Identification of phase transition kinetics from dilatometer measurements

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Abstract

The goal of this paper is a mathematical investigation of dilatometer experiments. These are used to detect the kinetics of solid-solid phase transitions in steel upon cooling from the high temperature phase. Usually, the data are only used for measuring the start and end temperature of the phase transition. In the case of several coexisting product phases, expensive microscopic investigations have to be performed to obtain the resulting fractions of the different phases. In contrast, it is shown in this paper that in the case of at most two product phases the complete phase transition kinetics including the final phase fractions are uniquely determined by the dilatometer data. Numerical results confirm the theoretical result.

Keywords: dilatometer, phase transitions, inverse problem

1. Introduction

The dilatometer is an instrument for measuring expansion and contraction of a solid during heating and subsequent cooling. It is often used in the determination of temperature driven phase transitions, occuring, e.g., in the heat-treatment of steel. The specimen is contained in a heating device, usually induction heating.Through a rod on its right-hand side, length changes $\lambda(t)$ due to compression or expansion are measured as a function of time t. In addition the temperature $\tau(t)$ is measured. In Section 2, we describe the governing equation (2.5) - (2.9) and then we have $\lambda(t) = u(1,t)$ and $\tau(t) = \theta(x_0,t)$ where x_0 is an observation point in a domain under consideration.

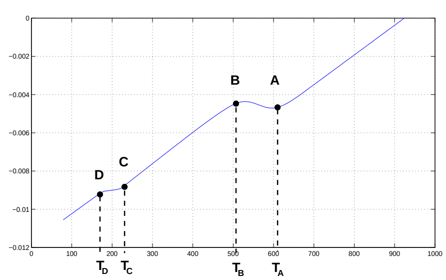

Figure 1: Dilatometer curve for steel C 1080 exhibiting 2 phase transitions.

Usually, the results are documented in a dilatometer curve, where length change is plotted versus temperature, parameterized by the time t. A typical dilatometer curve for the cooling of a specimen made of eutectoid carbon steel is shown in Figure 1. The part of the curve to the right of point A shows the normal contraction of the specimen during slow cooling for a steel in the austenitic phase. At point A a phase transition (from austenite to pearlite) starts and it ends at point B. Then again a period with linear contraction prevails followed by another phase transition (austenite to martensite) between C and D, and finally another linear contraction period. Therefore the main information drawn from such a dilatometer experiment usually are the start (T_A, T_C) and end (T_B, T_D) temperatures of the occurring phase transitions. Moreover, one knows that above T_A the state is purely austenitic. Between T_B and T_C there is a constant mixture of austenite and pearlite and below T_D we have a mixture of the product phases pearlite and martensite. Usually, these data are used to derive so-called Continuous-Cooling-Transformation (CCT) diagrams, which illustrate the beginning and end of a phase transition during continuous cooling.

This approach has two drawbacks. First of all, depending on the curvature of the respective dilatometer curve, it might become rather difficult and erroneous to fix transformation points $A, \ldots, D$. Secondly, in the case of two phase transitions as in Figure 1, the actual phase fractions of the different product phases cannot be drawn directly from the dilatometer curve. Therefore, usually costly polished micrograph sections have to be made and investigated under the microscope. The precision of the predicted phase fractions then highly depends on the experience of the respective experimenter.

From a mathematical point of view deriving just the four critical temperatures is like a waste of information. Indeed it is the goal of this paper to prove that one can uniquely identify the evolution of two product phases $y(t)$ and $z(t)$ from the measurements $\tau(t)$ and $\lambda(t)$. A detailed discussion of the problem can be found in [1].

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2. Mathematical Analysis

The standard shape for dilatometer specimen is a cylinder. Since the diameter is small compared to its length, we will neglect radial variations of the physical quantities and just consider variations along the symmetry axis. For convenience, we define $\Omega = (0, 1)$, and assume small deformations which will allow us to write down the equations in the undeformed domain.

We assume that at most two phase transitions may occur during cooling, with phase fractions $y(t)$ and $z(t)$, respectively, depending only on time t but not on space.The simplest model to describe a thermal expansion as indicated in Figure 1 is assuming a mixture ansatz for the thermal strain

$$\varepsilon^{th} = y\varepsilon_1^{th} + z\varepsilon_2^{th} + (1 - y - z)\varepsilon_0^{th},$$

where the thermal strain in each phase is given by the linear model

$$\varepsilon_i^{th} = \delta_i(\theta - \theta_{ref}^i).$$

Here the constants $\delta_i > 0$ is the thermal expansion coefficient and θ_{ref}^i the reference temperature. For convenience we define

$$\alpha_i = \delta_i - \delta_0, \quad \beta_i = \delta_i\theta_{ref}^i - \delta_0\theta_{ref}^0, \quad i = 1, 2.$$

With $w = (y, z)$ and

$$\delta(w) = \alpha_1 y + \alpha_2 z + \delta_0, \quad \eta(w) = \beta_1 y + \beta_2 z + \delta_0\theta_{ref}^0,$$

we obtain for the overall thermal strain

$$\varepsilon^{th} = \delta(w)\theta - \eta(w).$$

Assuming furthermore an additive partitioning of the overall strain into a thermal and an elastic one, i.e. $\varepsilon = \varepsilon^{el} + \varepsilon^{th}$ we obtain the following quasi- static linearized thermo-elasticity system:

$$\begin{aligned}
\left(u_x - \delta(w)\theta + \eta(w)\right)_x &= 0,\\
\rho c\theta_t - k\theta_{xx} + \Lambda\delta(w)u_{xt} - \rho L_1 y' - \rho L_2 z' &= \gamma(\theta^e - \theta),\\
u(0,t) = 0, \quad u_x(1,t) - \delta(w)\theta(1,t) + \eta(w) &= 0,\\
\theta_x(0,t) = \theta_x(1,t) &= 0,\\
\theta(\cdot, 0) &= \theta_0.
\end{aligned}$$

Here, ρ is the density, c the heat capacity, k is the thermal conductivity, Λ is the bulk modulus, L_1 and L_2 are the latent heats of the phase transitions. Since the cooling happens all around the specimen, we have chosen a distributed Newton type of cooling law, with the heat exchange coefficient γ, and θ^e is the temperature of the coolant. The second boundary condition for u just states that the specimen is stress-free at $x = 1$. All other constants have been normed to one without loss of generality. Inforcing standard assumptions on the data [1] there holds the following

Lemma. The state system admits a unique classical solution (u, θ). Moreover, it satisfies $\theta(x, t) \geq \theta^e$ in $\Omega \times (0, T)$.

Our main result is the following global stability estimate:

Theorem. Let (y_i, x_i), $i = 1, 2$ be two sets of phase fractions and let (u_i, θ_i), $i = 1, 2$, be the corresponding solutions to the state system.

Then there exists a constant $C > 0$ such that

$$\begin{aligned}
&\|y_1 - y_2\|_{C^1[0,T]} + \|z_1 - z_2\|_{C^1[0,T]}\\
&\leq C(\|(u_1 - u_2)(1, \cdot)\|_{C^1[0,T]} + \|(\theta_1 - \theta_2)(x_0, \cdot)\|_{C^1[0,T]}).
\end{aligned}$$

3. Numerical results

In this section we present some results for the numerical identification of phase fractions $y(t), z(t)$ from dilatometer curves, or more precisely, from measurements $\hat{\lambda}$ of the overall displacement $\lambda(t) = u(1, t)$, as well as measurements $\hat{\tau}(t)$ of the temperature in one point, $\tau(t) = \theta(x_0, t)$. To this end, we solve the optimal control problem

$$\min\left\{\omega_1\int_0^T(u(1,t)-\hat{\lambda}(t))^2dt+\omega_2\int_0^T(\theta(x_0,t)-\hat{\tau}(t))^2dt\right\}$$

subject to the state sytem and the control constraint $y, z \in U_{ad}$.

The state system is discretized using finite differences. The phase fraction functions to be determined are represented as cubic splines. Defining the parameter vector $p = (y(t_1), \ldots y(t_n), z(t_1), \ldots y(t_n)$ we solve the resulting nonlinear optimization problem by a Gauss-Newton method.

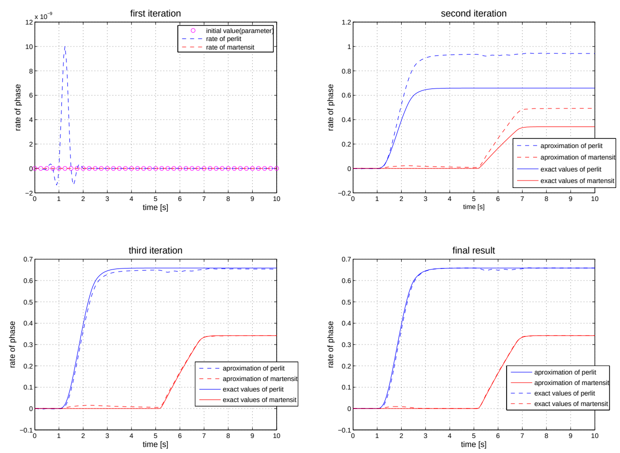

Figure 2: Three iterations and final resulting phase fraction curves in the case of moderate cooling.

Figure 2 shows the result of the optimization process in the case of model data for a plain carbon steel. Starting from initial values $y_0 = z_0 \equiv 0$, already after three iterations the correct final phase fraction has ben reached. Further numerical tests show that the method also works for real measurement data [2].

References

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