X-ray photoelectron diffraction analysis of the epitaxial growth of titanium on the (110) surface of aluminum
by Volker Krasemann

A thesis submitted in partial fulfillment of the requirements of the degree of Master of Science in Physics
Montana State University
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Abstract:
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The Ti overlayer exhibits no long-range order for coverages of eight monolayers but still shows short-range order according to XPD.

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Master of Science

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Physics

MONTANA STATE UNIVERSITY-BOZEMAN
Bozeman, Montana

June, 1996

N378
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of a thesis submitted by

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This thesis has been read by each member of the thesis committee, and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for submission to the College of Graduate Studies.

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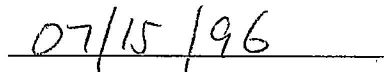
Date 

I wish to dedicate this thesis to

David Letterman

ACKNOWLEDGMENTS

I wish to acknowledge Dr. Richard J. Smith to whom I am very thankful for his advice and guidance and for all his support not only in regards to this thesis. It was a great pleasure to be a student under Dr. Smith's supervision. I am thankful to the Physics Department, especially Dr. J. C. Hermanson for the help and financial support I received during my graduate studies at Montana State University. Also I would like to thank N. R. Shivaparan for all his collaboration and continuous assistance. My thanks go to N. Williams and E. Andersen for their technical support and assistance.

This work was supported a grant by the National Science Foundation – Grant No. NSF Grant DMR - 9409205.

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ABSTRACT

The growth of Ti films on the Al(110) surface at room temperature has been studied using X-ray photoelectron diffraction (XPD) and low energy electron diffraction (LEED). The overlayer coverages were determined with RBS. The area under the Al $2p$ and the Ti $2p\frac{1}{2}$ and $2p\frac{3}{2}$ photoelectron peaks was recorded in dependence of the polar angle.

Both LEED and XPD support an epitaxial growth of Ti on Al in form of islands. The structure of the Ti overlayer is pseudomorphic *fcc*. A distortion of the overlayer was measured with LEED. The interlayer distance of the first two Ti overlayer is smaller than that for the bulk Al as can be concluded from a shift of the forward-scattering peak in the diffraction pattern for Ti.

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CHAPTER 1

Introduction

For the development of state-of-the-art electronic device technology the growth of ultrathin epitaxial films on single-crystal substrates becomes more and more important. The growth of epitaxial films and the formation of metastable phases of the overlayer material lead to new directions in the search for new materials. These phases are occasionally the result of lattice mismatches which lead to a structure that is different from the equilibrium one at room temperature.

For example, face-centered cubic (*fcc*) Co films grow epitaxially on Cu(001) substrates [1], [2]. Both Al and Au can in principle grow epitaxially on GaAs(001) by nucleating an *fcc* overlayer in which the surface unit mesh of the overlayer is rotated by 45° with respect to that of the substrate [4].

Saleh and coworkers as well as Jona et.al. previously studied the growth of thin Ti films on Al(110) and Al(100) surfaces [6], [21]. Thin Ti films are of considerable interest for their application as diffusion barriers in semiconductors. The main conclusion of the work by Saleh et.al. was that Ti deposition on Al(110) results in the formation of an epitaxial Ti overlayer. A critical thickness of this metastable overlayer is reached for approximately 5 monolayers (ML) as reported by Saleh [6]. Saleh showed that the Ti adopts the surface symmetry and lattice constant of the underlying Al and forms an *fcc* overlayer. This is particularly interesting since the *fcc* structure of Ti does not occur in the equilibrium phase diagram of Ti at any temperature [3]. Saleh further concludes using the VEGAS-simulation of shadowing of the Al atoms by overlaying Ti atoms that the Ti does not form islands.

The situation for coverages exceeding 5 ML is not clear however. Two different growth modes are suggested: Island formation for Ti coverages above 5 ML and interdiffusion of Ti and Al atoms at the interface.

The present study examines the growth of Ti films on Al(110) surfaces using X-ray photoelectron diffraction (XPD) to supplement the results by Saleh. XPD is well suited to the study of the structure of very thin transition-metal films, because XPD diffraction patterns are collected from each chemical species in the sample independently. These patterns arise primarily from the local structure of a few atomic shells surrounding the emitting atom, so that long-range order is not necessary.

Through XPD we hope to gain an even better understanding of the epitaxial growth of Ti on the Al(110) surface. Ti films of up to 9 ML were deposited on the Al single crystal and the atomic structure of both the overlayer and the substrate were studied *in situ*. XPD measurements were done for clean Al and at coverages of 1.8 ML, 2.5 ML, 4.8 ML and 8.9 ML of Ti. A separate set of LEED experiments were carried out at 1.9 ML, 5.5 ML, and 8.1 ML coverages of Ti.

CHAPTER 2

Experimental Setup

A very clean surface free of contaminants is necessary for conducting experiments that involve surface phenomena. The experiments were therefore carried out in an ultrahigh vacuum chamber.

In our study the chamber is connected to a VAN DE GRAAF accelerator allowing the use of RUTHERFORD backscattering to determine the thickness of the deposited layer. Furthermore the chamber is equipped with an X-ray photoelectron spectroscopy system to do X-ray photoelectron diffraction and with reverse view low energy electron diffraction (LEED) optics for LEED analysis.

The present chapter gives an overview of the system and the experimental setup.

The Ultrahigh Vacuum Chamber

All experiments were performed in an ultrahigh vacuum (UHV) chamber. The use of such a vacuum chamber is crucial for the investigation of surface phenomena because it minimizes contamination of the sample. At pressures below 10^{-10} *Torr* (UHV environment) it takes several hours for the sample surface to become contaminated by the adsorption of residual gas atoms. In addition, UHV environments are beneficial for all ion- and electron-based surface analytical techniques, because the mean-free-path of electrons and ions is very short at atmospheric pressure.

Schematic views of the chamber and its components are shown in Figures 1 and 2. The UHV chamber is made of stainless steel and the inside is lined with *mu metal*

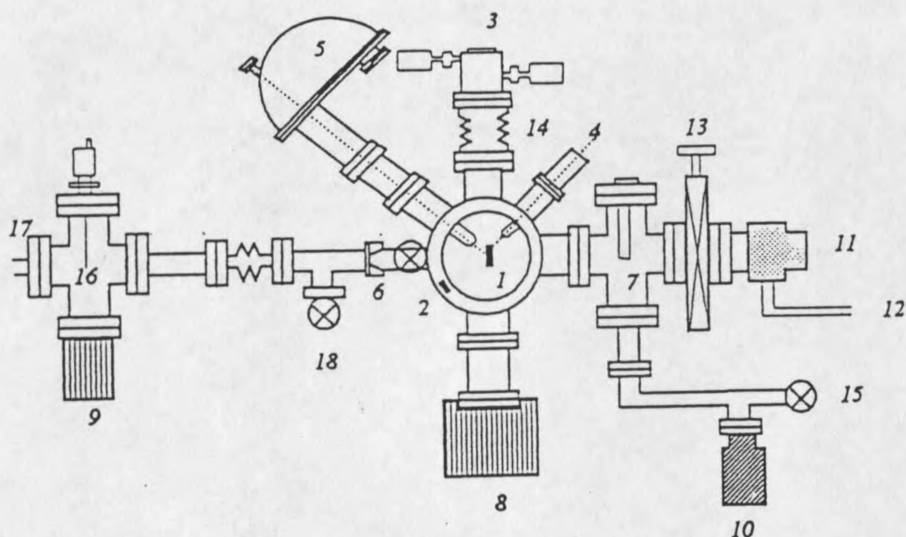

Figure 1: Schematic side view of the chamber

1. Sample
2. Detector
3. Goniometer
4. Sputter Gun
5. Hemispherical Analyzer
6. Collimator Slits
7. Ti sublimation pump
8. Ion Pump
9. Ion Pump
10. Sorption Pump
11. Turbo Pump
12. Foreline Pump
13. Gate Valve
14. Bellows
15. Leak Valve
16. Beam Blocker
17. Ion Beam Line
18. Valves

to provide magnetic shielding.

The chamber is equipped with a turbomolecular pump (*Balzers TPU 160*), an ion getter pump (*Varian*), and a titanium sublimation pump. To reach a regime in which the UHV pumps are operable a roughing pump with a foreline trap is connected to the chamber through the turbomolecular pump. An ionization gage and a quadrupole residual gas analyzer (*Dycor Electronics M100M*) are mounted to the chamber to monitor the pressure as well as the gas composition in the chamber. A pressure in the low 10^{-10} $Torr$ range is obtained by baking the chamber at 150^0C for about 48