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Multilayer Ti–Cu sputter films for the Gravity Probe B gyroscope housings

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Abstract

Multilayer Ti–Cu films with three, five, and seven individual layers have been prepared by sputtering on fused quartz substrates. The multilayer films have been studied by electron field emission, X-ray diffraction, scanning electron microscopy (SEM), and X-ray photoelectron spectroscopy. Trilayer Ti–Cu–Ti electrode films exhibit reduced electron field emission. Ti–Cu–Ti trilayer films have been applied to the Gravity Probe B gyroscope housings. The thin films have been deposited by sputtering onto the fused quartz housings to form suspension electrodes and lands. The gyroscopes have been successfully spun to 170 Hz.

1. Introduction

Gravity Probe B is a general relativity experiment which will use electrostatically suspended quartz gyroscopes in a 600 km polar orbit to measure geodetic and frame dragging effects with an accuracy of 0.3 marc-s year^{-1}. In a previous paper [1], we reported that Cu–Ti coatings have been applied by sputter deposition to fused quartz housings to form suspension electrodes and lands. Niobium-coated fused quartz gyroscope rotors [2] have been suspended and spun up in these housings coated with Cu–Ti bilayers. This paper focuses on the multilayer Ti–Cu films for the Gravity Probe B gyroscope housing suspension electrodes. The gyroscopes are electrostatically suspended by three orthogonal pairs of circular electrodes which are sputter deposited on the optically polished spherical cavity of the gyroscope housing. The requirements (and our solutions) for the thin film electrodes over the temperature range 2 K to 450 K are as follows:

(i) Good film adhesion to the quartz substrate (0.10 μm Ti binding layer to quartz).

(ii) Film stresses which do not exceed the tensile strength of quartz (suitable sputtering parameters).

(iii) Electrical resistivity of less than 27 μΩ m (2.5 μm Cu film).

(iv) Thermal conductivity greater than 0.5 W cm^{-1} K^{-1} (2.5 μm Cu film).

(v) Less than 1 pA enhanced electron field emission in a field of 4×10^7 V m^{-1} (0.20 μm Ti outer layer with low β factor).

(vi) Resistance to arcing damage for an arc energy of less than 3×10^{-4} J (0.20 μm Ti outer layer with low β factor and high melting temperature).

(vii) Film uniformity of better than 10% (suitable sputtering geometry).

(viii) Superconducting transition temperature of less than 1.5 K.

(ix) No damage under repeated thermal cycling between 2 K and 450 K.

2. Experimental details

The multilayer Ti–Cu films used in this study were prepared with a magnetron sputtering system. The detailed procedures for the coating technique and coating of housings have been previously described [1]. The base pressure was about 6×10^{-5} Pa, while the sputtering pressure was maintained at 0.9 Pa. The deposition rates are 0.4 nm s^{-1} for Ti and 2.2 nm s^{-1} for Cu. The Ti film was deposited using r.f. magnetron sputtering and the thickness was controlled by changing the deposition time. The Cu film was prepared using d.c. magnetron sputtering and the thickness was monitored with an Inficon IC-6000 quartz crystal monitor. In systematic studies, multilayers with 3, 5 and 7 individual layers have been prepared, with an overall thickness of about 2.5 μm. The first Ti layer thickness was 100 nm. Intermediate Ti layer thicknesses were 25 nm, 50 nm, and 100 nm for different samples, while the outer Ti layer thickness was 200 nm. The thicknesses of Cu layers were chosen so as to keep a total thickness of about 2.5 μm. The films were deposited without breaking vacuum between the deposition of the successive layers. Trilayer Ti–Cu–Ti films have been coated onto the fused quartz gyro housings and the fused quartz sample substrates.

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3. Results and discussion

The layer thicknesses of samples that have been studied are summarized in Table 1. The base Ti layer provides good adhesion to the fused quartz substrate. No adhesion failures were found in these coatings, as contrasted with the case of the Cu binding layer to quartz where failures were observed. The intermediate Cu layer provides low electrical resistance and good thermal conductivity. The outer Ti layer provides a high melting point material and improves electron field emission characteristics.

3.1. Electron field emission

We studied a number of films in order to find an electrode coating with low enhancement factor for electron field emission. The Cu (2500 nm)–Ti (100 nm), Mo (250 nm)–Cu (2500 nm)–Ti (100 nm), and Ti (250 nm)–Cu (2500 nm)–Ti (100 nm) films were tested by a field emission system apparatus. A polished rounded tungsten tip was positioned close to the surface of the sample using a microstepping servo motor and a capacitance bridge. The emission current was measured as a function of the electric field for distances of 25 μm to 50 μm at a pressure of about 5×10^{-6} Pa. Figure 1 shows the Fowler–Nordheim plot for a Ti(250 nm)–Cu(2500 nm)–Ti(250 nm) sample. The field enhancement factor β is typically around 20 for these trilayers, while the niobium gyroscope rotor coating has a β factor 10%–20% lower [3].

Figure 2 shows the electric field required for an electron field emission current of 2 pA for three film types. It can be seen from Fig. 2 that the corresponding field for the Ti (250 nm)–Cu (2500 nm)–Ti (100 nm) film is about 10^8 V m^{-1} and is higher than the other two films. Trilayer Ti–Cu–Ti films have been coated onto fused quartz gyroscope housings. The trilayer Ti–Cu–Ti electrode coatings exhibit reduced electron field emission, which in turn reduces gyroscope rotor charging. The Ti–Cu–Ti electrode coatings have greater resistance to electrical breakdown. The gyroscopes have been successfully spun to about 170 Hz [4].

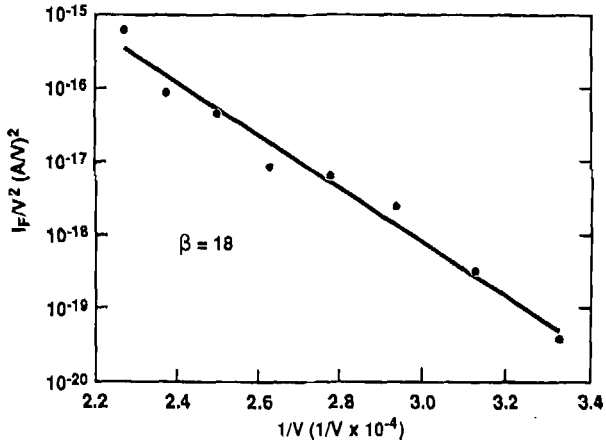

Fig. 1. Fowler–Nordheim plot for a trilayer film Ti (250 nm)–Cu (2500 nm)–Ti (100 nm) at 25 μm.

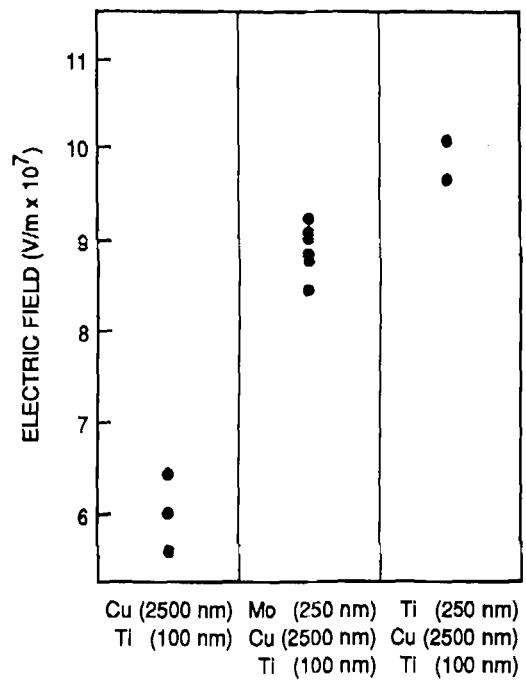

Fig. 2. The electric field required for an electron field emission current of 2 pA for three different films.

TABLE 1. Layer thicknesses of multilayer Ti–Cu films

	Film thickness (μm)						
	Three layers	Five layers			Seven layers		
	3A	5A	5B	5C	7A	7B	7C
1st layer Ti	0.10	0.10	0.10	0.10	0.10	0.10	0.10
2nd layer Cu	2.56	1.15	1.21	1.24	0.82	0.79	0.71
3rd layer Ti	0.20	0.025	0.05	0.10	0.025	0.05	0.10
4th layer Cu		1.15	1.21	1.24	0.82	0.79	0.71
5th layer Ti		0.20	0.20	0.20	0.025	0.05	0.10
6th layer Cu					0.82	0.79	0.71
7th layer Ti					0.20	0.20	0.20
Total	2.86	2.63	2.77	2.88	2.81	2.77	2.63

3.2. Film morphology

The surface morphology of multilayer Ti–Cu films was studied by scanning electron microscopy (SEM). Scanning electron micrographs of the surfaces of samples 3A, 5B and 7B are shown in Fig. 3. The grain sizes are about 0.35 μm for the 3-layer sample 3A, about 0.25 μm for the 5-layer sample 5B, and about 0.20 μm for the 7-layer sample 7B. From Fig. 3 it can be seen that the

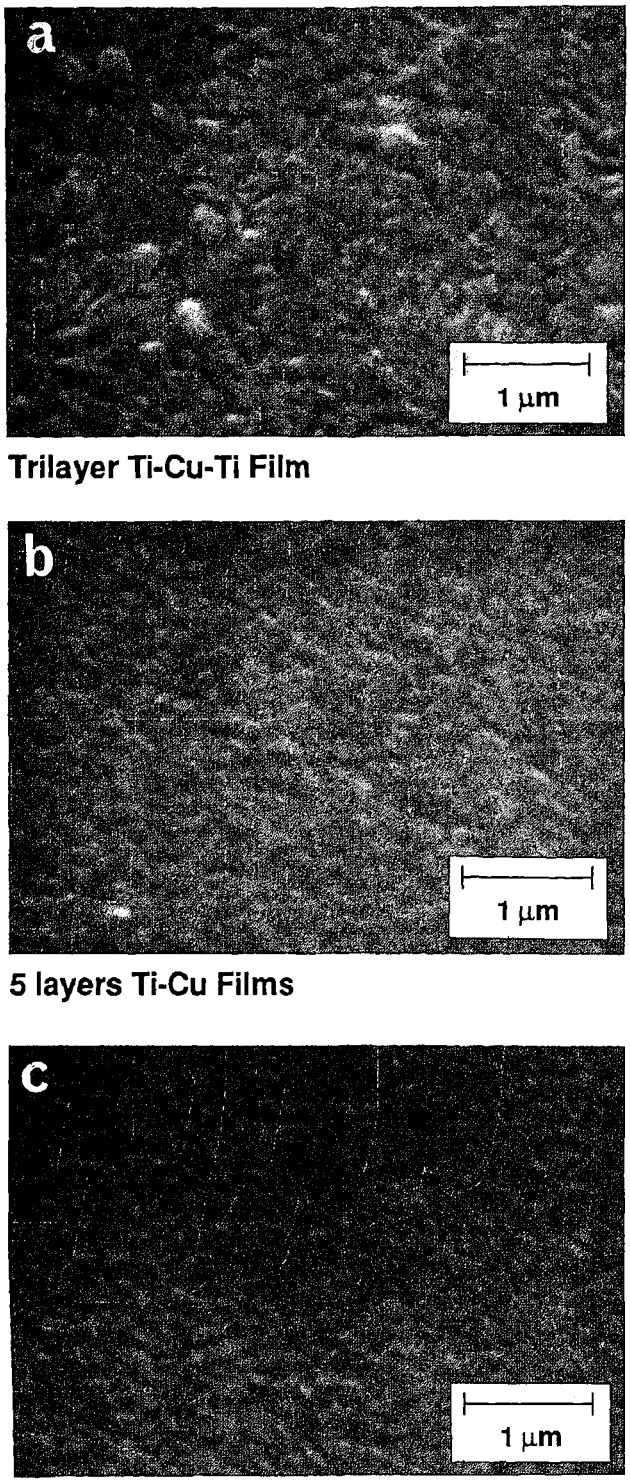

Fig. 3. Scanning electron micrographs of the surface for samples: (a) 3-layer Ti–Cu–Ti film; (b) 5-layer Ti–Cu film; (c) 7-layer Ti–Cu film.

surface morphology of the Ti–Cu films varied with the number of layers. Figures 3(a)–3(c) show the variations in surface smoothness. The 5- and 7-layer Ti–Cu films produced smoother surfaces.

3.3. *X-ray diffraction analysis*

The structure of multilayer Ti–Cu films was examined using an X-ray diffractometer with Cu Kα radiation. The X-ray diffraction patterns of samples 3A, 5B and 7B are presented in Fig. 4. The strong central peak is associated with Cu (111), and the satellite peaks contain Ti(002) and Cu(200). The samples 3A and 5B have a Cu (111) strong preferred orientation. The sample 7B shows a reduction in the Cu (111) orientation and increase in the Cu (200) orientation.

3.4. *X-ray photoelectron spectroscopy*

The trilayer Ti–Cu–Ti sample 3A was analyzed by X-ray photoelectron spectroscopy (XPS). For XPS data Al Kα radiation was used. High resolution XPS spectra were obtained for the Ti 2p binding energy region. Peaks for the surface after exposure to air and for the interior are presented in Figs. 5(a) and 5(b) respectively. Three peaks are observed in Fig. 5(a): Ti metal Ti $2p_{3/2}$, and the Ti $2p_{3/2}$ and Ti $2p_{1/2}$ states with TiO_2. The signature of TiO_2 is clearly observable in the surface sample. Only the two peaks associated with Ti ($2p_{3/2}$ and $2p_{1/2}$ states) are observed in Fig. 5(b) after sputtering for 22 min with argon to remove about 13 nm from the surface. This indicates that the interior of the Ti layer is free of oxidation. The thickness of the TiO_2 layer formed in air is approximately 6 nm, as determined from the 70° tilt XPS data for the sample 3A.

4. Conclusions

Ti–Cu–Ti trilayer films have been applied to the Gravity Probe B gyroscope housings. The thin films have been deposited by sputtering onto fused quartz

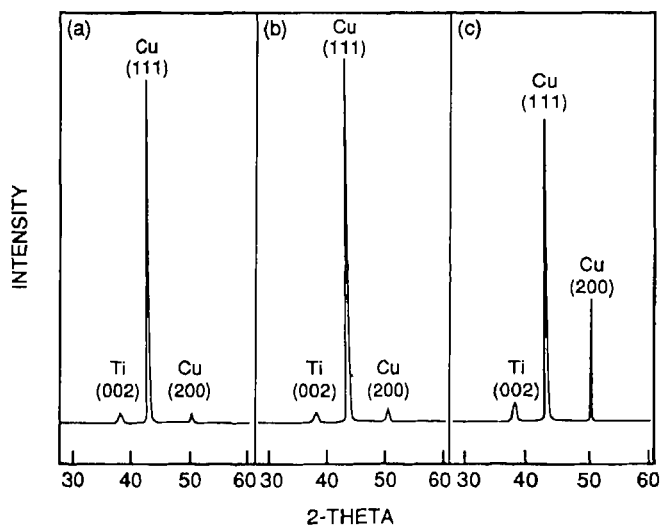

Fig. 4. X-ray diffraction patterns of samples: (a) 3A (3-layer Ti–Cu–Ti film); (b) 5B (5-layer Ti–Cu film); (c) 7B (7-layer Ti–Cu film).

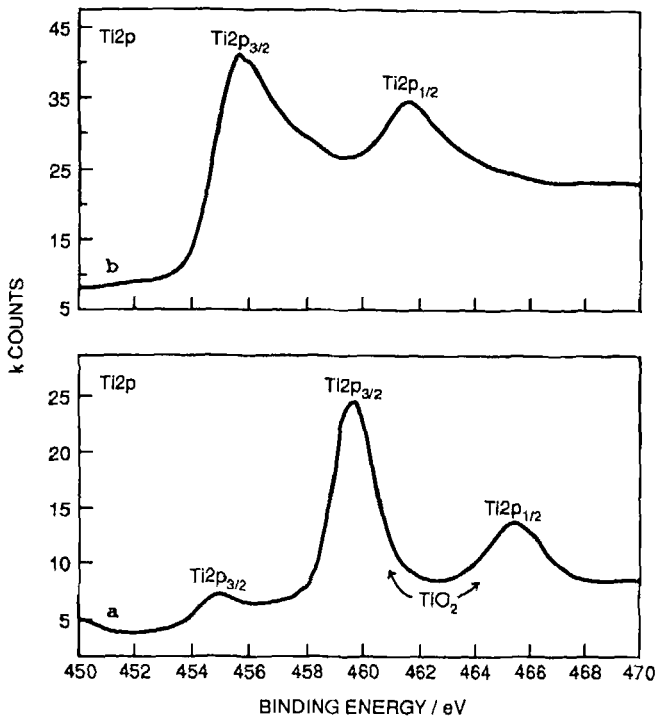

Fig. 5. XPS spectra in the Ti 2p binding energy region for sample 3A (3-layer Ti–Cu–Ti film): curve a, at the surface; curve b, after sputtering for 22 min with argon to remove about 13 nm.

gyroscope housings to form suspension electrodes and lands. Trilayer Ti–Cu–Ti electrode coatings exhibit reduced electron field emission, which in turn reduces gyroscope charging. The gyroscopes have been successfully spun to 170 Hz. The Ti outer layer has greater resistance to electrical breakdown and reduces damage when arcs do occur. The five and seven layer Ti–Cu films have smoother surfaces than the trilayer Ti–Cu–Ti film and are presently under evaluation for the Gravity Probe B gyroscope electrode coatings.

Acknowledgments

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