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SURFACE ENERGY ANALYSIS: PREDICTING ADHESION IN FIBER REINFORCED COMPOSITES

Fiber-reinforced composites are a rapidly growing part of the revolution in advanced materials development of the 80's and 90's. There are numerous examples of fiber-reinforced materials:

- A carbon fiber reinforced thermoplastic such as PEEK (polyethyletherketone) can form a composite material much lighter and stronger than steel that is ideal for automotive, aerospace, and sporting goods applications.
- A glass fiber-reinforced material such as fiberglass is a commonly used composite with a variety of applications which require a tough, lightweight, corrosion-resistant product.
- Organic fibers such as the aramid fiber Kevlar 49 (poly-p-phenylene terephthalamide) are commonly used as a reinforcement in a rubber matrix to make tires or in an epoxy matrix to make a high strength plastic.
- Wood, a fibrous blend of cellulose, lignin, and other polymeric materials is a "natural" composite material that is often adhesively bonded with a resin to make a plywood or paperboard product.
- Ceramic fibers such as alumina or silicone carbide are being used as a reinforcement in a metal-matrix composite (i.e. aluminum or titanium) for low-density structural material fabrication.

A common design feature of any composite material that must be understood, engineered, and controlled is the interfacial bond strength. Essential properties of the composite such as interlaminar shear and flexural strength can be achieved by controlling interfacial bond strengths. Adhesion at the fiber/matrix interface is, therefore, a critical property of the composite that must be measured.

Often the interface can be engineered by modification of the fiber surface chemistry to optimize the adhesion between fiber and matrix. The surface energy of a reinforcing fiber is a measure of the adhesive properties of the fiber surface that can be used to predict adhesion at the fiber/matrix interface.

SURFACE ENERGY MEASUREMENTS ON SINGLE FIBERS

The surface energy of a fiber, or any solid material, is a fundamental thermodynamic property that can be measured in the laboratory with the proper instrumentation. Surface energy calculations are derived from contact angle measurements made between the fiber and standard liquids of known surface tension. To make these measurements on a small diameter fiber, however, requires a high degree of sensitivity. The Thermo Cahn DCA 322 microbalance-based system offers the sensitivity required for single fiber analysis. Fibers as small as 5 µm in diameter are routinely studied with the system, with minimal sample preparation required.

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To prepare a single fiber sample for analysis on the DCA, a nichrome support wire (0.1 mm diameter, 50-60 mm long) is first cut and a hook is formed on one end to facilitate a convenient attachment point with the balance. Next, a 10-20 mm sample of the fiber to be analyzed is attached to the other end of the nichrome wire with a small drop of a fast drying (i.e. cyanoacrylate) adhesive. A microscope may then be used to verify that only one fiber has been attached and the experiment may then proceed with the first probe liquid. Typically, several probe liquids are chosen (i.e. water, methylene iodide, DMSO, bromonapthalene, form amide, and glycerol are common) to improve the reliability of the surface energy measurement. Surface tensions and polar and dispersive components for these liquids are well-known and can be found in the literature, or measured against polyethylene or Teflon with the DCA. The diameter of each fiber may be subsequently determined with the aid of a low surface tension liquid (i.e. hexadecane) that will wet-out the surface of the fiber, and thus permit a direct calculation of fiber perimeter.

The work of adhesion is, by definition, the work required to reversibly separate one bulk phase (i.e. a liquid) from another bulk phase (i.e. a solid). It is easily calculated from a contact angle measurement:

$$WA = \gamma L(1 + COS\ \theta) = \gamma L + \gamma S + \gamma LS$$

Where γL is the liquid surface tension, theta (θ) the contact angle, γS is the fiber surface energy, and γLS is the interfacial tension between fiber and liquid surfaces.

Useful expressions for the work of adhesion have been derived by Fowkes, Kaeble, Wu, and others, to separate the surface energy of a solid into two or more terms to classify the polarity of a solid in a similar fashion as is done with a liquid. References to the Geometric Mean (Fowkes, Kaeble), and the Harmonic Mean (Wu) equations are abundant in the literature.

In a similar fashion as described above, the work of adhesion for a hypothetical composite interface can be calculated from contact angle data, by using the same set of liquids to study the wettability of both reinforcing fiber and matrix surfaces. If the calculated work of adhesion between fiber and matrix exceeds the so-called work of cohesion for the matrix itself, a good adhesive bond is predicted to occur, with cohesive failure of the matrix predicted to occur before an adhesive failure at the fiber/matrix interface would occur. An elegant example of this work of adhesion concept applied to a carbon fiber composite can be found in the work of Briggs et al. (1)

FIBER SURFACE TREATMENT PROCESSES

A variety of surface treatment processes can be applied to a fiber to change the physical and/or chemical nature of the fiber surface. Among the more common are the electronic treatment processes - gas plasma and corona discharge. Physically, the fiber surface can be modified by roughening or etching of the surface which effectively increases the available surface area for bonding with the matrix.

If reactive gas plasma is used, however, the fiber surface can be chemically modified by the introduction of a process gas such as oxygen, argon, ammonia, or a hydrocarbon with reactive functional groups (i.e. hydroxyls, carbonyls, carboxyls). The resulting ultra-thin coating (less than 10 angstroms thick) that is deposited onto the fiber surface, is readily characterized by the DCA, but may not be detectable with other surface spectroscopy techniques (i.e. ESCA) which are not sensitive below 40-50 angstroms.

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