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CARLETON UNIVERSITY

A Study of Collimated Sputtering for VLSI Metallization

by

Tomas Janacek

A thesis submitted to the Faculty of Graduate Studies and Research
in a partial fulfillment of the requirements for the degree of Master
of Engineering.

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research for acceptance, a thesis entitled *A Study of Collimated Sputtering for VLSI Metallization* submitted by Tomas Janacek in partial fulfillment of the requirements for the degree of Master of Engineering.

Dr. T.J. Smy, Thesis Supervisor

Prof. J.S. Wight, Chairman
Department of Electronics

July 20, 1993

Abstract

This thesis is a part of the joint research project between Carleton University, Ottawa, University of Alberta, Edmonton, and Varian Associates, Palo Alto, CA. Its focus is on the properties of thin metal films deposited by *collimated sputtering*. Due to Varian's requirements, the bulk of the experimental work was done with titanium thin films. All experiments described in this thesis were carried out in the Alberta Microelectronic Centre, Edmonton, unless indicated otherwise.

This thesis studies the basic properties of the collimated sputtering technique and its impact on the thin film properties and microstructure. It has been shown that a substantially higher step coverage can be achieved. The main effect of collimation seems to be the entrapment of particles with high lateral velocities, i.e., a collimator acts as a *filter* for particles arriving at oblique angles. Most of the experimental data supports this simple explanation. Moreover, collimated sputtering also changes a film microstructure and increases the level of the compressive stress.

A software simulation package SIMSPUD/SIMBAD was used in order to simulate some of the experimental results described in this thesis. Although this software package has successfully been used elsewhere to simulate microstructure of thin films deposited by evaporation, magnetron sputtering, bias sputtering, CVD..., this thesis reports the first successful application of the SIMSPUD/SIMBAD package for films deposited by collimated sputtering. The sticking coefficient issue was studied in order to properly simulate our experimental results. The value of the sticking coefficient has been found very close to unity for all materials investigated (titanium, aluminum, copper, and tungsten).

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Chapter 1

Introduction

The postwar development of mankind has been to a large extent determined by two great inventions made during 1940's – the introduction of the first transistor by Jack Kilby (1947) [1] and the development of electronic computers. One of the first computers, ENIAC (Electronic Numerical Integrator And Computer), consisted of 18,000 tubes and 1,500 relays, its weight was 30 tons and power input 140 kW [2]. The comparison with today's computers shows quite convincingly the development achieved in this area. A significant part of this progress was due to the diminishing dimensions of the transistor, which shrank from centimeters to microns. This in turn increased the speed and memory capabilities of computers. The relationship between the transistor feature size and memory capability is shown in the table 1.1.

Seemingly endless growth in the capability and speed of integrated circuits (IC) enabled the increase in the number of microelectronic devices packed onto a single silicon “chip” and a respective size reduction of a single device. The number of devices per chip has grown since the period of Small Scale Integration (SSI), from approximately 100 components per chip up to today's VLSI (Ultra Large Scale Integration) levels, where more than a million devices can be found on a single chip.

The new *Pentium* chip, recently introduced by Intel, contains about 3.1 million devices on a chip, and performs 112 millions operations per second [3]. *Pentium*'s feature size is $0.8\ \mu\text{m}$, but a $0.56\ \mu\text{m}$ future version is being currently designed.

<i>Feature size (μm)</i>	<i>Year</i>	<i>Capacity (bits)</i>	<i>Integration level</i>
10	1970	1 K	LSI
8	1973	4 K	
6	1975	8 K	
4.5	1976	16 K	
3	1979	64 K	
2	1982	256 K	VLSI
1.2	1986	1 M	
0.7	1989	4 M	
0.5	1992	16 M	ULSI

Table 1.1: Decrease in standard feature size within the past two decades for DRAM memory circuits. [4, 5]

As a recently published Semiconductor Industry Association forecast [6] shows, it is widely believed that this trend will continue (please see table 1.2), although that will require a substantial improvement in the manufacturing technology. One of the areas of VLSI technology where this is especially true is IC metallization.

<i>Feature size (μm)</i>	<i>Year</i>	<i>DRAM capacity (bits)</i>	<i>Chip size (mm^2)</i>	<i>Gates per chip</i>	<i>Interconnect levels</i>
0.35	1995	64 M	200	800 K	4-5
0.25	1998	256 M	320	2 M	5
0.18	2001	1 G	500	5 M	5-6
0.12	2004	4 G	700	10 M	6
0.10	2004	16 G	1000	20 M	6-7

Table 1.2: Forecast of the future development of DRAM memory circuits. [6]

The main topic of this thesis is a study of *collimated sputtering*, a recent modification of the most popular VLSI metallization technique – sputtering. Chapter one serves as an introduction to the basic principles and terminology of IC metallization. Chapter two offers a detailed description of the sputtering process, while chapter three investigates the basic properties of collimated sputtering. Chapter four describes some interesting aspects of the interaction of the sputtered atoms with the surface, which are important for the computer simulation of our experimental results. Chapter five deals with the internal stress in thin films and how it is affected by collimation. Finally, chapter six summarizes the results and suggests research directions that could be pursued in the future.

1.1 Metallization

The term “metallization” has two accepted meanings – a *system level* metallization, which deals with chip-to-chip interconnections, and a *device level* metallization, which is focused on the deposition of low resistance reliable contacts/interconnections between devices.

Since this thesis deals only with the device level metallization, that is what will be understood whenever the expression “metallization” is used.

The metallization step in silicon device and IC processing is one of the last ones and involves the process of the deposition and etching of thin metal lines onto a wafer. It is a rather critical and important processing step. Up to 80% of failures in the VLSI circuits may be attributed to the metallization technology [7], which certainly leaves space for future research and improvement. The choice of the metal itself was

originally quite simple - *aluminum* was the material of choice mainly because of its extremely low resistivity (typical values of Al resistivity would be about 2.7 - 3.0 $\mu\Omega cm$ at room temperature for a 1 μm thick film) and was also easy to deposit [8]. A good overview of the recent status of VLSI metallization can be found in [9], while general aspects of metallization are described in [10, 11].

1.1.1 Requirements for metallization

The main requirements for the metal lines would be a low resistivity, low cost, easy fabrication (deposition, etching, lithography...), stability, conformal step and bottom coverage over VLSI topography (please see fig 1.1), low stress, good electromigration and corrosion resistance. A more detailed description of these requirements can be found in subsequent sections.

1.1.2 Multilevel metallization

The use of multiple layers of metals separated by dielectrics (see fig. 1.2) became a necessity by the end of 1970's, when packing density began to reach a level, where almost all of the chip area would have been covered by metal interconnection lines. This would have caused a serious manufacturing problem as well as a substantial increase in RC delay times.

An obvious solution was the move to the third dimension, although it brought along a need for a substantially improved manufacturing technology. The first two-level MOS IC appeared in the beginning of 1980's [12], circuits using three-level metallization appeared in the mid-1980's [13] and today, four-level metallization is frequently used [14, 15]. One of the problems that multilevel metallization has made worse, was the

Figure 1.1: Definition of aspect ratio and bottom/step coverages.

possibility of a circuit failure due to the instability of one of many material interfaces in the circuit. Generally, multilevel metallization involves deposition and etching of sandwiched layers of conductors and dielectrics. Interconnection between metal lines is provided by etched *via*-holes, which have increasingly higher and higher aspect ratios caused by a decreasing feature size of standard devices.

1.2 Deposition techniques

The task of the deposition of thin metal lines can be achieved via two different principles – *Physical Vapour Deposition (PVD)*, (i.e. evaporation, sputtering) and *Chemical Vapour Deposition (CVD)*. This thesis deals only with PVD, but a brief description of CVD will be given because of its increasing importance for refractory metals [16].

Figure 1.2: Illustration of two-level metallization structure. [10, 17]

1.2.1 Evaporation

The physical principle of this technique is quite simple – it involves the heating of the metal which is to be deposited in a vacuum environment until it evaporates and a portion of the evaporated atoms will cover a sample placed in the proximity of the source. More on this technique can be found in [18]-[21]. Evaporation was the method of choice in the past. A photograph of an evaporated titanium film is shown in fig. 2.4.

1.2.2 Sputtering

This is the most popular and widespread deposition technique in microelectronic circuit manufacturing, but its applications can be found literally everywhere - from biological and medical applications, to textile production or military applications (i.e. USAF Bomber Stealth 2). One of the main reasons for the broad area of applications of sputtering is its universality - almost all solid materials can be sputter-deposited without any serious problems. However, because of its vacuum requirements, sputtering is also a quite expensive deposition technique. Fig. 2.1 shows a schematic cross-section of a current sputtering system.

Despite the fact, that mechanism of sputtering was discovered some 140 years ago by W.R. Grove, who studied the processes in fluorescent light tubes [22], its widespread use is relatively recent. One of the reasons for this “temporary oblivion” may have been the lack of a sufficient vacuum technology. Currently, sputtering is the most important deposition technique in VLSI metallization. A detailed description of a sputtering process will be given in the next chapter.

1.2.3 CVD

CVD processes are generally gas phase chemical processes [23] wherein reaction chemistry and deposition conditions largely determine the film growth and its microstructure. CVD requires gaseous reactants, which are not always available for all materials (i.e., alloys). Basic parameters are the reaction rate and the transport of reactants onto substrate (diffusion).

The reason why CVD processes are so attractive for microelectronic manufacturing is due to the fact that they offer conformal step coverage and also have a high through-

put. One of the most important modifications of basic CVD is Low Pressure CVD (*LPCVD*) [24], but so far it remains limited to refractory metals, and even there its results are lagging behind comparable results achieved by sputtering – mainly because of the film contamination [25]. CVD processes also tend to be dangerous and quite expensive.

1.2.4 Current problems

Continuous shrinking of device features (please see tables 1.1 and 1.2) leads to a proportional increase in the aspect ratio of *via* contacts (oxide thickness is held constant because of the RC delay). This trend, however, is causing many difficulties. From the point of view of metallization, it is becoming increasingly difficult to deposit metal conformally over complicated VLSI topography (please see fig. 1.2). The biggest problems (please see fig. 3.1) are caused by low bottom and sidewall coverage which can cause premature diffusion barrier failure, lower film density (and thus higher resistivity) at *via* sidewalls, and film discontinuity at the *via* corners.

Ideal deposition process should be fast, inexpensive, universal, reproducible and provide conformal step coverage over any topography.

1.3 Computer simulations

Because all deposition processes involve complex multi-parameter physical and/or chemical interactions, any reliable computer simulation tools are very useful for their optimization. However, the same process complexity makes it very difficult to develop such a program. A very good overview of available software simulation packages, as well as their underlying principles can be found in [11, 17].

A brief description of a Monte-Carlo simulation package developed by M. Brett, T. Smy and S. Dew will be given here [26]-[31]. This software will be used in order to simulate some of the experimental results described in this thesis. Simulation of a sputter-deposition process is accomplished in two stages – a vapour transport simulator SIMSPUD and a film growth simulator SIMBAD (see fig. 1.1).

1.3.1 SIMSPUD

The first stage of the software simulator package is a vapour transport model SIMSPUD (**S**imulation of **S**putter **D**istributions), which simulates the three dimensional path of an individual atom from its emission from the target, through the working gas, until its arrival at the substrate plane.

The position, angle, and energy of each atom are tracked in order to collect statistics on the corresponding sputter distributions. To account for gas scattering, the model employs an energy-dependent collision cross-section with a hard sphere scattering model. For each particular set of the sputtering parameters, such as the substrate-target distance, target material, target erosion profile, deposition pressure, working gas etc., SIMSPUD can generate the corresponding angular distribution of the sputtered flux arriving at the substrate.

This angular distribution is then used in the second stage model, SIMBAD, to predict the film profile and microstructure over topography.

1.3.2 SIMBAD

SIMBAD, which stands for the **S**imulation by **B**allistic **D**eposition, is a two dimensional model which simulates the microstructure and profile of thin films deposited

over topography. The model employs a ballistic deposition algorithm and represents a film as a sequential aggregation of $10^4 - 10^5$ identical hard discs.

SIMSPUD - SIMBAD package

Figure 1.3: Schematic depiction of simulation software.

These discs are generated just above the substrate with the appropriate angular distribution¹ predicted by SIMSPUD. Linear trajectories are followed until the disc

¹SIMBAD uses a default cosine angular distribution, unless a user-specified one is supplied (i.e., from SIMSPUD).

hits the substrate which is initialized to the appropriate topography by a series of straight line segments. The disc is then allowed to diffuse over the film surface to a nearby cradle site which is chosen to minimize local surface curvature. This model incorporates the effects of local and topographical shadowing and limited surface diffusion which are the fundamental sources of the columnar microstructure and which lead to the development of the specific film profile.

1.4 Diffusion barriers

The concept of diffusion barriers dates back to the 1930's [32]. The main goal of the introduction of a diffusion barrier into VLSI and ULSI technology is to physically separate two adjacent layers whose contact would be otherwise unstable and could lead to a circuit failure. This failure can be caused either by a chemical reaction, or by interdiffusion (which is always present because most of the interfaces are not in thermodynamical equilibrium). The illustration of such a barrier is shown in the fig. 1.4. A general description of diffusion barriers can be found in [33, 34, 35].

Naturally, the mere presence of the diffusion barrier will not remove the cause of the chemical reaction, nor of the interdiffusion, and so it will not prevent the ultimate failure. However, this failure can be significantly delayed by a careful choice of a diffusion barrier.

Here is the list of properties of the ideal diffusion barrier X which blocks the mass transport between two different layers A and B [36]:

- negligible transport rate of A and B across X
- small loss rate of X into A and B

Figure 1.4: The illustration of the hypothetical diffusion barrier X.

- X should be thermodynamically stable with respect to A and B
- X should have an excellent adhesion to A and B
- X should not increase contact resistance
- X should have no stress and be stress resistant
- X should be deposited by IC compatible techniques
- X should have a high thermal and electrical conductivity

It is, naturally, impossible to find a material that would meet all the abovementioned conditions. There are, however, some materials that have most of the desired properties outlined above.

1.4.1 Common diffusion barriers

Although there are many different materials used as diffusion barriers, such as silicides, refractory metals, refractory metal compounds, the most popular diffusion barriers are TiN and TiW [37].

TiW

This is an alloy of two refractory metals [38]. The most typical composition is 10 wt % Ti (which amounts to 30 at %). Ti is easily soluble in W, while W is not soluble in Ti. Titanium is thus in the solid solution, distributed along the grain boundaries. This type of diffusion barrier is also called a *stuffed barrier*. Properties of TiW barriers are described elsewhere [39, 40].

TiN

TiN is perhaps the most common diffusion barrier² currently used in VLSI metallization [41, 42]. It can exist in two forms – at low concentration N exists in solid solution with Ti, while at the higher N concentration TiN and Ti₂N co-exist. The quality of the TiN barrier is sensitive to the film stoichiometry, which may be a problem when TiN is deposited by reactive sputtering over complex VLSI topography [43].

1.4.2 Titanium

Titanium is one of the most important materials for VLSI metallization. Usually, a very thin titanium layer (around 50-100 nm) is deposited as a starting point for subsequent processing steps.

²In fact, the most common diffusion barrier is a Ti/TiN one, described below.

A very common application is a Ti/TiN tandem barrier, wherein the titanium layer improves the TiN adhesion to the silicon dioxide. This underlayer also improves corrosion resistance [44]. The TiN barrier is usually deposited by reactive sputtering, or by thermal nitridation. A CVD TiN commercial process was recently introduced [45].

Another example of a diffusion barrier is a TiSi_2 deposited by the reaction of thin titanium film with silicon [46]. This barrier can also be deposited by reactive evaporation [47].

In all these cases, the thickness of the titanium film is very small. It is thus very important to ensure a conformal step coverage of the titanium film. And it is precisely the problems associated with the deposition of this titanium layer which represent the main focus of this thesis.

Chapter 2

Sputtering

Sputtering is a PVD process, where material is ejected from the surface of a solid target as a result of momentum transfer from bombarding ions. A source of the coating material (in our case a metal target) is placed into a vacuum chamber¹ together with the substrate (in our case a silicon wafer) and chamber is then evacuated to a base pressure of at least 10^{-6} Torr². The chamber is then backfilled with a *working gas* (defined below) in order to reach a working pressure (typically 1-50 mTorr), at which a plasma can be started and maintained using an electric discharge. Here the target serves as a cathode with typical voltages from ≈ -300 V (usual laboratory equipment) up to -1000 V. The discharge is typically dc. For sputter-deposition of dielectric materials, an rf discharge is used. Plasma is a source of gas ions which bombard the target. This bombardment leads to a dislodgement of target particles. Sputtered material is ejected primarily in form of single atoms, however, appearance of atomic clusters was also reported [48, 49, 50]. The fraction of sputtered clusters is related to the target material, and in case of polycrystalline metal targets seems to be below 5% [51]. Most commonly, these clusters are *dimers*, i.e., two-atom clusters.

¹A typical laboratory system is described here, which may differ from systems used for large scale manufacturing.

²Torr is the pressure unit used in this thesis. SI unit is Pascal, $1 \text{ Torr} \approx 133 \text{ Pa}$.

The ratio of clusters containing more than two atoms is usually about one order of magnitude smaller than that of dimers. It has also been suggested, that most of these clusters actually form *after* their dislodgement from the target [52]. It thus appears reasonable that sputtered particles will be further considered to be in the form of single atoms. This approach is also commonplace in most handbooks. The substrate is placed usually below or in front of target to intercept maximum of the sputtered flux. An illustration of a typical sputtering system can be seen in the fig. 2.1. In order to study the sputtering process in greater detail, it seems to be advisable to divide the whole process into three sequences and describe them separately – these are events occurring at the target, transport of sputtered particles from target to substrate, and finally events associated with the film growth. However, prior to this description, basic terms and expressions associated with sputtering will be introduced.

2.1 Basic definitions

Sputtering yield is the number of particles ejected by the impact of one incident ion. This number, naturally, depends on number of other parameters, such as angle of incidence, energy and mass of bombarding ion, target material, target voltage etc. Typical values of sputtering yield for several common metallization materials can be found in the table 2.1 together with values of binding energy U_b .

Sputtering threshold is a minimal energy needed for the sputtering event to occur.

It is usually about $4 * U_b$, where U_b is a binding energy of an atom in the lattice.

For most materials the sputtering threshold is from 15 to 30 eV³.

³This value assumes an argon bombardment.

Figure 2.1: Cross-section of a sputtering system.

Working gas is a gas used to backfill the chamber prior to deposition. This gas has to be inert, inexpensive and have a suitable mass in order to obtain an optimal sputtering yield for given target material. The most common choice is argon, which is also used for the experiments described in this thesis.

Base pressure is the vapour pressure in the chamber after the sputtering system had been evacuated, but prior to the chamber backfilling by working gas. It is a figure of merit of the sputtering system. Table 2.2 captures the classification

<i>Target Material</i>	<i>Sputtering Yield for Argon at</i>				<i>Binding energy per atom (eV)</i>
	<i>100 eV</i>	<i>300 eV</i>	<i>600 eV</i>	<i>1000 eV</i>	
Al	0.11	0.65	1.7	1.9	3.39
Au	0.32	1.65	2.8	3.6	3.81
Cu	0.50	1.60	2.3	3.2	3.49
Ni	0.28	0.95	1.5	2.1	4.44
Pt	0.20	0.75	1.6	—	5.84
Si	0.07	0.31	0.5	0.6	4.63
Ti	0.08	0.33	0.41	0.7	4.85
W	0.12	0.41	0.75	—	8.90

Table 2.1: Example of sputter yields and binding energies. [7, 20, 53]

of vacuum quality.

<i>Degree of vacuum</i>	<i>Base pressure range (Torr)</i>
low	25 – 760
medium	10^{-3} – 25
high	10^{-6} – 10^{-3}
very high	10^{-9} – 10^{-6}
ultra high (UHV)	10^{-12} – 10^{-9}

Table 2.2: Vacuum system classification with respect to base pressure.

Reactive sputtering is a modification often used for deposition of oxides and nitrides [42]. In addition to the working gas, a reactive gas such as oxygen or nitrogen is deliberately introduced into the chamber. For more description please see [54].

Magnetron sputtering is a very common version of sputtering, wherein a magnet is placed behind the target in order to confine the secondary electrons. The reason for this configuration will be explained in section 2.3.

Bias sputtering is a modification where a substrate is intentionally biased in order to attract positive Ar ions. These should enhance sample surface ion bombardment and contribute to *resputtering* (please see section 4.4).

2.2 Target

Because sputtering is an extremely versatile and universal process, almost any solid can be used as a target material. Once a bombarding ion hits the target, one of the following things may occur (see fig. 2.2):

- the ion may be reflected, most probably having been neutralized prior to the impact [55, 56] — this is a basis of *Ion Scattering Spectroscopy*, a surface analysis technique
- the ion may be buried deep into the target (*ion implantation*)
- the impact of ion may eject a *secondary electron*
- the impact of ion may cause a “collision cascade” (see fig. 2.3) leading to the ejection of an target atom (*sputtering*)
- the impact of ion may contribute to a structural rearrangements of the top surface layer (i.e., simple vacancies, interstitials), damaging the original lattice structure of the target
- target heating – sputtering is a very inefficient momentum transfer process, because sputtered atoms leave target with only about 1% of the original ion energy [57]

Figure 2.2: Illustration of a sputtering process.

Structural rearrangements and target heating may alter the composition of surface monolayers, which in turn can change conditions of the collision cascade and have an impact on the sputter yield.

2.2.1 Sputtered flux

Until now, all events occurring at the target were looked upon from the point of view of an individual particle. However, since we are interested in the process of sputtering as a whole, it is also desirable to consider the overall effect of continuous multi-particle bombardment of the target. Because of the non-uniformity of a stream of bombarding

Figure 2.3: A collision cascade leading to the ejection of a sputtered atom.

ions, it is reasonable to assume, that sputtered particles do not have identical energy nor velocity. Rather, they are described by specific *angular* and *energy distributions*. Unfortunately, there is very little experimental data available about these distributions. Instead of exact data, it is usually supposed that these distributions are more or less “cosine”, i.e. random ones. More about this particular topic can be found in [17, 58, 59]. However, knowledge of these distributions is extremely useful for thorough understanding of sputtering. As far as metal targets are concerned, it has been suggested that the angular distribution of sputtered flux consist of two components – randomly ejected particles (i.e., cosine distribution) and particles ejected in close-packed directions which reflect the structure of crystal lattice of respective material.

Naturally, this parameter depends on the particular crystal structure of each respective target material, and there is very little experimental data available. One of the very few exceptions is aluminum, where the emission characteristics were measured [60]. To further complicate the issue of sputtered flux distributions, working gas pressure must be taken into account because the shape of these distributions is subject to the scattering of the sputtered particles by argon ions and neutrals. What we need to know, however, is the particular shape of the angular distribution *at the substrate*, because this will affect the properties of a growing film. These considerations will be further elaborated in following sections.

2.3 Glow discharge

As was already mentioned, a glow discharge is a frequently used source of ions for sputtering processes. Although a glow discharge is not, strictly speaking, a plasma, both expressions are used interchangeably, mainly because many of general physical principles governing plasma behaviour can be also applied to glow discharges. One of the fundamental works on plasma physics is [61], while an overview of glow discharge processes can be found at [62, 63, 64].

2.3.1 Glow discharge properties

Ideally, glow discharge should provide a uniform, homogeneous supply of bombarding ions over the whole target area with minimal heat transfer to the target (especially at higher voltages, as cooling is rather difficult and expensive process). The discharge is usually ignited by applying high voltage to a metal electrode (target) in the vacuum chamber. This is called a diode discharge. These discharges have quite low ionization

fraction, usually about 10^{-4} . Electrons are the dominant charge carriers, since the heavy ions are usually considered immobile (this is a common theoretical treatment in most cases). There are two important collision processes, which sustain the discharge, and a subsequent relaxation process responsible for the visible glow:

Impact ionization of an Ar ion, which can be described as

has a threshold energy of 15.8 eV. Both electrons can – providing they still have sufficient energy – ionize other Ar atoms. There is also a possibility of double ionization of Ar ion, that, however, requires substantially larger energy and hence is much less probable. Triply ionized argon ions have not been observed.

Excitation of an Ar ion,

has a threshold energy of 11.56 eV and its product, Ar^* is metastable, which lifetime, however, may be surprisingly long (up to milliseconds).

Relaxation of excited Ar atom, Ar^* , is a real cause of the glow. Transition of an excited Ar atom,

into a ground state is accompanied by the emission of a photon of specific energy $h\nu$. A typical argon glow is purple, while the glow of oxygen is white.

There is a number of other collision processes (recombination, Penning processes...), their description, however, would be well beyond the scope of this thesis.

Electrons have a crucial role in the collision processes needed for a discharge maintenance. However, the configuration of a classical diode discharge is unable to guarantee efficient use of electron energy for impact ionization processes, because of rather short electron lifetime in the plasma. Given the small substrate-target distance, most electrons end at the substrate or chamber walls after only a few collisions. A solution, which dramatically increases the path of electron in the discharge, and thus a probability of impact ionization or excitation, is to introduce magnetic field into vacuum chamber.

This type of discharge is called *magnetron sputtering* (please recall its definition in section 2.1). One of the direct benefits of magnetron discharge is the fact, that it can operate at a lower working gas pressure than a diode discharge. If the gas pressure is too low, then there is an insufficient number of collision processes, if, on the other hand, the gas pressure is too high, electrons lose their energy too fast. In any case, the glow discharge cannot be sustained. Moreover, the magnetic field keeps discharge closer to the target and enhances thus target bombardment.

Nowadays, the use of a magnetron discharge is the most common type of sputtering and is used in industrial manufacturing as well as in research laboratories. There are a number of various configurations [63] for such a system, but only a planar cathode will be considered further.

2.3.2 Angular distribution in a glow discharge

Until now, only collision processes responsible for glow discharge maintenance were described. However, from the point of view of a sputtering process, collision processes affect also the sputtered atoms.

It has been explained that sputtered atoms have certain angular and energy distributions as they were emitted from the target. In order to investigate the process of thin film growth, it is necessary to have a knowledge of the shape of angular distribution *at the substrate*⁴.

Figure 2.4: SEM photograph of evaporated titanium film.

Obviously, the issue of the evolution of the original distribution which takes place between target and substrate, has to be addressed. Fig. 2.4 shows a SEM photograph of thin titanium film deposited by evaporation. Evaporated films are deposited at pressures corresponding to high vacuum. The film shown at the fig. 2.4 was deposited at 2×10^{-7} Torr, nominal power was 2 kW and evaporation time was 6 min. The

⁴Energy distributions will not be further considered because of problems associated with their experimental measurement.

original angular distribution of the evaporated flux remains practically unchanged.

Figure 2.5: SEM photograph of sputter-deposited titanium film.

The sharp edge of the film caused by shadowing is very obvious. A similar photograph of a titanium film deposited by sputtering under very similar conditions⁵ can be seen in figure 2.5. The main difference is the presence of the working gas for case of sputter deposition, which can significantly alter original angular distribution of sputtered flux due to the scattering. This argument can also explain the inability of evaporated films to coat non-planar substrate.

In case of sputtering, the working gas pressure is of crucial importance in the evolution

⁵Argon pressure was 10 mT.

of angular distributions, as it determines the collision frequency of the sputtered atoms with the gas particles. This problem is addressed in [17] and in the vapour transport simulator SIMSPUD.

2.4 Film growth and microstructure

Since there is a vast amount of literature available on this topic [7, 21, 63, 65, 66] only a brief description of metal film growth will be given.

2.4.1 Phases of film growth

Upon arrival of a sputtered atom onto the substrate surface (see fig. 2.6), one of the following events may occur. An atom can essentially

- reflect off the substrate
- temporarily adsorb and later re-evaporate
- adsorb and remain at the surface

The first case is in our situation (i.e., sputter-deposited metal) very unlikely [67]. So we essentially have only two options, which will be further investigated. An atom, which gets adsorbed at the surface, is called an *adatom*. This adatom may diffuse over the film surface (for a more complex description of surface diffusion please see section 4.2). During this process, an adatom can either return to the vapour phase or interact with other adatoms. This interaction may lead to the creation of atomic clusters. The dimension of such a cluster is of crucial importance – there is a certain *critical size*, above which the future cluster growth is energetically favourable. On

Figure 2.6: Species arriving at the sample surface during sputtering.

the other hand, clusters with smaller than critical size will decompose. This is the *nucleation* stage [68] of the film growth (please see fig. 2.7).

An important factor in the process of a film growth is the relative growth speed with respect to the substrate normal. If clusters grow faster in the direction parallel to the surface, so called *island stage* growth occurs. Here, respective islands have well-defined crystallographic orientations. Further island growth is called *coalescence*, where respective islands will grow into physical contact.

2.4.2 Origin of film microstructure

Once a continuous film layer forms, it will become a “substrate” for future layers or islands. Hence, the surface diffusion will occur under different conditions than it was the case with first few monolayers. There is also another process that greatly affects the film structure – *geometrical shadowing*.

Figure 2.7: Illustration of thin film growth process.

Parts of the substrate, which are relatively “higher” will intercept proportionally more flux than their immediate neighborhood, and this effect will in turn enhance their future preferential growth at the expense of sites in their vicinity. A direct consequence of this shadowing process is a columnar structure observed for vapour-deposited thin films (see fig. 2.8). This process is extremely important for industrial

manufacturing, because the topography of VLSI circuits is rarely smooth. Thus, a process called *planarization* is usually employed – the surface of deposited metal is planarized in order to make the deposition of subsequent layer easier [13].

Figure 2.8: Structure model. [69]

Essentially, the surface diffusion and geometrical shadowing will determine the microstructure and properties of the film. While surface diffusion tends to re-distribute the adatoms, shadowing tends to concentrate growth on the top parts of columns. Most of the parameters used for the optimization of the film properties, such as substrate temperature, gas pressure, substrate bias, target-substrate distance etc. will eventually lead to a change in one of these effects.

The deposition temperature has a dominant effect on the thin film microstructure. The parameter used for the evaluation of the temperature impact is a T/T_m ratio,

where T is a deposition temperature of the substrate, and T_m is the melting point of the respective material. One of the first and most important models which classified a film microstructure as a function of T/T_m is shown in the fig. 2.8.

Figure 2.9: Surface of zone 1 film. Titanium film deposited at 10 mT argon pressure.

Zone 1 films have a columnar structure separated by open boundaries as a result of geometrical shadowing. Films have usually poor reflectance due to the large surface microroughness. This is a result of insufficient surface diffusion.

Refractory metals usually exhibit a zone 1 structure when deposited at room temperature. An example of the surface microroughness of zone 1 film can be seen in fig.

2.9. Zone T (the transition zone) consists of a dense array of fibrous grain films with a reflective surface. Example of zone T film surface is shown in the fig. 2.10.

Figure 2.10: Surface of zone T film. Titanium film deposited at 2 mT argon pressure.

Films in both zone 1 and zone T can have large intrinsic stresses. Zone 2 structure consists of columnar grains with significantly higher density, as a result of dominant surface diffusion. Zone 2 films also tend to have lower resistivity as compared to films with zone 1 or zone T structure.

Finally, zone 3 films are dominated by the influence of bulk diffusion. Film grains no longer exhibit a columnar shape, probably as a result of recrystallization.

SEM photographs convincingly show the impact of working gas pressure on the film microstructure. Films deposited at lower pressure have a smoother and brighter surface as a result of more pronounced surface diffusion (zone T structure). Increased substrate heating caused by higher energy of incident particles (sputtered atoms, argon particles) will also increase the surface diffusion length. A higher working gas pressure will result in more frequent scattering, and a considerable loss of the original kinetic energy of the sputtered particles and the argon ions and neutrals. This will affect the surface diffusion range and as a result, zone 1 films will have higher surface microroughness and lower reflectivity.

Chapter 3

Collimated sputtering

Many problems associated with the premature failure of diffusion barriers sputter-deposited onto contacts/vias (see fig. 3.1) are caused by uneven barrier thickness.

The main trouble spots are:

- the bottom part of the contact/via (low bottom coverage)
- the bottom corners of the via (film discontinuity at the corner)
- the top of via, where preferential lateral growth caused by atoms arriving onto the substrate at oblique angles causes the reduction of the via dimension and aggravates the problem of insufficient film coverage at the via walls and bottom.

A simple modification of the standard sputtering process has been proposed [70]. This technique employs a physical collimator (see fig. 3.2) to control the flux of sputtered particles onto the substrate, and still retains almost all advantages of conventional planar magnetron sputtering. The ability of this technique to coat high aspect ratio contacts/vias has attracted considerable interest [71]-[74].

There is, however, still a great deal of uncertainty as far as the evaluation of overall impact of the collimated sputtering on properties of deposited thin films is concerned.

Figure 3.1: Typical problems of *via*-contact.

Therefore, a set of experiments was performed with the goal to further investigate the impact of collimation on properties of thin metal films. The focus of these experiments was on titanium and aluminum, two highly important metallization materials.

3.1 Computer simulation

Since PVD techniques are complex multi-parameter processes, reliable computer simulation tools are invaluable for their optimization.

The philosophy of the Monte-Carlo simulation programs SIMSPUD and SIMBAD was described in the section 1.3. These programs, however, were never previously used for

Figure 3.2: Illustration of collimated sputtering.

the simulation of collimated sputtering. The vapour transport simulator, SIMSPUD, was modified to include the effect of rectangular collimator (August 1992 by Prof. Dew, author of SIMSPUD). Further modification enabled the simulation of effects caused by hexagonally shaped collimator (October 1992 by author of this thesis).

SIMSPUD was then used in order to predict the angular distributions of sputter-deposited Ti films. Most of the input parameters required for a vapour-transport computer simulation were obtained from the literature. However, one parameter

that was obtained experimentally was the energetic collision cross-section of Ti-Ar interaction, which is required for the correct modelling of the transport of Ti atoms from the target to the substrate at realistic pressures. The collision cross-section varies considerably with energy of the sputtered atom, and, unfortunately, the detailed energy dependence for Ti-Ar collisions is not well established.

Therefore, SIMSPUD assumes the relative energy dependence of Ar-Ar collisions [75], which requires only two values to cover their energy dependence over the complete regime of interest. The first one is a collision cross-section of thermalized atoms, σ_T , (those with energy below 1 eV), and the other one, σ_o , is a collision cross-section of particles with energy higher than 1 eV. A detailed description of the scattering processes can be found in [17, 75, 76, 77].

3.1.1 Pinhole experiment

This experiment was necessary in order to determine the values of collision cross-sections of Ti-Ar collisions, as described above. In this experiment, a film was deposited at 10 mTorr argon pressure through a small hole in a mask just above the substrate (see fig. 3.3).

The thickness profile of the deposited film is highly sensitive to the angular distribution arriving at the pinhole, which in turn is a strong function of the collision cross-section of sputtered atoms. This thickness profile was directly measured using a profilometer (Tencor Alphastep 200) with resolution of 5 nm and compared with the SIMSPUD results. The resulting fit using the experimental results is shown in fig. 3.4.

Figure 3.3: Pinhole experiment. The illustration of film profile is not proportional.

By fitting the SIMSPUD results using the experimental data obtained from this experiment, values of both respective parameters were determined to be $\sigma_T = 68 \text{ \AA}^2$, and $\sigma_o = 48 \text{ \AA}^2$. Fig. 3.4 demonstrates a very good agreement between experimental data and the computer simulation.

Angular distributions obtained from SIMSPUD simulations were then subsequently used in film growth simulator, SIMBAD. No further calibration nor adjustment¹ of SIMBAD was necessary. However, these experiments represent the first test of SIMBAD's ability to model the microstructure of films deposited by collimated sputtering. Results of these experiments are discussed in following sections. It should be noted, that one more assumption was imposed on the SIMBAD modelling process – that there is no re-emission of already deposited atoms back to the vapour phase. This

¹SIMBAD has a default angular distribution, which is a cosine one. This cosine distribution is used, unless a user-specified angular distribution is provided (i.e., SIMSPUD result).

Figure 3.4: Pinhole experiment: SIMSPUD's fit into experimental values. Error bars are double the standard deviation.

assumption will be discussed and justified in the following chapter.

3.2 Titanium thin films²

There are two reasons why titanium films were a principal focus of this work. One of them is the overall importance of titanium as a metallization material. The other reason was that this thesis is a part of the joint research project, which was focused on sputter-deposited titanium thin films.

3.2.1 Experimental set-up

To deposit films for this set of experiments, high purity (99.95%) Ti was dc sputtered from a 5.0 cm diameter target using a planar magnetron source at 500 W constant

²Material in this section was accepted for publication in *Journal of Applied Physics* [78].

power with nominally 400 V applied, at 4, 10, and 16 mTorr of argon pressure, respectively. The base pressure for the vacuum system prior to deposition was between 5×10^{-7} and 1×10^{-6} Torr. The substrate profile consisted of trenches 0.8 μm wide with 85° sidewalls and aspect ratio of 1.2, formed in thermal oxide on a silicon wafer. The substrate temperature was not controlled during the deposition. Square grid collimators with 0.5 cm pitch were fabricated from 0.1 mm thick stainless steel foil. The depths of the collimators were 0.25 cm, 0.5 cm, 0.75 cm, and 1 cm, corresponding to aspect ratios of 0.5, 1, 1.5, and 2, respectively³.

The distance between the bottom of the collimator and the top of the substrate was 1.30 cm. The distance between the target and the substrate was 7.66 cm. The deposition time was 60 min in case of collimated sputtering and 15 min in case of uncollimated (conventional) sputtering, unless noted otherwise. After sputter deposition, the samples were cleaved to produce cross-sections of the trenches and a scanning electron microscope (SEM) was used for evaluation of deposition rates and film coverages. Wafers were cleaved manually, no wafer saw was used. Samples were not polished. All samples were immersed into liquid nitrogen (77 K) for the period of 60 seconds prior to cleaving in order to obtain a sharper cross-section image. Samples were then glued to a steel stub by silver epoxy and baked for 10 minutes at 130°C . A thin gold layer of approximately 15 nm was sputter-deposited onto the sample cross-section in order to prevent sample over-charging during SEM.

³One set of experiments was performed at the constant Ar pressure of 10 mT with different collimator aspect ratios, while the other one was performed with a constant collimator aspect ratio of 1.0 and at different pressures.

3.2.2 Effect of collimator aspect ratio

The principal effect of collimation is thought to be the effective elimination of particles with high lateral velocities (see fig. 3.2). All particles whose trajectory intersect the collimator vanes are supposed to be trapped there (here an assumption of zero re-emission is invoked again).

Since the majority of sputtered particles have their original velocity vector almost parallel to the target-substrate axis, it can be argued that particles entrapped by the collimator are likely to have undergone a proportionally higher number of collisions than those that are allowed to pass through collimator. If this is true, then the higher the collimator aspect ratio is, the "better" is the collimator effectivity, and the more particles will end their trajectory at the collimator vanes.

If only those particles with relatively "straight" trajectory could pass through collimator, then we could intuitively expect a higher bottom coverage and a lower deposition rate. This is, indeed, the case. The deposition rate of titanium was found to be $\approx 7.9 \text{ \AA/sec}$ in case of conventional (uncollimated) sputtering at 10 mT Ar pressure. However, when a collimator of aspect ratio 2.0 was employed (from now on marked as C2), the deposition rate dropped to $\approx 2.1 \text{ \AA/sec}$.

From the point of view of angular distributions at the target plane, one could expect higher proportion of particles arriving at deposition angles almost normal to the substrate at the expense of those with oblique angles of incidence. This functional dependence is depicted at fig. 3.5. The only varying parameter in those simulations was the collimator aspect ratio - 0.0 (no collimation, curve A), 1.0 (collimator a.r. 1.0 (C1), curve B), and 2.0 (C2, curve C). These results convincingly show a sharp

Figure 3.5: SIMSPUD's prediction of angular distributions of titanium sputtered flux. The number at the respective curve shows the aspect ratio of collimator employed.

increase of the ratio of atoms arriving at the substrate with trajectory very close to the target-substrate axis. This effect is in turn related to the increase of collimator aspect ratio.

Fig. 3.6 shows a decrease in the deposition rate as a function of the collimator aspect ratio. "Relative deposition rate" means a ratio of deposition rate for the case of collimated sputtering vs. the one of uncollimated sputtering under otherwise identical conditions. This ratio is expressed in %. Experimental data were obtained by measurement of the film thickness from SEM photographs. Simulated data were calculated from the number of particles deposited at the specified area of the substrate (please recall the description of SIMSPUD from the section 1.3). Since SIMSPUD

Figure 3.6: Collimation induced decrease in the deposition rate.

keeps statistical data about every single particle, two simulations, with and without collimator were performed. All parameters including the seed for a random number generator were identical. Number of the generated particles was in both cases very high (10 million) in order to minimize statistical error. The relative deposition rate was then calculated as the ratio of collected particles obtained from both experiments. This value was then compared to the experimental result, and results of this comparison (made for different aspect ratios of collimator) is shown in the fig. 3.6.

Good overall agreement of experimental results and SIMSPUD predictions, enabled us to perform further simulation of the thin film growth using the SIMBAD program, where appropriate angular distributions (see fig. 3.5) corresponding to the different collimator aspect ratios were used. Results can be seen in figures 3.7 and 3.8.

Figure 3.7: Thin Ti film sputter deposited without collimator – SEM photograph (top) and the respective SIMBAD prediction (bottom).

Figure 3.8: Thin Ti film deposited with collimator (C2) - SEM photograph (top) and the respective SIMBAD prediction (bottom).

SEM photographs show a cross-sections of thin Ti films deposited without collimator (see fig. 3.7), and with collimator C2, (see fig. 3.8). These photographs clearly illustrate the advantage of collimation. Bottom coverage is substantially improved from about 50% for conventionally deposited (uncollimated) film to about 82% in case of collimated film.

Another important feature of collimated film is a sharp decrease in the lateral growth at the via-opening (also called “mushrooming”). Collimation is able to substantially alleviate this undesired effect. Another impact of collimation is a slightly altered film microstructure at the trench sidewalls. Measurements averaged over ten columns for each SEM photograph yield sidewall column angles of 30° and 40° for films deposited without collimator and with C2 collimator respectively. This alleviates the effects of film discontinuity at bottom trench corners. On the other hand, this effect indicates worse step coverage when compared to the films deposited by conventional (uncollimated) sputtering. The reason of this change in columnar orientation is the more directional angular distribution for the case of collimated sputtering.

Respective SIMBAD predictions are in very good agreement with the experimental data. Gradual improvement of the bottom coverage as a function of the collimator aspect ratio is summarized in the table 3.1. The lateral film growth can also be observed (please see fig. 3.7).

To make a quantitative comparison, we have measured the opening ratio, which we define as the ratio of the width of narrowest opening at the top of the trench to that at the bottom. For the uncollimated sputtering case, this ratio is about 0.75 for the experimental film and 0.77 for simulated film. For the collimated sputtering with

<i>Bottom coverage</i>		<i>Collimator aspect ratio</i>
<i>SIMBAD</i>	<i>Experiment</i>	
52 $\pm$ 5 %	50 $\pm$ 5 %	0.0
58 $\pm$ 5 %	62 $\pm$ 5 %	1.0
77 $\pm$ 5 %	82 $\pm$ 5 %	2.0

Table 3.1: Bottom coverage increase as a consequence of collimation – comparison of experimental and simulated values.

a collimator of aspect ratio 2 (fig. 3.8), this ratio is very close to 1.0 for both the experimental and simulated films. The simulation shows again a very good agreement with the experimental data in this particular case. Both, the deposition rate and the film microstructure were successfully simulated using the SIMSPUD/SIMBAD package.

3.2.3 Effect of argon pressure

Working gas (Ar) pressure is another important parameter which determines thin film microstructure and properties. For case of collimator sputtering, this parameter is even more crucial. As it was shown, the main purpose of collimator is to entrap those particles, whose trajectory differs significantly from the vertical target-substrate axis, i.e., those, which have presumably undergone proportionally larger number of collisions. As the collision frequency is directly proportional to the working gas pressure, so is the number of those particles. In other words, the higher the working gas pressure is, the more particles will be entrapped on collimator vanes (and again, the lower the overall deposition rate will be).

The following experiments were performed under the experimental conditions described in the section 3.2.1. The only exception was the target. Instead of the

circular, 5.0 cm diameter target, a rectangular, 12.5 cm x 20 cm one was used. The collimator C1 was used for all experiments. Gas pressure was 4, 10 and 16 mT respectively. The results are shown in the table 3.2. Clearly, an increase in working gas pressure also increases the collimator "effectiveness". The experimental value of relative decrease in the deposition rate was calculated as an average from five measurements (as was the case with the data shown in the fig. 3.6). Typical standard deviation did not exceed 5 %.

Titanium	Pressure (mTorr)		
<i>Deposition Rate</i> (Å/s)	4	10	16
- No collimation	7.6	8.0	8.0
- Collimation C1	2.2	2.1	1.6
<i>Deposition rate ratio</i> (coll/ncoll)	27%±5%	25%±5%	20%±5%
<i>SIMSPUD prediction</i>	38 %	34 %	19 %

Table 3.2: Deposition rate decrease as a consequence of collimation – comparison of experimental and simulated values. Titanium sputter-deposited at various Ar pressures. Collimator aspect ratio was 1.0 (C1).

Ultimately, collimator aspect ratio as well as working gas pressure are the two important parameters in the process of the optimization of deposition rate. The above described results indicate that the collimator seems to induce the effect of "lower working gas pressure", i.e., the angular distribution of the sputtered flux for the case of collimated sputtering has a shape similar to the distribution of uncollimated flux of titanium atoms sputtered at lower argon pressure. The problem whether this "lower pressure effect" has any impact on film properties is dealt with in chapter five.

Unfortunately, SIMSPUD prediction does not quite match the experimental data.

There may be several reasons for this discrepancy. Lower gas pressure also means lower scattering frequency. Thus, angular distribution of the sputtered flux will be significantly less affected at low argon pressure (4 mT) than it would be the case at the higher argon pressure (16 mT). (Tables 4.1 and 4.2 in chapter four show SIMSPUD's prediction for an average mean free path of sputtered particle as a function of pressure.) Hence any problem with the shape of the *original* angular distribution of the sputtered flux will be more pronounced for the case of lower argon pressure. (Please recall the discussion about target material rearrangement and target emission data in chapter 2.) Another reason may lie in the fact that SIMSPUD assumes a zero pressure gradient in the vacuum chamber, which is not quite true.

3.3 Aluminum thin films⁴

Given the prominent position of aluminum as a metallization material, some of the experiments described above were performed to study the effect of collimation on aluminum films.

Aluminum	Pressure (mTorr)		
Deposition Rate (Å/s)	4	10	16
- No collimation	10	7.6	9.0
- Collimation C1	3.1	2.1	1.8
Deposition rate ratio (coll/ncoll)	31%±5%	27%±5%	20%±5%
SIMSPUD prediction	33 %	20 %	13 %

Table 3.3: Decrease of deposition rate of aluminum as a consequence of collimation at different Ar pressures. Collimator C1 was used.

⁴Material in this section together with material in section 5.4 was presented at International Conference on Metallurgical Coatings, San Diego, CA in April 1993 and subsequently accepted for publication in *Thin Solid Films* [79].

All experimental data were the same as described in the section 3.2.1. This time, a rectangular Al target 12.5 cm x 20 cm was used. Collimator C1 was used for all experiments and the effect of varying pressure was studied. Again, aluminum film was deposited over a substrate described in the section 3.2.1.

Figure 3.9: SEM photograph of thin Al film sputter deposited without collimator.

The experimental evaluation of the deposition rate decrease was also performed and the results were compared to the respective SIMSPUD predictions. All data are shown in the table 3.3. This time, however, there is much better agreement between the experiment and simulation, namely in case of lower gas pressures. It may be partly due to the fact that the emission characteristics for the aluminum target are known [60]. This result seems to indicate that the real cause of the discrepancy between the simulated relative deposition rate and experimental data for the case of titanium

(please see table 3.2) may have been due to the missing data describing respective emission characteristics for titanium targets. However, the relationship between the experimentally measured deposition rate and pressure remains unclear.

Figure 3.10: SEM photograph of thin Al film sputter deposited with collimator C2.

Figures 3.9 and 3.10 show SEM photographs of thin aluminum films deposited without collimator and with collimator C2. Please see also figures 3.7 and 3.8. The “mushrooming effect” is again very obvious. The film structure is also very different from the structure of titanium films. The main reason for this difference lies in the different T/T_m ratios for titanium and aluminum films. Unfortunately, since our sputtering system does not have the capability to measure the substrate temperature *in situ*, the value of deposition temperature may be only estimated⁵. However, given the dif-

⁵According to our estimate, substrate temperature can during deposition reach approximately 250° C.

ference in the values of melting points for aluminum and titanium (T_m (Al) = 660° C vs. T_m (Ti) = 1670° C), we are probably dealing with a zone 1 film (titanium) vs. a zone 2 film (aluminum).

SIMBAD simulation of the aluminum films was not attempted. The reason is in the T/T_m ratio. Basic SIMBAD code for thin film modelling is designed for low-temperature deposition. In case of aluminum, however, this is no longer true. High-temperature aluminum deposition is a recent addition to the SIMBAD code, so it can be modelled [80]. However, the modelling of the experimental results shown in fig. 3.9 and fig. 3.10 would go beyond the scope of this thesis.

Chapter 4

Surface effects

This chapter deals with events that occur at the film surface during the process of atom impingement and surface diffusion. In the previously described experiments, it was implicitly assumed that all sputtered particles that hit the surface would remain there, i.e. none of them would return to the vapour phase. This assumption was also used for the computer modelling of film structure in that chapter (see fig. 3.7 and fig. 3.8) and the agreement between the SIMSPUD/SIMBAD simulation and experimental result was very good. This could be taken as an indirect proof of this assumption. However, it will be shown directly that this assumption is correct, that in the experimental conditions described in the previous chapter, indeed, no measurable fraction of the adsorbed atoms returns to the vapour phase. It will also be shown theoretically as well as experimentally when and why will this cease to hold. In order to do that, some important definitions will be introduced.

4.1 Basic definitions

Physisorption is a bond that holds the physisorbed atom (adatom) on the surface (see fig. 4.1). The physical mechanism of this bond is the van der Waals force.

with the typical bond strength anywhere between 0.1 - 0.5 eV [57]. This is a metastable state, because of the very low bond strength. Physisorbed atoms are also very mobile.

Figure 4.1: Simplified model of physisorption and chemisorption energy states [81].

Chemisorption is a bond that exists between atoms in the crystal lattice. This bond is much stronger, in the case of common metals anywhere between 3 - 10 eV (please see table 2.1). Chemisorption involves valence electron sharing. A chemisorbed state is associated with a potential well (see fig.4.1) with depth of U_b . For atom to break loose, an energy U_b must be supplied.

Resputtering is an effect caused by the flux of bombarding particles onto the substrate [82] – some atoms may be sputtered (much the same way as it was the case at the target). Resputtering may be caused either by energetic argon particles or by metal atoms themselves.

Atomic peening (also called *forward sputtering* or *recoil implantation*) is also a result of a continuous bombardment of the evolving lattice structure on the sample surface. Bombarding particles simply knock some of the surface particles deeper into the crystal. Since no momentum reversal is required as it was the case at the real sputtering, the sputtering threshold is much lower and hence – especially at lower working gas pressures – this effect can have an important impact on the structure of deposited film.

4.2 Surface diffusion

This is a mechanism responsible for mass transport over the surface, which has a crucial importance for the microstructure of a thin film. Unfortunately, it is also an extremely complicated subject, many facets of which are not well understood. Hence, only very simple description will be given here. An excellent treatment of this topic can be found in [35, 65, 83].

4.2.1 Diffusion length

One of the most important parameters which controls the film microstructure is the surface diffusion length. A physisorbed atom, seeking the most suitable “cradle site”, i.e., the site where the surface curvature would be minimal, will “test” only a limited number of neighborhood sites. The extent of this “search” is characterized by the

surface diffusion length. In the simplest situation, the adatom encounters a periodical surface potential, as shown in the fig. 4.2.

Figure 4.2: Simplified two-dimensional model of surface energy.

The atoms are in constant motion, which has been represented as “atomic hopping”. However, in order to “jump over the potential barrier”, i.e., diffuse to the neighbouring site, two conditions must be met. First, the site must be empty. Since we are dealing with the surface diffusion, this condition is not a problem. (This is, however, not true for the bulk, or crystal diffusion, and this is the main reason why bulk diffusion proceeds at much slower pace than surface diffusion.) The second condition is that the adatom must have enough energy to surpass the barrier. The higher energy the adatom will have, the more potential cradle sites it will explore and the higher effect surface diffusion will have in terms of surface mass-redistribution.

If the substrate consists of *vias* or *trenches* (grooves) there is a chance that at least part of the material can reach the sites where the arriving flux density is very low (i.e., step corners). One of the ways to increase the average adatom energy and hence the surface diffusion length is heating the substrate, which is often used in microelectronic circuit fabrication. (Please recall the discussion of T/T_m impact on film microstructure in the section 2.4). Another possibility is simply keep the working gas pressure as low as possible, so that sputtered atoms can retain a maximum of their original kinetic energy.

Sticking coefficient

There are also other things that may happen during the process of surface diffusion. An adatom may be “buried” by the arriving flux prior to its finding the most appropriate cradle site. This will shorten its surface diffusion length and may cause a crystal imperfection. An adatom can also re-evaporate to the vapour phase during the diffusion process and get subsequently deposited elsewhere. If this happens, the material is said to have a low sticking coefficient. *The sticking coefficient s is defined as $s = 1 - p$, where p is the re-emission probability of a physisorbed atom.* The value of the sticking coefficient for the metals used in VLSI technology is one of subjects of current research.

Resputtering

There is, however, yet another mechanism that may cause atoms to return to the vapour phase – resputtering [82]. Unlike low sticking coefficient, however, resputtering affects chemisorbed atoms, and there is little possibility to avoid this completely. It is simply the same sputtering process as in the target, except that it is the *substrate*

itself that is being bombarded. The main problem lies in the fact, that although both effects have completely different origins, their result is the same – a fraction of the film atoms are re-emitted from the surface. However, for a process optimization it is important to know the real cause of the re-emission.

4.3 Overhang experiment

In order to measure the fraction of re-emitted atoms from the sample surface, the following experiment was designed and performed (please see fig. 4.3 and fig. 4.4).

Figure 4.3: Cross-section of the overhang structure manufactured at AMC.

The “overhang” structure was manufactured in the Alberta Microelectronic Centre (AMC) specifically for the purpose of this thesis. A detailed description of processing steps can be found in Appendix A. The aim of this experiment was to verify whether

there is any measureable re-emission from the film surface during the deposition. If this is the case, then there should be a thin layer underneath the overhang structure. This layer can be only caused by following mechanisms:

Figure 4.4: SEM photograph of the overhang structure prior to deposition.

1. *Surface diffusion* – given the structure dimensions, and temperature involved, it is not possible for adatoms to reach the area under the silicon dioxide beam via surface diffusion. The distance that would have to be travelled by adatoms is too large. Respective SEM photographs shown in the following subsections support this conclusion.
2. *Scattering within the structure* – again, given the pressure involved and structure

dimensions, it is extremely unlikely. Assuming room temperature, 16 mTorr Ar pressure, and the average diameter of Ar atom being $\approx 3 \text{ \AA}$, the total number of argon atoms within the structure is about 4,500 and the fraction of total volume those atoms occupy is only about 1.6×10^{-9} .

3. *Resputtering* will have a certain impact, however, similar experiments indicate, that this effect will not exceed $\approx 4\%$ [82]. (Please see section 4.4).
4. *Low sticking coefficient* now remains as the only possible explanation.

Because the issue of material re-emission is a crucial one, apart from titanium, three other important metallization materials (Al, W, and Cu) were also sputter-deposited at various pressures (4, 10 and 16 mT). The experimental set-up was described in the previous chapter.

4.4 Resputtering

It was suggested, that impact of resputtering will be limited to a few percent under our current experimental conditions [82]. Since resputtering is very sensitive to the working gas pressure, it may be argued that the lower the working gas pressure is, the higher its effect will be. Lower gas pressure also means lower scattering frequency, hence sputtered atoms will retain a larger part of their initial kinetic energy. Following tables shows SIMSPUD predictions for mean free path (mfp), average particle energy at the substrate and number of collisions between target and substrate as a function of energy for the case of collimated and uncollimated sputter-deposition. It can be argued, that even a small decrease in the argon pressure, such as from 4 mTorr to 2 mTorr, can increase average energy of incident particles.

<i>Pressure (mT)</i>	<i>Mfp (cm)</i>	<i>Energy (eV)</i>	<i>Average collisions</i>
0.5	32.76	17.54	0.31
1.0	15.31	15.98	0.66
1.5	9.44	14.50	0.70
2.0	6.42	12.41	1.67
2.5	4.73	11.40	2.36
3.0	3.70	10.20	3.17
4.0	2.42	8.23	5.35
5.0	1.74	6.54	8.27
6.0	1.36	5.35	11.74
7.0	1.10	4.12	16.10
8.0	0.92	3.13	21.00
9.0	0.79	2.91	26.60
10.0	0.69	2.09	32.86

Table 4.1: SIMSPUD prediction of properties of sputtered atoms at the substrate in case of uncollimated sputter-deposition of titanium.

<i>Pressure (mT)</i>	<i>Mfp (cm)</i>	<i>Energy (eV)</i>	<i>Average collisions</i>
0.5	34.59	18.29	0.25
1.0	16.61	16.36	0.54
1.5	10.45	15.31	0.90
2.0	7.46	14.51	1.29
2.5	5.68	14.15	1.73
3.0	4.45	12.81	2.27
4.0	3.00	11.26	3.59
5.0	2.16	9.62	5.37
6.0	1.67	8.31	7.40
7.0	1.34	7.54	9.83
8.0	1.10	6.47	12.70
9.0	.93	6.05	16.10
10.0	0.81	5.45	19.67

Table 4.2: SIMSPUD prediction of properties of sputtered atoms at the substrate in case of collimated (collimator C1) sputter-deposition of titanium.

The comparison of tables 4.1 and 4.2 suggests that the introduction of a collimator into a vacuum chamber seems to induce the effect of the lower working gas pressure and also that the higher the working gas pressure is, the higher the collimator effectivity is (in terms of the entrapment of particles arriving at oblique angles). Films deposited by collimated sputtering should have properties similar to the films deposited by uncollimated sputtering *at lower working gas pressure*.

Another set of experiments was performed with the goal to maximize the resputtering effect. Experimental conditions were similar to those described in the previous chapter, with the following changes:

- *argon pressure* was kept as low as possible – 1.7 mT
- *dc power* was increased to 700 W from original 500 W
- *material* – only titanium was used for those experiments
- *collimator* – only collimator C2 was employed in all experiments

The aim of these changes was to maximize the kinetic energy of sputtered particles.

Both SEM photographs (see fig. 4.5 and fig.4.6) show small titanium clusters deposited under the overhang structure.

Since surface diffusion and scattering inside the structure were ruled out, only remaining possibilities are either resputtering or low sticking coefficient. If the real cause was low sticking coefficient, then this result should be reproducible under similar deposition conditions (described in the section 3.2.1).

Experiments described in the following section will show that this is not true. Results shown in figures 4.5 and 4.6 were achieved only under the deposition conditions

Figure 4.5: Illustration of resputtering - titanium film deposited at 1.7 mTorr argon pressure.

described above. Under any other set of deposition conditions (which invariably meant higher argon pressure and lower dc power) similar results were *never* repeated. Also, it could be argued that low sticking coefficient of titanium would have probably produced more continuous film. The two photographs (see fig. 4.5 and fig. 4.6) convincingly demonstrate the consequence of resputtering in thin titanium films.

Figure 4.6: Illustration of resputtering – detail of the overhang structure.

4.5 Sticking coefficient¹

This set of experiments should determine, whether a value of sticking coefficient for particular material is less than unity. Experiments described in the previous section shown the impact of resputtering. Following experiments will show that the value of sticking coefficient for our vacuum system is very close to unity for titanium, aluminum, copper and tungsten.

All experiments were performed under the deposition conditions described in the

¹Material in this section together with materials in sections 3.2 and 4.6 was presented at VLSI Multilevel Interconnection Conference, June 1993, Santa Clara, CA [84].

section 3.2.1. Collimator aspect ratio was 1.0. Argon pressure was 4, 10, and 16 mT respectively.

4.5.1 Titanium

SEM image of thin titanium film (thickness $\approx 0.6 \mu\text{m}$) can be seen at the fig 4.7. Clearly, there is no layer underneath the overhang structure, which seems to indicate that only a negligible (or none) re-emission is taking place during the film deposition.

Figure 4.7: Titanium film deposited without collimation at 10 mT argon pressure.

Following fig. 4.8 shows the slow decrease of the film thickness corresponding to the growing importance of shadowing by the overhang structure. This supports our previous conclusion, that surface diffusion cannot be the cause of possible film presence

underneath the overhang structure. Both SEM photographs also reveal the columnar structure of the titanium film, which was discussed in the section 2.4.

Figure 4.8: Detail of the decreasing film thickness shows the limited ability of surface diffusion to re-distribute the film into the area below the overhang structure. Titanium deposited at 10 mT argon pressure, no collimation.

4.5.2 Aluminum

Since the melting point of aluminum is much lower than that of titanium ($T_m = 660^\circ \text{C}$ vs. $T_m = 1670^\circ \text{C}$) a different film structure as well as higher surface diffusion length (see fig. 4.9) could be expected. Film thickness is $\approx 0.35 \mu\text{m}$. The film surface is substantially rougher than it was the case with titanium films.

Figure 4.9: Aluminum film deposited without collimation at 16 mT argon pressure.

However, despite the increase in the surface diffusion length, the area underneath the overhang structure remained out of reach. Since aluminum has the lowest melting point T_m among all materials described in this section, contribution of surface diffusion to any possible deposition in the area of interest can be safely neglected.

4.5.3 Tungsten

This is a refractory metal often used as a diffusion barrier, with very high melting point ($T_m = 3406^\circ \text{C}$). It is not surprising that the film structure (see fig. 4.10) is similar to the structure of titanium – it is again zone 1 of the Movchan-Demchisin model (please see fig. 2.8). The film thickness is $\approx 1.0 \mu\text{m}$.

Figure 4.10: Tungsten film deposited with collimation C1 at 16 mT argon pressure.

Tungsten films exhibit the lowest surface diffusion length (for the metals described in this chapter). Tungsten is also one of the few materials commonly deposited by CVD [16].

4.5.4 Copper

Copper is sometimes used as a top metal in VLSI metallization, its melting point is $T_m = 1084^\circ \text{C}$. More often, copper is added to aluminum in order to improve electromigration resistance. Film thickness is $\approx 0.7 \mu\text{m}$.

Figure 4.11: Copper film deposited with collimation C1 at 10 mT argon pressure.

Again, the bottom part of the overhang structure does not exhibit any observable deposition layer.

One of the serious problems of copper films is their poor adhesion to silicon. This effect is shown in the fig. 4.12.

Figure 4.12: Detail of the bottom of the overhang structure. Please note the poor adhesion of copper films to the silicon substrate.

4.5.5 Conclusions

It is now clear, that previous experiments have confirmed our original assumption – that there is no measureable re-emission of titanium atoms from the film surface during the deposition under the conditions described in the section 3.2.1. The value of the sticking coefficient for titanium is for our experimental conditions unity. Naturally, the same holds true for aluminum, copper and tungsten.

The estimated minimal thickness of the film that would be visible by SEM is about 15 nm, which would correspond to the value of sticking coefficient $s = 0.97$. The

experiments have also convincingly shown the differences in the impact of surface diffusion – in soft metals, with low melting point (Al, Cu) was its range much more pronounced as it was in refractory metals with very high melting points (Ti, W). Regardless of surface diffusion length, however, for our experimental conditions the sticking coefficient can be safely taken as one.

4.5.6 Vacuum quality

It can be argued that one of the main reasons for the unity sticking coefficient of titanium (and possibly other metals) is the presence of contaminants in the vacuum chamber. If a partial pressure of the contaminant gas is about 1×10^{-6} Torr, the flux density of contaminants is approximately *equal*² to the flux density of the sputtered material! [64] Since the residual gases are mainly water vapour, CO, N₂, and O₂, there is a significant presence of highly reactive oxygen, which may react with titanium and decrease thus its mobility. This would explain the absence of re-emission attributable to the sticking coefficient in our experiments. To pursue further this direction, the following experiment was performed.

4.5.7 UHV titanium deposition³

This experiment involved the magnetron sputter-deposition of thin titanium film in the Varian M2000 vacuum system. This system is capable of the base pressure $\approx 5 \times 10^{-9}$ Torr, has a circular target with 20 cm diameter, applied nominal power was 3.4 kW, and argon pressure 10 mT. The deposition time was 25 min. Collimator with

²This estimate is valid only for our laboratory system, because it depends on the power supplied to the target.

³This experiment was performed in the E.R. Ginzton Research Center, Varian Associate, Palo Alto, CA.

aspect ratio 1.50 and hexagonal shape was used (this collimator is a standard feature in Varian's manufacturing process). The substrate-target distance was 12.7 cm, collimator height above the substrate was 7.62 cm (this is the distance between the bottom of the collimator and the substrate). The substrate used for this experiment was manufactured in Alberta Microelectronic Centre (it was the same substrate with overhang structures used for the experiments described in the previous section).

Figure 4.13: Titanium μm deposited at Varian Research Center, Palo Alto, CA. Ar pressure was 2 mTorr, collimator aspect ratio 1.5, base pressure $\approx 5 \times 10^{-9}$ Torr.

The result was the same (see fig. 4.13) - no visible layer underneath the overhang structure. This is not to say, that the abovementioned hypothesis is wrong. The hundred times lower base pressure does not necessarily imply hundred times lower

partial pressure of oxygen. There are several possible sources of oxygen, such as desorption from the chamber walls, surface bombardment by Ti-O ions (see fig. 2.6) which have to be considered.

In order to measure the exact partial pressure of oxygen, a sensitive Residual Gas Analyzer [85] would be necessary. Unfortunately, at the time when the experimental part of this thesis was being carried out, such a device was not available.

4.6 Modelling

Even though it was shown that the sticking coefficient of titanium and other materials of interest (W, Al, Cu) is very close to unity for our experimental conditions, it is useful to have software capable of modelling material re-emission.

Figure 4.14: Simulation of thin film deposited onto overhang structure with sticking coefficient $s = 1.0$. Please note the decreasing film thickness. This simulation is in excellent agreement with our experimental results.

Figures 4.14 and 4.15 show the simulated profile of the film deposited onto the overhang structure. The first figure illustrates the case of zero re-emission, while the

second one the effect of 10% re-emission.

Figure 4.15: Simulation of thin film deposited onto overhang structure with sticking coefficient $s = 0.90$. This simulation also shows a substantial deposit beneath the overhang structure, which has never been observed throughout our experiments.

Clearly, fig. 4.15 demonstrates the capability of the SIMSPUD/SIMBAD package to simulate the effect of material re-emission. It is important to realize, that during the modelling both intrinsic re-sputtering and re-emission due to the low sticking coefficient were lumped together.

Chapter 5

Stress

The phenomena of stress is one of the most complex issues associated with thin films. Almost all metallic films are in the state of stress, i.e. they are not in the thermodynamic equilibrium. This can seriously affect other film properties (electromigration resistance, adhesion, density etc.), thus the understanding of stress represents an important (albeit unfinished) part of thin film science [83, 86]. Force produced by stress may in some cases exceed the adhesion force between the film and substrate and the film “peels off” (see fig. 5.1). The character of the force produced by stress may be either *compressive* or *tensile*. If the film is trying to expand alongside the substrate, it is said to be under compressive stress. By convention, this stress has a negative sign. If, on the other hand, the film is trying to shrink, then it is said to have tensile stress, which has a positive sign.

5.1 Stress origin

There are two main components of total accumulated film stress - thermal stress and intrinsic stress. Their importance largely depends on the T/T_m ratio (see fig. 5.2). While thermal stress dominates at large T/T_m ratios, it is usually the intrinsic stress

which is prevalent at low T/T_m ratios. (Please recall the discussion of the zone model in the section 2.4.)

5.1.1 Thermal stress

Thermal stress is a consequence of different thermal expansion coefficients of film and substrate. Because during sputter-deposition the temperature of substrate is elevated as a result of the energy transfer from the bombarding ions and substrate pre-heating, thermal stress develops during the period of the sample cooling, i.e., *after deposition*. (Naturally, the same holds true in case of any subsequent heat treatment, such as annealing).

Figure 5.1: Illustration of peeled off titanium film.

However, since this thesis deals only with high melting point materials, from the point of view of our interest this component of total stress is negligible. A good overview of thermal stress can be found in [87].

5.1.2 Intrinsic stress

This is a very general expression, in fact, the term intrinsic stress covers all stresses except for the thermal one. In some cases the expression *growth stress* is used, because this type of stress is associated with the film structure and - unlike thermal stress - is produced during film deposition [88].

Figure 5.2: Contributions of thermal and intrinsic stresses. [89]

Intrinsic stress is a result of accumulating effects of crystal imperfections occurring

during the film growth. It is thus logical that intrinsic stress is very sensitive to the deposition conditions. It also explains why intrinsic stress is predominant at low T/T_m ratios. An increase in deposition temperature will enhance the diffusion process and this will act as a stress relief mechanism.

5.2 Influence of deposition conditions

There is a vast amount of literature with respect to this particular topic [88, 89], so only the most important conclusions will be presented here.

The most important deposition parameters affecting film stress are:

- working gas pressure and type (see fig. 5.3)
- angle of incidence (see fig. 5.4)
- substrate temperature
- vacuum chamber geometry
- deposition rate

By no means is this list complete, because in such a complex process as a sputter-deposition, virtually anything depends on everything else. This list should rather be taken as a summary of the parameters that can be employed in the process of controlling film stress.

It is known, that most evaporated and sputter-deposited films are in the state of tensile stress. This is usually explained as a consequence of film shrinkage which occurs during the process of film growth. One of the mechanisms responsible for such

a shrinkage could be the volume contraction associated with the process of cluster coalescence [90].

Figure 5.3: Film stress as a function of argon pressure. Normal incidence angle, film thickness $\approx 2,000$ Å, deposition rate ≈ 10 Å/sec. 1 N/m = 1,000 dyne/cm. [89]

Another such process could be the redistribution of crystal imperfections caused, for example, by high deposition rates (adatoms buried prior to their incorporation into lattice would represent one source of such crystal imperfection) [91, 92].

Compressive stresses are believed to be caused by the presence of impurities in the lattice [93]. An example could be argon atoms embedded in the lattice during sputter-deposition. Another mechanism contributing to the compressive stress is atomic peening (see section 4.1).

Effect of argon pressure on accumulated stress is shown at fig. 5.3. Higher deposition pressures yield generally tensile stresses, while low deposition pressures would result in increasing compressive stress. This is consistent with the conclusions described above. For each set of deposition conditions and a particular material, there is a pressure which would yield stress-free film.

Figure 5.4: Film stress as a function of angle of incidence. Ar pressure 1 mT, film thickness $\approx 2,000 \text{ \AA}$, deposition rate $\approx 6.5 \text{ \AA/sec}$. [89]

Increasing angle of incidence results in increased level of tensile stress. Again, this is consistent with our theoretical assumptions. The effect of the atomic peening, which is largely responsible for the compressive stress, will be maximal under normal incidence angle due to the most effective momentum transfer.

Given those facts, collimation should have a significant impact on the film stress. Following sections describe some of the experiments investigating this theory.

5.3 Overhang experiment

Some of conclusions with respect to the pressure effect to the level of accumulated stress in thin films discussed in the previous sections were also confirmed during the experiments described in chapter four. The effect of argon pressure on stress in tungsten films is shown in fig. 5.5 and 5.6.

Figure 5.5: Tungsten film deposited at 16 mT argon pressure.

Fig. 5.6 clearly shows the impact of the compressive stress of the tungsten film on the overhang structure, while similar film deposited at 16 mT argon pressure (see

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NATIONAL BUREAU OF STANDARDS
STANDARD REFERENCE MATERIAL 1010a
(ANSI and ISO TEST CHART No. 2)

fig. 5.5) but at otherwise identical conditions does not exhibit any tendency to shrink nor to expand.

As previously suggested, collimator works as an effective trap for the particles travelling at oblique angles (with respect to target-substrate axis). Elimination of those particles from the vapour phase would presumably increase the fraction of particles arriving at substrate at nearly normal angles.

Figure 5.6: Tungsten film deposited at 4 mT argon pressure is under compressive stress.

This is also supported by SIMSPUD simulations (see fig. 3.5). If this is the case, then the introduction of a collimator into a sputtering system should result in increase in compressive stress. This, indeed, has been observed in case of titanium film deposited

at 10 mT argon pressure with and without collimation. Fig. 5.7 shows titanium film in the state of compressive stress, while film deposited under the same conditions, but without collimator, is in the state of tensile stress (see fig. 5.8).

The normal formula for stress measurement from the beam deflection assumes a thin film deposited on thick beam [94]. This is inappropriate for our experiments since the thicknesses of the film and beam (overhang) are of the same order.

Figure 5.7: Titanium film deposited with collimator C1 at 10 mT pressure is under compressive stress.

To consider these factors and analyze the overhang deflection under planar stress

condition, we use the following multiple-layer stress equation [95, 96]

$$\sigma = \frac{\delta}{3L^2 t_f (t_f + t_s)} \left[\frac{E_f t_f^3}{(1 - \nu_f)} + \frac{E_s t_s^3}{(1 - \nu_s)} \right], \quad (5.1)$$

where E , t , and ν are Young's modulus of the layer, layer thickness, and Poisson's ratio, respectively and with the subscript f denoting the parameters of the Ti film layer and the subscript s denoting the parameters of the SiO_2 layer; δ is the displacement at the end of the overhang; and L is length of the overhang.

Figure 5.8: Titanium film deposited without collimator at 10 mT pressure is under tensile stress.

Substituting the measured δ , L , t , and the bulk¹ values of E and ν into the equation,

¹It has been shown, that the value of Young's modulus of thin film can be replaced by the bulk value [67].

we obtained the approximate stresses listed in table 5.1.

These values, however, were determined from the single experiment, so there may be a significant error margin there. There is also a large error margin caused by the small overhang dimensions and the problems associated with evaluation of exact values of overhang deflection from SEM photographs.

<i>Pressure (mTorr)</i>	2	2	10	10
<i>Collimation (C1)</i>	Yes	No	Yes	No
<i>Film thickness t_f (μm)</i>	0.54	0.44	0.40	0.60
<i>Overhang thickness t_s (μm)</i>	0.40	0.40	0.40	0.40
<i>Overhang length L (μm)</i>	5.2	5.88	3.84	4.2
<i>Overhang deflection δ (μm)</i>	0.32	0.14	0.56	0.80
Total stress (10^9 dyne/cm ²)	-2.15 (compressive)	-0.62 (compressive)	-8.23 (compressive)	9.18 (tensile)

Table 5.1: Stress in titanium films as a function of collimation and argon pressure.

However, these results do confirm the abovementioned hypothesis that collimator introduction increases the level of compressive stress.

5.4 X-Ray Diffraction experiments

In order to study the possible impact of collimation on the lattice spacing and preferred orientation, titanium films were deposited on the flat glass substrate (7059 glass was used) under the conditions described in the section 3.2.1. Argon pressure was 2 and 10 mTorr, deposition time 10 min in case of uncollimated deposition and 40 min in case of collimated one. Collimator C2 was employed for these experiments.

The following table summarizes the results. Graphs from the respective XRD experiments can be found at the Appendix B.

The positions of the peaks (and, hence the spacings between crystal planes) are also affected by the collimation. XRD results (please see Appendix B) show that the peaks in these spectra have been shifted with the deposition conditions. Data obtained from these experiments are summarized in the table 5.2.

The results show that the lattice constants in the collimation films are always higher than those in the conventional films at any given pressure. The lattice expansion is also dependent on the deposition pressures. The changes in the lattice constants indicate the impact of collimation on the film stress. These results support the conclusion, that both collimation and changes in the deposition pressure affect the intrinsic stress of thin films.

<i>Crystal planes</i>	<i>Ideal d ($\text{\AA}$) values</i>	<i>Measured d ($\text{\AA}$)</i>			
		2 mTorr C2	2 mTorr NC	10 mTorr C2	10 mTorr NC
010	2.557	2.563	2.558	N/A	2.548
002	2.342	2.356	2.345	2.349	2.341
011	2.244	2.260	2.244	2.244	2.240

Table 5.2: XRD experiments summary.

The X-ray diffraction spectra indicates that collimation has only a small effect on preferred crystal orientation at a given pressure. However, collimated sputtering increases the compressive stress in the film probably due to enhanced effect of atomic peening. This suggests that collimation can also be used to modify and control physical properties.

5.5 Long silicon dioxide cantilever beams

To further analyse impact of collimation on the accumulated film stress and to verify the preliminary results indicating that collimated films tend to have more pronounced compressive stresses, a following set of experiments involving titanium deposition on the substrate featuring long silicon dioxide beams was performed. These beams are commonly used in accelerometer sensors manufactured by the Alberta Microelectronic Centre. They were 80-260 μm long, 30 μm wide and 2.2 μm thick.

The experimental set-up was described in the section 3.2.1. Titanium was deposited at 2, 4, 6, 8, and 10 mT, respectively with and without collimation. Collimator C1 was employed. Together with a beam structure, a standard overhang structure was placed on each respective sample-holder. SEM photograph of each overhang sample was taken in order to measure film thickness. The deflection of the silicon dioxide beams was measured by the depth of the field of a standard optical microscope. At least four beams from each chip were measured and the stress was calculated from the formula 5.1. The value for total accumulated stress for each chip was then taken as average value of those measurements.

<i>Pressure (mTorr)</i>	<i>Stress (10^9dyne/cm^2)</i>	<i>Deposition rate ($\text{\AA}/\text{sec}$)</i>
10	5.36 ± 0.24	1.81 ± 0.10
8	4.93 ± 0.21	1.94 ± 0.10
6	3.83 ± 0.32	1.88 ± 0.10
4	0.27 ± 0.25	1.92 ± 0.10
2	-2.17 ± 0.21	1.83 ± 0.10

Table 5.3: Stress transition in titanium films deposited with collimator C1 and respective deposition rates.

This experiment was also used to evaluate the deposition rate at each set of deposition conditions. Data are summarized in the tables 5.3 and 5.4.

The results convincingly show that films deposited by collimated sputtering exhibit a larger degree of compressive stress than is the case with conventionally deposited films. The only exception is the experimental result achieved for 10 mTorr argon pressure. This may either be an error, or there well may be yet another effect that was not observed at lower gas pressures. From the manufacturing point of view, however, it is the pressure range between 2 and 8 mTorr² which is the most important. And in this region of interest, the results clearly demonstrate the collimator impact on the film stress.

<i>Pressure (mTorr)</i>	<i>Stress (10^9 dyne/cm²)</i>	<i>Deposition rate ($\text{\AA}/\text{sec}$)</i>
10	4.69 ± 0.58	4.77 ± 0.25
8	5.81 ± 0.83	5.30 ± 0.25
6	4.92 ± 0.34	6.50 ± 0.25
4	3.58 ± 0.17	6.10 ± 0.25
2	3.31 ± 0.38	6.67 ± 0.25

Table 5.4: Stress transition in titanium films deposited without collimator and respective deposition rates.

Numerical results from the tables 5.3 and 5.4 are shown in the fig. 5.9, which can be compared to the fig 5.3. Results obtained from this experiment confirm the conclusion that the lower the gas pressure is, the higher the level of compressive stress (or the lower is the level of tensile stress). The introduction of a collimator into a vacuum chamber will also cause the increase in the level of compressive stress, i.e., it will induce the "lower argon pressure effect" already suggested in the previous chapter.

²This is the common pressure range in Varian's manufacturing process.

This may be due to an increased impact of atomic peening. Since the collimator will remove the particles with oblique incident angles, fast argon neutrals will likely undergo less collisions and once they hit the substrate, they can transfer more kinetic energy increasing thus the effect of atomic peening.

Figure 5.9: Stress transition in titanium films as a function of argon pressure and collimation. Error bars are double the standard deviation.

Results described in this chapter demonstrate the impact of the collimator on the accumulated intrinsic stress of the film. They also show that collimator can be used as an effective tool for the process of stress-engineering of thin metal films.

Chapter 6

Conclusions and suggestions

6.1 Conclusions

This thesis has explained the fundamental principles of collimated sputtering, a technique which is already used in commercial processing [97, 98].

It has been shown that films deposited by collimated sputtering can achieve much higher bottom coverage, which is an important advantage as device feature shrink below $0.5\ \mu\text{m}$. Films deposited by collimated sputtering also exhibit improvement in the film density at the *via* corner (please see fig. 1.1), which represents one of the frequent points of the diffusion barrier failure.

On the other hand, a substantially lower deposition rate is a major limitation of this technique. However, for the titanium films deposited as a part of a Ti/TiN diffusion barrier this does not pose any problem due to their small thickness, which is mostly below 100 nm. For other applications this problem could be alleviated by an increase in the dc power supplied to the target.

The SIMSPUD/SIMBAD software package was successfully used for the simulation of the experimental data. This simulator was used for the first time to model films

deposited by collimated sputtering. A set of experiments was specifically designed in order to measure the fraction of atoms re-emitted from the film surface. It was shown, that for our depositions condition no such re-emission is taking place for titanium, aluminum, copper and tungsten.

The principle effect of collimation is thought to be the entrapment of particles with high lateral velocities, i.e., a collimator acts as a *filter* for particles arriving at oblique angles (with respect to target-substrate axis). Those are the particles which presumably underwent more collisions. So, only the particles, whose trajectory does not differ too much from target-substrate axis are allowed to reach substrate. Since these particles did not undergo too many collisions, they retain a proportionally larger amount of their original kinetic energy.

In other words, films deposited by collimated sputtering have grown mostly from the atoms which have retained a proportionally larger fraction of their initial kinetic energy. Hence, their microstructure will reflect this situation. Since more energetic atoms are also a result of a decrease in working gas pressure, it can be expected, that a collimation will introduce a similar effect. Moreover, collimator will not affect fast argon neutrals which have significantly contribute to the film bombardment. In fact, by entrapping the particles arriving at oblique angle, collimator will effectively *increase* the effect of argon bombardment of the growing film.

Both these effects, i.e. an increased argon bombardment and a higher kinetic energy of incident particles will contribute to the increase in a compressive stress and alter the film microstructure. This has been experimentally proven in the chapter five.

6.2 Suggestions for future research

Surface microroughness, also called a *ripple effect* is a result of small distance of the collimator bottom from the substrate. Area directly under the collimator vanes receives proportionally less flux, and a result is an irregular film thickness over the wafer.

Argon content in the films deposited by collimated sputtering could be higher compared to conventionally deposited films. This has also the direct impact on the level of compressive stress.

Sticking coefficient vs. oxygen partial pressure is another crucial issue with respect to the material re-emission. An UHV system and a sensitive RGA (Residual Gas Analyzer) is necessary to determine whether there is a relation between those two phenomena.

Poor adhesion at the via top corner is commonly observed in films deposited by collimated sputtering. Most films exhibit a small “crack” which can become the place of a diffusion barrier failure. Such a crack was not observed in conventionally deposited films. It is believed, that it is a direct result of a geometrical shadowing.

Re-emission from the collimator walls may be an important factor which has to be considered in case of low sticking coefficient. However, this effect can be for our deposition cases safely neglected (as is shown in chapter four).

Material accumulation on the collimator top is also a serious problem. Continuous collimator use will result in the film growth and alter thus geometrical dimen-

sions of collimator, which in turn can lead in changes in the film properties.

Although there are some problems associated with the collimated sputtering, it is a perspective deposition technique which can significantly improve conformal coverage of Ti/TiN diffusion barriers¹. Experimental data shown in the chapter five demonstrate the possibility of "stress engineering" wherein a collimator dimensions and position can be used as variables.

Experiments described in this thesis support the viability of the collimated sputtering as a deposition technique which can improve the bottom coverage in sub-micron microelectronic circuits. This technique can be also employed at engineering of multi-layer films with minimal stress. Moreover, this thesis reports a successful attempt to simulate microstructure of films deposited by collimated sputtering using the software SIMSPUD/SIMBAD simulation package.

¹Collimated sputtering has been so far implemented only into commercial processes for the Ti/TiN barrier deposition [97, 98].

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Processing steps for overhang structure

1. Plain Silicon Wafer $\langle 100 \rangle$ (cross-section)

2. Thermally grown Oxide (0.5 micron)

3. PR deposition

4. Patterning

5. Development

6. Buffered Oxide Etch (removes oxide but leaves PR)

7. PR stripping (acetone)

9. Thin film deposition

XRD spectra

Following pages show XRD spectra obtained from experiments described in the section 5.4. A more detailed description of this experiment can be found in [79].

All films were deposited on the 7059 glass. Respective codes denote the material, deposition pressure and collimation, respectively. Thus, a spectra marked T2C2 shows the result for titanium film deposited at 2 mTorr argon pressure with a collimator C2, while a spectra marked T10NC describes the result for titanium film deposited at 10 mTorr argon pressure without a collimator.

Z04703 2/23/93 S= 0.050 T= 1.000 T2NC, T.JANACEK

Z04705 2/23/93 S= 0.050 T= 1.000 T2C2. T.JANACEK

Z04706 2/23/93 S= 0.050 T= 1.000 T10NC, T.JANACEK

Z04704 2/23/93 S= 0.050 T= 1.000 T10C2. T.JANACEK

END

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