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Accelerated Vibration Life Tests of Threadlocking Adhesive

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Threadlocking adhesives are widely used in industry to reduce self-loosening of threaded fasteners in dynamic environments. However, accelerated vibration life tests for such adhesives have not been reported in the literature. This paper reports on the development of an apparatus and test procedure to evaluate threadlocking adhesive life when subjected to accelerated vibration testing. Data from five different threadlocking adhesives are presented and compared. The data for each adhesive is found to fit a Weibull distribution. The relationship between threadlocking adhesive life and applied vibration level is shown to be modeled quite well with an inverse power relationship.

Nomenclature

$F(t)$ = cumulative distribution
g = acceleration constant
k = empirical constant
n = empirical constant
p = percentage
t = time
V = vibration level
γ = Weibull shape parameter
θ = Weibull scale parameter
τ_p = percentile

Introduction

THREADED fasteners are used when a load-bearing connection that can be disassembled without destructive methods is required in a design. To some extent, all fasteners are subjected to dynamic loading, which can result in self-loosening. Self-loosening is commonly caused by vibration but can also be caused by temperature or pressure cycles.[1]

One method to resist self-loosening is to use threadlocking adhesives.[2] Such products have found widespread use in industry. However, accelerated vibration life tests for such adhesives have not been reported in the literature. Customers as well as users of machinery assembled with threadlocker are interested in dynamic life data to estimate its useful life. In fact, this work was initiated in response to an expressed need for such data by one of the leading aircraft manufacturers in the U.S. Manufacturers could use such data to develop product warranties and service schedules.

The traditional method for assessing the strength of threadlocking adhesives is to measure the break and prevail torque of an assembly consisting of a bolt and nut with adhesive.[3] The resistance of these products to adverse environments, e.g., heat, humidity, and solvents, is assessed by immersing the assembly in the environment for a period of time and then measuring the break and prevail torque. The main limitation of this traditional method is that the fastener assembly test specimens do not bear any load.

A more recent method of assessing the strength of thread adhesives that use load-bearing or pretorqued fastener assembly test specimens has been developed.[4] However, this method has not been used enough to provide a significant history and, as a result, has not replaced the traditional method.

Rich[3] developed a procedure for evaluating the thermal resistance of threadlocker. In his procedure, pretorqued fastener assembly specimens are subjected to various elevated temperatures for a fixed period of time. The specimens are then subjected to the minimum torque, which they should be able to resist. A specimen fails if it does not resist the torque. A specimen passes if it does resist this test torque. When a specimen passes, it is returned to an elevated temperature for a fixed amount of time and again tested with the minimum test torque. This process is repeated until all specimens fail. Life data are modeled with an Arrhenius-temperature relationship that provides life estimates at a normal temperature.

The focus of this paper is to present a test method to evaluate threadlocking adhesives through accelerated vibration life testing. It is anticipated that the accelerated vibration test described in this paper could be developed into a standard vibration life test for threadlocking adhesives.

The test apparatus used in this work consists of a compound cantilever beam similar to the directly loaded side-slider developed by Haviland[5,6] for demonstrating loosening. This apparatus provides a realistic vibration test because it represents the most common type of structures that causes shear loading on a fastener. It includes joints and panels of most major buildings, aircraft, cars, homes, and household appliances. Kerley[7,8] performed experiments using this type of apparatus with inertial loading to study the problem of vibration-induced loosening. More recently, Dong and Hess[9] used this type of apparatus with inertial loading to investigate the effect of dynamic shear force on fastener loosening and fastener placement in assemblies. In addition, this apparatus provides more realistic and repeatable results than the more severe existing standard tests, such as MIL-STD-1312-7A[10] and NAS 1675,[11] which are dynamic tests used to assess loosening of threaded fasteners. These tests generate large shock and impact loads.

Experiments

Test Apparatus

The test apparatus used in this work consists of a compound beam mounted on a test fixture. Figure 1 shows the test apparatus. The compound beam consists of two pieces of 316L stainless steel that are 11.5 in. (292.1 mm) long, 1.5 in. (38.1 mm) wide, and 0.117 in. (3.0 mm) thick. The combined mass of the two pieces is 1.14 lb (0.52 kg). The fixed end of the beam is attached on the test fixture with four 0.25–20 UNC socket head cap screws with a torque of 150 in.-lb (16.95 N-m). The overhang length of the cantilever beam is 10 in. (254 mm) because 1.5 in. (38.1 mm) of the beam is secured on the test fixture. There is a 0.394 ± 0.002 in. (10.00 ± 0.05 mm)

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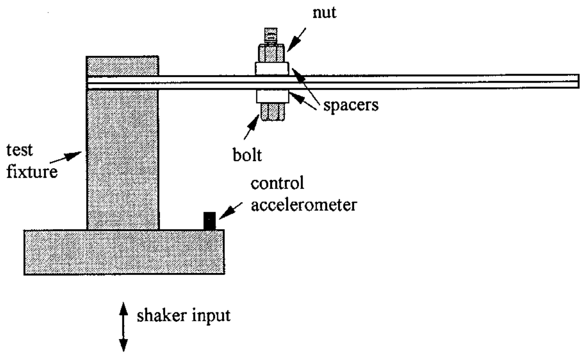

Fig. 1 Test apparatus.

diameter hole at the center of the beam, 7 in. (177.8 mm) from the free end. The test bolt, test nut, and two spacers are attached to the compound beam through this hole. The test bolts are M-10 × 1.5, 60 mm long, hex cap, class 10.9 steel bolts coated with phosphate and oil. The test nuts are M-10 × 1.5 hex, GM510M(9) steel nuts with a plain finish.

The test spacers are made of 1018 carbon steel. Their specification are as follows: 0.394 ± 0.002 in. (10.00 ± 0.05 mm) i.d., 0.75 in. (19.1 mm) o.d., and 0.65 in. (16.5 mm) thickness. Both ends of the spacers are finished to 0.004 in. CLA (0.1 mm Ra) and heat-treated to a hardness of 47 to 50 RC to a depth of 0.0025–0.0030 in. (0.635–0.762 mm).

The test fixture is made of aluminum and consists of three pieces. The top piece is a cover block of dimension 1.5 in. × 2 in. × 0.5 in. (38.1 mm × 50.8 mm × 12.7 mm) with four 0.25-in. (6.35-mm) holes. The middle piece is a 1.5 in. × 2 in. × 2.5 in. (50.8 mm × 38.1 mm × 63.5 mm) block with eight 0.25 × 20 UNC tapped holes, 1.5 in. in depth. The bottom piece is a cylindrical base of 5 in. (127 mm) diameter and is 1 in. (25.4 mm) thick with four 0.25-in. holes and four 0.375-in. holes. The middle block is attached on the base with four 0.25–20 UNC socket head cap screws with a torque of 150 in.-lb (16.95 N-m). The bottom piece is fixed on a shaker table with four 0.375 in.-20 UN socket head cap screws with a torque of 250 in.-lb (28.25 N-m). Twelve complete test fixtures and beams were built for this work.

Test Plan

A brief description of the test procedure is given as follows. Firstly, all of the test components were cleaned with acetone. The test beam was lubricated with naphthenic oil. Threadlocking adhesive was applied to the test specimen. The test specimen was installed on the beam with a tightening torque of 300 in.-lb (33.9 N-m) and allowed to cure for 48 h. Then the test specimen is tested with a test torque of 250 in.-lb (28.25 N-m) after one test interval. If the test specimen fails to resist the test torque, the test time is recorded. If the test specimen passes the test torque, the vibration test is continued for another test interval until the test specimen fails or the test time reaches the censored time of 30 min. The failure is assumed to occur at the upper endpoint of the last test interval. The defined failure does not necessarily correspond to an actual failure, e.g., complete loosening and/or bolt breakage, in a practical application.

Five threadlocking adhesives, TL242, TL262, TL271, TL290, and TL272 from Loctite Corporation are tested in this work. In most of the tests, a torque wrench with a specified test torque is used to assess the test specimen. Established recommendations and guidelines for good test plans for accelerated life tests are followed.[12] As a result, two or three levels of vibration are used. In these tests, the level corresponds to the amplitude of an applied sinusoidal vibration at a constant frequency of 50 Hz. Also, at least 10 test specimens are tested at each vibration level. Table 1 shows the test matrix.

Table 1 Test matrix

Vibration level, *g*	Test specimens				
	TL242	TL262	TL271	TL290	TL272
10	13	——	——	——	——
20	13	11	10	11	——
30	12	12	10	10	——
40	——	12	10	10	10
50	——	——	——	——	10

During preliminary testing it is found that the range of time to failure for the five adhesives is very large. The main test parameters that affect the time to failure include the tightening torque, test torque, and vibration level. Because it is desirable to compare the life-vibration data for the five adhesives, it is necessary to use the same test parameters for each adhesive. The task of determining test parameters that provide a reasonable range of time to failure is accomplished through preliminary tests.

The preliminary tests revealed that failure is caused by a combination of preload loss and adhesive degradation. For a given vibration level it is found that the time to failure and the test torque for the weakest adhesive could be increased by increasing the tightening torque. At the other extreme, the time to failure for the stronger adhesives could be decreased by increasing the test torque. Through trial and error the optimum values for the tightening torque and the test torque for the five adhesives are determined as 300 in.-lb (33.9 N-m) and 250 in.-lb (28.25 N-m), respectively.

With these values of tightening torque and test torque, all of the test specimens with TL262, TL271, and TL290 adhesives fail before the selected censored time, i.e., maximum test time of 30 min, at the vibration levels of 20, 30, and 40 *g*. The test specimens with TL242 adhesive usually fail in less than 10 s at 40 *g*. In an effort to keep time to failure between 10 s and 30 min, test specimens with TL242 are tested at 10, 20, and 30 *g* instead of 20, 30, and 40 *g*. Similarly, because many test specimens with TL272 do not fail after 30 min of testing at 20 and 30 *g* and, because beam fatigue failure occurs at 60 *g*, test specimens with TL272 are tested only at 40 and 50 *g*.

With the bolt tightened to the selected tightening torque of 300 in.-lb (33.9 N-m), it has a load of 2900 lb (12,899 N), as measured with a load cell. This corresponds to 21% of the bolt yield strength. Also, through impact hammer tests, the fundamental natural frequencies of the compound beams with the test specimens fastened to this torque are found to be within 58.5 ± 0.5 Hz. The preliminary tests show galling of the beams for a tightening torque of 500 in.-lb (56.8 N-m) or higher.

Another important test parameter, which is not shown in the test matrix, is the test time interval. After a test specimen is run for one test time interval, it is checked with the test torque to see whether it fails. In this work, the test time interval varies with test product or adhesive and vibration levels, and is set to a maximum 25% or less of the average time to failure found in the preliminary tests. For example, a test time interval of 5 s is used for test specimens with TL242 adhesive at 30 *g*; a test time interval of 2 min is used for test specimens with TL272 at 40 *g*. During testing, if a test specimen appears to be coming loose, the test interval is reduced.

Although the test matrix presented in Table 1 provides sufficient data to develop a life-vibration statistical model for adhesive threadlocking products, the data provide no information about the relative amounts of preload loss and adhesive degradation that occur during a life test. In an effort to quantify these failure modes, two additional specimens are tested at each vibration level. These tests utilize an ultrasonic instrument to measure bolt elongation before and after being subjected to vibration. The duration of the vibration for these tests

Table 2 Failure times[a]

TL242			TL262			TL271			TL290			TL272	
10 *g*	20 *g*	30 *g*	20 *g*	30 *g*	40 *g*	20 *g*	30 *g*	40 *g*	20 *g*	30 *g*	40 *g*	40 *g*	50 *g*
60	30	10	120	60	30	300	150	100	300	120	70	480	360
80	30	15	130	70	35	360	150	120	360	140	80	540	420
80	40	15	140	80	35	360	180	120	360	140	80	600	480
100	40	15	160	80	40	360	180	120	360	160	85	600	480
100	40	15	160	80	50	420	210	140	420	160	90	720	600
120	40	20	160	90	50	480	210	140	480	180	90	780	600
120	40	20	170	90	55	540	270	140	480	240	100	960	600
120	50	20	180	100	60	540	270	160	570	260	100	1500	720
120	50	25	190	100	60	540	300	180	780	300	120	1560	780
140	50	25	210	120	65	660	330	210	840	360	120	1740	840
140	60	25	230	120	65	——	——	——	840	——	——	——	——
140	60	25	——	120	65	——	——	——	——	——	——	——	——
160	60	——	——	——	——	——	——	——	——	——	——	——	——

[a]Failure times in seconds.

is selected to equal the 10% characteristic time. The characteristic times correspond to the estimated times at characteristic reliability levels, e.g., a 10% characteristic time corresponds to a characteristic reliability or survival level of 0.1, computed using the statistical life distribution model.[13] In addition to the elongation measurements that provide an estimate of preload, the breakloose torque of the test specimens are measured and compared to static breakloose torque measurements, to provide an estimate of adhesive degradation. The static break-loose torque is the maximum torque required to loosen a cured bolt that is not subjected vibration.

Test Data Analysis and Modeling

Table 2 lists the failure times for each adhesive and vibration level tested. A minimum of 10 tests is performed for each configuration. The failure times are listed in increasing order, not in the order in which they are obtained.

A statistical model of an accelerated life test consists of a life distribution and a relationship between test specimen life and the accelerated stress. Such a model depends on the product, the test method, the accelerating stress, the form of test specimen, and other factors.

The life distribution for a given test configuration represents the scatter in product life. Commonly used life distributions include the normal, exponential, lognormal, Weibull, and extreme value distributions. The most widely used model for life test data, the Weibull distribution, is used in this work. In many applications, both Weibull and lognormal distributions fit the same set of data well, which is found to be true in this work. However, the Weibull model is a little more conservative than the lognormal model in the lower tail of its distribution. Also, the lognormal hazard function has a strange property seldom seen in products. Therefore, the lognormal model is not used in this work.

The cumulative distribution for the two-parameter Weibull model is defined as

$$F(t) = 1 - \exp[-(t/\theta)^{\gamma}], \quad t > 0 \tag{1}$$

Once this Weibull model is fit to the data in Table 2, Eq. (1) can be used to estimate the fraction or percent of test specimen failures at any time t. A Weibull model is fit to each of the 14 data sets presented in Table 2.

There are many methods available for estimating the Weibull model parameters, θ and γ, and associated confidence intervals. The maximum likelihood (ML) method of estimation is considered the most accurate parameter estimation method and is used in this work. Before estimating the Weibull parameters using the ML method, a goodness-of-fit test is performed to check the adequacy of the Weibull model. Two goodness-of-fit tests, probability plotting and hazard plotting, have been used to check for all 14 populations listed in Table 2. All of the R-squared values fall within 0.85–0.96, which indicates the Weibull model fits the test data well.

Table 3 Maximum likelihood method estimates of Weibull distribution parameters

Product	Vibration, *g*	θ	γ
TL242	10	124.5	4.863
	20	49.4	5.045
	30	21.1	4.556
TL262	20	181.5	5.665
	30	100.4	5.378
	40	55.6	5.001
TL271	20	499.0	4.618
	30	248.3	4.101
	40	155.8	4.698
TL290	20	592.1	2.936
	30	231.7	2.906
	40	100.4	6.219
TL272	40	1077.0	2.291
	50	655.2	4.028

Weibull parameters are estimated via the ML method. Table 3 shows the estimates of Weibull distribution parameters using the ML method. All of the Weibull shape parameters, γ, are greater than 1, which indicates that the test specimen failure rate increases with age under the vibration environment.

Now that the life distributions have been modeled for each of the 14 test configurations, the next step is to develop a life-stress relationship, i.e., life vs vibration model, for each adhesive tested. The life-vibration model can be used to predict the life of an adhesive over a wide range of vibration levels. There are several life-stress relationships available. Although the Arrhenius life-stress relationship is generally used to predict life as a function of temperature (thermal stress) for thermal life tests, the inverse power relationship is the most widely used relationship to model product life as a function of most other accelerating stresses. It is found that the life-vibration relationships for the five different adhesives are best modeled with the inverse power law. The statistical model for each adhesive, which consists of Weibull life distributions for each test configuration and an inverse power-law life-vibration relation, is referred to as a power-Weibull model. For an inverse power-law life-vibration relation for each adhesive, the Weibull characteristic life θ as a function of V is defined as

$$\theta(V) = 1/(kV^{n}) \tag{2}$$

where k and n are model parameters that depend on the product and test method.

The two assumptions for the power-Weibull model are that the product life has a Weibull distribution for each test V, and that for a given product the γ is constant and independent of vibration level. The first assumption is satisfied because the adhesive life for each of the test configurations has been shown to have a Weibull distribution. For the second assumption one finds that the expected Weibull shape parameters for each adhesive, except for TL272 and TL290, are essentially constant (Table 3). The shape parameters for adhesives TL272 and TL290 show more variation, but not atypical of data fitted to the power-Weibull model.

The inverse power-law parameters, k and n, are estimated with the typical life data for each adhesive. The estimates are summarized in Table 4, together with their corresponding R-squared values that indicate or measure how well the model fits the data.

The inverse power model can be used to predict the adhesive life at any vibration level. The life-vibration relation is usually presented with percentiles. For the Weibull model, the percentile τ_p is the solution of

$$p = F(\tau_p) = 1 - \exp[-(\tau_p/\theta)^\gamma] \tag{3}$$

which results in

$$\tau_p = \theta[-\ell n(1 - p)]^{1/\gamma} \tag{4}$$

Substituting Eq. (2) into Eq. (4) leads to an expression for percentile as a function of vibration level

$$\tau_p(V) = [1/(kV^n)][-\ell n(1 - p)]^{1/\gamma} \tag{5}$$

Taking the logarithm of both sides of Eq. (5) results in

$$\log[\tau_p(V)] = \{\log[-\ell n(1 - p)]^{1/\gamma} - \log k\} - n \log V \tag{6}$$

Because the actual shape parameters for a given adhesive are slightly different at different vibration levels, the average shape parameter for a given adhesive is used to compute the percentile lines defined by Eq. (6). The data and associated percentiles for adhesive TL242 are shown in Fig. 2. The inverse power relationship with percentile lines for products TL262, TL271, TL290, and TL272 have also been computed.[13] With these models, one can readily predict the product life at any vibration level. For example, using Fig. 2 one finds that the 63.2th percentile of TL242 at 0.1 g is predicted as 2×10^5 s.

For comparison, Fig. 3 presents the typical (63.2th percentile) life-vibration relationships for all five threadlocking adhesives. It shows that over the range of 0.1–100 g vibration, a test specimen life increases with adhesive type from TL242, TL262, TL271, to TL272. The test specimen life with adhesive TL290 falls between that of TL271 and TL272.

The different behavior of product TL290 may be explained as follows. Because it has a much lower viscosity than the other adhesives tested, it is more difficult to control the amount applied to a test specimen. This may also explain why this product had a relatively large variation in the Weibull shape parameter for different vibration levels.

Product TL272 is found to be the strongest adhesive among the five products. A small variation in the amount applied to a test specimen may lead to a significant variation of failure test time, which could explain the relatively large variation of the Weibull shape parameter for this product.

It also is useful to know what fraction of product fails by time t at any V. An expression for the cumulative distribution as a function of time and vibration level is obtained by substituting Eq. (2) into Eq. (1):

$$F(t;\, V) = 1 - \exp\{-[t(kV^n)]^\gamma\} \tag{7}$$

Figure 4 shows the computed cumulative distribution of product TL262 for vibration levels of 1, 20, 30, and 40 g. The

Table 4 Inverse power model parameters

	Inverse power model: $\theta(V) = 1/(kV^n)$		
Product	k	n	R-squared values
TL242	1.9839E-04	1.58655	0.984
TL262	3.3961E-05	1.68966	0.991
TL271	1.3045E-05	1.68210	0.999
TL290	8.0538E-07	2.54393	0.996
TL272	2.5096E-07	2.22724	1.000

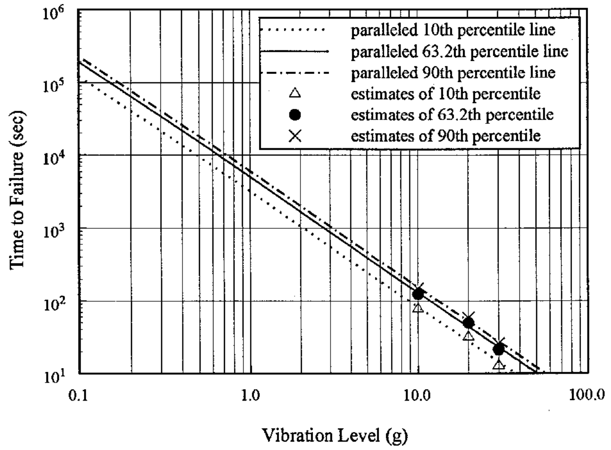

Fig. 2 Inverse power relationship with Weibull percentile lines for TL242.

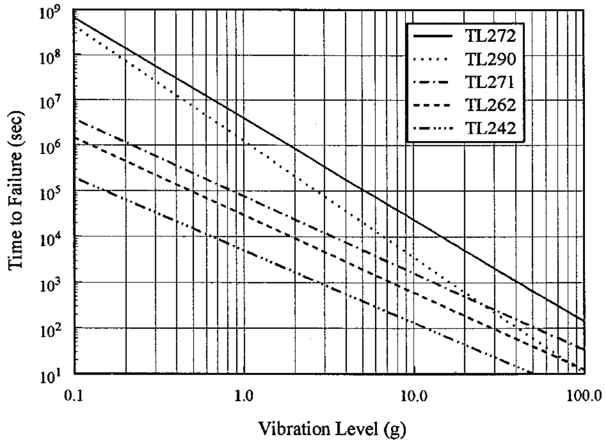

Fig. 3 Typical (63.2% failure) life-vibration relationship for threadlocking adhesives.

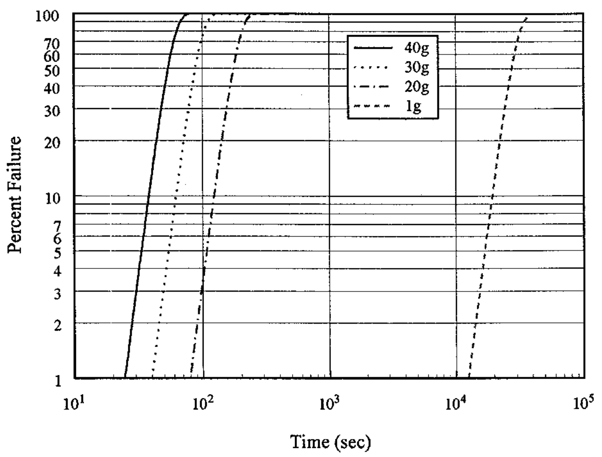

Fig. 4 Cumulative distribution of TL262 with power-Weibull model.

average shape parameter for this adhesive is used to calculate the cumulative distribution function. The cumulative distribution functions for TL242, TL271, TL290, and TL272 have also been calculated.[13] Using these plots, one can determine the percentage of product failure by *t* for the different vibration levels. For example, if test specimens with TL262 are tested at 1, 20, 30, and 40 *g* for 60 s, the corresponding percent of failure as predicted with Fig. 4 are less than 1, less than 1, 8, and 87%, respectively.

The results from the ultrasonic measurement tests show that the bolt elongation of all test specimens are reduced by 19–64% due to vibration.[13] This corresponds to reductions in bolt loads between 16 and 68%. The reduction in elongation and load appear to be somewhat higher for the stronger adhesives. The average break-loose torque obtained from specimens subjected to vibration were found to be anywhere from 18 to 85% less than the break-loose torque measured from specimens not subjected to vibration. Assuming a correlation between adhesive degradation and change in break-loose torque from static to posttest measurements, the stronger adhesives experience a much more significant degradation during the life tests than the weaker adhesives. This is perhaps an obvious result, because for the tests described in this work, the stronger adhesives have to degrade more significantly to fail.

Conclusions

An apparatus and test procedure have been developed to evaluate threadlocking adhesive life under vibratory conditions. It has been found that the life test data obtained using the developed test apparatus and procedure can be modeled using Weibull distributions and inverse power-law equations. To illustrate the approach, data have been presented for the evaluation and comparison of five threadlocking adhesives. The results show that the test apparatus and procedure offer a viable approach that could develop into a standard life test for threadlocking adhesives.

Acknowledgments

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