ADVANCES IN

FRACTURE RESEARCH

Editors

K. SALAMA, K. RAVI-CHANDAR,
D. M. R. TAPLIN, P. RAMA RAO

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Thermal Cycling of Carbonfiber-reinforced Resin Systems

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ABSTRACT

The effects of thermal cycling between +100 °C and -160°C on C-fiber reinforced resin systems have been investigated. The associated damage manifests itself largely in the form of matrix cracks which affect strengths and stiffnesses as well as thermal expansions. Initial and residual properties after various cycle numbers were established and compared. In view of the high cost of realistic space simulations, the replacement of radiative cooling in vacuum by gaseous nitrogen at atmospheric pressure and the feasibility of accelerated cooling rates were studied. The investigations are continuing with emphasis on high-performance resin systems for the HERMES program.

KEYWORDS

Thermal cycling, carbonfiber-reinforced resins, space simulations, matrix cracking, property degradation.

INTRODUCTION

Space structures operating in geostationary or low earth orbits may experience thousands of thermal cycles with high amplitudes. Under such conditions, carbonfiber-reinforced resin systems are affected because of discrepant thermal expansions of fibers and resins. Especially in high-performance systems, the substantial difference between their curing and their lower service temperature can induce thermal strains of such magnitude that matrix cracks, interfacial delaminations and degraded fiber/matrix interfaces may result and threaten their thermal fatigue performance.

In a series of tests conducted previously and reported in Ref. 1, the effects of up to 3500 thermal cycles between +100 °C and -160 °C were investigated by comparing initial and residual strengths and stiffnesses of parallel-sided ± 45°-laminates made from various epoxy resins reinforced with T300 fibers. In these tests the space environment was simulated in a space-rated facility in which, under vacuum, the heating was effected by infrared radiation and the cooling by radiation from an N_2- filled source.

In connection with the European space program, the issue of thermal cycling has recently been revived. The Institute for Structural Mechanics of the German Aerospace Research Establishment (DLR), under contract by the European Space Agency (ESA), has evaluated the response to thermal cycling of a new set of materials as well as the feasibility of reducing the cost of realistic space simulations.

SCOPE OF TEST PROGRAM

The investigations included the five material systems identified in Figure 1. Consistent with the previous test efforts, the testing was confined to parallel-sided ± 45°-laminates of 1 mm thickness and 60 % fiber content. From each of the five materials, 195x65 mm large coupons were prepared and grouped into two sets which were thermally cycled under different conditions, and one set which served as an uncycled reference basis. Additionally, laminates were prepared for the determination of their thermal properties.

The investigation of simplified test conditions centered on three questions:

1) Are the mechanical properties of the laminates affected by the absence or presence of a vacuum, or by the replacement of radiative cooling by convective cooling with gaseous nitrogen?
2) Are significant differences in the damage patterns introduced by increasing the rate of the cooling cycles?
3) Do matrix cracking and fiber debonding cease after relatively few cycles because of the associated stress relief?

In order to provide answers to these questions, the thermal cycling tests were conducted in vacuum with radiation cooling as well as at atmospheric pressure in gaseous nitrogen in a standard thermal chamber. In both cases the majority of the test coupons was exposed to 1500 cycles between +100 °C and -160 °C. The temperature vs. time relationships of the different cycling modes are given in Figure 2. Prior to the beginning of thermal cycling and after 10, 50, 150, 400, 800 and 1500 cycles the test coupons were radiographically examined and then subjected to bending tests in order to determine their initial and residual stiffnesses. To guard against damage of the coupons, their acoustic emissions were recorded and the maximum strains in the surface plies limited to 0.2 %.

Upon completion of various cycle numbers, test coupons of each set were dissected to provide specimens for residual tension tests, thermal expansion measurements and microscopic investigations.

The thermal cycling tests in gaseous nitrogen were monitored by in-situ acoustic emission measurements to provide a continuous record of damage initiation and progression.

STRENGTH AND STIFFNESS DEGRADATIONS

Test results of specimens cycled in vacuum

All tests in the space-rated facility were conducted with cycle durations of ca. 60 min. Figure 3 shows the decline of the residual strengths of the five material systems after 400 cycles. The severity of the degradations is seen to depend on the curing temperatures of the resin system - it is negligible for the 120 °C Code 92 material and quite substantial for the 210 °C 65 FWR bismaleimide which, on account of its higher degree of cross-linking, is also more brittle. The test data in both figures are similar to those obtained in a previous test program (Ref. 1), confirming the fact that the rate of crack development is high during the first few cycles and then declines at a diminishing rate without ceasing completely even after 1500 cycles. The nature of the test facility precluded the operation of acoustic emission equipment.

Test results of specimens cycled in gaseous nitrogen

In order to obtain comparable test data, one set of specimens was tested under conditions as identical as possible to those in vacuum. A small change in the cycle duration was necessitated by the different cooling approach but, as shown in Figure 2, the cooling rates in the critical temperature ranges could be kept similar. The majority of the tests was monitored by acoustic emission evaluations.

With respect to both strength and stiffness degradation, very similar results were obtained in both test facilities. As an example, the test data depicting the measured residual properties after 400 thermal cycles are shown in Figure 4 for comparison with Figure 3. While the strength values are quite coincident, the stiffness data displayed modest but inconsistent differences. Considering that the stiffness tests were conducted under low loads and therefore reflect test deviations disproportionally, it was concluded that the evaluation of mechanical property degradations due to thermal cycling does not require the use of space-rated facilities.

On this premise it was considered permissible to include data from both test facilities in the investigation of the effect of different cooling rates, i.e., to compare the degradations occurring under fast cycling in N_2 (cooling rate 32.5 k/min) to those of slow cycling in vacuum (cooling rate 4.7 k/min). In general, the losses in strength and stiffness tended to be somewhat higher after fast cycling. This observation was supported by a study of the emission rates of acoustic energy recorded during both cycling modes. It should be understood that acoustic energy counts represent energies released during the development of a crack, a debond or the rupture of a fiber. The integral of all acoustic energy counts, therefore, is an indicator of the damage state accumulated at a certain number of cycles. These data, in support of the mechanical test data, point to the probability that the damage development does not cease after a relatively few cycles but continues steadily even at high cycle numbers although at diminishing rates.

DAMAGE DEVELOPMENT

The dominant failure mechanism in thermally cycled laminates is the development of matrix cracks during the cooling phases. Their number and distribution through the thickness of the test coupons were determined by means of enlarged x-ray records and by microscopic inspection of polished cross-sections. For the purpose of comparison, a mean crack density was introduced defined as the total number of cracks detected in all plies of a laminate of unit length divided by the number of plies. Consistent again with previous test results, distinct effects of different cycling speeds on the damage pattern were noted, the rate of crack development being slower in the longer cooling phases with correspondingly lower temperature gradients as shown, typically, in Figure 5.

In all cases the cracking commenced in the surface plies and, depending on the characteristics of the resin system, under continuing cycling extended to various degrees into the intermediate plies. This sequential nature of crack formation is probably due to temperature gradients in the laminate thickness direction.

DEGRADATION OF THERMAL STABILITY

The need for thermal stability is an essential requirement for many space applications. The question whether and to what extent the thermal expansions are affected by thermal cycling is, therefore, of considerable importance.

Dilatometrical thermal expansion measurements were performed on both unidirectional and 45°-laminates. The test specimens were 45 mm long and 9.5 mm wide with thicknesses of 3 mm and 1 mm for the unidirectional and cross-plied laminates, respectively. No effects due to thermal cycling were noted in the longitudinal or transverse direction of the uniaxial specimens, while the crack formation in the cross-plied laminates tended to reduce the thermal expansions as shown, for example, in Figure 6. As expected, the reductions of the thermal expansions are in correspondence with the density of the crack patterns.

SUPPLEMENTARY TEST PROGRAM

The specific needs of the "HERMES" program, where the use of carbon-fiber-reinforced bismaleimide and poloyimide resin systems with curing temperatures of 210 °C and 250 °C is under consideration to reduce the weight of thermal insulation, has recently added another dimension to the issue of thermal cycling. Although in this case the lower service temperature has been tentatively defined as -120 °C and the ductility of newly developed resin systems may be expected to be higher, the reason for concern is obvious. It was reinforced by the acoustic energy records in Figure 7 which indicate that matric cracking in a 65 FWR Compimide commences already at approximately -60 °C. New tests with candidate materials for primary structural components for "HERMES" are currently in preparation and will be reported upon completion. First indications point to matrix cracking to commence in similar ranges as in the 65 FWR Compimide material.

SUMMARY

Thermal cycling tests have been performed with different material systems with temperature extremes of +100 °C and -160 °C. In carbon-fiber-reinforced epoxy resins strength and stiffness losses of up to 10 % were noted, whereas a bismaleimide resin suffered 20 % degradation. In all cases, the formation of matrix cracks was observed, the severity dependent on the curing temperature of the resins.

Identical tests were conducted in a space-rated facility under vacuum and with radiative cooling, and in a standard chamber under atmospheric pressure and with convective cooling by gaseous nitrogen. No significant differences were noted in regard to the strength and stiffness degradations of the test specimens. Substantial variations in the cooling rates lead to only moderate effects on the mechanical properties. The major part of the damage manifested itself in the form of matrix cracks during the early cycles with additional damage formation still detectable after 1500 cycles.

With respect to cooling rates, moderate differences were noted between fast and slow cooling, the development of damage being more severe at high cooling rates.

While the majority of the observed damage occurred in the early stages of cycling, some degradation was noted be take place even after 1500 cycles.

The development of cracks affects the thermal expansion coefficients in the sense that with increasing crack densities the expansion coefficients are lowered.

It must be expected that in the bismaleimide and polyimide systems under consideration for the HERMES vehicle, matrix cracks will occur before reaching the -120 °C lower temperature limit. In laminates of substantial thickness, thermal gradients in the thickness direction will accelerate the formation of cracks in the outside plies. These cracks are not necessarily detrimental to the safety of the vehicle but their existence cannot be ignored and their consequences should be explored further.

Of concern also is the potential weakening of the fiber/matrix-interface due to thermal cycling which, as observed in Ref. 1, may lead to a change from cohesive to adhesive matrix failure modes.

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Material Designation	Conpimide 65 FWR-IM6	Fibredux 914C-TS-5	LY 556/ HT976-IM6	Hercules 4502-IM6	Carboform SO 60/92/51
Fibres	Intermediate IM 6 - 12K	T300 - 6K	Intermediate IM6 - 12K	Intermediate IM 6 - 12K	High Modulus GY 70 SE
Resin	Bismaleimide	914-type Epoxy	LY 556 Bisphenol A	4502 Epoxy	Code 92 Epoxy
Curing Temperature	210 °C	175 °C	175 °C	180 °C	120 °C

Figure 1: List of tested materials

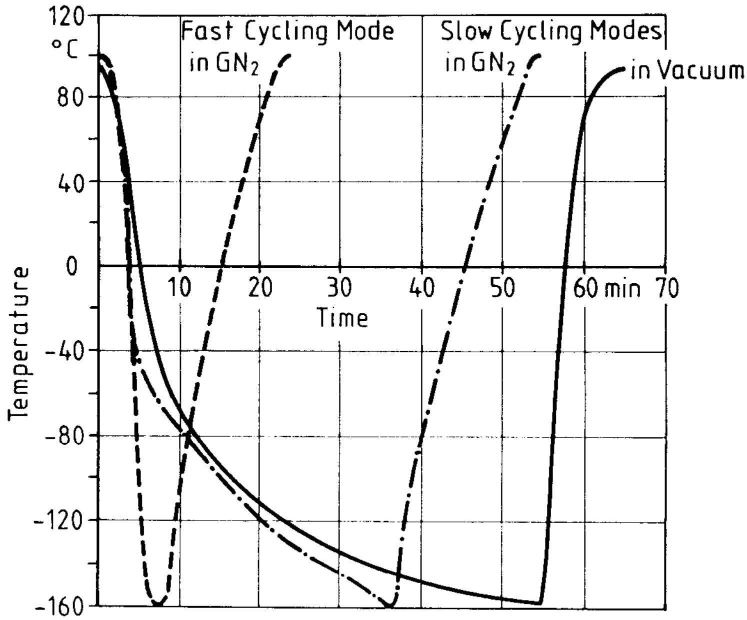

Figure 2: Temperature vs. time relationship at different cycling modes

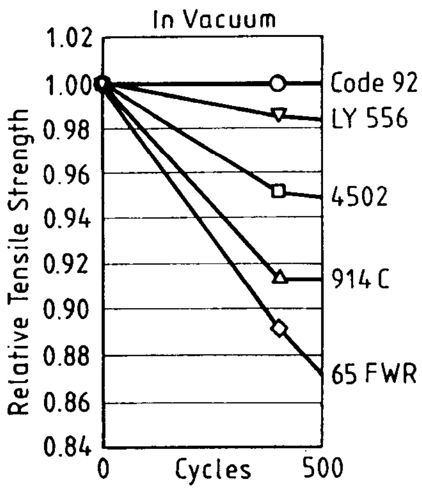

Figure 3

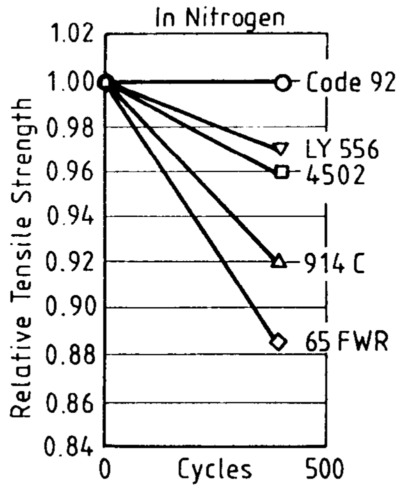

Figure 4

Strength degradation in two different cycling modes

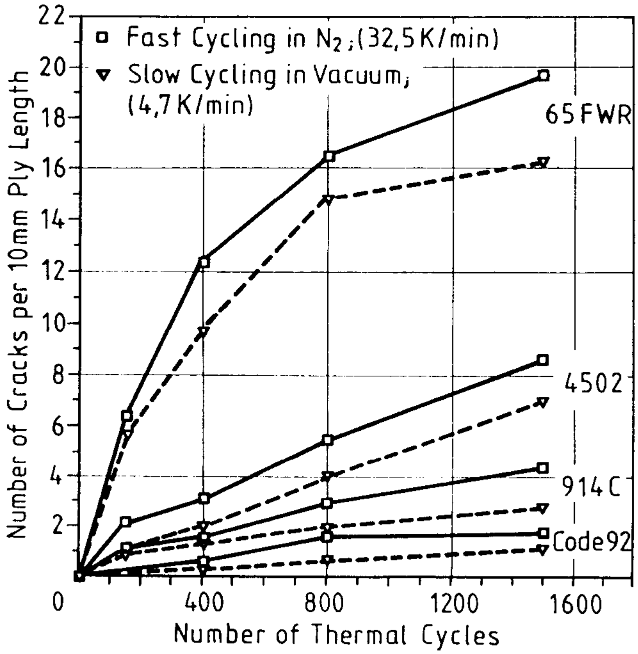

Figure 5: Number of cracks developed in various laminates vs. number of cycles at two different cycling speeds

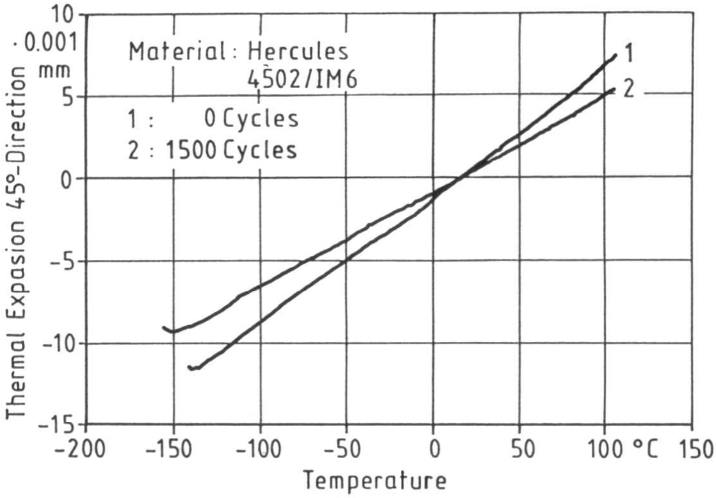

Figure 6: Effects of thermal cycling on thermal expansion

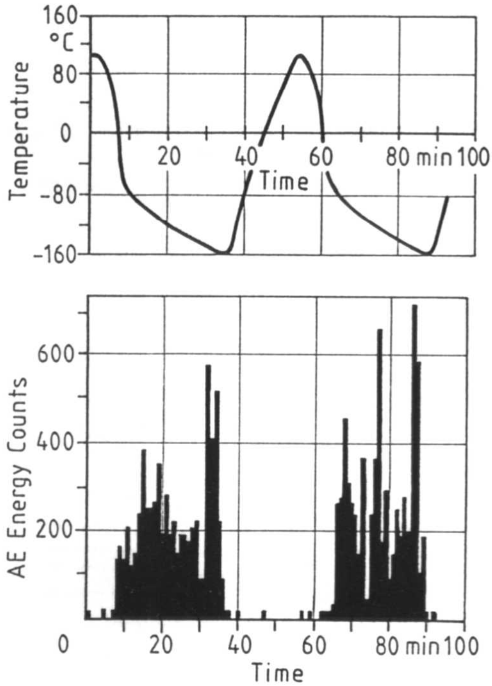

Figure 7: Release of acoustic energy during cycling of Compimide 65 FWR