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Aging Characterization of Adhesives and Bonded Joints by Non-Destructive Damping Measurements

Klara Shiloh, Avraham Eliahu, Avraham Rosenberg and Raphael Zilber

SOREQ NRC, Yavne 81800, Israel.

and

Aliza Buchman

RAFAEL ADA, Haifa, Israel.

ABSTRACT

Aging of adhesives is usually characterized by preparing standard bonded samples and destructively measuring shear, tensile or peel adhesion strength. Similarly the integrity of bonded devices is ascertained by destructive testing of duplicate samples, manufactured under identical conditions. The method requires a great number of samples in order to monitor the aging process.

A new non-destructive testing method is suggested based on many experimentally determined correlations between the internal friction (or damping) properties of materials and their strength, as a tool of characterizing the aging status of adhesives in bonded devices.

Damping measurements were performed on simple flat and cylindrical joints made out of metallic adherents and various adhesives. The joints were aged in natural environment as well as through thermal accelerated treatments. Vibrations were introduced into the samples by a brief impact with an electromagnetic shaker and detected with a microphone. Special care was devoted to the fastening of the samples to insure reliable results. The NDT measurement results were corroborated by destructive shear tests of new and aged bonded joints.

The results show that aging of adhesives can indeed be non-destructively characterized. Very good correlation was found between the specific damping capacity of the samples and the shear strength of the joints.

Keywords: vibration analysis, damping, aging, internal friction, adhesive joints, ndc, ndt, nde.

1. INTRODUCTION

Evaluation of materials by vibration techniques is unique among the other non destructive techniques in its being rapid and also by its ability to measure material quality as opposed to mere defect detection

Propagation of sound in materials closely simulates mechanical processes occurring in those materials (1). This statement is accurate as far as the elastic regime is concerned. Therefore, the various elastic moduli (or stiffness constants) can be evaluated by properly applying ultrasonic velocities or modal resonance frequencies measurements (2).

It is less known, and thorougly established theoretically, that damping capacity measurements can reflect material ultimate strength. Still, there is more and more evidence of direct correlation between material strength (or other material properties), and damping capacity, measured either by modal analysis or by acousto-ultrasonics in: metals (3-6), ceramics and ceramic composites (7-10), plastic materials (11-13), interfacial strength (14,15), adhesive joints (16,17). Increased specific damping capacity (equivalent to increased internal friction during the vibration movements) indicates degradation or reduced strength (negative correlation). The mechanism responsible for the internal friction is the vibration energy absorbed by sliding inner layers or void deformations. Those same voids and/or free layers are also responsible for strength degradation.

Establishing the correlations mentioned above, for newly developed materials or materials subject to aging or environmental degradation (otherwise non -destructively undetectable) can provide means for quick material evaluation. Moreover, the method can potentially become a powerful tool for the evaluating the aging status of materials incorporated in industrial devices.

Glues are among the most sensitive to aging materials. The strength of entire bonded devices depends on the strength and durability (reduced sensitivity to aging) of their adhesive joints. The research, (as well as the quality assurance) on aging of bonded joints is mostly conducted using destructive testing such as tensile, shear, flexural, fracture toughness or wedge experiments. All those data are collected on samples, rather than on the actual loaded components.

Periodic non-destructive strength evaluation of bonded devices is most welcome for both research and quality assurance.

In the work presented here, the possibilities of non-destructive quality assurance of adhesive joints by vibration tests is investigated. The results of destructive shear tests of bonded joints (DT) were compared to those of vibration (NDT) ones after sample exposure to identical aging conditions, with very satisfactory results.

2. EXPERIMENTAL

2.1. Vibration Measurements - Experimental Method

The experimental system is shown in Fig. 1a.

The method is based on impact excitation of the sample (fixed at one end and free at the other) and monitoring the displacement of the free end. The impact is activated by a pulse triggered electromagnetic shaker (Wilcoxon F4 Driver) and the vibration monitored by a microphone (Shure, SM98). A forcemeter and an accelerometer were attached to the hammer. Special attention was given to the sample holder (shown in Fig, 1b), to prevent any influence of the fixture on the measured results. It was verified that the overall damping was dominated by the sample.

The microphone readings were digitized by a CIO-AD16Jr-AT (Computerboard) board loaded in a PC.

Figure 1a
Experimental Setup

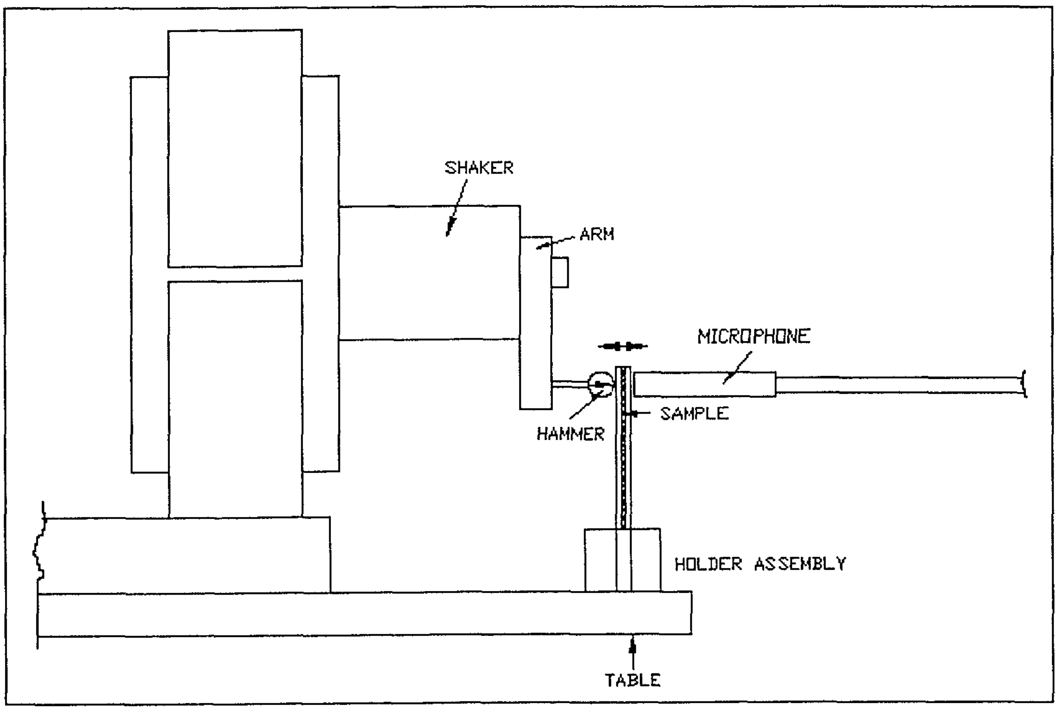
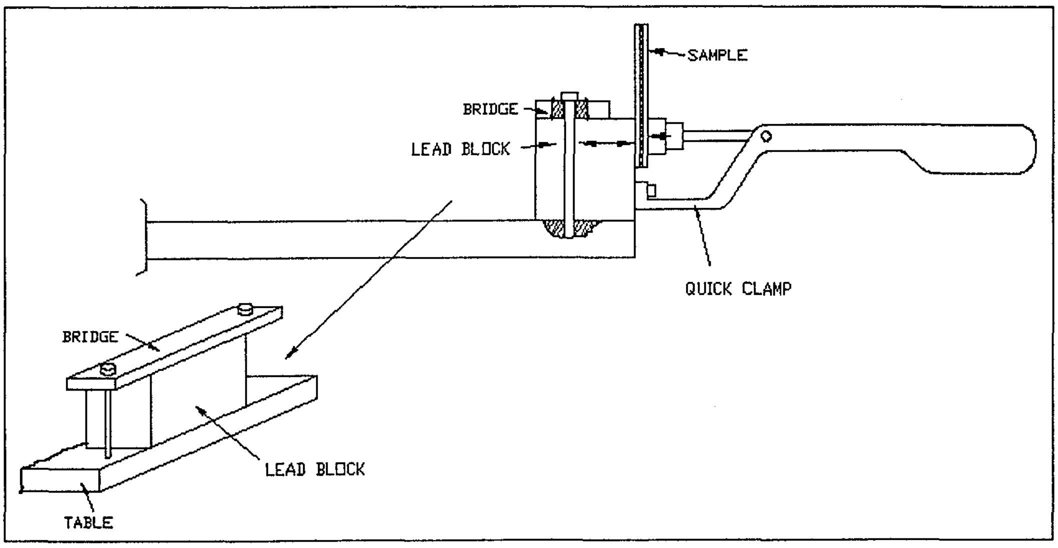


Figure 1b
Sample Holder Assembly



2.2. Materials

Two adhesives were tested:

An Epoxy adhesive - Epon 828 (Miller Stephoson) cured with an excess of DPM 30 and modified with a sulfide LP3 (Thiokol Chem. Ltd.). Curing was at room temperature (R.T.) for 24 hr. This adhesive is sensitive to aging, with quickly degrading properties even at ambient conditions. It will be related as LP.

The second adhesive is a polyurethane, based on MDI with a polyol curing agent (SW-4943 of Grace Co.). Curing was also at R.T. This adhesive is much less sensitive to aging. It will be related as SW.

The adherent used was a model adherent Al-2024 treated with Chromic anodization.

2.3. Destructive Tests Data

Shear tests of bonded joints, based on LP and SW were carried out, as a part of glue aging research and glue verification. The joints were exposed to various forms of aging, natural and accelerated, as described next. Extensive statistics was accumulated for the shear strength of the joints of both glues, after exposure to the natural and the two types of accelerated aging conditions. Shear tests were taken from non aged reference samples too. The shear tests (DT) results were compared with the vibration (NDT) ones, as seen in the section on the results.

2.4. Accelerated aging

The accelerated aging procedures, which simulated best the natural processes are given in Table 1:

Table 1
Accelerated Aging Procedures

mild accelerated aging	4 hours at 65 °C
intensive accelerated aging	48 hours at 65 °C, followed by 48 hours at -10 °C

2.5. Specimen

2.5.1. Flat Samples:
The basic test coupons were 1"x6"x1/8" thick Al plates glued all along with the specified adhesives. The glued joins were first tested with NDT damping method and then machined into Schlickelman test samples and tested in shear by an Instron machine at a rate of 2mm/min.

2.5.2 Cylindrical Samples:
The next set of specimen consisted of, composed of two rounded Al adherentsglued together by use of a vacuum bag, used in order to apply uniform pressure on the whole surface.

2.6. Calculations of Modal Frequencies and Damping

Matlab was the software package used for the calculations. The Modal Frequencies (in most cases only one frequency survived) were calculated by FFT from the recorded microphone waveforms (after deconvolution from the hammer forcemeter ones).

The damping coefficient was calculated in the time domain, since the frequency resolution for the relatively short duration of each waveform was too low to yield meaningful results.

3. RESULTS

3.1. Non-Destructive Tests

3.1.1. Accelerated Aging
Each specimen was tested by the vibration method shortly after preparation and then a frequent follow-up was carried on. Groups of specimens were taken at chosen times to be exposed to accelerated aging conditions. Those specimen were tested shortly after the aging procedure and again follow-up was carried on.
In all cases it was found that the damping coefficient of the LP samples was increased by a large factor, whereas the damping coefficient of the SW samples was slightly decreased. Typical results of the flat samples are shown in Fig. 2 and of the cylindrical ones - in Figure 3. The arrows in the figures in all cases point to the change which occurred in the damping coefficient before and after the accelerated aging procedure.

The modal frequency of the flat samples was not affected by the accelerated aging procedure in the case of the flat samples, whereas in the case of cylindrical LP samples, the frequency dropped by about 50 Hz (20%). The reason for that difference is the glue content of the sample. In the flat samples the amount of the glue was negligible. In the cylindrical ones - 10 to 20 volume %. In the flat ones, only the strength and not the modulus was affected. In the cylindrical ones - the modulus too.

3.1.2 Natural Aging
Part of the specimen was not exposed to accelerated aging but was left for frequent follow-up. The damping coefficient of both the LP and SW samples was increased, but the slope of the LP was ~3 times higher. Typical results can be seen in Fig.4. T he reference samples are made of aluminum only.

Figure 2
Accelerated Aging of LP and SW
(Flat Samples)

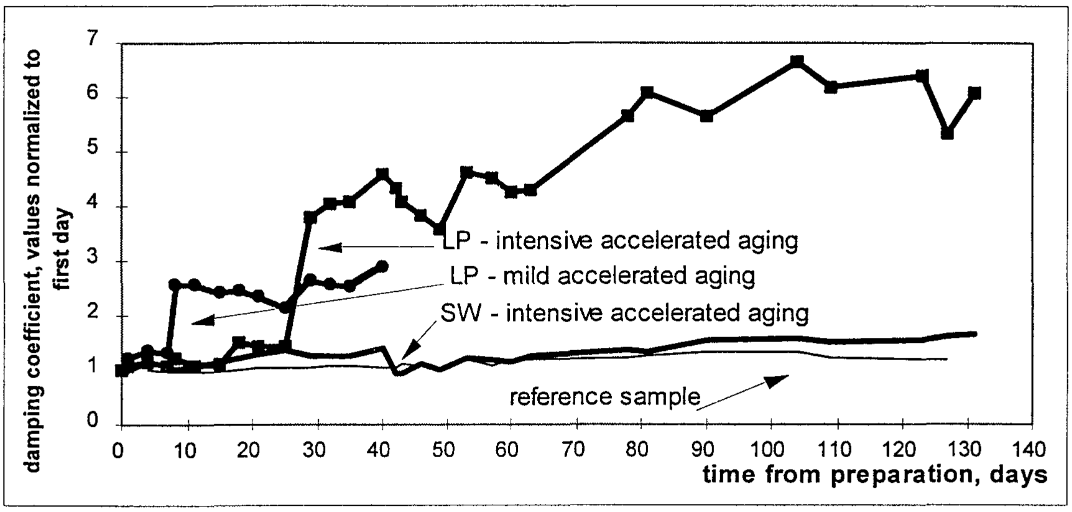


Figure 3
Three Types of Aging of LP
(Cylindrical Samples)

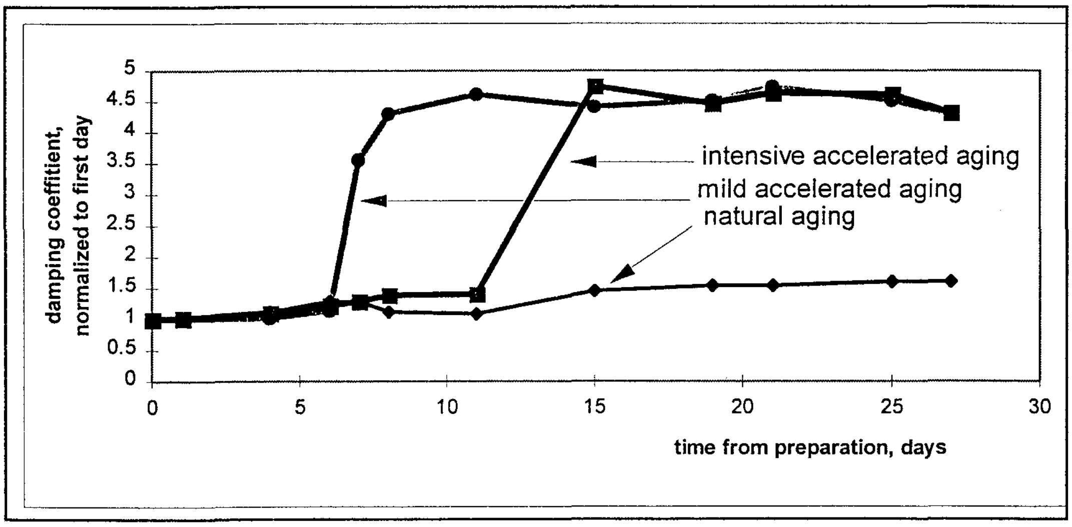
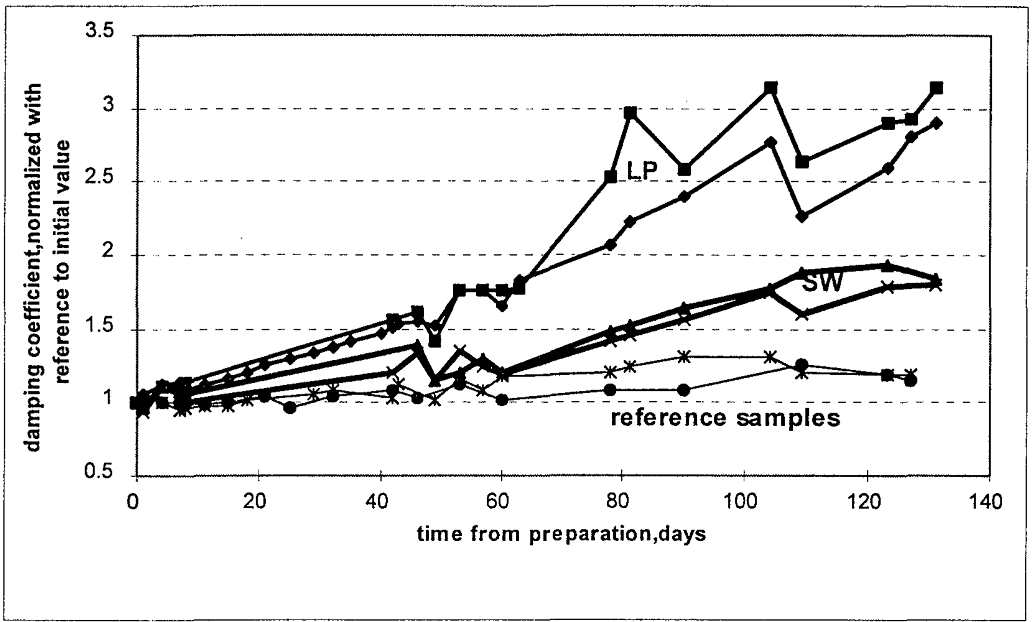


Figure 4
Natural Aging of LP and SW
(Flat Samples)



3.2. Comparison between DT and NDT

3.2.1. LP Specimen
The average shear strength tests results and the non destructive damping coefficients are compared Table 2:

Table 2
Comparison Between Average DT and NDT Results
Flat Samples - LP

aging conditions	shear strength average absolute values psi	shear strength, average values normalized to new samples	damping coefficients, average values normalized to new samples
new samples	1778±120	-	1
natural aging, 42 days	1778±200	1.4	1.5
mild accelerated aging	900±20	2.0	1.9
intensive accelerated aging	687±170	2.6	2.9

3.2.2. SW Specimen
The accumulated statistics of the destructive tests of was less extensive than the LP one, therefore the comparison will be more qualitative.

a. The natural aging of SW, according to the destructive tests was much slower than that of LP. This is comparable to the NDT results (about 3 times slower, as shown in Fig. 3).
b. The accelerated aging caused, according to the destructive tests results, a slight increase in strength (instead of the drastic decrease in the LP samples). This is explained by additional curing occurring in the glue as a result of the heat treatment. This again fits the NDT tests, where the accelerated aging causes a slight decrease in the damping coefficient (Fig. 2).

4.CONCLUSIONS

The possibility of non-destructive periodic monitoring of the aging of glue in adhesive joints and in bonded devices, using vibration analysis was investigated.
a. Two kinds of glues were investigated: fast aging - LP, and slowly aging - SW.
b. The samples were exposed to three kinds of aging: natural, intensively accelerated and mildly accelerated.
c. The damping coefficients of all samples were monitored frequently, before and after the accelerated aging and also during the natural aging.
d. It was shown that there is a very good correlation between damping coefficients of the samples (NDT) and the accumulated information of the glue strength, based on shear tests (DT). The strength decrease found in the destructive tests is mirrored by increase of the damping coefficient, and vise versa.

5. REFERENCES

1. A. Vary, "Overview of Applications and Potentials of Acousto-Ultrasonics", 2nd Int. Conf. On Acousto-Ultrasonics, Atlanta, GA, June 24-25, pp. 5-6,(1993)..
2. S. Timoshenko & D.H. Young "Vibration Problems in Engineering", D.Van Nostrand Co., Princeton,NJ,(1955). Chapter V.
3. G Canella & M Taddei," Correlation Between Ultrasonic Attenuation and Fracture Toughness of Steels", Conf. of Nondestructive Characterization of Materials, Montreal, pp 261-9,(1987).
4. V.S. Postinkov, "Internal Friction in Metals", Internal Friction in Metals and Alloys, ed. V.S.Postinkov, F.N.Tavadze, L.K.Gordienko, Consultant Bureau, NY, pp. 1-13, (1967)
5. G Canella & M. Taddei, "Correlation Between Ultrasonic Attenuation and Fracture Toughness of Steels",Conf. of Nondestructive Characterization of Materials, Montreal, pp 261-9,(1987).
6. R.W.Howard, "Vibration Damping Approach to Corrosive Fatigue Studies in Type 403 Stainless Steel", Stainless Steel Conference, Goteborg, Sweden, 3-4 Dec. pp.527-32,(1984).
7. F. Ali and C. Semler, "Prediction of Refractory Strength Using Nondestructive Sonic Measurements", Am. Ceram. Soc. Bull 64, [12] 1555-58 (1985).
8. R.A. Bemis, K. Shiloh and W.A. Ellingson, "Nondestructive Evaluation of Thermally Shocked SiC by IAR", The 40th Gas Turbine & Aeroengine Congress, Houston,Tx. June 5-8, (1995).
9. T.Ono et al,"Internal Friction,Crack Length of Fracture Origin and Fracture Surface Energy in Alumuna-Zirconia Composites", J. Of Mat. Sci 24,pp1974-8,(1989)
10. J.E. Grady and B.A. Learch, "Effect of Heat Treatment on Stiffness and Damping of SiC/Ti-15-3",NASA Technical Memorandum 105564.
11. B. Tang, C.T.Liu and E.G. Heneke II,. "Acousto-Ultrasonic Technique Applied to Filled-Polymer Damage Assessment", Journal of Spacecraft and Rockets, Vol. 32, No 5, pp. 866-9 (1995).
12. C.L. Rohn, "Dynamic Mechanical Measurement Yields a Better HDPE Fuel Tank", Modern Plastics, pp. 129-32,October(1989)
13. A.A Higazy et al., "Ultrasonic Characterization, Hardness and Dielectric Properties After γ-Irradiated Polyvinil Chloride", Materials Letters 20 pp. 237-44 (1994)
14. C.M. Su and H.G. Bohn,"Interface Constraint on Grain Boundary Relaxation in Thin Al and Al-Alloy Films on Si-Substrates", Mat.Sci. Forum Vol. 119-121 pp. 285-90 (1993).
15. K. Shiloh and W.A. Ellingson, "Non Destructive Evaluation Of Nickalon/Sic Composites By Vibration Analysis", 14th WCNDT Conf., Dec. 8-13, New Delhi, (To be Published), (1996).
16. H.L.M.Dos Reis and H.E. Krauz,"Nondestructive Evaluation of Adhesive Bond Strength, Using Stress Wave Factor Techique", Journal of Acoustic Emission, Vol 5, No. 4 , pp. 144-7 (1986).
17. A.A. Khalil and A.N. Kagho, "Non-Destructive Testing of Adhesively Bonded Joints Using Vibrational Analysis, Int. J. Adhesion and Adhesives, Vol. 11, No. 2 pp.191-27 (1991).