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Detection of Defects in Composite Pyrotechnic Materials and Products by IR NDT Methods

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Abstract

Pyrotechnic composite materials are widely used in military and civil applications as solid composite (heterogeneous) propellants and gas-generators in rocket motors. Non-destructive testing (NDT) methods are used to detect defects which can disturb in reliable and safe functioning of these materials and products or distinctly decrease the level of their performance characteristics. These defects are caused by intrusions, voids, caverns (e.g. air bubbles), cracks and stratifications of different size, having different shapes and densities than the main (matrix) material. Defects in composite pyrotechnic materials and products can be created in their production stage or in service time esp. during long-term storage. The diverse and changeable environmental conditions appearing in life cycles of composite pyrotechnic materials and products including such materials, decide in considerable degree about their physicochemical properties such as thermal and mechanical characteristics, physicochemical stability, material compatibility (esp. chemical reactivity) and durability. In this paper both computer simulations to define defects which could be detected by using IR thermography method, and selected experimental investigations are presented.

Keywords: infrared NDT, composite pyrotechnic materials, inhibitor coating, propellants, surface and subsurface defects

1. Introduction

Solid rocket propellants are usually divided into two main groups taking into consideration their material structure and composition i.e. homogeneous and heterogeneous (composite) multi-component systems. Homogeneous solid propellants are consisted of fibrous, polymeric nitrocellulose (NC) matrix contained intra-molecular oxygen (oxidizer) and ca. 12.5 % by weight of intra-molecular nitrogen which plays role as fuel agent in NC. NC is usually gelatinized by nitroglycerine. To this double-base material system there are also introduced technological additives like plasticizers, stabilizers and burning rate modifiers. Heterogeneous solid propellants are composed of oxidizer crystals of inorganic salts distributed (suspended) in polymeric binder matrix (of organic origin). Analogously as for homogeneous propellants, heterogeneous ones include low molecular substances like plasticizers, burning rate modifiers and additionally energetic ingredients like metal powders (very common is Al) and even explosives like hexogene (RDX) and octogene (HMX).

Above mentioned propellants undergo physicochemical changes (e.g. chemical reactions like decomposition, oxidation, phase transitions) during their production process and life cycle period mainly including storage and transport. The rate of these physicochemical processes in the propellant grains in high degree depend on thermal (temperature), mechanical and other physicochemical stimuli of environment like action of wet and/or salt existed in air. The exposition of propellants to such stimuli, especially to thermal and mechanical ones, results in generation and growth of mechanical stresses and structural defects in their grains (charges).

From statistics given in literature data it appears that basic reasons of unstable burning of solid propellant in rocket motors often leading to their failures, there are such defects like cracks, voids in propellant and de-bonding between propellant charge and its inhibiting layer (inhibitor coating). It was established that during operation of rocket motors these defects are able to cause rapid rise of propellant burning surface which induces violent increase of combustion products pressure in motor chamber sometimes leading to instabilities of rocket motor thrust course and/or to destroying even leading to catastrophic rupture of rocket motor. To avoid above instabilities accompanying burning process of propellant charges caused by structural defects of propellant and ensure high level of probability / confidence of appropriate safety and operation reliability/suitability of rocket motor, there were evolved several non-destructive test (NDT) methods to detect, measure and assess defects in structure and integrity of propellant charges. These NDT inspection methods for solid rocket propellant charges are mainly: radiographic (X-ray) testing, computed tomography, neutron radiography, ultrasonic testing, infrared (IR) thermography, acoustic emission testing, optical holography and of course – visual inspection. Among NDT methods, radiography techniques have the widest usage, but they indicate some inconveniences/disadvantages because in dependence on X-ray absorption abilities of tested samples, these techniques require careful selection and adjustment of operation mode of X-ray source within the range of its radiation intensity. In several cases, X-ray techniques also need troublesome and time consuming measurements connected with determination of defects size and their positioning, bringing in consequence some difficulties in interpretation of X-ray images esp. in terms of defects size, areas in the region of surface, subsurface layers and boundary surfaces.

2. IR Thermography

IR thermography applications as NDT method is not a new concept in evaluation of objects. IR thermography is one of many techniques used to “see the unseen” [1] because it makes possible to detect structural changes inside material of tested object, which are not visible on its surface. Its physical principle is based on the analysis of transient thermal fluxes (or temperature distributions) that appear in objects under thermal stimulation. The presence of structural irregularities causes local thermal anomalies of whose amplitudes and temporal behavior depend on anomalies properties and their depth. The development of IR thermography is caused by the new generation of quick thermal cameras, which make possible to watch/measure quick processes of heat transfer, and by the development of data processing algorithms in last years[2, 3].

Defects in composite pyrotechnic materials and products/goods containing such materials can appear in their production stage or during their life cycle esp. during long-term storage. The diverse, changeable environmental conditions appearing in life cycles of composite pyrotechnic materials and products including composite pyrotechnic components decide in considerable degree about physicochemical characteristics such as thermal, mechanical and chemical stability, physicochemical compatibility and durability of such products and materials.

The main factors influencing the deterioration of these products and materials quality during their service time are of an environmental type including conditions of storage and operation i.e.:

- humidity of air, changes of temperature and contamination agents in atmosphere like dusts, salts, oxides of inorganic elements like sulphur or nitrogen ;
- mechanical stimuli like dynamic impacts, loads, stresses, accelerations appearing mainly during transport and operation of above materials and products.

Nowadays radiographic (X-ray) testing methods are used widely as the basic diagnostic methods to prevent the malfunction of above composite pyrotechnic items, but sometimes it is difficult to detect some types of defects by these methods. Therefore there are other NDT methods than X-ray techniques which can improve the detection of above mentioned defects[4-6].

IR thermography can be divided into the passive and active one. The passive methods are used to test materials and structures which have usually different temperature than ambient one while in the case of active methods an external or internal optionally thermal or cooling stimuli are necessary to induce relevant thermal contrasts. Because composite materials including pyrotechnic components should be safe for use at temperatures between - 40°C to + 55ºC esp. in military equipment used in climatic zones attributed to Poland[7], the application of active IR thermpgraphy as NDT method is potentially attractive for their testing.

3. Computer simulation

3.1. Programme

In order to determine the potential usefulness of thermal methods for non-destructive testing of composite pyrotechnic materials the computer simulations were carried out with the deployment of the specialized software ThermoCalc-6L™ (developed by V.Vavilov for needs of Military Institute of Armament Technology in Poland - MIAT). The ThermoCalc-6L™ software is intended for calculating three-dimensional (3D) temperature distributions in anisotropic six-layer solid bodies which may contain up to nine subsurface defects. The corresponding mathematical heat conduction problem is modeled in Cartesian coordinates and solved by using an implicit finite-difference numerical scheme. Originally, ThermoCalc-6L™ was developed for simulating thermal nondestructive testing (NDT) problems where transient temperature signals over subsurface defects are of a primary interest. These signals evolve in time and diffuse in space. The unique numerical algorithm implemented in ThermoCalc-6L™ which unlike to currently available commercial software, enables the modeling of very thin defects in rather thick materials without loosing the computation accuracy. It allows to analyze up to nine defects within a specimen being heated uniformly or non-uniformly with a square or cosine shape of heat pulse that gives possibilities to study defect cross-influence and lateral 3D heat diffusion.

ThermoCalc-6L™ is suitable to find the solution of transient heat conduction problem for a six-layer parallelepiped-shaped body that contains up to nine parallelepiped-shaped defects. The body is heated or cooled down on the front surface with an external 'heat' pulse. The front-surface heat flux is assumed to be uniform or Gaussian-distributed in space. The heat flux center can be located at any point on a front surface. Along with the external thermal stimulation of the front surface, both the front and rear surfaces are cooled down according to the Newton's law. Thermal properties of the specimen and the defects can be specified separately in three spatial directions, thus modeling *fully anisotropic* material. The specimen side surfaces are adiabatic. On the boundaries between the specimen layers and between the host materials and the defects, the temperature and heat flux continuity conditions take place. The conception of the so-called *capacitive* defects is realized in ThermoCalc-6L™. This means that, unlike *resistive* defects involved in some other NDT models, both defect thermal diffusivity and conductivity are taken into account. This provides the most correct description of physical phenomena occurring in the spaces occupied by defects.

The mathematical description of the program is given with the following expressions.

1. 3D parabolic equation of heat conduction;

$$\frac{\partial T_i(x,y,z,\tau)}{\partial \tau} = \alpha_i^x \cdot \frac{\partial^2 T_i(x,y,z,\tau)}{\partial x^2} + \alpha_i^y \cdot \frac{\partial^2 T_i(x,y,z,\tau)}{\partial y^2} + \alpha_i^z \cdot \frac{\partial^2 T_i(x,y,z,\tau)}{\partial z^2};$$

$$i = 1 \div 15 \quad (six \quad layers \quad + \quad nine \quad defects); \tag{1}$$

2. The initial condition;

$$T_i(\tau = 0) = T_{in}; \tag{2}$$

3. The boundary condition on a front surface (heating + cooling);

$$-K_1^z \cdot \frac{\partial T_1(x,y,z=0,\tau)}{\partial z} = Q(x,y,\tau) - h_F \cdot [T_1(x,y,z,\tau) - T_{amb}]; \tag{3}$$

4. The boundary condition on a rear surface (cooling only);

$$K_3^z \cdot \frac{\partial T_3(x,y,z=L_z,\tau)}{\partial z} = -h_R \cdot [T_3(x,y,z,\tau) - T_{amb}]; \tag{4}$$

5. The adiabatic conditions on side surfaces determined by coordinates x and y;

$$\frac{\partial T_i(x,y,z,\tau)}{\partial x} = 0 \quad \text{for } x = 0, y = 0 \div L_y; x = L_x, y = 0 \div L_y; \tag{5}$$

$$\frac{\partial T_i(x,y,z,\tau)}{\partial y} = 0 \quad \text{for } y = 0, x = 0 \div L_x; y = L_y, x = 0 \div L_x;$$

6. Temperature and heat flux continuity conditions on the boundaries between the layers and between the layers and the defects;

$$T_i(x,y,z,\tau) = T_{i\pm1}(x,y,z,\tau) \text{ and } K_i^{q_j} \cdot \frac{\partial T_i(x,y,z,\tau)}{\partial q_j} = K_{i\pm1}^{g_j} \cdot \frac{\partial T_{i\pm1}(x,y,z,\tau)}{\partial q_j} \tag{6}$$

where:

T_i is the temperature in the i-th region counted from the initial object temperature (i=1÷6 corresponds to specimen layers, i=7÷15 corresponds to nine defects);

T_{in} is the specimen initial temperature;

$\alpha_i^{q_j}$, $K_i^{q_j}$ are the thermal diffusivity and the thermal conductivity in the i^{th} region by the coordinate q_j;

x, y, z are the Cartesian coordinates; q_j is one of the Cartesian coordinates x, y or z (j=1÷3);

τ is time;

$Q(x,y,\tau)$ is the heat flux power density that in a general case, varies both in time and space;

h_F, h_R are the heat exchange coefficients on the front and rear surfaces respectively;

T_{amb} is the ambient temperature;

L_x, L_y, L_z are the specimen dimensions.

3.2. Models

In order to analyze a possibility of nondestructive testing by IR thermography methods used for detecting defects in composite pyrotechnic materials two models of a heterogeneous solid propellant with an inhibitor layer (coating) in the form of rectangular prism plate were simulated. The size of the model plates were 30x50 mm and 20 mm thick (layer of inhibitor was 2 mm thick). In Model 1 (Fig. 1) there were simulated two defects parallel to the surface of the sample: Defect 1 (D1)size was10x10 mm and 0.5 mm thick and Defect 2 (D2) size was 5x20 mm and 0.5 mm thick. These defects simulate delaminations in the layer of inhibitor and between propellant and inhibitor. The defects are located at different depths. In Model 2 (Fig. 1) there were simulated two defects perpendicular to the surface of the sample: Defect 3 (D3) size was 30x0.1 mm and 0.5 mm thick and Defect 4 (D4) size was 30x1 mm and 0.5 mm thick. These defects simulate cracks in layer of inhibitor and were located at different depths. Computer simulations were performed by means of the ThermoCalc-6L software. Table 1 shows thermal data of materials and the air (which simulates a defect) used for the computer simulation.

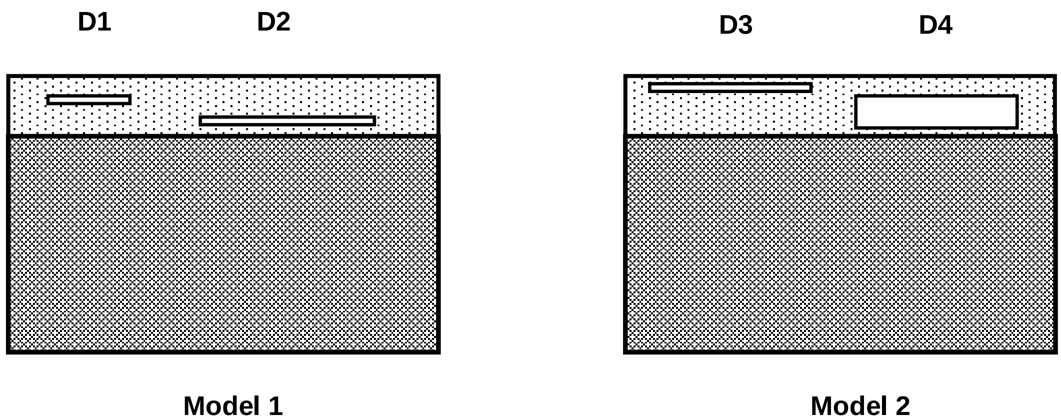

Fig.1. Models of samples

Material	Specific heat kJ/kgK	Thermal conductivity W/mK	Specific density kg/m^3
Propellant	1255	0.29	2000
Inhibitor	1884	0.33	1100
Air (thin gaps)	1.005	0.07	1.2

Table 1: Thermal data for tested/used materials

3.3. Results

It was introduced a model of samples (Fig.1) to simulate heating stimulations by computer programme. The heating of all samples was modeled in three variants. In the first variant of heating it was applied a thermal pulse on the front surface of the sample with pulse heat duration $\tau_h = 0.01$ s and power density $Q = 3{\cdot}10^5$ W/m^2. In the second variant the sample was heated to 50°C and the cooling was simulated to ambient temperature (20°C). The cooling process was going on through a "normal" convection. A specimen was continuously cooled by the ambient air. In the third variant the sample was also heated to 50°C but at the beginning of the convection process a cooling pulse was used. In this simulation two kinds of cooling pulse were used, in the first case the cooling pulse duration was $\tau_c = 2$ s and power density of cooling pulse was $Q = 5 \cdot 10^3$ W/m^2, and in the second case the cooling pulse duration was $\tau_c = 10$ s and power density of cooling pulse was $Q = 10^3$ W/m^2. Selected results of optimum parameters for detection of defects D1-D4 can be calculated as shown in Table 2-4 respectively.

Method	Defect	C, %	τ_m, s	ΔT, °C
Heating pulse	D1 D2	68 55	13.5 11.4	0.37 0.33
Convection	D1 D2	8 6.9	35 33	-3.0 -2.49
Cooling pulse $\tau_c = 10$ s $Q = 10^3$ W/m^2	D1 D2	15 14	14.2 13.6	-5.37 -4.47
Cooling pulse $\tau_c = 2$ s $Q = 5 \cdot 10^3$ W/m^2	D1 D2	11 10	8.7 7.6	-4.47 -4.01

Table 2. Expected detection parameters in the front-surface (defects at depth of 1mm)

Method	Defect	C, %	τ_m, s	ΔT, °C
Heating pulse	D1 D2	31 23	28,8 25.5	0.15 0.12
Convection	D1 D2	4.8 3.6	73 70	-1.57 -1.19
Cooling $\tau_c = 10$ s $Q = 10^3$ W/m^2	D1 D2	6.9 5.4	25.4 23.1	-2.67 -2.07
Cooling $\tau_c = 2$ s $Q = 5 \cdot 10^3$ W/m^2	D1 D2	3.4 2.6	23.5 20.5	-1.47 -1.14

Table 3. Expected detection parameters in the front-surface (defects at depth of 2 mm)

Method	Defect	C, %	τ_m, s	ΔT, °C
Heating pulse	D3 D4	67 12.7	0.36 8.23	-4.84 0.11
Convection	D3 D4	6 2.1	10 32	-2.55 -0.64
Cooling $\tau_c = 10$ s $Q = 10^3$ W/m^2	D3 D4	-11 -4	1 12.9	-3.62 -1.35
Cooling $\tau_c = 2$ s $Q = 5 \cdot 10^3$ W/m^2	D3 D4	-7 -3	0.3 6.5	-1.99 -1.12

Table 4. Expected detection parameters in the front-surface (D3 and D4)

It was assumed that a defect can be reliably detected by its surface temperature “footprint” if the corresponding sample excess temperature T and the signal ΔT meet the following conditions:

- a sample maximum excess temperature $T(\tau_h)$ that occurs at the end of heating is lower than the destruction temperature of the sample material T_{destr} (+ 55ºC);
- a ΔT signal must exceed a temperature resolution of a used IR system ΔT_{res};
- a running temperature contrast $C = \Delta T(\tau)/T(\tau)$ must exceed the noise level that adheres to each material and surface condition (up to 2%).

Assuming that T_{destr} = + 55ºC, $\Delta T_{res} = 0.1^oC$ and $C_n = 2\%$ and applying the detection criteria to the data in given in Table 2-4, it can be stated the following:

- the sample maximum surface temperature will not exceed 50°C;
- all defects produce $\Delta T > 0.1$°C and $C > 2\%$.

4. Experimental Testing

4.1. Method

The nondestructive IR thermography testing method was selected by taking into consideration the results of computer modeling. Method of natural convection was chosen to initial experimental investigations because this type of composite material was tested the first time. The sample have a cylinder shape, 20 mm diameter and 100 mm length. Pyrotechnic material was covered by a layer of inhibitor that is about 1.5 mm thick. The sample of composite pyrotechnic material was conditioned in climatic chamber at + 50°C for 3 hours. After heating whole structure of the sample has the same temperature + 50°C. Next the sample was taken out from the chamber and changes of temperature on the surface of the sample were recorded by IR camera AGEMA 900 LW. The changes of temperature were recorded until the sample achieved the ambient temperature.

4.2. Results

Fig.2 and 3 show selected results obtained by nondestructive testing of a composite pyrotechnic material sample possessing defects. Fig. 2 presents X-ray image of the sample with a detected defect between inhibitor and pyrotechnic material. Fig.3 shows thermogram of this sample with a detected delamination. In the thermogram it is visible the position, size (area) and shape of this delamination. These details are not visible on the X-ray picture.

5. Conclusions

(1) Results received from the computer simulation as well as preliminary experimental testing have shown that active IR thermography is promising method and sometimes more efficient in detection of some kind defects and their size assessment in comparison with radiography (X-ray) methods because of better detection of surface and subsurface defects particularly in the inhibitor layer and in the region of boundary surfaces i.e. between inhibitor and propellant materials.

(2) The computer simulations showed, that the largest values of temperature contrast are used for the stimulation of material by thermal pulse. However this method requires a precise selection of the thermal pulse power density to prevent the growth of temperature on the surface of material over the critical value i.e. below temperature of thermal decomposition of tested material and/or even its ignition .

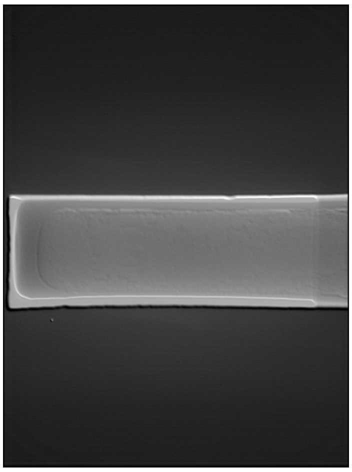

Fig.2. X-ray image of tested gas-generator sample

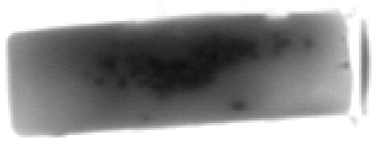

Fig.3. IR thermogram of tested gas-generator sample

(3) The surface cooling method provides both the shorter time to reach the best conditions to detect defects as well as it gives greater value of temperature contrast when using "natural" convection method. However surface cooling method requires further investigations dealing with power density and types and rates of cooling to avoid any generation of defects by sharp changes or local surges of temperature.

(4) Taking above into consideration the "natural" convection method was applied for preliminary experimental testing. Future investigations will be focused on the improvement of recognition of these methods used to specific types of composite pyrotechnical material.

6. Acknowledgements

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