Experimental Measurements and Numerical Modelling of Conductive and Radiative Heat Transfer in Polytetrafluoroethylene

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Polytetrafluoroethylene (PTFE, or Teflon) components are used in propulsion and power systems due to their resistance in high temperature environments. Heat transfer in PTFE samples is investigated, both experimentally and numerically, to characterize the thermal protection behavior of PTFE samples under conductive and radiative heat sources. PTFE is known to undergo a sharp, reversible transition at 600 K, changing from the white, virgin substance to a transparent, amorphous material. As the temperature of PTFE is raised above 620-670 K, pyrolysis occurs and the polymer decomposes into gaseous products. Experiments performed using a CO_2 laser show that the internal absorption of radiation causes a continous increase of both the internal temperature and the thickness of the gel layer. These experimental data are then used to validate a model of the thermal response and degradation process of PTFE samples. To model the ablation processes, under the same operating conditions of the experimental approach, a transient one-dimensional model, including variable thermal properties and radiation absorption is written. Energy, mass conservation equation and the decomposition rate kinetic equations are discretized by means of a finite difference technique. The comparison between experimental and numerical results is then discussed. Conclusions stress the importance of a coordinated approach to investigate the design and the sizing of PTFE thermal protection systems under ablating and non-ablating conditions.

Nomenclature

A	:	sample cross section
B	:	pre-exponential factor
c_p	:	specific heat
E_a	:	activation energy
h	:	enthalpy
H_d	:	depolymerization energy
I_0	:	external incident radiation intensity
m_a	:	mass ablation rate
n	:	reaction order
P	:	sample perimeter
r	:	reflectivity
R	:	universal gas constant
Q	:	heat of decomposition
r_a	:	ablation rate
t	:	time
T	:	temperature
X	:	abscissa
α	:	absorption coefficient
ε	:	surface emissivity
η	:	reaction completeness
λ	:	thermal conductivity
ρ	:	density
Φ	:	source term in energy equation
ψ	:	convective and radiant losses term

subscripts:

f	:	final
g	:	gas (decomposition products)
j	:	index in space
n	:	index in time
s	:	surface
o	:	initial

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1. Introduction

Ablation is an effective and reliable method, widely accepted in the aerospace applications, for protecting heat-sensitive structures against the effects of an external high temperature energy source [1]. Its use is restricted to applications where the structure needs to be protected only for a limited period of time. The term ablation denotes the loss of matter from the surface exposed to the heat flux, usually due to the action of a hot moving gas stream or to the action of a radiant flux. The most widely used ablation materials are polymers or polymer-containing composites. This is because polymers provide such advantageous properties as low density, low thermal conductivity, high specific heat and controlled decomposition which yields low molecular weight gaseous products.

The wide range of heating conditions which must be accommodated by ablative thermal protection systems necessitates a variety of materials whose properties are tailored to meet specific performance criteria. These criteria include controlled surface recession, heat transfer through the ablator to the substrate, heat shield weight, material and fabrication cost and heat shield reliability. Since most ablators are composites of several constituents including polymer matrices, fiber reinforcement, low density fillers, gas formers and endothermically decomposing species, material variations can be readily achieved. Single materials systems which are the exception to this rule are polytetrafluoroethylene and fused silica. They find only a limited use, mainly as ablating antenna window materials, but they have been treated extensively in the literature since they represent classic examples of ablators whose mass loss occurs by depolymerization, melting, vaporization and oxidation. These mechanisms lend themselves to theoretical analyses.

The accurate knowledge of the thermal response of these materials is essential for the optimum design of thermal protection systems. Specific purposes of this work are to develop a diagnostic technique to characterize the macrokinetic degradation of polytetrafluoroethylene and to develop an appropriate mathematical model for the thermal response of Teflon, undergoing decomposition without the surface recession. In order to use the model, the decomposition kinetics under very high heating rates, not available in the literature, has to be determined, while temperature dependent thermal and transport properties are taken from well established data in the literature.

The accuracy of the model is estimated by comparing the computed and experimental transient temperature profiles, in decomposing Teflon cylindrical specimen, exposed to a radiant heat flux of known intensity.

2. Applications and properties of polytetrafluoroethylene

Polytetrafluoroethylene decomposes primarily by a depolymerization mechanism and therefore leaves no char residue. Thus, it is suitable for ablating antenna windows which must be RF-transparent during and after ablation. The literature on Teflon ablation far exceeds its practical usage, because the depolymerization facilitates the analytical treatment of the ablation process and of the associated heat absorption mechanism.

Ablation of polytetrafluoroethylene by depolymerization appears to occur by a first-order reaction [2-3]. The monomer has a very high vapor pressure so that it tends to flash directly to vapor. A phase transition from the semicrystalline state to amorphous gel occurs at approximately 325 °C with an energy of phase transition of approximately 14 cal g^{-1} . The process of depolymerization absorbs approximately 420 cal g^{-1}. However, if the reaction temperature exceeds 538 °C, the decreased viscosity of the surface gel layer will allow diffusion of incompletely depolymerised species through the surface. Once formed, the gaseous monomer can react with itself to produce higher molecular weight species ($2\ C_2F_4 \rightarrow C_4F_8$, $3\ C_2F_4 \rightarrow 2\ C_3F_6$), whose concentration is a function of pressure. When exposed to air at elevated temperature, the monomer may also dissociate and combine with oxygen [3] ($C_2F_4 + O_2 \rightarrow 2\ COF_2 \rightarrow 2\ CO + F_2$).

The main thermal, chemical and optical properties of Teflon are shown in Tab. 1. These properties are assumed as reference properties in order to obtain the numerical results discussed in Sec. 8 of this paper.

Property	Value	Dimension	Reference
density, ρ_v	$2.119 + 7.92 \times 10^{-4}$ T/K - 2.105×10^{-6} T^2/K^2	g cm^{-3}	[2]
density, ρ_a	$2.07 - 7 \times 10^{-4}$T/K	g cm^{-3}	[2]
Thermal conductivity, λ_v	$(1.2 \times 1.467 \times 10^{-2}$ T/K) x 10^{-4}	cal s^{-1} cm^{-1} K^{-1}	[1]
Thermal conductivity, λ_a	$(21.04 - 3.34 \times 10^{-2}$T/K + 1.39×10^{-5} $T^2/K^{2)} \times 10^{-4}$	cal s^{-1} cm^{-1} K^{-1}	[1]
Specific heat, $c_{p,v}$	$0.123 + 0.3733 \times 10^{-3}$ T/K	cal $g^{-1}K^{-1}$	[1]
Specific heat, $c_{p,a}$	$0.216 + 0.156 \times 10^{-3}$ T/K	cal g^{-1} K^{-1}	[1]
absorption coefficient, α_v	0.056	cm^{-1}	[6]
absorption coefficient, α_a	0.22	cm^{-1}	[6]
surface emissivity, ε	0.1		[6]
depolymerization energy, H_d	$424 - 6.67 \times 10^{-2}$ T/K	cal g^{-1}	[4]
	76.6	Kcal mol^{-1}	[1]
	83.0	Kcal mol^{-1}	[3]
	75.6 +- 5.7	Kcal mol^{-1}	[2]
activation energy, E_a	80.5	Kcal mol^{-1}	[5]
	75.6	Kcal mol^{-1}	[2]
	83.0	Kcal mol^{-1}	[4]
	$1.5 \cdot 10^{17}$	s^{-1}	[1]
	$4.7 \cdot 10^{18}$	s^{-1}	[5]
pre-exponential factor, B	$5.0 \cdot 10^{18}$	s^{-1}	[1]
	$3.0 \cdot 10^{19}$	s^{-1}	[3] and [4]
	$3.1 \cdot 10^{19}$	s^{-1}	[2]
	$3.1 - 3.7 \cdot 10^{19}$	s^{-1}	[2]
heat of transition, h_t	14	cal g^{-1}	[6]

Tab. 1

Properties of Teflon (subscript v: virgin material; subscript a: amorphous phase).

3. The approach suggested by Zenin

The strategy assumed in this paper follows the approach suggested by Zenin [7] in the study of steady deflagration waves in solid propellant combustion. The temperature vs time trend, in a solid reacting condensed-phase sample, under the hypotheses of 1-d and steady conditions, is described by the following energy conservation equation:

$$\frac{d}{dx}(\lambda \frac{dT}{dx}) - \rho r_a c_p \frac{dT}{dx} + \Phi(T) = 0$$

with the boundary conditions: $x = 0 \quad T = T_s$

$x \rightarrow \infty \quad T = T_0$

and where:

$$\Phi(T) = Q^* \eta_s \rho B e^{(-\frac{E_a}{RT})} (1 - \eta_s)^n$$

If a first order reaction (n = 1) and the completeness of the reaction are assumed, the integration of the equation leads to:

$$(\frac{m_a Q}{T_s})^2 = \lambda\rho \frac{R}{E_a} QBe^{(-\frac{E_a}{RT_s})}$$

and the logarithm form is:

$$\ln(\frac{m_a Q}{T_s}) = -\frac{E_a}{2R}\frac{1}{T_s} + \frac{1}{2}\ln(\lambda\rho \frac{R}{E_a} QB)$$

where:

$$Q = c_p(T_s - T_0) + \frac{H_d}{2}$$

The unknown variables in the previous equations are the temperature of the heated surface T_s, the mass ablation rate m_a, the pre-exponential factor B and the activation energy E_a in the Arrhenius term. The surface temperature (T_s) considered in the previous equation is that corresponding to the onset of the depolymerization reactions; the experimental trend of the temperature vs time curve, clearly shows such occurrence, characterized by a macroscopic change in the slope of the curve. The time instants at which this temperature is reached in-depth, measured by two different thermocouples placed at a known distance from the heated surface, allow to determine the thermal wave propagation rate (r_a) and consequently the mass ablation rate ($m_a = r_a \rho$). A plot of the procedure is shown in Fig. 1. The logarithmic form of the equation, in a $\ln(m_a Q/T_s)$ vs $1/T_s$ plot is a straight line equation, whose slope is correlated to the activation energy ($m = -E_a/2R$) while the intercept with the vertical axis is correlated to the pre-exponential factor ($q = ½ \ln(\lambda \rho R/E_a Q B)$). Fig. 2 show the described correlation.

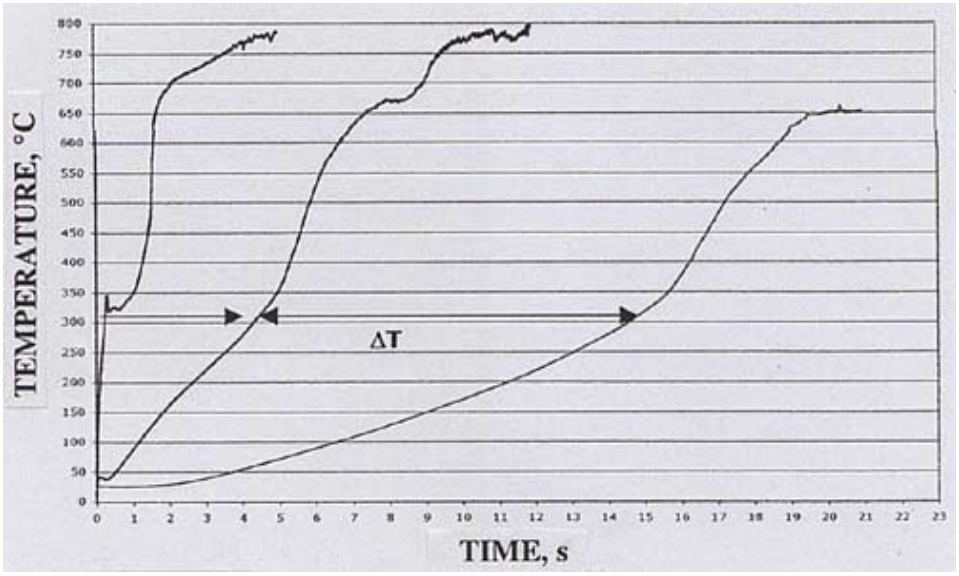

Fig.1 – Evaluation of surface temperature (T_s) and mass ablation rate (m_a).

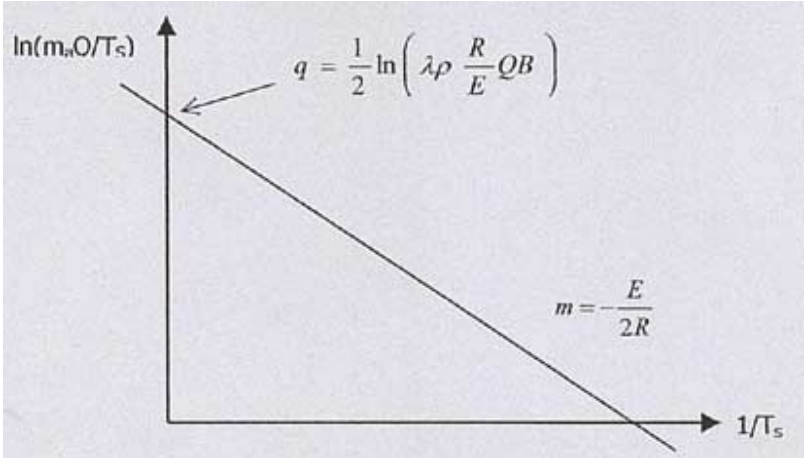

Fig. 2 – Evaluation of activation energy (Ea) and pre-exponential factor (B).

4. Experimental set-up

The set up used to measure the thermal response in Teflon samples is shown in Fig. 3. A CO_2 laser (adjustable power until 200 W), is used to heat a cylindrical Teflon specimen (10 mm diameter, 20 mm length), placed in a closed chamber in which a nitrogen atmosphere prevent the onset of a flame due to the oxidation of the depolymerization gases. The power impinging on the surface of the Teflon sample is measured, for each test, by a power meter. The temperature curves, shown in Fig. 1, are measured by means of Pt-Pt/Rh microthermocouples (50 μm wires), inserted in the sample following the procedure shown in Fig. 4. The temperature signal acquisition is performed by a 16 bit four channel acquisition card (National Instruments) placed in a HP Vectra PC. A Labview program is developed to control the output coming from the three microthermocouples inserted in the Teflon sample. A sampling rate of 5000 Hz has been used during the tests.

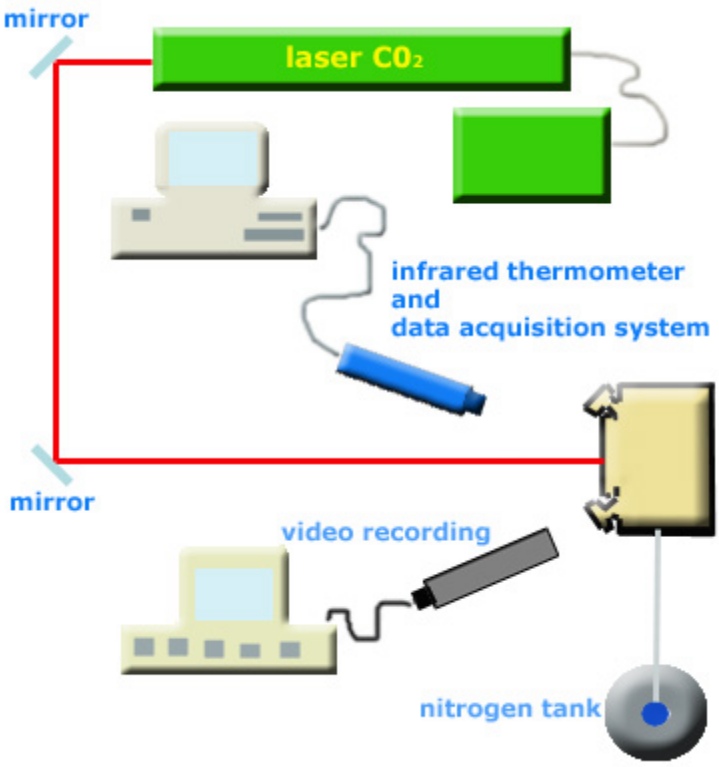

Fig. 3 – Set up used to measure the thermal response.

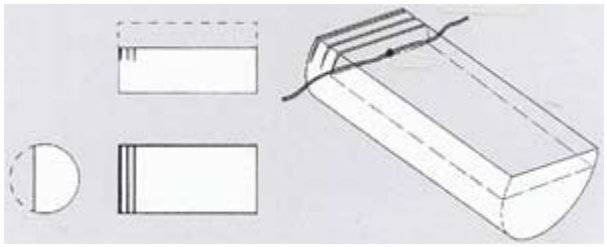

Fig. 4 – Microthermocouples arrangement in cylindrical Teflon samples.

5. The measurement of the surface temperature

The initial temperature rise, due to a heat flux impinging on the surface of a chemically reacting material, depends on the heat conduction rate into the material and on boundary conditions. The thermal response during this initial phase is typical of a material without the occurrence of chemical reactions. Chemical reactions, forming gaseous products, begin to occur at a temperature of approximately 300 °C, depending of course on the nature of the material. These gaseous products diffuse back attenuating the heat conduction to the reaction zone; as time increases a wider pyrolysis zone, which further penetrates in the original virgin material, can be observed. The pyrolysis process causes large changes in both thermal and transport properties. It should also be noted that these processes are strongly dependent on the heating rate, usually shifted to higher temperatures as the heating rate is increased. To model the thermal behavior of the heated material, under the effects of the degradation processes, the occurrence of chemical reactions and the presence of gaseous products, variable thermal and transport properties, thermochemical expansion or contraction must be considered.

The measurement of the surface temperature represents a crucial point in the approach followed in this paper. This temperature can be determined looking at the change in the slope of the temperature curve, related to the change from the situation of a nonreacting, to the situation of a reacting solid material heating. Typical trend, shown in Fig. 1, can be detected in each test. In order to measure accurately the trend of the surface temperature, an infrared thermometer (Raytek, model MA2SC), designed to measure temperatures in the range 350-2000 °C, was used. The response time of the device is 10 ms (to reach the 95% of the final temperature), reduced to 1 ms in fast modulation, which is used in the tests. Tests performed at 30 and 50 W laser power, are shown in Fig. 5 and Fig. 6, respectively. In both cases the temperature, recorded by the infrared thermometer is very close to the trend of the microthermocouple in the region of the fast heating (until 600 °C), but it is lower at high temperatures. This trend can be motivated by the presence of depolymerization gases in the region of the surface, along the infrared thermometer optical axis, and by the impinging of the laser beam on the bead of the microthermocouple. Approximately 1 s after the starting of the heating process, because of the ablation rate, the bead of the microthermocouple placed on the surface is not in contact with the Teflon but it is directly hit by the laser beam.

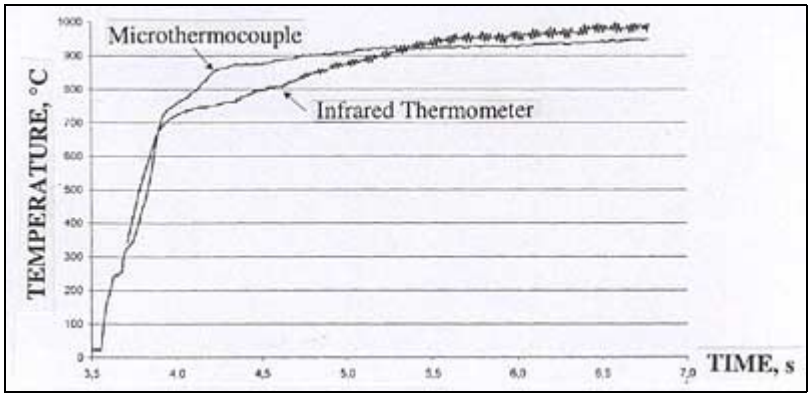

Fig. 5 – Temperatures measured by microthermocouple and infrared thermometer (laser power: 30 W).

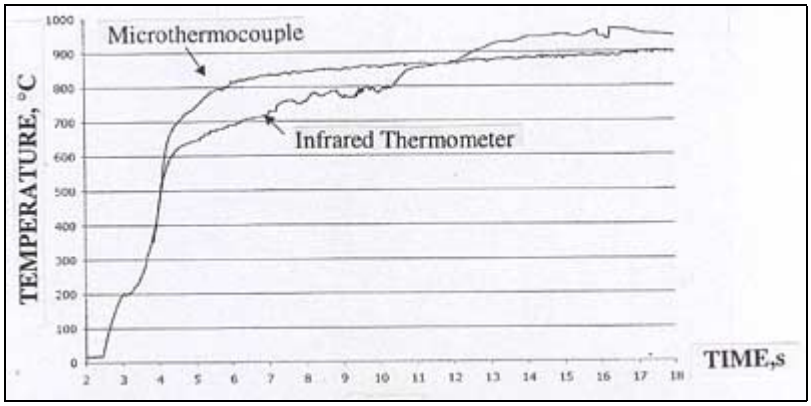

Fig. 6 – Temperatures measured by microthermocouple and infrared thermometer (laser power: 50 W).

6. Experimental results

Following the procedure previously described, Tab. 2 summarizes measured and computed quantities during the tests. Three selected tests for each laser power, in the range between 30 and 50 W, are shown in the tables. A general plot of the performed tests is shown in Fig. 7. The equation of the fitting curve is: y= -11773 +16.837 with R^2 = 0.958. The values of the pre-exponential factor and of the activation energy are B = 3.83 10^{19} s^{-1} and E_a = 46786 *cal/mole*. In comparison with the values suggested by Holznecht [2] (B = 3.1 10^{19} s^{-1}; E_a = 75600 *cal/mole*) the value of B is approximately the same but the activation energy of the macrokinetic scheme is much lower. It is important to stress that the macrokinetic parameters found in the literature are obtained by a thermogravimetric approach [8], characterized by heating rates of few degrees per minute, while in the present approach the heating rate is of the order of seven hundred degrees per minute. The heating rate seems to have a remarkable effect on the degradation process.

Power	test no.	distance T2 - T3	ρ [g/cm^3]	r_a [cm/s]	m_a [g/ cm^2 s]	Q [cal / g]	$1/T_s$ [K^{-1}]	ln (m_a Q/Ts)
30 W	19	0,19	1,68	1,7E-02	2,9E-02	268,6	1,780E-03	-4,293
	35	0,14	1,68	1,4E-02	2,3E-02	265,2	1,813E-03	-4,503
	38	0,08	1,68	1,2E-02	2,1E-02	265,1	1,818E-03	-4,589
35 W	55	0,13	1,67	2,5E-02	4,2E-02	386,2	1,765E-03	-3,915
	56	0,13	1,68	1,8E-02	3,1E-02	386,6	1,782E-03	-4,224
	57	0,13	1,68	2,0E-02	3,4E-02	386,5	1,780E-03	-4,123
40 W	58	0,09	1,67	3,3E-02	5,6E-02	385,8	1,746E-03	-3,650
	59	0,05	1,67	3,2E-02	5,4E-02	385,8	1,747E-03	-3,688
	60	0,12	1,68	1,6E-02	2,6E-02	387,1	1,805E-03	-4,360
45 W	61	0,10	1,67	3,4E-02	5,7E-02	385,5	1,733E-03	-3,634
	62	0,10	1,68	1,3E-02	2,1E-02	387,3	1,815E-03	-4,567
	63	0,10	1,66	4,2E-02	7,0E-02	385,3	1,726E-03	-3,426
50 W	64	0,20	1,67	2,9E-02	4,8E-02	385,7	1,743E-03	-3,799
	65	0,09	1,67	2,5E-02	4,2E-02	386,1	1,759E-03	-3,935
	66	0,15	1,67	3,1E-02	5,2E-02	385,7	1,741E-03	-3,728

Tab 2. – Measured and computed quantities during the experimental tests.

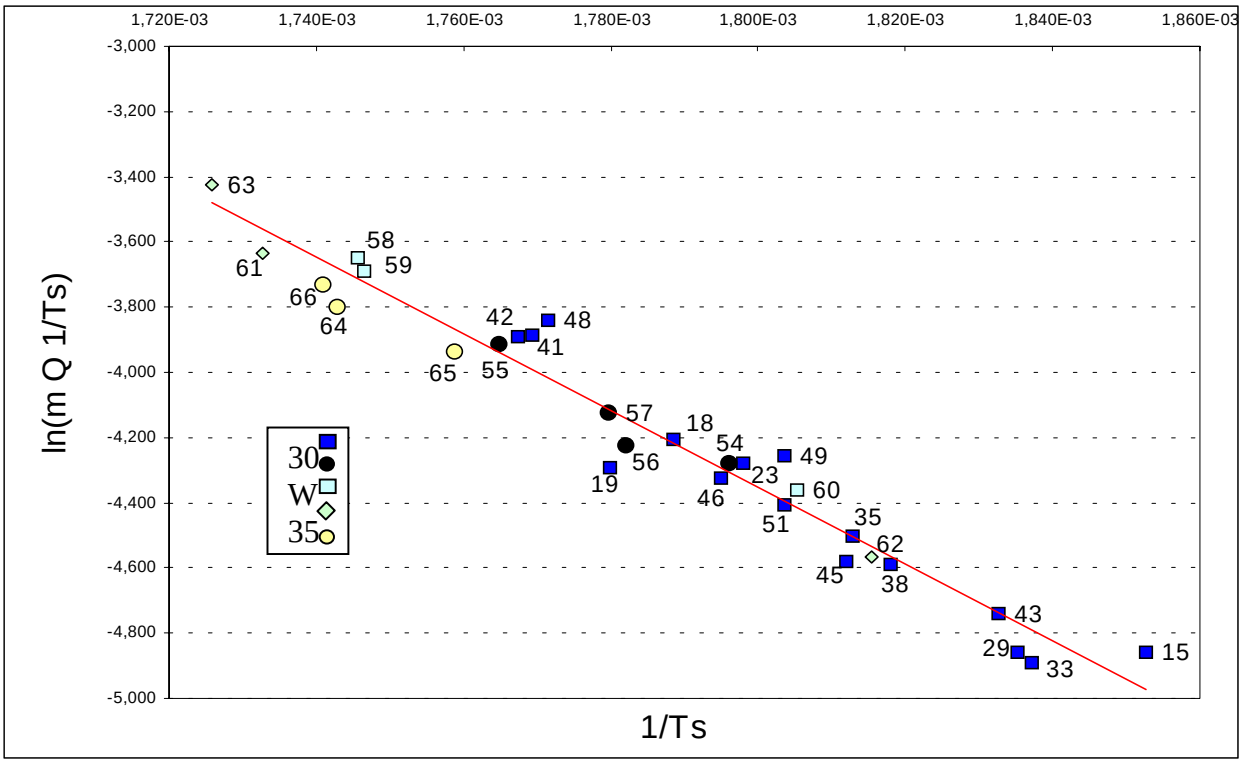

Fig. 7 – Experimental results plotted in order to evaluate the activation energy and the pre-exponential factor.

7. Numerical approach

Some typical assumptions, usually accepted [9], are introduced to simplify the model: thermochemical expansion or contraction is not considered; decomposition gaseous products do not accumulate in the heated material; pyrolysis gases are in thermal equilibrium with the solid. Under these assumptions the governing equations of the model, written to obtain the thermal response to a radiant energy flux, impinging on a Teflon sample, include the energy conservation equation, the mass conservation equation and the degradation rate equation, according to the following system:

$$\frac{\partial}{\partial t}(\rho h) = \frac{\partial}{\partial x}\left[\lambda \frac{\partial T}{\partial x}\right] - \frac{\partial}{\partial x}(\dot{m} h_g) - Q\frac{\partial \rho}{\partial t} + \frac{1-r}{d} I_0(t) \exp(\frac{x-x_s}{d}) - \psi \frac{p}{A} \tag{1}$$

If the accumulation of gases is not considered, the mass conservation may be expressed by:

$$\frac{\partial m_g}{\partial x} = -\frac{\partial \rho}{\partial t} \tag{2}$$

The degradation rate is given by an n^{th} reaction order (= 1 for Teflon) kinetic equation:

$$\frac{\partial m}{\partial t} = -B m_0 \left[(\frac{m-m_f}{m_0})\right]^n \exp(\frac{E_a}{RT}) \tag{3}$$

Under the assumption of no expansion or contraction of the solid heated material, the degradation rate equation also yields the rate of the density change, $\partial\rho/\partial t$, contained in Eq. 1.

Eq. 1 can be written in terms of specific heats; substituting the continuity equation, expanding the derivative terms, rearranging the factors, the following form is obtained:

$$\rho c_p(T)\frac{\partial T}{\partial t} = \lambda \frac{\partial^2 T}{\partial x^2} + \frac{\partial \lambda}{\partial x}\frac{\partial T}{\partial x} - m_g c_{pg}(T)\frac{\partial T}{\partial x} - \frac{\partial \rho}{\partial t}(Q + h - h_g) + \frac{1-r}{d} I_0(t) \exp(\frac{x-x_s}{d}) - \psi \frac{p}{A}$$

This equation, together with Eqs. 2 and 3, form a system of non-linear partial differential equations. It has to be solved simultaneously for unknowns m, m_g and T. Thermal conductivities and specific heats, for both crystalline and virgin materials, are assumed temperature dependent, according to the expressions of Tab. 1. Initial and boundary conditions are assumed as follows:

initial condition: $T(x, t=0) = T_a$

boundary conditions:

$$\left.\begin{aligned} &(\lambda(T)\frac{\partial T}{\partial x})_{x=0} = \varphi(T(x=0,t) - T_a) \\ &m_h = 0 \end{aligned}\right\} \quad x = 0, t > 0$$

$$\left.\begin{aligned} &(\lambda(T)\frac{\partial T}{\partial x})_{x=x_s} = f(t) \\ &f(t) = (1-r)I_0(t) - \psi \end{aligned}\right\} \quad x = L, t > 0$$

Ψ: convective and radiant losses term.

A finite difference approach is used to discretize the equations, which can be written in the form:

$$-A_j T_{j+1}^{n+1} + B_j T_j^{n+1} - C_j T_{j-1}^{n+1} = D_j$$

This equation can be applied to all the nodal points of the domain, except the points of the boundary, giving a system of jf-2 algebraic equations whose coefficients form a tridiagonal matrix. The Thomas algorithm is used to solve the system, using the well known recurrence formula:

$$T_j^{n+1} = E_j T_{j+1}^{n+1} + F_j$$

where: $E_j = \dfrac{A_j}{B_j - C_j E_{j-1}}$ j = 2,3,4,…jf-1 and $F_j = \dfrac{D_j + C_j F_{j-1}}{B_j - C_j E_{j-1}}$ j = 2,3,4,… jf-1

To initialise the procedure the boundary conditions have to be used.

8. Numerical results

Numerical experiments are performed at the same power intensities of the laser beam which are experimentally investigated; temperature is monitored vs time at the same depths, from the heated surface, at which microthermocouples are placed in the experimental tests. For the numerical simulation it is needed to know thermal, kinetic and optical properties of polytetrafluoroethylene. Some properties are known from the literature (thermal properties), others are obtained experimentally in this project, others are unknown. Unknown properties are found with a parametric study. Preliminary results obtained with the numerical code developed in this project, are presented in Fig. 8 and Fig. 9. They show a comparison between the experimental and the numerical thermal response of polytetrafluoroethylene samples heated with a thermal power of 60 and 80 W. Several aspects, mainly the significant influence of the optical properties of the virgin and amorphous materials have to be further investigated, but the macrokinetic parameters, experimentally obtained, allow to reproduce the experimental temperature profile. It seems impossible, for the heating rates considered in this study, to reproduce this trend using the activation energy values available in the literature.

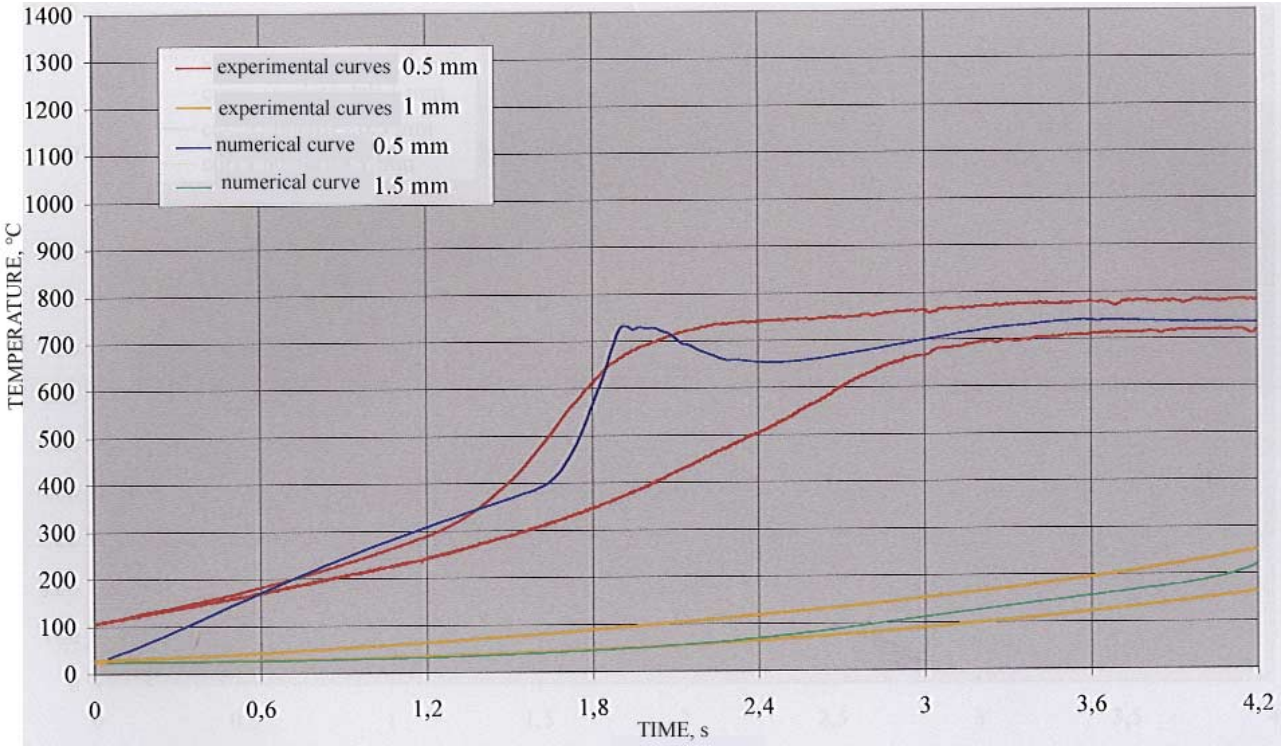

Fig. 8 – Comparison between experimental and numerical results. Thermal power: 60 W.

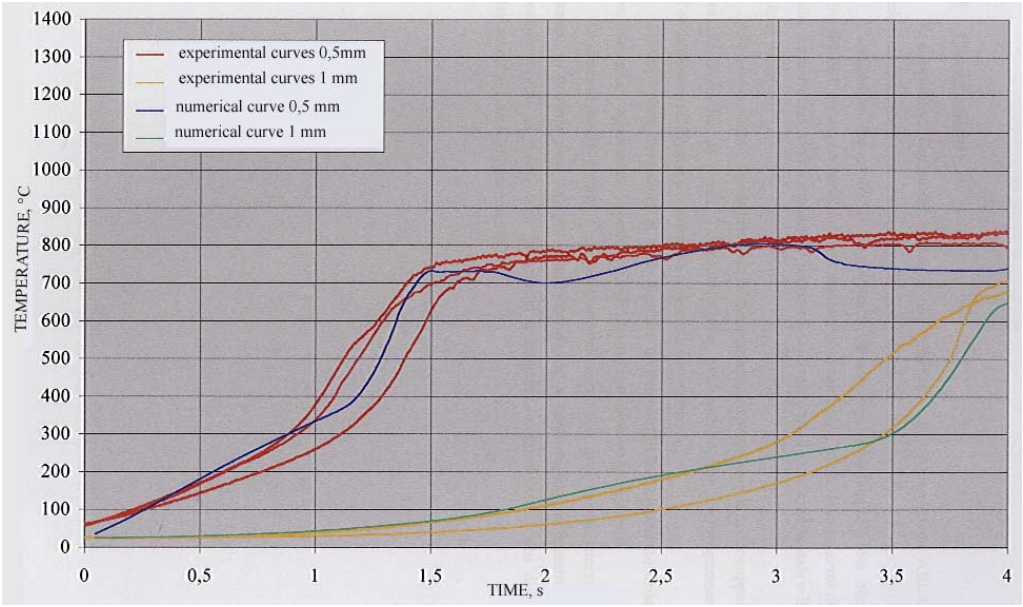

Fig. 9 – Comparison between experimental and numerical results. Thermal power: 80 W.

9. Conclusions

An experimental set up has been designed to measure the macrokinetic parameters which characterize the Teflon ablation under conditions of very high heating rates, obtained with a laser source. To perform such task, time dependent temperature profiles were measured at the surface and in-depth, in cylindrical Teflon samples. These temperature measurements also provided the experimental data base useful to evaluate the accuracy of the simulations of the numerical model. Considering the preliminary stage, the thermal model developed in this paper satisfactorily predicts the experimental thermal response. The accuracy of the model depends upon the accuracy of the input properties, as well as the boundary conditions. The investigation of this dependence is the next step of this project, in the framework of an accurate validation of the model.

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Paper Number: 20

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Question:
The pryrolysis of TEFLON is an exothermic reaction. Is it not better to use a material with endothermic reaction?

Answer:
Most ablators are composites including polymer matrices, fiber reinforcements (for mechanical properties), low density fillers end species which decompose endothermically.
For such materials the knowledge of properties, end the dependence of properties on temperature is rather uncertain. Teflon finds on to a limited use in ablative thermal protection systems, but it has been treated extensively in the literature.
Properties of Teflon are well known and this is the reason to choose Teflon in order to check the proposed approach for the evaluation of the thermal degradation rate at high heating rates.

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