Magnetron discharge volt-ampere characteristic investigation at thin film coating process

N F Kashapov, A G Luchkin and G S Luchkin
Kazan (Volga Region) Federal University, 18 Kremljovskaja str., Kazan 420008, Russian Federation
E-mail: AGLuchkin@kpfu.ru

Abstract. Magnetron discharge at reactive and working gases mixture atmosphere current-voltage characteristic (I-U) for different sputtering parameters is investigated. It is shown, that form of volt-ampere characteristic doesn’t depend on gas supply scheme at vacuum chamber pressure 4-6·10⁻² Pa. Reactive gas (oxygen) flow increasing leads to making I-U transition part wider and amplification of difference between top and bottom parts of hysteresis loop I-U. Discharge voltage is less at oxygen and argon gases mixture atmosphere than at argon atmosphere.

1. Introduction

It is well known that magnetron sputtering is applied for making functional coatings including biocompatible on different materials [1-4]. Example of biocompatible coatings are thin films of titanium and it’s compounds (TiOₓ, TiN, etc.) [5,6]. TiOₓ coatings can be made by titanium oxide sputtering at argon atmosphere or by titanium sputtering at oxygen and argon gases mixture atmosphere. During metals magnetron sputtering at oxygen and argon gases mixture atmosphere on metal target surface appears oxide thin film [7], and volt-ampere characteristic has hysteresis shape [8,9].

Experimentally determined that depending on target surface oxide covering degree there are several sputtering regimes. If target surface totally covered by oxide film, metal oxide parts are sputtered. If target surface covered partly – metal parts. Transition between regimes is uncontrolled. Difference between regimes is well shown at shape of I-U – it is hysteresis. There some methods to avoid hysteresis [10]. Also there is opportunity to use both regimes.

I-U hysteresis has metallic, transition and oxide parts. For sputtering at both regimes it is necessary to investigate transition part of I-U hysteresis. The article aim is to determine discharge parameters influence on transition part of I-U hysteresis.

2. Experimental plant and methods

Functional schema of experimental plant is showed at Pic. 1. It consists of: Vacuum chamber 1, door 2, pump system 3, two magnetrons 4, power supply of magnetron and source of ions 5, system of gas input, system of photometric control of film thickness that include spectrophotometer S-100, moving system 8 of substrates 9, heating system 10, windows 11, valve for atmosphere air 12, filled-system transducer 13, cooling system 14, ion source 15 [11,12].

There is control stand for all systems. Pumping system includes mechanical pump 2NVR90B and diffusion pump ND-200 and provides Vacuum at chamber about 10⁻³ Pa. Pressure registration system consists of vacuum-gauges VT1-4 (thermocouple method) and VMB1-2.
(ionization method). Gas input in chamber realized by leak valves VTT02.01.31.000 and controller RRG-8. Gas flow counted by constant volume method [11].

![Functional schema of experimental plant](image)

Pic. 1 Functional schema of experimental plant: Vacuum chamber 1, door 2, pump system 3, two magnetrons 4, power supply of magnetron and source of ions 5, system of gas input, system of photometric control of film thickness that include spectrophotometer S-100, moving system 8 of substrates 9, heating system 10, windows 11, valve for atmosphere air 12, filled-system transducer 13, cooling system 14, ion source 15.

Current-voltage characteristic measured for different reactive and working gases ratios. Plant permits two cases of gas supply: into target region (schema “1”) and into substrate region (substrate “2”). Discharge parameters were: voltage from 100 to 700 V, current density from 4 to 24 mA/sm$^2$, chamber pressure 0.1 – 0.8 Pa. Target material – titanium, argon flow 0.4 – 1.2 mg/sec, oxygen flow was 0.2 – 0.7 mg/sec.

3. Results and discussion

Discharge $I$-$U$ characteristics at oxygen and argon atmosphere (gas supply schema “1”) are shown at Pic. 2. Oxygen flow into vacuum chamber was 0.4 и 0.7 mg/s. Pressure in vacuum chamber was 4·10$^{-2}$ Pa. Current density was from 4 to 20 mA/sm$^2$, voltage increased from 200 to 600 V.

At increasing current density of discharge at oxygen and argon atmosphere (“O$_2$+Ar ↑”) voltage was less than in case of discharge at argon atmosphere (“Ar ↑”). Transparent oxide films was coated at this $I$-$U$ region, because oxygen presence caused oxide film on magnetron target surface and discharge power was not strong enough to sputter it. This regime ended at current density about 18 mA/sm$^2$, when high discharge power caused full sputtering of oxide film on magnetron target surface. Than began metal (elemental) regime (metal film sputtered). Metal regime voltage deviation from sputtering at argon atmosphere was insignificant. Therefore were identical processes. At decreasing current density (“O$_2$+Ar ↓”) was reverse transition at 8 mA/sm$^2$. 
Oxygen flow increasing (Pic. 2, b) didn’t change qualitatively $I$-$U$ form, but increased difference between metal and oxide regimes and transition region. Transition to metal regime was at 20 mA/sm$^2$. Voltage of oxide regime was lower 500 V.

Discharge $I$-$U$ characteristics at oxygen and argon atmosphere (gas supply schema “2”) are shown at Pic. 3. Oxygen flow into vacuum chamber was 0,4 и 0,7 mg/s. Pressure in vacuum chamber was 4-6·10$^{-2}$ Pa. Current density was from 4 to 24 mA/sm$^2$, voltage increased from 200 to 680 V.

Oxygen flow leads to lower voltage (30-50% less) at current increasing and to hysteresis appearing (Pic. 3). At discharge current increasing oxide deposits. Transition to metal regime was at current density 12 mA/sm$^2$. Reverse transition to oxide coating deposition regime at decreasing current density was at 6 mA/sm$^2$. 
Pic. 3 I-U for gas supply schema «2»:
a), b) – argon (flow 0,4 mg/s) and oxygen (flow 0,4 and 0,7 mg/s respectively) mixture

Oxygen flow increasing in 2 times (Pic. 3 b) didn’t change I-U from qualitatively, but voltage difference between metal and oxide regimes enlarged. Transition state also increased. Transition to metal regime was at 14 mA/sm². Voltage of oxide regime was lower than 400 V.

Therefore reactive gas (oxygen) flow rate significantly effects on appearing I-U hysteresis loop and on metal magnetron sputtering ability at presence of reactive gas in vacuum chamber.

4. Conclusions
Magnetron discharge at reactive and working gases mixture atmosphere I-U characteristic for different sputtering parameters is investigated. It is shown, that form of volt-ampere characteristic doesn’t depend on gas supply scheme at vacuum chamber pressure 4-6·10⁻² Pa. Reactive gas (oxygen) flow increasing leads to making I-U transition part wider and amplification of difference between top and bottom parts of hysteresis loop I-U. Discharge voltage is less at oxygen and argon gases mixture atmosphere than at argon atmosphere.

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