POSTER

HIGH TEMPERATURE THERMAL AND MECHANICAL PROPERTIES OF HIGH TENSILE CARBON SINGLE FILAMENTS

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INTRODUCTION

Due to its refractory properties, carbon is widely used for thermal protection in aerospace applications, mostly under the form of carbon carbon composite materials. This behavior is strongly dependent on the intrinsic thermal properties of each component of the composite, but also on the good adequation of their thermal characteristics.
Most of these intrinsic properties have to be measured on a single filament. Nevertheless measurements on single filaments, because of the handling difficulties combined with the needed high temperatures are scarcely found in the literature; they give very accurate information on the properties of the fibers and on the reliability of their making process.
This work is devoted to thermal and mechanical properties of the recent high resistance **T900** compared to the **T300** and **T700S**, all fibers from TORAY which are expected to better fit the needs for thermal protection applications.
We have examined "in situ", from 20°C to more than 2000°C on a single filament set-up :
- the thermal expansion (longitudinal and radial)
- the fracture strain with the "loop test" method.

EXPERIMENTAL

1- Sample heating and thermometry :
The carbon fibers being more or less good electrical conductors, we have used the Joule effect to heat them up to 2500°C. Taking into account the emissivity factor of a carbon surface and the discrepency between an 8 µm filament and the ideal black body, we observe a correlation between the electrical power **P** given to the sample filament and its temperature **T** under the vacuum of a turbomolecular pump (10^{-6}mbar). This correlation can be checked only above 600°C with a vanishing filament pyrometer. **P** plotted versus T^4 gives a straight line and thus a very practical and accurate "thermometer".

2- Thermal expansion
This property can be expressed through two different coefficients $\alpha_{//}$ along and $\alpha_{\perp}$ across the fiber, both expressed by a linear combination of the theoretical expansion coefficients α_a and α_c [1] of graphite. But in fact, the useful coefficients for application purposes are the mean values between 20°C and T°C noted $\overline{\alpha_{//}(T)}$ and $\overline{\alpha_{\perp}(T)}$ called secant expansion coefficients [2].
The experimental set-up is based on the "sag method" from V.P. WASAN [3] : a single filament of around 14 mm of length is sticked by silver paste to a sample holder and thus connected to an intensity regulated generator. It is slightly stretched by a small pin-spring and either placed in a small vacuum cell over an optical microscope, or inside a Scanning Electron Microscope (SEM). This very simple device gives a mechanical amplification of the linear expansion of the fiber geometrically deduced from the variation of the sag observed and recorded versus time and temperature. The values of the coefficients are given with a relative error lower than 5%.
With the greater magnification of the SEM, we can reach the radial expansion of the fiber (T900 only because of its regular shape) by measuring its apparent diameter temperature dependence. Both methods are comparable in accuracy.

3- Bending test :
The "loop test" is the method used to get a fast measurement of the bending strength of the fibers [4]. The set-up holds a single loop-shaped filament between a fixed electrode and a moving one fixed to the tip of a bellow valve. The filament is kept flat inside a double weaved net of alumina fibers. When moving the head of the valve, we stretch the loop up to failure. The last recorded image of the unbroken fiber gives the tensile strain ε which can be calculated from the width **D** of the loop before failure and the diameter **d** of the filament : $\varepsilon = d / D$
Measurements can be performed "in situ" at high temperature, the alumina network avoiding the heat-losses by thermal conduction with the holder.

RESULTS AND DISCUSSION

1. Thermal expansion :
1. 1. Longitudinal expansion coefficient : $\overline{\alpha_{//}(T)}$
All the fibers show a very similar behavior (Fig. 1), T900 giving the "cleaner" results : first a contraction from room temperature to 400°C, then an increasing expansion up to 0.6 10^{-6} °C^{-1} at around 2000°C. The first step shows that the average orientation of the

crystallites is perpendicular to the axis of the fiber. At higher temperatures, above 1500°C, the increase of $\overline{\alpha_{//}(T)}$ slows down, showing that the nature of the fiber is modified (as seen also by X-ray diffraction). The graphitic planes expanding, stiffness increases and the thermal expansion coefficient decreases as shown by BUTLER & coll [2].

1. 2. Radial expansion coefficient : $\overline{\alpha_{\perp}(T)}$

According to the graphite values, it is expected to be around 20 times the longitudinal one. There also, we observe the same competition between two causes of the variation of the diameter (Fig. 2): the chemical and crystallographic modifications of the fiber offset its thermal expansion.The measure made along a first temperature rise does not show any variation of the diameter up to 1500°C, then there is a small contraction. In fact, if cooled down to room temperature after each step, the fiber shows a decreasing diameter. From this we can calculate a "composite coefficient" almost constant around 1.5 10^{-5} °C^{-1} in the range of 500°C to 2000°C.

2- Bending test :

At room temperature, the loop test gives very accurate and reproducible results. In comparison to a regular tensile test, the working volume of the sample is much smaller (about 10^5 smaller at failure)[5]. The value of the tensile strength σ, directly related to the number of internal and surface defects and flaws in the working volume, is twice the one found in the literature for any of the studied fibers (also for M40, a high modulus one).

σ (GPa)	T300	T700S	T900	*M40*
tensile test	3,5	4,8	5,4	*2,7*
loop test	6,4	11,6	12,3	*5,4*

At high temperature, we can measure a more important size of the loop at failure. This observation in itself cannot lead to a straightforward explanation.

We have already observed during the thermal expansion experiments an apparent non-Hookean behavior at temperatures above 1000°C, the overheated fibers keeping the memory of their last shape. A similar observation was also made during the high temperature loop tests.

Complementary experiences on loop-shaped fibers heat treated at various temperatures have shown a critical temperature around 850°C over which a plastic deformation starts. This temperature step has already been evidenced in thermogravimetric experiments, explained by the partial departure of the nitrogen atoms. A molecular rearrangement of the fiber induces this apparent inelastic behavior.

CONCLUSIONS

The experimental set-up, worked out for the purpose of testing the thermal characteristics of the carbon fibers, has shown a very good ability to give accurate and reliable information on the very intrinsic properties of these materials over a wide temperature range. The determination of thermal expansion coefficients appears to be very powerful and particularly accurate. The loop test can only give accurate information about the fracture strains of the fibers at room temperature. The exploitation of the high temperature experiments cannot be done without any further theoretical work about non-Hookean behaviors.

The major result is to make evident the chemical effect of low temperature treatment on fibers manufactured at around 1500°C. Nevertheless comparative studies can be achieved giving helpful practical information for the composite manufacturer. In particular, the thermoplasticity observed during our experiments at low temperature (<1000°C) shows the good adaptability of this family of fibers to the thermal stresses involved in the different steps of the composite materials processing.

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[3] V.P. WASAN, Carbon 17 (1979), p55-58

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[5] W. R. JONES and J. W. JOHNSON, Carbon 9 (1971), p 645-655

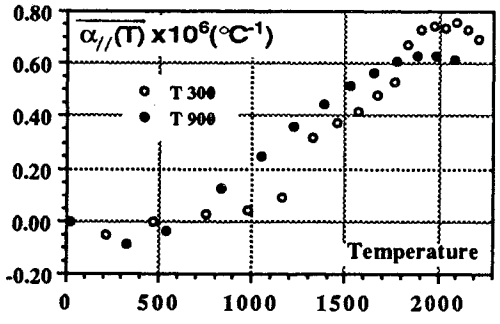

Fig 1 : T900 / T300 longitudinal expansion

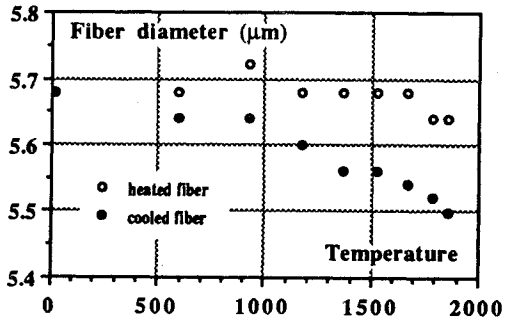

Fig 2 : T900 radial expansion