## DYNAMIC ERRORS IN NONSTATIONARY THERMOPHYSICS MEASUREMENT SYSTEMS

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A method is given for calculating the dynamic error in nonstationary thermophysical measurements as influenced by inertial characteristics.

Nonstationary methods are at present the best ones for examining thermophysical characteristics [1] primarily because the thermal parameters are determined together and the measurements are rapid. However, a nonstationary method involves an additional error source absent from a stationary one, which is due to the inertial characteristics in the primary sensor and in the measurement components. The measurement system here is a combination of devices providing signal amplification and transformation running from the sensor itself. There are fairly many papers such as [2, 3] on estimating dynamic errors in thermal measurements due to primary sensor lag, whereas there has been neglect of the selection or development of electronic systems to provide a given dynamic error. The topic is considered here. The approach is all the more necessary because there is a tendency in such experiments to reduce specimen and probe dimensions [1], which means that the transient thermal processes have elevated rates and thus there are tighter specifications for system dynamic characteristics.

In such measurements, the temperature sensors are often thermocouples or resistance thermometers. Their signals are very weak (tens of microvolts to a few millivolts), so the next device is usually an amplifier. When such low-voltage measurements are made, there is inevitably an error due to noise and interference, and the error is reduced by fitting a low-pass filter (LPF) after the amplifier [4], which suppresses interference and noise at frequencies much higher than the signal. One most often uses active filters, since they provide the required cutoff above the limiting frequency and the necessary uniformity within the passband.

Laplace transformation is frequently applied to the transient response [2, 3], and we have applied it to the dynamic error.

The essence is as follows.

The Laplace transform for the output signal is

$$U(s) = K_{ms}(s) T(s) = K_{ts}(s) K_{a}(s) K_{lpf}(s) T(s),$$
(1)

where  $K_{ms}(s)$  and  $K_{ts}(s)$  are the transfer characteristics for the measurement system and temperature sensor, while  $K_a(s)$  and  $K_{lpf}(s)$  are those for the amplifier and low-pass filter, with T(s) the Laplace transform for the input temperature.

There are several papers [2, 3] on temperature-sensor transfer-characteristic determination; methods based on heat sources [3, 5] may employ instantaneous, pulsed, or constant-output ones, where the sensor is located directly on the heater or the heater is at the same time the thermometer. Then the sensor lag is incorporated when one corrects for the heater thermal capacity.

The filter transfer characteristic can be derived, as for example, in [6].

It is somewhat more difficult to determine the amplifier transfer characteristic because it often contains not only several amplifying stages but also stages that transform the signal, e.g., by modulation and demodulation [7]. However, the amplifier is usually faster in response than the LPF, and thus a thermal transient lasting more than a few

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Fig. 1. Error  $\delta$  (%) introduced by the amplifier as a function of time  $\tau$  (sec) for various materials (w<sub>o</sub> = 200 W/m<sup>2</sup>, c<sub>h</sub> = 200 J/m<sup>2</sup>•K, r<sub>o</sub> = 10•10<sup>-3</sup> m, f<sub>t</sub> = 10<sup>6</sup> Hz, and G = 10,000): 1) rubber ( $\lambda$  = 0.05 W/m•k, a = 0.14•10<sup>-6</sup> m<sup>2</sup>/sec); 2) lucite (0.196 and 0.128•10<sup>-6</sup>); 3) glass (1.17 and 0.58•10<sup>-6</sup>).

Fig. 2. Error  $\delta$  (%) introduced by LPF as a function of time  $\tau$  (sec) for various materials (w<sub>o</sub> = 200 W/m<sup>2</sup>, c<sub>h</sub> = 200 J/m<sup>2</sup> •K, r<sub>o</sub> = 10 • 10<sup>-3</sup> m, f<sub>t</sub> = 10<sup>6</sup> Hz, G = 10,000, f<sub>co</sub> = 5 Hz): 1) rubber ( $\lambda$  = 0.05 W/m•k, a = 0.14 • 10<sup>-6</sup> m<sup>2</sup>/sec); 2) lucite (0.196 and 0.128 • 10<sup>-6</sup>); 3) glass (1.17 and 0.58 • 10<sup>-6</sup>).

i	Vi	ī	V <sub>i</sub>
1 2 3 4 5	$\begin{array}{c} 0,08333333333\\ -32,083333333\\ 1279,000076\\ -15623,66689\\ 84244,16946\end{array}$	6 7 8 9 10	$\begin{array}{r}236957,5129\\ 375911,6923\\340071,6923\\ 164062,5128\\32812,50256\end{array}$

TABLE 1. Values for the First Ten Coefficients

hundred milliseconds enables one to replace the amplifier transfer characteristic by that of a single-stage amplifier for estimates [8]:

$$K_a(s) = \frac{G}{1 + \frac{Gs}{2\pi f_*}},$$

where G is the gain, while  $f_t$  is the unit-gain frequency (the frequency at which the gain is one).

When one examines fast processes, one needs a refined derivation for the transfer characteristic or measurements on the lag in a particular amplifier, which can be approximated by a higher-order transfer characteristic.

These transfer characteristics and the input temperature transform are substituted into (1) to get the output Laplace transform; the dynamic error can be estimated for each component and for the system as a whole if one transfers from the Laplace transform to the time domain by inverse Laplace transformation, although the resulting expressions are very often cumbersome. Numerical inversion methods are then required, where we consider a detailed case as an illustration.

We examine the effects of component lag in determining thermal parameters by means of a circular source having a constant output [9].

The sensor (thermocouple) is mounted directly on the heater, so  $K_{ts}(s)T(s)$  can be put [10] as

TABLE 2. Transient Response in the Measurement System for Polymethylmethacrylate ( $w_0 = 200 \text{ W/m}^2$ ,  $c_h = 200 \text{ J/m}^2 \cdot \text{K}$ ,  $\lambda = 0.196 \text{ W/m} \cdot \text{K}$ ,  $a = 1.28 \cdot 10^{-7} \text{ m}^2/\text{sec}$ ,  $r_0 = 10 \cdot 10^{-3} \text{ m}$ ,  $f_{CO} = 5 \text{ Hz}$ ,  $f_t = 10^6 \text{ Hz}$ , and G = 10,000;  $A_1 = 1.8478$ ;  $B_1 = B_2 = 1.0$ ;  $A_2 = 0.7654$ )

τ, sec	<i>T</i> , K	r, K	<sup>U</sup> a, V	<sup>8</sup> a, %	Ulpf. V	åzpf'%
$\begin{array}{c} 0,1\\ 0,3\\ 0,5\\ 1,0\\ 2,0\\ 3,0\\ 5,0\\ 10,0\\ 15,0\\ 10,0\\ 15,0\\ 10,0\\ 10,0\\ 20,0\\ 30,0\\ 50,0\\ 100,0\\ 200,0\\ 500,0\\ 1000 0\end{array}$	$\begin{array}{c} 0,134383\\ 0,232758\\ 0,300489\\ 0,424956\\ 0,600979\\ 0,736045\\ 0,950231\\ 1,34383\\ 1,64585\\ 1,90046\\ 2,32736\\ 2,99906\\ 4,15323\\ 5,45438\\ 7,03860\\ 7,98383\\ \end{array}$	$\begin{array}{c} 0,0605105\\ 0,134973\\ 0,196090\\ 0,311967\\ 0,480831\\ 0,608126\\ 0,826055\\ 1,21756\\ 1,51676\\ 1,76926\\ 2,19019\\ 2,85455\\ 4,02753\\ 5,35411\\ 6,99062\\ 7,96209 \end{array}$	$\begin{array}{c} 0,0597915\\ 0,134376\\ 0,195633\\ 0,311639\\ 0,480591\\ 0,607939\\ 0,825922\\ 1,21744\\ 1,51668\\ 1,76918\\ 2,19013\\ 2,85450\\ 4,02750\\ 5,35409\\ 6,99062\\ 7,96209 \end{array}$	1,190,440,230,110,050,0300000000000000000	0,0132406 0,101618 0,170732 0,292730 0,468662 0,597119 0,818303 1,21253 1,51189 1,76546 2,18706 2,85172 4,02575 5,35301 6,99055 7,96200	78,1 24,7 12,9 6,07 2,53 1,81 0,94 0,41 0,32 0,21 0,14 0,10 0,04 0 0

$$K_{ts}(s) T(s) = \frac{w_0 \left[1 - \exp\left(-\sqrt{s} \frac{r_0}{\sqrt{a}}\right)\right] E}{bs \sqrt{s} + c_h s^2 \left[1 - \exp\left(-\sqrt{s} \frac{r_0}{\sqrt{a}}\right)\right]},$$
(2)

where  $w_0$  is the specific source power,  $r_0$  the heating spot radius,  $c_h$  the surface heat capacity, a and b are correspondingly the thermal diffusivity and thermal activity for the material, and E is the thermocouple's emf coefficient. It is assumed in (2) that the thermo-emf per degree remains constant for small excess-temperature increments in T(s) up to 10 K.

One might employ an F-7024S/1 amplifier, for which G = 10,000 and  $f_t \approx 10^6$  Hz. When this amplifier works with a thermocouple, it is found that the output voltage (signal) has a superimposed line-frequency interference (100 Hz), amplitude about 300 mV. To provide the required measurement error (about a microvolt), one needs at least a third-order active filter [6] providing a slope of -60 dB/dec. We selected a fourth-order Butterworth filter, since this provides good flatness in the passband and sufficiently steep cutoff. The transfer characteristic is [6]

$$K_{lpf}(s) = \frac{1}{\left(1 + A_1 \frac{s}{2\pi f_{co}} + A_2 \frac{s^2}{4\pi^2 f_{co}^2}\right) \left(1 + B_1 \frac{s}{2\pi f_{co}} + B_2 \frac{s^2}{4\pi^2 f_{co}^2}\right)},$$

where  $f_{co}$  is the cutoff frequency and  $A_1 = 1.8478$ ;  $A_2 = 0.7654$ ;  $B_1 = B_2 = 1.0$ .

Then the Laplace transform for the output signal is

$$U(s) = \frac{w_0 \left[1 - \exp\left(-\sqrt{s} \frac{r_0}{\sqrt{a}}\right)\right] EG}{\left[bs\sqrt{s} + c_{c0}s^2 \left(1 - \exp\left(-\sqrt{s} \frac{r_0}{\sqrt{a}}\right)\right)\right] \left(1 + \frac{Gs}{2\pi f_t}\right)} \frac{1}{(1 + A_1y + A_2y^2)(1 + B_1y + B_2y^2)}, \quad (3)$$

where  $y = s/2\pi f_{co}$ .

For convenience, E per degree Kelvin has been taken as 100  $\mu$ V/K, so EG = 1 V/K (temperature increment of 1 K causes an output voltage increase of 1 V).



The inverse transformation from (3) cannot be based on tables; numerical inversion is possible via the following algorithm [11]:

$$Fa = \frac{\ln 2}{\tau} \sum_{i=1}^{N} V_i P\left(\frac{\ln 2}{\tau} i\right),$$

where

$$V_{i} = (-1)^{\frac{N}{2}+i} \sum_{k=\left[\frac{i+1}{2}\right]}^{\min\left(i,\frac{N}{2}\right)} \frac{k^{\frac{N}{2}}(2k)!}{\left(\frac{N}{2}-k\right)!k!(k-1)!(i-k)!(2k-i)!},$$

and Fa is the approximate value for the original  $F(\tau)$ , with P(s) the transform of it.

The least error in the original is attained for N = 10 with ordinary computer accuracy [11]; Table 1 gives the values for the first ten  $V_i$ .

The algorithm was checked by reversing transforms for known originals, which showed that the reversal error may be as much as  $\pm 3\%$  for transforms analogous to (3). Increasing N and double-length working did not improve the accuracy much, so we recommend the following technique. It is necessary to invert only the corrections introduced by each element, and appropriate analytic expressions are required. For example, before one calculates the transient response after the sensor, one needs to obtain an expression representing the difference between the transform for the theoretical temperature—time dependence without correction for sensor inertia and that with it:

$$U_{ts}^{g}(s) = T(s) (1 - K_{ts}(s))$$

Similarly, before one calculates the transient response after the amplifier, one needs an expression for

$$U_{a}^{g}(s) = K_{te}(s) T(s) (1 - K_{a}(s))$$

In the same way, one can derive  $U_{lof}^{-g}(s)$ .

The transient-response curves are then obtained by subtracting the numerical inversion results for the corresponding corrections from the theoretical dependence; this improves the accuracy to 0.1-0.2% for N = 10, which is quite sufficient.

Table 2 gives calculations on the response for polymethylmethacrylate, where T is the temperature increment (from the initial value) at the central point in the circular heater without allowance for the heater's thermal capacity,  $T_{ch}$  is the excess temperature at that point with allowance for the heater's capacity,  $U_a$  is the amplifier output, and  $\delta_a$  is the error introduced by the amplifier,

$$\delta_{a} = \frac{T_{ch}GE - U_{a}}{T_{ch}GE} \cdot 100\%;$$
(4)

with  $U_{\text{lpf}}$  the output from the LPF,  $\delta_{\text{lpf}}$  the error introduced by the LPF,

$$\delta_{lpf} = \frac{T_{ch}GE - U_{lpf}}{T_{ch}GE} \cdot 100\%.$$
<sup>(5)</sup>

TABLE 3. Values for the Low-Pass Filter Dynamic Error for Various Filter Orders:  $w_0 = 200 \text{ W/m}^2$ ,  $c_h = 200 \text{ J/m}^2 \cdot \text{K}$ ,  $\lambda = 0.196 \text{ W/m} \cdot \text{K}$ ,  $a = 1.28 \cdot 10^{-7} \text{ m}^2/\text{sec}$  (polymethylmethacrylate),  $r_0 = 10 \cdot 10^{-3} \text{ m}$ ,  $f_t = 10^6 \text{ Hz}$ , G = 10,000, and  $f_{CO} = 5 \text{ Hz}$ 

τ, sec	First-order filter $A_2 = B_1 = B_2 = 0; A_1 = 1, 0$		Second - order filter $A_2=B_2=0; A_1=1,4142; B_1=1,0$		Fourth-order filter $B_1=B_2=0; A_1=1.8478; A_2=0.7654$	
	v <sub>lpf</sub> , v	<sup>8</sup> lpf•;%	Ulpf, V	°lpf•%	v <sub>lpf</sub> , v	<sup>8</sup> lpf• %
0,5 1,0 2,0 3,0 5,0 10,0 15,0 20,0 30,0 50,0 70,0	$\begin{array}{c} 0,187123\\ 0,305050\\ 0,476443\\ 0,604160\\ 0,823258\\ 1,21572\\ 1,51500\\ 1,76788\\ 2,18905\\ 2,85352\\ 3,39377 \end{array}$	4,57 2,22 0,91 0,65 0,34 0,14 0,12 0,08 0,05 0,04 0,02	$\begin{array}{c} 0,174245\\ 0,295559\\ 0,470440\\ 0,598746\\ 0,819452\\ 1,21327\\ 1,51262\\ 1,76602\\ 2,18752\\ 2,85214\\ 3 9291 \end{array}$	11,1 5,26 2,16 1,54 0,80 0,34 0,27 0,18 0,12 0,08 0,04	0,170732 0,292730 0,468662 0,597119 0,818303 1,21253 1,51189 1,76546 2,18706 2,85172 3,39265	12,9 6,07 2,53 1,81 0,94 0,41 0,32 0,21 0,14 0,10 0,05

These calculations show that the transient response in the amplifier is the same as that in the filter, and after a certain instant, the effects can be neglected, with the LPF introducing much more error than the amplifier.

To reduce the lag effects from the electronic components, one has to exclude the initial part of the temperature curve, over a length determined by calculation.

As the error due to the thermal capacitance in the heater can be corrected [10], we consider only the additional error introduced by the electronic components relative to the transient response after the sensor (in our case, relative to the transient-response curve with allowance for the heater's capacity).

Figures 1 and 2 show the time dependence of the errors introduced by the amplifier and LPF.

The values in Figs. 1 and 2 have been derived from (4) and (5).

These calculations show that the errors due to the electronic-device lag for a given measurement time are somewhat larger for materials showing better insulation, because of the steeper temperature curve and consequently the presence of higher-frequency components.

Figure 3 shows the error introduced by the LPF as a function of time for various cutoff frequencies for polymethylmethacrylate as derived from (5). One can thus choose the optimum cutoff frequency as regards error. In our case, it is sufficient to have a cutoff frequency not more than 5 Hz for measurements after 5 sec (this provides a measurement error not more than 1%).

Table 3 shows that the filter order (damping) affects the error much less than the cutoff frequency, so it is undesirable to use an order above the fourth, as this increases the filter size without reducing the error substantially.

Filter error estimation should always be accompanied by error estimation for the noise after the filter. In our case, the fourth-order Butterworth filter had an input noise amplitude of 300 mV, absolute noise error  $\delta_n = 0.3$  K for thermo-emf of 100  $\mu$ V/K, with  $f_{co} = 10$  Hz giving a noise amplitude of 8 mV at the filter output,  $\delta_n = 0.08$  K; with  $f_{co} = 5$  Hz, the output noise amplitude is 1.5 mV and  $\delta_n = 0.015$  K; and for  $f_{co} = 1$  Hz, the values are about 0.5 mV and  $\delta_n = 0.005$  K. It is thus sufficient to have  $f_{co} = 5$  Hz to measure temperature differences of 1.5-2 K with an error less than 1%.

This approach provides exact estimates for the dynamic error and enables one to choose lag characteristics optimal from the viewpoint of measurement error, so one can establish the suitability of the components for particular purposes.

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## NONLINEAR OSCILLATIONS IN BULK CRYSTALLIZATION PROCESSES

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Nonlinear oscillations generated by losses of stability of steady-state bulk crystallization are investigated.

Heat- and mass-transfer processes, which are used extensively in chemical technology, metallurgy, and heat engineering and which result in the formation of particles of a new phase, are often accompanied by various kinds of oscillations and fluctuations of the characteristic parameters such as the temperature, concentration of the two-phase system, supersaturation, etc. [1-4]. These unstable regimes are manifested in diverse ways because of the vigorous ongoing development of highly promising technologies that make effective use of major departures of the system from equilibrium phase conditions and operate with metastable states. The investigation of mechanisms for the changeover of heat- and mass-transfer regimes is especially timely in the determination of ways to control transient processes, because the onset of instability can have a significant influence on the nature and the results of the processes involved.

In the present article we investigate the laws governing the loss of stability of steady-state regimes and the inception of highly nonlinear self-excited oscillatory regimes of bulk crystallization from supersaturated solutions or supercooled melts, where the twophase mixture is subjected to intense mixing, and the finished crystals are extracted from the system at a rate that depends on their dimensions.

## DERIVATION OF THE EVOLUTION EQUATION

For definiteness, we shall discuss crystallization from supersaturated solutions.

The physical mechanism of the formation of self-excited bulk crystallization regimes in a metastable medium under steady-state external conditions is described in [1]. The principal cause of instability of steady-state crystallization is a highly nonlinear

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