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A tensile test device for in situ atomic force microscope mechanical testing

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Abstract

The microstructure and mechanical behavior of polymeric-based materials can be controlled at the micro- and nanometer length scales through blending, copolymerization, and the incorporation of micro- and nanometer particles. To facilitate the study of morphology, deformation mechanisms, and mechanical properties of micro- and nanocomposite materials, a tensile testing machine with an integral commercial atomic force microscope (AFM) was designed and built. This testing machine determines the macroscopic stress–strain behavior of materials under different controlled loading conditions, and simultaneously allows the microscopic structure changes to be observed using the AFM.

Keywords: Atomic force microscope; Nanocomposites; Microcomposites; Stress–strain behavior; Chevron effect; Triblock copolymer; Mechanical testing; Tensile testing

1. Introduction

This paper focuses on the design, manufacture, and testing of an in situ testing machine that in combination with a commercial atomic force microscope (AFM) measures the macroscopic stress–strain behavior of polymeric-based micro- and nanocomposite materials under controlled loading conditions; it also allows the straining microscopic structure to be observed with nanometer resolution.

Nanocomposites are polymeric-based materials that have significantly enhanced mechanical performance as well as other properties such as electrical conductivity, resistance to permeability and abrasion resistance while maintaining the low inherent density of polymers. The microstructure and mechanical behavior of polymeric materials can be tailored via the incorporation of second phase particles into the polymer, which can be done, for example, through the blending of two or more polymers, and through copolymerization. These processes act to produce multi phase morphologies where the length scales of the different underlying phases may range anywhere from nanometers to tens or hundreds of micrometers. The microscopic geometry and properties of the constituent phases governs the resulting macroscopic mechanical behavior [1–5].

In order to better design and tailor polymeric-based blends, micro- and nanocomposites, a better understanding is needed of the connections between microstructure and mechanical behavior. Typically, specimens are evaluated before and after tensile testing using instruments such as atomic force microscopes [6–12]. Bobji and Bhushan [7] successfully performed in situ tensile testing on magnetic tapes in order to study the growth of microcracks. The experiments observed the growth of these cracks with an AFM as a function of strain, but did not include any measurements of the occurring stresses. Oderkerk et al. [6] used a manual stretching device capable of producing a maximum strain of 100% to test 20 μm thin nylon-6 samples. The stretching

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device did not include any force measurements but included a support underneath the sample to avoid vibration during the AFM scan. Opdahl and Somorjai [9] examined thin samples of polyethylene (LDPE and HDPE) with a strain of up to 150% and were able to detect changes in both surface structure and its properties using an AFM. Vogel et al. [12] used an AFM to characterize materials whose properties were altered by the addition of nanosized particles.

Ideally, however, a tensile testing machine is needed that can perform standard tensile test measurements in an incremental manner while between steps allowing an atomic force microscope to directly observe the strained sample microstructure.

Atomic force microscopes are readily commercially available, and thus it makes good design sense to design the system around a commercially available instrument. Scanning of the microstructure surface would be done in tapping mode where the AFM measures the topography of the sample by slightly tapping the surface with the tip of a vibrating cantilever beam at high frequency while scanning in the x- and y-directions [13]. To give an idea of the requirement for nesting the AFM with the tensile tester, consider that the AFM's silicon cantilever is typically 125 μm long × 45 μm wide × 4 μm thick. The tip is also made of silicon with a height of 17.5 μm and a tip radius of less than 10 nm. A piezoelectric driver excites the cantilever that holds the tip at resonance frequency (typically 100–300 kHz). In addition, the AFM scans the tip in a 3 μm × 3 μm plane.

The changes in oscillation amplitude of the scanning tip due to the attractive forces between the surface being scanned and the tip, describe the topography of the sample. Therefore, any vibration or oscillatory motion that comes from the tensile testing machine or the sample in its tensile test fixture can inadvertently affect the resolution quality of the images. In addition, because the scan typically takes about 5 min, the specimen cannot be stretched and scanned at the same time: the specimen must be incrementally stretched and held with great stability between increments.

2. Design of the tensile testing machine

2.1. Specifications

The performance requirements for the machine were established by Prof. Boyce who is an active materials researcher interested in observing the microstructure and mechanical behavior of polymeric materials. The functional requirements for the machine were:

- *Maximum sample size*: 2 mm thick, 8 mm wide, 100 mm long.
- *Minimum sample size*: 10 μm thick, 1 mm wide, 20 mm long.
- *Maximum strain*: 500% (for a 25 mm long sample).
- *Maximum tensile force*: 4.4 kN.
- *Minimum strain resolution*: 0.2%.
- *Sample support*: optional for extremely thin samples.

2.2. Strategy and concept

Since the AFM is the dominant part of the system, it leads to the strategy of making the tensile test machine be able to nest with the AFM so it can pull the specimen under the AFM tip. The center of the sample, which is undergoing high strain, must therefore not translate so that there can be registration between AFM scans. This leads to the need for machine concepts that can pull each end of the sample at the same precise rate.

In order to minimize parasitic error motions of the sample, the machine must have not only accurate geometric motion, but minimal deflections under load. Given that the maximum tensile force requirement is 4.4 kN and an accuracy on the order of 1 μm is required to enable the sample to stay within the scan range of the AFM tip, the machine's structural loop stiffness needs to be on the order of 4400 N/μm. This is an exceptionally stiff machine with a modest range of motion, which therefore seems to preclude the use of flexures.

This leads to three design considerations:

- Use rolling element bearings for the carriages that hold each end of the specimen and their centers of stiffness should be as close as possible to the plane of the specimen.
- The tensile forces applied by actuators on the carriages should be in-line with the specimen.
- The position measurement of the carriages should be in-line with the specimen to minimize Abbe errors.

The first consideration conceptually is not difficult to imagine; however, the second poses a problem because it implies that two actuators are needed, one on either side of the sample which requires two coordinated servo systems with effective loop stiffnesses on the order of 5000 N/μm. This would be a very expensive design to accomplish. In addition, measuring the motion of the carriages exactly in-line with the sample would imply the use of a differential interferometer which would greatly increase the cost and complexity of the system.

An alternative is to use a combination of self-help and increased stiffness:

- Use a single ballscrew that is ground with a left- and right-hand thread, such that any deflection of the screw affects both ends of the specimen simultaneously and equally [14,15]. The ballscrew will thus need to be located below the plane of the sample, but in the vertical plane of the sample so as to minimize yaw-induced Abbe errors at the sensor.
- Position the bearings such that their plane of stiffness is as close to the plane of the specimen as possible, and size the bearings and the carriages such that the moment created by the ballscrew force being below the plane of the specimen only causes an acceptable deflection, on the order of 1 μm