

2-Color LII measurements of carbon black: Interpretation for quantitative measurement of fineness

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The 2-color LII technique is utilized to provide on-line measurement of the fineness level of carbon black during its manufacture. The BET surface area does not follow the expected " $1/d$ " behavior, and the soot volume fraction measured by LII has been found to be dependent upon laser fluence. Reasons for these anomalous behaviors are not yet known.

Introduction

The specific surface area of aggregated nanoparticles, related to primary particle diameter, is a key parameter in the production of carbon black. Currently, there is no available technique to provide on-line, real-time measurement of this parameter during the production of carbon black. Rather, periodic sampling, followed by adsorption measurements (such as the BET method), is typically utilized by the carbon black industry to provide this information. It is well known that 2-color LII has been utilized to measure soot volume fraction (svf) and primary particle size of an aerosol cloud (see e.g. [1-2]).

Experimental Equipment

A small amount of carbon black aerosol is continuously drawn from a commercial carbon black reactor system using a venturi eductor sampling device. A critical orifice, attached to the inlet of the venturi eductor, controls the flow rate of aerosol that is sampled. Because of the high dilution ratio utilized (approximately 200:1), the dilution gas comprises >99% of the background gas during the LII measurements. The gas and aerosol stream are cooled to ambient temperature prior to entering the LII instrument.

A commercial, 2-color LII instrument (Artium Technologies) is utilized to make measurements at a pulse repetition frequency of 20 Hz. For each laser pulse, the incandescence signals are measured at wavelengths of 400 nm and 780 nm. The incandescence signals, measured over a duration of approximately 1000 ns at intervals of 10 ns, are converted to intensity values using previously determined calibration factors. From the intensity values, the soot volume fraction and the particle temperature are calculated. The decay rate of the particle temperature is utilized as a measure of particle fineness [1].

Measurements

The LII measurements were performed during the production of two different grades of carbon

black (N234 and N299). During the production process, the specific surface area of the carbon black was purposely varied a small amount from the target to determine the capability of the LII technique. For comparison, the BET measurements of specific surface area were performed on carbon black samples collected during the tests. Although the 2-color LII technique is able to resolve small changes in specific surface area, BET surface area does not exhibit the expected " $1/d$ " dependence. Furthermore, the power of the exponent seems to be different for different grades of carbon black.

One of the benefits of using 2-color LII (as opposed to a single detector technique) is the ability to utilize low laser fluence levels, where carbon sublimation is negligible [3]. In this regime, the soot volume determined by LII should not vary with laser fluence. As seen in Figure 1, the indicated svf shows a marked and unexpected dependence upon laser fluence.

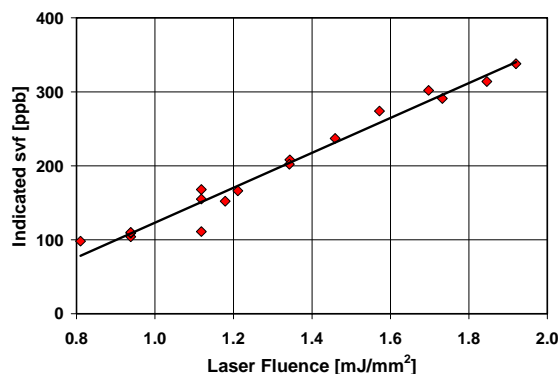


Fig. 1: Indicated Soot Volume Fraction

References

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