A COMPACT ELLIPSOMETER FOR CHARACTERIZATION OF SIZE AND SHAPE OF NANO-PARTICLES AND AGGLOMERATES

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With exponentially increasing demand for nanopowders, the need for improved particle characterization technology for nano-particles and agglomerates is more acute than ever before. In order to produce nanopowders consistently and in a cost-effective way, on-line monitoring of particle size, size distribution, and shape is needed. In addition, nanopowders have a strong tendency to agglomerate and form highly irregularly shaped structures. Agglomeration presents a major problem to nano-material based processes. Thus, the characterization of particle clusters (number of monomers, cluster shape, delineation of monomer sizes and size distribution) is also important for understanding process production mechanisms and eventual process refinement and optimization.

Standard techniques used to characterize micron and sub-micron size particles do not have the fine resolution needed for nano-sized particle detection. Methods based on electrical resistance (Coulter counters), sieving, turbidimetric techniques, diffraction based light scattering techniques and photon correlation spectroscopy are all inadequate for on-line characterization of nano-sized particles. Sedimentation methods for characterizing nano-sized particles are commercially available; however, they are slow and yield only the information about effective diameters. Newer methods, such as acoustic spectroscopy and electroacoustic spectroscopy, have become commercially available and show promise, but they also have their drawbacks (e.g., limited size range, inability to measure dry powders or dilute systems, cannot characterize particle shape). Currently, reliable characterization of nano-materials has been mostly by electron microscopy (e.g., TEM, SEM, atomic force microscopy). These techniques provide qualitative information, but are extremely tedious and not appropriate for quality control applications. Because they are unable to sample a statistically significant number of particles, they cannot give accurate values for average size or the size distribution.

Recently, the Synergetic Technologies teamed up with the University of Kentucky to develop a practical and fast particle characterization system for on-line particle diagnosis. Characterization of nanopowders is realized using the measurement of angular light scattering signals. The idea is based on the quantification of how the irregularly shaped particles alter the polarization of incident light. This compact instrument is designed following the laboratory studies conducted at the University of Kentucky over the years [1-5]. With this tool, particle size and size distribution can be determined precisely and accurately for a wide range of nano-scale particles sizes and types. The methods developed in this project can be used to distinguish between single and agglomerated particles.

THEORY

The theory behind the present technique is discussed in the literature [1-5]. Here, we will only summarize the main concept. Imagine a set of four filters, each of which, under natural illumination, will transmit half the incident light. The first filter is isotropic, letting waves in all polarization settings go through. The second filter is a linear polarizer oriented horizontally. The third filter is a linear polarizer oriented at 45 degrees from horizontal in the clockwise direction. The fourth is a circular polarizer opaque to cylindrical polarization states. By measuring the irradiance that passes through each of these filters individually, (I_1, I_2, I_3, and I_4) we can construct the Stokes vector.

$I = 2I_1$ This is simply the irradiance of the original beam.
$Q = 2(I_2-I_1)$ Tendency of to be horizontally (>0) or vertically (<0) polarized.
$U = 2(I_3-I_1)$ Tendency to be linearly polarized to +45° (>0) or –45° (<0).
$V = 2(I_4-I_1)$ Tendency to be circularly polarized right (>0) or left (<0).

Therefore, if polarized light is incident on a particle cloud, the scattered light will have the finger-prints of the particles that scatter the light. These finger-prints then can be used to identify the particle characteristics.

We can write the relationship between the incident light and the scattered light as:

$$\begin{Bmatrix} I_s \\ Q_s \\ U_s \\ V_s \end{Bmatrix} = \frac{1}{k^2 r^2} \begin{pmatrix} S_{11} & S_{12} & S_{13} & S_{14} \\ S_{21} & S_{22} & S_{23} & S_{24} \\ S_{31} & S_{32} & S_{33} & S_{34} \\ S_{41} & S_{42} & S_{43} & S_{44} \end{pmatrix} \begin{Bmatrix} I_i \\ Q_i \\ U_i \\ V_i \end{Bmatrix}$$

The 4 x 4 matrix is the *scattering matrix* for a single particle. The sixteen scattering matrix elements are functions of scattering angle and the physical and morphological details of each particle. For a single particle, only seven of these elements can be independent, corresponding to the four moduli $|S_j|$ (j=1,2,3,4) and the three differences in phase between the S_j.

The Stokes parameters of the light scattered by a collection of randomly separated particles are the sum of those of the individual particles. Therefore, the scattering matrix for such a collection is merely the sum of the individual particle scattering matrices. For particles in a sample that have a preferential orientation, S_{ij} elements do not, in general, vanish, e.g., cylinders or scattering at oblique angles. Thus, by identifying each of the S_{ij} elements, a particle system can be: 1) more accurately characterized (sized) compared to systems which just measure the upper left-hand quadrant; and 2) more and better information can be retrieved regarding particle shapes.

Key to obtaining the various matrix elements and, thus, characterizing the particle system, is the ability to modulate the polarization before and after scattering. This can be performed by placing a series of polarizers and retarders (half-wave and quarter-wave plates) between the light source and the particles and between the particles and a light detector. Such an arrangement is crucial to obtaining variable ellipticity of both incident and scattered beams. By adjusting the orientation of

the optical axes of the polarizers and retarders, the measured light intensity contains the fingerprints of the different scattering matrix elements. The orientation of each retarder and polarizer, which determines the phase differences between the transverse peaks of the light intensity, can be expressed in matrix form.

In this poster, we will discuss the particle characterization system and outline the preliminary results obtained from the experiments for various particles.

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