Measurement of Paint Layer Thickness with Photothermal Infrared Radiometry

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Abstract

Photothermal infrared radiometry has been used for the measurement of thermo-physical, optical and geometrical properties of multi-layered samples of paint on a metalic substrate. A special data-normalization is applied to reduce the number of sensitive parameters which makes the identification task for the remaining parameters easier. The normalization stabilizes the evaluation of the photothermal signal and makes the infrared radiometry more attractive for applications in the industrial environment. It is shown that modeling and multi-parameter-fitting can be applied successfully to the normalized data for the determination of layer thicknesses. A second approach is presented to verify the adaptability of reconstruction algorithms for thickness measurements. An algorithm which uses the affinity of the thermal waves to the acoustic waves and the inverse scattering problem demonstrates the applicability in general.

1 Introduction

The determination of coating thickness is an important requirement for the industry [1, 2]. As input for remote control systems, thickness information has to be supplied in realtime. Data acquisition has to be perfomed in a non-contact manner. Not only non-destructive and remote control are required, but also methods allowing to determine the composition of a multilayer-coating. Furthermore the calibration should be as easy as possible. In conventional modulated photothermal infrared radiometry, paint-film-thickness is measured using lock-in amplifiers. The phase-difference between modulated laser-excitation and detected infrared signal that is emited by the sample [3, 4] is sensitive to changes of the paint-layer thickness and therefore used as calibration variable. The phase- and amplitude data to a series of excitation frequencies is called a frequency-scan and can be used to identify thermo-physical parameters of the sample [5, 6]. It is represented by a function of complex value with the excitation frequency as argument. If - as in our instrumentation - a mechanical chopper is used to generate a periodical heat source on, respectively within, the sample then we waste a lot of information if only the first harmonic part of the infrared signal is used: Since the chopped laser beam can be approximated by a square wave, we have significant amplitudes at odd multiples of the chopper frequency. The heat equation, which is the mathematical model of the heat diffusion in the sample, is a linear differential equation in the case of temperature independent thermo-physical properties. Furthermore the radiation depends approximately linearly on the temperature in the case of small temperature variations. Therefore we will have significant amplitudes of the periodical radiation-intensity-function of the sample only at odd multiples of the chopper frequency. In this paper we describe a general concept how this information can be extracted from the measured signal and used in infrared radiometry, we call it *simultaneous calibration*. In a first step we derive

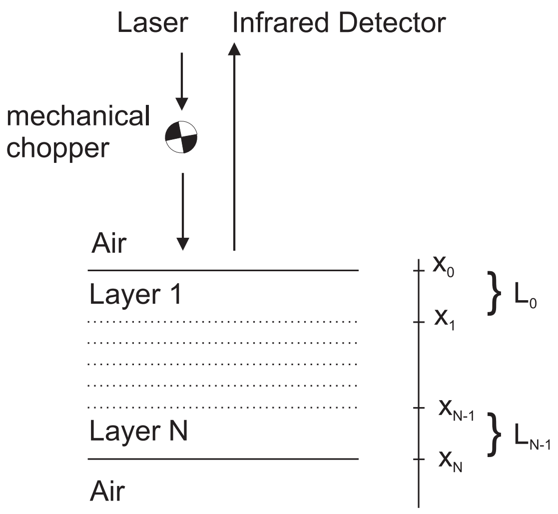

Figure 1: Schematic illustration of the experimental set-up

a mathematical model of the heat flow in N-layer samples. Several approaches to multi-layer-systems can be found in literature [3, 7, 8]. It is assumed that the first layers are light-absorbing and that the last is a metallic substrate, and the layered sample is surrounded by air, which is assumed to be a thermal isolator,

see figure (1).
In the next section we present the general method that we use to extract higher harmonic information from the time signal. This is done theoretically as well as experimentally. The inverse problem of parameter identification is presented with some remarks on the ill-posedness of the problem. Parameter studies performed on measured data sets are used to verify our modeling and normalization procedure.
A second approach makes no assumption on the special geometrical structure of the sample. The diffusivity profile of the sample is assumed to be a function of depth. The actual instrumentation does not allow to make frequency scans in real time. But assuming this would be possible, we were able to show that reconstruction of the diffusivity- respectively effusivity-profiles can be used to determine the paint layer thickness. All experiments were performed by the com-

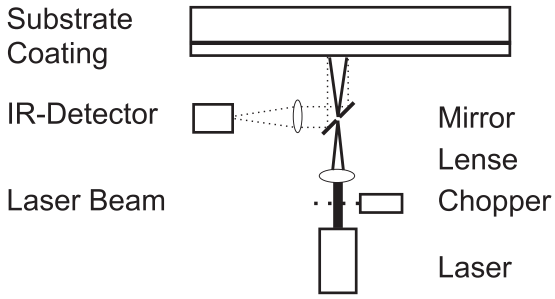

Figure 2: Schematic illustration of the instrumentation

mercial system PS-Online of the Phototherm Dr. Petry GmbH. An illustration of the experimental setup is shown in figure (2).

2 Mathematical Model I

We consider a sample composed of N layers; see figure (1). The laser spot is assumed to be very large compared to the sample thickness and to the detection spot. Since in addition the detection spot is assumed to be large compared to the thermal diffusion length $\mu = \sqrt{2\alpha/\omega}$, our problem can be reduced to a one-dimensional one [3]. α is the thermal diffusivity, ω the angular frequency. The sample will be heated by a square wave heat-source function $q(x,t)$, $0 \leq x \leq x_N, 0 < t$ induced by a time periodic chopper at frequency f_0

$$q(x,t) = q_0 \beta \exp(-\beta x) \mathrm{square}(f_0, t), \tag{1}$$

where β is the infrared absorption coefficient of the first layer and $square(f_0,t)$ the square wave function at frequency f_0. For the sake of simplicity we assume that scattering and reflection of the induced laser beam can be ignored and that all layers have the same absorption coefficient β. Then q is also time periodic with frequency f_0 and the temperature T in the sample is the solution of the heat equation, which can be written in the frequency domain as [9]:

$$\frac{\partial^2}{\partial x^2}T(x,\omega) - \sigma^2(\omega)T(x,\omega) = -q(x,\omega), \tag{2}$$

where $\sigma = (1+i)\sqrt{\omega/2\alpha}$ is the thermal wave number, α the thermal diffusivity and q_0 the maximum induced power. The heat equation is transformed into the Helmholtz equation which shows the similarity between thermal and acoustic waves. Here the change of variables t and ω denotes the transition to frequency domain. Since q is a square wave function we know that $q(x,\omega)$ has amplitudes different from zero only for frequencies which are odd multiples of the chopper frequency $\omega_0 = 2\pi f_0$. From now on we write $q(x,\omega)$ and $T(x,\omega)$ and ω is assumed to be an odd multiple of the chopper frequency ω_0. We assume the sample layers to have constant physical properties . In each layer j the solution of (2) has the form

$$T_j(x) = A_j e^{-\sigma_j(x-x_j)} + B_j e^{\sigma_j(x-x_j)} + C_j e^{-\beta(x-x_j)}, \tag{3}$$

where $x_0 = 0$, $T_j(x), 0 \leq x \leq L_j$ and $q_j := q_0 e^{-\beta x_j}$. We are searching for a smooth solution, where T_j satisfies the following boundary conditions:

$$\begin{aligned} \frac{\partial}{\partial x}T_0(0) &= \frac{\partial}{\partial x}T_{N-1}(x_N) = 0 & (4)\\ T_j(x_{j+1}) &= T_{j+1}(x_{j+1}) & (5)\\ k_j\frac{\partial}{\partial x}T_j(x_{j+1}) &= k_{j+1}\frac{\partial}{\partial x}T_{j+1}(x_{j+1}) & (6) \end{aligned}$$

$j = 0, \ldots, N-2$. This linear system of equations (4-6) for the coefficients A_j and B_j can be solved analytically. The surface temperature is then $T(0) = A_0 + B_0 + C_0$. The value C_0 is determined by equation (2). More differentiated models can be derived in the same way as presented here. In practical application the absorbed power q_0 is difficult to quantify and dependent on environment conditions as well as apparatus components. We remove the influence of q_0 using the fact that all coefficients A_j, B_j contain the factor C_j. So q_0 can be factored out and is then cancelled through normalization. This is the subject of the next section.

3 Simultaneous Calibration

In the case of a one dimensional model the radiometric signal is [10]

$$S(\omega) = K\beta_{IR}\int_0^L \exp(-\beta_{IR}x)T(x,\omega)dx, \tag{7}$$

where K is an instrumental constant depending on geometrical factors, the emissivity averaged over the spectral bandwidth of the detector, the Stefan Boltzmann constant, and the ambient temperature. Additionally we have the influence of electronic devices such as amplifiers in the parameter K so that $K = K(\omega)$ may depend on the frequency and has complex value. We suggest a new way to normalize the measured data which is based on a multi-harmonic thermal excitation (which in turn produces a multi-harmonic infrared signal). This allows to use one harmonic part as reference and one or all the other harmonic parts as information carrier [11]. In the case of a square wave excitation we apply the following transformation to the measured time data $S(t)$:

$$S(t) \to \hat{S} = \left(\frac{S(\omega_0)}{3\sqrt{3}S(3\omega_0)}, \frac{S(\omega_0)}{5\sqrt{5}S(5\omega_0)}, \ldots, \frac{S(\omega_0)}{(2n+1)^{3/2}S((2n+1)\omega_0)}, \ldots \right)^T \tag{8}$$

We call the mapping $S(t) \to \hat{S}$ *simultaneous calibration* [11] because information

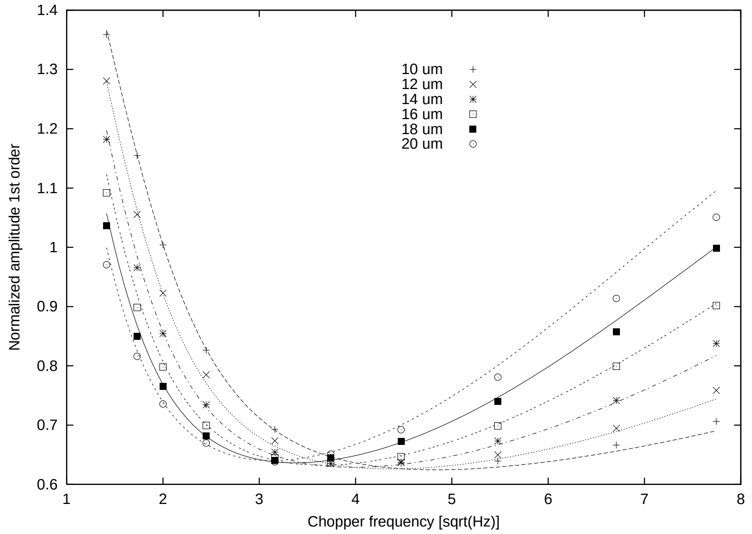

Figure 3: *Multi-Parameter-Fit: Normalized amplitudes* $|\hat{S}_1|$. *Six samples of different and known paint layer thickness were used to fit the thermophysical parameters of the samples.*

retrieval and calibration are carried out in parallel. The factors $(2n+1)^{3/2}$ are chosen so that a semi-infinite sample with surface absorption has a normalized phase of zero and a normalized amplitude of one in all entries of $\hat{S}$. Compared to traditional normalization there are some serious advantages of this method: first the gathered information is not affected by changes of the induced maximum power, which may result from changes of the ambient terms or replacement of apparatus components. Second, we get information about different thermal depth from only one measurement.

4 Multi-Parameter-Fitting

It is well known that the inversion problem of parameter identification for thermal wave data is an ill-posed problem [8]. One consequence of this effect is that the

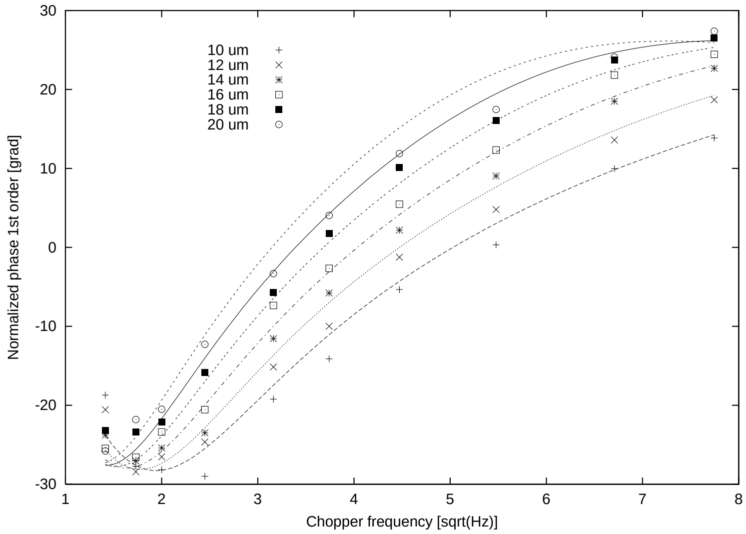

Figure 4: *Multi-Parameter-Fit: Normalized phases* $arg(\hat{S}_1)$.

excitation frequency has to be chosen well so that data is sensitive to changes of sample parameters. Moreover, ill-posedness limits the amount of information that can be retrieved from noisy data. Let us consider the case of a two-layer system paint on metallic substrate. Let $T(x, \beta)$ be the temperature distribution to the absorption coefficient β in the thermally excited sample. Then it can be shown using the mathematical model derived in section (2) that

$$\lim_{\beta \to \infty} \int_0^L e^{-\beta_{IR} x} T(x, \beta) dx = \lim_{\beta_{IR} \to \infty} \int_0^L e^{-\beta_{IR} x} T(x, \beta) dx = T(0, \beta).$$

when we insert the same value for β and β_{IR}. This means that the influence of absorption and emission is interchangeable in the common case of small optical penetration depths. For that reason we omit one of the parameters to stabilize the inversion procedure. Since the remaining parameter β - in our examples we set $\beta_{IR} = \infty$ - compensates the influence of the absorption and emission effect, it cannot be seen as quantitative variable. But since we are interested in layer-thickness this is not a disadvantage. With the introduction of the compensation-parameter we decrease the number of free parameters from three to two per layer.

5 Experimental results: measured data

The experimental set-up is presented in figure (2): we have a CO_2 laser whose output beam is first modulated by a chopper, then focused by a lens of ZnSe, so that the beam can pass a small hole in a mirror. Futhermore this lense controls the diameter of the laser-spot on the sample. The function of the mirror is to deflect the emited radiation in direction of the IR-detector. Another lense, a CAFl lens focus the radiation on the InSb detector which is cooled by a Stirling cooling system. Digitized infrared time data were sampled and analyzed. We present the results for a three layer sample: two different coating layers of paint on a steel substrate. The sum of the coating thicknesses and substrate thickness (430μm) were measured by other methods and therefore approximately known. They were used to verify our mathematical model and normalization approach. Time data acquisition was done on six samples. Paint layer thickness varied between 10 and 20μm, ten chopper frequencies between 2 and 75 Hz were applied. The second layer was a ground coat layer. It was thin compared to the first layer. The substrate layer thickness and the thickness of the ground coat layer were supposed to be the same for all samples. Digitized time series of the pre-amplified detector signal were sampled at a rate of 200 MHz. No Lock-In amplifier was used.
First of all it has to be mentioned that we were not able to differentiate the two paint layers with this data in a stable and reproduceable way. Fitting procedure with the three-layer model converges to a two-layer sample. For that reason we used the two-layer model for all parameter estimations.

We want to verify the approximation quality of our mathematical model. The

calibration with	10 μm	12 μm	14 μm	16 μm	18 μm	20 μm
all samples:	10.0	12.1	14.2	16.1	18.1	19.4
10 μm sample:	10.0	11.9	13.9	15.5	17.3	18.4
12 μm sample:	10	12	14	15.7	17.5	18.7
14 μm sample:	9.7	11.9	14	15.8	17.8	19
16 μm sample:	9.5	11.7	14.0	16	18.0	19.3
18 μm sample:	9.4	11.6	14	16.0	18	19.3
20 μm sample:	8.7	11.2	14	16.3	18.6	20

Table 1: *Computed thicknesses of the first layer for all samples. Thermo-physical properties were computed with the calibration data. All parameters but the first layer thickness were frozen. One parameter fitting for each sample gives the computed quantitative layer thickness. Given thickness was measured with a non-contact-free ultrasound method*

verification method is as follows: Using the knowledge of layer thicknesses we can replace the model parameter *thermal thickness* $L_0/\sqrt{\alpha_0}$ of the first layer with the diffusivity α_0 because L_0 is known as mentioned before. Now we fit the frequency scan data to all samples simultaneously. We use the normalized data $S_1, \ldots, S_4$ (8). In figure (3,4) we see the respective plots for normalized amplitudes and phases. The test presented in table (1) provides information about the stability of our approach as a single-sample-calibration method. One calibration sample per row with known thicknesses was used to determine the thermophysical parameters of the sample. For the other samples solely the thickness L_0 remains as fitting parameter. It can be seen that all samples deliver stable parameters, solely the 20μm sample leads to slightly different results. This is probably caused by less accurate thickness information. But we find our method to be stable, at least in the sense that the computed thicknesses are monotonicly increasing when thickness increases.

6 Mathematical Model II

The first approch uses the layered structure of the samples. The concept presented here does not make such assumptions. We reconstruct a profile of some thermophysical properties of the sample. Thermal diffusivity is very different for metal and for paint, so that our aim will be to find the position of the interface between the layers. Some methods can be found in literature concerning the reconstruction of conductivity or effusivity profiles of hardened steel. We compare one of these methods [12] with a new one which uses the analogy with the acoustic inverse scattering problem. Starting from the Psdeudo Helmholtz equation [13]

$$\left(\frac{d^2}{dx^2} - \sigma_0^2\right) T(x) = F(x)T(x) \tag{9}$$

where

$$F(x) = \begin{cases} \sigma_0^2(n(x)^2 - 1) & : \quad 0 \leq x \\ 0 & : \quad x < 0 \end{cases}$$

and the thermal refraction index $n(x) = \sqrt{\alpha_0/\alpha(x)}$ Then T is a solution of (2) and can be written as $T(x) = T_i(x) + T_s(x)$

$$\begin{aligned} T_s(x) &= -\alpha_0 \int_0^\infty G(x,y)F(y)T(y)dy && (10) \\ T_i(x) &= \frac{I}{\sigma_0 k_0} e^{-\sigma_0 x} && (11) \end{aligned}$$

the sum of incident and scattered thermal wave. G is the Greens function of equation (9)

$$G(x,y) = \frac{1}{2\sigma\alpha}\left(e^{-\sigma|x-y|} + e^{-\sigma|x+y|}\right)$$

We assume the temperature values at the surface were available to different excitation frequencies. With these data we have to reconstruct the profile $F(x)$.

Using the Born-Approximation

$$
\begin{aligned}
T_s(0,\omega) &\approx -\alpha_0 \int_0^\infty \frac{1}{2\alpha_0\sigma_0} 2e^{-\sigma_0 y} T_i(y,\omega) F(y) dy && (12)\\
&= -\frac{\alpha_0}{2k_0} I \int_0^\infty e^{-\sigma_0 y} \underbrace{\left(\frac{1}{\alpha(\frac{y}{2})} - \frac{1}{\alpha_0}\right)}_{:=f(y)} dy, && (13)
\end{aligned}
$$

this problem becomes linear. We see that our data can be approximated by the laplace transform of the object f, which is directly connected to the original object function $F(x,\omega) = i\omega f(x)$. The concept called approximative inverse [14] has been applied for the numerical inversion of the the laplace transform. This problem is extremely ill posed, and the algorithm for the numerical solution very complex, so that we cannot present further details here. One important fact is, that we need a frequency scan, that includes the sensitive frequencies. If not - there will be a lack of information: Since the excitation frequency triggers the penetration depth of the thermal wave, we cannot reconstruct a profile above a certain maximum depth which depends not only on the frequency but also on the diffusivity of the object itself.

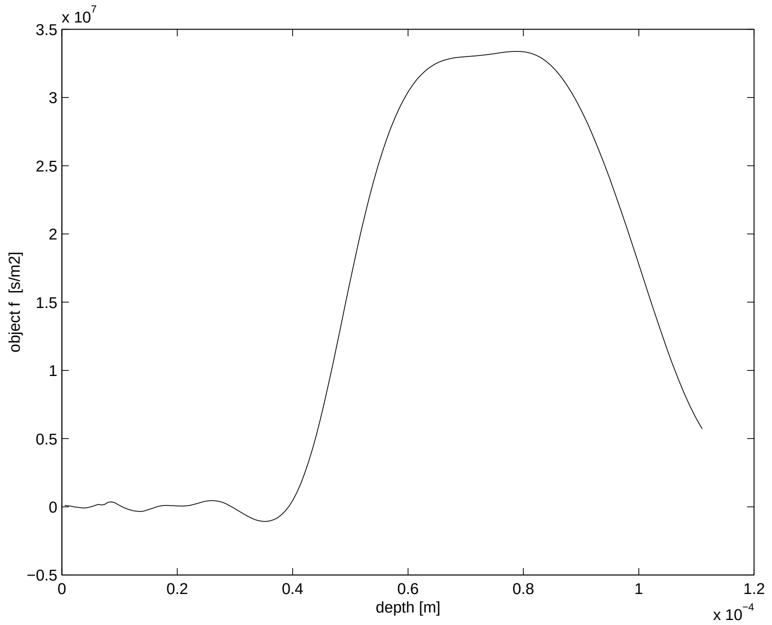

Figure 5: *Diffusivity profile reconstruction using born approximation. 100 Freqeuncies between 0.3 and 400 Hz were used. Regularization with truncated singular value decomposition and smoothing with gauss-kernel.*

7 Experimental results: simulated data

A chopped laser beam which is used for the thermal excitation of the sample does not provide the frequency-scan data with more than 50 excitation frequencies in real time which is necessary for profile reconstruction. Real time measurements of such a frequency-scan cannot be realized with the given apparatus. For that reason we use synthetically data which was generated using the (nonlinear) layered model presented in section (2). Our aim is to show what could be possible, if the measurement technology is able to produce real-time frequency-scans. As a benchmark we examine five samples of paint on a metallic (semi-infinite) substrate. Again we were not able to distinguish mutiple paint layers when the data is disturbed by (artificially introduced) noise. But the location of the interface between paint and metal was successful. A comparison between the born-approximation solution and the results of the state-of-the-art algorithm developed by Bertolotti et al. shows, that both algorithms can be used for thickness computation. We have to mention, that the bertolotti algorithm was originally

exact thickness	computed thickness Bertolotti algorithm	computed thickness Born-approximation
40 μm	38.8	40.1
42 μm	40.1	42.1
44 μm	42.8	44.5
46 μm	44.2	46.3
48 μm	48.4	48.4
50 μm	48.8	50.1

Table 2: *Comparision of reconstruction algorithms. Synthetically generated data for excitation frequencies between 0.3 and 400 Hz, Gaussian noise of 1%. Bertolottis algorithm uses maximum of effusivity contrast $e'(x)/e(x)$ to identify interface location between paint and metal. Born-approximation algorithm uses maximum of differentiated diffusifity profile $\alpha(x)'$.*

designed for the reconstruction of smooth effusivity profiles of hardened steel. We showed, that it can be adopted for the determination of layer thickness. In figures (5,6) we compare the reconstruction quality of the two algorithms. While Bertolottis algorithm (figure 5) is very good in the region of constant effusivity we see better results in the reconstruction of the constant profile of the substrate for the born-approximation algorithm (figure 5). Examples have shown, that the localization of the interface is more stable under the influence of noise for the born-approximation. Both algorithm were able to demonstrate, that linearized models are quite good approximations for the nonlinear problem of profile reconstruction - at least if we are only interested in the location of interfaces within the sample.

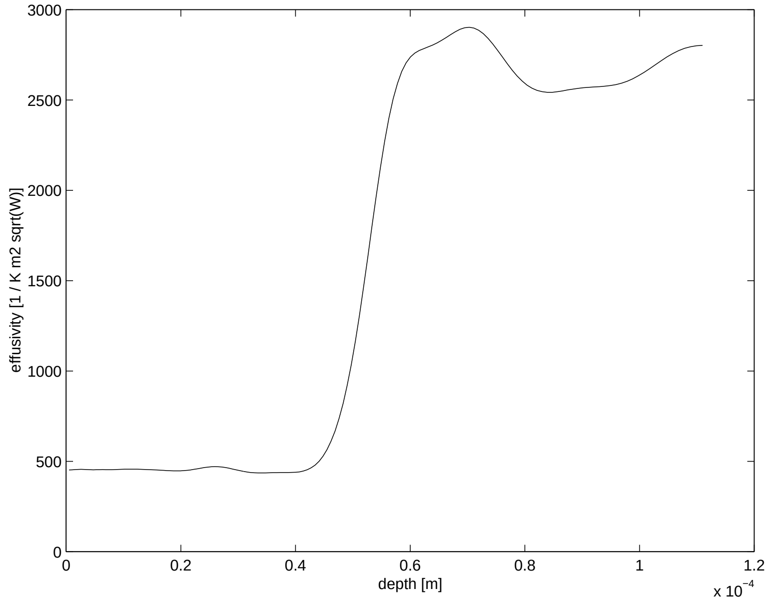

Figure 6: *Effusivity profile reconstruction using bertolottis algorithm. 100 Freqe-uncies between 0.3 and 400 Hz were used. Regularization with truncated singular value decomposition and smoothing with gauss-kernel.*

8 Conclusion

Photothermal radiometry has been used to identify samples of a paint layer on a metallic substrate. A very robust method consisting of a signal analysis - and modeling module has been presented. It makes it possible to identify parameters with very few data, acquired in a rough industrial environment. We were able to show that multi-parameter fitting can be a valuable tool in the measurement technology if we reduce the free parameters to those with the highest sensitivity. The development of suitable data transforms - like simultaneous calibration - in combination with reduced mathematical models leads to measurement methods with some kind of intelligence. Compared to a neural network approach, the modeling-multi-parameter-fitting process has significant advantages: only one frequency scan to only one sample is enough training for our method. After the parameter-identification is performed, the thickness of any other sample - consisting of the same materials - can be identified. We want to emphasize particularly the normalization scheme that we presented. It is the key part of the procedure since it reduces the number of parameters with a strong influence on the (normalized) photothermal signal to four free parameters for a two-layer sample. Moreover the photothermal information - the transformed frequency scan data - is independent of the instrumentation; replacing instrument components does not induce the need of new calibration or multi-parameter-fitting.

Furthermore we were able to show that profile reconstruction as a tool for thickness determination could also be successful, if necessary modifications of the measurement apparatus are technically feasable. But linear models as presented here are not sensitive enough to distinguish different paint layeres, a case which is important in industrial applications. Therefore future work has to deal with nonlinear models, and nonlinear profile reconstruction.

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