1995116600
N95- 23037

OUTGASSING CONSIDERATIONS FOR COMPOSITES IN SPACE APPLICATIONS

By

Petar Arsenovic and Huai-Pu Chu
Goddard Space Flight Center
Greenbelt, MD 20771

ABSTRACT

Composites have been increasingly used in the construction of spacecraft. However, unlike metals, composites must be used with particular discrection in space applications because of their outgassing properties. For example, the outgas materials may cause serious contamination problems and affect the performance of delicate instruments. This paper presents an overview of the testing procedure and acceptance criteria for outgassing selection of spacecraft materials. Since composites can contain and absorb moisture which will outgas in space as water vapor, the test results of moisture absorption and desorption of a composite material are discussed also.

INTRODUCTION

The design of modern spacecraft has posed evermore stringent demands on materials. Advanced composites with unique properties have shown great promise to meet such demands. For example, composites have been selected for spacecraft structures, optical benches and instruments due to their high modulus, high strength and dimensional stability. Numerous successful applications of composites in spacecraft have been summarized in Reference 1.

However, it is important to note that certain composite materials may be disqualified for space applications because of their outgassing characteristics even though they do have other desirable properties. Thermoset and thermoplastic materials tend to be outgassers, especially at increased temperatures or in vacuum. They may emit gases and water vapors which would deposit, for instance, on lenses, mirrors and other parts of optical instruments and advsersely affect their performance. Therefore, all composites should be tested for outgassing before they are selected for use in spacecraft in order to prevent contamination. This paper presents a general discussion on outgassing, and summarizes the results of a recent study on moisture absorption and desorption of a composite, which is closely associated with the outgassing problem.

PRECEDING PAGE BLANK NOT FILMED

OUTGASSING

Test Method

The ASTM Standard E595-90 (Ref. 2) is commonly used for conducting outgassing tests for space applications. Briefly, the test utilizes a "microvolatile condensable system" which mainly consists of isolated sample chambers and collector chambers. Samples are heated to 125 ^{o}C for 24 hours in a vacuum jar to accelerate the outgassing process. Also, testing the materials in vacuum is compatible with their use in space environment. The outgas products in each sample chamber travel through a hole to a corresponding collector chamber, wherein a portion of the outgas products will condense on a collector plate which is maintained at 25 ^{o}C. Test results are determined from the condensed materials and the total amount of outgas from the samples. After testing in vacuum, the samples may be kept in 50% relative humidity at 25 ^{o}C for 24 hours for an optional test to determine the amount of water reabsorbed by each sample. It is termed as water vapor regained (WVR) and expressed as a percentage of sample mass before the test. Because of the micro-quantities involved in the tests all procedures detailed in the ASTM Standard should be followed closely so as to obtain consistent and accurate results.

Acceptance Criteria

Referring to the above, the mass of condensate on the collector plate is calculated as a percentage of the mass of the original sample; and this is the collected volatile condensible material (CVCM). Also, the total mass of material outgassed from the sample is determined by measuring the sample before and after the test. The total mass loss (TML) due to outgassing is expressed as a percentage of the initial sample mass. In general, materials which have CVCM $\leq$ 0.10% and TML $\leq$ 1.00% as specified in ASTM E595-90 are acceptable for space applications.

Outgassing Data

Goddard Space Flight Center (GSFC) has extensive experience in the study of outgassing properties of materials for spacecraft applications. A wealth of GSFC test data has been made available to the space industry through a series of NASA publications over a period of some 20 years. The latest is NASA Reference Publication 1124, Revision 3, which includes GSFC outgassing data on many materials generated through July 1993 (Ref. 3). The data are also available through the Materials and Processing Technical Information Service (MAPTIS) data bank in Marshall Space flight Center, Huntsville, Al. There are other sources of outgassing data, but they may not always conform to ASTM Standard E595-90.

Materials Selection

Although the outgassing data from Reference 3 or other sources can greatly facilitate the selection of materials for use in spacecraft, such test data should still be examined for each application to make sure that they are suitable for the particular functions and design requirements of the spacecraft. For example, the data may become questionable if a composite material is to be used at a temperature below 25 °C, because the CVCM is determined from outgassing products that are condensable only at or above 25 °C in the ASTM Standard test.

Sometimes outgassing data from different sources on one composite material may disagree with each other although they all have used the same ASTM Standard test method. This is quite possible for a number of reasons. For example, if the samples were taken from different batches with some slight variations in the manufacturing process they could have different outgassing properties; and this could happen even though the variations were all within the producer's specifications. For this reason, after a composite is selected for space application, it is still necessary to perform outgassing test on each batch of the material for quality conrol purposes.

In case a selected composite has all the preferred properties for a particular application except for outgassing, a thermal-vacuum treatment may be used to remove its outgassing materials. Also, some modifications in materials processing, such as a suitable change in the cure cycle, may bring about enough improvement in the outgassing property. If no method could sufficiently reduce its outgassing the composite should be replaced by an alternate material, or it may be used with some shielding or venting devices to protect the instruments from outgassing contamination.

MOISTURE

Experimental Procedure

The material tested was T50/ERL1962 graphite - epoxy coupons. For moisture absorption, twelve samples were thoroughly baked out at 90 °C for 120 hours and then placed in different environments with respect to humidity in groups of three. The mass of each coupon was measured periodically over time at each humidity level with a high precision Ohaus analytical microbalance having a readability to 0.00001 grams. The coupons were exposed to 21%, 38%, 65%, and 100% relative humidity at 25 °C.

Moisture desorption testing was accomplished by first preconditioning a set of graphite - epoxy coupons at 80 ^{o}C in an environment of 100% humidity for a period of 670 hours to ensure that the specimens were fully saturated with water. Following this, a Cahn vacuum balance was used to record the mass loss over time of the samples under a vacuum of 10^{-5} Torr at temperatures of 40, 60, and 90 ^{o}C.

Results and Discussion

Moisture Absorption

Experimental data on moisture absorption are summarized in Figure 1. The vertical coordinate represents the average mass gains of specimans in three tests in each of the four differet relative humidity levels. The mass gain is defined as the measured increase in mass of a specimen during exposure to controlled humidity and expressed as a percentage of the initial mass of the dry specimen. Figure 1 shows that moisture absorption started with high rates which decreased quickly in low humidity and gradually in high humidity. Each curve appears to level off eventually toward a point of saturation which is dependent on the relative humidity.

It is interesting to note that the mass gain versus time curves become linear in log-log coordinates with approximately the same slope as shown in Figure 2. Therefore, an empirical equation can be established for the tested composite as

$$M = kt^{0.33} \qquad (1)$$

where M = mass gain, %
t = time, h

The quantity k is a function of relative humidity and is equal to the mass gain at unit time. Figure 3 shows that a plot of log H versus log k is linear. Thus,

$$k = (H/1238.40)^{0.85} \qquad (2)$$

where H is the relative humidity. After substitution into Equation 1, the overall moisture absorption behavoir can be described by

$$M = (H/1238.40)^{0.85}\ t^{0.33} \qquad (3)$$

This equation is simple and accurate, and represents the experimental data quite well, as seen in Figure 1.

Moisture Desorption

Test data for moisture desorption are presented in Figure 4. The vertical coordinate represents the mass loss which is expressed as a percentage of the initial mass of the specimens after preconditioning in 100% relative humidity. Figure 4 shows that moisture desorption started with high rates which decreased quickly at high temperature and gradually at low temperature. The curves appear to level off eventually to a point when the coupons would be completely dry. It is interesting to note that the maximum mass loss is about the same magnitude of the mass gain shown in Figure 1. This indicates that all the moisture absorbed in the composite could outgas as water vapor in vacuum.

The moisture desorption data can be analyzed by a diffusion model (Ref. 4) as follows:

$$m = m_t[1-\exp(-7.3(t/t_c)^{0.75})] \qquad (4)$$

Where
m = mass loss, %
m_t = total change in mass, %
t = time, h
t_c = characteristic time, h

The change in mass, i.e. mass loss, is related to the "characteristic" time, which is in turn a function of the diffusion parameters. Thus

$$t_c = x^2/(D_o\exp[-Q/RT]) \qquad (5)$$

Where
x = thickness of the material
D_o = diffusion constant frequency factor
Q = activation energy
R = gas constant
T = absolute temperature

Based on the test data, the activation energy associated with the diffusion process is determined as Q=8.5 KCal/Mol. The total change in mass m_t is a parameter which is determined as m_t = -1.2% by its best fit to the experimental data using Equation 4. The number m_t is given a negative sign to account for moisture desorption. A value of D_o = 0.85 in^2/hr is used. This value is also determined by the best fit to the data and is typical for an epoxy compound of this type (Ref. 5). The t_c values are calculated for the three test temperatures as follows:

T (^oK)	t_c (hours)
313	402
333	175
363	62

Figure 4 shows that there is a good agreement between Equation 4 and the experimental data.

SUMMARY

The testing procedure and acceptance criteria for outgassing selection of materials to be used in spacecraft has been reviewed. Outgassing testing should be conducted according to ASTM Standard E 595-90. In general, materials with CVCM $\leq$ 0.10 % and TML $\leq$ 1.00 % are acceptable for space applications.

Test data on a composite material T50/ERL1962 are presented over time at various relative humidity levels at room temperature for moisture absorption, and under vacuum at several temperatures for moisture desorption (outgassing). The data can be accurately represented by simple equations which are useful for materials characterization.

REFERENCES

1. V. P. McConnell, " Progress Report: Composites in Space", Advanced Composites, July/August 1991, p.26

2. ASTM Standard E 595-90, Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment, Annual Book of ASTM Standards, Vol. 15.03, American Society for Testing and Materials, 1992

3. W. A. Campbell, Jr., and J. J. Scialdone, "Outgassing Data for Selecting Spacecraft Materials", NASA Reference Publication 1124, Revision 3, September 1993

4. G. S. Springer, "Environmental Effects, Section 16 in Composite Design, 4th Edition, Edited by S. W. Tsai", Think Composites, 1988

5. C. Barrett, W. Nix, and A. Tetelman, "The Principles of Engineering Materials", Prentice Hall, 1973, p. 155

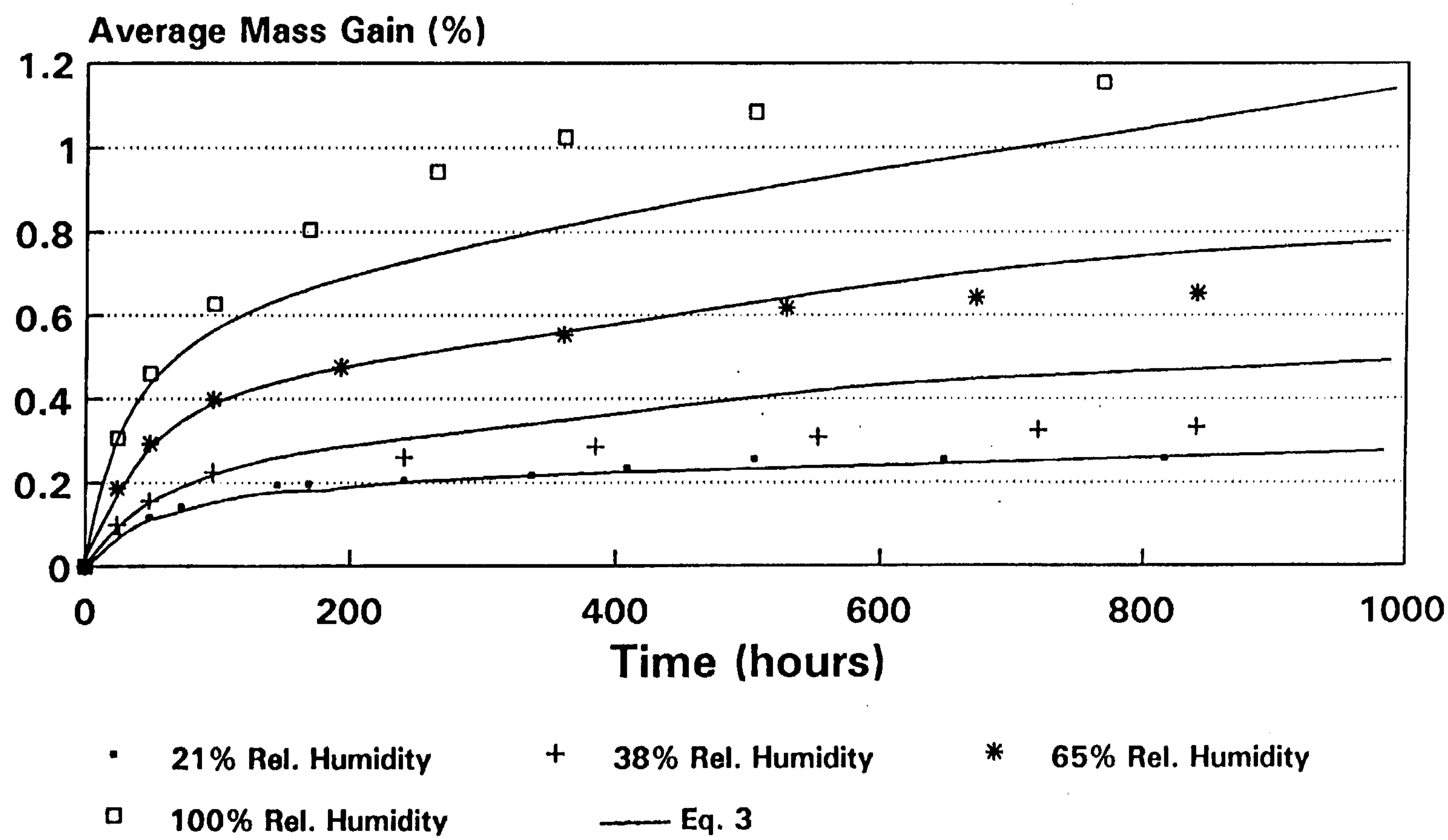

Fig. 1: Moisture absorption of T50/ERL1962 at four relative humidity levels

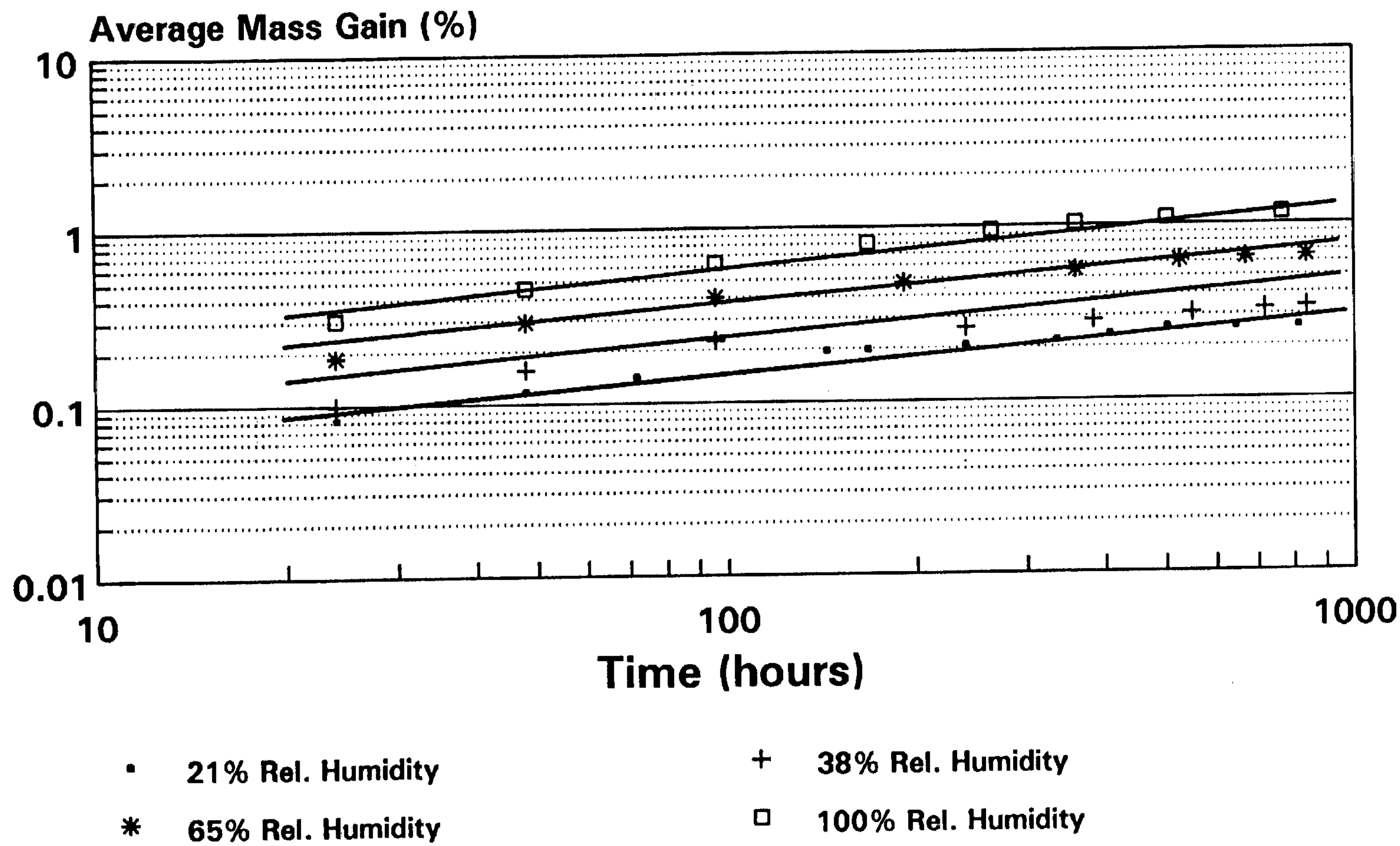

Fig. 2: Log-Log plot of moisture absorption data on T50/ERL1962

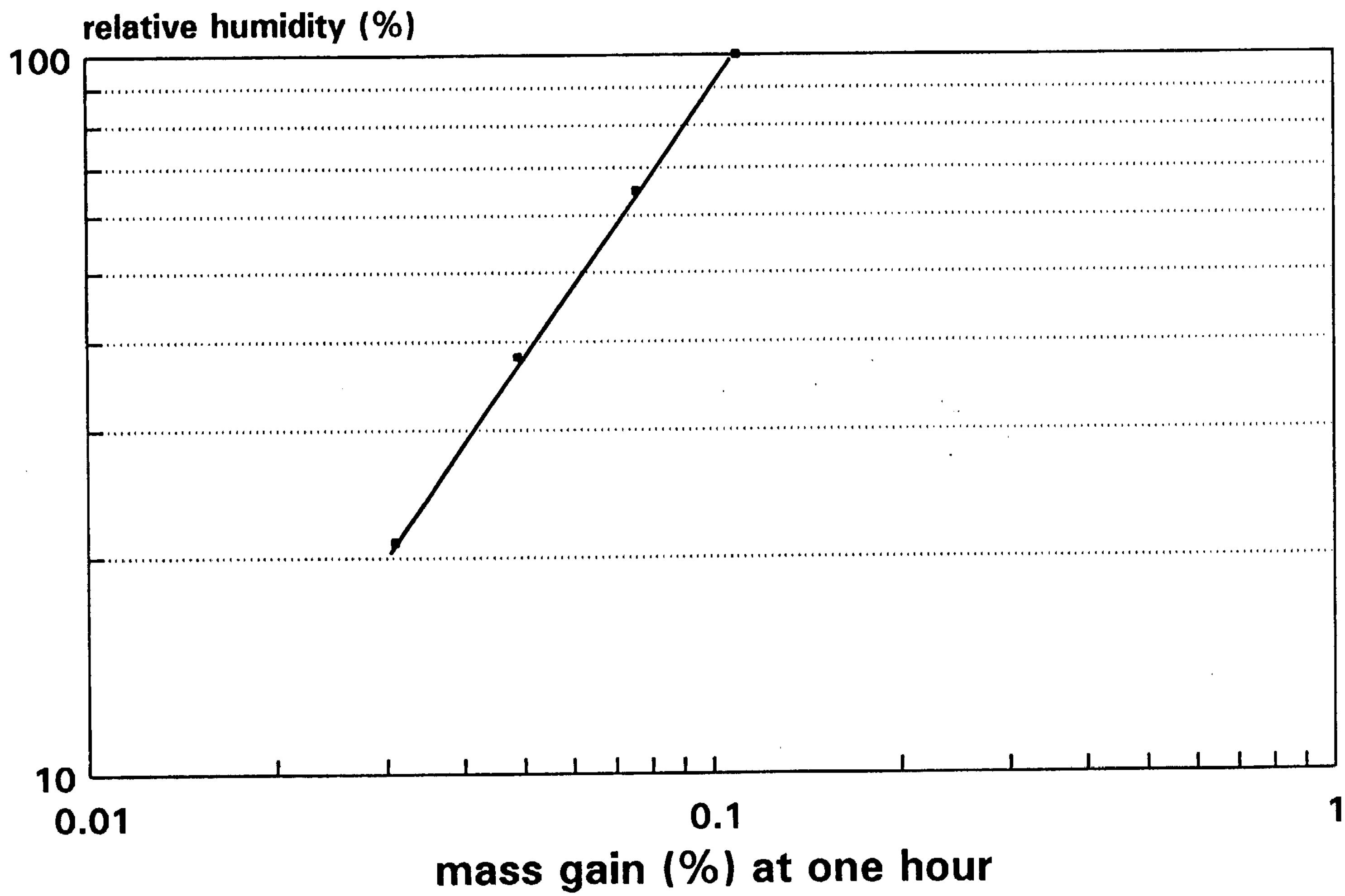

Fig. 3: Moisture absorption data on T50/ERL1962 at unit time

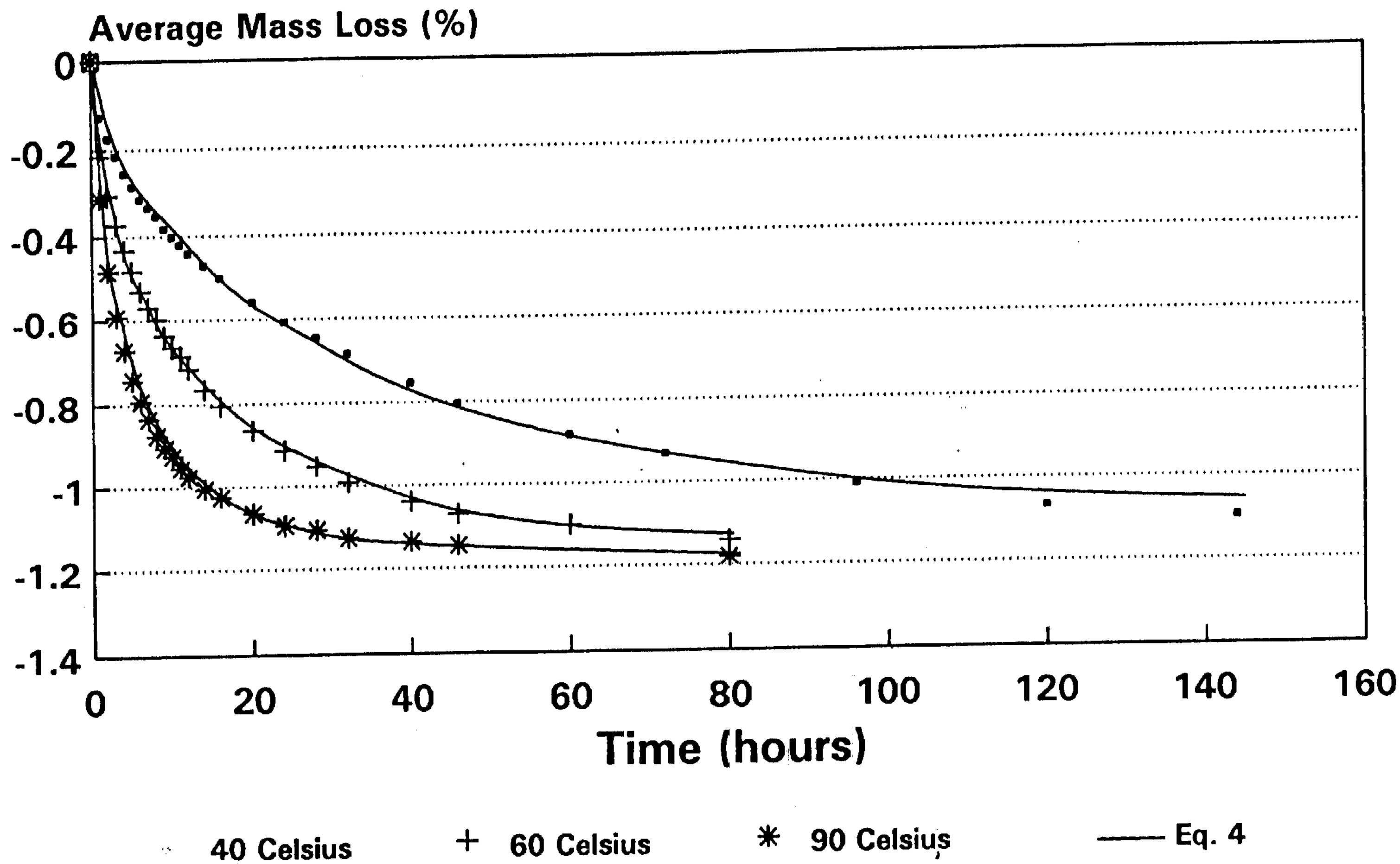

Fig. 4: Moisture desorption of T50/ERL1962 at 3 temperatures