Jul 29th, 11:00 AM - 1:30 PM

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Joshua Wolff
University of Northern Iowa

Jeff Carlson
University of Northern Iowa

See next page for additional authors

Author

Joshua Wolff, Jeff Carlson, and Timothy E. Kidd Ph.D.

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University of Northern Iowa
Department of Physics

Joshua Wolff, Jeff Carlson, and Dr. Timothy E. Kidd, Ph.D.
Department of Physics, University of Northern Iowa, Cedar Falls, Iowa 50613

Abstract

We have developed a method to control the surface roughness of ultrathin Au surfaces on MoS_2 crystals through the use of annealing. Through this method, we are now able to utilize Au surfaces for a variety of research, such as research on self-assembled monolayers (SAM) as well as with surface-enhanced Raman spectroscopy (SERS).

Introduction

The study of ultra-thin, ultra-flat Au film on MoS_2 substrates is prevalent due to their prime candidacy for use as a base in SAMs, as gold is among the least reactive transition metals.

Being able to control the surface roughness is particularly of interest for SERS. In SERS, the light enhancement is influenced by the surface roughness of the film. Gold is useful for SERS due to its biocompatibility and its ability to enhance light at its surface, so tuning the Au surface roughness can be used to optimize the sensitivity of Raman spectroscopy.[4] Thus, finding a method to control surface roughness increases the utility of gold films for a wide variety of research techniques.

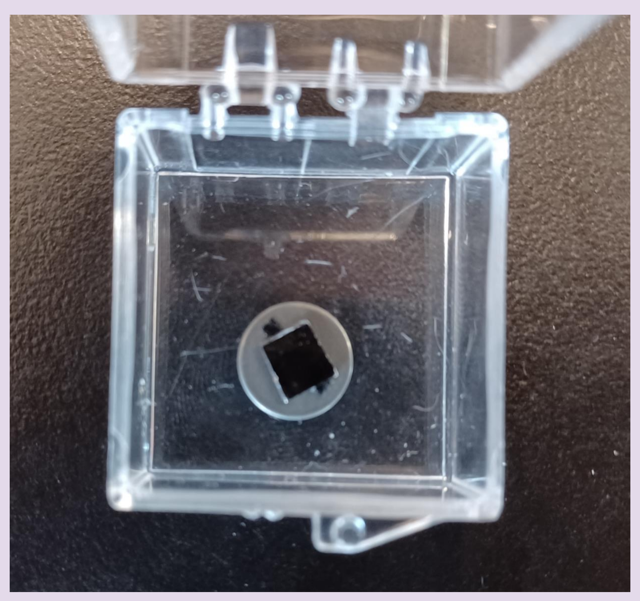

Figure 1: Sample of 12nm gold film on an MoS_2 crystal that was attached to a silicon wafer via double-sided thermal tape. Samples were placed onto a sample disk and scanned inside of the Atomic Force Microscope, followed by analysis utilizing the Gwyddion software.

Method

Each sample is a 12 nm Au film deposited onto a 1.3 nm thick MoS2 film. The underlying substrate is a highly doped Si(100) single crystal covered by a 0.5nm native oxide (SiO2).

MoS2 films are sputtered at 700C at 5 millitorrs Ar in a chamber with a base pressure of 10^{-7} Torr vacuum. Au films were sputtered at room temperature at 400 millitorrs in a system with a base pressure of 10 millitorrs.

After deposition, the same sample was annealed in ambient conditions for 30 minutes at room temperature, 100C, 200C, and 300C, then allowed to cool..

After cooling, the sample was scanned using an Agilent 5500 SPM Atomic Force Microscope (AFM) at a variety of different sizes ranging from .5μm to 10μm to measure the surface roughness at different temperatures. Once the topographical images were obtained using the AFM technology, results were analyzed using the Gwyddion software.

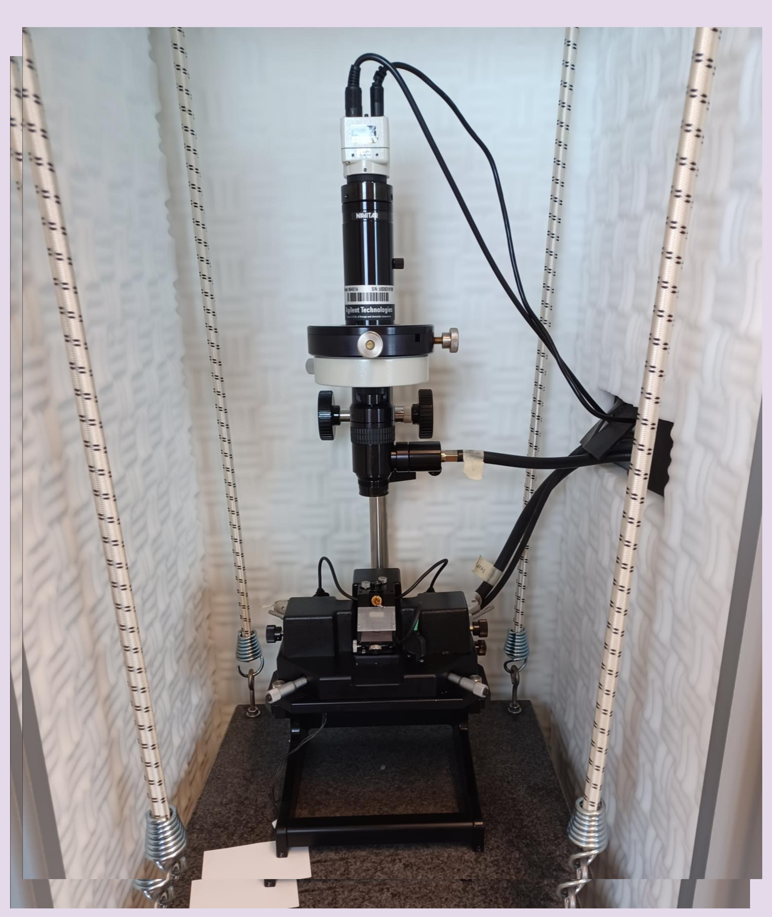

Figure 2: Agilent 5500 SPM Atomic Force Microscope. Used to scan samples and get a measure of surface roughness in combination with analysis via Gwyddion software. Topographic images are generated using a pointed tip that scans the surface of the sample while laser deflection caused by the movement is measured using a photodetector.[1]

Results

Topographical View of Au Surface via AFM

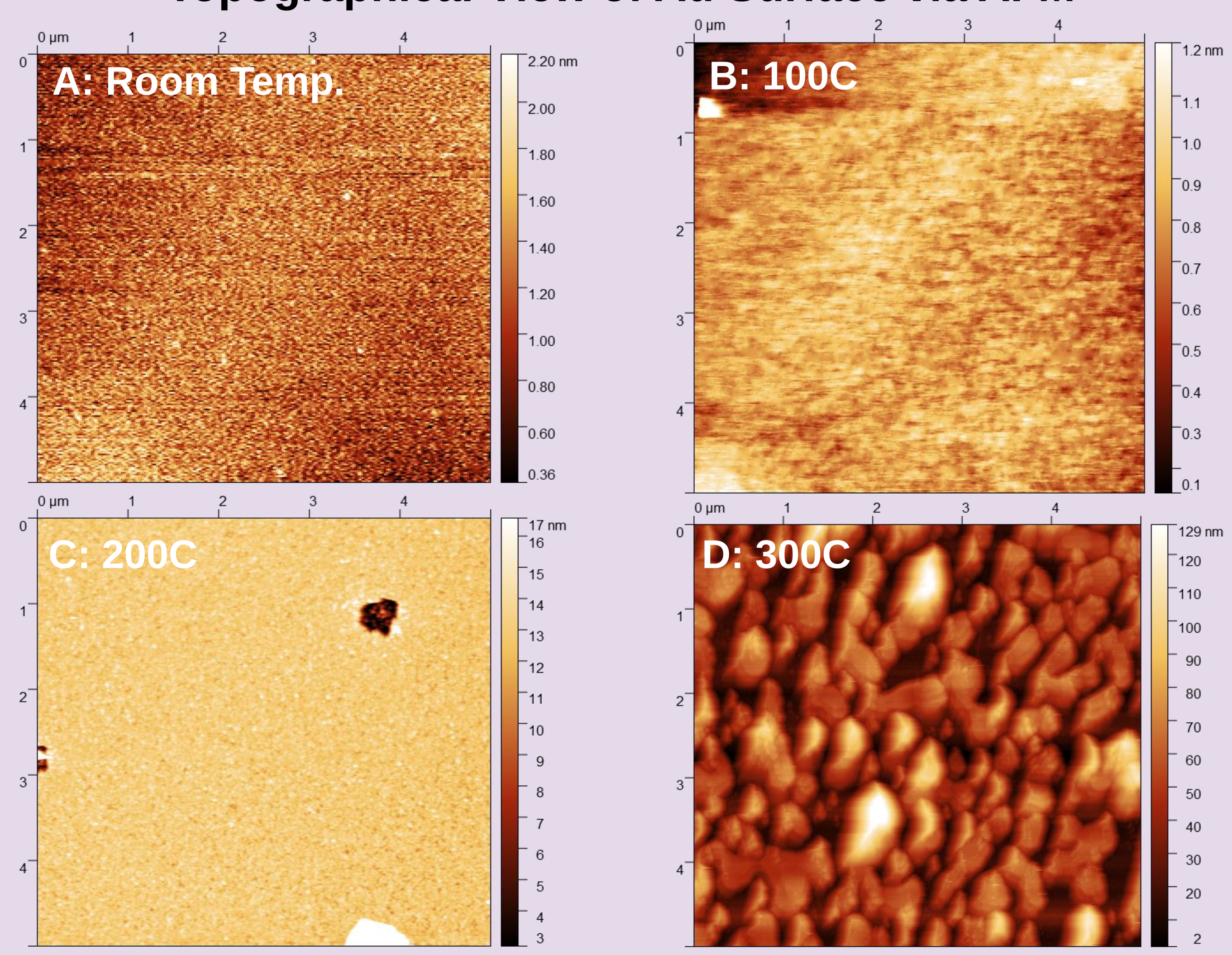

Figure 3: AFM topography of samples annealed to different temperatures. Each sample is a 12 nm thick Au film deposited onto a 1.3 nm thick MoS_2 film. Each temperature is shown in the top left of the images. The large chunk present in image C is understood to be a "boulder" that rolled off from the hole above. Surface roughness for C was calculated in areas without this defect.

Sample	Temperature (°C)	Surface Roughness (nm)
A	Room Temperature	0.288
B	100°C	0.153
C	200°C	0.613
D	300°C	20.62

Figure 4: Table representing the surface roughness corresponding with each temperature the sample was annealed to, Data shows that there must be some threshold between 200C and 300C at which surface roughness increases dramatically.

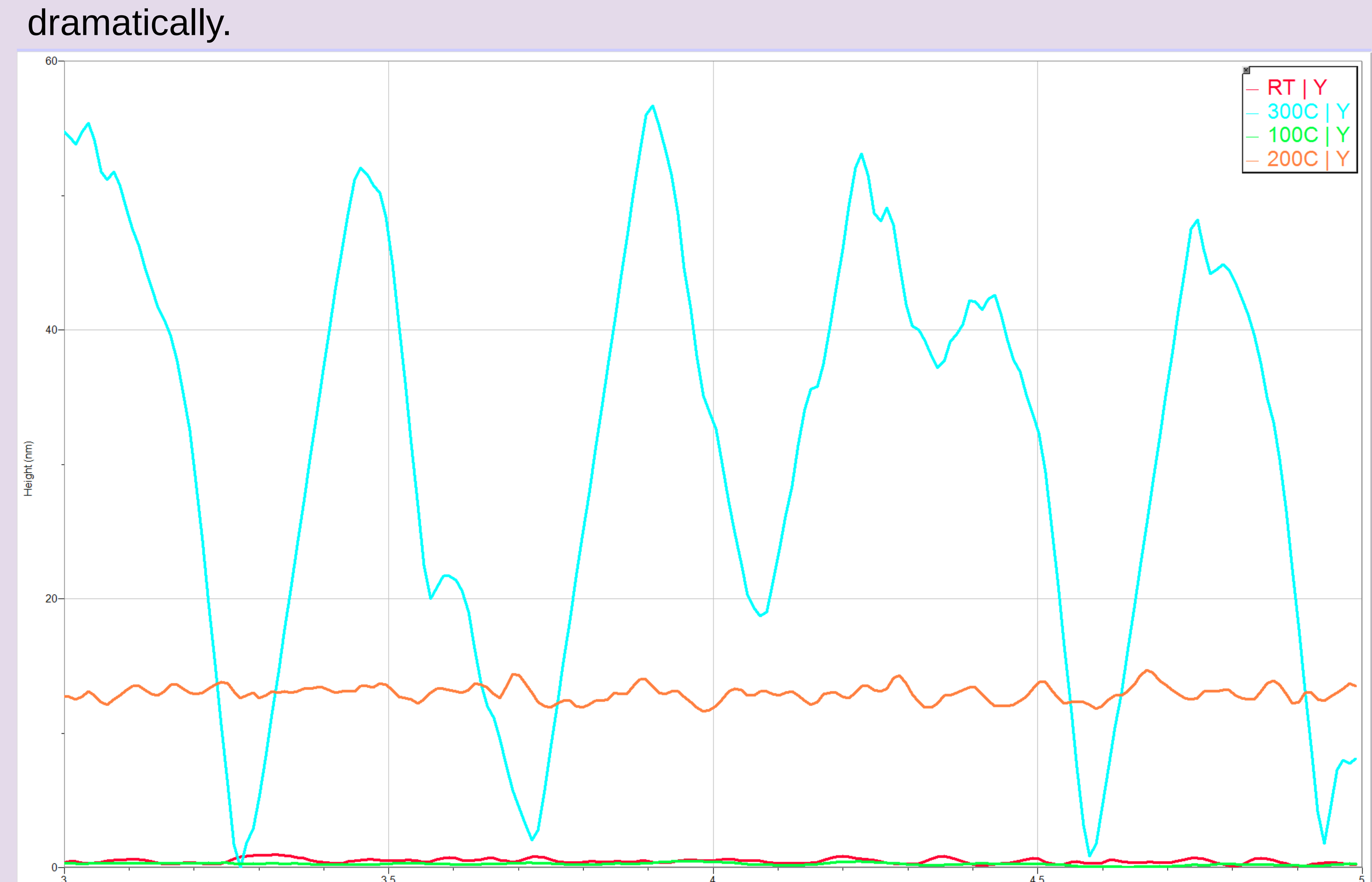

Figure 5: Line profiles taken from the topography maps above, Each peak is understood to be a clump of Au. Data shows that at 300C, the peaks of each clump of material are significantly larger than those of lower temperatures. Features seen at RT and 100C are atomic scale.

Results (Cont.)

Samples were stable in air but did show significant changes in surface roughness with annealing at 200C. From the scanned images in Figure 3, the surface roughness over a 5μm area at room temperature and 100C appears to approach atomic flatness, as the height of one Au atom is 250 pm. Differences between 100C and room temperature are on the order of experimental resolution. At 200C, the surface roughness increases to the nanoscale, and at 300C the surface roughness increases by over 30x.

Discussion

The sample used in this research showed that it is possible to control the surface roughness Au film through the process of annealing. Previous to annealing, these films were prime candidates for use in SAMs, due to their atomic scale flatness. Once annealed at 300C, this film developed several particles around 50nm, which is an optimal measurement for use in SERS.[2] Therefore, by utilizing annealing, we have developed a way for a gold film to be used for a variety of purposes that require drastically different surface roughness measurements.

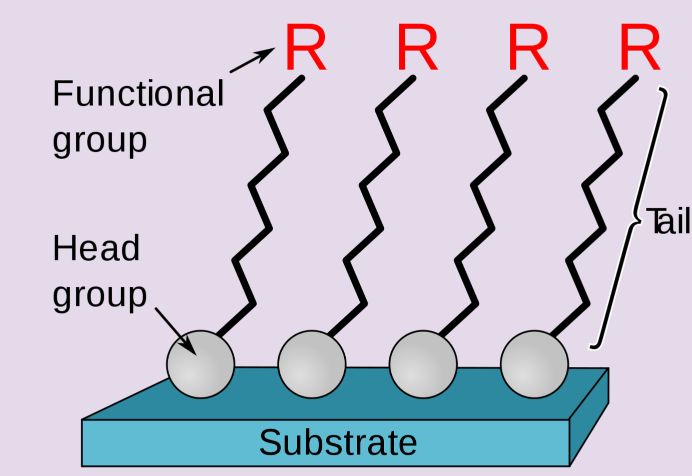

Figure 6: An image depiction of SAMs attaching to a substrate.[3] The Au film in its original state, and at 100C, is a prime candidate for use in SAMs due to its ultra-flat nature. The Head group would require a thiol to be applied in order to stick to the Au surface.

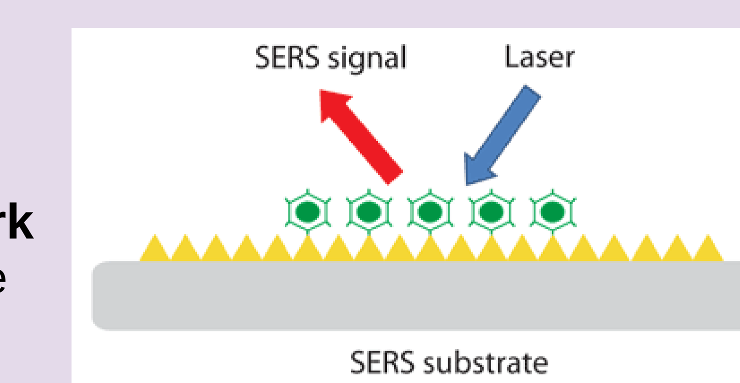

Figure 7: An image depiction of SERS utilizing a substrate, such as gold, to create a localized light field via laser excitation. With this light field, weak Raman signals are amplified.

Conclusions and Future Work

A method of controlling the surface roughness of gold films has been created using the process of annealing for use in both SAMs and SERS.

Future works include determining the temperature threshold at which the gold films will undergo the most rapid change. This work will also include time trials where the gold is left their temperatures for differing amounts of time in order to determine how this would affect the surface roughness measurements,

Literature Cited

(1) "Agilent 5500 SPM User's Guide." Agilent Technologies, Sept. 2008

(2) Hong, S., & Li, X. (2013). Optimal size of gold nanoparticles for surface-enhanced Raman spectroscopy under different conditions. *Journal of Nanomaterials*, *2013*, 1–9. https://doi.org/10.1155/2013/790323

(3) By Vladsinger - Own work, CC BY-SA 4.0, https://commons.wikimedia.org/w/index.php?curid=68470397

(4) *What is surface-enhanced Raman scattering?* Semrock. (n.d.). Retrieved July 22, 2022, from https://www.semrock.com/surface-enhanced-raman-scattering-sers.aspx

Acknowledgments:

The work was supported by Grant No: DE-SC0020334 funded by the U.S. Department of Energy and the Army Educational Outreach Program, Office of Science. J.C. was also supported by Dr. Gerald Intemann Undergraduate Research Fund.