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Development of Laser Lock-In Thermography for Plasma Facing Component Surface Characterisation

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ABSTRACT.

InfraRed (IR) photothermal techniques are candidates for in-situ characterisation of tokamak Plasma Facing Components (PFC) surfaces, by means of an external thermal excitation coupled with an IR temperature measurement. Among these techniques, the Laser Lock-In Thermography (LLIT) uses a modulated laser excitation which gives 2 major advantages: enhancement of signal to noise ratio and emissivity independence, which is a plus when the components have various and unpredictable surface quality. With this method, it is possible to develop a process which could be used remotely, either mounted onto an in-situ inspection device (articulated arm) or in a PFC test bed. This paper presents the results obtained with a continuous modulated laser heat source on particular samples (W coating on CFC substrate, C layer on graphite substrate). The identification of the experimental data with a theoretical model allows a quantitative characterisation of the layers.

1. INTRODUCTION

The LLIT is proposed to analyse either co-deposited layers or coatings on PFC. Co-deposited layers are frequently observed on PFC surfaces resulting from the erosion/re-deposition of materials caused by the harsh plasma radiation and particle outflow. These undesirable layers which can trap tritium and/or affect plasma operation can be removed by an ablation system [1]. A preliminary layer characterisation by means of LLIT helps to adjust the ablation parameters to make it more efficient. Coatings are used to enhance PFC surface features (e.g. W coating for JET ITER-like wall). The LLIT technique could be used to characterise coating adhesion and their evolution (ageing) during wall lifetime.

The modulated laser source provides a high excitation frequency range [1Hz–10kHz]. This capability allows determining thermal properties of thin layers of micrometres to millimetres thickness. LLIT gives access to several layer thermal parameters, and mainly to the surface thermal resistance, which is linked to the layer thickness in the case of porous and thermally resistive co-deposited layers, and to the contact thermal resistance between coating and substrate related to the coating adhesion quality.

First tests have been performed using halogen lamps as heating source, and an IR camera to measure the surface thermal response [2]. Due to the low frequency response of the lamps (<2Hz), this device was restricted to non destructive examination of PFC. To extend the test to the characterisation of thin layers, a modulated continuous-wave laser and an IR detector sensitive to low temperature variations near room temperature are used.

2 DESCRIPTION OF THE EXPERIMENT

2.1 TEST BED SETUP

The test bed setup shown in Fig.1 consists of two main parts: the thermal excitation and the temperature measurement. The thermal excitation is generated by a CW Ytterbium fibre laser (50W CW, 1.1mm wavelength), which can be driven by a signal generator to provide a periodical heat flux. The driven signal is a sinusoid of frequency (f) in the range [1 Hz – 10 kHz]. A fast photodiode

measures the laser beam reflected on the sample surface, and gives the phase lag of the laser, which depends on the power and the frequency.

The thermal response of the heated sample is measured at its surface with a fast IR photovoltaic detector, (HgCdZn, thermo-electrically cooled, 3-11µm wavelength, 5µs time constant). A biconvex lens (50mm focal length) focuses the sample radiation on the detector window. The detector is supposed to be insensitive at the laser beam wavelength. However, diffuse laser reflection is very high compared to IR radiation, particularly on reflective surfaces. Thus, a Ge filter is placed in the optical path to cut off laser reflection. The laser output collimator and the IR detector are carefully positioned, so that the image of the detector ($\Phi_m = 1mm$) focused by the lens is centred into the laser heating spot ($\Phi_0 = 5mm$ @ $1/e^2$ intensity).

The lock-in amplifier measures the photodiode and the IR detector signal phases with respect to the reference signal. The IR detector phase minus the photodiode phase gives the phase-shift between the sample surface IR radiation and the thermal excitation.

Thanks to the high sensitivity of the IR detector and of the lock-in amplifier, a low laser power is sufficient to perform a test without any material alteration. 5W is typically used for common surfaces, which gives a mean an incident flux of 250kW/m^2 on the laser spot. The steady state mean temperature reached by the sample is only a few degrees above room temperature for a large sample with a high diffusivity, and remains lower than $\approx 80^{\circ}$ C in the contrary case. The steady state regime is reached in few seconds maximum. The temperature magnitude at the excitation frequency strongly varies with the sample thermal characteristics, and decreases like $1/\sqrt{f}$. For a 5W laser power, it can roughly vary from 0.01° C at 10kHz for a high diffusivity material to 10° C at 1Hz for a low diffusivity material. Such low temperature variations ensure a non destructive measurement.

For reflective materials, the laser power must be increased, because only a small fraction of the energy is absorbed, and the IR radiation emitted by the sample is lowered again by the low emissivity.

With this test bed, we observe a very good reproducibility between successive measured data $(<2^\circ)$ except for the higher frequencies (\geq 5kHz) due to a low signal to noise ratio. It has been checked that the measured data do not depend on the laser power.

2.2 TESTED SAMPLES

The aim is to determine layer properties of various types.

First a TEXTOR tokamak tile from ALT-II (Advanced Limiter Test II), made of isotropic fin grain graphite (Toyo Tanso, IG-430U) with various deposited carbon layers of different thickness is studied.

Then, a CFC sample with W-layer deposited onto the substrate by plasma spray has been studied. The W layer has been characterised beforehand with a thickness in the range [90 - 190] mm, and a density of 15000kg/m³, i.e. 78% to that of bulk W.

3 SIMULATION

3.1 HEAT TRANSFER MODELLING

The heat transfer of a laser beam heating a sample as shown in Fig.2 is a 3D axisymmetrical problem. The heat conduction equations of this problem can be written in cylindrical coordinates. Using the Hankel and Laplace transforms enables to solve the equations, and gives the transfer function Z of the surface temperature q_0 in the Fm diameter, over the absorbed heat flux j_0 , expressed in the Laplace domain, as described in [3]:

$$\theta_0(\mathbf{p})/\phi_0(\mathbf{p}) = \mathbf{Z}(\mathbf{p}, xyz) \tag{1}$$

xyz being a set of thermal and geometrical parameters describing the sample (thermal conductivity λ , volumetric heat capacitance ρ Cp, contact thermal resistance at the interface Rc inverse of heat transfer coefficient which characterizes the layer adherence, thickness e), and the test bed (ϕ_0 , Φ_m , Φ_0).

 ϕ_0 being a sinusoidal heat flux, one can replace the Laplace variable p by $2\pi fi$, which gives the transfer function Z versus the frequency. The argument of Z gives the phase-shift between q_0 and ϕ_0 .

The following assumptions are posed:

- Substrate and layer materials are homogeneous and isotropic,
- Linear behaviour, justified by the low temperature magnitude at excitation frequency,
- Optical absorption and emission are taken into account, although they are negligible. For instance, the absorption depth at 1 micron wavelength is about 30nm [4] for W,
- Convective heat exchange $h = 10 \text{ Wm}^{-2}\text{K}^{-1}$ is applied at front and rear surfaces (not at the sides), but is negligible for the phase-shift, except for very low frequencies not studied here (<<1Hz).
- IR radiation cooling is negligible, (equivalent h < 5).

With this model, it is possible to simulate an unlimited number of distinct layers piled up on the substrate, each of them being linked to the preceding one with a different Rc.

The theoretical model gives the temperature phaseshift, whereas the measured data by the IR detector is proportional to the IR radiation luminance. However their phase-shifts are rigorously the same.

With this method, there is no need to calculate the magnitude of Z, because the surface temperature is sensitive to the surface emissivity ε and the heat flux magnitude. e can vary strongly with the material and the surface roughness, and is hardly known [5]. The heat flux φ_0 is the absorbed part of the incident flux from the laser excitation, and is roughly proportional to $(1 - \varepsilon)$. φ_0 can not be known with accuracy, as well as the IR radiation which is proportional to ε . To the contrary, the phase-shift is not sensitive to φ_0 and ε , which is the major advantage of LLIT.

3.2 IMPLEMENTATION

A MATLAB® routine has been developed to calculate Z. The argument of Z versus f is a curve which depends on the parameters described on Fig.2. If the substrate is known, it is possible to determine some of the layer parameters. The solution is not unique and involves some a priori on the range of

the parameters to be identified. The retained solution is the most plausible one. The identification of the layer unknown parameters is solved by fitting, in the least squares sense, the non linear function of the temperature phase-shit, using a MATLAB® algorithm (lsqcurvefit) included in the optimisation toolbox.

4. RESULTS AND DISCUSSIONS

4.1 VALIDATION ON A STAINLESS STEEL SLAB

In order to check the consistency of the simulation, measurements are performed first on a thin Stainless Steel (SS) foil of 250 μ m thickness, split into 2 equal layers linked with a perfect contact thermal resistance (Rc=0).

Due to the shiny surface of SS, a 30W laser power is used to obtain a convenient IR signal. In such conditions, the sample reaches a steady temperature of $\approx 300^{\circ}$ C.

The experimental data Fig. 3 are consistent with the calculated curve from the 3D model, using thermophysical parameter values of stainless steel (Table 1).

The 3D thermal behaviour is usually perceptible only for low frequencies, when the thermal wavelength $\Lambda = 4\pi\lambda (\sqrt{f\rho Cp})$ [6] is much lower than the laser diameter Φ_0 . For the SS foil, the 3D behaviour is observed for f < 20Hz, instead of 3.3Hz due to the thinness of the sample. The advantage of being in the 1D behaviour range is that the material orthotropy does not affect the thermal response. The semi-infinite wall behaviour, depicted by a -45° phase-shift, is observed when $\Lambda/2$ is lower than the layer thickness (f ≥ 320Hz for the SS foil).

Discrepancies for higher frequencies (\geq 5kHz) could be explained by the volumetric absorption and emission caused by surface roughness, when the thermal penetration depth m = Λ /2p, is in the order of magnitude of the roughness, e.g. 14mm at 10kHz for the SS. An additional thin surface layer (table 1) added in the model to improve the fit can also explain the high frequency behaviour.

4.2 GRAPHITE TILE

The graphite tile is tested with a 5W laser power, on 3 different zones on which various C deposits are observed:

- Zone 1: clean zone, plasma erosion
- Zone 2: adherent deposit
- Zone 3: fragile deposit, near flaking

The erosion zone is first fitted with parameters closed to the original graphite, with reduced conductivity, due to the ageing of graphite in the tokamak. However, for high frequencies, the fit does not match well with the semi-infinite wall behaviour (-45°) , and a thin layer added on the surface is necessary for a convenient fit. No thermal resistance (Rc) is needed, so this layer could be viewed as a surface state modification due to plasma erosion.

The zone 2 is fitted with a thin layer with low conductivity, a reduced heat capacitance due to porosity, and poor Rc.

For the zone 3, any simple surface layer is able to fit the experimental data above 1kHz, and it

is necessary to split the layer to achieve a convenient fit. Four identical layers linked with the same Rc have been simulated. Table 2 indicates the total Rc and the total thickness of the 4 layers. This simple simulation could not reproduce the complexity of the 3 dimensional deposit structure, but it can be viewed as a representation of such porous deposits with a kind of lamellar form [7], which makes a thermal resistance between the lamellas distributed through the layer.

This simulation reveals that the C layer couldn't be considered as a simple thermal resistance added on the surface: the role of the heat capacitance is more and more important when the laser frequency increases.

4.3 CFC TILE WITH W COATING

Three distinct zones have been tested:

- Zone 1: bulk CFC, W coating have been pulled up.
- Zones 2 and 3: distinct areas with W coating as described in §2.2.

The W volumetric heat capacity is assumed to have the same ratio compared to the bulk W as for density, i.e. 2.1 J/K/m3. A 5 W laser power is used.

Once again, a simple 2-layer model is not able to fit the experimental data of zones 2 and 3 at higher frequencies. 5 layers have been arbitrary used for the simulation Fig. 5, with the same thicknesses and contact thermal resistance at the interfaces. W porosity observed on micrography [8] probably makes thermal resistances distributed along the layer thickness.

CONCLUSION

The LLIT is a convenient technique for contactless non destructive characterization of various layers covering PFCs. The independence of the phase-shift from surface emissivity and heat flux magnitude is its major advantage.

The theoretical model of phase-shift calculation allows determining thermo-physical properties of layers provided the substrate is known, and the materials are rather homogeneous and isotropic. The multilayer model is indispensable to find a convenient simulation fitting the experimental data, when the layers are quite porous or in lamellar form.

Further development of the theoretical model should include the layer surface roughness, which could explain discrepancies observed at high frequencies, and then extend the identification to thinner layers.

The use of a larger heat flux zone, with a defocused laser or other excitation would increase the 1D behaviour frequency span. The advantages are to make the model and the experiment independent of laser and detector diameters and to reduce drastically the calculation time.

The evolution of the test bed for longer distance measurements is the next step which will enable its possible operation in tokamaks.

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REFERENCES

- [1]. A. Semerok et al., Journal Applied Physics **101**, 084916 (2007)
- [2]. X. Courtois et al., Physica Scripta T128, 189-194 (2007)
- [3]. J.L. Battaglia et al., Int. J. Thermal Sci. 45, 1035-1044 (2006)
- [4]. Handbook of Optics, ISBN 0-07-147740-X II.35.12-27
- [5]. M. Richou et al., submitted Journal Nuclear Materials (2009)
- [6]. W.J. Parker et al., Journal Applied Physics Vol. **32** n°9, 1679-1684 (1961)
- [7]. P. Gasior et al., Physica Scripta T123, 99–103 (2006)
- [8]. A. Semerok et al., JET Technology Work-programme, Task JW8-FT3.45, 2009

parameter	Standard values from literature	Optimized values	Additional layer
λ W/m/K	15 - 30	23	36
ρCp MJ/m³/K	3.5	3.5	3.5
e μm		232	9
Rc m ² .K/W			0.3 x 10⁻ ⁶

	Bulk	Additional	C layer	C layer
	graphite	layer	zone 2	zone 3
parameters		zone 1		4 layers
λ W/m/K	96	50	7	36
ρCp MJ/m³/K	1.5	1.5	1.3	1.3
e μm	10 x 10 ³	10	10	25
Rc m ² .K/W	-	10 ⁻¹⁰	6 x 10⁻ ⁶	18 x 10⁻ ⁶

Table 1: Thermo-physical	properties of	f stainless stee	l foil.
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narameters	Bulk graphite	Additional layer	C layer zone 2	C layer zone 3
γarameters	96	50	7	36
oCp MJ/m ³ /K	1.5	1.5	, 1.3	1.3
e μm	10 x 10 ³	10	10	25
Rc m ² .K/W	-	10 ⁻¹⁰	6 x 10⁻ ⁶	18 x 10 ⁻⁶

Table 2: Thermo-physical properties of graphite tile + C deposit.

		Additional	W	W
	Bulk	layer	coating	coating
parameters	CFC	zone 1	zone 2	zone 3
λ W/m/K	220	196	42	39
ρCp MJ/m³/K	1.5	1.5	2.1	2.1
e μm	8 x 10 ³	15	100	110
Rc m ² .K/W	-	0.046 x 10 ⁻⁶	13 x 10⁻ ⁶	21 x 10⁻ ⁶

Table 3: Thermo-physical properties of CFC tile + W coating.



Figure 1: Schematic description of the experimental setup.



Figure 2: 3D axisymmetrical problem for LLIT simulation.



Figure 4: Experimental data (marks) and best fitted curve with 3D model (lines) for following surfaces: (1) bulk graphite (dashed line) and bulk graphite + additional layer (solid line) (2) graphite + C deposit zone 2 (3) graphite + C deposit zone 3, 1 layer (dashed line), 4 identical layers (solid line).



Figure 3: LLIT test on SS foil compared to 1D and 3D simulation. 1D simulation is described in [2].



Figure 5: Experimental data (marks) and fitted curve with 3D model (lines) for following surfaces: bulk CFC + additional layer and plasma spray W coating on CFC.