Groupe de Recherche en Sciences pour l’Ingénieur

GRESPI-ECATHERM

Caractérisation Thermophysique Multiéchelles

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Thermophysical characterisation by modulated photothermal methods: size effects in micro-structured materials

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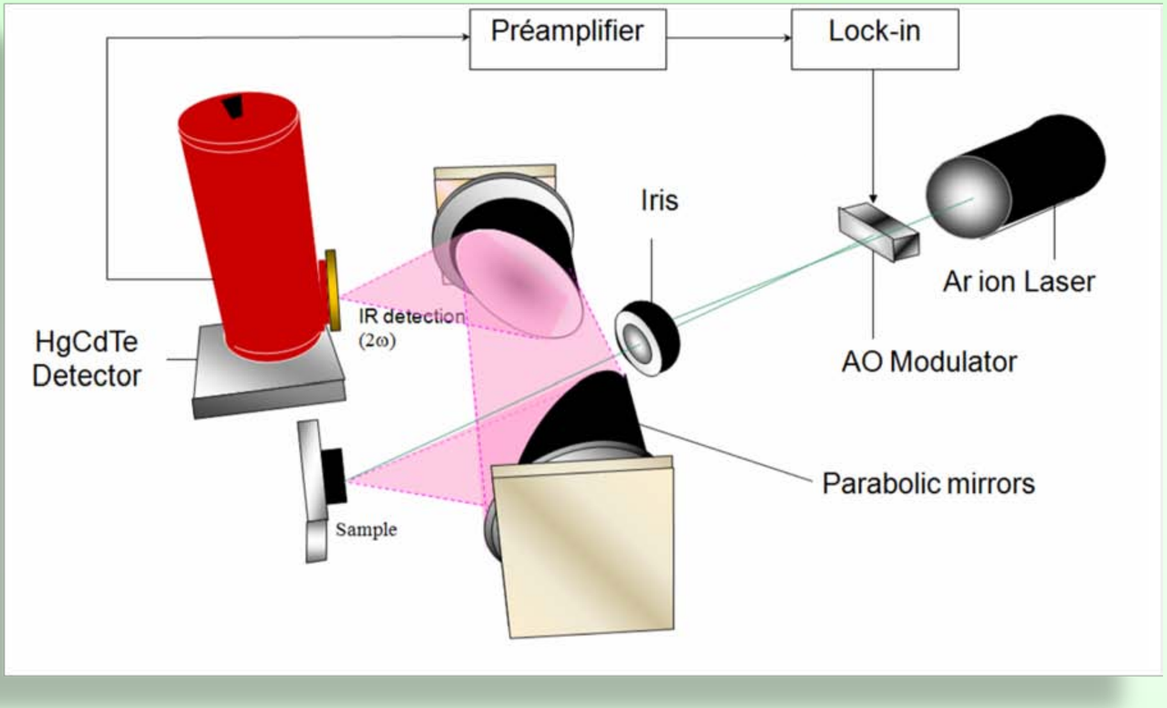
Photothermal radiometry (PTR)



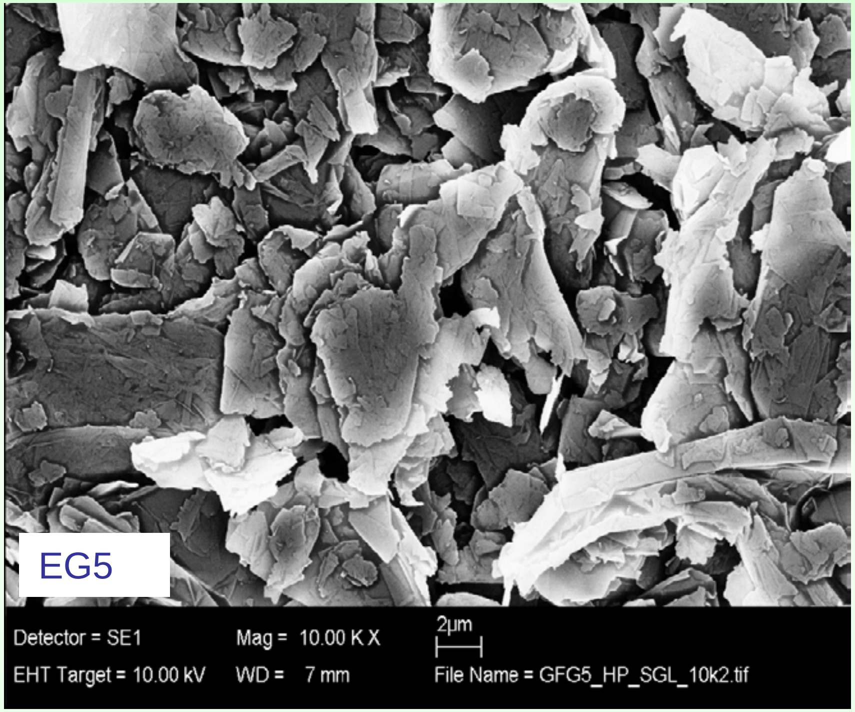
Use of microlens beam-shaper for uniform sample irradiation

$\Rightarrow$ 1-D model down to 0.1 Hz

$\Rightarrow$ homogeneous phase up to 1 MHz

Observation scale $\propto \mu$, thermal diffusion length: mm ... <μm

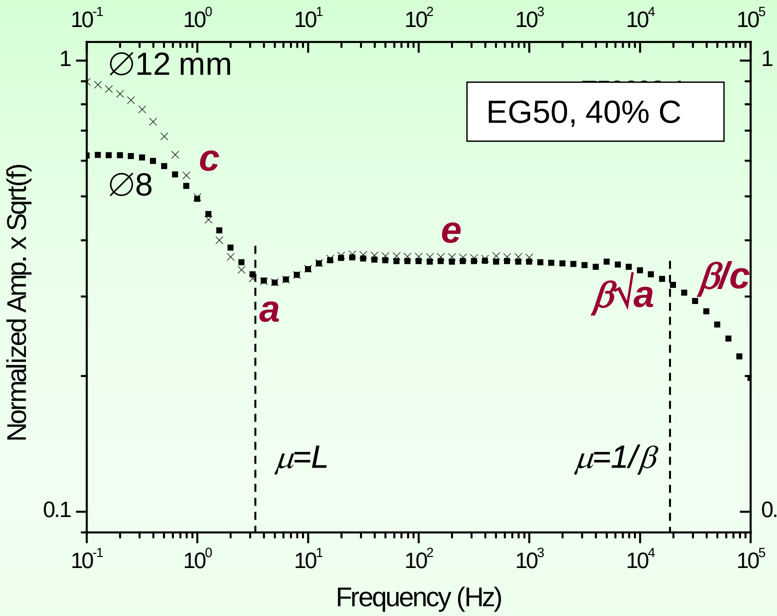
1. Thermal percolation and size-effect in HDPE-EG

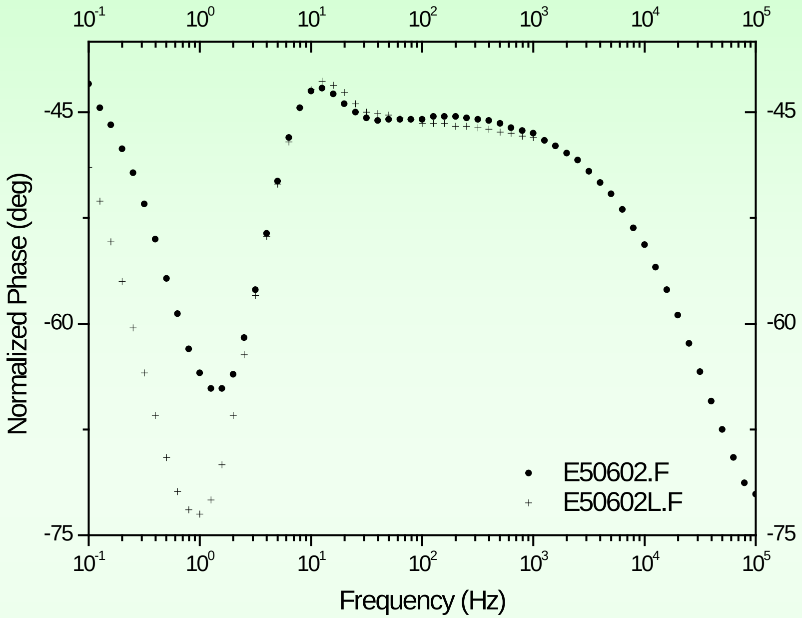


Materials:

- High density polyethylene HDPE
- Expanded graphite (EG) with particle sizes about 5 and 50 µm
- Samples: sheets of few cm^2, L = 0.25 ... 0.45 mm
- Produced by compression molding at 120°C, 40 kPa pressure, 1 min.

Front detection FD-PTR





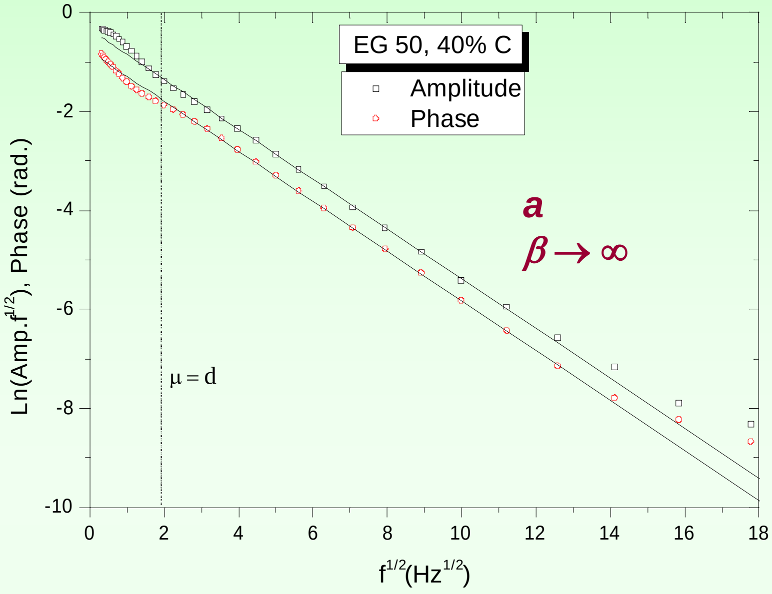
1-D model: semi-transparent layer (m) on thick substrate (b)

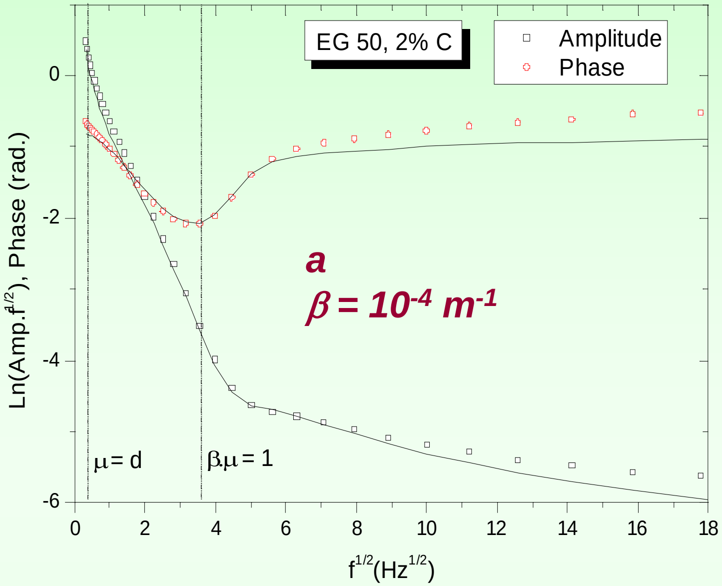
z_{gm} [m^2KW^{-1}], specific thermal impedance:

$$\frac{z_{gm}(FD)}{z_0} = t\,\frac{-e^{-\beta L}[(t-1)+(t+1)R_{mb}]M^{-1}+(t-1)+(t+1)R_{mb}M^{-2}}{(t^2-1)(1-R_{mb}M^{-2})}$$

$t = (1/2)(1-i)\beta\mu$, $M = \exp[(1+i)L/\mu]$, R_{mb} = thermal reflection coefficient

Back detection BD-PTR





1-D model: semi-transparent layer (m) on thick substrate (b)

z_{gm} [m^{2}KW^{-1}], specific thermal impedance:

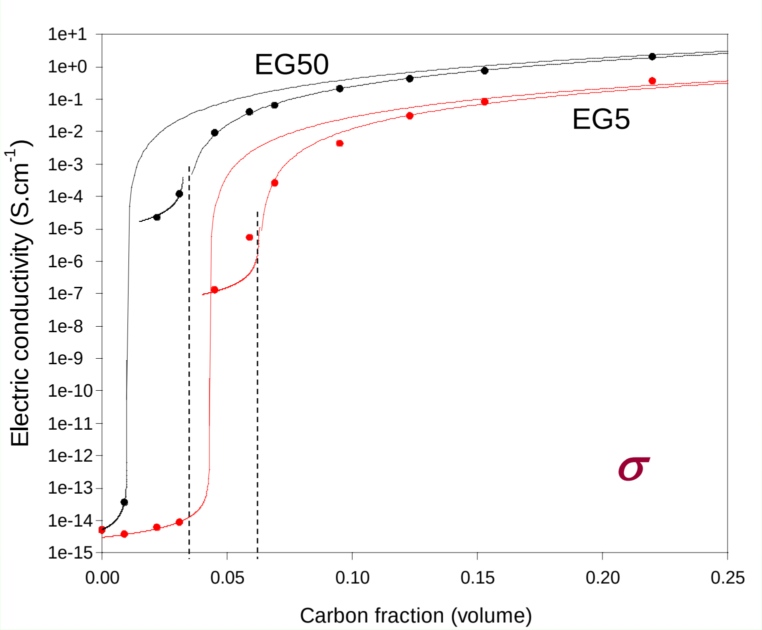
$$\frac{z_{gm}(BD)}{z_0} = t\,\frac{[(t+1)+(t-1)R_{mb}]M^{-1} - e^{-\beta L}[(t+1)+(t-1)R_{mb}M^{-2}]}{(t^2-1)(1-R_{mb}M^{-2})}$$

$t = (1/2)(1-i)\beta\mu$, $M = \exp[(1+i)L/\mu]$, R_{mb} = thermal reflection coefficient

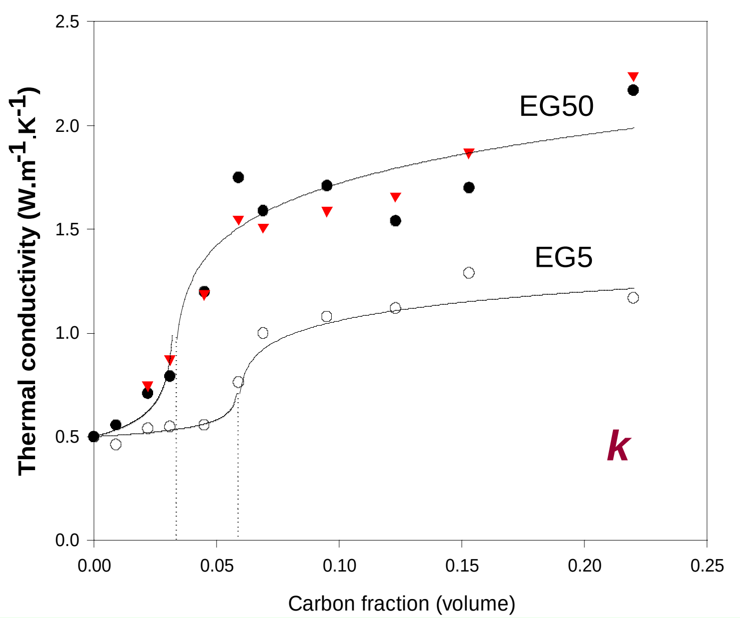
Electrical and thermal percolation in HDPE / EG

Classical percolation theory : $k = k_1\left(\frac{\phi_c - \phi}{\phi_c}\right)^{-s}, \quad \phi < \phi_c$ and $k = k_2\left(\frac{\phi - \phi_c}{1 - \phi_c}\right)^{t}, \quad \phi > \phi_c$

ϕ_c , threshold volume fraction - depends on particle sizes, shapes, composite topology
s = 0.87 and *t* = 2 , universal values of critical exponents



s = 0.87, *t* = 2



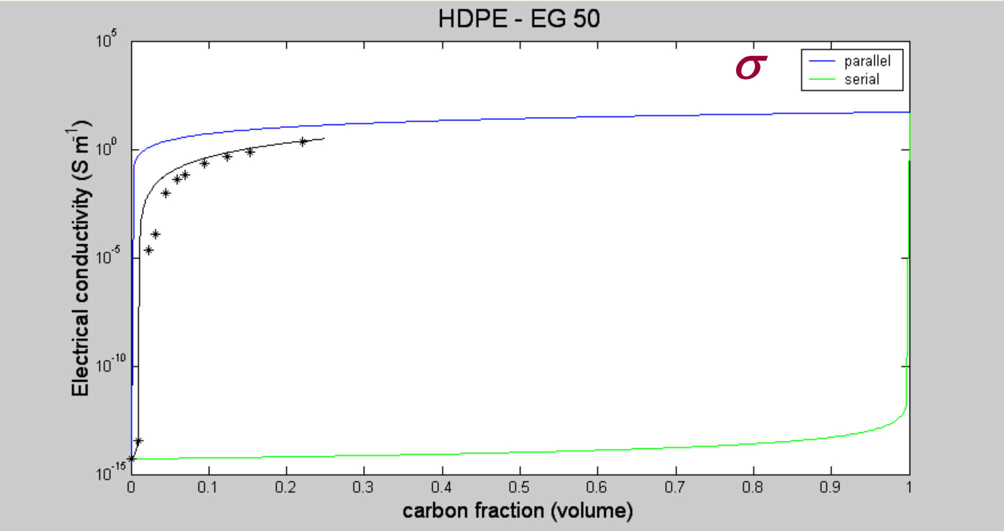
s = 0.20, *t* = 0.14 (EG50)
s = 0.08, *t* = 0.10 (EG5)

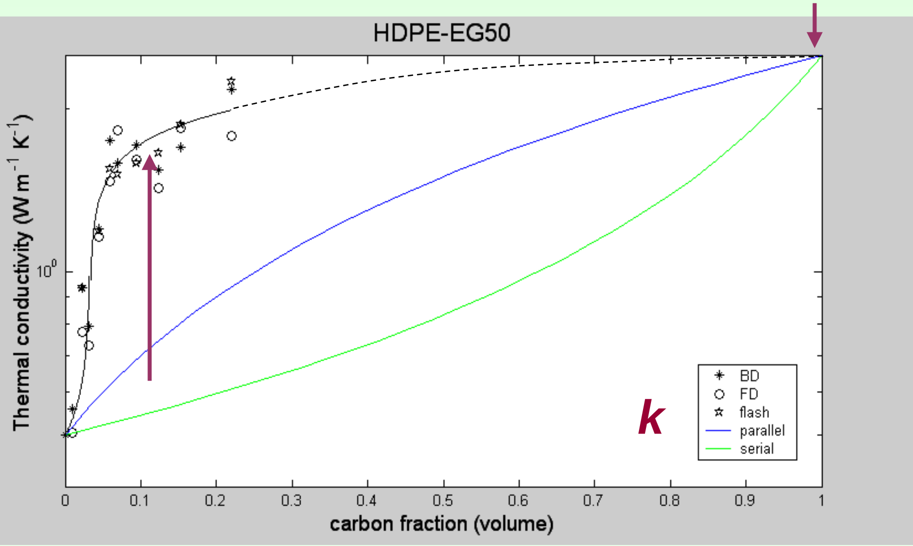
Experimental results (*k* computed from *a* and *c*):
- Two-step electrical percolation behavior
- Shifted thermal percolation due to tortuousity of the connectivity between particles
- Larger particle sizes are more effective in enhancing thermal and electrical transport

Role of grain boundary thermal resistance R_{th}

High local k_{micro}

R_{th} limits k_{macro}





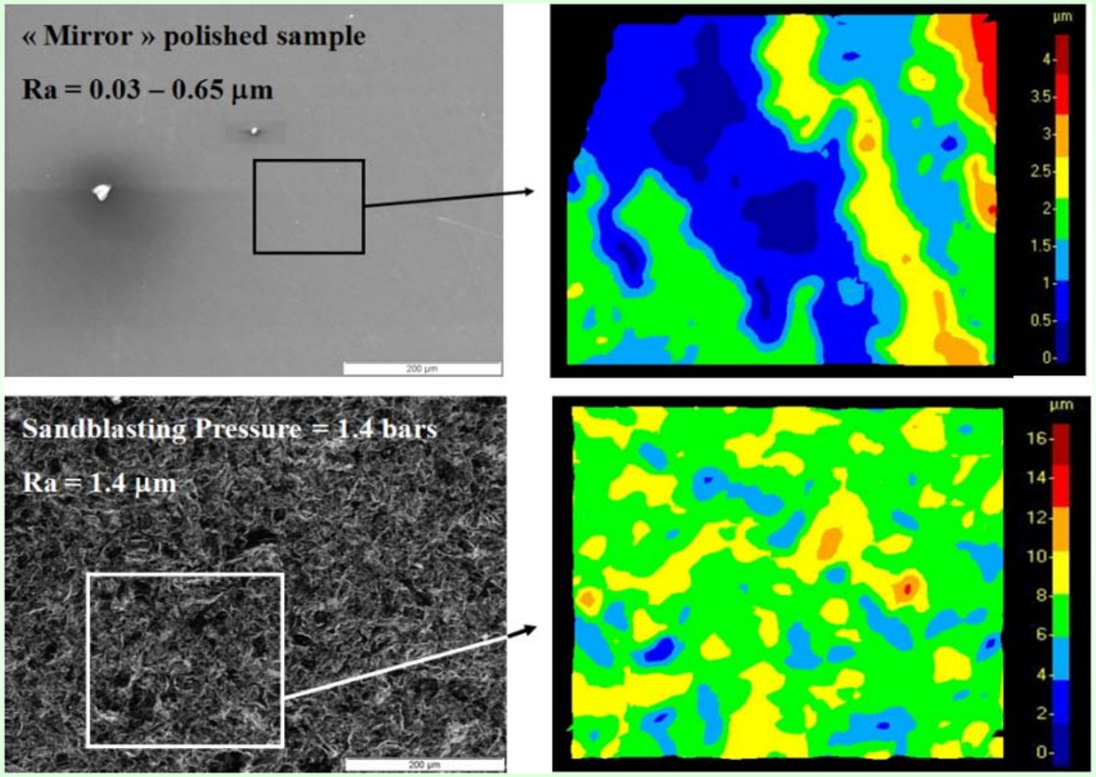
$$k_{\|} = (1-\phi)k_{low} + \phi k_{high}$$

$$k_{\perp} = 1/[(1-\phi)/k_{low} + \phi/k_{high}]$$

Conclusion: Thermal percolation and size-effects in HDPE/EG are associated with R_{th}

2. Thermo-optical characterization of surface roughness

SEM topography of sandblasted $Ti_{0.9}Al_{0.06}V_{0.04}$ surface

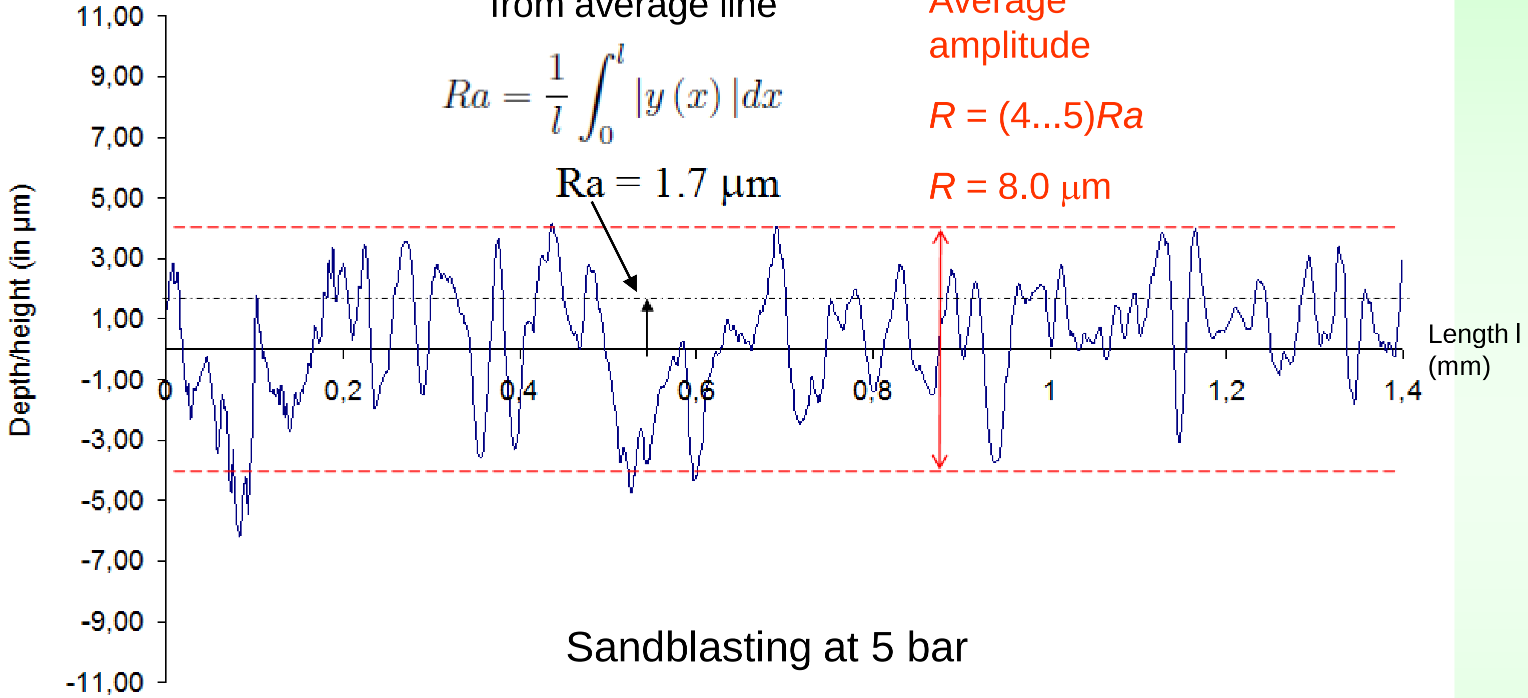


Applications:

Rough surface + hydroxyapathite → biocompatible coatings for prostheses in orthopaedic surgery

N. Dumelié, H. Benhayoune, C. Rousse-Bertrand, S. Bouthors, A. Perchet, L. Wortham, J. Douglade, D. Laurent-Maquin and G. Balossier, "Characterization of electrodeposited calcium phosphate coatings by complementary scanning electron microscopy and scanning-transmission electron microscopy associated to X-ray microanalysis", *Thin Solid Films*, 492, 131-139 (2005).

Profile reconstructed by X-ray microscopy



Mean deviation from average line

$$Ra = \frac{1}{l}\int_0^l |y(x)|dx$$

Ra = 1.7 µm

Average amplitude

R = (4...5)*Ra*

R = 8.0 µm

Sandblasting at 5 bar

Sandblasted (bar) ---------------- Reference	- ------------- Polished	1.5	2	3	4	5	Corrindon-blasted
Ra (µm)	0.03-0.65	0.70-1.23	1.0	1.2	1.4	1.7	3.6

Traditional PTR approaches of roughness

Models:

- Open pores (statistical)
- Spatial noise (statistical)
- Step-like layer
- Phase lag extremum
- Effective layer with frequency-dependent *k*, *c*

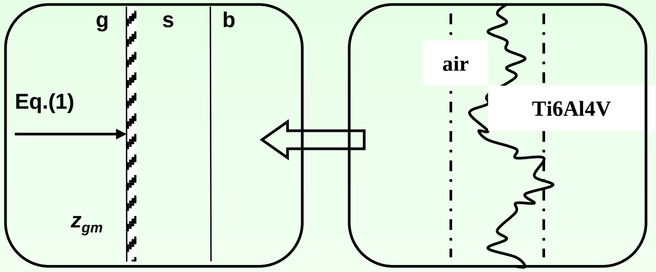
J.A. Garcia, A. Mandelis, B. Farahbakhsh, C. Lebowitz, and I. Harris, Thermophysical Properties of thermal Sprayed Coatings on Carbon Steel Substrates by Photothermal Radiometry,
Int. J. Thermophysics, Vol. 20, No. 5, 1999, p. 1587-1602.

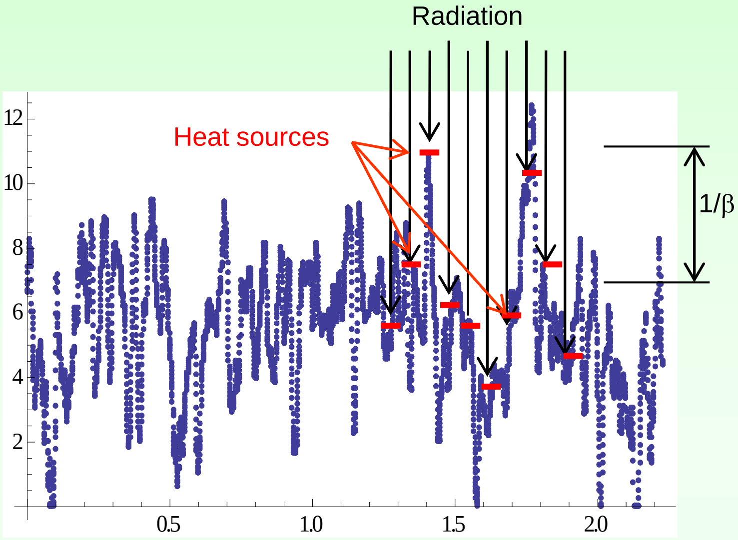
H. G. Walther, Photothermal inspection of rough steel surfaces,
J. Apply. Phys., Vol. 89, 5, 2001, p. 2939-2942

J.A. Garcia, Lena Nicolaides, Peter Park, Andreas Mandelis, and B. Farahkbahsh, Photothermal Radiometry of Thermal Sprayed Coatings: Novel Roughness Elimination Methodology,
Anal. Sci., Vol. 17, 2001, p. 89-92.

F. Macedo, A. Gören, F. Vaz, J.L. Nzodoum Fotsing, J. Gibkes and B.K. Bein, Photothermal characterization of thin films and coatings,
Vaccum, Vol. 82, 12, 2008, p. 1461-1465.

Model: equivalent layer with effective thermo-optical properties





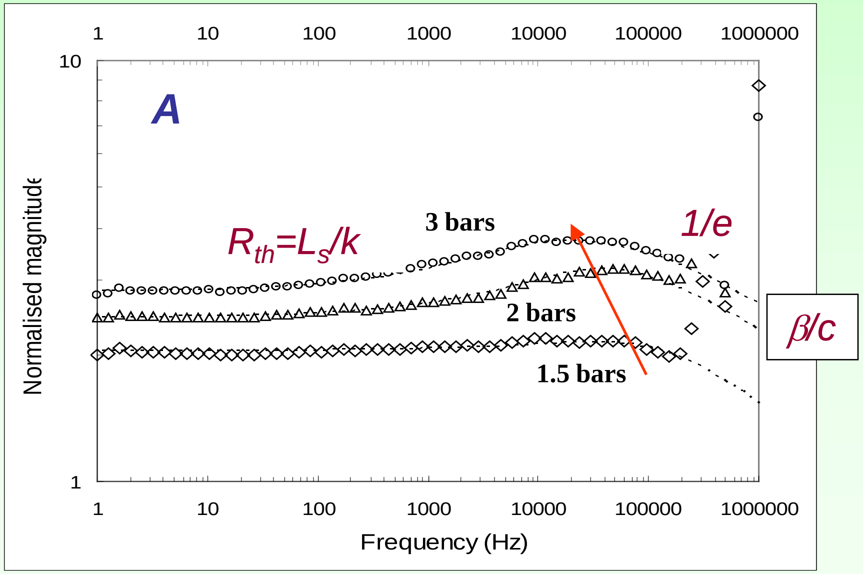
Equivalent layer (s):

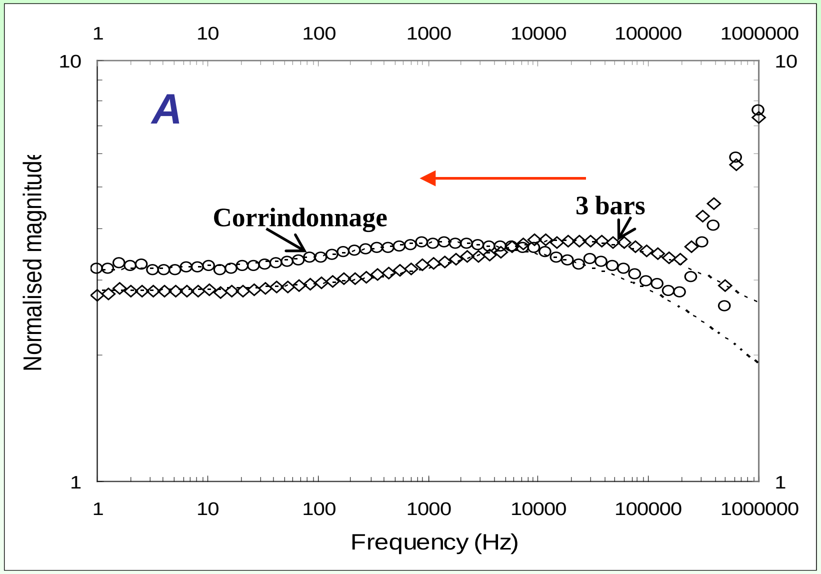
- homogeneous thermophysical properties
- "mixture" of bulk + air with volume fraction ϕ

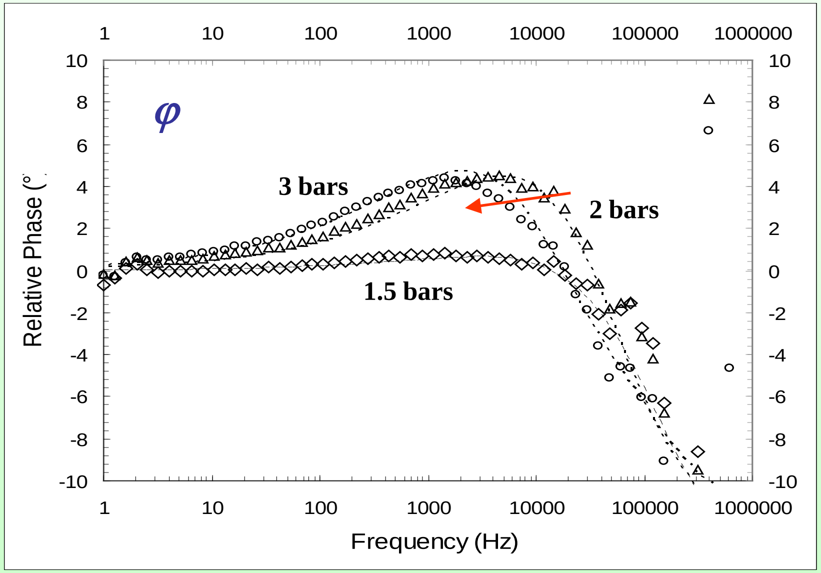
- topography → optical penetration depth $1/\beta$

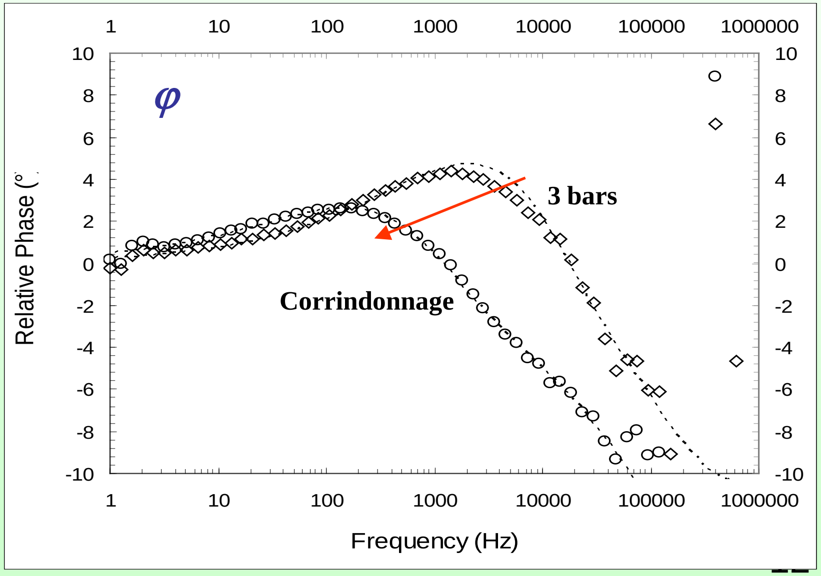
1-D analytical model: Eq. for FD-PTR

Experimental results and fits

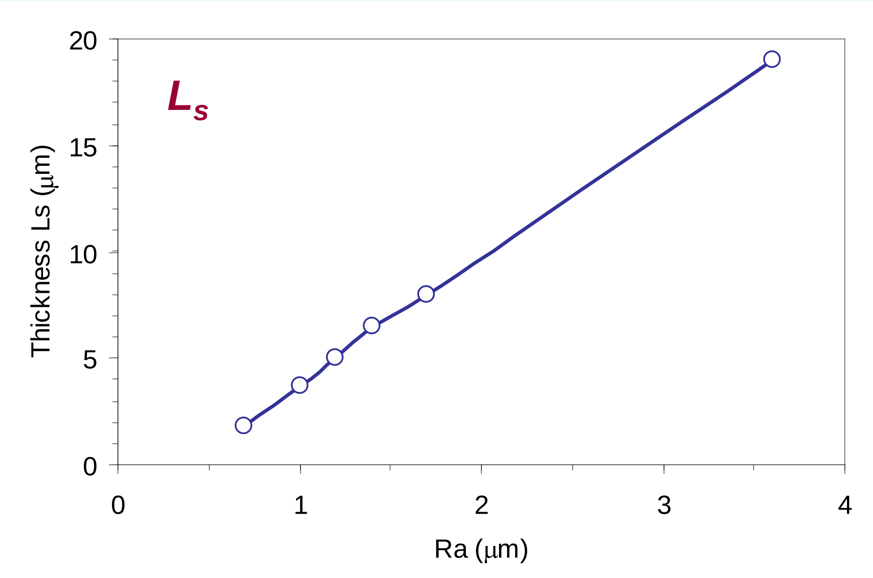


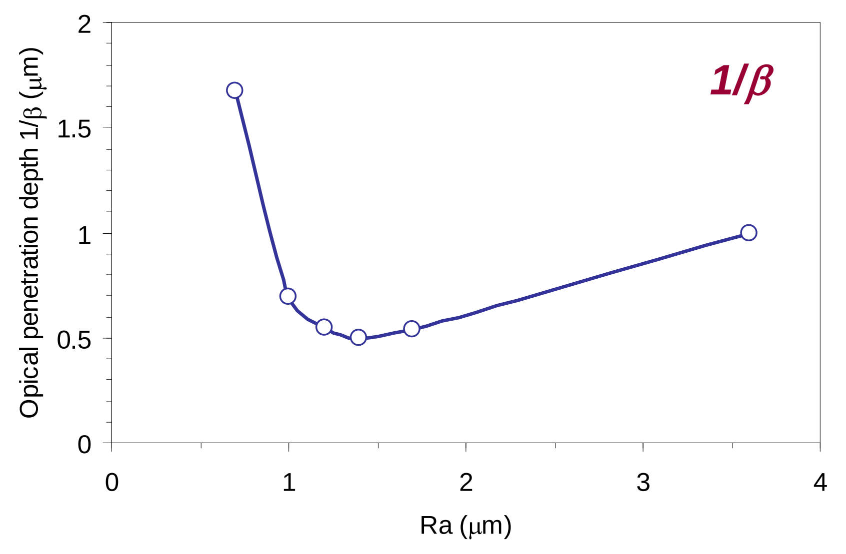


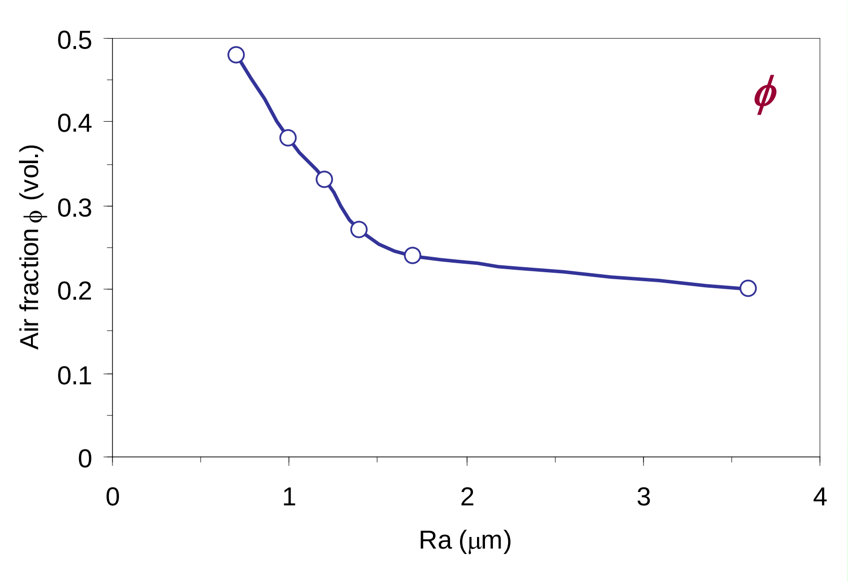




Fit parameters *vs.* roughness R_a





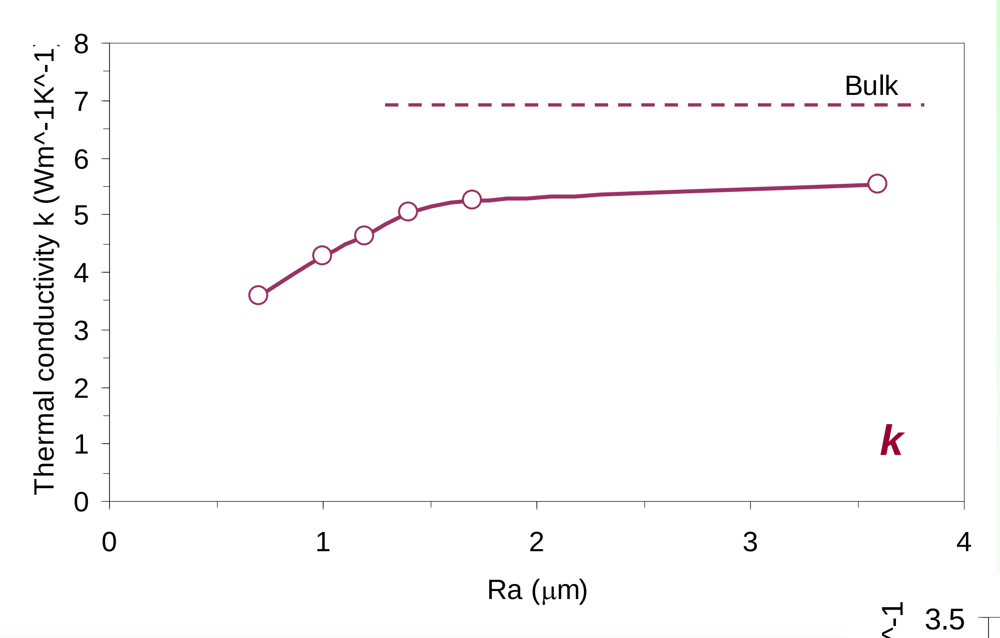


$L_s = 5.88\ R_a - 2.07$

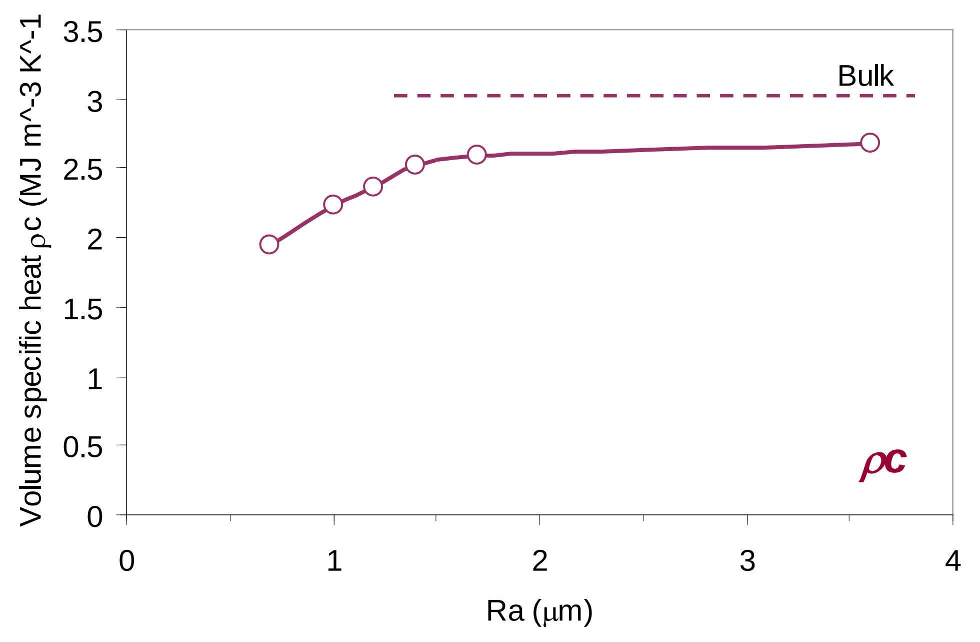
$R = (4...5)\ R_a$ (average roughness amplitude)

$\Rightarrow L_s \approx R$: roughness estimation is possible

Size-effect of equivalent thermophysical properties



$$k_{\parallel} = (1-\phi)k_{bulk} + \phi k_{air}$$



$$\rho c = (1-\phi)(\rho c)_{bulk} + \phi(\rho c)_{air}$$

Conclusions (2)

- Roughness *R* can be characterized by PTR
- Effective thermo-optical properties are size-dependent for $R_a < 2\ \mu\text{m}$

Other research subjects:

- **Thermal microscopy**
- **Thermal interface resistance of metallic coatings**
- **3ω hot wire method: nanofluids, glass transitions, anemometry**
- **Multilayer modelling**