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**STUDIES OF ENCAPSULATION MATERIALS
FOR TERRESTRIAL PHOTOVOLTAIC ARRAYS**

Fourth Quarterly Progress Report, June 16, 1976—September 15, 1976

September 28, 1976

Work Performed Under Contract No. NAS-7-100-954328

**Battelle Columbus Laboratories
Columbus, Ohio**

MASTER
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
Division of Solar Energy

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FOURTH QUARTERLY PROGRESS REPORT
Covering the Period June 16, 1976, to September 15, 1976

on

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FOR TERRESTRIAL PHOTOVOLTAIC ARRAYS**

JPL Contract No. 954328

Encapsulation Task
Low-Cost Silicon Solar Array Project

to

JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY

This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, under NASA Contract NAS7-100 for the U.S. Energy Research and Development Administration, Division of Solar Energy.

The JPL Low-Cost Silicon Solar Array Project is funded by ERDA and forms part of the ERDA Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays.

September 28, 1976

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ABSTRACT

This Fourth Quarterly Progress Report on JPL Contract No. 954328 covers work conducted in the period from June 16, 1976, through September 15, 1976. The research is part of a five-task development project, the Low-Cost Silicon Solar Array Project (LSSA Project), being managed by the Jet Propulsion Laboratory for the Energy Research and Development Administration, Division of Solar Energy. The Encapsulation Task (Task III) of this project includes four studies assigned to Battelle-Columbus. The status of the studies active during this quarter is summarized in this report.

Study 1 of this contract is entitled "Evaluation of World Experience and Properties of Materials for Encapsulation of Terrestrial Photovoltaic Arrays". The objectives of this study are to provide a summary of world experience related to polymeric and glass protective systems for terrestrial arrays and to recommend candidate encapsulation materials and processes which can be expected to provide a service life of 20 years. The reviews and analyses of the information collected in this study were completed this quarter. A draft of the final report has been submitted to JPL for review, and it is expected that the report will be issued early in the next quarter. Contractually, no quarterly report on this study is required at this time; however, statements of the objectives and status are included in the report in order to identify the scope of the total contract.

The objective of Study 2, "Definition of Encapsulant Service Environments and Test Conditions", is to determine the frequency and duration of climatic and other environmental conditions for a 20-year array lifetime and identify conditions that can be used in testing candidate encapsulant materials for a service life of 20 years in terrestrial environments. Study 2 also is in its final stages. A draft of the final report has been prepared and is being reviewed by JPL. While no technical discussion of Study 2 is to be included in this report, a brief statement of scope is included.

Study 3 is concerned with evaluation of the properties of candidate encapsulation materials which have been identified based on the results of Study 1. The initial portion of the effort of Study 3 is directed toward identifying and evaluating relevant measurement methods and techniques for determining properties of materials as described in test standards and other similar sources. The major portion of the study will be devoted to the evaluation of important properties of potential encapsulation-system materials under selected conditions. This effort is now planned to include determining degradation-rate data on properties of selected materials and materials interfaces for use in the development of aging tests in Study 4. This quarter, the planning of the experimental phase of the study was emphasized. Material interfaces identified in Study 1 as the most important sources of failure for presently produced photovoltaic modules will be the subject of the initial experimental efforts. These interfaces are identified in this report along with initially selected materials which have been ordered. Experimental work has been initiated on the materials received to date.

Most of this report describes the work being conducted on Study 4, "Development of Accelerated and Abbreviated Testing Methods for Predicting Performance of Encapsulation Materials Over a 20-Year Lifetime". The outputs of the study are to be (1) the specification of a methodology for developing the experimental designs for aging tests, and (2) the development of the experimental designs for aging tests for selected materials and material interfaces. Pursuant to these goals, the aging behavior of polymeric materials has been reviewed so as to identify possible failure mechanisms and appropriate choices of characteristics to be measured and stresses to be used in accelerated/abbreviated tests. A similar review has been made for glasses. The results of these reviews are discussed in previous quarterly reports (ERDA/JPL-954328-76/1 and -76/2), along with aging data and data analysis methods (models) which have been used in previous investigations. This quarter, the thrust was on the further development of the methodology for aging tests. Specifically, methods of discriminating among possible mathematical models which fit early aging data were treated. Several methods are discussed in this report, with emphasis on predicting future results from historical data.

STUDY 1. EVALUATION OF WORLD EXPERIENCE AND PROPERTIES OF MATERIALS FOR ENCAPSULATION OF TERRESTRIAL PHOTOVOLTAIC ARRAYS

F. A. Sliemers, C. W. Kistler, Jr., R. E. Sharpe,
G. B. Gaines, R. D. Igou, and D. C. Carmichael

INTRODUCTION

Achievement of acceptable low-cost, silicon photovoltaic arrays with a life of 20 years requires the selection and/or development of encapsulation materials and processes that are compatible with the photovoltaic array and that afford the required encapsulant life under terrestrial climatic conditions. The final process for producing the active silicon arrays and the engineering design of the array are yet to be evolved. The output of Study 1 is formulated to contribute to design evolution and materials and process selections through a review and analysis of world experience in the area of materials for encapsulation/protection systems for solar cells and related devices. Out of the analysis will come a list of the most promising candidate materials and processes for encapsulating terrestrial photovoltaic arrays and their properties. Such criteria as cost, known and potentially achievable properties, availability, and ease of automation will govern the selection of candidate materials and processes. Data on world experience and materials properties have been drawn from published and unpublished literature, personal contacts, and site visits.

STATUS OF STUDY 1

The collection and analysis of the information on the world experience with photovoltaic modules in terrestrial environments were completed this quarter, and candidate materials have been identified for each of the various functional components of the encapsulation system. A draft of the final report has been submitted to JPL for review. In addition to the major text of the report, a compilation of the known relevant properties of recommended materials was prepared. This compilation will constitute a major appendix to the report. The complete final report is expected to be issued early next quarter.

In accordance with the contract, no quarterly report is to be prepared at this time; this brief section has been included to indicate the scope of the total investigation.

STUDY 2. DEFINITION OF ENCAPSULANT SERVICE ENVIRONMENTS AND TEST CONDITIONS

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R. C. Behn, P. R. Sticksel, S. G. Talbert,
R. B. Bennett, G. B. Gaines, and D. C. Carmichael

INTRODUCTION

The objectives of Study 2 are to develop the statistical methodology, determine the frequency and duration of climatic and other environmental conditions for a 20-year array lifetime, and identify corresponding conditions that can be used in testing candidate encapsulation materials for a service life of 20 years in terrestrial environments.

The methodology for accomplishing these objectives involves several stages. Climatic data (temperature, relative humidity, wind speed, etc.), insolation data, air-pollution data, and data related to miscellaneous hazards are being gathered for the following geographic locations: Albuquerque, New Mexico; Bismarck, North Dakota; Boston, Massachusetts; Brownsville, Texas; Cleveland, Ohio; Fairbanks, Alaska; Los Angeles, California; Miami, Florida; and Phoenix, Arizona. These data are being statistically analyzed to quantify past environmental conditions and to make 20-year forecasts. Special attention is being focused on the frequency and duration of combinations of environmental conditions that are likely to initiate and/or promote material degradation. These projected conditions furnish the basis for selecting environmental factors and levels that can be used in testing candidate encapsulation materials.

STATUS OF STUDY 2

A draft of the final report on Study 2 has been prepared and is being reviewed by JPL. It is planned that final modifications of the report will be carried out during the next quarter. Although no quarterly report on Study 2 is to be provided at this time, this brief section has been included to state the scope and status of the study.

STUDY 3. EVALUATION OF TEST METHODS AND PROPERTIES OF CANDIDATE ENCAPSULATION MATERIALS

F. A. Sliemers, M. C. Brockway, R. B. Bennett,
G. B. Gaines, and D. C. Carmichael

INTRODUCTION

To date, the experience with encapsulation of photovoltaic solar arrays for terrestrial applications has been rather limited. Moreover, the encapsulant materials used so far have been employed based on criteria not entirely appropriate for the goals of the LSSA project, especially cost and ease of processing using automated methods. There is a considerable need, therefore, for important information on the properties and aging behavior of materials which are candidates for encapsulating low-cost photovoltaic arrays and are prepared by methods suitable for automated processing. Also, because of the newness and some unique requirements of the terrestrial photovoltaic arrays, standard methods of measuring the properties of candidate encapsulation may not be useable.

The objectives of this study, therefore, are to

- (1) Review test standards for appropriateness to the requirements of the candidate materials and processes,
- (2) Measure properties of selected materials under conditions which will make feasible a choice among candidate materials and processes for use in future encapsulation systems.

As part of the second area, materials and measurement techniques will also be experimentally evaluated to obtain information for use in development of aging tests in Study 4.

STATUS OF STUDY 3

Efforts in Study 3 this quarter have been directed primarily to planning and initiating the experimental work to meet Objective 2, above. These planning efforts have resulted in a suggested change in technical emphasis from that originally conceived with regard to some aspects of the study. The initial emphasis is being placed on investigation of the major problem areas which have been identified in encapsulation systems, particularly on the properties and characteristics of materials interfaces.

Results of the analysis of the world experience with existing modules (Study 1) indicate that major problems with material interfaces have overshadowed changes in bulk properties of materials. The experience shows that

many module failures have occurred at the seals attaching the bottom cover to top cover and at those interfaces forming the seal of the electrical leads to the protective covers. Further encapsulation system developments depend significantly on finding solutions to the seal problems. Thus, a substantial portion of the initial Study 3 efforts is being directed toward material properties involved in these problems, including the following:

- (1) Thermal coefficient of expansion
- (2) Mechanical moduli
- (3) Moisture permeance
- (4) Bond strength (adhesive, metal, cover material)
- (5) Effects of processing on these properties
- (6) Effects of environmental parameters on these properties.

The environmental parameters which will be of first concern are ultraviolet radiation, temperature (including temperature cycling), and the relative humidity. Clearly, other environmental parameters can affect the seal performance, but those mentioned expectedly warrant major attention. Other parameters might be added later once a first-order selection of materials and processes is effected.

Specific materials which have been selected to date for initial experiments involving problem interfaces are identified in Table 1. All of these materials have been ordered and some have been received. Exploratory experiments have been initiated which are concerned with techniques and methods for determining the desired property measurements, and initial results will be available next quarter.

TABLE 1. MATERIALS SELECTED FOR USE IN INITIAL
EVALUATIONS OF ADHESIVE/SEALANT
SUBSYSTEMS

Type of Component	Material	Supplier
Adhesive	Acryloid B-7 Scotch-Weld 2216 B/A Scotch-Weld RTC-3810	Rohm & Haas Company 3 M Company 3 M Company
Adhesive/Sealants	RTV 108 RTV 118	General Electric Co. Ditto
Film	Lexan (5 mil) "Teflon" FEP (5 mil) Korad A Tedlar Mylar	Cadillac Plastics K-Plastics Excel Corporation E. I. duPont de Nemours Ditto
Sealants	MONO Tremco 440 Lasto-Meric Dimeric Weatherban 1103	Tremco Manufacturing Co. Ditto " " 3 M Company
Sheet	Plexiglas G or K (1/8") Lexan 9030 (UV stabilized) (1/8")	Cadillac Plastics Ditto
Glass covers	Soda-Lime-Silica Borosilicate	
Leads	Copper Silver	

STUDY 4. DEVELOPMENT OF ACCELERATED AND ABBREVIATED TESTING METHODS FOR PREDICTING PERFORMANCE OF ENCAPSULATION MATERIALS OVER 20-YEAR LIFETIME

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D. M. Bigg, R. D. Igou, G. B. Gaines,
and D. C. Carmichael

INTRODUCTION

To be economically feasible, photovoltaic solar arrays should have a service life of at least 20 years. Silicon solar cells, if properly protected from the atmosphere, are expected with reasonable confidence to have such a lifetime. The success of the Low-Cost Silicon Solar Array Project depends substantially on identifying a low-cost encapsulation material system that will protect the solar cell for that period of time with little loss in electrical output. The success of an encapsulation material, therefore, depends on its ability to withstand 20 years of outdoor service in various environments. Since 20 years of service testing is obviously impractical, methods for short-term (abbreviated/accelerated) aging tests must be developed from which service life can be predicted with acceptable confidence.

Although a specific accelerated/abbreviated test for the ultimate encapsulant system cannot be verified until the system is defined, the objectives of this study are to accomplish the following significant steps in establishing such a test.

- (1) Developing a suitable methodology for designing the statistical and experimental techniques for such a test
- (2) Using this methodology to specify detailed test designs for aging tests on specific materials and material interfaces which are representative of candidate encapsulation systems.

Subsequent work would involve conducting the tests and analyzing the data in accordance with the test design ("test plan") to verify the methodology and techniques and to obtain high-confidence predictions of service life.

The methodology to be developed will describe all important factors that must be considered in the detailed test design and describe suitable statistical and experimental techniques for treating the important factors including statistical data analysis and prediction methods.

The test designs will specifically describe the total elements of a test plan, including material selection, specimen design and number, environmental parameters and operating conditions, properties to be measured, instrumentation to be utilized, and data analysis methods. Inputs regarding the selection of materials and interfaces to be considered will come from Study 1. Study 2 will furnish environmental descriptions of various geographical sites of interest.

Also, as indicated previously, Study 3 will be directed, in part, to furnishing experimental data on selected materials, materials interfaces, and measurement techniques to aid in formulating the methodology and test designs and in validating the methodology in Study 4. This information is necessary because the initial efforts in Study 4 indicated a lack of data obtained under acceptable conditions and on relevant materials for use in this study.

TECHNICAL DISCUSSION OF RESULTS THIS QUARTER

The major effort this quarter on Study 4 concentrated on the development of one of the major facets of the methodology for developing experimental designs for aging tests, viz model discrimination during the acquisition of aging data. It is to be recalled that the identification and discussion of possible aging models have been contained in previous reports, as have been the aging behaviors of glasses and polymeric materials in various environments. The discussion that follows describes the effort this quarter on model discrimination. Subsequent work will be concerned with the methodology for determining the precision in property/performance measurements which will be required.

Designing an accelerated or abbreviated life test involves many factors such as determination of sample size, setting of stress levels for accelerated degradation, determination of test accuracy, and other considerations. The major difficulty with such tests lies in the necessity for extrapolating a fitted equation to unmeasured conditions to predict life at conditions of actual use. For abbreviated tests, the extrapolation is usually over time whereas for accelerated tests, extrapolation is generally over one or more stresses. Either way, extrapolation is the most critical aspect of life testing methodology and is always done with considerable reservation.

A sensible approach to the extrapolation problem was proposed by McCallum, Thomas, and Waite⁽²⁾. They proposed that instead of waiting until all data are collected and then performing one grand extrapolation, it would be better to practice extrapolating and predicting during the course of the experiment. In this way, the method of extrapolating should improve as the experiment progresses.

Extrapolation is most meaningful when the fitted equation is derived from known physical laws operative in the degradation process. Such equations are generally termed mechanistic as opposed to empirical models which have no such basis. Mechanistic models have been described as empirical models with an infinite domain implying validity over all levels of the independent variable. As Box and Hill⁽¹⁾ put it, "In practice such a (true mathematical) model accounting for the phenomenon under study in terms of its exact mechanism is seldom obtainable. Many examples occur, however, where the main characteristics of a mechanism can be elucidated and a close mechanistically based mathematical representation is possible... The chances of meaningful extrapolation are very

much greater with such models." As this implies, it is not particularly useful or accurate to dichotomize equations into mechanistic and empirical categories. It is more useful to consider a continuous spectrum of models from the crudest empirical model to a theoretical model which takes into account all phenomena acting on the system. In one sense, the domain (i. e., range of validity) of a model is the criterion which determines how mechanistic it is. The larger the domain the more mechanistic is the model, and, conversely, the closer a model approaches the mechanistic end of the spectrum the more accurate extrapolations from that model become. With the above in mind, the extrapolation problem can be considered a model discrimination problem where the goal is to select from a set of candidate models, one which appears most mechanistic. Several criteria will be discussed for achieving this goal including a formalization of the idea of using a predictive track record as a measure of extrapolatability.

Selection and Evaluation of Models

Clearly, the first step in applying the model discrimination methodology is to formulate a set of candidate models. For extrapolations over stress in accelerated testing, one would, of course, want to be sure that the set included models which have been used successfully in the past. Some of these are as follows:

Arrhenius Equation

$$k = \exp \left(A - \frac{B}{T} \right) , \quad (1)$$

Generalized Eyring Equation

$$k = AT \left[\exp \left(\frac{-B}{kT} \right) \right] \left[\exp \left(CV + \frac{DV}{kT} \right) \right] , \quad (2)$$

Inverse Power Law

$$\ln k = \frac{A}{VB} , \quad (3)$$

where T is temperature, V is a generalized stress variable (a nonthermal stress for the Eyring equation), k is a rate-of-degradation parameter and all other terms are fitted constants.

In fitting property, P , versus time, t , data, some candidate models might be kinetic models for various orders. For example, for first-order degradation, one has

$$P_t = P_0 \exp (-kt) , \quad (4)$$

where P is property level at time t , P_0 is the initial property level, and k is the first-order rate constant. Of course, here again, empirical and mechanistic models which have been applied in the literature should be included, examples of which are (3, 4, 5)

$$P = P_0 + bt \quad (5)$$

$$P = P_0 + k \log t \quad (6)$$

$$P = P_0 + k_1 \log t + k_2 \log^2 t \quad (7)$$

$$P = b_1 \exp \left[- \left(\frac{t+b_2}{b_3} \right)^{b_4} \right] + b_5 \quad (8)$$

where P_0 and P are property levels at time zero and t , respectively, and all k 's and b 's are fitted constants. Aside from candidates from the literature, low-order polynomials should be evaluated as yardsticks by which candidate mechanistic models can be evaluated. Various metrics should be evaluated for the polynomials by applying transformations to independent and/or dependent variables. A useful transformation family for this purpose was proposed by Box and Cox⁽⁶⁾ as follows:

$$T = \begin{cases} (Y + \lambda_2)^{\lambda_1} & (\lambda_1 \neq 0) \\ \ln (Y + \lambda_2) & (\lambda_1 = 0, Y > \lambda_2) \end{cases} \quad (9)$$

where Y represents the dependent or independent variable, T is the corresponding transformed variable, and λ_1 and λ_2 are variable parameters. Variable transformation often results in better fitting and/or more meaningful models.⁽⁷⁾ Furthermore, when two or more stresses are involved, variable transformation can result in the elimination of interactions between stresses. Box and Cox⁽⁶⁾ point out that such linearization often tends to make extrapolation more valid than for the unlinearized model. The same argument applies to second-order effects (i. e., V^2) in a single stress where a curvilinear relationship is transformed into a linear relationship which is more amenable to extrapolation.

Once a set of models is selected and fitted to the data, standard statistical tests can be employed to eliminate the poorest models.⁽⁸⁾ For example, for linear regression models, a nonsignificant regression F ratio, a significant lack of fit F ratio, and/or an abnormal pattern in the residuals would disqualify the poorest models. Discriminating among the remaining models is more difficult and can be accomplished by establishing a prediction track record as discussed previously. With the exception of the prediction track record method and residual analysis, screening models for nonlinear regression models is done in a different way as discussed in Reference (8).

Study With Artificial Data

To illustrate and evaluate the model discrimination methodology described above, artificial data were generated from the "true" equation

$$P = 850 - 40 t \quad , \quad (10)$$

where P is property level at time, t , in years. Property levels, P_i , were calculated for various times and duplicates were taken for each value of t up to 2 years. Normally distributed error with mean zero and variance, $\sigma^2 = 1$ was added to each P_i . The resultant "data" are shown in Table 2. This model was chosen to illustrate the problem of predicting 20-year life from 2 years' data. The results, however, are applicable to other extrapolation problems, including extrapolation of property level or degradation rate over one or more stresses. The candidate models are listed in Table 3 and are simply transformed versions of Equation (10). As can be seen, Model 20 is the "true" model.

All of the equation forms shown in Table 3 were fitted to data shown in Table 2. Five regression analyses were run for each model for data covering 0.4 year ($N = 8$ data points), 0.8 year ($N = 16$), 1.2 years ($N = 24$), 1.6 years ($N = 32$), and 2 years ($N = 40$), respectively. After each set of 20 regression analyses for each N , the fitted equations were judged according to two criteria: (1) F ratio for lack of fit and (2) nonrandom residual patterns. All models exhibited F ratios for regression significant at the 5 percent level of significance. The results for the first discrimination phase are shown in Table 4. The earliest eight data points covering a 0.4-year period resulted in no models being rejected. In other words, all 20 fitted models were consonant with the earliest eight data points. After 16 data points (0.8 year) had been collected, the procedure was repeated and resulted in elimination of Models 1 and 2. This procedure was repeated in 0.4-year increments up to 2 years and 40 data points. After 40 data points, all but four models had been rejected: Models 5, 10, 15, and 20. These gave 20-year predictions of 416, 228, 315, and 47, respectively, where the "true" 20-year value was 50. It is interesting and perhaps distressing that three equations which exhibited excellent fit to the data gave extremely poor predictions! At this point, if the poorest 20-year prediction were acceptable, no further discrimination would be necessary. Suppose, however, that further discrimination was desirable; then one must look at the predictive track records for the four remaining models.

The track records for the four remaining models are shown in Table 5. Here for each model a matrix was constructed. The row indices represent the time interval over which the data were collected. The column indices represent times for which predictions were made. The mean of the two data values for each prediction time are shown at the bottom of the table. The first entry in the prediction matrix for Model 5, i.e., 834.9, is then the predicted value of P at 0.4 year made from data up to 0.2 year. The number in row three, column four, i.e., 789.1, is the P predicted from data for time, $t = 1.6$ year from data covering 0.8 year, etc. The 20-year predictions for each time interval are shown in the column beside the matrix. Since the quantities of real interest are the differences between predicted values and actual data values (i.e., residuals), residual matrices were constructed for each matrix of Table 5 and are shown in Table 6. The residual matrices correspond to the prediction matrices. For example, the first element in the matrix for Model 5, i.e., -1.95, represents the difference between the mean of the two actual data values at $t = 0.4$, 832.95 and the corresponding predicted value, 834.9. The same correspondence applies to all other matrix entries.

TABLE 2. ARTIFICIAL 20-YEAR PROPERTY DATA

Time, years	Actual Property Level, P_i	Artificial Data, Actual Property Level + Error	
		$P_i + \epsilon$	$P_i + \epsilon$
0.1	846	846.6	845.2
0.2	842	843.3	841.1
0.3	838	838.9	838.0
0.4	834	833.0	832.9
0.5	830	830.1	830.6
0.6	826	824.9	825.9
0.7	822	823.5	822.4
0.8	818	819.6	816.8
0.9	814	814.1	815.7
1.0	810	809.6	808.2
1.1	806	805.7	806.4
1.2	802	804.3	802.2
1.3	798	797.3	797.9
1.4	794	795.4	793.5
1.5	790	790.2	788.1
1.6	786	785.5	785.2
1.7	782	783.4	782.3
1.8	778	777.4	776.6
1.9	774	775.6	773.6
2.0	770	770.0	768.7

TABLE 3. CANDIDATE MODELS

Model No.	Model
1	$1/P = b_0 + b_1/(t + 1)$
2	$= b_0 + b_1 \sqrt{t}$
3	$= b_0 + b_1 \ln(t + 0.5)$
4	$= b_0 + b_1/\sqrt{t + 1}$
5	$= b_0 + b_1 t$
6	$\sqrt{P} = b_0 + b_1/(t + 1)$
7	$= b_0 + b_1 \sqrt{t}$
8	$= b_0 + b_1 \ln(t + 0.5)$
9	$= b_0 + b_1/\sqrt{t + 1}$
10	$= b_0 + b_1 t$
11	$\ln P = b_0 + b_1/(t + 1)$
12	$= b_0 + b_1 \sqrt{t}$
13	$= b_0 + b_1 \ln(t + 0.5)$
14	$= b_0 + b_1/\sqrt{t + 1}$
15	$= b_0 + b_1 t$
16	$P = b_0 + b_1/(t + 1)$
17	$= b_0 + b_1 \sqrt{t}$
18	$= b_0 + b_1 \ln(t + 0.5)$
19	$= b_0 + b_1/\sqrt{t + 1}$
20	$= b_0 + b_1 t$

Note: b_0 = fitted intercept.
 b_1 = fitted slope.

TABLE 4. RESULTS OF MODEL DISCRIMINATION FOR VARIOUS
SUBSETS OF DATA COVERING A 2-YEAR PERIOD

No. of Data Points Time, years	8 0.4	16 0.8	24 1.2	32 1.6	40 2.0
Model					
1	X	*	*	**	**
2	X	X	*	**	**
3	X	X	*	**	**
4	X	X	*	**	**
5	X	X	X	X	X
6	X	X	**	**	**
7	X	X	**	**	**
8	X	X	**	**	**
9	X	X	**	**	**
10	X	X	X	X	X
11	X	X	**	**	**
12	X	*	**	**	**
13	X	X	**	**	**
14	X	X	**	**	**
15	X	X	X	X	X
16	X	X	**	**	**
17	X	X	**	**	**
18	X	X	**	**	**
19	X	X	**	**	**
20	X	X	X	X	X

Key: X - Model retained.

* - Model disqualified due to significant lack of fit F ratio.

** - Model disqualified due to abnormal residual pattern.

TABLE 5. PREDICTION MATRICES FOR MODELS 5, 10, 15, AND 20

		Times For Which Predictions Were Made, years					
		0.4	0.8	1.2	1.6	2.0	20
Model 20 ("True Model")	0.2	834.8	820.0	805.2	790.4	775.6	109.6
	0.4		816.445	779.405	782.36	765.325	-1.5
	0.8			802.416	786.61	770.806	59.6
	1.2				786.57	770.76	59.26
	1.6					769.80	46.4
Model 10	0.2	834.82	820.17	805.64	791.25	777.0	269
	0.4		816.60	799.88	783.33	766.96	209
	0.8			802.66	787.21	771.91	239
	1.2				786.95	771.57	237
	1.6					770.34	228
Model 15	0.2	834.77	820.265	806.0	792.0	778.2	472
	0.4		816.730	800.334	784.425	768.496	308
	0.8			802.89	787.786	772.964	329
	1.2				787.31	772.34	326
	1.6					770.863	318
Model 5	0.2	834.9	820.6	806.9	793.6	780.7	451
	0.4		817.0	801.2	786.0	771.3	419
	0.8			803.5	789.1	775.1	432
	1.2				787.9	773.7	427
	1.6					771.7	420
Mean of Two Duplicate Data Values		832.95	818.2	803.25	785.35	769.35	50 ^(a)

(a) True 20-year value with no error.

TABLE 6. RESIDUAL MATRICES CORRESPONDING TO
PREDICTION MATRICES IN TABLE 5

		Times For Which Predictions Were Made, years				
		0.4	0.8	1.2	1.6	2.0
Model 20 ("True Model")	0.2	-1.85	-1.8	-1.95	-5.05	-6.25
	0.4		1.755	3.845	2.99	4.025
	0.8			0.834	-1.26	-1.456
	1.2				-1.22	-1.41
	1.6					-0.451
Model 10		1.85	1.80	2.2	2.58	2.50 ← CSEP ^(a)
	0.2	-1.87	-1.97	-2.39	-5.90	-7.65
	0.4		1.6	3.37	2.02	2.39
	0.8			0.59	-1.86	-2.56
	1.2				-1.6	-2.22
Model 15	1.6					-0.99
		1.87	1.82	2.14	2.69	3.15 ← CSEP
	0.2	-1.82	-2.065	-2.75	-6.65	-8.85
	0.4		1.47	2.916	0.925	0.854
	0.8			0.36	-2.436	-3.614
Model 5	1.2				-1.96	-2.99
	1.6					-1.513
		1.82	1.80	2.08	2.84	3.50 ← CSEP
	0.2	-1.95	-2.4	-3.65	-8.25	-11.35
	0.4		1.2	2.05	-0.65	-1.95
	0.8			-0.25	-3.75	-5.75
	1.2				-2.55	-4.35
	1.6					-2.35
		1.95	1.92	2.18	3.43	4.53 ← CSEP

(a) Cumulative standard error of prediction.

Cumulative Standard Error of Prediction (CSEP)

At each measurement time - 0.4, 0.8, 1.2, 1.6, and 2.0 years - one can compute a cumulative standard error of prediction as follows:

$$\text{CSEP} = \left\{ \left[\sum_{i=1}^c (r_i)^2 \right] / c \right\}^{1/2}, \quad (11)$$

where c is the number of residuals calculated up to the most recent time for which residuals were calculated, and r_i is the i th residual. For example, for Model 5 at $t = 0.4$, one has $c = 1$, $r_1 = -1.95$, so

$$\text{CSEP} = [(-1.95)^2 / 1]^{1/2} = 1.95.$$

Likewise, at $t = 0.8$, one has $c = 3$, $r_1 = -1.95$, $r_2 = -2.4$, and $r_3 = 1.2$. Then

$$\text{CSEP} = \left[\frac{(-1.95)^2 + (-2.4)^2 + (1.2)^2}{3} \right]^{1/2} = 1.92.$$

The CSEP values are given below each residual matrix for each of the four models (Table 6). Plots of CSEP versus time are shown in Figure 1 where Model 17 was included to illustrate the poor predictive power of a noncandidate model discarded in the initial model screening. At the end of 1.6 years, Models 10, 15, and 20 remain candidates since they all are close together on the CSEP scale. At the end of the 2-year period, however, Model 20, the true model, emerged as the best predictive model.

Regression F Ratio as a Model Discrimination Statistic

When a regression equation is fitted to data, three criteria are most commonly applied to judge its adequacy: (1) regression F ratio, (2) lack of fit F ratio, and (3) examination of residuals (i. e., actual-predicted values). If the regression F ratio is significant at a preselected significance level, the lack of fit F ratio is not significant at a preselected significance level and the residuals do not exhibit abnormal behavior, it was concluded that the fitted equation is an adequate representation of the data. Put another way, one says that the data are consonant with the fitted equation. Notice that nothing is said about how useful the equation can be expected to be for extrapolation or to what degree the fitted equation is of correct functional form to be a mechanistic model. For example, suppose that two fitted equations exhibit acceptable residual behavior and nonsignificant lack of fit F ratio and significant regression F ratios of, say, 10 and 1000. Instinctively, one feels that the equation with an F ratio of 1000 is more likely to be the true model than the one with an F of 10. However, current statistical practice generally would not admit to such a conclusion. Recent work by Box and Wetz⁽⁹⁾, Draper and Smith⁽⁸⁾, Derringer⁽¹⁰⁾, and Suich and Derringer⁽¹¹⁾,

FIGURE 1. CUMULATIVE STANDARD ERROR OF PREDICTION VERSUS TIME FOR MODELS 5, 10, 15, 17, AND 20

however, indicate that the higher the regression F ratio, the better the prediction accuracy of the fitted equation. Put another way, the higher the regression F ratio, the "closer" the fitted equation approaches the "true" equation. Clearly then, when extrapolation is the goal, a large regression F ratio should give more assurance than a smaller but statistically significant F ratio. As an illustration of this, consider Table 7, where predictions from Models 17, 5, 10, 15, and 20 fitted to 1.6 years of data ($N = 32$) were made for 3 through 10 years. As can be seen, the models can be ranked in their order of increasing prediction accuracy as $17 < 5 < 15 < 10 < 20$, which is the same ranking as their respective regression F ratios, i. e., 920 (Model 17) < 7654 (Model 5) < 8889 (Model 15) < 9420 (Model 10) < 9847 (Model 20). This strongly suggests that the size of the regression F ratio is an indicator of predictive accuracy.

Regression F ratios for Models 1, 5, 10, 15, and 20 are given in Table 8 as a function of time and number of data points covering a 7.5-year period. Model 1 was representative of the poorest fitting equations eliminated in the first discrimination phase. After 2 years of data were accumulated, all five models exhibited very large F ratios for regression, Model 20 exhibiting the largest in agreement with the CSEP plots. Aside from the magnitude of the F ratio statistic discussed above, its rate of change with the number of data points is quite interesting. The F ratio for the true model increased continuously with N at a very high rate. For the other three models, the F value increased to a point and then began to decrease. This behavior is in agreement with statistical theory. For the linear case, the regression F ratio is equal to MSR/s^2 if the model is of correct functional form, where MSR is the mean square for regression and the residual mean square, s^2 , is an estimate of the true experimental error, σ^2 . The expected value of MSR is $\sigma^2 + b \sum (x_i - \bar{x})^2$, where b is the slope of the true linear relationships, and $\sum (x_i - \bar{x})^2$ is the sum of squared deviations of independent variable settings from their mean. As N increased, s^2 approached the constant, σ^2 , whereas MSR increased continuously as did the resulting F ratio. If, however, the model being fitted is not of correct functional form, the expected value of residual mean square is equal to $\sigma^2 + (\sum B_i^2)/(N-2)$, where B_i is a measure of the discrepancy between the fitted and the true model at each point. Instead of approaching σ^2 , in this case, the residual mean square increases continuously at a rate dependent upon the size of discrepancies, B_i . The regression F ratio then increases at a lower rate for an incorrect model and will generally peak and begin to decrease as the B_i becomes larger as data are collected for longer times.

The F-regression versus N values in Table 8 are plotted in Figure 2 for Models 1, 5, 10, 15, and 20. Model 1 was included as representative of the models rejected by conventional statistical measures. Following the curve for increasing N , the first model to be eliminated was Model 1 which peaked out at $N \cong 18$. Models 5, 15, and 10 were eliminated in that order so that eventually only Model 20, the true model, remained in the set.

In summary then, in the early stages of the model-discrimination process, conventional statistical tests were employed to weed out the poorest predictor equations leaving only those

TABLE 7. PREDICTIONS FOR YEARS 3 TO 10 FOR VARIOUS
MODELS FITTED TO DATA FOR 1.6 YEARS

Time, years	Predicted Value For The Property (P) For Given Model Numbers					True Value of P
	17	5	10	15	20	
3	760	736	732	734	730	730
4	743	704	694	699	689	690
5	727	675	658	665	649	650
6	713	648	622	633	609	610
7	700	161	587	603	569	570
8	688	600	554	574	529	530
9	677	579	521	546	489	490
10	666	559	490	520	448	450

TABLE 8. REGRESSION F RATIO VERSUS N AND TIME
FOR MODELS 5, 10, 15, AND 20

Time, years	Data Points, N	F Regression			
		Model 5	Model 10	Model 15	Model 20
0.2	4	8	8	8	8
0.4	8	187	189	189	190
0.8	16	1298	1291	1295	1286
1.2	24	3819	3887	3882	3872
1.6	32	7654	9423	8889	9847
2.0	40	13206	18338	16828	19304
3.0	45	16432	34618	29223	34766
4.0	50	14239	69597	42134	70690
5.0	55	10013	83341	34544	136930
7.5	60	4451	76393	19257	273551

(Models 5, 10, 15, 20) which were consonant with the data. These models were then assessed with plots of CSEP and regression F ratio versus N (or time). Both methods succeeded in selecting Model 20 as the "best" equation in roughly the same amount of time (≈ 2 years). The discrimination for F , however, does not show up well on the logarithmic scale used in Figure 2. Figure 3 shows plots of the true model data points without error (i. e., column P_i in Table 2) fitted to Models 5, 10, 15, and 20. The resulting equations were as follows:

$$P = 850 - 40 t \text{ (Model 20)} \quad (12)$$

$$\sqrt{P} = 29.16136 t \text{ (Model 15)} \quad (13)$$

$$\ln P = 6.74616 - 0.04953 t \text{ (Model 10)} \quad (14)$$

$$P^{-1} = 0.0011742 + 0.61358 \times 10^{-4} t \text{ (Model 5).} \quad (15)$$

As Figure 3 shows, there is very little difference among these four equations when plotted as P versus t in the first 2-year period. In view of this, it is quite encouraging that the correct model was isolated with the proposed methods.

Literature Example and Extrapolation Over Stresses

In a study by Welch⁽¹²⁾, service life of an epoxy resin was studied as a function of two stresses - humidity and temperature - at levels higher than use conditions. The actual data are given in Table 9 rearranged in real-time order to simulate the order in which data were obtained. Regression analyses were run for 8, 10, 12, 14, 15, 16, 17, 18, and 19 data points. Each regression analysis included all points available up to that particular time. The models to which each data set was fitted were as follows:

$$\ln t = b_0 + b_1 H + b_2 C \quad \text{Model A}$$

$$\ln t = b_0 + b_1 \ln H + b_2 C \quad \text{Model B}$$

$$\ln t = b_0 + b_1 H + b_2 / (C + 273) \quad \text{Model C}$$

$$\ln t = b_0 + b_1 \ln H + b_2 / (C + 273) \quad \text{Model D}$$

$$\ln t = b_0 + b_1 \ln H + b_{11} \ln^2 H +$$

$$b_2 / (C + 273) + b_{22} / (C + 273)^2 +$$

$$b_{12} \left[\frac{\ln H}{(C + 273)} \right] \quad \text{Model E}$$

Model D is derived from kinetic considerations including an Arrhenius relationship between rate of degradation and reciprocal temperature. This is the relationship which Welch used although he treated the data graphically in two phases instead of straightforward fitting of Model D. Model E is Model D with second-order terms added to account for any deviations from the assumed semimechanistic relationship. Models A, B, and C were modifications of Model D. The resultant plots for F regression and CSEP versus number of data points (i. e., time) are shown in Figures 4 and 5.

FIGURE 2. REGRESSION F RATIO VERSUS NUMBER OF DATA POINTS FOR MODELS 1, 5, 10, 15, AND 20

FIGURE 3. PLOT OF PROPERTY LEVEL (P) VERSUS TIME FOR MODELS 5, 10, 15, AND 20 FITTED TO EQ. (10) WITHOUT ERROR

TABLE 9. WELCH DATA

Temperature, C	[H ₂ O], moles/ liter x 10 ²	Time to Failure, hours
95	2.62	54
95	2.26	73
95	2.04	89
95	1.57	112
82	1.65	140
82	1.42	220
82	1.30	230
95	0.523	295
82	1.00	330
66	0.879	690
66	0.750	910
66	0.695	1080
66	0.575	1260
82	0.331	1460
49	0.416	2920
49	0.355	4240
66	0.167	4550
49	0.329	4880
49	0.299	5800

FIGURE 4. CUMULATIVE STANDARD ERROR OF PREDICTION VERSUS N FOR WELCH DATA AND MODELS A, B, C, D AND E

FIGURE 5. REGRESSION F RATIO VERSUS N FOR WELCH DATA AND MODELS A, B, C, D AND E

The F plot indicated that Models B, D, and E would be the best candidates for prediction purposes since F generally increased with N as opposed to the relatively flat behavior with Models A and C. The CSEP plot, however, eliminated Model E early in the course of data collection, and at the end of the program, only Models B and D remained candidates. Model D is the semimechanistic model used by Welch for prediction, whereas Model B is Model D with a linear term for temperature replacing reciprocal temperature. Ideally, additional experiments would have been run to discriminate between these two models. In the absence of additional data, a minimax procedure might be used, the prediction would be made from both models and the least favorable result accepted. In any case, the example is in general agreement with the findings from the simulated study with the exception of the plots for Model E. This model gave a high F value but showed poor predictability (high CSEP). The poor predictability is not unexpected since it is generally acknowledged that the higher the order of the polynomial, the poorer the extrapolation accuracy. It appears, therefore, that the F criterion is good for comparison of models of the same order whereas the CSEP procedure is independent of the order of the polynomial. Of course, further simulations would be required to verify this behavior.

Discussion

The proposed data analysis methodology consists of formulating a list of candidate models for property-versus-time data, degradation-versus-stress data, etc. Through various discrimination techniques, the number of viable models is narrowed down as more and more data are obtained. For all potentially viable models, an extrapolation is made over time, stress, or other variable. At any point in the model-discrimination procedure, if predicted values made from all remaining models are acceptable, a decision can be made based on this minimax criterion and further experimentation discontinued.

Of course, this methodology is based upon the assumption that the "true" model is in the set of candidates. Actually, the assumption is not as restrictive as it appears. In all life testing that includes an extrapolation step, a successful extrapolation depends, whether stated or not, upon the model being correct. The criterion used to judge this model adequacy is typically how well four or five data points fit a straight line, such as the Arrhenious relationship; a criterion which, this work has shown, is quite hazardous. At least, in the method proposed in this study, great care is taken to give the "true" model a chance to emerge. To make sure the "true" model is an element of the candidate set, the candidate set can be made infinite in a sense. For example, at each data-analysis juncture in the procedure, the models remaining in the candidate set can be put through transformations from the family of transformations shown in Equation (9) using dependent and/or independent variable(s). If an index of fit such as regression F ratio is plotted as a function of λ_1 and/or λ_2 , the resulting function

is continuous and can be maximized. At each data-analysis junction in the methodology, the F statistic (or other statistic indicator of model adequacy such as CSEP applied retroactively) can be maximized over a domain of λ_1 , λ_2 values. This is, in a sense, equivalent to looking at an infinite set of models each time the data are analyzed, and retaining the best one(s), thus maximizing the probability that the true model will be found.

Data Analysis When Degradation Mechanism Changes Over Time

A major concern with most accelerated life testing is how to extrapolate in instances where the degradation mechanism and the resultant rate of degradation change over time. If the rate of degradation changes discontinuously at some point in time for property-versus-time models or at some stress level for property-versus-stress models, an inaccurate extrapolation will result unless only data collected after the mechanism change are used. For example, in predicting a 20-year property level from 2 years of data, one has no hope of an accurate extrapolation if the degradation mechanism changes discontinuously after 2 years. From chemical kinetic considerations it is more reasonable to assume, however, that if the degradation rate changes as a function of time or stress level, it will do so in a continuous manner. This is still a problem when it has been decided a priori to use a particular model (such as the Arrhenius relationship) for extrapolation, since the extrapolation will be from a linear equation fitted to data generated by a nonlinear process. The many literature examples of data plotted as $\ln k$ versus $1/T$ which show a hint of curvature support the existence of this problem.

In the extrapolation methodology described in this report, a continuous degradation mechanism change with time or stress presents no barrier because the model would change continuously to reflect the change in mechanism. A weighted regression analysis which weighs data points inversely with their age would probably lead to even better results in such situations.

Alternative Procedure

Another procedure for model discrimination, although not employed in the present study, should be mentioned since it is receiving considerable attention in the statistical literature. The procedure was developed by Box and Hill and is based upon the concept of entropy, as defined by Shannon⁽¹³⁾, in relation to information theory. Shannon defined entropy S as

$$S = - \sum_{i=1}^k \pi_i \ln \pi_i ,$$

where π_i is the probability associated with a symbol i , in this instance a model i . Without repeating here the mathematical details, the resultant discrimination proceeds as follows. A set of candidate models ($M_1, M_2, \dots M_i, \dots M_k$) is proposed and each is assigned a prior probability π_i where, of course,

$\sum_{i=1}^k \pi_i = 1.0$. When no reason exists to favor any model(s) over the rest, equal

probabilities, $\frac{1}{k}$, are assigned to each model. After a few initial experiments, the probabilities for each model are updated. Then the experimentation and probability updating are continued until one model emerges as clearly the most probable. The example from Reference (1), shown in Table 10, illustrates the evolutionary process. Here n is the experiment number, x_1 and x_2 are independent variable settings, y is the response and the π_i 's are the model probabilities for four models. In this instance Model 2 evolved as the most probable. In a modification of this method, Hill, Hunter, and Wichern⁽¹⁴⁾ added to the entropy-based model discrimination a criterion of the optimal setting of the independent variables for the next experiment resulting in a joint experimental-design and model-discriminating methodology.

TABLE 10. EXAMPLE FROM BOX AND HILL AS DISCUSSED IN TEXT

n	x_1	x_2	y	π_1	π_2	π_3	π_4
0	(Prior Probabilities $\rightarrow$)			0.25	0.25	0.25	0.25
1	25	575	0.396				
2	25	475	0.723				
3	125	475	0.422				
4	125	575	0.130	0.0069	0.4290	0.5008	0.0633
5	125	600	0.098	0.0019	0.5602	0.4291	0.0088
6	125	600	0.056	0.0018	0.8639	0.1339	0.0004
7	50	450	0.797	0.0021	0.9736	0.0243	0.0000
8	100	600	0.033	0.0032	0.9956	0.0012	0.0000

Although these entropy-based methods are rather complex mathematically, they could be programmed on a computer for routine use given sufficient resources to do so. It is, of course, not known at this time whether they would be superior to the track-record or regression F-ratio-based methods. These entropy-based discrimination methods would be a potentially valuable subject for future work.

PLANS FOR NEXT QUARTER

During the next quarter, efforts toward both objectives of Study 4 will be pursued. With regard to the methodology portion, the effort will turn immediately toward the development of an analysis scheme to determine the precision requirements in property characteristics measurements to assure detection of changes within appropriate time periods and with statistical validity. Following this effort, the methodology for selecting stress levels (multiple levels), sample size for each stress level, and an appropriate test sequence will be emphasized.

With regard to the experimental design of aging tests (EDAT), attention will be directed toward selecting the materials and materials interfaces to be investigated, the properties to be measured, the instrumentation to be used, and the stress levels.

NEW TECHNOLOGY

No new technology items have been reported thus far in the investigation.

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