# STRESS AND ITS EFFECT ON SURFACE MORPHOLOGY IN MULTI-LAYER Ti-Cu FILMS

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## ABSTRACT

We have characterized the stress and its effects on surface morphology for multi-layer thin films of about 3μm total thickness, consisting of three or seven layers of Ti and Cu. These films constitute the electrostatic suspension electrodes for the gyroscope housings of the Relativity Mission Gravity Probe B. Full understanding of surface morphology is critical for meeting the complex requirements of this application.

The residual stresses have been measured using a laser curvature technique, while the surface morphology was studied by scanning electron microscopy (SEM). We find that the surface morphology depends strongly on the stress, which evolves with the Ti-Cu multi-layer period. Average stress and the resulting surface roughness decrease for thinner Cu layers (increased total number of layers). Seven layer Ti-Cu films with low stress and very smooth surface have been successfully used for the electrodes of the gyroscope housings.

## INTRODUCTION

Metal multi-layer thin films are important components in many materials applications because the mechanical properties of these structures differ from those of the bulk materials. Multi-layer Ti-Cu films have been successfully used in the Gravity Probe Relativity Mission gyroscopes.[1] The Gravity Probe B (GP-B) experiment is designed to measure the geodetic and frame -dragging precessions predicted by Einstein's general theory of relativity. The goal of the experiment is to measure these precessions to better than 0.01% and 1%, respectively.[2] The GP-B gyroscopes are electrostatically suspended, niobium-coated fused quartz or single crystal silicon spheres of 2 cm radius. They are spun to 170 Hz, in a quartz housing at 2K, and in ultrahigh vacuum ($10^{-9}$ Pa).

Figure 1 is a exploded view of the gyroscope and its housing. The gyro housing has six electrodes arranged in three mutually orthogonal pairs. The requirements for the electrode films are: (1) Low tensile stress in the film, (2) good adhesion to the quartz substrate, (3) good thermal and electrical conductivity, (4) low electron field emission in a field of up to $3\times10^{7}$ V/m, (5) resistance to arcing damage for an arc energy of less than $3\times10^{-4}$ J, and (6) film uniformity of better than 10% over the electrode surface. These requirements insure the mechanical, and electrical integrity of the gyroscope housings under the operating conditions, spanning the temperature range from 2 K to 300 K, and the voltage range from 20 mV to 2,000 V. The uniformity of the coating is required to limit the electrostatic forces and therefore the disturbance torque. Electrodes made of multi-layer Ti-Cu films sputtered on the quartz housing have met all requirements in 100,000 hours of gyroscope testing.

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In this paper, we describe the stress and its effects on the surface morphology of multi-layer Ti-Cu films. The residual stress has been measured using the laser curvature technique. The surface morphology is examined by scanning electron microscopy (SEM).

## EXPERIMENTAL PROCEDURE

All multi-layer Ti-Cu films were prepared by a magnetron sputtering system on fuse quartz substrates. The base pressure was about $6 \times 10^{-5}$ Pa after a liquid nitrogen trap w cooled. The layers are deposited in 0.9 Pa with an Ar flow of about 22 standard $cm^3$ $min^{-1}$ without substrate heating. The Ti film was prepared using r.f. magnetron sputtering at a power of 400W. The Cu film was deposited using d.c. magnetron sputtering . Both the copper and the titanium targets were of 99.995 purity. For the multi-layer Ti-Cu films, a Ti layer with 100 nm thickness was used as a binding layer t the quartz substrate. The intermediate Ti layer thicknesses were 50 nm. The outer Ti layer thickness was 200 nm. The thickness of Cu layers were adjusted in order to keep the total film thickness at about 2.7 μm. The films were deposited onto the fused quar substrates and silicon wafer without breaking vacuum between the deposition of successive layers. The film thicknesses were measured by a DEKTAK 3030ST profilometer.

Stress measurements were performed by measuring the substrate curvature using a Laser Scanning Technique in the as-deposited films. The film stress was determined b measuring the change in substrate after film deposition, using Stoney's formula:[3]

$$\sigma_f = \frac{E_s t_s^2}{6 t_f (1 - v_s)} \left[ \frac{1}{R} - \frac{1}{R_0} \right] \qquad (1$$

where $\sigma_f$ is the biaxial stress in the film, $E_s$ and $v_s$ are Young's modulus and Poission's ration of the substrate, $t_f$ and $t_s$ are the thickness of the film and substrate, and $R_0$ and are the substrate curvature before and after film deposition. For stress measurements, initial wafer bow was measured prior to film deposition.

### TABLE

Table 1. Stress in Ti-Cu multi-layer films

| Sample | Pre-Radius (m) | Post-Radius (m) | Film Thickness (μm) | Stress (MPa) |
|---|---|---|---|---|
| (A) Ti/Cu/Ti | 43.4 | 25.0 | 3.04 | 79.0 |
| (B) Ti/Cu/Ti/Cu/Ti/Cu/Ti | 69.7 | 38.4 | 3.04 | 54.5 |

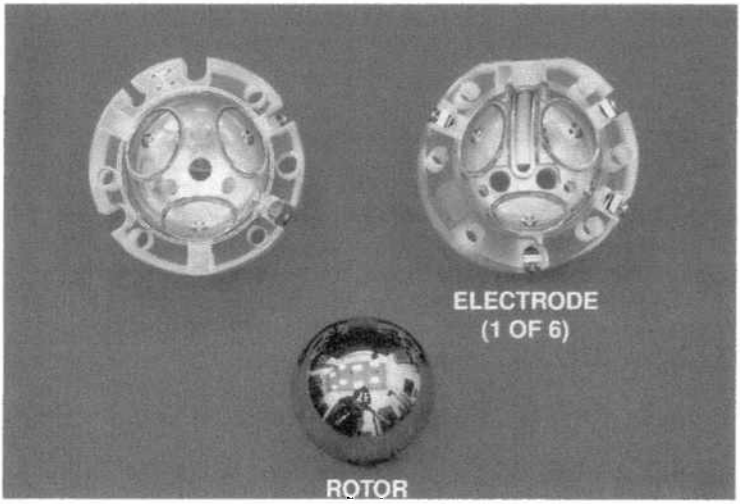
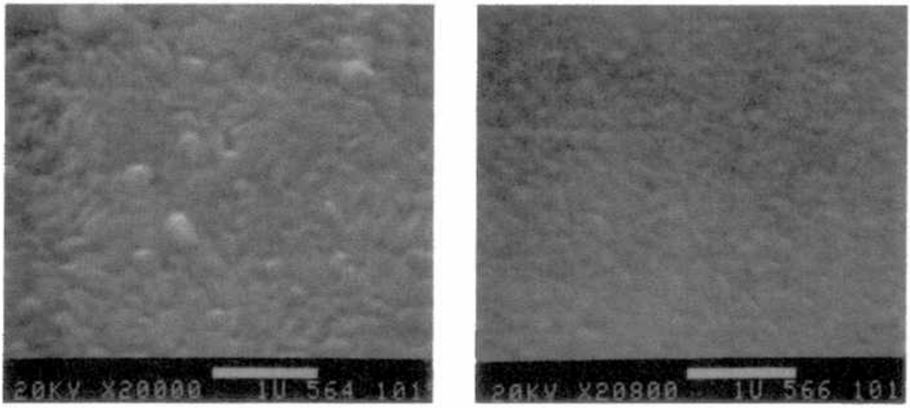


Fig. 1 Exploded view of the gyroscope and its housing.



(a) (b)

Fig. 2 SEM surface morphology of sample:
(a) tri-layer Ti-Cu-Ti film; and
(b) seven-layer Ti/Cu/Ti/Cu/Ti/Cu/Ti film.

## RESULTS AND DISCUSSION

Multi-layer Ti-Cu films for the Gravity Probe B gyroscope housings were reported previously.[1] The first Ti layer provides good adhesion to the fused quartz substrates. No adhesion failure was found in room temperature and after thermal cycling to 77 K. The intermediate Cu layer provides good thermal and electrical conductivity. The outer Ti layer provides a high melting point material and has greater resistance to electrical breakdown.

Several films have been studied using a Laser Scanning Technique, Tencor Instruments, Santa Clara, CA. They include the tri-layers Ti (0.1 μm)/Cu (2.74 μm)/ Ti (0.2 μm) and the seven-layer film Ti (0.1 μm)/Cu (0.88 μm)/Ti (0.05 μm)/ Cu (0.88 μm)/Ti (0.05 μm)/Cu (0.88 μm)/Ti (0.2 μm). The average stresses for the two samples are summarized in Table 1.

Sputter deposited films are subject to growth stresses that depend on the deposition pressure and on the source to substrate distance. For the present sputtering system (Sputtering Film Inc., Santa Barbara, CA), the stresses of multi-layer Ti-Cu films are tensile over the entire film, which is desirable for the hemispherical surface of gyroscope housing electrodes. The 3.25 μm copper film is found to have a tensile stress of 194 MPa. The exact stress in the Titanium films has not been measured in this study. It is clear that the thicker, multi-layer structures do not sustain much stress. The stresses in the tri-layer and seven-layer films are below 100 MPa. As seen from the data in Table 1, increasing the number of layers reduces the stress, consequently the seven-layer films have a lower stresses than the tri-layer film.

Surface and interface can play a role through the surface or interface tension[4]. When a layer of Cu is deposited on a Ti surface, the Ti surface is replaced with an Cu surface and a Cu/Ti interface. The substrate can curve either upwards or downwards, depending on the balance of the interface tensions. In the multi-layer Ti-Cu films average stresses decrease for thinner Cu layers (increased total number of layers).

The surface morphology of the multi-layer Ti-Cu films was examined using scanning electron microscopy(SEM). Figures 2(a) and 2(b) are SEM scans of the surface of samples trilayer Ti/Cu/Ti film and seven-layer Ti/Cu/Ti/Cu/Ti/Cu/Ti film respectively. The grain sizes are about 0.35 μm for the tri-layer film A, and about 0.20 μm for the seven-layer film B. The seven-layer film appears to have smoother surfaces than the tri-layer film. The resulting surface roughness decreases for thinner intermediate Cu layer

## CONCLUSIONS

Multi-layer Ti-Cu films have been prepared by sputtering on fused quartz substrates. The residual stresses have been measured using the laser curvature technique, and the surface morphology has been examined by scanning electron microscopy(SEM). We find that the surface morphology depends strongly on the stress, which evolves with the Ti-Cu multi-layer period. Average stress and the resulting surface roughness decrease for thinner Cu layers. Seven layer Ti-Cu films with low stress and very smooth surface have been used for the gyroscope housing electrodes. The gyroscopes have been successfully spun to 170 Hz, and have operated for more than 100,000 hours, therefore fully proving their capability to operate for two years at 170 Hz in the GP-B experiment.

## Acknowledgements

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## References

[1] P. Zhou, S. Buchman, K. Davis, C. Gray and J.P. Turneaure, Surf. Coat. Technol., 54/55, p. 548 (1992).

[2] J.P. Turneaure, C.W.F. Everitt and B.W. Parkinson, Adv. Space Res., 9, p. 29 (1989).

[3] G.G. Stoney, Proc. R. Soc. London A82, p. 172 (1909).

[4] J.A. Ruud, A. Witvroum, and F. Spaepen, Mat. Res. Soc. Proc., p. 209 (1991).