

Local Viscoelastic Measurements using Interfacial Force Microscopy

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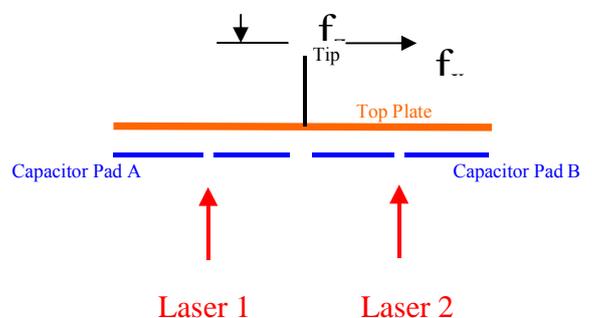
The detailed analysis of the mechanical properties of many materials, for example fiber reinforce composites, often requires being able to make measurements locally, for example, in the region near the interface between the fiber and its matrix, i.e., within the “interphase” region. An important example involves studies involving the aging of propellants. These materials are normally composed of high-energy particulates distributed in an elastomer matrix. The role of the elastomer is to maintain a constant burn-front velocity, i.e., constant thrust, even under the enormous temperatures and pressures present during the burn. However, elastomers often oxidize and become brittle over time, which can lead to cracking, especially under the influence of elevated pressures and temperatures. Open cracks expose additional fuel particles, which further accelerates the burning process until, at some point, the system switches from being a propellant to a very dangerous explosive.

In order to study in detail the aging process, it is necessary to be able to measure the mechanical properties of the matrix in the region very near the fuel particles, i.e., the “interphase” region, as a function of the aging period. With the rapid development of scanning-probe techniques like the atomic force microscope (AFM), measurements of such local properties have become reasonably routine. However, many of these techniques rely on the deflection of an elastic member, such as the weak cantilever beam in the case of the AFM, in order to measure forces and displacements and this creates problems in obtaining quantitative values for mechanical parameters. The worst of these problems involves the inherent instabilities that give rise to the so called “snap-to-contact” and “pull off

force” phenomena, that is, the value of the force just before the tip suddenly snaps out of contact. These shortcomings make it impossible to quantify the nature of the interfacial bond, as well as, the quantitative “work of adhesion”.

In this presentation, I introduce an alternative scanning-probe technique, i.e., the Interfacial Force Microscope (IFM), which overcomes these difficulties by utilizing an electrostatic force-feedback technique to eliminate the instabilities and permit the measurement of stable and quantitative values of adhesion and local viscoelastic properties. These advantages will be illustrated with applications to measurements of the local adhesive and viscoelastic properties for several material systems.

The IFM sensor consists of a kind of “teeter totter” (TT) supported by torsion bars above two capacitor pads with a sharp tip located in its center. Forces on the tip cause the TT to displace slightly in vertical or rotary motion, depending on the nature of the force. The displacement is detected by two independent laser interferometers and the displacement is balanced by a force-feedback technique. Thus, the instability is stabilized and the voltages required yield quantitative force values. A schematic of the sensor is shown schematically in the following sketch.



The tips generally consist of FIB shaped diamond, etched tungsten or fired glass, depending on the material being studied.

In terms of local viscoelastic measurements, we chose as a demonstration what has to be one of the most extreme examples of a dilatant material, one that is often referred to as a “solid liquid”. This is the children’s toy “Silly Putty” (SP). Here we are dealing with a material that can be slowly strained to extreme levels but, rolled into a ball, will bounce off hard surfaces with high coefficients of restitution. The assumption was that if you could analyze the local viscoelastic properties of Silly Putty, you could analyze these for any viscoelastic material.

For this difficult material, in order to satisfy the contact-mechanics constraints and obtain quantitative results, we chose to bring the tip into a brief, stable contact, take a quick micro-photo of the contact, allowing a value for the initial contact area to be obtained, and immediately perform a relaxation measurement by suddenly stepping into the surface by a nm-level tip displacement, while recording the resultant time-dependent force. The relaxation behavior reveals the details of the viscoelastic properties. A typical example is shown by the squares in Fig. 1 for a tip step of 100 nm. This result was then characterized by a two-component Maxwell spring/dashpot model. shown as the solid line in Fig. 1, in order to obtain both the real and imaginary terms of the viscoelastic response. After proving linearity, by demonstrating that the behavior was identical when scaled by the initial contact area and the value of Δz taken over a large range of Δz , the Fourier transform of the relaxation behavior allows the frequency response for both the real and imaginary components to be calculated, as shown in Fig. 2. The validity of this approach is verified by a direct comparison with data obtained from a classic rheometer provided by the Cambridge Polymer Group. Considering the contact-mechanics problems, this fit is truly remarkable and clearly demonstrates the ability of the IFM to obtain very local and quantitative data on viscoelastic materials.

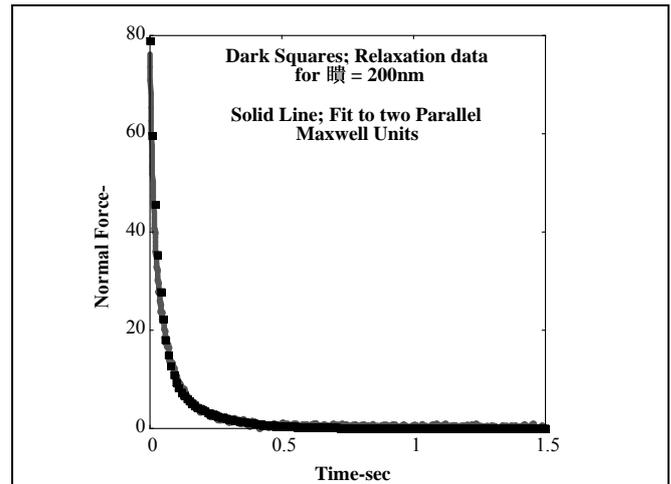


Fig. 1, Fit to relaxation data for a displacement of 200 nm with a parallel, two-element Maxwell model consisting of springs and dashpots in parallel.

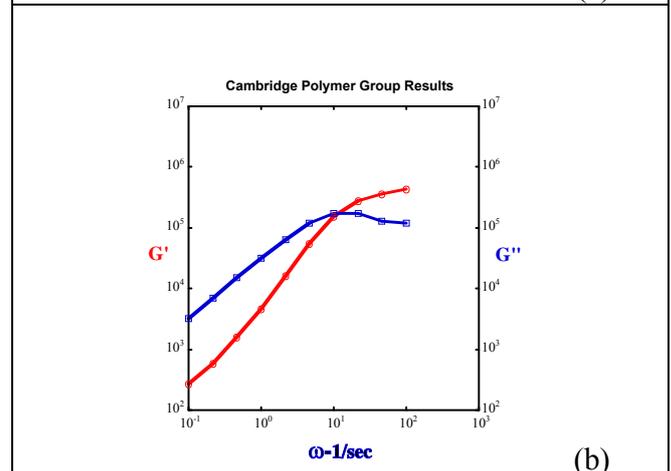
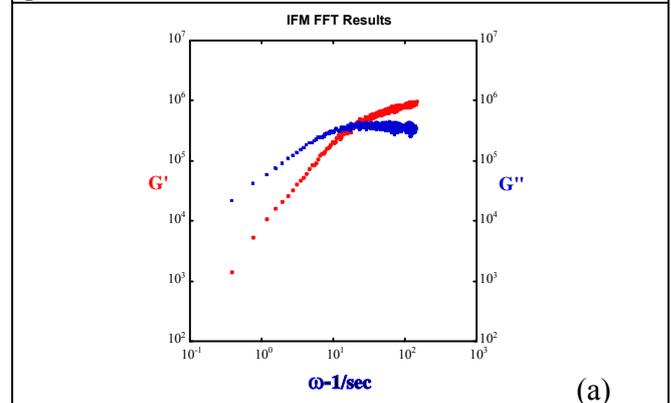


Fig. 2, A direct comparison of the IFM FFT results (a) and those from the Cambridge Polymer Group (b). The former involved a single two second experiment, while the latter represents 10 individual measurements with a significant amount of signal averaging at each point.