

Fatigue Test and Lifetime Prediction of Implantable Biocomposite Materials

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Abstract

Biocomposite materials consisting of Poly-L-Lactic-Co-Glycolic Acid (PLGA) and Hydroxyapatite (HA) were synthesized and mechanical properties have been observed by carrying out the fatigue test, for which accelerated tests have been conducted such that 20%, 30% and 40% of the yield strength have been applied in a repeated mode until a crack occurred on the load-applied points of test specimen. Based on the fatigue test results, the lifetime of test specimen (B_{10} life) was estimated. It was found that Weibull distribution (shape parameter=8.79) was the most proper one to describe the failure data obtained from the accelerated fatigue tests. B_{10} life at 5% of yield strength was estimated to be 335,764 cycles (Confidence Level=95%, lower limit) by applying an inverse power model.

Keywords: Fatigue test; Lifetime assessment; Implantable; Biocomposite; Reliability

Introduction

Recently, implantable medical devices consisting of biodegradable polymers such as PLGA or Poly-L-Lactic Acid (PLLA) have been attracting a great deal of attention due to their ease of operation, excellent mechanical properties and, no need of subsequent surgical removal after being implanted in bodies, which is in turn reflected in a significant increase in world-wide market share [1-5]. In spite of their biodegradable properties, extra benefits such as body constituents cannot be obtained due to their intrinsic nature of chemical formula. There have been challenges to overcome this drawback as well as maximize the usefulness of the implantable medical devices. Among others, bioceramic materials such as β -Tricalciumphosphate (β -TCP) or HA have been mixed with biodegradable polymers to form biocomposites. In these types of biocomposite-based medical devices, bone formation can be more facilitated by the absorption of bioceramic components at the implanted sites during the healing period, during which the polymers are completely biodegraded to form CO_2 and H_2O . When developed, one of the most important tests that implantable medical devices must satisfy is the biological safety test. However, it is also of great importance to assess the performance durability for manufacturers, especially to assure the quality of the products after sales. One of the most widely accepted and implemented durability test is Q10 test. However, this test only gives the information related to temperature stress conditions. For manufacturers, reliability information [6] related to mechanical properties is also very important ones to consider since they need to assure the performance of their products before they were applied in the human body. Therefore, the lifetime of biocomposite materials in terms of mechanical performance was estimated by conducting accelerated fatigue tests in the present study.

Experimental

The biocomposite materials used in the present study were made by mixing PLGA and β -TCP in a 70:30 ratio. Details of the synthetic procedures can be found in the previous study [7]. The yield strength of the biocomposite materials were measured by using Universal Testing Machine (UTM) (TENSILON RTF-1325). The fatigue tests were conducted such that 20%, 30% and 40% of the yield strength were applied in a repeated mode on a test specimen. The failure of the specimen was determined to be out of the range of $\pm 10\%$ of initial value (yield strength) as recommended by the manufacturer as described in Table 1.

Sample	Load Applied (% of yield strength)	Failure Determination
Biocomposite materials	20	out of the range of $\pm 10\%$ of initial value
	30	
	40	

Table 1: Conditions of accelerated fatigue test on biocomposite materials.

For the estimation of lifetime of biocomposite materials in normal use condition (5% of yield strength), a statistical approach was adopted by using a commercial software program, ALTA[®] (ReliaSoft, USA). Weibull distribution was applied as the lifetime distribution of the biocomposite materials in the present study. Lifetime of biocomposite materials in normal use condition was predicted using the failure time data obtained from the accelerated fatigue tests by adopting an inverse power model.

Results and Discussion

The dimension of test specimens prepared for the yield strength was $100 \times 20 \times 1.5 \text{ mm}^3$ as shown in Figure 1(a). The yield strengths of the biocomposite materials were measured as shown in Figure 1(b) and the average value was 116.03 N.

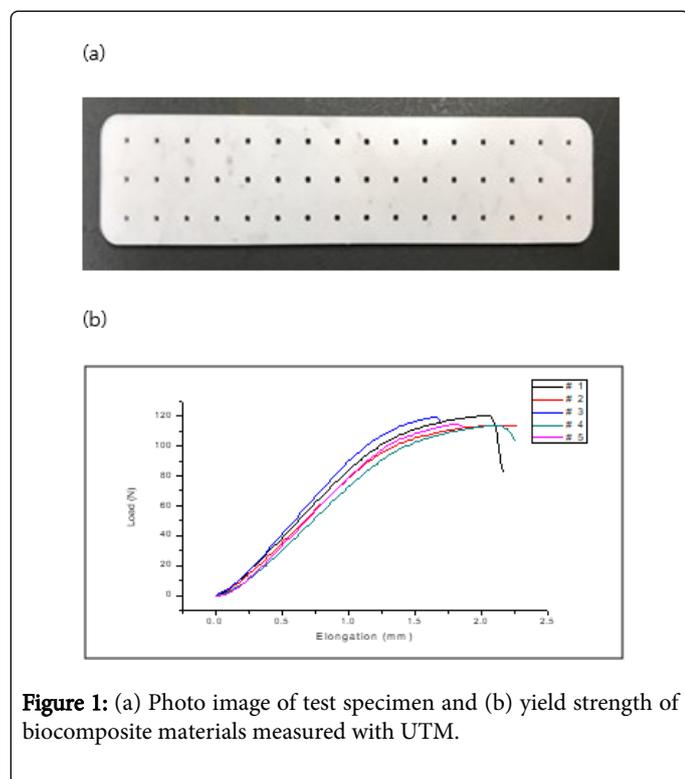


Figure 1: (a) Photo image of test specimen and (b) yield strength of biocomposite materials measured with UTM.

The fatigue test of biocomposite materials was conducted on the middle position of the test specimens in a repeated mode at the rate of 1/3 Hz (load was applied 1 time during the period of 3 seconds) using the fatigue test setup shown in Figure 2(a). The failure time was collected by setting the failure at the point in which the measurement reading was out of the range of $\pm 10\%$ of initial reading and the failure cycle numbers of the test specimens at each stress level were shown in Table 2.

Stress Level	Test Specimen	Failure Cycle
20%	#1	13,364
	#2	20,274
	#3	19,163
30%	#4	5,483
	#5	7,014
	#6	7,603
40%	#7	2,529
	#8	3,099
	#9	3,414

Table 2: Failure cycles of biocomposite materials at each stress level.

By using the failure distribution pattern estimated in Figure 2 (b-d), the probability of life (Inverse Power Law-Weibull Distribution) and the relationship between life vs. stress was plotted in Figures 3 and 4 using ALTA[®]. In Figure 3, it was confirmed that the accelerated conditions such as 20%, 30% and 40% of the yield strength were in the linear range of acceleration. The solid straight line on the right-hand side of Figure 3 indicates the probability of failure at the normal use condition (5% of the yield strength). With this regard, the common shape and scale parameters were evaluated at the normal use condition (5% of the yield strength) as shown in Table 3.

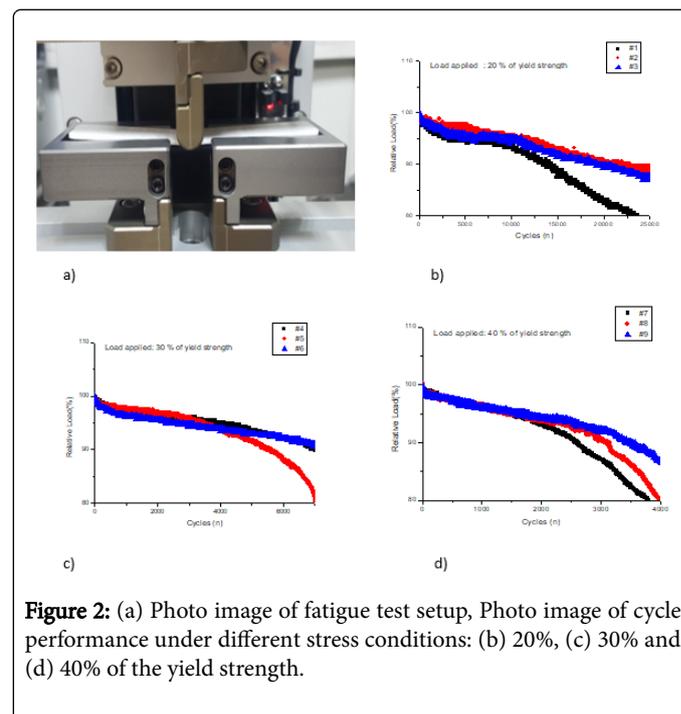


Figure 2: (a) Photo image of fatigue test setup, Photo image of cycle performance under different stress conditions: (b) 20%, (c) 30% and (d) 40% of the yield strength.

Stress level (%)		Life distribution	Acceleration model	Scale Parameter (Eta(cycles))	Shape Parameter (b)
Accelerated stress	Normal use stress				
20	5	Weibull	Inverse Power	681,575	8.79
30					
40					

Table 3: Prediction of scale and shape parameters.

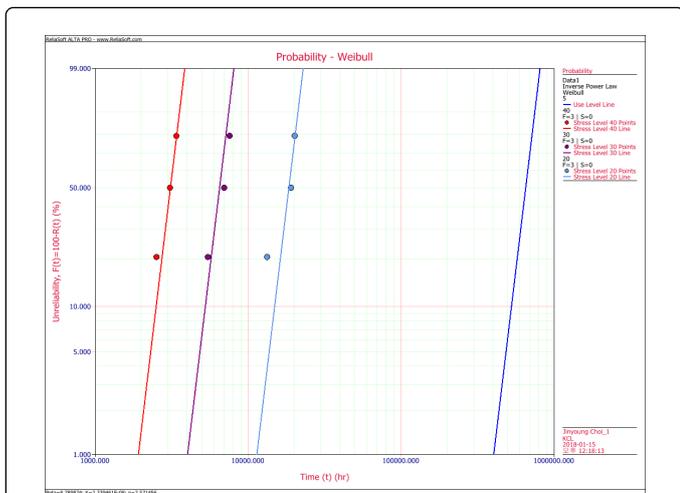


Figure 3: Probability of life (Inverse Power Law and Weibull Distribution).

The applied strength was changed from 20% to 40% of the yield strength as shown in Figure 4 and it was found that the overall behavior of failure cycle decreased as the applied strength increased. The reliability and scale parameter lines were shown in straight lines. The upper top line indicated the 90% unreliability line while the lowest line indicated the 10% unreliability line, respectively. The middle line indicated the shape parameter line. For example, the reliability began to drop at 20% approximately after 20,000 cycles, and the reliability began to drop at 30% approximately after 6,700 cycles, and the reliability began to drop at 40% approximately after 3,200 cycles. Therefore, the lifetime of the biocomposite materials was getting shorter in the yield strength conditions as the stress levels were getting more severe, which could be verified by the area of the parabola of each stress condition since the lifetime represented by the area was appearing in the shorter time as the stress levels were getting more severe. In addition, the upper limit of lifetime was also appearing in the shorter time as the stress levels were getting more severe.

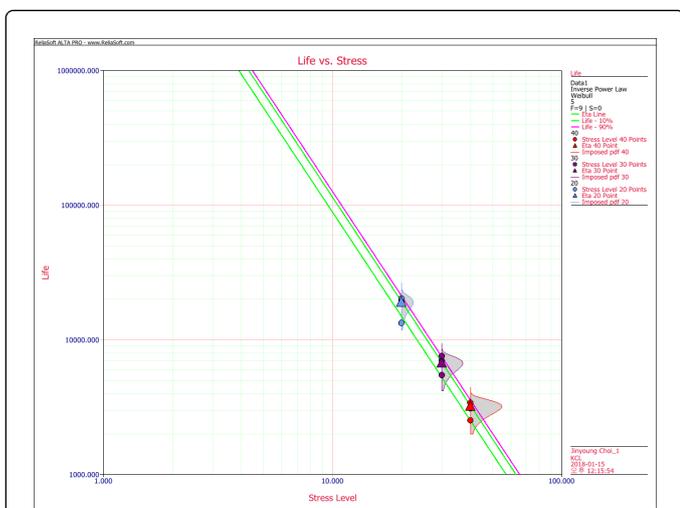


Figure 4: Life-Stress under different stress conditions.

The behavior of probability density function and failure rate with cycle was shown in Figure 5 when the Weibull distribution was applied as the lifetime distribution of the biocomposite materials in the present study. Particularly, Figure 5(a) the failure of the biocomposite materials did not occur at the initial stage of the fatigue test, but it drastically appeared at around 263,000 cycles, and was clouded at around 665,000 cycles. This was also verified by the failure rate in Figure 5(b), since there was an abrupt increase in the failure rate after 900,000 cycles, which is indicative of a typical wear-out failure. It was found that the failure rate showed and IFR (increasing failure rate) mode as shown in Figure 5(b), which indicated that the degradation of the biocomposite materials developed as the stress conditions (yield strength) were getting severe and the failure of the biocomposite materials caused by the degradation drastically were progressed within around 2,000,000 cycles.

The lifetime prediction was made by adopting an inverse power eq. model (1) for life-stress relationship.

$$L=AV^{-n} \quad (1)$$

- L: Life,
- A, n: constant,
- V: Stress factor.

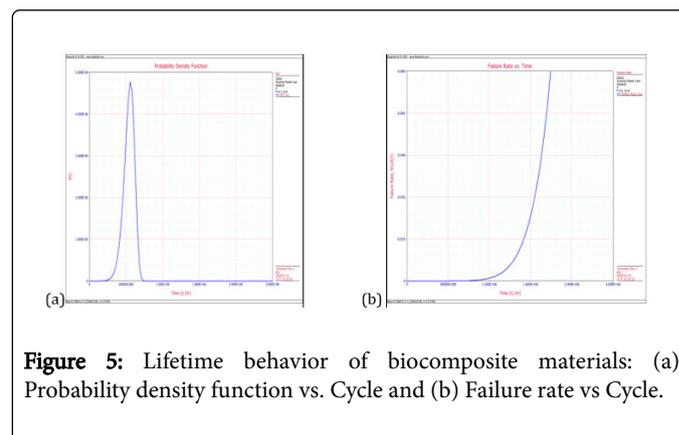


Figure 5: Lifetime behavior of biocomposite materials: (a) Probability density function vs. Cycle and (b) Failure rate vs. Cycle.

Using the model equation, the B_{10} life of biocomposite materials was estimated to be $B_{10}=335,764$ cycles (lower limit) with confidence level=95% as shown in Table 4. The AF were calculated for each accelerated condition from the Stress-Life relationship by setting 5% of the yield strength as the actual field condition for use.

Load applied (% of yield strength)	B_{10} Life (CL=95%, lower limit)
5%	335,764 cycles

Table 4: Prediction of Lifetime.

$$AF = \frac{L_{use}}{L_{accelerated}} = \frac{AV_u^{-n}}{AV_a^{-n}} = \left(\frac{V_a}{V_u}\right)^n \quad (2)$$

- L_{use}: the life at use stress level,
- L_{accelerated}: the life at the accelerated stress level,
- V_u: use stress level,

- V_a : accelerated stress level,

- A , n : constant.

The effect of stress conditions on the AF were shown in Figure 6. It should be noted that it was not recommended to go over the 40% of the yield strength since the linearity of the accelerating test condition did not hold at 50% of the yield strength, for example, shown in the Figure 6, which was due to the different failure modes of 20%, 30% and 40% of the yield strength from that of 50% of the yield strength.



Figure 6: Effect of stress on AF.

Conclusion

The life of biocomposite materials were estimated through fatigue tests under 3 different accelerated conditions of yield strength. The failure time of the biocomposite materials was determined to be the time when the value at the time of the measurement was out of the range of $\pm 10\%$ of the initial yield strength. The lifetime prediction was made by adopting an inverse power eq. model for stress-life relationship, and the B_{10} life (with $CL=95\%$) was estimated to be 335,764 cycles (lower limit).

Symbol

β : Shape parameter,

A : Constant,

B_{10} : a measurement of the time by which ten percent of population of a product will have failed,

CL : Confidence Level,

AF : Acceleration Factors

E_{ta} : Scale parameter,

L : Lifetime,

$L_{accelerated}$: The life at the accelerated stress level,

L_{use} : The life at use stress level,

n : Constant,

V : Stress factor,

V_a : Accelerated stress level,

V_u : Use stress level.

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