

The Storage Life Assessment of PEEK Based on Accelerated Degradation Test

Zhi-Peng Zhang, Jun-Feng Wang, Bei Wang and Xiao-Guang Wang

China Academy of Engineering Physics

Abstract

Degradation data plays a significant role in storage lifetime assessment. In this paper, the accelerated degradation test is performed to obtain the degradation data of the Polyether Ether Ketone (PEEK) under multiple temperature stresses. Then, accelerated degradation analysis is conducted by combining the exponential degradation model and the Arrhenius acceleration model. Based on the results, the activation energy of PEEK is calculated, and the performance level after 20 years' natural storage of PEEK is also obtained.

Keywords: PEEK, Degradation data, Arrhenius acceleration model, Activation energy, Natural storage.

1. Introduction

It is an urgent need to assess the storage life of electromechanical products with the characteristics of "Long-term storage and first using effectiveness" under the high combat requirements. However, the influence factors of the failure of electromechanical products can be numerous for its complex physical structure. Moreover, the performance of such products is closely related to its composed parts and the material property. To accurately predict the storage life of the entire electromechanical products, it necessitates a clear analyzation of its key materials (or parts), and the life characteristic parameters of these key materials (or parts). Therefore, based on the results of the key materials, it can provide valuable data for the whole product storage life prediction. As one of the typical material for special engineered plastics, the Polyether Ether Ketone (PEEK) is widely utilized for its good heat resistance, excellent water resistance, corrosion resistance and good processability (see Zhang et al. [13]). In recent years, the PEEK material is implemented to the insulting plate of multiple types of switches. As one of the key parts of the switches, the insulting plate mainly applied to isolate two switches from contacting with each other (such as, the spring contact and the electrical plate contact). Then, as one of the main influence factors to switching threshold, the switching path, i.e., the distance between spring contact and the surface of electrical plate contact, can

be directly influenced. In General, switching threshold is positively correlation with switching path. Put another way, when switching path increases 1particular type of switch, and its natural storage effectiveness of 20 years, we have to accurately analyze the lifespan of its key components or materials. Therefore, it is necessary for us to carry out the research on the PEEK materials of insulting plate. To do so, we have to master the law of its natural storage, and predict its natural storage performance level of a 20-year duration, and eventually provide data for the entire products' storage life assessment.

In recent years, many attempts of analyzing the lifespan of the PEEK material have been made for special engineering plastics. For instances, Stuart and Briscoe [7] investigated the sliding friction of a PEEK and polyether imide (PEI) compounded blend. It was showed that the frictional behavior of the blend follows a similar trend to that of the unblended PEEK, but the overall friction was higher than the blend. Moreover, a potential reduction in the failure of PEEK material can be achieved when PEI was incorporated. Hu and Li [5] analyzed the PEEK's friction mechanism and performance from the phenomenon on the surface of a PEEK after friction. Furthermore, the melting process of the PEEK particles and the crystallization of the wear debris was also analyzed through the differential scanning calorimetry (DSC) experiment. Niu et al. [6] obtained the relation between the temperature and the tensile properties of PEEK composites from a tensile test. Based on the test results, the influence of the environment temperature on the tensile properties of PEEK composites and the fatigue life has been studied. Dandy and Oliveux [2] studied the accelerated degradation behavior of PEEK composites with the accelerated stress of temperature. Recent research of PEEK, including its chemical modification and physical modification, such as fibre reinforcement, inorganic fillers, surface modification, blending, have been reviewed (see Zhang et al. [12]). Application and prospect of (PEEK) and its composites in the biomedical field were also reviewed (see Zong et al. [16]). Moreover, Wu [9] provided a comprehensive introduction on the course of research and development, main characteristic, application fields and new progress in new century of PEEK resin. Yang et al. [10] instrumentally investigated various properties of PEEK exposed to heat and gamma rays in air. As observed in their work, if PEEK was aged by heat and gamma irradiation in air, chemical bond scission, oxidation, crosslinking, and char formation could simultaneously occur as a competing mechanism. Moreover, the temperature plays a decisive role in degradation, while the irradiation accelerates it. The mechanical properties deteriorated much earlier than the dielectric properties with the progress of aging.

Notwithstanding the above, there is a limited research on natural storage life assessment and natural storage effectiveness of 20 years of PEEK even though the degradation performance theory of PEEK has been established for years. To properly address the aforementioned issue, in this work, the accelerated test of PEEK with temperature stress is conducted. Based on the experimental data, we obtain the natural storage effectiveness of 20 years through degradation process modeling and accelerated modeling.

The remainder of this paper is rolled out as follows: Section 2 provides the accelerated degradation test and modeling analysis of PEEK. A storage life assessment method based on Arrhenius model and exponential degradation model is presented in Section 3. It is followed by a conclusion and a brief discussion in Section 4.

2. Accelerated Degradation Modeling Analysis of PEEK

2.1 Profile of accelerated degradation test

The traditional accelerated life test adopts the accelerated stress to the life test of products. It could shorten the test time, improve the test efficiency, and reduce the test cost (see Chen et al. [1] and Zhang et al. [11]). But for some high reliability and long life products, such as gyroscope, aircraft engine and attitude control flywheel, it is difficult to get the failure data even through the accelerated life test method. This leads the accelerated life test method based on the failure data can not get expected results, and can not meet the needs of reliability engineering. It is in this context, accelerated degradation test technology based on the degradation modeling came into being, and quickly get widespread attention and extensive research. Accelerated degradation test speed up the performance degradation by raising stress level, and collect the accelerated degradation data that is used to predict the storage life under the routine use. Accelerated degradation test which is an emerging method in long life prediction, overcomes the difficulty of the application of accelerated life tests in zero failure (see Deng et al. [3], Deng et al. [4], Wang et al. [8], Zhao et al. [14], and Zhao et al. [15]). It will have broad prospect of application on the study of high reliability and long life products, and become the new ways to solve the reliability problems of the modern high reliability and long life products.

2.2 Accelerated degradation test and data analysis of PEEK

The basic procedures of PEEK's storage life assessment is shown in Figure 1.

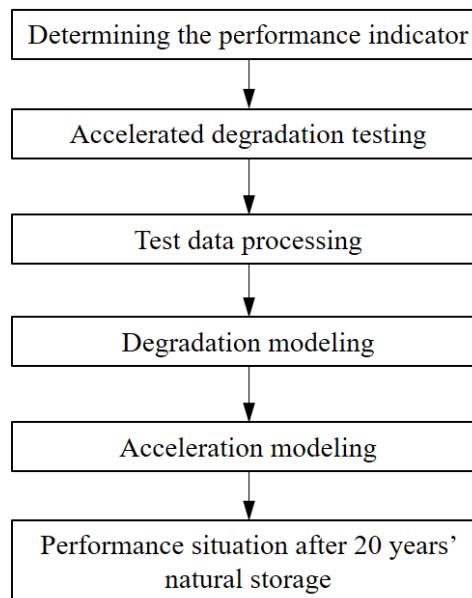


Figure 1: The route of PEEK's storage life assessment.

In the process of accelerated degradation test implementation, considering the PEEK's good hydrolysis resistance, only 0.5% of saturated water absorption under 25°C, the lowest in engineering plastic materials, we believe that the humidity have little effect on the accelerated degradation test. And early wet and hot aging test of PEEK showed that the high humidity of 90% RH did not have significantly influence on PEEK. In conclusion, humidity can be neglected, and temperature can be taken as the single accelerating stress.

During the accelerated degradation test, various properties, such as yield strength, tensile strength, elastic modulus, and volume resistance of PEEK samples were tested respectively, and 5 samples were tested for each performance indicators.

The test values (the mean of 5 samples) of various properties of PEEK after accelerated degradation test under 65°C is illustrated in Figure 2. as observed in Figure 2, the indicators of tensile strength, elastic modulus, and volume resistance do not change obviously, and do not present a trend of degradation. However, only the indicator of yield strength decreases with the increase of the test time, and presents an obvious law of degradation. Therefore, we finally select the yield strength as the only performance indicators to describe the performance degradation process of PEEK.

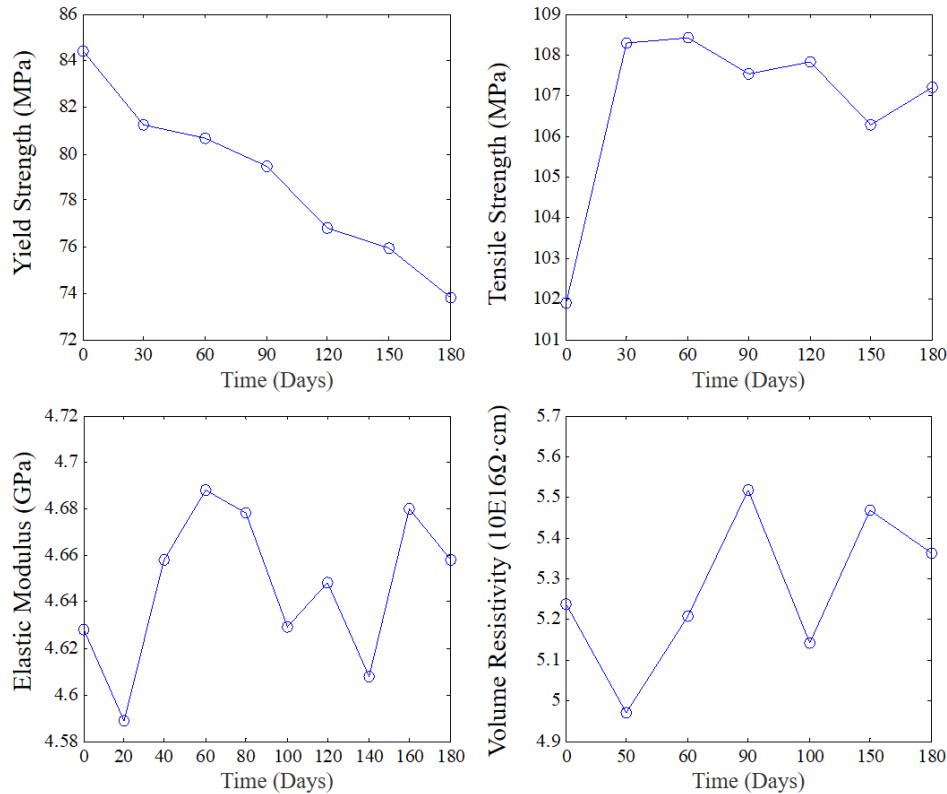


Figure 2: The values of PEEK's various properties.

The PEEK had experienced accelerated degradation tests under 65°C, 85°C and 120°C respectively. During the test, the values of yield strength are measured, and the

values of 5 test samples and the mean values of the samples are shown in Figure 3. Figure 4 gives the corresponding average degradation path of the yield strength.

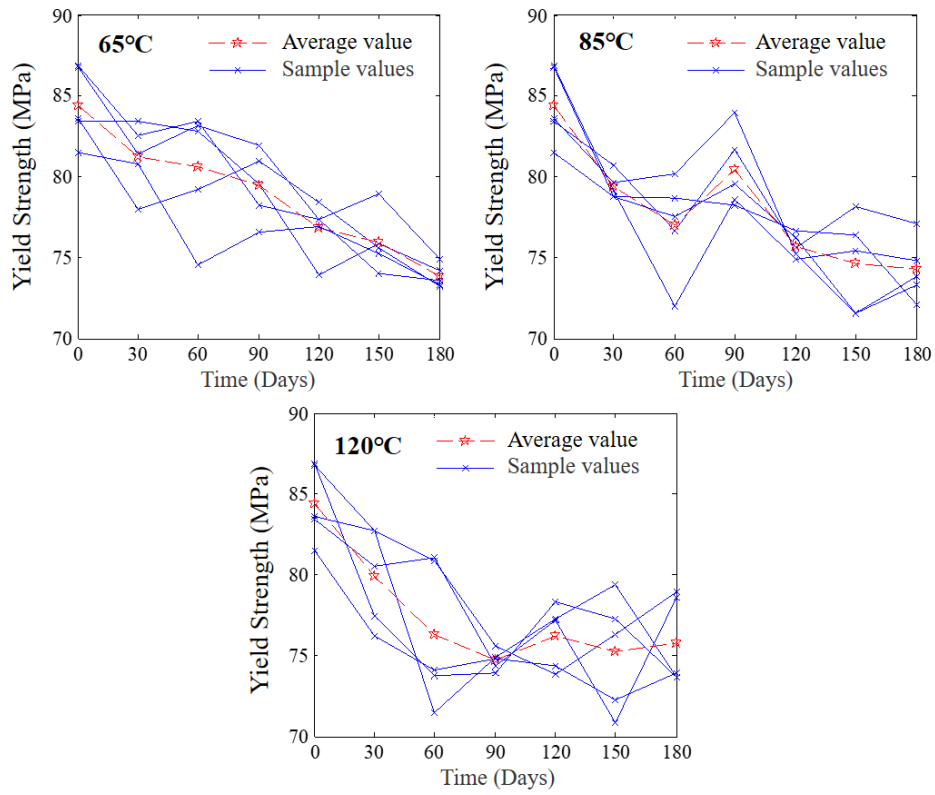


Figure 3: The degradation of the PEEK's yield strength.

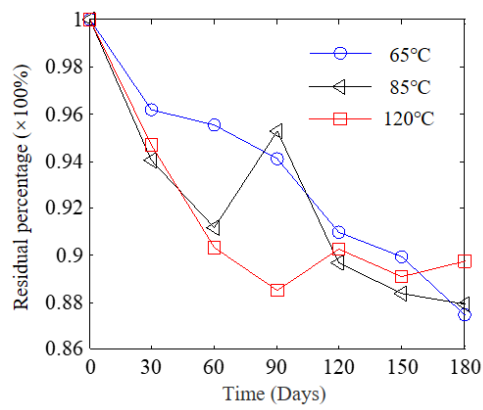


Figure 4: Remaining percentage of PEEK's yield strength.

2.3 Degradation modeling analysis based on exponential distribution

The exponential distribution is used to model the degradation process of yield strength. And widely applicable to mechanical performance degradation model of exponential distribution can be described as:

$$y = B \cdot \exp(-k \cdot t^a) \quad (2.1)$$

where, y is the remaining percentage of the yield strength; B is a constant, k represents the aging rate coefficient; t is the time (unit: days); a is the time index. Considering that when $t = 0$, the remaining percentage of the yield strength y is 100%. Then the constant B equals to 1. Under this circumstance, the performance degradation model of exponential distribution is given by:

$$y = \exp(-k \cdot t^a). \quad (2.2)$$

We fit the accelerated test data of the yield strength under 65°C, 85°C and 120°C respectively with exponential distribution model that shown in Eq.(2.2). And the fitting effect is shown in Figure 5, the corresponding parameters' value under different temperature are shown in Table 1.

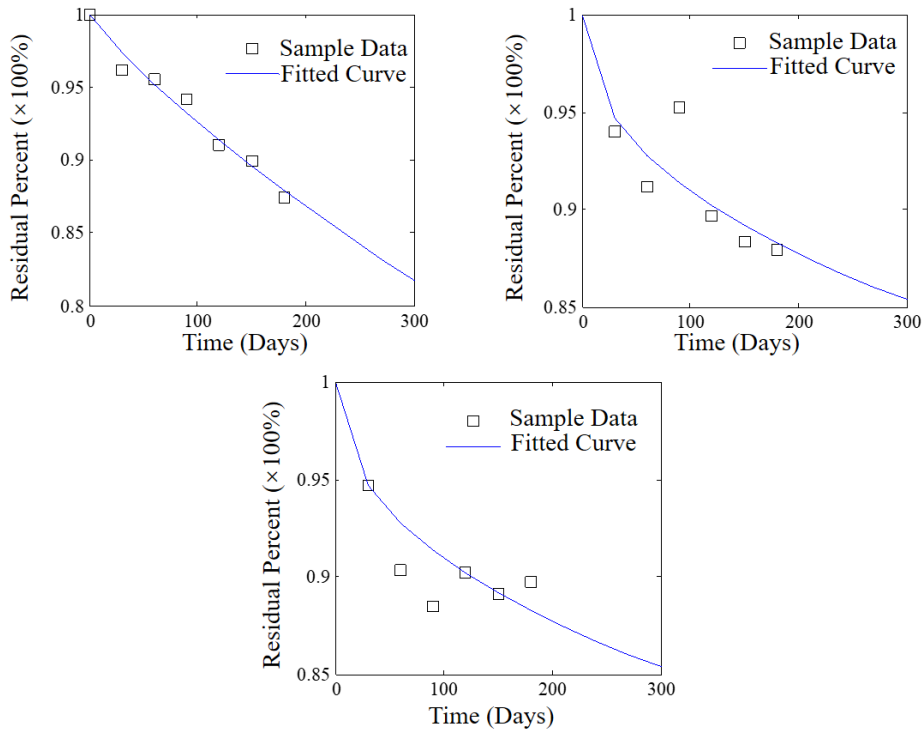


Figure 5: Exponential fitting effect at different temperature.

Table 1: The results of Parameter estimation.

Parameters	Accelerated temperature (°C)		
	65	85	120
k	0.0014	0.0111	0.0285
a	0.8745	0.4659	0.2784

3. Storage Life Assessment of PEEK

In most of the cases, the influence of temperature on the components or materials is described by Arrhenius model. And when the Arrhenius model is used to describe the accelerated degradation process, the aging rate coefficient k of the PEEK's yield strength under the Kelvin temperature T can be further obtained as follows:

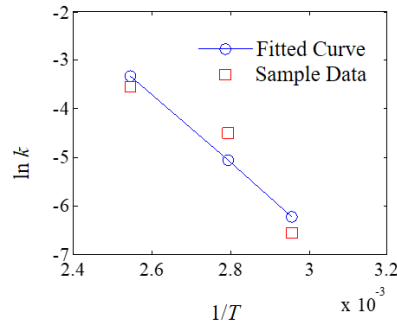
$$k = A \cdot \exp\left(-\frac{E_a}{RT}\right) \quad (3.1)$$

where, A is a constant and independent to the temperature; E_a represents the activation energy (eV); R is the Boltzmann constant (8.623×10^{-5} eV/K); T is the corresponding accelerated Kelvin temperature.

Taking natural logarithm on both sides of Eq. (3.1), we have following equation:

$$\ln k = -\frac{E_a}{RT} \cdot \frac{1}{T} + \ln A \quad (3.2)$$

Eq. (3.2) can be explained that the natural logarithm of aging rate coefficient $\ln k$ has a linear relation with the reciprocal of temperature $1/T$. Thus, combining the parameter values of the aging rate coefficient showed in Table 1, a linear fitting can be taken for $\ln k$ and $1/T$. The results is presented in Figure 6.

Figure 6: Linear fitting results of $\ln k$ and $1/T$.

In Figure 6, the slope of the fitted curve is the value of $-E_a/R$, then we can calculate the value of the activation energy E_a . And it's 0.6041eV.

At this point, the accelerated factor AF of the accelerated temperature T related to the natural storage temperature T_0 can be represented as:

$$AF = \exp \left[\frac{E_a}{R} \left(\frac{1}{T_0} - \frac{1}{T} \right) \right]. \quad (3.3)$$

Thanks to Eq.(3.3), we could calculate the AF of the different accelerated temperature (65°C, 85°C and 120°C) related to the natural storage temperature (25°C). The corresponding results are shown in Table 2.

Table 2: Results of the accelerated factor.

Accelerated factor	Accelerated temperature (°C)		
	65	85	120
AF	16.116	51.2379	292.3325

Then, the remaining percentage of PEEK's yield strength $y_{25^\circ C}$ after natural storage (25°C) of N years can be calculated through the following equation:

$$y_{25^\circ C} = \exp[-K_T \cdot (365N/AF)^{a_T}] \quad (3.4)$$

where, k_T is the aging rate coefficient, and a_T is the time index under accelerated temperature T . AF is the accelerated factor of the accelerated temperature relative to natural storage temperature (25°C). Then the remaining percentage of PEEK's yield strength after 20 years' natural storage can be calculated from the values shown in Table 1 and Table 2. The results are shown in Table 3. And the average value of the remaining percentage is 85.73%. It indicates that after 20 years' natural storage, the remaining percentage of PEEK's yield strength is 85.73% (about 72.38Mpa).

Table 3: Remaining percentage of the yield strength after 20 years' natural storage.

	Accelerated temperature (°C)			Average value
	65	85	120	
Remaining percentage	74.50%	89.42%	92.94%	85.73%

In order to verify the accuracy of our model, the accelerated aging test on PEEK was carried out under 85°C and 120°C respectively. After 150 days of aging test under 85°C (about 20.95 years' natural storage), the yield strength dropped from 84.43 MPa to 74.61 MPa. The corresponding remaining percentage is 88.37%. And after 30 days of aging test under 120°C (about 23.85 years' natural storage), the yield strength dropped from 84.43 MPa to 76.20 MPa. The corresponding remaining percentage is 90.25%.

The accelerated aging test results and our model's calculation results are shown in Table 4.

The accelerated aging test results and our model's results are very close. It suggests the credibility of the calculation results of our model.

Table 4: Accelerated aging test results contrast to our model's calculation results.

Remaining percentage of yield strength	Accelerated temperature (°C)	
	85	120
Accelerated aging test	88.37%	90.25%
our model	89.20%	92.93%

4. Conclusions and Remarks

In this paper, we conducted the PEEK's accelerated degradation test under three different accelerated temperature (65°C, 85°C and 120°C), and carried out the degradation modeling analysis with exponential distribution. To calculate the activation energy, the Arrhenius model was introduced. Finally, the remaining percentage of PEEK's yield strength after 20 years' natural storage was given, and the verification tests show that the assessment result is effectiveness. It provides the data support of storage reliability assessment for electromechanical products with its key material is PEEK.

References

- [1] Chen, X., Zhang, C. H. and Wang, Y. S. (2013). *Accelerated Life Testing Technology and Application*, Beijing: National Defense Industry Press.
- [2] Dandy, L. O. and Oliveux, G. (2015). *Accelerated degradation of Polyetheretherketone (PEEK) composite materials for recycling applications*, *Polymer Degradation and Stability*, Vol. 112, 52-62.
- [3] Deng, A. M. (2006). *Research of Reliability Technology on High-reliability and Long-lifetime Products*, Changsha: National University of Defense Technology.
- [4] Deng, A. M., Chen, X., Zhang, C. H. and Wang, Y. S. (2007). *A comprehensive of accelerated degradation testing*, *Acta Armamentarii*, Vol. 28, 1002-1007.
- [5] Hu, J. and Li, W. (2016). *The study on frictional performance of PEEK and CF/PEEK*, *NCCM-16*, Vol.8, 701-705.
- [6] Niu, Y., Yang, Y. and Yao, J. (2016). *Effect of temperature on tensile properties and fatigue life of carbon fiber reinforced Polyether-Ether-Ketone composites*, *Aerospace Materials and Technology*, Vol.46, 63-67.
- [7] Stuart, B. H. and Briscoe, B. J. (1996). *Sliding friction studies of a poly (ether ether ketone)/ poly (ether imide) blend*, *High Performance Polymer*, Vol.8, 275-280.
- [8] Wang, F. K. and Chu, T. P. (2012). *Lifetime predictions of LED-based light bars by accelerated degradation test*, *Microelectronics Reliability*, Vol.57, 1332-1336.
- [9] Wu, Z. W. (2010). *International and domestic development course and new progress of poly (ether ether ketone) resin*, *New Chemical Materials*, Vol.38, 1-4.
- [10] Yang, L. Q., Ohki, Y. and Hirai, N. (2017). *Aging of poly (ether ether ketone) by heat and gamma rays - Its degradation mechanism and effects on mechanical, dielectric and thermal properties*, *Polymer Degradation and Stability*, Vol.142, 117-128.
- [11] Zhang, C. H., Wen, X. S. and Chen Xun. (2004). *A comprehensive of accelerated life testing*, *Acta Armamentarii*, Vol.25, 485-490.
- [12] Zhang, Y., Shao, C. and Zhang, R. (2012). *Reinforced modification of PEEK and its applications*, *Polymer Bulletin*, Vol.9, 56-62.
- [13] Zhang, Z. D., Xu, J., Sun, K. Y. and Ning, C. C. (2013). *Research development on the wear-resisting performance of PEEK composites*, *Fiber Reinforced Plastics / Composites*, No.1, 94-98.

- [14] Zhao, J. Y., Sun, Q., Zhou, J. L., He, S. B. and Wei, X. F. (2006). *Failure analysis of metallized film pulse capacitors based on accelerated degradation data*, High Power Laser and Particle Beams, Vol.18, 1495-1498.
- [15] Zhao, J. Y., Sun, Q., Peng, B. H. and Zhou, J. L. (2005). *Reliability analysis for accelerated degradation tests*, Electronics Quality, Vol.7, 30-33.
- [16] Zong, Q. Y., Ye, L. and Zhang, A. Y. (2016). *Applications of polyether ether ketone and its composites in biomedical field*, China Synthetic Resin and Plastics, Vol.33, 93-96.

Institute of Electronic Engineering, China Academy of Engineering Physics, Mianyang, Sichuan, 621900, P.R. China.

E-mail: 641465790@qq.com

Major area(s): Mechanical design, reliability assessment.

Institute of Electronic Engineering, China Academy of Engineering Physics, Mianyang, Sichuan, 621900, P.R. China.

E-mail: wangjunfeng@caep.cn

Major area(s): Mechanical design, reliability assessment.

Institute of Electronic Engineering, China Academy of Engineering Physics, Mianyang, Sichuan, 621900, P.R. China.

E-mail: 609012061@qq.com

Major area(s): Mechanical design and manufacture.

Institute of Electronic Engineering, China Academy of Engineering Physics, Mianyang, Sichuan, 621900, P.R. China.

E-mail: wxgrain@163.com

Major area(s): Material mechanics and design.

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