Development of low roughness, low resistance bottom electrodes for tunnel junction devices

Designing and assembly of a new annealing setup for 150mm wafers

David Filipe Coelho de Almeida Aurélio

Setembro de 2007

1. INTRODUCTION

Deposition of complex structures such as spinvalves (SVs) or magnetic tunnel junctions (MTJ’s), require excellent film grain growth control, and its structural stability upon annealing, is of major importance for device integration. In particular, devices operating in a current-perpendicular-to-plane (CPP) geometry use low resistance films to meet the increasing area densities, which requires low resistance contact electrodes, (in series with the sensor stack), to avoid current crowding effects. The choice of these electrodes is based on aluminum (Al), copper (Cu), or gold (Au) films, which are usually thick (above 100 nm) and rough.

State of the art

Conventional metallization processes at INESC-MN uses 300 nm thick Al films deposited by high rate magnetron sputtering, in up to 150 mm wafers substrates. Although deposited at high rate, 60nm thick Al films have been successfully used in the bottom contacts, after grazing ion milling smoothing which reduces its roughness, (height RMS) from 1,2 nm down to 0,2 nm. However, and despite the low surface roughness of these buffers, high-resolution transmission electron microscopy (HRTEM) analysis of tunnel junctions shows local defects at the oxide barrier induced by the grain boundaries of the Al buffers underneath.

Objectives

In this work it was studied the optimization of the deposition conditions of the Al bottom electrodes in a magnetron sputtering system, (Nordiko 7000) in order to reduce its roughness through the method of insertion of thin layers of other materials during the Al film deposition in order to avoid grain growth (lamination).

A new annealing system able to anneal 150 mm wafers up to 400°C, is to be developed during this work, in order not only to test the annealing properties on the buffers developed, but also to be able to anneal big wafers in a static system, until the date inexistent at INESC-MN were this work was developed.

Fabrication of magnetic tunnel junction devices incorporating laminated and non-laminated Al films, in order to compare the effectiveness of these buffers as low resistance, smooth materials for CPP devices.

2. THEORETICAL BACKGROUND
Grains

A film deposited by a physical vapor deposition method is often composed by many small polygons denominated grains, delimited by surfaces named grain boundary. These boundaries have an interface energy, and represent a metastable state, which can be dislocated by the effect of temperature, and are the privileged places for the segregation of impurity atoms, and also constitute an obstacle to the propagation of dislocations. Therefore these structures play an important role in the mechanical proprieties of the polycrystal.

Each grain is in fact a single crystal, and the orientation of each grain in the crystal lattice is for most materials different, and the material is thus designated polycrystalline.

Grain Growth

Grain growth refers to the grain size increase in a material, usually due to a thermal treatment of the material. However in this section it will be referred as the polycrystalline structure growth of thin films as a result of physical vapor deposition, where each grain deposited might grow more or less than the grains at its borders depending on the deposition conditions. This difference in grain growth will be reflected as the overall roughness of the deposited film.

Film growth is influenced by many factors, like the nature of the deposited material, deposition power, pressure, temperature and substrate in which it is grown. The combination of these different factors is what determines the crystal structure of the end film.

On this work it was searched the optimal deposition conditions in a magnetron sputtering system (Nordiko 7000), for the bottom electrode, usually denominated as buffer, in order to reduce the differences between grain boundaries thus reducing the buffer roughness.

Roughness

It is a measurement of the small-scale variations in the height of a physical surface. This is in contrast to large-scale variations, which may be either part of the geometry of the surface or unwanted waviness.

Roughness is sometimes an undesirable property, as it may cause friction, wear, drag and fatigue, but it is sometimes beneficial, as its texture allows surfaces to trap lubricants and prevents them from welding together. It is measured in different ways for different purposes.

Two of the most commonly used are;

- Average roughness (Ra); which represents the average height of the bumps on a surface.
- Root mean square (RMS) roughness; which is the statistical measure of the height of the bumps, which gives a somewhat larger value than Ra. It is given by:

\[ x_{rms} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} x_i^2} = \sqrt{\frac{x_1^2 + x_2^2 + ... + x_n^2}{n}} \quad (2.1) \]

The measurements of the film roughness done during this work were made using an atomic force microscopy technique, and the dimension used the height RMS of the bumps.
**Annealing**

Annealing, as defined in metallurgy and materials science, is a heat treatment wherein a material is altered, causing changes in its properties such as strength and hardness. It is used to induce softness, relieve internal stresses, refine the structure and improve cold working properties.

The annealing process is characterized by three stages, with the first being the recovery phase, in which results the softening of the metal through removal of crystal defects and the internal stresses which they cause. The second phase is denominated recrystallization, where new grains nucleate and grow to replace those deformed by internal stresses. If annealing is allowed to continue once recrystallization has been completed, grain growth will occur, in which the microstructures start to coarsen, since further reduction in the internal energy can only be achieved by reducing the total area of grain boundary. This may cause the metal to have less than satisfactory mechanical properties.

### 3. LAMINATED ALUMINUM BUFFERS

**Introduction**

In order to try and achieve the desired low roughness low resistance bottom electrodes (buffers), for MTJ, various different deposition conditions were tested using the lamination method, where the standard aluminum bulk buffers were intercalated with thin layers of a different material. Titanium tungsten nitride (TiWN), was the material available at the machine to use as the thin laminating layer material.

It was used a high rate magnetron sputtering deposition device, (Nordiko 7000), to deposit the buffers, whose deposition conditions varied in order to optimize the buffers to low roughness low resistance films, were the DC power source, the deposition time, (and hence the thickness of the film), and the type of lamination.

**Experimental method and analysis**

Since different deposition powers were to be tested in the magnetron sputtering machine, calibration samples were first deposited on one inch glass substrates, in order to assert the different deposition rates and see if they are linear as expected.

The deposition conditions for the referred sputtering system usually used at INESC-MN for the MTJ aluminum buffers are the following:

- Injection of 50 sccm Argon gas flow for plasma creation.
- Chamber pressure of 3 mTorr.
- DC deposition power of 2000 watts.

With these conditions the corresponding thickness is of 600 Å, when depositing for 12 seconds, which results in a typical rate for a high deposition sputtering machine of ~50 Ås⁻¹.

Throughout this work the deposition conditions used for aluminum, were the same as the ones previously referred, except for the deposition power and times, which were the variables changed in order to discover the deposition conditions that would result in the lowest roughness and resistivity films. In the deposition of TiWN layers, it was always used an
injection of 50 sccm argon plus 10 sccm of nitrogen gas flow for the plasma creation, with a chamber pressure of 3mTorr, and a DC deposition power of 500 W.

**Buffer resistivity**

Knowing the deposition rates for each of the deposition powers to test, several thickness and lamination types were tested, in order to discover which set of deposition conditions results in the lowest roughness films.

First however, it would be showed the resistivity results for those conditions, since it is desirable low roughness films but not at the cost of much higher film resistance.

The lamination of the films was made by first depositing a layer of Al of a given expected thickness, followed by a thin layer of TiWN in order to prevent the Al grain growth, and repeating this process until the desired total thickness of the buffer. The buffer was always finished with a layer of TiWN, since it helps protects the film from oxidation.

Due to the TiWN high resistivity, only thin layers were deposited, (of either 30 or 35Å).

The results of the resistivities vs Al laminated thickness of all the different buffers deposited are plotted in the next graph.

![Resistivity vs Lamination type](image)

**Figure 1 - Example of the lamination type**

The results of the resistivities vs Al laminated thickness of all the different buffers deposited are plotted in the next graph.

**Figure 2 - Resistivity dispersion for the different deposition powers and lamination types.** The Al laminated thickness represents the thickness of Al on each layer of the buffer. The values with Al laminated thickness higher than 1000Å represent control samples of non-laminated Al buffers.

By observing the previous results, it is possible to conclude that for a thicker layer of Al in the buffer, the resistivity decreases as expected since the TiWN has a higher resistivity than Al, so when the percentage of Al to TiWN increases the resistivity decreases. Thus in the
search for low roughness buffers one has to consider that for a 30 to 35 Å layer of TiWN one should use a layer of Al with a thickness around 400Å or more, in order not to raise the resistivity too much in respect to the non-laminated aluminum buffers previously used.

**Buffer roughness**

The roughnesses of the films were analyzed by AFM (model Veeco/DI Dimension 3100) using silicon nitride tips (OMCL-AC160TS-W from Olympus), and the resulting data treated with the software Gwyddion 2.4.

The next graphs show the dispersion values of the sample’s roughness in respect to the deposition power and lamination type.

![Graph showing the results of the laminated films and of the bulk aluminum films](image)

*Figure 3 - Roughness values for each type of lamination; a) Graph showing the results of the laminated films (left side), and of the bulk aluminum films*

By interpretation these results one can see that by laminating the films the roughness lowers as expected, the only exception are in some samples deposited at 200 W. Another result that can be taken is that it seems that for a higher deposition powers (until a certain point) the roughness is lower. The best lamination parameters in terms of roughness are the films laminated with, more or less 400Å of aluminum thickness, deposited at 1900 and 2000 watts.

**Magnetic tunnel junction (MTJ) results**

Four samples with an identical MTJ structure, were processed, in a Nordiko 3600 ion beam system. On each of the samples a different buffer was deposited above the glass substrate, with the objective of asserting if the lower buffer roughness implies better TMR signals. (One MTJ deposited directly on a glass substrate without the buffer; One with a standard aluminum buffer of 600Å thick, deposited by the magnetron sputtering system, Nordiko 7000, with the following conditions; DC source 2kW at 3mTorr with 50sccm of Ar, deposition time 12 seconds; One with the standard aluminum buffer with the same deposition conditions as the previous one, but heated in vacuum in the same machine one a different module up to 450ºC
for 2 minutes followed by a heat treatment at 150ºC for 30 minutes. The buffer was then taken from vacuum to be submitted to a ion beam smoothing in a 30º pan for 60 seconds, in the Nordiko 3600 system; One of the samples was deposited with the one of the best laminated buffer obtained, in terms of film roughness.

The full structure of the junction deposited is illustrated in the next figure along with a statistical box chart of all the MTJ measured on each sample in terms of tunneling magneto resistance (TMR) versus type of buffer used in order to try and assert which is the best buffer.

![Figure 4 - Deposited MTJ structure and statistical box chart, TMR vs Type of Buffer, whose interpretation helps in the determination of the best buffer.](image)

**Discussion**

In terms of lowering the buffer roughness, it was possible to reduce the bulk Al roughness from ~1.2nm to ~0.62 nm height RMS, using the lamination method. It was also showed that exists a small range of optimal deposition powers that give lower film roughness. For the Nordiko 7000 sputtering system used, this range was determined to be between 1900 and 2000 watts. With the machine’s available target materials, the lamination of two layers of ~400Å Al /~30Å TiWN, is the one which gives the lowest roughness, with the advantage of not increasing the buffer resistivity to much due to the presence of TiWN. One has to keep in mind that the discovered optimal deposition conditions are for the tested machine, since the internal geometry, like cathode–anode distance and dimensions, influence the plasma and thus the deposition.

Comparing the TMR distribution and statistical analysis of the different samples, it is possible to conclude that the buffer which contributes with a better uniformity that results in more MTJ’s with higher TMR is the ion milling smoothen buffer sample. The sample with the laminated buffer developed during this work revealed to be only slightly better than the
sample with the bulk aluminum buffer, having a higher TMR average, for the measured MTJ’s.

Since the roughness of the laminated buffer is about half of the a bulk Al buffer, it would be expected that the uniformity in terms of more MTJ with higher TMR, would be more evident in the sample with the laminated buffer. The reason for this might be due to problems during the junction’s process that somehow affected more the laminated buffer.

4. NEW ANNEALING SYSTEM FOR 150mm WAFERS

Introduction

A new annealing system able to anneal 150mm wafers up to 400°C, was developed during this work, in order not only to test the annealing properties on the buffers developed, but also to be able to anneal big wafers in a static system, until the date inexistent at INESC-MN. It was used an already existing permanent magnet with 0.8 Tesla bought to this end.

Annealing system for nanotechnology applications

Besides the beneficial characteristics of annealing a material already seen in 2.5, in device applications like MTJ it is known that with the help of a strong external magnetic field during the annealing process, in order to force the desired magnetization into the magnetic layers of the junction, thus optimizing the spin polarization at the barrier interfaces, the TMR increases significantly. The overall device resistance after the annealing process also diminishes, due to the oxygen redistribution by the effect of temperature, which usually flows into the MgO barrier thus reducing the resistance.

In these applications the annealing is usually done in a high vacuum to prevent the oxidation of the devices during the process.

![Annealing schematic](image)

Figure 5 - Annealing schematic used in device anneals. The heating stage is done slowly, ~5°C per minute, in order to guarantee a homogeneous heating of the sample and to avoid the overshoot of the desired annealing temperature. The annealing stage is done at a sufficient temperature and time so the desired stages described in section 2.5 can take place. The cooling down stage is normally let to run freely until the samples reach the room temperature.

Ideas and technical difficulties

Note: All of the drawings were done using the computer programs, Autocad 2004 (2D) and Autodesk Inventor Professional 8 (3D and final 2D schematics)

Various ideas and difficulties arose during the development of the annealing system until its final apparatus, where the choice of non magnetic materials due to the magnet’s strong field,
and the magnet’s cooling method due to it’s demagnetization temperature were among the principal concerns.

Having already a permanent magnet to use with the following characteristics:

- Demagnetization temperature: 80°C
- Magnetic field: 0.8 Tesla

![Permanent magnet used, with corresponding dimensions in mm](image)

It was desirable to maximize the number of wafers that could be annealed in one run. The effective area inside the magnet where the magnetic field is the strongest and most uniform is in the center of the hole with a height of 20mm, (blue area in the previous figure), which could accommodate four wafers in a suitable cassette, which was drawn to that end.

Several sketches were made for the system; the first one’s being composed of separated modules in vacuum, with a load lock, heating chamber and cool down inside the magnet, however and despite the advantage of not having to cool down the magnet, this ideas were abandoned to the automatisms that had to be incorporated into the system, and do to the fact that it was desirable to have a static system, were the annealing process followed by the cool down would take place inside the magnet, since some of the materials currently being used in the MTJs at INESC-MN seem to be sensible to the temperature gradient from taking the samples from the heating module to the magnet for the cool down.

So a simple design was made incorporating the heating module inside the magnet that now could only anneal one 150mm wafer.

Has for the heating source, it was first designed a system composed of halogen lamps, however they presented several problems like, non-homogeneities, non-existing of the necessary ceramic supports or inadequate size. So the heating source that was finally chosen was a 1000 W inductive coil resistance similar to the ones used on cooktops plates, since it solves the problems seen in the other sources. They exist in the market for a low price coming with a respective ceramic support and the resistance wire is made of a non-magnetic alloy of Cr20%Ni80%. Above this heating source a copper disc is laid to allow homogeneous heat conduction up to the silicon wafer.
Since the heating of the wafer is to be done inside the magnet up to 400ºC, a cooling system needed to be implemented in order to avoid the demagnetization of the magnet. The cooling system chosen was a water-cooling system composed of a double copper spiral.

A vacuum chamber was also drawn to be implemented into the system to avoid the oxidation of the devices during the annealing process.

The final apparatus of the system is presented in the following figure.

To power the inductive resistive load it is used a Eurotherm Power Thyristor unit Model 461, and for the temperature control, a Eurotherm PID, (proportional-integral-derivative) Temperature Controller Model 3204. Next is presented the electrical circuit used to power and control the inductive resistive load.

Discussion

All the technical difficulties were surpassed, and the annealing system is fully projected. To test the system first it is necessary to confirm if the signals of the thyristor unit and temperature controller are correct, then a test run should be done outside the magnet’s to ensure all is working well, including the water cooling system. During this test the water
temperature should be measured to ensure that when the copper disc is at its highest
temperature (~400°C), the water’s temperature does not exceed a the desired cooling
temperature, since the magnet's demagnetization temperature is 80°C.

Since the materials of which the thermocouple is made are sensible to the magnetic field
of the magnet, a calibration is needed to determine the error of the thermocouple’s
measurement while immerse inside the strong magnetic field. Thus a test run should also be
made inside the magnet and with the help of an infrared thermometer the calibration curve
can be determined.

Until the date however it was not possible to test the projected system (except the heating
using the thyristor module to heat up the resistance, which has been done successfully) due
to delays in some of the necessary parts, in particular the cooling spiral without which the
annealing system cannot be used. The vacuum chamber was yet to be ordered due to it's
cost, but the annealing process can be done without it, being only necessary to protect the
samples from oxidation by other means.

5. CONCLUSIONS

In respect to the development of low roughness low resistance buffers for device
applications, it was possible to optimize the deposition conditions for the Nordiko 7000
magnetron sputtering system in such a way as to lower the buffer roughness from ~1,2 nm to
~0,62 nm height RMS, without considerably raising the buffer resistivity. However it was not
possible to reach the desired 0,3nm of roughness, already possible to obtain using a grazing
ion milling smoothing technique [5], nevertheless the grain growth of the buffers was
improved by use of the investigated lamination deposition conditions.

When applying the low roughness laminated buffers in the MTJ devices process it
revealed not to be better than the pre-annealed buffer grazed by ion milling, in terms of the
uniformity in the TMR signal, but better than the sample with the bulk Al buffer and the
sample without buffer. The fact of not having more junctions with higher values of TMR using
the laminated buffer, might be due to problems during the junction process and not
exclusively because of the buffer topography. The ion milling technique and the pre-anneal
should be implemented into to the laminated buffers in order try to improve the buffer.

Concerning the projected annealing system for 150mm wafers, all the technical difficulties
were surpass and the final apparatus is presented. It was not possible to go into the test
phase of the system, beyond the test of the thyristor module and controller due to delays in
the process of some of the necessary parts of the system.