

Critical Review of the Methods to Measure the Condensed Systems Transient Regression Rate

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Abstract

Accurate knowledge of steady state and transient burning rate of solid fuels and energetic materials is very important for evaluating the performance of different propulsion and/or gas generator systems. The practical demands imply accuracy of available burning rate data on the level of 1% or better and proper temporal resolution. Unfortunately, existing theoretical models do not allow predicting the magnitude of the burning (regression) rate with needed accuracy. Therefore, numerous burning rate measurement methods have been developed by various research groups over the world in the past decades. This paper presents a critical review of existing techniques, including basic physical principles utilized for burning rate determination, an estimate of the temporal and spatial resolutions of the methods as well as their specific merits and limitations. There are known the methods for measuring linear regression rate via high speed cinematography, X-ray radiography and ultrasonic wave reflection technique. Actually, none of those methods could satisfy the practical demands. As an alternative is the microwave reflection method, which potentially possesses high spatial and temporal resolutions and may solve the measurement problem. In addition, there exist methods for measuring transient mass or weight of the burning material. They are based on recording the frequency of oscillations of elastic element with attached specimen or a cantilevered rod with a strain gauge pasted to the base. Practically, these methods could not provide needed accuracy. Much better parameters can be obtained when using the recoil force or microwave resonator techniques. Recommendations for special applications of certain methods are formulated.

1. Introduction

When designing different propulsion devices, it is necessary to know the burning rate (linear regression rate) of the energetic material and its dependency on the pressure and initial temperature. In particular, upon designing solid-propellant rocket motor, the error in the propellant stationary burning rate should not exceed 1%. Consequently, accurate determination of the burning rate and its functional dependency is highly critical. When going to transient burning rate measurements, due to great technical difficulties the needed accuracy can be slightly decreased but it is necessary to provide proper temporal resolution, for example, 1 kHz and higher.

These requirements were formulated many years ago in review [1] and main conclusions of it were confirmed later in review [2]. Note that at present time none of the theoretical models is able to predict the burning rate with the accuracy required because detailed physical and chemical mechanisms of transformations occurring in the reaction zones above and below the burning surface are not fully understood and the values of the energetic materials characteristic parameters at high temperatures are unknown. Thus, it is quite evident that the development of reliable experimental methods for accurate measurement of the energetic material regression rates is extremely desirable to satisfy the stringent demands in the design of propulsion systems.

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This paper summarizes the characteristics of various burning rate measurement techniques available in the literature. A comparative analysis of the different techniques is presented, and recommendations for the application of these methods are provided.

2. Experimental methods for measuring transient burning rates

For transient regression rate measurements several experimental approaches are often employed.

2.1. Measurement of the instantaneous web thickness

One of the broadly used methods for measuring the instantaneous web thickness is based on high-speed cinematography of the transient combustion event. Despite the apparent simplicity of the method, its use for oscillatory combustion situations can produce significant error, which substantially sets the limit to their application. A spatial resolution associated with this technique is approximately 10–20 μm . If one wishes to measure the instantaneous burning rate under oscillating pressure and/or heat flux conditions, a much higher spatial resolution is required. This point can be illustrated by considering a simple example with the following conditions: nominal burning rate of 1 cm/s, frequency of oscillation of 50 Hz, and the amplitude of burning rate fluctuation of 20%. During a half period of oscillation (0.01 s), the burning surface regresses at average distance of $100 \pm 20 \mu\text{m}$. A spatial resolution of 10–20 μm translates into an error of 50–100% in the measurement of the burning rate fluctuation. This error is clearly unacceptable for the conditions stated. In many combustion instability studies, the frequencies of oscillation are often higher than 50 Hz; therefore, the cinematography methods are inadequate for oscillatory burning rate measurements.

For studying the combustion behavior under high-pressure environment, real-time X-ray radiography coupled with high-speed movie/video cameras has been used [3, 4]. This technique is especially useful for studying combustion processes in closed vessels. In this method, a continuous X-ray beam is used to penetrate the propellant sample and its enclosure. The attenuated beam strikes on an image intensifier which transforms the X-ray images into visible light images. These images are recorded on a high-speed, high-resolution video

camera. The recorded images are then analyzed using an advanced image processor to determine instantaneous burning surface locations, grain motion as well as any anomalous behaviors, such as grain fracture. The time resolution of this system depends upon the framing rate of the camera. The spatial resolution depends on many factors, such as 1) the relative attenuation of the combustion chamber walls and the propellant grain, 2) the ratio of the distance between the X-ray source to the object and the distance between the source and the screen of the image intensifier, 3) the focal spot size of the X-ray source, and 4) the magnification scale of the image intensifier. The spatial resolution of real-time X-ray radiography is on the order of 100 μm .

In addition to direct photographic techniques, optical projection methods have been used for the measurement of instantaneous length of a burning propellant sample in a windowed bomb [5]. The sample image is focused by optical lenses on an array of photodiodes which is masked by series of pin holes (0.5 mm in diameter) to increase the spatial resolution. As the propellant regresses, light from the flame illuminates a larger number of photodiodes. The time response of each photodiode is recorded and analyzed for the determination of the time variation of the burning surface location. The burning rate is then deduced by the use of a linear regression analysis. The spatial resolution of this method is estimated to be 100 μm .

The instantaneous burning surface location can also be determined by ultrasonic wave reflection methods [6–10]. The first experiments using ultrasonic waves for measuring transient burning rates of solid propellants were performed in the USA [6] and later the ultrasonic wave reflection technique was further developed by researchers in Europe [7]. The principle of the method is to measure the time elapsed between an emitted sound pulse and its echo generated from the reflection at the burning surface. The pulse repetition frequency can be as high as 5 kHz. Further, the known value of the sound velocity is used to deduce the distance traveled by the ultrasonic wave. Numerical differentiation of the distance vs. time data generates the information of burning rate vs. time. Both the ultrasonic emitter and receiver are located in the unburned base of the sample. In this setup the ultrasonic wave first propagates through the cold unheated substances (consists of the propellant and coupling material located between the ultrasonic transducer and propellant) and then travels through

the higher temperature region composed of the pre-heat and reaction layer.

One of the uncertainties of this technique is associated with the unknown solid-phase thermal wave profile under transient conditions. Since the speed of sound is a function of temperature, the ultrasonic wave accelerates as it approaches the burning surface. Difficulties can also appear due to distortion of reflected wave signal upon passage through the boundaries of phase transitions and gas-filled reacting layers (foam layer). Anisotropy of mechanical characteristics of the propellant material and the dependency of sound velocity on the stress level in the propellant material also introduce some uncertainties. Errors in determining the precise time at which the echo wave returns to the sensor are caused by the non-ideal shape of reflected signal and the occasional weakening of signal intensity. For a nominal sound speed of 2.5 km/s in a solid propellant sample with a frequency of ultrasonic source of 2.5–5 MHz, an error of 1/4 of the oscillation period corresponds to an error of 100–500 μm for determining the burning surface location. For relatively slow transients in combustion conditions, the accuracy of this method is estimated to be 5–10% [9]. It is difficult to estimate the accuracy of highly transient regression rate measurements using ultrasonic methods.

The microwave reflection method of measuring the instantaneous propellant web thickness is at present the most accurate one; however, its setup and operation are technically complex. The initial development of this method, based on the measurement of the Doppler frequency phase shift between the initial 30 mm band microwave signal and that reflected from the burning surface of the solid propellant, was published in 1967 [11]. Afterwards, interest in this technique stimulated subsequent work with further developments [12–14]. Phase resolution can be enhanced via using shorter microwave wavelength or employing interferometry [12] when the electronic setup includes two klystrons operating at frequencies (10 GHz) and (10 GHz + 500 kHz) and a pair of double balanced mixers. In this setup, any drifting of klystron difference is self-compensated in the two mixers and the phase resolution of the recording system is within 0.08–0.16 milliradian. This corresponds to a spatial resolution of about 0.2 μm allowing a very detailed measurement of the instantaneous burning rate, which is a much more accurate than other methods. The potential limitations of the system are caused by the influence of propellant compress-

ibility, signal distortion resulting from the reflection of electromagnetic waves from the ionization zone in the flame, noises generated from vibrations of the test stand.

It was experimentally verified that under small and rapid perturbations of pressure, the error caused by the effect of propellant compressibility is negligibly small, and the vibrations can be minimized to a suitable level by a special design of the experimental test rig. In addition, it was experimentally demonstrated [12, 14] that the distortions of signal due to the reflection from flame zone are insignificant.

Methodical problems of burning-rate measurements with use of microwaves are discussed in [15, 16]. It has been shown that the microwave meters can be successfully used in measurement of burning rate of solid propellants. The 2-mm band microwave meter can be applied for testing non-metallized and weakly metallized propellants. The 8-mm band microwave meter can be used for testing the propellants with the metal content up to 20% by mass.

2.2. Measurement of instantaneous mass and weight of the burning material

The instantaneous mass of the burning specimen can be measured using a well-known physical principle according to which the period of resonance oscillations (or natural frequency) of an elastic element depends on its mass, physical dimensions and mechanical properties. This method can be implemented using a vibrating mechanical element with an attached mass (propellant specimen), an electronic data acquisition system to record the harmonic motions of the assembly, and an electromagnetic actuating device to sustain non-decaying oscillations. Several different versions of this device were utilized by various researchers. In Ref. [17], a mechanical element has a form of a thin metallic membrane with a diameter of 50 mm. The propellant specimen was located on a rod attached to the center of the membrane. In Ref. [18], the mechanical element represented a cantilevered quartz rod with a diameter of 8 mm; the propellant specimen was fastened at the free end of the rod. The nominal natural frequency of oscillation of these two systems was 1 kHz. The propellant samples studied had a nominal mass of about 100 mg.

Very small masses of gasifying solid propellants were measured in Ref. [19] using a mechanical element in the form of a cantilevered quartz tube; a

stainless steel tip-end was attached to its free end. A specimen of energetic material with 1 mg mass was affixed (thin layer of 20–60 μm) to the tip-end. The eigenvalue frequency of cross oscillations of the quartz tube was 130 Hz. The data were collected and processed using a personal computer.

In general, the choice of the resonance frequency depends on the accurate measurement of oscillation period and the desired temporal resolution, which should be at least two times higher than the characteristic frequency of the process under study. In addition, there is a vague physical restriction: an oscillating propellant specimen must retain its mechanical characteristics (Young's modulus) during transient combustion. Otherwise, these measurements become questionable as was demonstrated by our experiments on the transient combustion of sodium nitrate based pyrotechnic mixtures which form a thick liquid layer on the combustion surface. This layer prevents correct determination of the mass of the oscillating specimen.

The weighing methods technical implementation has its own peculiarities. The existing methods display very low temporal resolution, although, very small specimens can be utilized with this method. In Ref. [20], a method was described for the continuous weighing of a 1–2 mg specimen. The sensing element used was a cantilevered rod with strain gauges pasted to the base. The propellant specimen was located at the free tip-end. The natural frequency of oscillations of the system was 120 Hz, and the weight sensitivity was 10–20 mg. The apparatus can be used to study processes whose frequency is no more than 30–50 Hz. The sensitivity and frequency range of the method can be substantially increased by using more sensitive strain gauges and data acquisition system. An improved design of the force transducer is given in [21]. The solid propellant specimen was fastened to the upper tip-end of a movable electrode which was attached to the inner surface of the gauge case by a pair of metallic membranes. The change in specimen weight produced a change in the distance between the movable electrode and the stationary base which in turn causes a change in the capacitance of a condenser. The condenser was a part of an oscillating inductance-capacitance (LC) circuit. A data acquisition system was used to record signals that were proportional to the specimen weight and its temporal derivative. A liquid damper was used to damp the natural oscillations of the mechanical system. The nominal weight of the specimen was 1 g, and the sensitivity was 2 mg. The

eigenvalue frequency of the system was 500–600 Hz, and the working frequency band was 0–400 Hz. In order to decrease the temperature errors, all metallic elements of the gauge were produced from Invar alloy with a very low thermal expansion coefficient.

The reaction forces produced by the combustion products can amount to tens of percent of the measured weight and can be a substantial source of error in the measurement of the instantaneous burning specimen weight. The influence of the reaction forces can be adequately subtracted from the data reduction procedure by placing the specimen in such a way that the reactive force vector is directed in the gravitational direction. In this arrangement, only an occasional variation in the direction of gasifying products from the burning surface can slightly affect the value of the instantaneous specimen weight.

2.3. Indirect methods for burning rate determination

The instantaneous mass of the burning propellant can be determined by an indirect method when the amount of unburned substance is deduced from the electrical capacity of the specimen in Hermance's capacitance method [22]. In his set-up, two opposing lateral surfaces of a rectangular propellant strand were covered with a combustible metallic foil, serving as the facing plates of a planar capacitor. The regressing propellant specimen, acting as a variable capacitor, was mounted in parallel with a capacitor and an inductor of known characteristics in an L-C circuit. When driven by an alternating current, the change in the resonance frequency of the L-C circuit gave the change in the value of variable capacitance. Therefore, the instantaneous length and the burning rate of propellant sample were deduced. One of the main errors of this method was caused by the unknown dependency of flame/plasma conductance on pressure [23]. According to a detailed analysis [24], special studies are necessary for estimating the real contribution of the flame conductance dependency on both the propellant formulation and the pressure level. It is suggested that the relative flame contribution can be decreased by increasing the resonance frequency of the L-C circuit. According to Hermance [22], the error analysis showed at least 10% error in the measurement of the instantaneous burning rates under oscillating pressure conditions (0.1–1.0 kHz).

Pressure diagram is another indirect method for determining transient burning rates at oscillating pressure conditions can be achieved by solving a set of equations describing the unsteady behavior of interior ballistics of rocket motors [25, 26]. In this method, the instantaneous chamber pressures at different locations should be accurately measured. The measured p-t traces are used as input information to the theoretical model for deducing the instantaneous burning rates. Essentially, the burning rates are solved in an inverse problem from the formulation and measured pressure-time data [26]. However, the accuracy and reliability of this method depend strongly upon the appropriateness of a large number of assumptions; e.g. spatially uniform distribution of pressure and temperature in the chamber, the absence (or formal account) of heat losses, the constancy of gas composition.

Reactive (recoil) forces generated from the combustion products gasifying from the burning surface were first measured by Muhlfeith, et al. [27]. They measured the response of the burning rate of a solid propellant to the perturbations of thermal radiation flux. Subsequently, their method was used in similar experiments by other researchers [21, 28–31]. The method is based on the relationship derived from the steady-state momentum equation:

$$F = mp^2/\rho_g$$

In equation the propellant recoil force, F , is directly proportional to the square of mass burning rate mp and inversely proportional to the gas density at the flame temperature, $\rho_g = PM_w/R_u T_f$. Assuming that the gas density is approximately constant during the experiment, the reactive force permits a reasonable estimation of the variations in mass burning rate.

The application of the recoil-force transducer (Fig. 1) considerably enlarges the volume of experimental data on transient burning rate behavior. The capacitor type transducer possesses force measurements with limit value of measuring force 5 g, and working frequency range 0–500 Hz.

The reactive force signal is recorded by turning the transducer axis into a horizontal position. In this manner the reactive force acts along the axis of a movable electrode. The weight of the specimen is compensated by the reaction of supports (membranes) which allows the measurements of sufficiently long (up to 10–20 mm for 10 mm diameter) specimens without losing the apparatus sensitivity. The gage sensitivity is 1–3 mg.

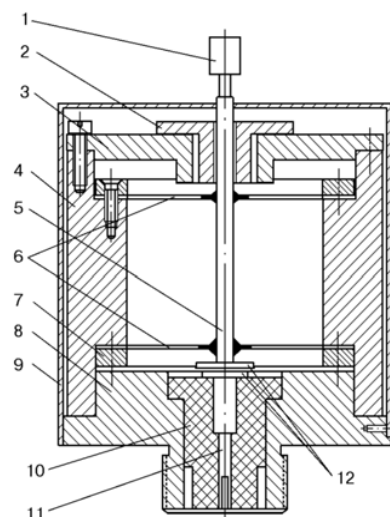


Fig. 1. Recoil-force transducer [21]: 1 – propellant sample; 2 – bushing for liquid damping; 3 – flange; 4 – case; 5 – movable rod; 6 – strings (membranes); 7 – pressing ring; 8 – flange; 9 – protective screen; 10 – insulating bushing; 11 – connective electrode; 12 – capacitor plates.

Note that the information obtained corresponds to the signal averaged over the burning surface, and for some cases, additional tests are needed to decipher the information. For example, the reactive force signal in the combustion of noncatalyzed double-base propellant in air exhibits frequencies of 100–200 Hz, which are much higher than those of the natural frequency of oscillations of burning rate (15–20 Hz) associated with periodic perturbations of radiant energy flux. The use of high-speed cinematography testifies to the chaotic appearance and disappearance of hot spots with high intensity chemical reactions on the burning surface. The simultaneous existence of several hot spots on the surface can generate complex spectra of the reactive force signals. The characteristics of the reactive force spectrum for a given propellant depend on the active reaction sites on the burning surface.

Specific features of registration of the recoil force using various types of transducers were discussed in [31]. The influence of various factors such as instrumental distortions, variable parameters of the ambient medium, and inhomogeneity of combustion was analyzed. It has been concluded that the method of registration of the recoil force for burning-rate measurement with the help of prior experimental calibration and visual control is preferable.

Special measures should be taken to protect the transducer from the vibrations and the thermal action produced by equipment and combustion

products, since these factors significantly affect the quality of the registered signal. These measures favor unique interpretation of the recoil-force signal. Finally, it can be noted that calibration is not necessary to obtain time characteristics such as the ignition delay, the combustion duration, the frequency of oscillations, or the phase shift. Thus, if the above measures for organization of an experiment are fulfilled, the method for registration of the recoil force can be effectively used to measure unsteady characteristics of the burning rate.

3. Comparison of measurement methods

The applicability of a prospective measurement method to a specific problem can be assessed by analyzing the characteristic parameters of both the instruments and the physical operating conditions. For all transient combustion events, the time resolution is the universal parameter to be considered in burning rate measurements. According to physical principles, any phenomenon can be detected by a recording system whose working frequency is at least two times higher than that of the physical phenomenon. Thus, the processes in the condensed phase with frequencies up to 500–600 Hz can be studied with a minimum time resolution of the recording system of about 1 ms. That requirement can be satisfied by the methods of cinematography, microwave probing, and the method of measuring instantaneous propellant mass. The method of measuring reactive forces can also be utilized in this range of operation. In fact, by decreasing the sensitivity of the force transducer, one can easily achieve a suitable time resolution.

The sensitivity of a given method can be characterized through the determination of its spatial resolution. As already mentioned, the microwave method has the highest spatial resolution (better than 1 μm) among all techniques. The spatial resolution of other methods, including the ultrasound, optical and weighing methods, is at least an order of magnitude lower. For a fixed weighing sensitivity, the resolution of the burnout layer thickness can be increased by enlarging the burning surface area of the specimen. However, this causes the increase in the total weight of the specimen and complicates the isochronic ignition over the surface. Therefore, the method for recording the reactive force is sometimes preferable, since its signal is directly related to the square of the burning rate value and its treatment does not involve differentiation of the signal value. However, there exist the problems

in application of recoil method for measuring the burning rate of real propellants. Several researchers revealed that theoretically predicted square dependency is not realized in static calibration runs and burning rate exponent may decrease up to the value of 1.2–1.5 [30, 32].

Various measurement methods provide different degrees of averaging over the burning surface. It is evident that the methods of weighing and those for measuring the reactive force give information which is averaged over the entire burning surface. Deviation from the nominal burning surface area can certainly introduce errors into the deduced values of the linear burning rate. The methods of ultrasonic and microwave probing allow one to extract information from a definite size burning area, but the optical methods are mainly meant for the measurements at a certain point or along a particular line. Thus, the question of inconsistency arises when comparing the data obtained via using different methods. The solution of these problems often requires the detailed knowledge of the combustion mechanism. The reliability and accuracy of the measurements of regression rate using various techniques depend on the knowledge of the mechanical and physical characteristics of the substance in question. For example, the thermal expansion of the substance and its deformation due to the effect of alternating pressure introduce errors in the measurement of the regression rate recorded by the change of specimen length. In this family of techniques, the error in the measurement of the instantaneous burning rate is of the order of tens of percent. The interpretation of the data of the ultrasonic method substantially relies on the knowledge of the dependency of sound velocity in the condensed substance on temperature, phase state, and chemical composition. Even empirically this information is difficult to obtain because the experiments involving reacting exothermic substances usually require a special technique with a fast response and a high sensitivity. A similar situation is observed for the microwave method. In this case, one also must know the dielectric constants of the solid fuel being studied and the coupling material as well as the coefficients of microwave signal reflection from the boundaries of different media. Thus, some claims of measurement accuracy are still debatable [33]. Only through the analysis of combined information of the substance properties and physical principles of measurements, one can reliably estimate and substantiate the accuracy of a given method.

4. Conclusions

The interest in the study of transient combustion of solid propellants in recent years has promoted the development of several new techniques for measuring instantaneous burning rates. No universal methods exist for measuring burning rates in broad range of operating conditions. The selection of a measurement technique depends on the nature of the problem and the availability of the particular instrument to the researcher.

The problems of the regression rate measurement in transient combustion conditions are generally categorized into small- and large-amplitude regression rate variations. For small-amplitude pressure oscillations, one anticipates small variations in regression rates. The problem usually becomes the measurement of the response function of burning rate to periodic oscillation of chamber pressure or heat flux at a known frequency. For this type of problem, either the microwave technique or reactive force measurement technique are preferable. When measuring large variations in burning rate associated with drastic changes in pressure or heat flux, it is desirable to use the method for measuring the instantaneous weight of the propellant sample in combination with the visualization of the combustion surface and flame behavior in order to interpret any non-uniform surface burning behavior. The flow visualization becomes especially important in transition regimes across which a slight change in test conditions can cause a drastic change in both the burning rate value and stability of the propellant flame. For high-pressure combustion environments with a large amplitude pressure excursion, the real-time X-ray radiography method is very attractive, since the history of surface re-

gression as well as grain motion and fracture can be clearly observed.

Some recently it was reported about novel microwave method for measuring transient mass gasification rate of condensed systems [34]. The microwave resonator method of dynamic measurement of mass of gasifying solid fuel samples is based on the measurement of the attenuation of a microwave signal passing through the resonator sensor (Fig. 2) loaded with investigated sample. Before firing experiments the sensor is calibrated via using samples of studied material having different channel radius.

The sensor is intended for measuring the instantaneous gasification rate of the samples of dielectric gasifying materials under intensive gas blowing with the space resolution about few microns and frequency resolution better than 1 kHz.

Finally, it should be noted that even though new breakthrough techniques using basic physical principles could still be developed in the future, it is believed that the further advancements in burning rate measurements will depend also upon improvement of the existing techniques.

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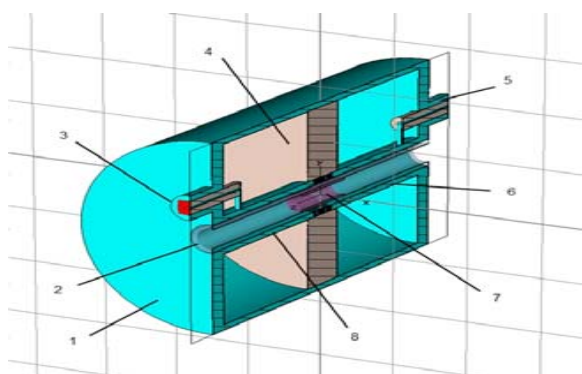


Fig. 2. The microwave resonator sensor: 1 – case; 2 – protective tube; 3 – input port; 4 – centering disk; 5 – output port; 6 – output resonator; 7 – sample; 8 – input resonator.

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