UTILIZING PERIOD-DOUBLING BIFURCATIONS TO LOCATE GRAZING IN ATOMIC FORCE MICROSCOPY

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Abstract

A nonlinear beam model is developed to study the cantilever probe of an atomic force microscope. With a multi-mode approximation, atomic interaction forces from molecular dynamics simulations are used to simulate the system's response. An excitation frequency of two and a half times the fundamental frequency is studied. Period-doubling occurs for this off-resonance excitation in support of previous findings from a larger scale-model. Through a better understanding of the qualitative change in the behavior, it is believed that the period-doubling can be utilized in the development of a near-grazing based operation mode of the atomic force microscope.

Key words

Atomic force microscopy, off-resonance excitation, period-doubling bifurcation

1 Introduction

The atomic force microscope (AFM) is a device that utilizes atomic interaction forces between the tip of the AFM probe and sample in order to "feel" the surface characteristics of the sample (Binnig *et al.*, 1986). This device is able to provide high resolution images of micro- and nano-scale materials with nanometer resolution. The operation method of interest to this work is the so-called amplitude modulation mode of atomic force microscopy. Tip-sample interaction forces from the surface of the sample affect the response of the AFM probe, which is generally monitored by reflecting a laser beam off the end of the probe and onto a photo-diode. These changes in the response amplitude are monitored and utilized within the control scheme to maintain a desired separation distance between the sample and the cantilever base (Martin *et al.*, 1987). The atomic force microscope uses this information to control the scanning of the sample and to discern surface characteristics. In the most common application of this operation mode, known as "tapping" mode AFM, the cantilever probe of the AFM is harmonically excited so that it oscillates above the surface of the sample with the probe tip coming into contact with the sample surface once per oscillation (Zhong *et al.*, 1993). Limited to only periodic contact, this method reduces the potential damage done to the sample, the amount of wear experienced by the probe tip, and the possibility of the probe tip adhering to the sample due to stiction.

In recent efforts to enhance the resolution of the atomic force microscope, work has been done to decrease the size of the tip radius of the AFM probe. This has been done by fabricating sharper silicon tips and by attaching carbon nano-tubes to the probe tips (e.g. (Choi *et al.*, 2000; Solares *et al.*, 2005)). While these actions do allow the atomic force microscope to obtain higher resolution images, by reducing the number of atoms interacting at the tip-sample interface, the effective stiffness of this interface is significantly decreased. For the same level of contact force applied to the surface of the sample by the probe tip, a smaller tip radius will result in a greater amount of penetration into the sample. This is a problem when analyzing delicate samples such as living biological specimens. When subjected to relatively large contact forces, the images of these samples can be significantly distorted and the samples may be damaged or destroyed. In order to benefit fully from the implementation of probes with smaller tip radii, it is necessary to improve the control of the atomic force microscope in order to enable operation with lower contact force levels. Some work has been done to address this issue in the theoretical development of the Frequency and Force Modulation (FFM) operation mode (Solares, 2007; Solares and Crone, 2007). Here, a nonlinear phenomenon for off-resonance operation of an atomic force microscope is investigated as the basis to develop a nonlinear dynamics based operation mode in order to reduce the contact force levels acting on the sample surface.

The nonlinear phenomenon that is of interest is the qualitative change in the dynamics response of the AFM probe that results from the transition from the inactive state, when the probe tip is not influenced by the sample, to the active state, when the probe tip is influenced by the surface forces of the sample once per oscillation. The qualitative change is characterized by a transition from period-one motion to period-two behavior. The presence and strength of the period-two component is determined by monitoring the magnitude of the half-frequency sub-harmonic of the excitation frequency. In previous work, similar methods have been employed to monitor harmonics of the excitation frequency in order to gain additional information about a sample being studied (Stark and Heckl, 2000; Sahin and Atalar, 2001; Sahin *et al.*, 2004; Crittenden *et al.*, 2005). In tapping mode AFM, the influence of the tip-sample interaction forces causes the periodic behavior of the probe to become anharmonic and higher harmonics exist within the response. By utilizing multi-degree of freedom representations, these studies found that the higher harmonics are sensitive to the characteristics of the tip-sample interaction force and can be used to obtain additional information about the samples.

At the macro-scale, this type of transition corresponds to a grazing bifurcation where the transit point is characterized by grazing. In previous work, a macro-scale model of the AFM cantilever probe, with a scale of approximately 1000:1, was utilized in a comprehensive study of the nonlinear behavior associated with the grazing bifurcation for off-resonance excitation (Dick *et al.*, n.d.). With regard to the atomic interaction forces, only the repulsive forces were represented within the macro-scale model. Since this is often the regime of the interaction force curve utilized for tapping-mode AFM, the results of this study are believed to be relevant to the micro-scale system. As the qualitative change in the system's response occurs when the cantilever probe begins to be influenced by the repulsive forces, the goal of this work is to utilize this phenomenon in the development of an operation mode that will maintain near-grazing behavior in order to reduce contact force levels during imaging. In this paper, the details of a numerical investigation into this nonlinear phenomenon by using a model of the micro-scale AFM cantilever probe are presented.

The remainder of this article is organized in the following way. The next section describes the development of the model. In the third section, the analysis of the nonlinear phenomenon is presented by using phase portraits, frequency spectra, and bifurcation diagrams. Contact force levels for the off-resonance condition are also compared with those of the standard excitation condition and the use of dual-frequency excitation with the off-resonance frequency is introduced. Concluding remarks and the direction of future work are addressed in the final section.

2 Modeling

The AFM probe modeled in this work is composed of single crystal silicon, has a geometry of $450\,\mu\text{m} \times 40\,\mu\text{m} \times 5\,\mu\text{m}$, and has a fundamental frequency of 28 kHz. In order to model the cantilever probe of the atomic force microscope, a nonlinear beam model is developed. Nonlinearities are included to ensure that the model is able to accurately represent the behavior of the system under the new excitation condition that is considered. Starting from the assumption that the beam is inextensional, the kinetic and potential energies of the beam are calculated to produce an augmented Lagrangian and the extended Hamilton's principle is used to derive the partial differential equation of motion for the transverse oscillations of the probe. The non-conservative work on the system is produced by the force from the harmonic base excitation and the atomic interaction force acting at the tip. Within this derivation, terms of orders greater than cubic are neglected. A linear damping term is also included in order to account for energy dissipation. The non-dimensional form of this equation is presented as equation (1). The "hat" symbol, $\hat{\bullet}$, will be used to identify dimensional parameters when necessary.

$$\begin{aligned} &w^{iv} + \left[w'\left(w'\,w''\right)'\right]' + c\,\dot{w} + \ddot{w} - \\ &\quad \frac{1}{2}\gamma\,w''\left(\int_0^1 \frac{d^2}{dt^2}\left(w'\right)^2 ds\right) + \\ &\frac{1}{2}\left\{w'\int_1^s\left[\int_0^s \frac{d^2}{dt^2}\left(w'\right)^2 ds\right] ds\right\}' = f_b. \end{aligned} \tag{1}$$

Within this equation, w is the non-dimensional transverse displacement of the beam. Derivatives with respect to the non-dimensionalized position along the beam s are indicated by the prime symbol and derivatives with respect to the non-dimensional time t are represented by over-dots. The variable c is the non-dimensional damping coefficient and γ is the ratio of the tip mass to the mass of the beam. The non-dimensional force f_b corresponds to the base excitation. For harmonic excitation, f_b is defined as

$$f_b = -X_0\,\omega^2\,\cos\left(\omega\,t\right), \tag{2}$$

where X_0 is the non-dimensional amplitude of excitation and ω is the non-dimensional excitation frequency. The boundary conditions are also obtained from the extended Hamilton's principle. The boundary conditions of the AFM probe are equivalent to those of a uniform cantilever beam with the exception that the shear force at the free end of the beam must be equal to the sum of the inertial force of the tip-mass and the force resulting from atomic interactions between the probe tip and the surface of the sample. When the mass of the probe tip is much smaller than the mass of the probe, the value of γ will become very small and the inertial term can be neglected.

$$\begin{array}{ll} w|_{s=0} = 0, & w'|_{s=0} = 0, \\ w''|_{s=1} = 0, & w'''|_{s=1} = \gamma \ddot{w}|_{s=1} + f_{ts}. \end{array} \quad (3)$$

Within the equations for the boundary conditions, given as equation 3, f_{ts} represents the non-dimensional tip-sample interaction force acting on the probe tip. This force f_{ts} is defined as equation (4) when the transverse deflection of the probe at the tip, $w\left(1,t\right)$, is less than or equal to $(\delta - \sqrt{W/S} - X\left(t\right))$. If the value of $w\left(1,t\right)$ is greater than $(\delta - \sqrt{W/S} - X\left(t\right))$, the non-dimensional atomic interaction force is defined as equation (5). In these equations, the term $X\left(t\right)$ defines to the motion resulting from the base excitation. The square root term is included so that the value of the non-dimensional separation distance δ corresponds to the point on the interaction force curve where the force is equal to zero at the transition from the attractive regime to the repulsive regime.

$$f_{ts} = \frac{-W}{1 + A\left(w\left(1,t\right) - \delta + \sqrt{\frac{W}{S}} + X\left(t\right)\right)^2}, \quad (4)$$

$$f_{ts} = S\left(w\left(1,t\right) - \delta + \sqrt{\frac{W}{S}} + X\left(t\right)\right)^2 - W. (5)$$

With these equations, the non-dimensional parameters W, A, and S provide a model of the interaction forces between the atoms of the probe tip and those on the surface of the sample. The values of these parameters are calculated with a curve-fitting process which is applied to the results of molecular dynamics simulations for a specific type and size of probe tip and a specific type of sample material. Within the molecular dynamics simulations, the Dreiding force field is employed to describe atomic interactions (Mayo *et al.*, 1990).

In order to perform numerical simulations, it is necessary to discretize the system model. This is done by utilizing separation of variables to define the transverse deflection as the sum of a finite number of mode shapes $\phi_n\left(s\right)$ multiplied by their corresponding modal amplitude functions $q_n\left(t\right)$. By using mode shapes from the linearized system, the Galerkin method is applied to transform the partial differential equation of motion into a set of coupled ordinary differential equations.

Values are then assigned to the parameters so that simulations can be performed. Table 1 contains the parameters used within the simulations. Many of these values correspond to the device geometry and material properties but others must be determined though experimental

Table 1. Dimensions and properties used to simulate behavior of atomic force microscope cantilever probe.

Parameter	Value
Probe Length, $\hat{L}$	450×10^{-6} m
Probe Width, $\hat{b}$	40×10^{-6} m
Probe Thickness, $\hat{t}$	5×10^{-6} m
Density, $\hat{\rho}$	$2330\, kg/m^3$
Elastic Modulus, $\hat{E}$	$123 \times 10^9\, N/m^2$
Tip Mass, $\hat{m}_{tip}$	2.1×10^{-12} kg
Damping Factor, ζ	1.45×10^{-3}
Force Curve Parameter, $\hat{W}$	0.4×10^{-9} N
Force Curve Parameter, $\hat{A}$	$10 \times 10^{18}\, 1/m^2$
Force Curve Parameter, $\hat{S}$	$8.0 \times 10^9\, N/m^2$

means. In order to quantify the damping within the system, experimental data collected from an AFM system is used. The response data was collected in the form of the output voltage from the photo-diode and a linear approximation of the damping is employed. From the free-vibration data, a damping ratio of 1.45×10^{-3} is calculated.

The other parameters necessary to perform simulations of this system are those that define the tip-sample interaction force curve. In the previous work with a macro-scale test apparatus, a "soft" foam rubber material was selected in an effort to represent the low stiffness of a delicate sample that might be imaged with atomic force microscopy (Dick *et al.*, n.d.). The parameter values used within this study are obtained by fitting the force curve model to force-displacement results from molecular dynamics simulations of the interaction between the tip of a triple-walled carbon nanotube and a single bacteriorhodopsin molecule on a silicon substrate. This provides a very good case of low stiffness for a delicate sample. The values of the dimensional parameters are: $\hat{W} = 0.4$ nN, $\hat{A} = 10$ nm^{-2}, and $\hat{S} = 8$ nN/nm^2.

3 Analysis

By using the discretized model developed in the previous section, a series of numerical experiments are conducted in order to study the simulated response of the cantilever probe. In this initial work with the model of the micro-scale AFM probe system, conditions are selected to verify the type of response observed of the macro-scale test apparatus. Two excitation frequency values are of interest: near the fundamental frequency of the probe and near two and a half times the fundamental frequency of the probe. The first excitation frequency is selected to correspond to the standard op-

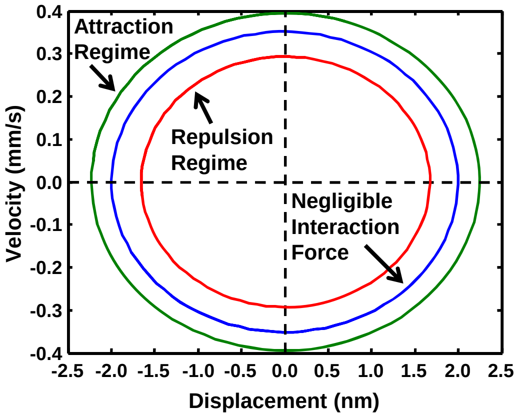

Figure 1. Simulated phase portraits of dynamic responses to excitation near the fundamental frequency for three different separation distances.

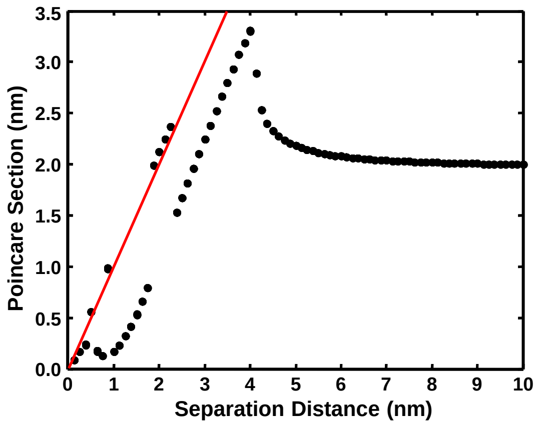

Figure 2. Simulated bifurcation diagram of the dynamic responses to excitation near the fundamental frequency. The separation distance is the control parameter.

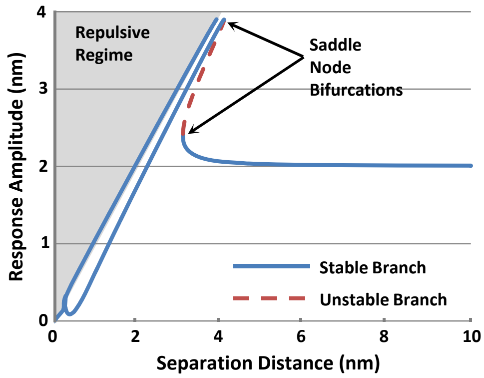

Figure 3. Bifurcation diagram produced using AUTO bifurcation and continuation software for excitation near the fundamental frequency. The separation distance is the control parameter.

eration of tapping-mode AFM. The second frequency is selected for the period-doubling phenomenon that has previously been identified when studying harmonically impacted elastic structures (Balachandran, 2003). Similar period-doubling behavior has also been identified at higher frequencies but this off-resonance frequency is selected in order to minimize the velocity of the probe. Experiments and simulations of the macro-scale model displayed period-two behavior for the second excitation condition for active conditions. Since the second excitation frequency is between the fundamental frequency and the second characteristic frequency, a multi-mode approximation is used. A three mode approximation was necessary to accurately simulate the response for the macro-scale system with the third mode providing additional detail to the response. This analysis investigates how the nonlinear response is affected by scaling the system down from the macro-scale and how the addition of the attractive regime in the interaction force affects the nonlinear response.

3.1 Standard Excitation

When the excitation frequency is near the fundamental frequency, the change in the response of the cantilever probe is limited to an increase and then decrease in the oscillation amplitude as the separation distance is decreased. For the AFM system, the relative oscillation amplitude is significantly smaller than that of the macro-scale system and as a result, the response is linear and consists of the first vibration mode with negligible contributions from the higher modes. The change in the amplitude is visible in the phase portraits presented in figure 1 for three different separation distances.

Aside from the change in the amplitude of oscillation, there is little change in the form of the response. For this reason, excitation near the fundamental frequency and the use of a Hookian approximation are standards for AFM. In order to further study how the response amplitude changes for different separation distances, Poincaré sections are calculated for a range of separation distances and combined to form a bifurcation diagram, shown in figure 2. Each simulation, and corresponding Poincaré section, is obtained by initializing the system with the same initial conditions and a separation distance of 10 nm, so that the influence of the surface forces are initially negligible. For cases of bistability, this process allows for the response to converge to either of the multiple stable solutions.

The system is excited so that the free amplitude, that is the amplitude of oscillation when the response is unaffected by the surfaces forces, is 2 nm. This is done to match the oscillation amplitude of the system when it is examined for the off-resonance condition. In addition to confirming the general trend in the amplitude due to the change in the separation distance, the bifurcation diagram also reveals the bistability of the system as a result of the nonlinear tip-sample interaction force curve. The diagonal line in figure 2 shows where the separation distance is equal to the Poincaré section amplitude and is considered to be the “surface” of the sample.

In order to confirm the bistability behavior produced by the simulation, AUTO bifurcation and continuation software is employed (Doedel, 1981). The resulting bifurcation diagram is shown in figure 3. While the general form of this bifurcation diagram agrees with the results in figure 2, there are some small differences which are attributed to the fact that the AUTO analysis is conducted with a linear, single-mode approximation of the AFM probe. The results of the AUTO analysis reveal two saddle node bifurcations on either side of an unstable branch and the presence of two stable branches that coexist over a range of separation distance values. At a small separation distances, 0.2 nm, a branch point is identified where the two stable branches split off from a single stable branch that exists for smaller separation distances. The shaded area of the plot indicates the influence of the repulsive regime of the tip-sample interaction forces on the cantilever probe. The upper of the two stable branches lies just inside the shaded area.

During normal operation, the free amplitude of the AFM probe is selected to be from 20 to 100 nm, depending on the material being studied and the properties of the probe that is being used. The amplitude set point, which is the amplitude value that the control scheme will work to maintain, is then selected to be up to 10 nm less than the free amplitude (Zhong *et al.*, 1993). This results in a response that is away from the unstable branch but within the range where the two stable branches coexist.

3.2 Off-Resonance Excitation

The second excitation frequency to be investigated has a value of two and a half times the fundamental frequency. It is determined that for this excitation condition, the oscillation amplitudes experienced by the AFM probe cause the nonlinear terms in equation 1 to produces values significantly smaller than those of the linear terms. However, the use of the multi-mode approximation is determined to be very important in order to accurately predict the behavior of the system. At this frequency, a different type of response is observed when the AFM probe approaches the sample. Initially, the amplitude of oscillation shows very little change before the attractive regime is strong enough to draw the probe toward the sample where the influence of the repulsive regime produces a qualitative change in the response. Phase portraits of the response for inactive and active conditions are shown in figure 4(a) and figure 4(b) for the macro-scale test apparatus and the micro-scale AFM cantilever probe, respectively. The change in the response is observed to occur very abruptly for both of these systems and produces period-two behavior. Additionally, comparison of the macro-scale system and the micro-scale system reveals strong agreement with regard to the relative increase in the response amplitude under active conditions.

In order to verify the period of the response, frequency spectrum information is examined for the responses in figure 4(b) for both inactive and active conditions. The frequency spectrum information, shown in figure 5, includes the expected significant frequency component at the frequency of excitation. However, for the active condition, the presence of a second significant frequency component, located at a frequency equal to half of the excitation frequency, confirms the period-two

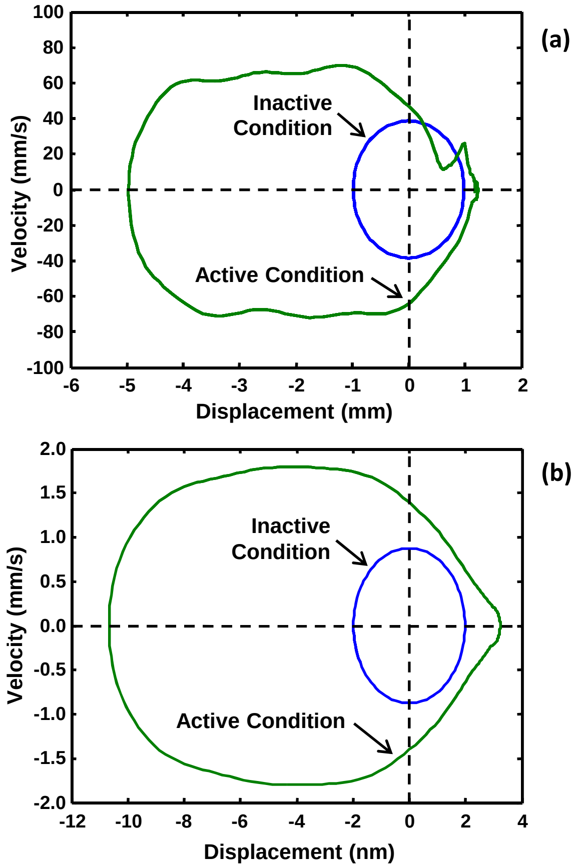

Figure 4. Simulated phase portraits of dynamic responses to excitation near two and a half times the fundamental frequency for (a) a macro-scale test apparatus and (b) a micro-scale AFM cantilever probe.

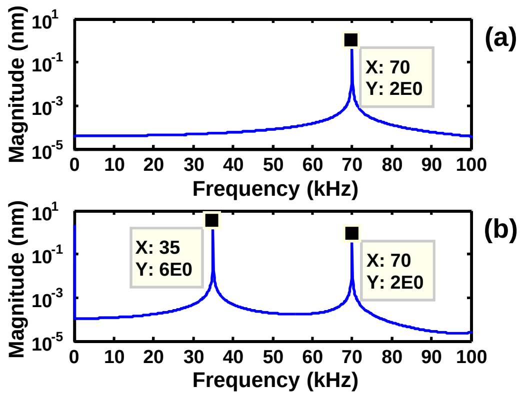

Figure 5. Frequency spectrum corresponding to dynamic responses of simulated AFM probe excited near two and a half times the fundamental frequency for (a) inactive conditions and (b) active conditions.

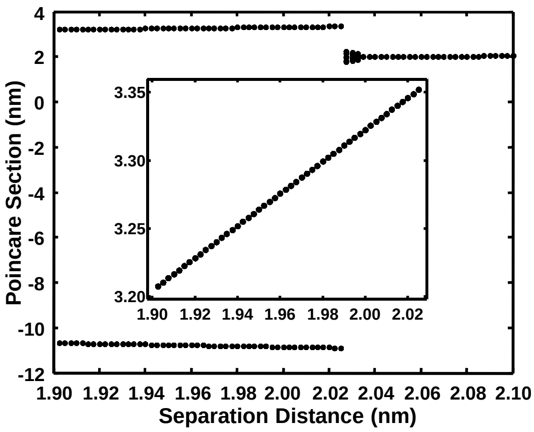

Figure 6. Simulated bifurcation diagram of the dynamic response to excitation near two and a half times the fundamental frequency. The insert contains an enlarged plot of the upper branch for the smaller separation distance values. The separation distance is the control parameter.

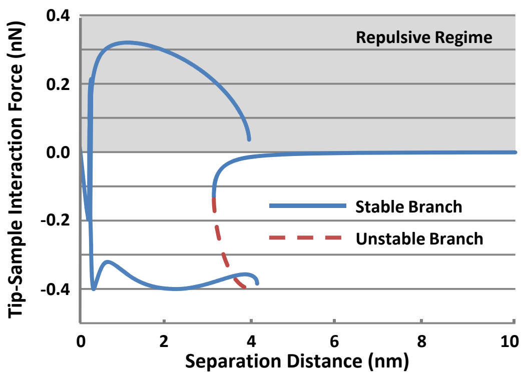

Figure 7. Maximum tip-sample interact force values calculated from the results of the AUTO analysis that were used to produce the bifurcation diagram.

behavior. A bifurcation diagram is also prepared for this excitation frequency in order to verify the period-two behavior and to provide insight into the relationship between the response and the separation distance. This bifurcation diagram is prepared in the same manner as was done for the previous excitation condition and is shown in figure 6. For these excitation conditions, this bifurcation diagram suggests the presence of a non-smooth bifurcation. This is believed to be a result of selecting the separation distance as the control parameter since changes in the separation distance affect the structure of the system. For the data presented, a separation distance increment of 0.025 nm is used in order to approximate the behavior trends corresponding to the vertical resolution of the atomic force microscope. Unlike the first excitation frequency, the nonlinear tip-sample interaction force curve does not appear to produce bistability for an excitation frequency of two and a half times the fundamental frequency. The insert included in figure 6 contains an enlarged view of the upper portion of the bifurcation diagram for active conditions and the Poincaré sections suggest the presence of a single, stable branch.

3.3 Contact Force

As the goal of this work is to reduce the level contact force applied to a sample being studied, the maximum tip-sample interaction force values corresponding to the two excitation frequencies are examined and compared.

The interaction force values corresponding to the first excitation condition are calculated from the results of the AUTO analysis and presented in figure 7. Within this plot, there is a maximum force value of 0.32 nN. The transition from the large separation distance values where the interaction forces are negligible, through the unstable branch, to the stable branch that lies in the attractive regime is shown within this figure. The positive, attractive force values of the second stable branch are also shown to exist within the same range of separation distance values.

In comparing the force values for the off-resonance excitation in figure 8 with those of the standard excitation method, they are significantly larger. The largest force value from the analysis of the off-resonance condition has a value of 18.9 nN. Despite the easily identifiable qualitative change that signals the onset of repulsive interaction forces, the off-resonance excitation condition is characterized by a significant increase in the contact force levels applied to a sample. Additionally, due to the rather significant change in the response of the probe, a much greater amount of time is necessary for the transient oscillations to decay when transitioning between inactive and active response states. For these reasons as well as the anticipated complexity of a control scheme to handle such different behavior, this excitation condition does not provide ideal conditions for the development of a new operation mode.

3.4 Dual-Frequency Excitation

Due to the various complications that arise when utilizing the discussed off-resonance excitation condition, another implementation of this nonlinear phenomenon is investigated and preliminary findings are positive. This method takes advantage of the predictability of the AFM probe's response for excitation near the fundamental frequency and the nonlinear phenomenon associated with the off-resonance condition through the use of dual-frequency excitation. In recent efforts, others have investigated the benefits of utilizing multi-frequency excitation to enhance measurement capabilities in various forms of scanning probe microscopy. By exciting multiple vibration modes, additional information is obtained from the response of the higher frequency mode in Kelvin probe force microscopy (Kikukawa *et al.*, 1996), electrostatic force microscopy (Stark *et al.*, 2007), and

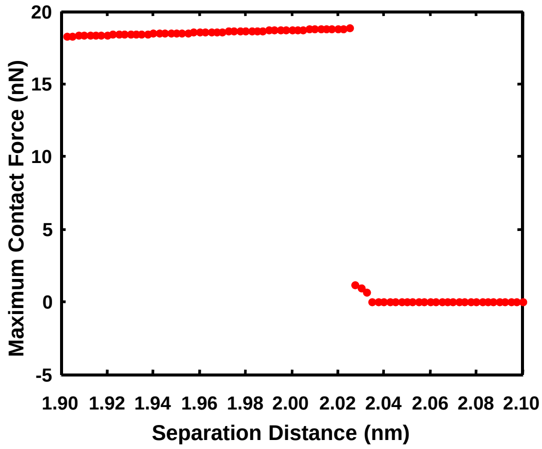

Figure 8. Maximum tip-sample interact force values for the separation distance ranges and response conditions presented in the bifurcation diagrams for (a) excitation at the fundamental frequency and (b) excitation at two and a half times the fundamental frequency.

atomic force microscopy (Proksch, 2006; Rodriguez *et al.*, 2007). While these methods benefit from having only a weak coupling between the responses of the orthogonal modes due to the influence of the sample being studied, the use of the off-resonance excitation condition relies on interaction between the two excitation frequencies. Through this interaction, the period-doubling phenomenon of the off-resonance condition is transferred to the response near the fundamental frequency. This phenomenon is visible in the spectral plots in figure 9. The response of the dual-excited system for inactive conditions is presented in figure 9(a) and the response of the AFM probe to active conditions is displayed in figure 9(b). Among the changes observed in these spectral representations of the response is a new component with a frequency equal to half of the primary excitation frequency suggesting that period-doubling has occurred. In order to minimize the negative effects associated with off-resonance excitation, the magnitude of the free response to the secondary excitation is less than one percent of the response to the primary excitation. In agreement with other recent studies (Thota *et al.*, 2007), preliminary results suggest that the addition of the off-resonance excitation signal also serves to successfully disrupt the bistability of the response. By using this dual-frequency excitation method, efforts will continue in the development of a near-grazing based nonlinear operation mode for atomic force microscopy.

4 Concluding Remarks

From this study, is has been verified that the period-doubling that was observed in the macro-scale system for the off-resonance excitation at two and a half times the fundamental frequency is relevant to the dynamic behavior of the micro-scale cantilever probe of an atomic force microscope. Also, the manner in which the qualitative change in the response occurs suggests that the material selected to represent the sample in the macro-scale experiment provided a good representation of a "soft" sample such as a single bacteriorhodopsin molecule. However, it is determined that exciting the AFM probe with the off-resonance frequency results in significantly higher contact force levels. In order to address this issue, the use of dual-frequency excitation is explored and it is determined that it may allow for use of the nonlinear phenomenon under more favorable conditions. Through additional numerical studies with this model and complementary experimental studies, it is believed that the period-doubling can be used to enhance the performance of the atomic force microscope in the development of a near-grazing based operation mode.

Future work will focus on investigating the system's response to dual frequency excitation with a weak secondary component at the off-resonance frequency. Additionally, the characteristics of the response transition will also be studied to determine their relationship with the non-dimensional sample stiffness.

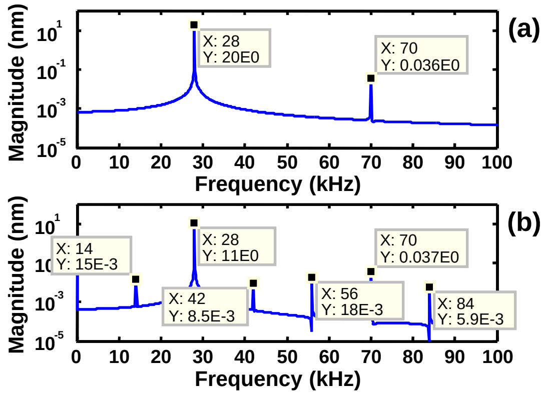

Figure 9. Spectral plots of simulated AFM response to dual frequency excitation for (a) inactive conditions and (b) active conditions.

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