetration of physiological fluids deep inside the material and to weakening of "fibre-matrix" bond.

Performed durability analysis containing lifetime predictions under conditions of simulated environment of a life body point at limited possibilities of fitting the experimental data with use of commonly applied micromechanical models. More accurate lifetime predictions require the formulation of much more complex model describing the fatigue behaviour of polymer and composite screws in the environment of physiological fluids.

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