

X-Ray Reflectivity Analysis of Multilayer Structures

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Abstract

The purpose of this study is to characterize the nanometer-scale structure of an Al₂O₃-W-Ge multilayer utilizing x-ray reflectivity. Attributes such as layer thickness, density, surface, and interface roughness are determined and explained. The reasons for using this particular multilayer are also reviewed, as well as the benefits to science and the semiconductor industry.

Introduction

As semiconductor electronic devices become increasingly smaller, the need to be able to study the properties of the layers and interfaces of these devices on the atomic scale arises. Since devices have become so miniaturized that the wavelength of light is too large to use as a detector,¹ another method of examination must be used to understand how atoms are arranged within an ultrathin-film multilayer structure. This research project used x-ray reflectivity (XRR), which uses reflected x-rays and theoretical models (explained in detail later) to determine physical properties of the multilayer structure. XRR is an ideal technique in the study of thin films for many reasons. The wavelength of x-rays

is on the same order of size as individual atoms, and materials can be studied in a nondestructive manner.^{1,2}

Many characteristics of a multilayer can be determined via XRR including thickness, density, and surface and interface roughness. Determining these characteristics can greatly affect the future performance of electronic devices.¹

The purpose of this study is to use XRR to determine the surface characteristics of an Al₂O₃-W-Ge multilayer film. Germanium is a common material used in the semiconductor industry and is slowly gaining popularity due to its ability to withstand higher voltages. It has been found that germanium *with* layers of thin films could last much longer and withstand even greater voltages. For this reason, advancing knowledge of the properties of germanium with various thin films is valuable.^{1,3}

Background

The development and availability of x-ray tools capable of characterization at the atomic scale has greatly increased understanding of surface and thin-film science in recent decades.² Basic geometry and knowledge of elemental electron densities have allowed scientists to develop equations that predict the composition of thin films. The Parratt recursion formula is one of these equations.⁴ The formula predicts the reflectivity of a multilayer as a function of angle of incidence based upon the knowledge that x-rays reflect and refract differently off materials with different electron densities (Figure 1).

The index of refraction of an x-ray region is $n = 1 - \delta - i\beta$, where δ is a known value proportional to electron density and β is

associated with absorption. These values, along with a known angle of incidence θ , are used to calculate reflectivity intensities for a range of θ , thus creating a simulation curve used later to fit experimental data.

Approach

The Al₂O₃-W-Ge multilayer sample was grown by atomic layer deposition (ALD) at Argonne National Laboratory by Jeffery Elam.

Also used in this study was a four-circle diffractometer with a 12 kW rotating anode x-ray source operated at 40 kV and 100 mA. Figure 2 shows a schematic of this setup. The specular reflected x-ray intensities are collected by a NaI detector for low angles of incidence and plotted on an intensity curve. Corrections are made for background radiation, and the data is loaded into a computer for analysis.

In order to analyze this data, a simulation curve (an assumed ideal situation) was generated using the Parratt formula.⁵ Figure 3 shows the simulation of a blank Ge sample. A program was developed that allows the user to enter different property values, such as interfacial roughness and layer thickness, for a particular sample.

Normally, the composition of a sample is known to a certain extent. This provides a starting point for simulation generation. For example, in this case the sample was known to contain layers of Al₂O₃ and tungsten on a germanium substrate. Thus, known δ and β values for these materials are entered into the program, and simulations are compared to experimental reflectivity data until the two are comparable to each other.

X-Ray Reflectivity Analysis of Multilayer Structures (continued)

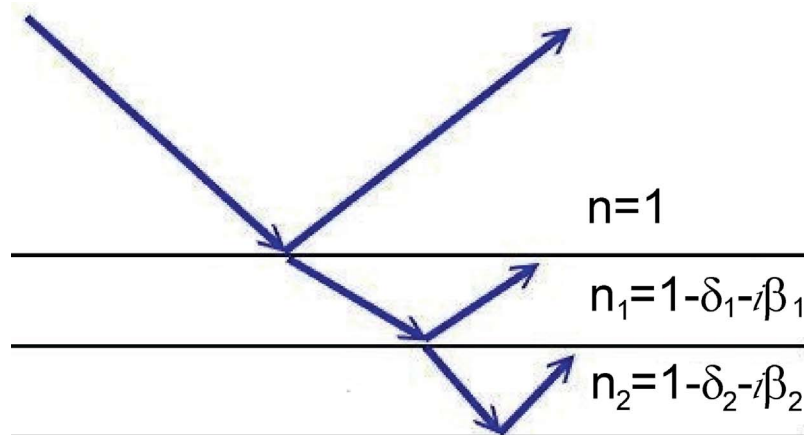


Figure 1: Diagram of X-rays reflecting and refracting from each interface of a multilayer.

NU X-ray lab monochromatic 12 kW Diffractometer

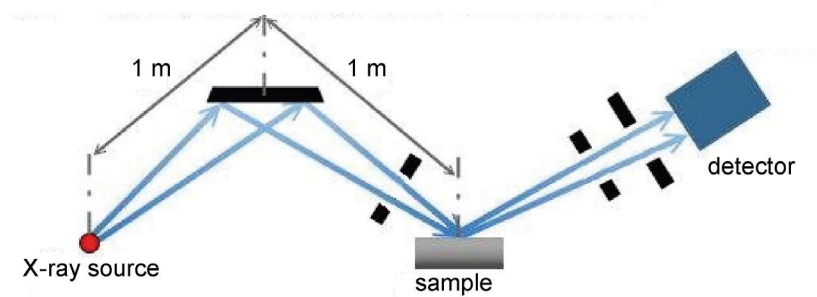


Figure 2: A diagram of the 12 kW Diffractometer.

Results and Discussion

After following the above process the assumption that the multilayer was composed of a layer of tungsten under a layer of Al_2O_3 on a germanium substrate was found to be incorrect. The most accurate fit for the experimental data occurred with a simulation of Al_2O_3 under tungsten (Figure 4).

It was also found that instead of well-defined layers with sharp interfaces, a certain amount of diffusion had occurred between the layers. As a result, the simulation curve had to be altered by adding 20 transition layers between the vacuum layer and tungsten and one layer between the Al_2O_3 and tungsten. The delta and beta values of the 20 transition layers were determined by a normalized form of the Gaussian function (error function) with values between the Al_2O_3 and tungsten values. This, in effect, accounted for the diffusion at the interface of layers.

Conclusions

XRR proved to be an ideal tool for the study of the Al_2O_3 -W-Ge sample. The Parratt Recursion Formulation provided a simulation that was used to fit experimental reflectivity data to characterize the multilayer. It was found that a certain amount of interfacial diffusion had occurred between layers, and an error function was used to simulate the actual density profile. Layer thickness of 88 Å for tungsten and 100 Å for Al_2O_3 were found. The interface thicknesses used were 10 Å for the vacuum-W interface using the error function generated δ and β values, and 10 Å for the W- Al_2O_3 interface. These interfaces were found

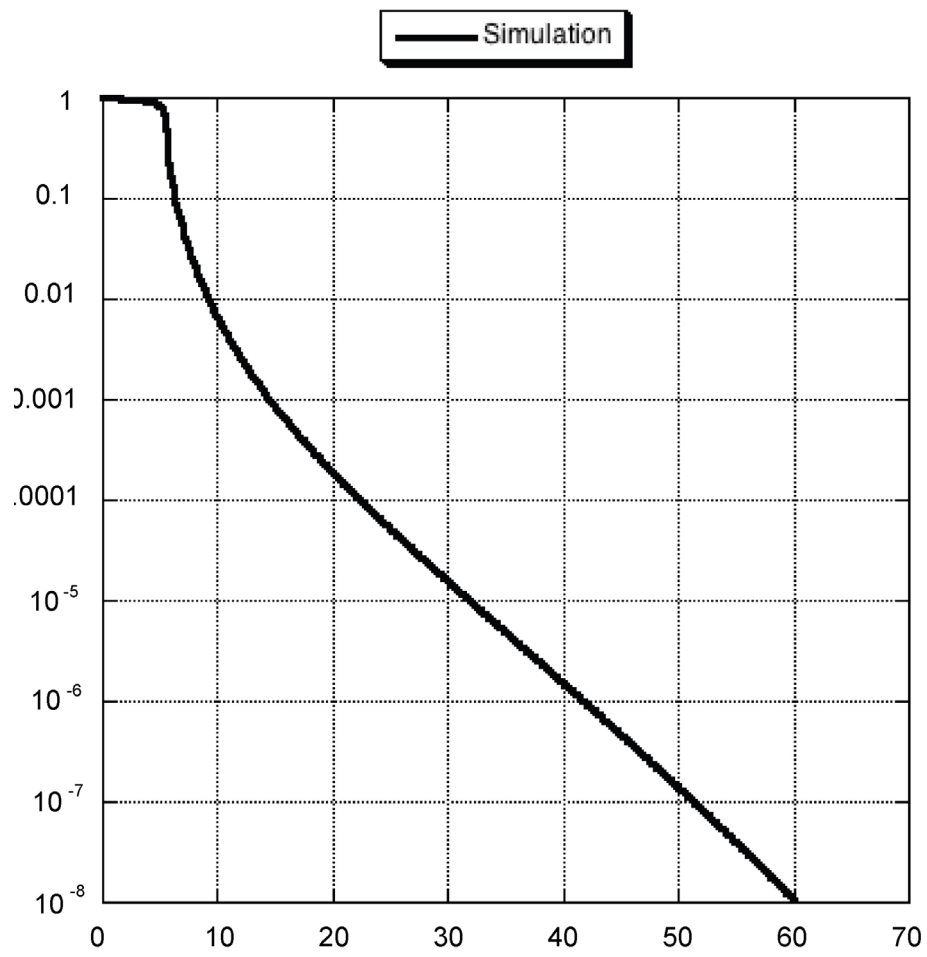


Figure 3: Simulation of blank Ge sample.

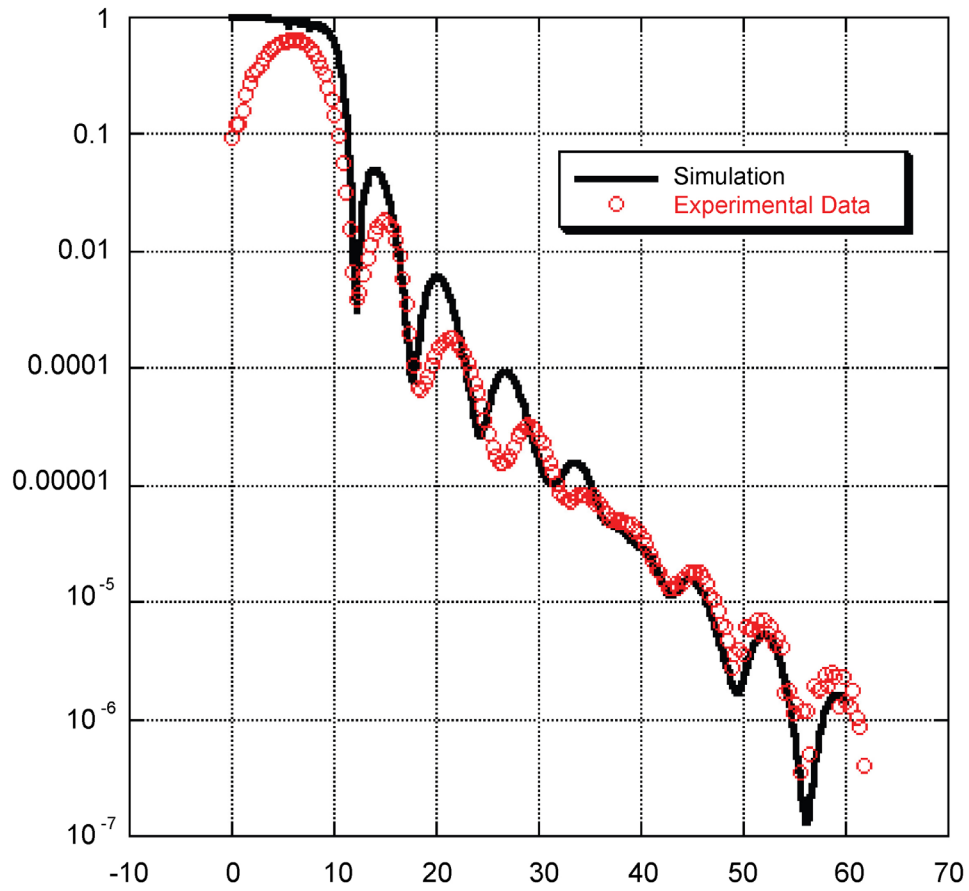


Figure 4: Al_2O_3 simulation and experimental data fit.

to have a roughness of 4 \AA . A model showing these findings is shown in Figure 5.

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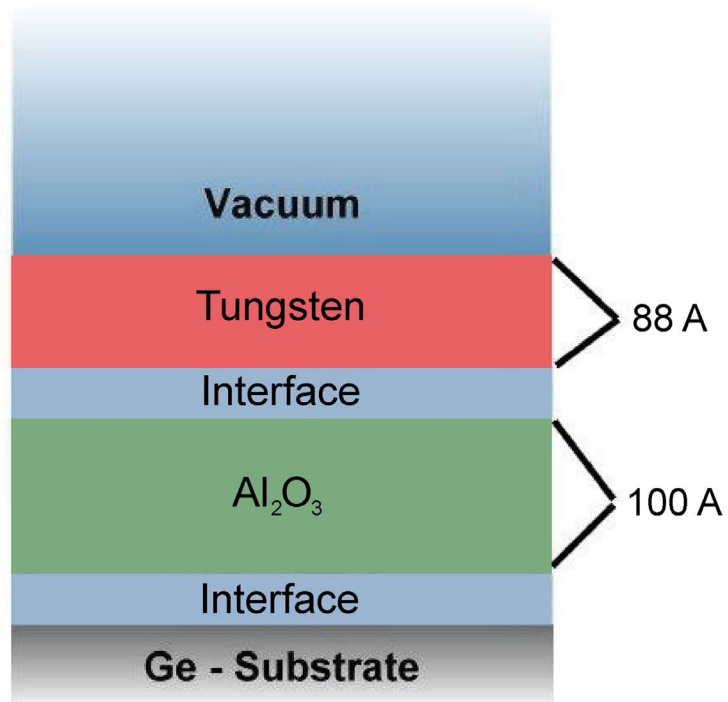


Figure 5: Diagram of the germanium multilayer.