



Using AFM Phase Lag Data to Identify Microconstituents with Varying Values of Elastic Modulus

D.N. Leonard*, A.D. Batchelor, P.E. Russell*,****

***Dept. of Material Science and Engineering, Box 7531, North Carolina State University, Raleigh, NC 27695-7531**

****Analytical Instrumentation Facility, Box 7531, North Carolina State University, Raleigh, NC 27695-7531**

Introduction

- The atomic force microscope (AFM) has allowed microscopists to observe surface topography of non-conductive samples on the atomic level. One AFM mode that scanned probe microscopists have recently shown an interest in is the phase lag imaging mode. It has already been demonstrated that the resulting phase data can be used to identify different densities of microlayered polyethylene samples.¹ However a quantitative understanding of phase lag imaging has yet to be fully developed. With a better understanding of the phase lag data it may be possible to estimate the modulus or other material properties of a sample from an AFM phase image alone

Introduction

- The need for better understanding phase image data is essential to increasing the knowledge of a how a material's microstructure behaves on the nanometer scale. This investigation focused on AFM phase images produced when scanning metallic alloys that posses microstructures with large differences in modulus values between the compositional phases. Being able to relate the phase lag data to a specific change in modulus can aid in the identification of hard and soft regions in a materials microstructure from a phase image. Although, the interpretation of phase lag data is complicated by several factors induced by sample topography and scan parameters

Introduction

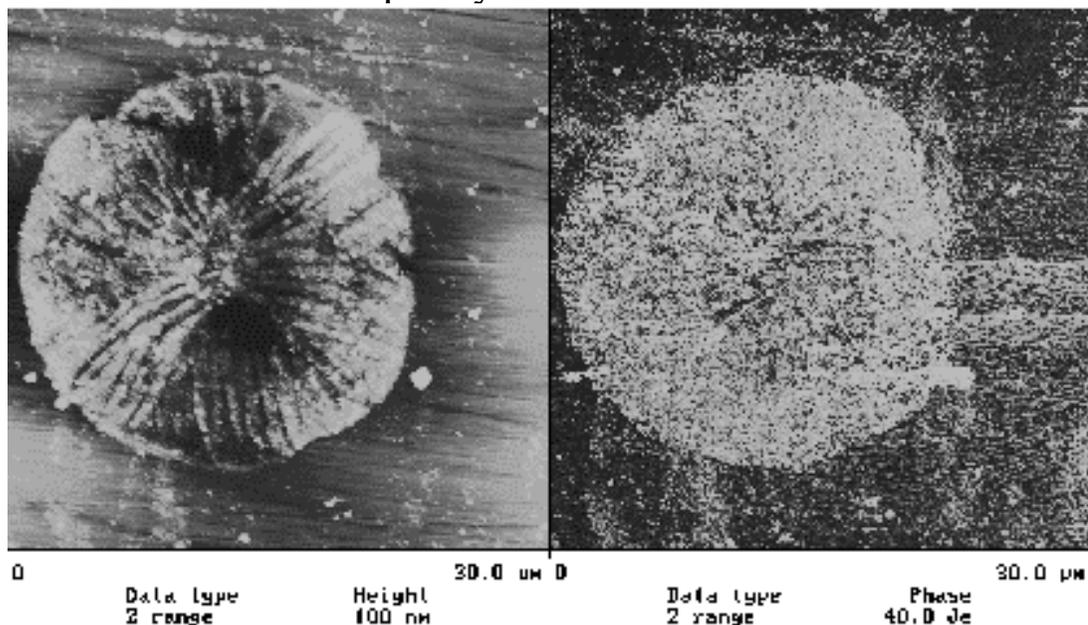
- Sample topography, and scanning parameters like drive frequency, degree of amplitude damping, and integral gain values all influence phase lag data and contribute to the difficulty of its interpretation. All variables affecting the phase lag image need to be controlled so that only material properties (i.e. elastic modulus) will produce contrast in the phase lag image. A 'point method' for data collection was developed for this experiment so that scan parameters could be controlled and as a result phase lag data interpretation simplified.

Procedure

- A Digital Instruments D3000 AFM with a Nanoscope III controller and extender module was used to record the phase lag data.
- The samples chosen for this experiment include 60Sn40Pb solder, an Al/Si alloy, high carbon steel and nodular cast iron.
- The samples are prepared using conventional metallographic techniques so surface topography is minimized (~2nm RMS for solder samples).
- To control scan parameters, the drive frequency chosen during cantilever tuning was kept constant throughout the data acquisition.
 - The drive amplitude of ~25nm was chosen.
 - The phase offset was then zeroed at the cantilever's drive frequency.
 - After cantilever tuning was completed a sample was scanned to locate the region being tested.
 - The tip was then centered over the compositional phase and the x and y scan directions were disabled.
 - The cantilever was then frequency swept through .00001 kHz from the offset it was driven at.
 - The phase offset was zeroed after the setpoint was decreased by 0.05V intervals, and the phase lag was recorded each time.
 - After the tip amplitude was completely damped (i.e. setpoint=0V) the setpoint was then increased by 0.05V and the phase lag recorded.

Results

- Figure 1 on the next page shows an resonance enhanced (AC) AFM scan captured during this experiment. The left (height) and right (phase lag) images were acquired simultaneously. The phase lag image on the right shows increased contrast when compared to the height image on the left. Although, it cannot be concluded that all of the contrast in the phase lag image is due to material properties. The topography in the height image (only 100nm z-range) results in the contrast enhancement of the phase lag image. It is necessary to reduce or eliminate all topography so that phase lag contrast is only contributed by resonator (cantilever) response relative to the resonator drive frequency.



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Figure 1.

Results

- In Figure 2 the results of the 'point mode' method on a nodular cast iron sample are graphed. The graph is able to illustrate large increases or decreases in phase shifts as the amplitude is damped. The decrease of setpoint from 2.42V to 2.32V produced a phase lag of $\sim 20^\circ$ in the softer iron matrix as compared to the phase lag of $\sim 10^\circ$ for the harder spherulitic graphite precipitate. this trend follows the theory of a larger phase lag for softer materials proposed by Magonov et al.1

