B.Sc. (Hons) Physics Project

Thesis

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Characterisation of Nanostructured Thin Films

Keywords: surface plasmons, nanostructures, spectroscopy, metallic-blacks
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1 Introduction

- Waffle about motivation for the project
  - Metal-Black films may have application for ... something.
    - Radiometer vanes, IR detectors
    - Number of applications where high absorbance into IR is required
    - These have all been studied before though.
  - The electron spectra of metal-blacks have not yet been examined.
  - Remarkable difference between Metal-Black films (bad vacuum) and normal metal films (UHV)
    - No (detailed/satisfactory) explanation (that I can find...) for difference
  - Talk about plasmonic based computing? Moore's law? Applications to thin film solar cells?
- Specific aims of project
  1. Surface density of states / band structure of Black-Au films using TCS (The main aim)
  2. Identification of plasmonic effects in Black-Au films (?) (If they even exist!)
     - Identify plasmonic effects in Au and Ag films with Ellipsometry (this is fairly simple to do)
  3. Combination of Ellipsometry and TCS to characterise thin films (not just Black-Au)
     - Ie: How can one technique be used to support the other?
- Structure of thesis

2 Overview of Theory

Summarise the literature, refer to past research etc

2.1 Electron structure of surface

- Overview of electron spectrum properties
  - Density of states $n(E)$
  - Energy band structure $E(\boldsymbol{k})$
- Properties of surface region
  - Difference between potential of surface and bulk
    - Change between the two limits in the "near-surface" region
  - Theoretical models for the potential, 1D vs 3D
    - Simplest case is a step potential.
    - Various improvements on this model, discussed in Komolov's book.
      - Possibly adapt CQM project to model these potentials, if I get time
  - Limitations of theoretical models
    - Real surface is not a step potential
    - Adsorption of foreign particles onto the surface also plays a large role in determining the electron spectrum.
- Main reference: Komolov "Total Current Spectroscopy"
- "Solid State Physics" textbooks and "Electron Spectroscopy" textbooks

2.2 Plasmonics

I really think I should actually find plasmonic effects before writing too much about them...

- Charge density oscillations
- Surface and bulk plasmons
- Pines and Bohm
- Review article from T.W.H Oates et al about using Ellipsometry to characterise plasmonic effects

2.3 Metallic-Black Thin Films

- How they are made (bad vacuum, in air or a noble gas)
  - If made in air, there are usually tungsten oxides present (from filament). Refer to paper by Pfund.
- Structural difference between Black-Au and "Shiny" (need a better term) Au
  - Can include electron microscopy images?
  - An actual photograph of a Black-Au film? Not necessary?

- Pfund (earliest publisher, preparation and general properties)
- Louis Harris (most research in 50s and 60s)
  - L. Harris mostly did transmission spectroscopy in the far infra red (well beyond the ellipsometer and Ocean Optics spectrometer ranges)
  - The really crappy measurements I did with the Ocean Optics spectrometer seem to agree with these measurements
    - L. Harris' λ has a range of 1nm to 100μm; my measurements are only to 1μm
    - Agreement in first 1μm anyway
    - I should probably re-do those measurements with a less crappy setup, if I actually want to use them
  - Harris related the optical properties to the structure of the film (condensor strands) via the electronic properties
- Plasmonic effects - Deep R. Panjwani (honours thesis)
  - Not sure if I can use an honours thesis as a reference.
  - Concluded that surface plasmon resonance in Black-Au film on solar cells lead to increase in solar cell efficiency
  - Used simulation that modelled Black-Au film as spherical balls to show E field increased by plasmon resonance
    - Was this model appropriate? Black-Au is more "smoke" or "strand" like according to other references. Images also do not show "blob" like structure.
  - Need to read this reference more thoroughly

3 Experimental Techniques

3.1 Preparation of samples

- Black-Au - 1e-2 mbar vacuum
- "Shiny" - 1e-6 / 1e-7
- Current of 3.5A through W wire filament spot welded onto Ta strips in turn spot welded to Mo posts
- Voltage through filament is 1 V; quote the power?
- Filament isotropically coats sample with desired material.
- Possibly get a curve of Au thickness estimated with Ellipsometry vs exposure time?
  - Probably too much work and too unreliable
  - Maybe do it, but only use 2/3 data points
  - Low priority

3.2 Total Current Spectroscopy

- Overview of technique
  - Low energy beam of electrons incident on sample
  - Measure slope of resulting I-V curve
  - Relate to density of states and electron band structure (Komolov chapter 3.2)
- Description of apparatus
  - Electron gun and filament
  - Electron gun control box
  - ADC/DAC control box and data processing
- Photographs vs Diagrams
  - Prefer diagrams to photographs
  - Especially for the ADC/DAC control box circuit. Because it looks like a horrible mess.

3.3 Ellipsometry and Transmission Spectroscopy

- Overview of techniques
- Description of apparatus (use VASE manual)
- Ocean Optics spectrometer? Usable?
- Application of Ellipsometry to finding plasmonic effects
  - Surface plasmons = E oscillation parallel to surface $\implies$ only p component of light excites plasmons

4 Experimental Results and Discussion

4.1 TCS Measurements

- TCS for Si
- TCS for Si + Au
- TCS for Si + Black-Au
- Affect of preparation pressure on TCS for Si + Black-Au
- Repeat for Si + Ag and Si + Black-Ag (?)

4.2 Ellipsometric Measurements

- Ellipsometry to estimate thickness of SiO2 layer on Si
- Estimate thickness of Au/Ag on Si+SiO2
- Ellipsometric measurements of Si+Black-Au/Ag
  - Modelling procedures to characterise Black-Au/Ag
- Ellipsometric measurements of Glass+Black-Au/Ag (?)
- Transmission spectra of Glass+Black-Au/Ag from earlier in year (?)

5 Achievements

- Deposition of thin films of Au and Black-Au in vacuum chamber
- Ellipsometric and spectroscopic measurements on these films
- Repurpose vacuum chamber for sample preparation and TCS experiments
- Designed and built electronics for TCS experiments
  - Electron gun control box
  - ADC/DAC box
- Wrote software for data aquisition and data processing

6 General notes

6.1 TCS

- Optimise setup of gun
  - Emission current. How much does it vary, why does it vary.
  - Why does Is/Ie curve shift with successive sweeps? Does sweep modify sample's surface?
  - Is sample holder acceptable? Are ceramic washers accumulating charge?
  - How do I tell when the setup is optimised... "The setup was optimised by looking for an S curve". Very scientific.
  - The gun was focused on the phosphor screen... and then I turned it around, changing the distance from the gun to the sample. Brilliant.
- Obtain TCS spectra for Si that compares well with literature
  - How to relate TCS spectrum to $n(E)$ and $E(\boldsymbol{k})$
- Prepare Au films, obtain TCS spectra that compares with literature
- Obtain TCS spectra of Black-Au films
- Use results to compare properties of films with results from other methods in the literature

- Uncertainties
  - Oscilloscope measurements of inputs to ADC channels under controlled conditions
    * Expected values are +/-3mV due to ADC channel, +/-300mV due to 610B, +/-1mV due to 602
    * 610B and 602 will probably be worse because they are ancient
    * There is about 200mV of noise between the GND of the ADC box and the electron control box.
    * How to reduce ground loops? Not much I can do. Rack is now also grounded to water pipe, but this doesn't seem to make a difference.
  - Stupid 50Hz AC noise... how to reduce with filters and/or averaging
- Create circuit diagrams for Electron gun circuit
- Create circuit diagrams for ADC/DAC box
  - Simulate behaviour of circuit
  - Use of instrumentation amplifier on ADC5 to make off-ground measurements
  - Use of low pass filter on ADC5
- Include references to all datasheets, etc
- Vacuum chamber
  - Base pressure with rotary pump? Was 1e-3 after 30 minutes at start of year, but probably introduced leaks since then
  - Lowest pressure achieved with turbo pump is 1.1e-7 mbar as of 25/07.
  - Viton gaskets on some seals. Copper on other.
  - Flanges:
    1. View window (large, view of sample & sputtering filaments)
    2. Rotation manipulator & sample mount
    3. Pump inlet
    4. Filament flanges 1 (used earlier in year, not anymore) and 2
    5. Inlet with leak valve (for introducing gases into chamber)
    6. Vent valve on turbo pump
    7. Electron gun flange
    8. View window (small, view of back of electron gun)

References