ABSTRACT

Experimental techniques have been developed to measure the compressive and shear stiffnesses and bonding strengths of nanostructured thin films consisting of arrays of Cu and Ag helical springs. Rectangular areas of films were loaded in uniform compression and shear and the film properties were obtained through the load versus displacement response. The compressive stiffness of the nanostructured Cu films was measured to be $258 \pm 12$ MPa while the shear stiffness was $15 \pm 3$ MPa for Ag films on Si substrates and $19 \pm 2$ MPa and $21 \pm 4.5$ MPa for Cu films on Si and W, respectively. The experimental results were compared to theoretical predictions by modeling the nanostructured film elements as perfectly helical springs. The measurements were found to be typically within 20% of the theoretically-predicted values, and spring equations consistently predicted the change in stiffness between films of different materials. Bonding strength measurements showed an interfacial strength dependence on the bonded area, which is consistent with literature reports of other interfacially-bonded systems. The design of nanostructured thin films was conceptually optimized by considering both qualitative observations and by applying constitutive spring equations. The thickness of the cap layer and the morphology of the nanostructured layer were found to significantly affect the mode of debonding of the films. Optimum geometric and material parameters were also determined to maximize the amount of energy stored in the films in the elastic regime. A spring index, which is the ratio of the mean coil diameter to the wire diameter, of 2.5-3 was found to maximize the amount of elastic spring energy stored in a nanostructured film under uniform compressive loading and an index of 2.3-2.5 maximizes the stored energy under combined compressive and shear (mixed) loading.
ACKNOWLEDGMENTS

While often credited to a single inventor or researcher, all of the great scientific advancements in the history of mankind have relied on the exchange and sharing of ideas and resources between people of varied backgrounds, knowledge, and expertise. Thinkers, workers, leaders, and learners come together to plant the seed of science and nurture it into a blooming idea. In that regard I would like to thank those who have lent me their hand in leadership and assistance in making the work of this thesis possible. I would like to primarily thank my advisor Dr. Ioannis Chasiotis, who served as the principal investigator of this research. His leadership and technical knowledge have been crucial to the success and propagation of this project.

I would also like to thank all of my past and present colleagues at the Nanomechanics & Materials Research Lab as my daily interactions with them promoted a positive work environment that fostered my success. Mr. Karanjgaokar and Dr. Oh were instrumental in developing the experimental tools required for my research, while Mr. Ozkan and Mr. Yagnanurthi consistently provided invaluable insights into problems and challenges faced throughout my experimental work. Dr. Naraghi, Dr. Chen, and Mr. Tusz provided insightful and wise advice at the onset of my graduate studies, whereas Mr. Arshad and Mr. Kolluru promoted a lighthearted and cooperative work environment.

I would like to acknowledge the support by General Electric Global Research under subcontract #400019573 with Dr. David M. Shaddock as the principal investigator of the Nanothermal Interfaces Program (NTI). This work was sponsored by DARPA under Contract N66001-09-C-2014 (approved for public release, distribution unlimited). I would also like to thank Dr. Marshall for providing the necessary training and assistance for my work with the FEI DB235 FIB, which allowed SEM imaging and FIB milling work to be carried out in the Frederick Seitz Materials Research Laboratory Central Facilities at UIUC, which are partially supported by the U.S. DoE under grants DE-FG02-07ER46453 and DE-FG02-07ER46471. Numerous thanks are also due to the Aerospace Department’s machine shop, which transformed paper drawings into quality parts in a timely fashion. I would finally like to thank my family, friends, and any individual I have met in my life that has helped shape the person I have become.
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<td>K</td>
<td>Stress correction factor</td>
</tr>
<tr>
<td>$K_{elementary}$</td>
<td>Wahl stress correction factor based on elementary theory</td>
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<tr>
<td>$K_{exact}$</td>
<td>Stress correction factor based on exact elastic solution</td>
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<td>m</td>
<td>Number of springs in a given area</td>
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<td>Axial force</td>
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<td>Yield strength</td>
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CHAPTER 1

INTRODUCTION

In the field of materials fabrication and development, it is often desired to control and tailor the constitutive thermal, mechanical and electrical behavior of a material. In metals, for example, the grain size, orientation and boundaries and the presence of point and line defects contribute significantly to the bulk-scale material response affecting engineering properties, such as hardness and toughness. Similarly, in composite materials the integration of soft and hard phases (e.g. polymers and carbon or glass fibers) merges the advantages of materials with orthogonal but advantageous properties and mitigates their weaknesses. However, the combination of dissimilar monolithic materials to achieve designer thermomechanical and thermal properties is not a straightforward task: metals are good thermal conductors but are also of high stiffness, thus resulting in prohibitive thermal stresses and interfacial failures. Although metals processing has achieved great strides to date, all efforts have been focused in controlling the material inelastic behavior, with their thermoelastic response remaining virtually unchanged. Porous metal films respond well to thermal mismatch loads but their thermal transport properties are abysmal [1]. This incompatibility of mechanical and thermal behavior has led to significant difficulties in several applications including microelectronics and Microelectromechanical Systems (MEMS) [2]. With these considerations in mind, it is evident that compliant interfaces between metals or metals and semi-conductors can be possible by capitalizing on the structural rather than the intrinsic material response of metals. A versatile method for the deposition of thin films that potentially addresses this requirement was developed in the mid 1990s: in this method, nanostructured, or nanosculptured thin films are deposited with control to the order of 10 nm and possess mechanical stiffness that is at least two orders of magnitude smaller than solid thin films of the same metals [3,4]. The fabrication parameters can be varied in such a way to adjust the film stiffness for a given thickness. With thicknesses on the order of hundreds to
thousands of nanometers, these films show promise as compliant interfaces to bridge thermal strain mismatches across an interface [4]. It is therefore of interest to study the mechanical behavior of these films in connection with the geometric parameters and the mechanical behavior of the individual nanostructures comprising them.

1.1. NANOSTRUCTURED THIN FILMS AND THEIR PROPERTIES

The method of glancing angle deposition (GLAD) produces nanostructured thin films as a collection of densely-packed three-dimensional structures with similar nanoscale geometric features. The latter are hundreds of nanometers to several microns in height and width while their smallest feature size (usually the wire diameter) is of the order of tens to hundreds of nanometers. The individual nanostructures are randomly dispersed or regularly spaced on a seeded substrate [5], and their geometry may be columnar, helical, zigzagged, or a combination of the three [3]. The GLAD process is scalable and compatible with many materials [6]. A final capping layer may be deposited on top to protect the underlying nanostructured layer [3]. An example of a helical Cu structure deposited onto a Si substrate with a Cu capping layer is shown in Figure 1.1.

![Nanostructured Cu thin film with helical spring structures and a Cu capping layer.](image)
1.1.a. Fabrication of Nanostructured Thin Films by GLAD

GLAD thin films are produced by physical vapor deposition onto a flat substrate positioned at an oblique angle while rotating to achieve the desired geometry of the nanostructures [5]. The GLAD process can be controlled to produce microstructures with features tailored to scales of less than 10 nm [3]. Even without rotation, deposition at an oblique angle results in distinct columnar features that form due to a self-shadowing effect that occurs when the incident material flux is not coincident with the substrate normal vector [5]. As the material deposits on the substrate, regions of deposited material shadow the incoming flux and promote the growth of columnar structures angled in the direction of the incoming flux. If the substrate is rotated, the incoming flux continuously changes direction and the direction of columnar growth varies as a result. The rotation may be at a constant angular frequency, in which case the structures assume the shape of helical springs with the precise geometry being determined by the deposition parameters, including the incident flux and the rotational speed of the substrate. The rotation may also occur at discrete time and angle intervals, thus producing zigzag structures [5]. In order to form the final capping layer, the incident angle of the flux is gradually decreased until the flux is normal to the substrate. Thereby a cap is formed with minimal residual stresses that could otherwise lead to fracture within the cap layer [3]. This gradual capping deposition is also required to minimize the amount of flux that penetrates into the nanostructured layer.

1.1.b. Nanosprings Fabricated by GLAD

The nanostructured thin films studied in this research most closely resembled arrays of helical springs and were modeled as such throughout this dissertation. The independent geometric parameters used to describe a helical spring are shown in Figure 1.2 and are used in the analysis of helical springs in Chapter 3.

The mean coil diameter, \( D \), in Figure 1.2 is the average of the outer and the inner diameters of the spring. In the GLAD process, the mean coil diameter is inversely related to the substrate rotation speed. Lower substrate rotation speeds lead to larger helical diameters, while higher rotation speeds lead to slender structures that are more columnar.
than helical [5]. The helix angle is primarily a function of the angle of the incident flux since spring growth occurs in the direction of the incoming flux; oblique deposition will result in small helix angles. When considering columnar growth without substrate rotation, the angle between the resulting columns and the substrate normal is smaller than the angle between the incident flux and the substrate normal. [5]. An incident flux of 80° to the normal, for example, will produce columns that grow at possible angles of 50-65° from the normal [5]. Spring lengths can be as small as 200 nm per coil turn [4] or as large as 2,600 nm per coil turn [7], while multiple turns are typically deposited in a single deposition. All of the above parameters describe perfectly helical springs, for which elastic solutions have been developed to predict their stresses and deflections. However, most nanosprings fabricated by the GLAD process deviate significantly from this perfectly helical spring geometry. As shown in Figure 1.3, while the general shape of the coil is similar to a helical spring, the swept cross-section is not perfectly circular and variations in the deposited material lead to non-uniformities across the coils. These geometric irregularities, coupled with the close spacing of the deposited nanosprings, make the application of theoretical spring equations less accurate. Therefore, experimental measurements are imperative to obtain the elastic response of the nanospring films.

Figure 1.2 – Geometric parameters and nomenclature for the helical springs used in this research.
While mechanical experiments with individual nanosprings are appealing to compare the ideal with the true nanospring response, the variation in the geometry of adjacent springs and their interpenetrating arrangement in a nanostructured film make such local measurements less useful compared to thin film level measurements where the collective response of a large number of springs is recorded.

![Image of nanowires](image.png)

Figure 1.3 – Comparison of the actual spring geometry (left) and perfectly helical geometry (right).

### 1.1.c. Applications of GLAD Materials

Several applications of nanostructured thin films have been reported in literature since their initial development in the 1990s. Uncapped nanostructured thin films with stacked layers of SiO$_2$ and TiO$_2$ have been fabricated as non-reflective film coatings [8]. These 600 nm tall nanostructured films had tailored refractive indices to create a quintic variation of the refractive index between the ambient air and the substrate onto which the films were deposited. Since the fraction of reflected light increases with an increasing mismatch of the refractive indices at an interface, the thin film acts as a gradual transition of the refractive index to minimize the amount of total reflected light, as shown in Figure 1.4. Other studies of the optical properties of nanostructured thin films have included
These films showed optical anisotropy and were studied to understand their spectra at various wavelengths. The results suggested numerous applications, including retardation plates, polarizers [9], and photovoltaic devices [10]. It was also found that the deposition angle has a strong effect on the optical properties of the films.

![Variation of refractive index across a geometrically-graded nanostructured thin film][8]

In a sensor application, uncapped nanostructured TiO$_2$ films produced by GLAD were used as relative humidity sensors by monitoring the change in capacitance of the film [12,13]. Two Au electrodes deposited between a SiO$_2$ substrate and the thin film served as connections across which the capacitance was measured. The sensors were capable of measuring the relative humidity over a range of 2 - 95%, in which the capacitance varied over three orders of magnitude. The same study also analyzed the effect of film thickness and deposition angle on the response time of the sensor, which varied from 64 – 1440 ms. Small film thicknesses of less than 1 μm and large deposition...
angles led to the fastest response times, although the coupling effect of film thickness and deposition angle was not investigated.

In a different application, the spring-like properties of nanostructured thin films have been taken advantage of to form compliant interfaces that eliminate stress singularities at interfaces [4]. In this reference, finite element analysis was performed to quantify the stress distribution at the interface between a rigid substrate and an elastic half-plane with and without a crack. The introduction of compliant nanostructured helical springs to the interface eliminated the stress singularity that is typical at a crack tip or sharp interface.

The aforementioned applications of nanostructured films rely on the geometry of the nanostructured unit elements to tailor the film properties. Among them, their application as compliant interfaces demands quantitative understanding of the mechanical response of the nanostructured films so that predictive design models can be developed. Experimental measurements of the compressive and shear stiffness and strength of these films are necessary because the elastic spring equations are only useful as a first order approximation. Furthermore, in the case of anti-reflective films, the geometry of the spring layer is directly related to the refractive index of the material, while in relative humidity sensors the porous nanostructure is crucial to the humidity measurement and is easily affected by mechanical deformations. The film porosity is also a crucial parameter in thin layer chromatography applications, where varying the nanostructure and film thickness determines the performance of the technique [14]. In all cases, mechanical stresses result in film deformations that inevitably affect performance.

1.2. MOTIVATION AND BACKGROUND RESEARCH

Early work on the mechanical characterization of nanostructured films involved the technique of nanoindentation to measure the film compressive axial stiffness [7]. Seto et al used rounded spherical indenters of 1 and 120 μm tip radii to compress 3 μm thick samples of SiO-capped nanostructured SiO thin films. Stiffness measurements were performed on films of varying pitch and spring diameter and comparison to bulk SiO
films showed the nanostructured film to be significantly more compliant than the bulk material. The results were used to calculate the resonance frequency of the springs, which was on the order of tens of MHz. While the indentations revealed elastic behavior of the material up to the point where the cap layer was damaged, this method could serve only as an approximate measure of the stiffness since the calculations assumed the indenter to be of a flat circular shape instead of hemispherical. The experiments also loaded a continuously capped material and it was acknowledged by the authors that lateral redistribution of load may involve large measurement uncertainties.

Further work on the mechanical characterization of nanostructured films attempted to improve on the nanoindentation method by loading defined rectangular areas of thin films instead of continuous films. One experiment focused on the lateral and axial stiffness of 1.9-13.2 μm² areas of three-turn Tantalum(v) Oxide (Ta₂O₅) springs of 560 nm height that were deposited onto a Ta₂O₅ substrate and capped with a 200 nm thick Ta₂O₅ layer [4,21]. By means of hemispherical or pyramidal diamond tips, Sumigawa et al measured the effective axial and shear stiffnesses of the spring layer to be 60 and 375 MPa, respectively. As shown in Figure 1.5, the hemispherical tip was used to load the sample under axial compression and the diamond tip was used for lateral shear. A Hysitron Triboscope provided the load/displacement response for 100 nm displacements and 100 μN forces. While this method was the first to measure the lateral stiffness of the film, it still suffered from multiple drawbacks. As with the nanoindentation method, this method was unable to impose a uniform displacement boundary condition along the entire surface of the sample area. In the case of axial compression, the hemispherical diamond tip has a finite radius of curvature that inherently introduces a non-uniform loading profile. In the case of lateral loading, the pyramidal diamond tip only loads one edge of the sample and it is not immediately evident to what extent the concentrated line load applied to the edge of the sample causes deformation of the sample far from the load. On the other hand, this method did improve on previous attempts by loading well defined areas of material, thus removing the effects of the lateral redistribution of load through the cap layer.
Another work investigated the issue of non-uniform loading by attempting to measure the spring constant of individual helical structures within an uncapped film [15]. Liu *et al* used a single crystal Si conical AFM tip to target individual helical springs within a patterned template. The individual springs were compressed and the load/displacement response was recorded by an AFM. The 4 μm tall Si springs were measured to have a spring constant of 3-10 N/m, depending on the spring geometry. As expected, springs with a 225 nm wire thickness were measured to be less stiff than springs with a 350 nm wire thickness. The force response of the spring was linear up to the maximum applied displacement of 40-80 nm. This method was unable to measure the transverse stiffness of the springs since it was not possible to load the spring in the transverse direction given the presence of neighboring springs. On the other hand,
translating the single nanospring data into film properties is not a simple task because of the high degree of interaction between adjacent springs.

As an improvement upon previous studies, this research aimed at loading rectangular sections of nanostructured thin films in uniform compression and shear to extract the effective elastic properties of the anisotropic films. The techniques applied in this research are independent of the geometry of the spring layer and provide an accurate measurement of the axial and lateral stiffness of the entire nanostructured film.

1.3. OBJECTIVES AND PROCEDURES OF DISSERTATION RESEARCH

The objective of this research was to quantify the axial and shear stiffness of nanostructured thin films and to compare the results with theoretical calculations predicted by elastic helical spring theory. The experimentally-determined values of stiffness could then be used in finite element models such as the one analyzed by Sumiwaga et al [4] to study how the film behaves when incorporated as an interface into a larger system. The methods applied in this research are an improvement upon previously reported methods due to the ability to uniformly load a clearly-defined section of the material and obtain measurements of the deformation of the interface without the influence of the compliance of the measurement system. The bonding strength of nanostructured thin films was also investigated both qualitatively and quantitatively and was compared across various geometries of the nanostructured film. Finally, equations that predict the elastic response of a helical spring were applied to understand and predict the response of a nanostructured thin film in shear and compression.

The individual objectives of this research were pursued by:

- Experimental measurements of axial stiffness, shear stiffness, and bonding strength were obtained by utilizing a custom-built experimental apparatus with the capabilities to load rectangular areas of thin films under uniform compression and in-plane shear and to measure the resulting force/displacement response.
• Theoretical predictions of axial and shear stiffness were obtained by applying previously-derived solutions for the elastic loading of a helical spring to the case of an array of springs loaded in parallel.

• The design of nanostructured thin films was analyzed by considering experimental observations and theoretical calculations to determine the optimum geometrical features of individual nanosprings in a film, which would result in maximum storage of elastic energy before nanospring yielding.
CHAPTER 2

MECHANICS OF NANOSTRUCTURED THIN FILMS

Uniform uniaxial compression and in-plane shear loading experiments were performed on rectangular areas of nanostructured thin films with thicknesses of 3 - 5 μm. Both the axial and shear stiffnesses were quantified by measuring the load versus displacement response of thin film samples and using it to extract the film stiffness. By assuming a uniform deformation of the anisotropic film, the film stiffnesses were related to the effective material stiffnesses in an analogy to the Young’s and shear moduli of a homogenous and isotropic material. All experiments were performed with the modular uniaxial testing apparatus shown in Figure 2.1, which was adopted and modified from [16] to allow for compressive and shear loading of nanostructured thin films. The experimental measurements were compared to theoretically-predicted values of stiffness based on an ideal helical spring model. The bonding strength of the films was also measured in some cases by loading sample areas under in-plane shear until they debonded from the substrate.

Figure 2.1 – Experimental apparatus used to measure force/displacement response of nanostructured thin films under compressive and shear loading.
2.1. COMPRESSIVE STIFFNESS OF NANOSTRUCTURED Cu FILMS

Uniaxial compression experiments were carried out on Cu-capped nanostructured Cu thin films obtained from General Electric Global Research (Niskayuna, NY) to measure the axial (compressive) stiffness of the nanostructured spring layer. The compressive stiffness was quantified by measuring the load versus displacement response of the samples and relating the sample stiffness (N/m) to a material stiffness (Pa) through a uniform compression assumption. The experimental results were compared to theoretical calculations based on the helical spring geometry of the nanostructured layer.

2.1.a. Materials and Methods

Cu-capped nanostructured Cu thin films deposited on a Si wafers or Tungsten foils were subjected to uniform compression to measure their axial stiffness. A Focused Ion Beam (FIB) milling technique was developed to create rectangular areas of the order of 300 × 300 μm² without subjecting the edges of the area of interest to significant damage. According to Figure 2.2, first, a rectangular border was milled through the sample to the Si substrate to define the shape of the desired area. Unlike other studies that used FIB milling to define an area of a nanostructured film [4], this study minimized the required milling time to define an area by cutting only a thin line around the region of interest and using a stiff probe to remove the surrounding material. This improvement allowed large areas to be precisely defined with short milling times: a 200 × 200 μm² sample area required less than 10 minutes of actual milling time. Compared to simple mechanical cutting with a razor blade or other sharp tool, this technique creates edges that are better defined that don’t suffer from the plastic deformation or local debonding seen in a razor blade cuts. Figure 2.3 shows close-up images of the edges of areas defined by FIB milling and razor blade cutting. The FIB-cut edge shows no signs of gross deformation or debonding of the material, while the edge cut with a razor blade exhibits signs of damage. The razor blade is unable to create a well-defined edge in many samples due to the strong cohesion of the cap layer.
Figure 2.2 – SEM images of the FIB area-definition technique. First a thin border is milled to the substrate to create the desired area (left). The surrounding material is then removed with a probe to provide a well-defined and isolated area (right).

After definition, each rectangular area was loaded with a home-built indenter that was slightly larger than the defined area to ensure uniform loading. The indenter was fabricated by fracturing a single crystal Si wafer into small rectangular pieces which were attached to an Al holder. The probe was attached to a linear actuator and the sample was in series with a miniature load cell with 50g, 250g, or 5 lb capacity, depending on the desired maximum force. The load cells had non-repeatability and hysteresis ±0.1% and ±0.15% of full range, respectively. The deformation of the nanostructured thin film was measured by optically recording the motion of the surface of the Si indenter, \( u_{\text{indent}} \), and the Si substrate, \( u_{\text{subst}} \), with a CCD camera at 465 nm/pixel resolution at 1024 × 768 pixels. Digital Image Correlation (DIC) was used to correlate the motion of the two surfaces, shown in Figure 2.4, and the deformation of the thin film layer was estimated as:

\[
\delta_{\text{film}} = u_{\text{indent}} - u_{\text{subst}} \tag{2.1}
\]

Equation (2.1) assumes that the displacement of the cap layer is much less than the displacement of the nanospring layer and can be neglected. This assumption is based on the realization that the cap layer has the stiffness of bulk Cu and that the nanospring layer is much more compliant, which was validated by the experimental results that
showed the overall response of the film to be three orders of magnitude less stiff than bulk Cu.

Figure 2.3 – SEM images of areas created by a FIB (left) and with a razor blade (right). The jagged edge seen in the razor blade cut is due to strong cohesion in the cap layer, which leads to the separation of large pieces of the film when loaded with a razor blade.

Figure 2.4 – Side view (not to scale) schematic of uniform compression loading of a nanostructured thin film.
2.1.b. Experimental Data

The axial stiffness of Cu-capped nanostructured Cu thin films was experimentally determined through the load vs. displacement curves of uniform axial compressions performed on FIB-defined rectangular areas of the films. The stiffness of a rectangular film area, $k_a$, was found from the unloading slope of the force-displacement data, and the effective film stiffness, $E^*$, was determined by:

$$E^* = k_a \frac{L}{A}$$ (2.2)

where $L$ is the length of the nanospring layer and $A$ is the area of the rectangular thin film specimen that was compressed.

Each rectangular area was loaded and unloaded multiple times between 0 - 15 MPa. The load range was chosen such that the stress was high enough to produce a measurable deformation of the film, but low enough such that the film remained in the linearly elastic regime. The unloading stiffness of each compression was calculated and used to obtain an average value for the film stiffness. Measurements from multiple areas resulted in an average value of $258 \pm 12$ MPa for the effective Young’s modulus of the thin film. A representative set of stiffness measurements across nine compressions of a $200 \times 200 \, \mu\text{m}^2$ area of Cu-capped nanostructured Cu film is shown in Figure 2.5, and the stress/strain response of the material is shown in Figure 2.6.
Figure 2.5 – Stiffness of a 2.5 μm thick nanostructured film based on nine compressions between 0 - 15 MPa.

Figure 2.6 – A series of nine compressions on a 200 × 200 μm² area of a Cu springs thin film.
The data showed random scatter in each series of compressions suggesting that there was no stiffening or softening over multiple compressions at the applied stress of 15 MPa. This can be further seen in Figure 2.6, where the stress versus strain response of the film is plotted for successive compressions. The curves are consistent across multiple compressions, although there is scatter in the displacement measurement on the order of a few percent of the sample strain due to the difficulty in resolving such small displacements with DIC. The curves in Figure 2.6 display an initial shallow slope up to 5% strain. This portion of the curve has been attributed to the gradual engaging of the flat probe to the thin film sample. Although the probe was aligned on two independent planes, the resolution of the optical microscope and local variations in the film and probe geometry placed a constraint on the degree of alignment that could be achieved. As a result, the initial segment of the loading curve represents a partial response as only part of the sample is loaded. It is also possible that the material experiences a stiffening effect due to the increased interaction between neighboring coils. Since this portion of the curve is typically seen at very small loads, the reported stiffness values are from the latter portion of the curve.

2.1.c. Comparison with Elastic Spring Theory

By modeling a nanostructured film as a collection of independent helical springs, an estimate of the theoretical axial stiffness of the thin film may be obtained and compared to the experimental results. To a first approximation, the axial stiffness of a helical spring is calculated by considering the spring as a torsion bar wound into a helix according to the analysis in [17]. The stiffness of an individual spring, $k_a$ [N/m], is then:

$$k_a = \frac{d^4 \mu}{8D^3N}$$

(2.3)

where $\mu$ is the material shear modulus [Pa], and $N$ is the number of coils in the spring. It then follows that an area $A$ [m$^2$] consisting of $m$ springs has stiffness of $k_a \cdot m$. Using Equation (2.2), the effective stiffness (modulus) of the constructed film is:
where $\xi$ is the area density of springs $= m/A$.

The parameters $D$, $N$, $L$, and $\xi$ can be readily obtained from SEM images of the springs and were $2.6 \, \mu m$, $2.5$ turns, $2.5 \, \mu m$, and $0.25 \, \text{springs/} \mu m^2$, respectively. $\mu$ was set to $48$ GPa which is the typically reported value for Cu. The value of $d$, however, is subjective since the geometry of the springs is not perfectly helical. Instead, it consists of an irregular cross-section that is swept from the spring’s axis to the outer perimeter of the spring. Figure 1.3 shows a visual comparison between the actual springs composing the nanostructured thin film and a perfectly helical spring. The value of $d$ is important to this calculation since it exhibits a $4^{th}$ power dependence on the stiffness. Since the wire cross-section is swept from the spring’s axis to the outer edge, a logical value for the wire diameter is half of the spring diameter. Taking this value of $d$ results in a stiffness of $290$ MPa, which is within $13\%$ of the experimentally-measured value of $257$ MPa for $2.5 \, \mu m$ Cu springs.

Equation (2.3) may be multiplied by a correction factor to account for the effects of non-zero helix angle, twisting moment, and direct shear [17], but this factor is typically no greater than $1.5 - 2$ and is overshadowed by the uncertainty in the wire diameter. This calculation also neglects the interaction of neighboring springs. As seen in Figure 1.1, there is significant overlap between neighboring springs and it is clear that there will be a contributing component of stiffness from this interaction.

2.1.d. Inelastic Deformation of Nanosprings

In order to assess the maximum stresses under which the film may be subjected during assembly and operation, the residual plastic strain at different applied loads was measured. The remnant plastic strain was quantified via confocal microscopy from the film height change between subsequent uniform compressions of a $200 \times 200 \, \mu m^2$ film area which was uniformly loaded to stresses of $250$, $375$, and $500$ MPa. The compressive
stress vs. plastic strain in Figure 2.7 suggests that the onset of plastic deformation occurred at stresses between 200 and 300 MPa.

![Figure 2.7](image)

Figure 2.7 – Plastic strain for compressive stress applied to a 200 × 200 μm² area of a 4.8 μm nanostructured Cu film. The horizontal error bars signify the uncertainty in the film height measurement.

This onset of plastic deformation was compared to the yield strength of solid Cu thin films that were fabricated by electron-beam evaporation and tested as composite films on polyimide under uniaxial tension [18]. The results showed a strong dependence of yield strength on film thickness, with the most significant strengthening observed for thicknesses under 1 μm. For film thicknesses ~5 μm, the yield strength was in the range of 200 - 400 MPa, which is consistent with the range of 200 - 300 MPa measured here.

### 2.2. SHEAR STIFFNESS OF NANOSTRUCTURED Cu FILMS

In-plane shear experiments were performed on Cu-capped nanostructured Cu thin films to measure their shear stiffness. As with the uniaxial compression experiments, the film compressive stiffness (N/m) was related to a material stiffness (Pa) through a uniform in-plane shear assumption. The experimental results were compared to
theoretical calculations that predict the transverse stiffness of a spring and the expected ratio of normal-to-transverse stiffness.

### 2.2.a. Materials and Methods

Ag and Cu nanostructured thin films with heights of 4.8 μm were obtained from General Electric Global Research (Niskayuna, NY) and used in the measurement of shear stiffness. The films were deposited on W or Si and rectangular areas of the films on the order of 0.1 mm² were isolated using the previously-described FIB milling technique. Each area was loaded under in-plane shear by attaching a flat glass grip to the cap layer with a UV-curable epoxy and by displacing the grip with a Picomotor™ linear step actuator. A schematic of the experimental procedure is shown in Figure 2.8.

![Figure 2.8 – Side view (not to scale) schematic of in-plane shear loading of nanostructured thin films.](image)

The applied force was measured by mounting the sample in series with a 50 g precision miniature load cell with non-repeatability and hysteresis of ±0.1% and ±0.15% of full range, respectively. The displacement of the nanostructured film was obtained by assuming the substrate and cap to be rigid when compared to the compliant nanospring
layer. This allowed for the shearing of the nanospring layer to be measured by taking the difference between the motion of the Si substrate and the loaded cap layer. Both surfaces were monitored throughout the experiment with a microscope-mounted CCD camera at a resolution of 465 nm/pixel and 1024 × 768 pixels and the motion was extracted by applying DIC to the recorded images. In order to bring the substrate to the same focal plane as the loaded cap layer, an identical glass probe was attached to an adjacent unloaded sample area. A top view of the experimental arrangement with overlaid the DIC calculated displacements in the y-direction, \( v \), is shown in Figure 2.9. The displacements measured had to be sub-pixel in size because of the small applied shear strains. Large shear strains were not always possible due to debonding of the glass grip from the Cu cap layer. DIC has been reported to have resolution of the order of \( 1/10^{th} \) of a pixel when rigid body motions are measured [19,20], which supports the sub-pixel resolution required in the present experiments.

![Figure 2.9](image-url)

**Figure 2.9** – Top view of vertical displacement fields of the specimen area (left) and unloaded substrate reference area (right). The contours show the point-wise calculated y-direction displacements in pixels (1 pixel = 465 nm).
2.2.b. Experimental Measurement of Shear Stiffness

The shear stiffnesses of Cu-capped Cu nanostructured films and Ag-capped Ag films were determined from the load vs. displacement curves of in-plane uniform shear loading tests. The stiffness of a rectangular specimen area was computed from the best-fit line of the force-displacement data and the material stiffness, $\mu^*$, was calculated by:

$$
\mu^* = \frac{k_t L}{A}
$$

where $k_t$ is the measured stiffness of the rectangular area [N/m], $L$ is the length of the nanostructured spring layer [m], and $A$ is the area of the tested region [m$^2$]. Since the angular deflections of the nanosprings are small and the solid cap layer is much stiffer than the spring layer, the above equation for uniform shear loading can be applied. The shear stress/strain curve for a $360 \times 280 \, \mu m^2$ sample is presented in Figure 2.10.

![Figure 2.10 – Shear stress vs. strain plot for in-plane shear loading of a 360 × 280 \( \mu m^2 \) rectangular area of a nanostructured Cu-capped Cu thin film.](image-url)
The shear behavior of the nanostructured films was nearly linear, due to small angular deformations that were of the order of a few degrees. The data showed a small-amplitude, low-frequency superposition to a linear curve that could be attributed to the convergence accuracy of the DIC algorithm arising from attempting correlation of areas with very small displacements. This low-frequency variation does not affect the integrity of the data since the overall large displacement allows for an accurate determination of the slope of the curve.

The epoxy used to bond the loading grip to the sample area was unable to sustain stresses high enough to obtain large displacements and strains in the non-linear regime of the film response. It is expected that large lateral displacements of the nanostructured films would result in stiffening of the film as neighboring springs become intertwined. Large deflections of individual springs would also cause rotation about the base of the spring, giving rise to vertical in addition to a lateral displacement.

Figure 2.11 – Average shear stiffness of nanostructured thin film samples. Error bars represent one standard deviation of the data.
A summary of the shear property measurements is presented in Figure 2.11 and Table 2.1 with an average shear stiffness of 19.3 MPa and 20.9 MPa for the two Cu samples and 14.5 MPa for Ag samples. The standard deviation across multiple areas on an individual sample die was 3 MPa for the Cu sample on Si, 4.5 MPa for Cu on W, and 2 MPa for Ag samples for 3-6 measurements in each case.

<table>
<thead>
<tr>
<th>Material</th>
<th>Shear Stiffness (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu springs on Si</td>
<td>19.3 ± 2</td>
</tr>
<tr>
<td>Cu springs on W</td>
<td>20.9 ± 4.5</td>
</tr>
<tr>
<td>Ag springs on Si</td>
<td>14.5 ± 3</td>
</tr>
</tbody>
</table>

The data scatter can be attributed to variations in the loading conditions across different experiments. Since epoxy coverage of the sample region varied across experiments, the loading conditions of the film varied accordingly causing the sample area to be loaded in a non-uniform manner. While these effects were minimized as much as possible, the inherent nature of the epoxy produced inevitable variations across experiments. Further scatter in the data can be simply attributed to local variations in the film morphology. Between the two Cu samples, the one deposited on the W foil had greater standard deviation of the measured stiffness than the sample on Si. Since a Si wafer is much more smooth and rigid than a W foil, this difference in data scatter can be attributed to local height variations in the W foil. Parts of the foil may not have been completely attached to the underlying fixture, thus introducing extraneous deformations in the film.

2.2.c. Comparison with Elastic Spring Theory

As with the case of compression, equations derived for the helical spring geometry may be used to obtain an approximation of the shear stiffness of the
nanostructured films. An approximation to the lateral stiffness of a single helical spring based on a beam bending model is given by [17,21]:

\[ k_t = \frac{Ed}{8Nc^3} \left[ 1 + \frac{1}{12} \left( \frac{2L}{D} \right)^2 \left( 1 + \frac{E}{2\mu} \right) \right]^{-1} \]

(2.6)

Using Equation (2.5), the effective shear modulus of a film constructed from an array of springs with lateral stiffness \( k_t \) and area density \( \xi \) is given by:

\[ \mu^* = \frac{Ed}{8Nc^3} L\xi \left[ 1 + \frac{1}{12} \left( \frac{2L}{D} \right)^2 \left( 1 + \frac{E}{2\mu} \right) \right]^{-1} \]

(2.7)

As with the expression for the axial spring stiffness, there exists a strong dependence on the wire diameter of the spring. Since the springs are not comprised of perfect helical coils, an approximation has to be made for the effective coil diameter. Assuming the effective wire diameter to be \( \frac{1}{2} \) of the spring diameter gives values of the effective shear modulus of 158 MPa for the Cu films and 105 MPa for the Ag films. These values are several times larger than the experimental estimates. If the effective wire diameter is taken to be \( \frac{1}{3} \) of the spring diameter, values much closer to the experimental are obtained: 31 and 21 MPa. In calculating the axial stiffness, it was found that taking the effective wire diameter to be half the spring diameter provided the closest approximation of the axial stiffness; here, letting the wire diameter be one-third the spring diameter provided a close correlation with the experimental data. It can be concluded that the actual geometry of the coils and their mutual entanglement are such that, compared to perfect and isolated helical springs, there is larger stiffening effect in the normal direction than the lateral direction.

The effect of material properties on the shear stiffness of the nanosprings can be analyzed by considering the ratio of lateral stiffnesses of individual helical springs made of different materials but with identical geometries, which depends both on the elastic moduli of the two materials and the length and diameter of the springs:
For the given spring geometry and elastic moduli, the expected ratio of shear stiffness for a Ag spring to a Cu spring is 0.670, which is within 11% of the experimental value of 0.751. Once again the deviation from helical spring geometry makes the calculation an idealization of the actual film, although the effect of the bracketed terms is minimal for the given geometry. The most significant term is the ratio of the elastic moduli of the two materials, which is what the expression condenses to for a simple beam bending model.

### 2.3. INTERFACIAL BONDING STRENGTH OF NANOSTRUCTURED FILMS

The interfacial bonding strength between Cu-capped nanostructured Ti thin films and a Si substrate was measured through in-plane shear loading. The bonding strength was quantified by measuring the average applied stress to the specimen area and taking the average stress at failure to be the bonding strength.

In these experiments, rectangular areas of Cu-capped nanostructured Ti thin films were defined and loaded under in-plane shear loading in a method identical to that of the experimental measurement of shear stiffness. Areas ranging in size from $1.5 \cdot 10^4 \, \mu\text{m}^2$ to $6.4 \cdot 10^4 \, \mu\text{m}^2$ were loaded at a rate of approximately 1.5 mN/s until catastrophic drop in load, at which point debonding from the substrate was observed. The average bonding strength was calculated by dividing the maximum sustained load by the total loaded specimen area. An example of a debonded sample area is shown in Figure 2.12.
2.3.a. Experimental Measurements of Bonding Strength

The average bonding strength was measured in multiple areas of nanostructured Ti thin films, and the results are plotted as a function of specimen area in Figure 2.13. There is a clear trend of decreasing bonding strength with increasing specimen area with smaller areas showing over 3.0 MPa average bonding strength and large areas showing bonding strengths of 0.5 MPa. This result can be rationalized by considering the process of film loading and debonding as the nucleation/initiation and propagation of a crack across the interface that starts at an edge of the film where the stresses are the highest.
This dependence of the average bonding strength on the interfacial area is commonly seen in adhesive bonding of fiber/epoxy composite systems [22]. Figure 2.14 shows the adhesive bond strength plotted as a function of the interfacial area for multiple fiber/epoxy systems, as reported in literature. Although the interfacial bonding mechanism is different in the two cases, the data for the bonding strength of nanostructured thin films follow the trend observed in the debonding experiments of fiber/epoxy matrix. Both cases also experience the same predominantly mode II shear loading conditions and the ductile cap layer of the nanostructured thin film is able to distribute the load across the area despite the presence of a discrete array of nanosprings underneath. These results suggest that a simple average stress model is not sufficient to characterize the bonding strength of nanostructured thin films and that the precise stress profile and the fracture toughness of the interface must be considered instead.
Figure 2.14 – Adhesive bond strength vs. interfacial area for multiple carbon fiber/epoxy systems. Chart reproduced from [22].
3. OPTIMIZATION OF NANOSTRUCTURED THIN FILMS

In the application of nanostructured thin films, it is crucial to understand how the morphology of the spring layer affects the overall mechanical response of the film. In homogenous metals, properties such as hardness and toughness are the manifestation of the metal’s unique grain and crystal structure and are tailored to meet the material demands of a specific application. Qualitative observations were used to predict how the interaction between the spring and cap layers determines the response of the film to concentrated loading. Theoretical spring calculations were then used to understand how the geometric parameters of the spring layer can be used to optimize the storage of elastic energy in the film.

3.1. EFFECT OF SPRING GEOMETRY ON QUALITY OF THE INTERFACE

The effect of the geometry of the nanostructured springs on the bonding quality with a Si substrate was explored through qualitative observations of the debonding sites. Debonding was achieved in a controlled manner by subjecting the thin film to a concentrated line load in the form of a sharp razor blade drawn across the surface. Three types of nanostructured thin films were studied: slender branched springs, helical springs with a thin cap and helical springs with a thick cap. Tilted side views of all three samples are shown in Figure 3.1. The first two samples were tested as-fabricated with the GLAD process by General Electric Global Research (Niskayuna, NY), while the third sample consisted of as-received nanostructured films on which a 3-4 μm coating of sputter-deposited Cu was added at UIUC.
Figure 3.1 – Comparison of three types of nanostructured thin films: (a) branched springs with thin cap, (b) helical springs with medium cap, and (c) helical springs with thick cap.
Figure 3.2 shows a comparison of the common types of failure seen within each type of sample. The slender branched springs offered little cross-linking and intertwining between adjacent springs so the main type of debonding that was observed was a uniform debonding at the substrate/spring interface. The thin cap layer held the springs together, while at the same time allowing large strips of material to be removed without causing the cap to crack or crumble. Helical springs with a high degree of intertwining showed the highest resistance to debonding from the substrate as the majority of the energy went into plastic deformation of the thin film and cracking and separation of the cap layer. The exact type of deformation depends on the applied loading and specifically the type of razor blade used. A dull, flat blade subjects the film to compressive loading, leading to plastic deformation, while a thin, tapered, blade tends to open the film/substrate interface, leading to cracking and separation of the cap layer. Depositing an additional 3-4 \( \mu \text{m} \) of Cu on top of this cap layer allowed the cap layer to be more cohesive and to hold the springs together more uniformly. The high bending stiffness and high resistance to cracking allowed the cap layer to “lift off” from the substrate, pulling the underlying helical springs with it.

These observations lead to the conclusion that the mode of debonding of a nanostructured thin film is highly dependent on the nanostructured spring geometry and perhaps more importantly on the thickness of the cap layer. The height of the springs is also important but it was not possible to study the effect in this work in an extensive way. It is, therefore, hypothesized that helical wires with large coil diameters and high degree of intertwining favor deformations that are localized and consist primarily of plastic deformation of the film or cracking of the cap layer, depending on the type of load applied. Slender branched wires with a thin cap layer can be peeled off in large sections since the majority of the load is distributed through the ductile cap layer. Having a thick cap layer further improves the cohesiveness of the thin film by preventing failure from initiating in the cap layer in the form of cracking.
3.2. NANOSPING DESIGN FOR STRAIN ENERGY DENSITY

In general, it is desirable to maintain the applied load in the elastic regime when loading an array of helical springs. Permanent deformation of the helical springs would change the overall film response under further loading. It is, therefore, desired to
maximize the strain energy that a spring layer can absorb prior to spring yielding by selecting the appropriate geometric and material parameters for the springs. This was investigated in the present research by considering axial and transverse loading with an initial axial preload. A schematic of the two types of loading is shown in Figure 3.3.

![Figure 3.3](image)

Figure 3.3 – Axial (left) and transverse (right) loading of a helical spring. The convention used in this analysis is that $P$ is the axial force and $F$ is the transverse force with respect to the spring axis.

### 3.2.a. Axial Spring Loading

The strain energy of a single helical spring loaded along its axis was calculated and the expression was then applied to a collection of parallel springs. To a first approximation, a single helical spring could be modeled as a torsion bar wound into a helix. The maximum shear stress, $\tau$, can then be calculated for the case of pure torsion of a circular cross-section and is given by [17]:

$$\tau = \frac{16P}{nD^2}$$
Since the helical spring is being modeled as a torsion bar, this expression was derived by considering the torsional stresses arising from a torque that is generated by a force $P$ applied to the spring at a moment arm $D/2$.

This elementary formulation assumes that the twisting moment due to curvature and direct shear effects do not contribute significantly to the maximum shear stress and that the helix angle of the spring is small. These effects can be compensated for by multiplying the expression in (3.1) with a stress correction factor, $K$, which is a function of the spring index, $c=D/d$, and the helix angle $\lambda$. The expression for $K$ can be obtained by considering curvature and direct shear effects or by solving the exact elasticity problem. The most commonly used expression for $K$ has been derived by Wahl by superposing the stresses due to curvature and direct shear and is given by [17]:

$$ K_{elementary} = \frac{4c - 1}{4c - 4} + \frac{0.615}{c} $$

The Taylor expansion of an exact elastic solution was obtained by Henrici [23] and was refined by Wahl to include the effect of a non-zero helix angle $\lambda$ [17]. The first five terms of the expansion and the correction for the helix angle are given by:

$$ K_{exact} = 1 + \frac{5}{4c} + \frac{7}{8c^2} + \frac{155}{256c^3} + \frac{11911}{24576c^4} + H.O.T. + \frac{1}{2} \tan^2(\lambda) $$

As shown in Figure 3.4, for small helix angles and large spring indices, the stress correction factor is nearly unity. As the wire diameter approaches half the coil diameter, the stress increases by a factor of 2 for small helix angles, and by as much as 2.5 for larger helix angles.
Figure 3.4 – Stress correction factor, $K$, from elementary theory and as a function of the spring index, $c$, for various helix angles.

The total strain energy of the spring can be calculated by considering the linear spring energy equal to:

$$W = \frac{1}{2} P_y \delta_y = \frac{1}{2} \frac{P_y^2}{k_a}$$  \hspace{1cm} (3.4)

where $k_a$ is the axial spring stiffness, $\delta$ is the axial deflection of the spring and the subscript $y$ denotes the state of yielding. The axial stiffness of the spring can be calculated by integrating the differential deflections of the torsion bar and is given by [17]:

$$k_a = \frac{\mu d}{8N c^3}$$  \hspace{1cm} (3.5)
where \( N \) is the number of turns in the spring. The force required to yield the spring, \( P_y \), can be calculated by evaluating the corrected form of Equation (3.1) for the Tresca maximum shear criterion \((\tau = \sigma_y/2)\). The resulting expression for \( P_y \) and the expression for \( k_a \) can then be substituted into Equation (3.4) to obtain an expression for the linear elastic spring energy of a helical spring at yielding:

\[
W = \left( \frac{\pi^2}{64} \right) \left( \frac{Nd^2}{K^2} \right) \left( \frac{\sigma_y^2}{\mu} \right) \tag{3.6}
\]

where \( \sigma_y \) is the yield strength of the material.

The above expression is applicable for a single spring loaded at yield stress. For an array of identical springs in parallel, the total strain energy is multiplied by the total number of springs. Since the energy per unit area, \( w \), is to be maximized, the expression to be maximized becomes:

\[
w = \left( \frac{\pi^2}{64} \right) \left( \frac{Nd^2}{K^2} \right) \left( \frac{\sigma_y^2}{\mu} \xi \right) \tag{3.7}
\]

where \( \xi \) is the areal spring density [springs/area]. Considering the GLAD process in which helical springs are generated from the shadowing of neighboring springs, it can be assumed that the spring spacing should be approximately equal to the coil diameter of the individual springs. This assumption is supported by the nanostructured spring layer in Figure 1.1. Since the linear spacing is nearly equal to the coil diameter, the areal density of the springs will be proportional to \( 1/D^2 \) and the expression becomes:

\[
w \approx \left( \frac{\pi^2}{64} \right) \left( \frac{Nd^2}{D^2 K^2} \right) \left( \frac{\sigma_y^2}{\mu} \right) \tag{3.8}
\]

The above expression is a product of numerical constants, geometric properties, and material properties. To maximize the material properties term, a material with the largest ratio of the square of the yield strength to the shear modulus should be chosen.
For a given material, post-fabrication thermal processing can be performed to modify the material yield strength.

The independent variables in the geometric term are the spring diameter, the wire diameter and the number of turns in the spring. The number of turns in the spring is typically dictated by the height of the coils, which is most likely determined by the specific application of the film. For a spring with a given number of turns, \( N \), the geometric ratio to be maximized can be written as:

\[
w_{geom} \approx \left( \frac{d^2}{DK^2} \right) = \left( \frac{d}{cK^2} \right)
\]  

(3.9)

Calculations show that a spring index of \( c=2.93 \) maximizes the function for the Wahl stress correction factor and \( c=2.80 \) maximizes the function for the Henrici stress correction factor for zero helix angle, as shown in Figure 3.5. This value is independent of the actual geometry since the expression for \( w_{geom} \) is strictly a product of independent terms. Setting \( K = 1 \) provides no maximum as the function monotonically increases with decreasing spring index. The optimum spring index decreases linearly with increasing helix angle and the relationship is plotted in Figure 3.6.
Figure 3.5 – Ratio \( \frac{d}{c \cdot K^2} \) for \( d=1 \) (arbitrary units) as a function of spring index \( c \) for uncorrected stress \((K=1)\), Wahl stress correction and exact theory solution for multiple helix angles.

As a result, the individual helical springs should have a coil diameter that is \(~2.5 - 3\) times greater than the wire diameter to allow for the most elastic energy to be stored before material yielding. The exact value depends on the helix angle and can be determined from Figure 3.6. This is the point at which the geometry is such that the wire is slender enough to avoid significant stress concentrations that would promote premature yielding, while at the same time allowing the stiffness to be high enough so that a significant amount of elastic spring energy can be stored prior to yielding.
3.2.b. Combined Axial and Shear Loading

In the analysis of combined loading, the strain energy of a single helical spring loaded transversely to its axis is analyzed and the expression is then applied to an array of springs in parallel. The calculation of stresses requires the presence of an axial load, \( P \). Unlike the case of pure axial loading, the maximum shear stress under mixed loading is not simply linearly proportional to the applied force. Instead the maximum shear stress depends on both the transverse displacement, \( \delta_t \), and the axial load \( P \) and can be approximated by [17]:

\[
\tau = K \frac{8PD}{\pi d^3} \left( 1 + \frac{\delta_t}{D} + \frac{FL}{PD} \right) \tag{3.10}
\]

where \( P \) and \( F \) are the forces shown in Figure 3.3. The transverse displacement is simply the transverse force divided by the transverse stiffness given by Equation (2.6) so Equation (3.10) can be written entirely in terms of forces as:
\[ \tau = K \frac{8PD}{\pi d^3} \left(1 + \frac{F}{k_t D} + \frac{FL}{PD}\right) \quad (3.11) \]

As before, \( K \) is the stress correction factor based on the geometry of the spring. For the purposes of this analysis, the axial force \( P \) is treated as constant (i.e. a pre-load) and the transverse load \( F \) is allowed to vary. Assuming linear superposition, the maximum elastic spring energy stored prior to yielding is:

\[ W = \frac{1}{2} \frac{P^2}{k_a} + \frac{1}{2} \frac{F_y^2}{k_t} \quad (3.12) \]

where \( F_y \) is the transverse force required for the maximum shear stress to reach a value of \( \sigma_y/2 \). Equation (3.11) can be evaluated for the Tresca maximum shear criterion at \( \tau = \sigma_y/2 \) to solve for the required force, \( F_y \), for initial yielding of the spring:

\[ F_y = \left( \frac{\pi d^3 \sigma_y}{16KPD} - 1 \right) \left( \frac{Dk_t P}{k_t L + P} \right) , \quad P < \frac{\pi d^3 \sigma_y}{16DK} \quad (3.13) \]

The derivation of Equation (3.13) assumes that the spring has not yielded due to the axial pre-load, \( P \), thus placing an upper bound to \( P \). Any pre-load exceeding the upper bound prescribed in Equation (3.13) will itself cause yielding without the presence of a transverse force.

The critical force, \( F_y \), can now be substituted into Equation (3.12) to develop an expression for the amount of linear spring energy stored in an individual spring for an axial preload \( P \) when loaded to the yield point via transverse loading:

\[ W = \frac{1}{2} \frac{P^2}{k_a} + \frac{1}{2} \frac{\left( \frac{\pi d^3 \sigma_y}{16KPD} - 1 \right)^2 \left( \frac{Dk_t P}{k_t L + P} \right)^2}{k_t} \quad (3.14) \]
As with the case of pure axial loading, when an array of springs is loaded in parallel the entire expression must be multiplied by \(1/D^2\) to obtain the energy stored per unit area under the assumption that the areal density of springs is proportional to \(1/D^2\). The final expression for the linear spring energy stored per unit area becomes:

\[
W = \frac{1}{2} \frac{p^2}{k_a D^2} + \frac{1}{2} \frac{\left( \frac{\pi d^3 \sigma_y}{16 K P D} - 1 \right)^2 \left( \frac{D k_t P}{k_t L + P} \right)^2}{k_t D^2}
\]  \(3.15\)

Unlike the case of pure axial loading where the optimum spring index was independent of all constants except the helix angle, in the case of combined loading the material properties and the aspect ratio, \(L/D\), affect the value of \(c\) at which the stored linear spring energy is maximum when the coil diameter is held constant and the wire diameter is varied. Figure 3.7 shows the spring energy in arbitrary units as a function of the spring index, \(c\), for varying values of the aspect ratio. The plots were generated for the material properties of Cu and for one coil per unit length of a spring. The optimum spring index increases with decreasing aspect ratio of a spring of a given coil diameter and is in the range of 2.25 - 2.5. The relative amount of energy increases significantly with decreasing aspect ratio (increasing coil diameter) such that a *single large spring has higher energy storage capacity than an array of smaller springs*. However, in the case of nanostructured thin films the coil diameter is largely limited by the fabrication process. In this case, the coil diameter should be maximized to the extent that the fabrication process allows and then the wire diameter should be chosen such that it is approximately 2.4 times smaller than the coil diameter.
Figure 3.7 – Total spring energy, w, absorbed at yield for an array of springs with varying aspect ratio.
CHAPTER 4

CONCLUSIONS

The mechanical properties of nanostructured thin films of thicknesses of 4 - 6 μm were measured by custom experimental techniques that addressed the challenges of defining and loading in uniform compression and shear small thin film domains attached to a substrate. In this method, rectangular areas with length and width on the order of 200 - 300 μm were isolated from continuous nanostructured Cu or Ti films deposited on Si or W substrates by FIB milling. The individual areas were then loaded under compression or shear and the force/displacement response of the nanostructured films was used to extract the effective film stiffnesses.

The average compressive stiffness of the Cu films was measured to be 258 ± 12 MPa with variability across successive compressions of 12 MPa. This value is nearly 500 times lower than the Young’s modulus of monolithic Cu, emphasizing the applicability of nanostructured thin films in compliant interfaces. While the actual spring geometry was not perfectly helical, modeling the springs as perfectly helical springs with an effective wire diameter assumed to be equal to half the coil diameter predicted a stiffness of 290 MPa, which is within 13% of the experimentally-calculated values. The experimental data suggested that the effective wire diameter should be equal to nearly half the coil diameter when predicting the compressive stiffness of the nanostructured films at hand. The compressive yield strength of the films was estimated to be in the range of 200 - 300 MPa, which follows closely with uniaxial tension results for the yield strength of homogenous Cu thin films. Further experiments are needed to validate this estimate, potentially combined with FIB pre- and post-compression cuts that will reveal the state of the compressed springs.
The average shear stiffness of Cu and Ag films on Si substrate was measured to be $19.3 \pm 3$ MPa and $14.5 \pm 2$ MPa, respectively, across multiple areas on a single sample die. Cu films on W had nearly the same stiffness as Cu films on Si, but the scatter in the data was much larger, owing to irregularities in the W substrate. The 33% increase in stiffness due to the material differences followed to the theoretical prediction of 49% increase arising from the relative elastic moduli of bulk Cu and Ag. Theoretical calculations predicted a shear stiffness that was eight times larger than the experimentally-measured value when the effective wire diameter was assumed to be half the spring diameter. However if the effective wire diameter was assumed to be one-third of the coil diameter, the theoretically-predicted values were within 30% of the experimentally-determined values. This disagreement implies that the imperfectly-helical geometry of the springs and the specific spring entanglements contribute more to stiffening in the axial direction than in the transverse direction. The result is that the effective wire diameter used in calculating the theoretical stiffness of the particular springs should be larger when evaluating the axial stiffness than when evaluating the shear stiffness of the films.

Qualitative observations and theoretical application of the constitutive spring equations were used to predict the optimal nanostructured spring film geometry that would provide a perfectly-bonded interface with the maximum elastic spring energy before material yielding. On the qualitative side, it was found that films for which the inter-spring spacing was smaller than the coil diameter experienced primarily local plastic deformation and cracking when subjected to a concentrated load, while films comprised of slender springs or a cap of thickness much larger than the springs height tended to debond uniformly from the substrate as the load was transferred efficiently from the loading point to the springs. Theoretical calculations showed that a spring index of 2.5 - 3 provided the optimal geometry for maximizing the elastic spring energy during compressive loading of a film until yielding. The optimal spring index decreased with increasing helix angle and was independent of the material properties, which can be separately maximized. Spring materials with a large ratio of the square of their yield strength to their shear modulus are best suited for storing elastic energy since stresses are
able to remain below the yield point at high loads. Under combined axial and shear loading, there is coupling between many of the geometric and material parameters. For Cu springs of a given length, the stored elastic energy reaches a maximum at a spring index of 2.3 - 2.4. Increasing the aspect ratio (lowering the coil diameter) reduces the optimum spring index.

Further work on material characterization of the nanostructured thin films may focus on developing a model for the mechanical response of the films based on varying fabrication parameters. Instead of attempting to model the structured layer as springs with particular geometric parameters, it is more convenient to understand the effects of incident flux, substrate angle and substrate rotation on the resulting mechanical properties of the film. Multiple films may be fabricated with varying fabrication parameters and a design of experiments (DoE) methodology can be applied to derive the effective film stiffness as a function of the significant fabrication parameters. This would allow a film to be fabricated with the desired mechanical properties simply by selecting the appropriate fabrication parameters.
REFERENCES


