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PHOTOTHERMAL RADIOMETRY – PRINCIPLE AND APPLICATIONS

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Abstract: Photothermal radiometry has been proved being a versatile tool for the non-contact and non-destructive material inspection in near-surface zones. Although these near-surface areas represent only a few percent of the workpiece's volume, they influence significantly its quality, functional behaviour, reliability and life time. In particular, they determine decisively the mechanical properties of precisely manufactured and/or surface finished parts. In photothermics, the propagation and penetration depths of optically excited thermal waves, the thermal response at the surfaces on optical excitation of opaque materials give sets of information about the outer and inner structures of the inspected materials. This paper focuses on photothermal radiometry and its potential to assess the thermal material properties and their changes which might be induced by a mechanical impact and/or by a thermal treatment. Special emphasis is given on the industrial applicability of the photothermal radiometric technique. Two problems of industrial relevance are illustrated: the thickness determination of optically opaque layers and the evaluation of hardness depths and profiles in steel specimens.

Keywords: Radiometry, Photothermal techniques, Non-destructive testing

1 INTRODUCTION

In modern manufacturing, on-line sensors are required which are able to detect mechanically or thermally induced material modifications, process influences, flaws and defects inside materials, at surfaces and/or in near-surface layers. Special emphasis ought be focussed on a contactless and non-destructive measuring principle. X-ray, ultrasound propagation and eddy current [1] are well-accepted inspection techniques and they are extensively used for various industrial inspection problems. This contribution takes special emphasis on the photothermal non-destructive technique [2]. Due to their basic principle, photothermal measurement techniques yield information about the material properties in near-surface zones. In subsurface region within the inspected material they cover the micrometer as well as the millimetre range [3,4]. In the last few years, photothermal measurement techniques have found considerable interest [5]; in particular, its potential to assess material properties in near-surface zones and their modifications due to a mechanical impact or a thermal treatment have opened important and very promising applications.

All over, photothermal measurements exploit the time oscillated heat flow induced by a time varying heat source when light energy is applied on a optically absorbing sample. The photothermal effect (see figure 1) is realised by a periodically modulated or by a pulsed excitation. Lasers are well-appropriated, and therefore, favourite energy sources. The time-varied excitation initiate propagating temperature oscillations, so-called thermal waves. Their propagation in the inspected sample and their interactions with surface and subsurface thermal properties (e.g., microstructural changes, thermal inhomogeneities, flaws and defects) determine the temperature rise (thermal response) at the surface of the inspected material. Thus, the temperature measurement, in particular its time structure or its dependency on modulation-frequencies, imply various sets of information about the outer and inner structures.

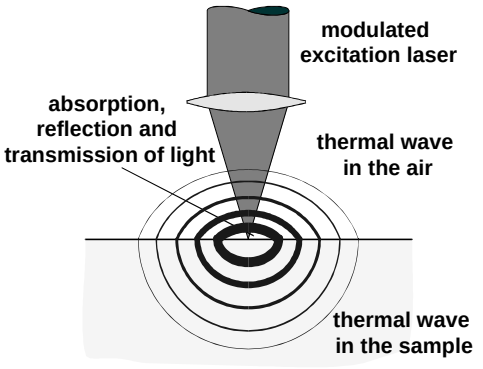

Figure 1. Excitation and propagation of thermal waves

Among the different available options in photoacoustics and photothermics, recently the photothermal radiometry (PTR) [6] has gained substantial progress. Today, the photothermal radiometric technique has been applied to numerous kind of materials and composites. Various material properties concerning coatings, its thickness or

adhesion strengths, hardness depths and hardness profiles, residual stresses, wear and subsurface affected material properties are accessible [2]. Radiometric imaging is included in the fast, reliable and non-contact material inspection. A microscopic resolution in the range of several ten micrometers can be obtained.

The following sections describe the fundamental of photothermal radiometry and an appropriate photothermal setup (section 2). Moreover, two industrial relevant problems will be elaborated: the thickness determination of opaque layers and the evaluation of hardness depths and profiles from photothermal measurements (section 3).

2 FUNDAMENTALS OF PHOTOTHERMAL RADIOMETRY

2.1 Principle and experimental setup

In photothermal radiometry (figure 2 for basic principle) the excitation of thermal waves affects the thermal emission on the heated surface. Following Kirchhoff's and Stefan's law the IR-radiation is increased and its maximum is shifted to higher frequencies. So far, photothermal radiometry links two functions. The first is the optical excitation which produces thermal waves within the sample. The two extremes are sinusoidal modulation or impulse excitation. The second is the radiometric detection of the associated emission of heat radiation. For modulated excitation, one monitors the narrow-band amplitude and phase of the signal at the same frequency as the excitation. The second detection scheme is a wide-band monitoring of the sample's thermal response on the pulsed excitation. Via Fourier transformation, the two detection schemes can be converted into each other.

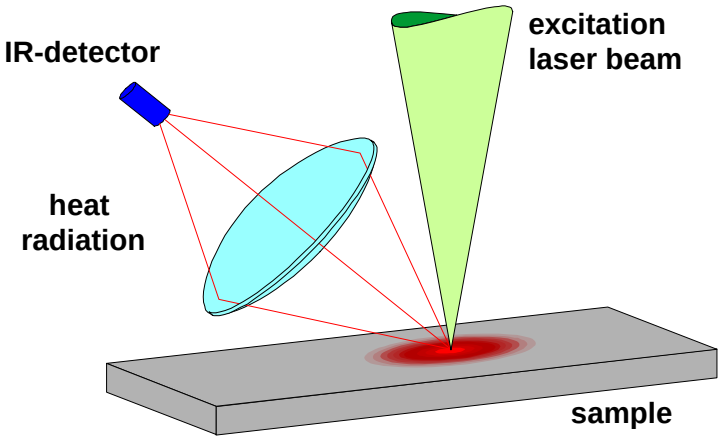

Figure 2. Excitation of thermal waves and their IR-radiometric detection

The temperature measurement is also affected by spatially concentrating the irradiation or localising the detection areas. This paper considers only the case of an one-dimensional heat flow: the excitation area is much wider than both the detection area and the thermal diffusion length (see section 2.2).

One also has to consider that in real samples, various measuring conditions affect the measured IR-flux: optical material properties, absolute temperature of the surrounding, surface roughness, heat flow from the sample's surface to the surrounding gas, etc. Usually, these influences are ignored in non-destructive photothermal applications, since only the signal changes during a lateral scanning across the surface or during a frequency sweep at one surface spot reveal the subsurface structures.

A compact photothermal radiometric setup is shown in figure 3. Herein, heat is generated by the absorption of intensity-modulated light, that is delivered by a laser diode. Generally in photothermal radiometry, a cooled IR-detection device (here a liquid nitrogen cooled MCT = mercury cadmium telluride-detector) monitors the thermal response and the variations of the surface temperature. The emitted IR-radiation is imaged onto the detector element by suitable optics.

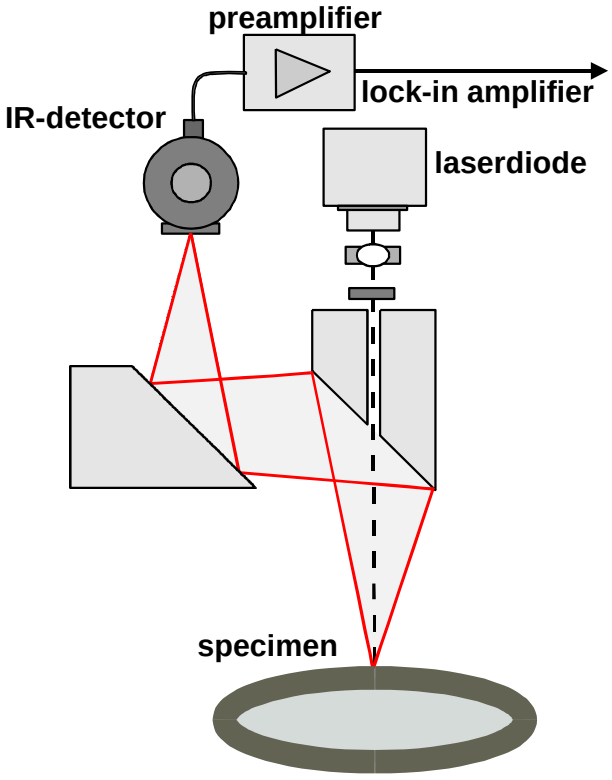

Figure 3. Experimental setup for photothermal radiometry

2.2 Thermal diffusion equation

The thermal diffusion equation (TDE) (eq.1) enables to quantify photothermal signals. Its solution describes the heat diffusion process, i.e. the temperature distribution T(r,t) with its spatial (r) and time (t) dependency after a thermal load from the time-structured exciting laser beam. The temperature distribution within the sample depends on the spatial distribution of the thermal parameters thermal conductivity k, specific heat C and mass density ρ.

$$\frac{d}{d\vec{r}}\left(k(\vec{r})\frac{d}{d\vec{r}}T(\vec{r},t)\right)-\rho(\vec{r})C(\vec{r})\frac{d}{dt}T(\vec{r},t)=-Q(\vec{r},t) \tag{1}$$

In general, an analytical solution for (1) cannot be achieved, only for special cases and regarding some restrictions. Considering only the one-dimensional case for a homogeneous surface absorbing (opaque) sample, the temperature profile within the sample is given by eq. 2.

$$T(z,t)=\frac{I_0}{2e\sqrt{\omega}}\exp(-z/\mu)\cos\left(\omega t-\frac{z}{\mu}-\frac{\pi}{4}\right) \tag{2}$$

The main quantities are the thermal effusivity $e=(k*\rho*C)^{1/2}$ and the thermal diffusivity $\alpha=k/\rho*C$. The last one quantifies the penetration depths of the thermal waves, the thermal diffusion length μ. It depends on the sample's thermal parameters and is inverse proportional to the square root of the exciting frequency f:

$$\mu=\sqrt{\frac{2\alpha}{\omega}} \qquad \text{with } \omega=2\pi f \tag{3}$$

Thus, by varying the modulation frequency, photothermal detectors can 'look inside' a surface zone down to a variable depth.

In thermal equilibrium, the total emitted power, W_{BB}, of a black body is given by eq. 4a. In photothermal radiometry, the measured signal results from transient enhancements of emission, due to absorption and the temperature oscillations at the sample's surface and inside the sample. If the temperature excursions are small compared with the equilibrium temperature, the emitted power-oscillations are given by eq. 4b. Obviously, the emitted IR radiation power depends linearly on the temperature variation. But due to both the detector's sensitivity on wavelength and the electrical response, the measurable signal gain varies and measurements at higher temperature might be preferred.

$$W_{BB}=\sigma T^4 \tag{4a}$$

$$\Delta W(t)=4\sigma T_0^3\Delta T(z=0,t) \tag{4b}$$

σ : Stefan-Boltzmann constant

3 APPLICATIONS OF INDUSTRIAL RELEVANCE

3.1 Thickness and porosity of plasma-sprayed coatings

Plasma sprayed coatings are used in technological applications to protect surfaces and to adapt the product surface to specific requirements. E.g., for medical applications, mainly in orthopaedics, plasma sprayed coatings on artificial hip joints are used to ensure a sufficiently good adhesion and sustainable bone growth into the artificial microstructure. During the plasma coating process the rapid cooling and solidification of the impact-flattened solid droplet cause a lamellar-stratified and highly porous structure. Certainly, the thermal properties of the plasma sprayed coating differ from those of the base material. Due to the damping, and therefore, the limited penetration depths of the optically excited thermal waves, the desired information about layer thickness and porosity can be achieved at different modulation frequencies. To evaluate the porosity (area/volume percentage of pores referred to the entire area/volume) of the layer, the selected modulation frequencies must be in the range between 75 Hz up to 90 Hz. where the thermal waves are damped inside the porous layers and do not reach the interface to the base material. In order to determine the layer thickness of porous plasma sprayed coatings, several frequencies from 5 Hz to 10 Hz are feasible. Of course, since the layers which are very close to the surface contribute always more to the overall signal than deeper subsurface zones, the porosity must be determined at first. Together with this information, the thickness of the porous layer can be determined fairly accurately.

Because the thermal parameters of porous layers are not known, the measuring system must be calibrated on the different base materials (e.g., titanium, $TiAl_6V_4$ or cobalt chrome steel alloy), whereat the layer thickness and porosity have to be determined destructively for different, especially designed samples. Figure 4 shows two calibration curves for the determination of porosity and thickness of

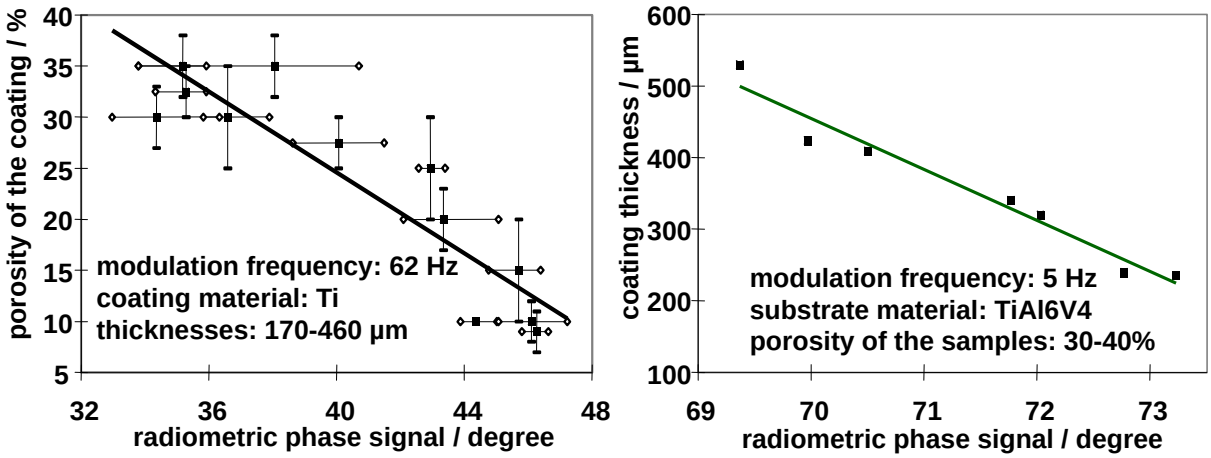

Figure 4. Calibration curves for the photothermal radiometric determination of porosity (left) and thickness (right) of Ti-plasma sprayed coating on Ti, or resp. $TiAl_6V_4$-substrate material

plasma sprayed titanium coating on Ti, or resp. $TiAl_6V_4$-base material. Both the porosity and the phase variations on different locations on the sample's surface mirror the manufacturing bandwidth.

Finally, the photothermal instrument allows to measure automatically the coating thickness and porosity in a fast non-destructive way and without any sample preparation. It can be used in an industrial environment, and so far, the implants can be inspected very close to the coating process. No special knowledge for the handling of the system is needed. A measuring instrument has been developed for the company AESCULAP AG, Tuttlingen, where it works successfully close to a production line.

3.2 Approach for the determination of hardness depths and profiles

The performance of surface hardened parts is a major issue in automotive and aerospace industries. There is, therefore, a strong need of hardening companies to improve the quality control of their products by introducing new measurement systems which allow for non-destructive, non-contacting hardness profile measurements as an alternative to the presently used destructive inspection methods. It is the aim of this section to describe very briefly the state of the art concerning the determination of hardness depths and profiles by means of the photothermal methodology.

The approach for photothermal hardness inspection is based on microstructural changes, as they appear in the steel hardening process (e.g. in the austenite/martensitic phase conversion at case hardening) together with modifications of the thermal properties. I.e., the increasing hardness in a near edge layer causes a decreasing thermal conductivity in this zone. Since hardening belongs to the diffusion processes, a smooth profile for the thermal conductivity within the hardened specimen can be assumed [7-9].

For simplification, one assumes also a surface absorbing sample, whereat the optical absorption length is much smaller than the thermal diffusion length.

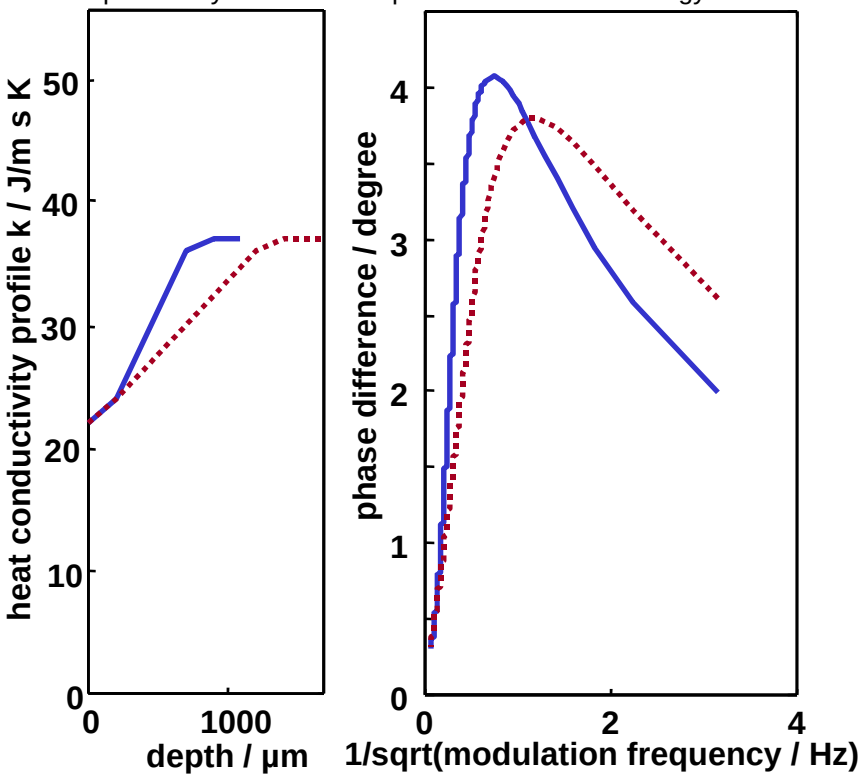

Figure 5. Profiles of thermal conductivity (left) and expected phase difference curves (right) in photothermal hardness inspection

This assumption is reasonable for all metal specimens. Furthermore, one can suppose that hardened specimens show a polygonal depth profile of thermal conductivity k, whereas the thermal density ρ*C remains constantly unaffected by the microstructural modifications. The region from the sample's surface to the unaffected base-material can be subdivided into virtual sublayers. In each layer, the thermal conductivity varies linearly (see figure 5 left), and the temperature field for each zone can be composed of various propagating thermal waves. Finally, a superposition of all thermal waves yields the surface temperature (amplitude) and time-delay between excitation and thermal response (phase) [10]. Figure 5 on the right demonstrates the calculated phase slope for two different hardness depths, that derives from the correlation between hardness and thermal conductivity [11]. Obviously, increasing hardness depths, or vice versa a slower rise in the thermal conductivity profile, result in a phase shift to lower modulation frequency and in a decreased phase maximum.

As an experimental verification, a photothermal hardness inspection has been carried out on various case hardened samples of 16 MnCr 5. These specimens were inspected in both ways, conventionally by Vickers indentations along the transverse cut and non-destructively by photothermal investigations at the surface. The phase curves (phase differences between hardened and soft, but heat treated specimens) are illustrated in figure 6. These results confirm the theoretical predictions that increasing hardness depths lead to a phase maximum shift and to lower phase values.

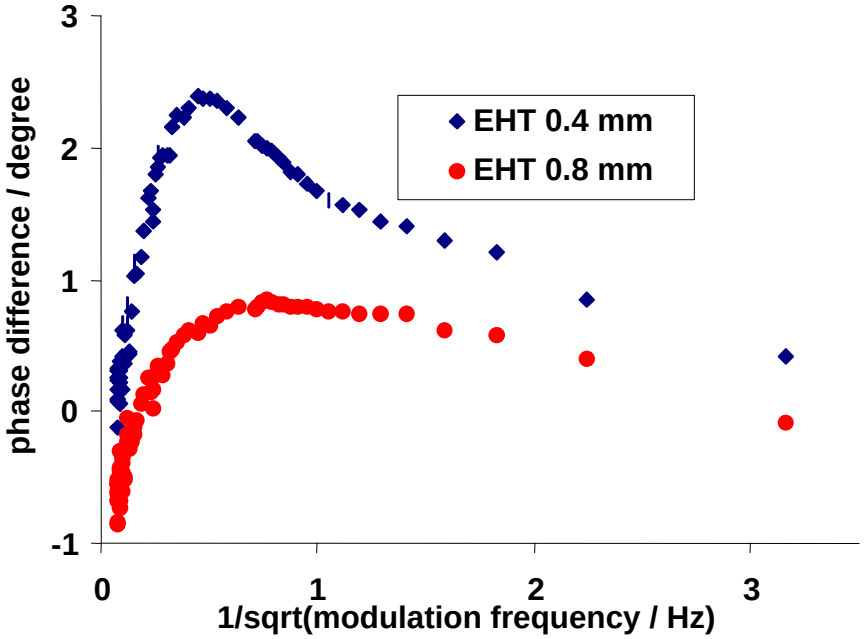

Figure 6. Phase difference curves for two case hardened 16MnCr5 specimens

In summary, photothermal radiometry allows for the determination of the hardness depths via calibration curves, e.g. conventionally destructively measured hardness profiles on cut cross sections via Vickers indentations. Recently, the mathematics to reconstruct the hardness profiles from photothermally measured phase curves has been developed [10, 11]. Still, this procedure must be verified on the shown samples.

Moreover, numerous photothermal experiments have been performed in various laboratories [11]. The future research efforts in photothermal hardness inspection have to be concentrated on the separation of disturbing influences at the photothermal hardness profile evaluation, such as grinding belts, scaled layers, surface carbon austenite content, weak spots, pitting and/or residual stress. With the same time, photothermal methodology for hardness inspection must be transferred to industrial use.

4 CONCLUSIONS

This paper intends to give a compressed survey about the state-of-the-art of the photothermal radiometric techniques meeting industrial purposes. It is a non-destructive and non-contact method correlating the material and structural properties of the near-edge zone with thermal and/or mechanical parameters and their spatial distribution. Today, photothermal inspection covers a wide spectrum of materials coating features (thickness, adhesion strength, local disturbances), hardness depths and profiles, residual stresses and wear. Using the described photothermal measuring device, one is able to determine the thickness of opaque layers, the thickness and porosity of plasma sprayed coatings (PLASMAPORE) on various base materials in a non-contact manner and independently of the sample's geometry. Several investigations performed on almost all known hardening processes like case hardening, nitriding, carbo-nitriding lead to promising results concerning measuring times, accuracy, robustness and costs. The use of photothermal calibration curves (e.g., destructively obtained Vickers data versus photothermal data) offers a simple and practical approach for the

hardness depth or compound layer thickness determination. So far, the photothermal measurement technique allows for a fast and accurate quality control. In addition, the findings can be transferred to other similar measuring problems in industry, whereat the system must be adapted to the individual measuring problem. Furthermore, the theoretical verification of the results must be the topic for further investigations, whereat various thermal profiles and their correlation with different mechanical properties should be considered.

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