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Multilayer Films of TiC, Ti and Cu for the Gravity Probe B Relativity Mission Gyroscopes

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Abstract

Single component and multilayer films of TiC, Ti and Cu have been produced by sputtering on fused quartz substrates and have been investigated for suitability as electrodes for the electrostatically suspended gyroscopes of Gravity Probe B. The main requirements for these films are low electron field emission in fields of 3×10^7 V/m, low film stress, and good thermal and electrical conductivity in the temperature range 2 K - 400 K. The films have been studied using x-ray diffraction, scanning electron microscopy (SEM), Auger electron spectroscopy (AES), Rutherford backscattering spectrometry (RBS), and four-point resistivity techniques. SEM measurements indicate that the TiC films have very smooth surfaces and that multilayer films of TiC, Ti and Cu have reduced roughness as compared to the Ti-Cu multilayer films. Seven layer 2.5 μm thick Ti-Cu films have been successfully used in the Gravity Probe B gyroscopes, meeting all requirements. Multilayer films of TiC, Ti and Cu are presently being tested with the expectation that they will further improve gyroscope performance.

I. Introduction

The Gravity Probe B Relativity Mission is an experimental test of general relativity using orbiting gyroscopes to measure the geodetic and frame-dragging effects with an accuracy of 3×10^{-4} seconds of arc per year (1). The gyroscopes are quartz spheres of 2 cm radius coated with a 2.5 μm niobium film (2) and suspended electrostatically in a quartz housing at low temperature, 2 K, and in ultra high vacuum, 10^{-9} Pa. Figure 1 is a photograph of the gyroscope and its housing.

In a previous paper (3), we reported a study of multilayer Ti-Cu sputtered films for the GP-B gyroscopes. Seven layer 2.5 μm thick Ti-Cu films for the gyroscope housing electrodes have met all requirements. The gyroscopes have been successfully spun to 170 Hz (10,000 rpm). The main requirements for these electrode films (in the 2 K to 400 K temperature range) are:

a) low electron field emission in a field of up to 3×10^{7} V/m,

b) resistance to arcing damage for deposited arc energies of up to 3×10^{-4} J,

c) low tensile stress in the film,

d) good thermal and electrical conductivity,

e) good adhesion to the quartz substrate,

f) film uniformity of better than 10% over the electrode surface,

g) no magnetic impurities that would produce a field in excess of 10^{-7} gauss.

This paper presents a study of the multilayer films of TiC, Ti and Cu for the Relativity Mission gyroscope electrodes, performed with the aim of improving the above characteristics of the films. The deposited films were characterized using X-ray diffraction, scanning electron microscopy (SEM), Auger electron spectroscopy (AES), Rutherford backscattering spectrometry

(RBS) and four-point resistivity measurements. Section II is a description of the experimental procedure used in producing the films, while in section III the results of the measurements of the film characteristics are presented, and their applicability to the improvement of the gyroscope performance is discussed.

II. Experimental Procedure

Single component and multilayer films of TiC, Ti and Cu have been produced by magnetron sputtering on quartz substrates. The facility and experimental techniques have been previously described (4). The sputtering chamber was evacuated to a base pressure of about 6×10^{-5} Pa using a turbopump with a liquid nitrogen trap. The sputtering pressure was 0.9 Pa at an argon flow of about 20 sccm.

Titanium carbide thin films were produced by non-reactive dc magnetron sputtering from a stoichiometric TiC target (99.5% purity). Before the deposition of the TiC films the target was cleaned by pre-sputtering for 30 minutes. The TiC films were deposited without a bias applied between the target and the substrates. Copper films were deposited using dc magnetron sputtering. The thicknesses of TiC and Cu films were monitored with an Inficon IC-6000 quartz crystal monitor. Titanium films were deposited using RF magnetron sputtering and their thickness was controlled by the deposition time. Both the copper and titanium targets were of 99.995% purity.

Deposition rates were 0.4 nm/s for Ti, 2.2 nm/s for Cu, and 0.3 nm/s for TiC. For the multilayer TiC, Ti and Cu films, a Ti layer with 100 nm thickness was used as a binding layer to the quartz substrate. The intermediate Ti or TiC layer thicknesses were 50 nm. The outer layer TiC thickness was 200nm. The thicknesses of Cu layers were adjusted in order to keep the total film thickness at about 2.5 μm. The films were coated on to the fused quartz sample substrates

film thickness at about 2.5 μm. The films were coated on to the fused quartz sample substrates and the fused quartz gyro housings without breaking vacuum between the deposition of the successive layers. In systematic studies, TiC films and multilayer films of TiC, Ti and Cu have been produced. No substrate heating was used, but substrate temperature did rise to about 100 °C during deposition, due to plasma heating.

III. Results and discussion

A. TiC/SiO_2

Figure 2 shows the AES spectra of the film of TiC as-deposited on fused quartz at two depths: (a) at the surface, and (b) in the bulk of the film after 22 minutes with a 2 keV argon ion sputter etching (about 180 nm deep). The AES spectra indicate that a top layer of about 5 nm was contaminated with oxygen but that the bulk of the film was oxygen free. The shape of the carbon peak in Fig.2(b) is characteristic of TiC, while the argon peak inside the film was primarily due to argon trapped during sputtering.

Rutherford backscattering spectrometry (RBS) was performed using 2.3 MeV helium ions for measuring the composition and thickness of the TiC films. Figure 3 shows the RBS spectra (overlaid with simulated fits) of a TiC film measured at (a) 170 degrees, and (b) 120 degrees. The 120 degrees measurement serves to separate the carbon signal from the film and the oxygen signal from the SiO_2 substrate. The RBS spectra show that the TiC sample has a constant composition with depth of $Ti_{44.5}C_{55.4}W_{0.1}$. The uncertainty in the stoichiometry is due to the height measurement and amounts to 6.3% for this film. There is a surface oxide of about 5 nm, which has been modelled as TiO_2, although other composition oxides are possible. The tungsten

which has been modelled as TiO_2, although other composition oxides are possible. The tungsten impurity is constant in concentration at all depths in the film. It is due to target impurity and it has no negative effect for this TiC film application. The thickness of the film is 338±5 nm.

The surface morphology of the TiC films was observed by SEM. The TiC film has very smooth surfaces and the average grain size is about 0.8 nm.

The electrical resistivity of the TiC film, as measured with a four-point probe, was about 200 μΩ -cm at room temperature. The structure of the TiC films was examined with an X-ray diffractometer using Cu Kα radiation. X-ray diffraction analysis shows that the TiC film is polycrystalline and has no preferred orientation.

TiC is intended to serve as an overlayer to the multilayer films of the gyroscope electrodes. As such it must fulfill the requirements for low field emission, resistance to arc damage, low magnetic impurities, low chemical reactivity, and good wear resistance. These measurements indicate that the TiC films are of high purity and quality, insuring the hardness and the low reactivity characteristic of this material, as well as the lack of magnetic contamination required by the gyroscope application.

TiC is characterized by its high melting point, 3067 °C, in contrast to the significantly lower melting temperature of Ti, 1668 °C, the material presently used as an electrode overlayer. It is therefore expected that the TiC overlaid electrodes will exhibit reduced arc damage. The 200 μΩ-cm electrical resistivity of the TiC overlayer is only a small contribution to the total electrode film resistivity, which is dominated by the conductivity of the copper layers.

B. TiC/Cu/Ti/Cu/Ti/Cu/Ti/SiO_2 and TiC/Cu/TiC/Cu/TiC/Cu/Ti/SiO_2

In seven layer TiC/Cu/Ti/Cu/Ti/Cu/Ti and TiC/Cu/TiC/Cu/TiC/Cu/Ti films, the binding Ti layer provides good adhesion to the fused quartz substrate. These films pass the Scotch tape adhesion test both before and after cycling to 77K. The intermediate Cu layers provide good thermal conductivity and low electrical resistivity. Table 1 summarizes the resistivity of a Cu film and the seven layer films of TiC, Ti and Cu. The resistivity of the TiC/Cu/Ti/Cu/Ti/Cu/Ti film is equal to that of the Cu film (2 μΩ -cm at room temperature), while the resistivity of the TiC/Cu/TiC/Cu/TiC/Cu/Ti film is 25% higher. This value is well below the requirement for the maximum acceptable resistivity for the gyroscope electrodes (4). The high thermal conductivity of the films, provided by the Cu layers, is crucial in limiting any arc damage by dissipating the heat generated by the bombardment of field emitted electrons.

Figure 4 shows the surface morphology of three types of seven layer films as imaged by SEM: a) TiC/Cu/Ti/Cu/Ti/Cu/Ti, b) TiC/Cu/TiC/Cu/TiC/Cu/Ti, and c) Ti/Cu/Ti/Cu/Ti/Cu/Ti (the film presently in use). The smoothest surface was observed for the a) film (Ti interlayers and an overcoat of TiC). Average grain size for this film is about 0.1 μm, while films b) and c) exhibit grain sizes of 0.2 μm. Note that the intermediate Ti (or TiC) layers are used to inhibit columnar growth in the thick Cu film, thus insuring a smooth outer film surface. The increased smoothness is expected to contribute to the reduction of field emission. Experiments to measure the field emission characteristics of this film are presently being performed.

The structure of the multilayer films was determined by X-ray diffraction analysis with Cu Kα radiation. Figure 5 shows the x-ray diffraction spectra of seven layer films of a) TiC/Cu/Ti/Cu/Ti/Cu/Ti and b) TiC/Cu/TiC/Cu/TiC/Cu/Ti, respectively. The strong central peak is associated with Cu(111), while the satellite peaks are associated with Cu(200) and Ti(002). These films have thus a strong Cu(111) preferred orientation. Similar highly oriented

Ti(002). These films have thus a strong Cu(111) preferred orientation. Similar highly oriented Cu(111) appears in the presently used seven layer Ti/Cu/Ti/Cu/Ti/Cu/Ti films, where it provides good Cu interconnections. The same high quality Cu interconnections are expected for the TiC overcoated films.

The seven layer films have been measured in ultrahigh sensitivity, low temperature magnetometers and have been proven to meet the requirements for low magnetic contamination. Stress measurements in conjunction with Scotch tape adhesion testing and temperature cycling (4 K-400 K) have shown that the films meet the requirements for the gyroscope electrode application. Thickness measurements of the electrodes performed with a Talyrond machine show that the thickness uniformity is better than 10%.

IV. Conclusions

TiC,Ti and Cu films have been produced by sputtering on fused quartz substrates. The TiC film has a constant composition with depth (Ti/C=0.8). The TiC film has very smooth surfaces. The seven layer TiC/Cu/Ti/Cu/Ti/Cu/Ti films have good adhesion, low resistivity and reduced roughness as compared to the seven layer Ti-Cu films. Seven layer 2.5 μm thick Ti-Cu films have been successfully used in the Gravity Probe B gyroscopes, meeting all requirements. Multilayer films of TiC, Ti and Cu are presently being tested with the expectation that they will further improve gyroscope performance.

ACKNOWLEDGMENTS

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Figure Captions

Figure 1. Gyroscope rotor and opened gyroscope housing halves.

Figure 2. AES profiles of TiC as-deposited film a) at the surface and b) in the bulk after 22 minutes argon ion sputter etching (about 180 nm deep).

Figure 3. RBS spectra of TiC as-sputtered film with overlaid simulated fits for a) 170 degrees and b) 120 degrees.

Figure 4. SEM surface morphology of various seven layer films: a) TiC/Cu/Ti/Cu/Ti/Cu/Ti, b) TiC/Cu/TiC/Cu/TiC/Cu/Ti, and c) Ti/Cu/Ti/Cu/Ti/Cu/Ti (the film presently in use).

Figure 5. X-ray diffraction spectra of seven layer films: a) TiC/Cu/Ti/Cu/Ti/Cu/Ti, and b) TiC/Cu/TiC/Cu/TiC/Cu/Ti.

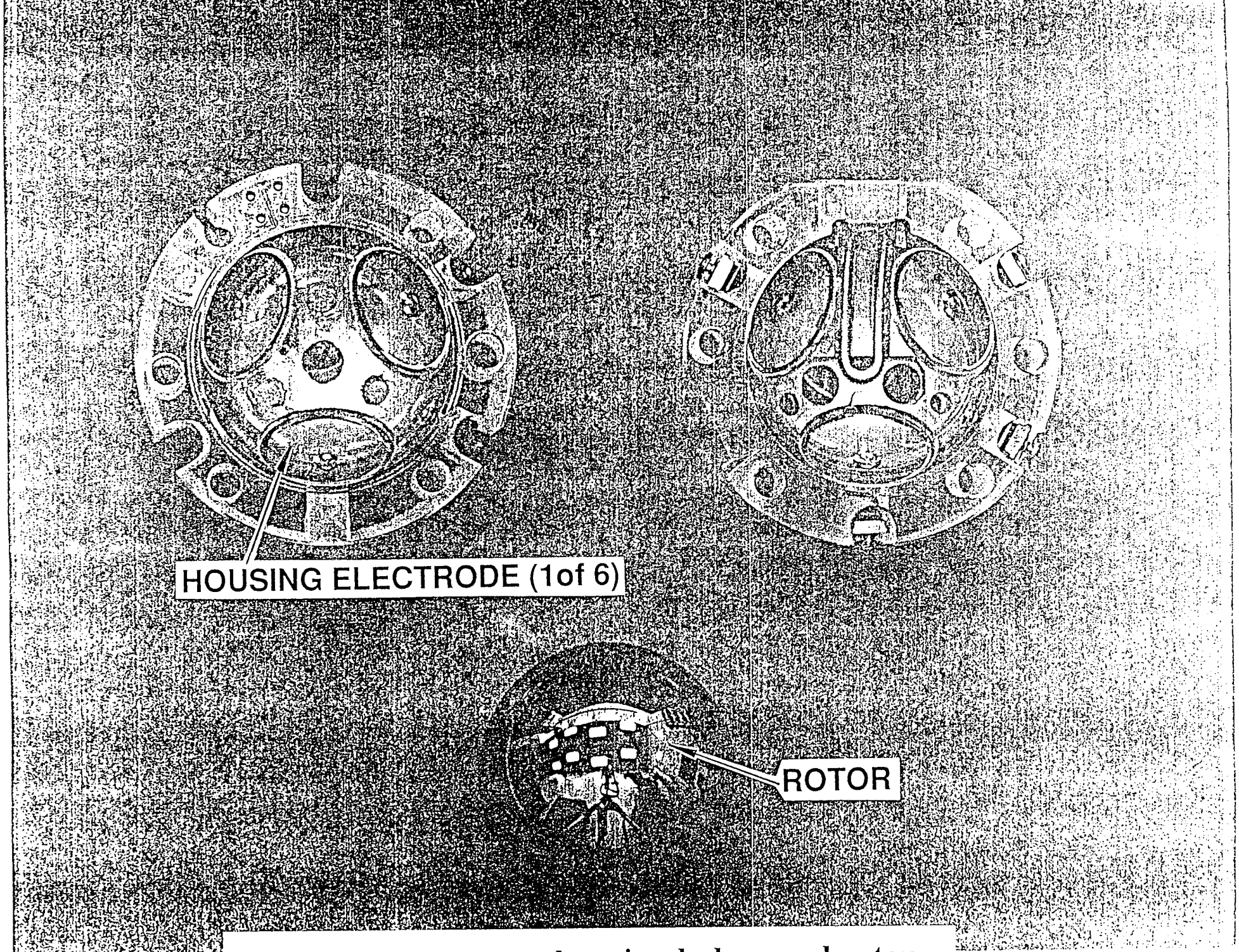

Fig. 1. Gyroscope housing halves and rotor

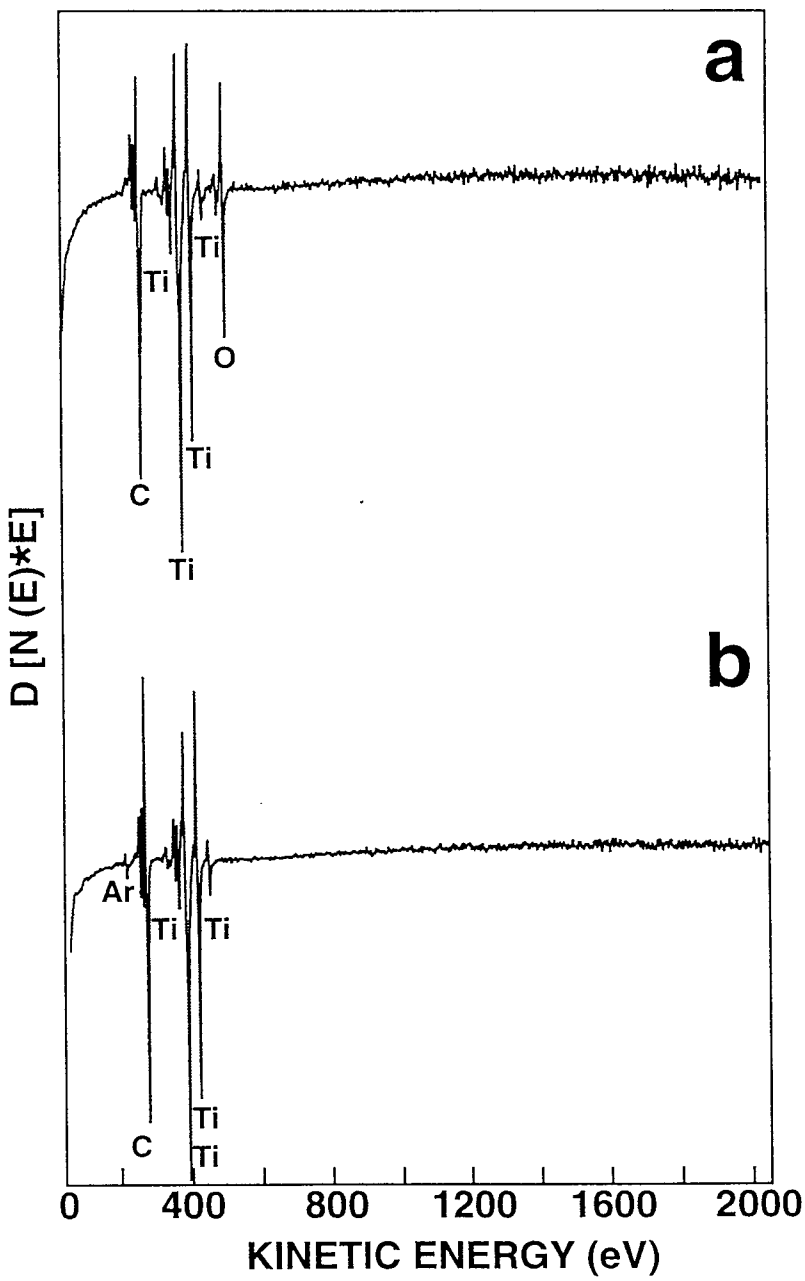

Fig. 2. AES profiles of TiC as-deposited film (a) at the surface and (b) in the bulk after 22 minutes Argon ion sputter etching.

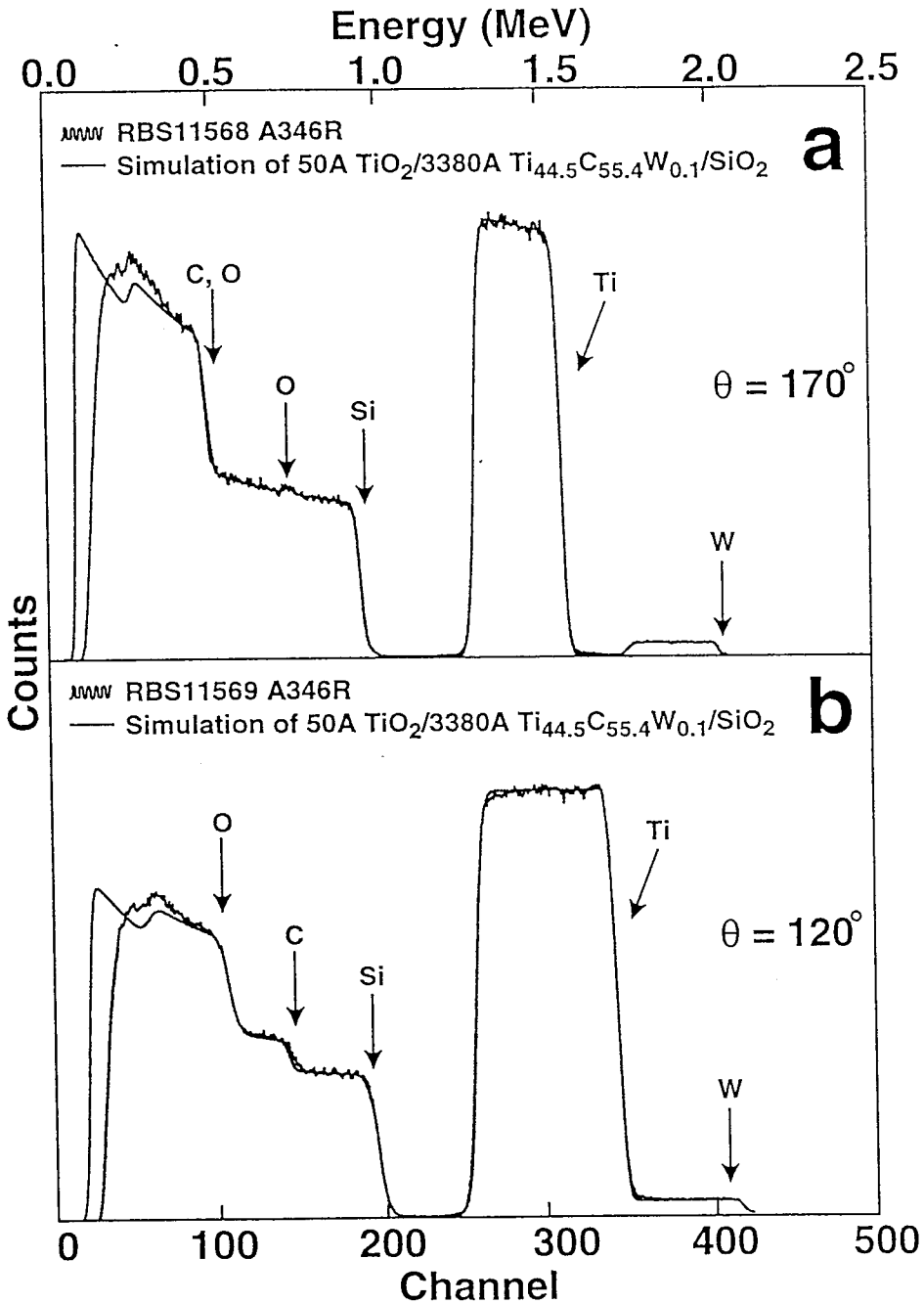

Fig. 3. RBS spectra of TiC as-sputtered film taken at (a) 170 degrees and (b) 120 degrees, overlayed with simulated fits.

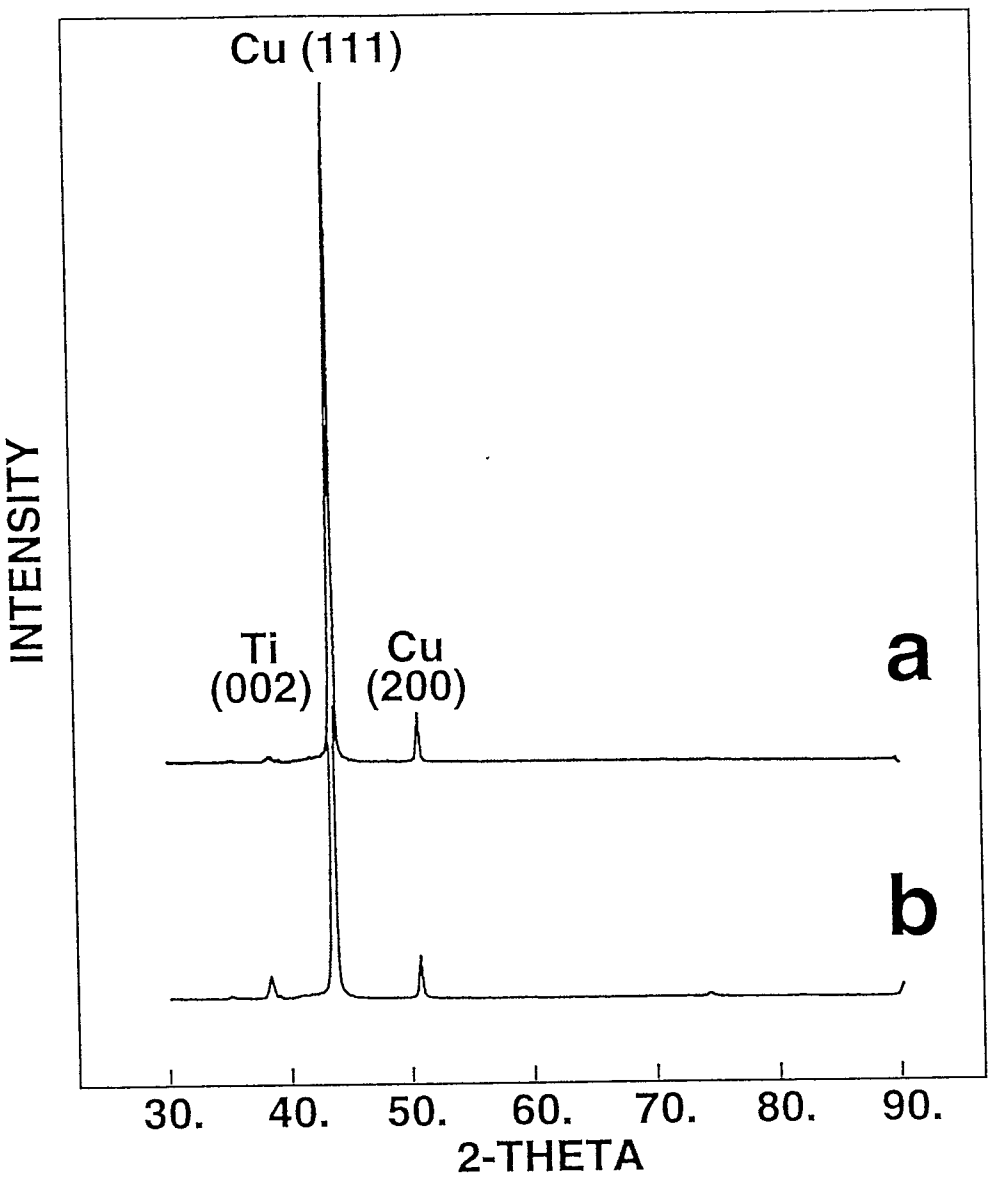

Fig. 5. X-ray diffraction spectra of seven layer films
(a) TiC/Cu/Ti/Cu/Ti/Cu/Ti film, and
(b) TiC/Cu/TiC/Cu/TiC/Cu/Ti.

Table 1.
Electrical Resistivity of Cu Film and Seven Layer Films of TiC, Ti and Cu at Room Temperature

SAMPLES		RESISTIVITY (μohm-cm)
Cu		2
TiC/Cu/Ti/Cu/Ti/Cu/Ti	1A	2
	1B	2
	1C	2
TiC/Cu/TiC/Cu/TiC/Cu/Ti	2A	2.5
	2B	2.5