Multilayer Cu-based Films for the Gyroscope Housings of the Gravity Probe B Relativity Mission

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Abstract

Multilayer Cu-based films have been manufactured using magnetron sputtering on fused quartz substrates including the gyroscope housing electrodes. A 100 nm Ti layer was used to provide good adhesion to the fused quartz. A 200 nm Ti layer was used to overcoat the Cu film, to reduce electron emission in high electric fields and to protect the easily corroded copper film. The seven layer Cu-based films, with smooth surface, low stress, good adhesion and low resistivity have been successfully applied to the electrodes of the gyroscope housings. The gyroscopes have been spun to 170 Hz for more than 100,000 hours testing.

Introduction

In recent years, copper has been investigated as an attractive material for interconnection metallization. It has the advantages of low resistivity, high thermal conductivity, and excellent electromigration resistance. Unfortunately, copper has poor adhesion to quartz substrates and it is easily corroded. So there are still many critical issues which remain to be resolved in the development of copper-based metallization.

In previous papers (1,2), we reported that multilayer Ti-Cu films have been successfully applied to the Gravity Probe Relativity Mission Gyroscopes housing electrodes. The Gravity Probe B Relativity mission (GP-B)(3) is a general relativity experiment to measure the frame dragging and geodetic relativistic precessions in a 650 km polar orbit with an accuracy of 0.3 marc-s year^{-1}, using orbiting gyroscopes. The GP-B spherical gyroscopes are made of fused quartz or single crystal silicon and coated with Niobium. The gyroscopes are electrostatically suspended and spun to 170 Hz in the quartz gyroscope housing at 2 K, and in ultrahigh vacuum(10^{-9} Pa). Figure 1 is a schematic view of an exploded gyroscope rotor and its housing.

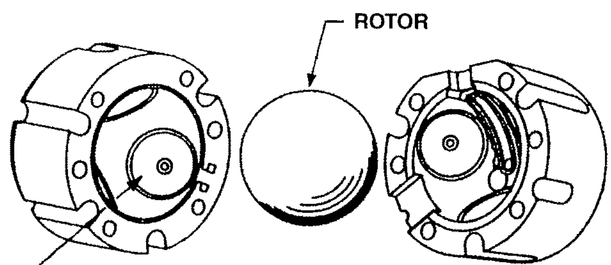

Fig. 1. Exploded view of a gyroscope rotor and its housing.

The quartz gyroscope housing has three orthogonal pairs of circular electrodes which are coated with multilayer Ti-Cu thin films. The requirements for these electrode films are: (1) good adhesion to the quartz substrate, (2) good thermal and electrical conductivity (3) resistance to arcing damage for an arc energy of less than 3×10^{-4} J, (4) low tensile stress in the film (5) superconducting transition temperature of less than 1.5 K, (6) Film uniformity of better than 10% over the electrode surface, (7) low field emission currents (< 1 pA) in fields of 3×10^{7} V/m. These requirements insure the mechanical and electrical integrity of the gyroscope housings under the temperature range from 2 K to 300 K, and voltages ranging from 20 mV to 2,000 V.

The copper-based film deposition processes, metal cleaning processes, usage of copper for advanced interconnection metallization, and Cu-based film low resistivity charac-terization are discussed in this paper.

Experimental

Sputter deposition is used to coat the flat, fused quartz sample substrates and fused quartz gyroscope housing for the suspension electrodes. Each electrode is a 2.5 μm thick metallic layer deposited on a circular section of a hemispherical surface with a chord diameter of 18.3 mm. Copper and titanium films were alternately deposited by a magnetron sputtering system [SFI Sputtering Inc., CA]. The system was pumped town to a pressure of $6x10^{-5}$ Pa using a turbopump with a liquid nitrogen trap. The sputtering pressure was 0.9 Pa at an Ar flow of about 22 standard cm^3 min^{-1}. The copper film was deposited by d.c. magnetron sputtering at a rate of 2.2 $nm.s^{-1}$ and the thickness was monitored with an Inficon IC-6000 quartz crystal monitor. The titanium film was prepared using r.f. magnetron sputtering at a rate of 0.4 $nm.s^{-1}$ and the thickness was controlled by varying the deposition time. Both the copper and the titanium targets were 99.9955 purity. For the multilayer Cu and Ti films, the first layer was Ti with 100 nm thickness. The intermediate layers were Ti with 50nm thickness or Cu and the thickness of Cu layers were adjusted in order to keep a total thickness of about 2.5 μm. The outer layer Ti thickness was 200 nm. The copper -based films have been coated onto the quartz sample substrates and the quartz gyro housing electrodes without breaking vacuum between the deposition of the successive layers. No substrate heating was added. After deposition, the gyro housing was carefully cleaned with urethane foam in MICRO detergent solution, immersing and rinsing in hot water and deionized water to remove particles and drying in a vacuum oven for three hours about 100°C.

Results and Discussion

A. Adhesion tests

Scotch tape peel test was used to verify adhesion. Adhesion failures were observed for the single layer Cu films at room temperature, indicating that copper films have poor adhesion to the quartz substrate. For Ti-Cu, Ti-Cu-Ti and seven layer Ti-Cu films, a thin Ti layer was coated on the quartz substrate, and no adhesion failures were observed in room temperature testing and in testing after thermal cycling to 77 K. Trilayer Ti-Cu-Ti and seven-layer Ti-Cu gyro housing electrode films have passed scotch tape adhesion testing after temperature cycling between 4 K to 400 K. The first Ti layer provides good adhesion to the fused quartz substrate and gyroscope housing, ensuring that copper provides a good interconnection material for gyroscope housing electrodes.

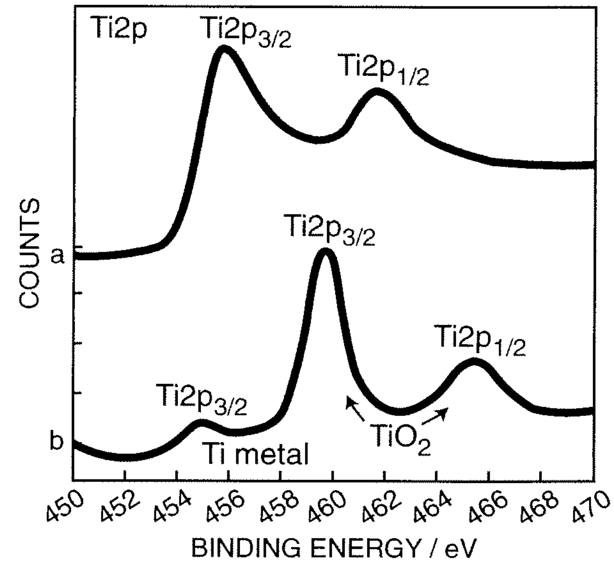

Fig. 2. High-resolution scans of the Ti (2p) XPS peaks taken (a) at the surface, (b) at the interfacial Ti layer for Ti-Cu-Ti film.

B. X-ray photoelectron spectroscopy (XPS)

High-resolution x-ray photoelectron data (Al kα radiation) were taken in Ti-Cu-Ti film in order to learn more about the chemical bonding in the surface and the interior of the outer Ti layer for the Ti 2p binding energy region. The Ti (2p) peaks for the surface after exposure to air, and for the interior are shown in Fig. 2 (a) and Fig. 2 (b), respectively. Three peaks appear in Fig. 2 (a): Ti metal Ti $2p_{3/2}$, and Ti $2p_{3/2}$ and Ti $2p_{1/2}$ states with TiO_2, The oxygen was associated with TiO_2, and the signature of TiO_2 is clearly observable in the surface. In Fig. 2 (b), the two peaks associated with Ti ($2p_{3/2}$ and $2p_{1/2}$ states) were observed after sputtering for 22 min. with argon to remove about 13 nm from the surface. The XPS data indicated that the interior of the outer Ti layer is free of oxidation. From the 70° tilt XPS data for the Ti-Cu-Ti film, we deduce that the TiO_2 layer is about 6nm thick. The outer Ti layer can protect the easily corroded Cu layers and also provides a high melting point material, thus improving the electron field emission characteristics of the film (1).

Table 1. Cu and multilayer Cu-based film resistivity.

Films	Film Thickness	Resistivity (μΩ-cm)	
		4.2 K	**280 K**
Cu	**0.9 μm**	**0.05**	**2**
Ti/Cu/Ti	**2.8 μm**	**0.23**	**3.6**
Ti/Cu/Ti/Cu/Ti/Cu/Ti	**2.7 μm**	**0.14**	**2**

Resistivity

A four-point method was used to measure the resistivity of the films. The resistivity data for Cu, Ti-Cu-Ti and seven layer Ti-Cu films are given in Table 1. The resistivity value of Cu film is slightly higher than that of bulk copper (1.67 μΩ-cm). At room temperature, the seven layer Ti-Cu film has a resistivity similar with that of the Cu film. The thickness of each Cu layer is about 0.8 μm, and the total thickness of copper layers is about 85% of the film thickness in the seven layer Ti-Cu films. Copper films are dominant in low resistivity multilayer films. The resistivity values of the multilayer Cu-based films at 4.2 K are below the GP-B requirement of 27 μΩ-m. The intermediate Cu layers provide low electrical resistance and good thermal conductivity.

Conclusions

Copper metallizations have been applied to the Gravity Probe B Relativity Mission Gyroscope housing electrodes. In the multilayer Cu-based films, the first Ti layer provides good adhesion to the quartz substrates and gyroscope housing electrodes. The easily corroded copper film can be protected by an outer layer coating of Ti. The seven layer Cu-based films provide good adhesion, low resistivity, good thermal conductivity and very smooth surfaces. Seven layer Cu-based films have been successfully used for the gyro housing electrodes. The gyroscopes have been successfully spun to 170 Hz for more than 100,000 hours testing, and are able to operate for two years at 170Hz in the GP-B experiment projected to fly around the year 2,000.

Acknowledgements

This work was supported by NASA Contract NAS8-39225.

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