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Characterization of reference standards for dirt by Laser Ablation Induced Photoacoustics (LAIP)

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Abstract. Measurements of surface cleanliness and dirt characterization are important problems in a wide range of processes in industry and production. Standard methods are in most cases cumbersome laboratory procedures that must be performed out of the production lines. Instruments and methods for cleanliness determination and dirt characterization require reference standards for calibration. For that purpose we built a possible dirt reference standard (DRS) made by films of graphite grease subjected to heat treatment for mechanical stabilization. The DRS characterization was performed by Laser Ablation Induced Photoacoustics (LAIP). The measurement of the thickness of the films was made by low-coherence interferometry.

1. Introduction

The measurement of the level of cleanliness of a surface is a problem of great importance in many industrial and technological processes and in scientific research, i.e fiber optics, airplane and siderurgical industries, in the conservation of objects of cultural value, in material science, etc.[1-3] We recently developed an on-line new method for measuring the cleanliness of any surface, by using laser ablation and acoustic detection. Based on this method we designed, patented (in Argentina and USA), and developed the first instrument in the world that measures on line the cleanliness degree of manufactured steel plate bobbins, during its production. [4-6]

Surface dirt deposited on an object may have different origins. It may come from handling, aging, pollution, or could be a consequence of fabrication or industrial procedures. In many cases surface dirt can be described as a dark thin film of organic substances as oil or grease in which a more or less homogeneously distribution of particles of different compounds is present. Most of these particles are black, giving the dirt the dark aspect. In other cases, like “clean industries” as micro-electronics, micromechanics, data storage, etc. usually, the main dirt is composed by small isolated particles added to the substrate.[3]

Surface dirt can be found on any object, but there is no simple way to produce it in controlled conditions. Therefore, most of the laboratory methods that measure the cleanliness of a surface use an indirect calibration reference. To our knowledge, there are not dirt reference samples, for calibration purposes, that can be manufactured in reproducible conditions, with well controlled thickness, particle size, and a homogeneous distribution of the dirt. For that reason we developed a possible dirt reference standard (DRS) made by films of graphite grease with concentrations up to 5% and thickness between 30-55 microns, to be used as reference patterns of dirt.

The DRS characterization was performed by using *Laser Ablation Induced Photoacoustics* (LAIP) and the measurement of the thickness of the films was made by low-coherence interferometry.

2. Experimental

2.1. Samples Design

2.1.1. Preparation of graphite grease. Dilutions were prepared with 0.5, 1, 1.5, 2.5 and 5 % w/w graphite in pure lithium grease, mechanically mixed and placed in vacuum for 24 hours while maintaining saturation vapor pure grease.

*2.1.2. Samples Manufacture***.** Films of graphite grease were made on steel AISI 4140 substrates, previously cleaned with ultrasonic solution 50/50 v/v % of isopropanol and acetone.The mixture was extended by sliding a glass in a single movement. Tapes were used as guides. The samples were heated in an oven at 180 º C for 10 minutes covered by a watch glass.

2.2. Experimental Set-Up used for LAIP.

A schematic of the set up used for LAIP is shown in figure 1. A Q-switched Nd: YAG laser with pulse duration of 7 ns (FWHM) operating at a wavelength of 1064 nm is directed normal to the sample surface. A neutral density wedge filter is used to change the energy of the laser pulse to produce laser fluences (energy per unit area), **F**, ranging $0 < F < 3.5$ J/cm^2. A microphone picks up the emitted sound, registered on a digital oscilloscope. The amplitude of first peak to peak of the Acoustic Signal (**S**) was measured as a function of the laser fluence. The pulse energy was measured using an energy meter with a pyroelectric detector, splitting the laser pulse by means of a calibrated beam splitter.

Taking into account the non-homogeneous spatial energy distribution of the laser pulse and then the effective area of the laser spot, the fluence values calculated in this work have an estimated uncertainty of c.a. 30%. The sample is moved perpendicularly to the laser beam so that each laser shot impinge on a different region.

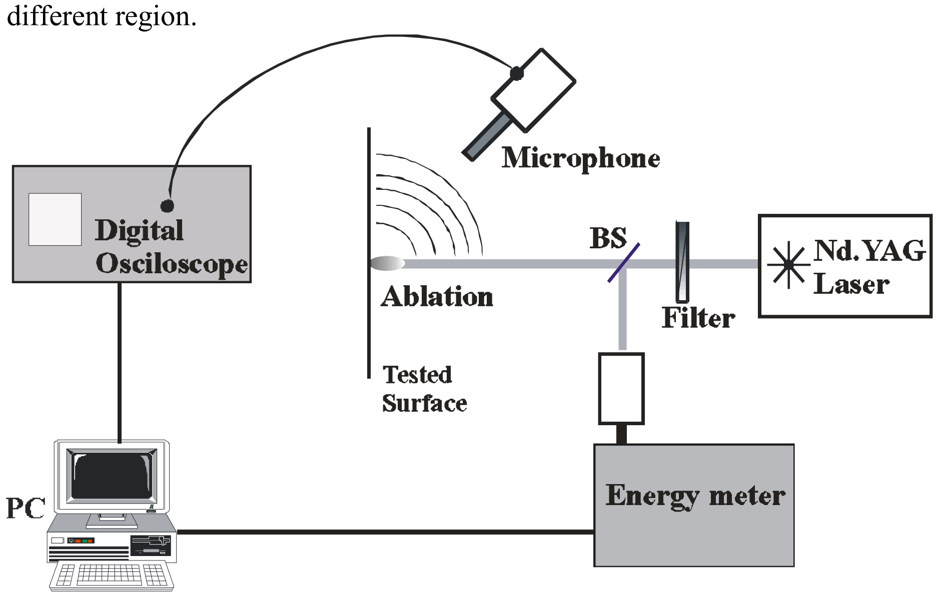

Figure 1. Experimental set-up used for LAIP

2.3. Thickness measurement of the sample.

The measurement of the thickness of the films was made by determining the step at the edge of the grease film by low-coherence interferometry [7, 8]. This experimental arrangement allows to measure thicknesses between 10 microns and 1 millimeter with an error of the order of one micron.

3. Characterization by LAIP

Figure 2 shows the dependence of the peak to peak amplitude of the acoustic signal as a function of the laser fluence for the DRS previously described. The laser fluence ablation threshold (**F_0**), changed between 0.9 - 1.5 J/cm^2 for different concentrations of graphite in the sample. A linear relationship **S** vs. **F** was observed for laser fluences larger than **F_0** for all the samples.

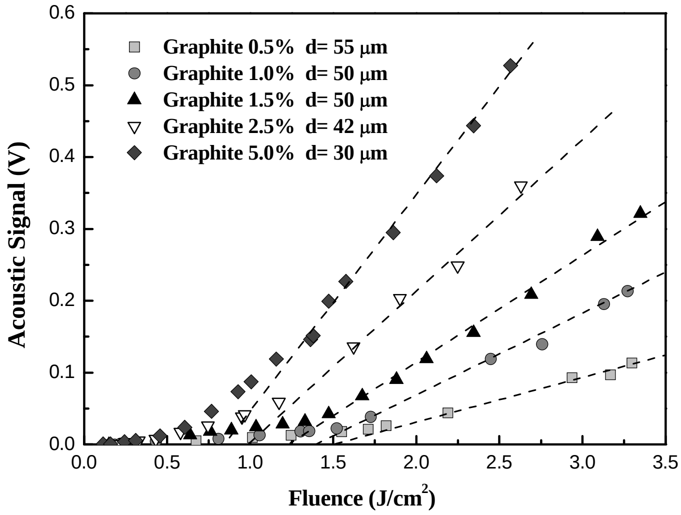

Figure 2. Peak to peak amplitude of the acoustic signal as a function of the laser fluence for five different concentration of graphite in DRS samples. **d** = thickness of the films measured by low-coherence interferometry.

4. Model for the acoustic signal

Figure 3 shows the dependence of **$S/(F - F_0)$,** obtained from the slopes of figure 2, as a function of the dirt absorption; **$(1- e^{-A})$**, where **$A= \alpha d C$** is the absorbance of the film; α is the average particle cross section (assumed constant); **d** is the film thickness, and **C** is the graphite concentration.

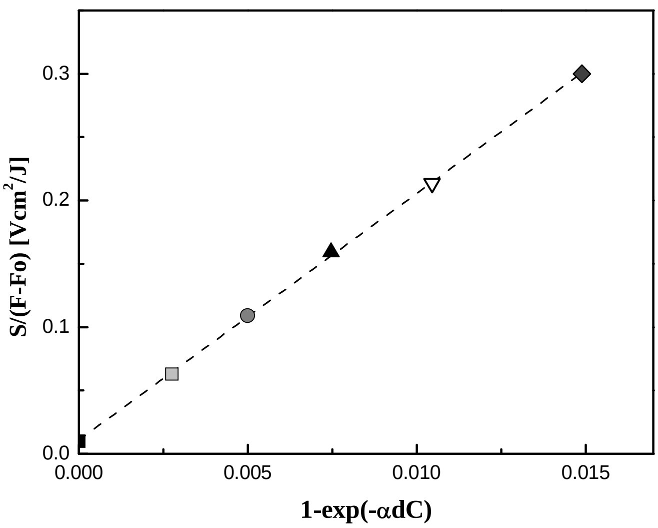

Figure 3. Dependence of the slopes **$(S/F-F_0)$** obtained from figure 2 as a function of the film absorption **A** of the DRS.

As it can be seen from figure 3 a linear relationship between the fluence normalized acoustic signal and the dirt absorption was found. The interception at the origin corresponds to the acoustic signal generated by the pure grease without graphite. Hence the Acoustic Signal can be represented by the equation [1]:

$$S = K\,(F-Fo)\,(1- e^{-\alpha C d}) \qquad (1)$$

Where $K = 19.50 \pm 0.14$ Vcm^2/J, is a constant that depends on the nature of the sample, the detector geometry and sensitivity.

5. Conclusions

Reproducible films of graphite grease with concentrations up to 5% and thickness between 30-55 microns were designed and manufactured to be used as dirt reference standards for cleanliness determination.

By using photoacoustic induced by laser ablation and a simple model [Eq. (1)] the characterization of the samples as a function of the average particle cross section, the graphite concentration and the thickness of the sample can be performed.

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