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Preparation of single-crystal TiC (111) by radio frequency magnetron sputtering at low temperature

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ABSTRACT

Single-crystal films of TiC (111) have been synthesized at room temperature on Al_2O_3 (0001) substrates by radio frequency magnetron sputtering using a compound Ti–C target. The substrate temperature and bias were varied to explore the influence of deposition parameters on the crystal structure. Both Al_2O_3 (0001) and Si (100) substrates were used for epitaxial growth of TiC films. A series of characterizations of TiC films were carried out, including Rutherford backscattering spectroscopy, X-ray diffraction, Raman and X-ray photoelectron spectroscopy. Single-crystal films of TiC (111) on the Al_2O_3 (0001) were demonstrated.

1. Introduction

TiC possesses many special properties, such as high melting point, high thermal stability, exceptional hardness, outstanding wear resistance and good corrosion resistance, which make it of interest in many fields including wear-resistant coatings on cutting and grinding tools, thermal barriers and corrosion resistance on metallic structure.

TiC thin films have been synthesized by various methods such as chemical vapor deposition [1,2], pulsed laser deposition [3], arc ion plating [4], and sputtering [5–8]. The disadvantage of these techniques, however, is that the microstructure of the film is highly dependent on the deposition temperature. Synthesis of single-crystal TiC films requires high temperature. Lower deposition temperature usually leads to amorphous or polycrystalline films. Recently it has been demonstrated that epitaxial carbide films could be deposited by co-evaporation of C_{60} and Ti at very low temperature [9,10]. Up until now no report on synthesis of single-crystal TiC films at room temperature has been made. This paper will introduce a method of growing single-crystal TiC films at room temperature and explore the effects of various parameters on crystal microstructure.

2. Experimental details

2.1. Specimen preparation

The TiC films were prepared by a radio frequency (RF) magnetron sputtering method. The films were deposited on 10 cm by 15 cm Al_2O_3 wafers. The wafers were cleaned several times in an ultrasonic bath containing a solution of ethanol and then were dried and loaded into the sputtering chamber immediately to avoid contamination. The compound target was fabricated by a Ti disk (purity 99.99% and 75 mm in diameter) and sector carbon sheets with the Ti/C area ratio of 2.429. The target was mounted on the RF electrode and located 50 mm from the substrate. The substrate was mounted in a stainless steel holder which was biased negatively to ground. The base pressure before deposition was less than 3×10^{-5} Pa, using a turbo-molecular pump. In order to decrease doping of H from H_2O adsorbing on the wall, the chamber was baked at about 200 °C for 3 h. The working gas used for discharge was argon (99.999% purity). In order to investigate the effects of the substrate and deposition temperature on the TiC films, both Al_2O_3 (0001) and Si (100) substrates were used and a series of substrate temperatures from room temperature to 700 °C were performed. The total pressure of the sputtered gas was controlled at 0.1 Pa in order to obtain stable plasma. In this experiment, the flux of Ar was 1.8 sccm (standard cubic centimeter per minute). The applied bias during the deposition was fixed at −60 V except for when the effects of bias voltage were studied. The RF power for all depositions was 100 W. The film thickness and deposition rate were about 200 nm and 3 nm/min respectively.

2.2. Ion beam analysis

The composition and thickness of the TiC films were measured by using non-Rutherford scattering (non-RBS) of 3.65 MeV He^+ ion beam. At this energy the scattering cross section of C is significantly enhanced over the standard Rutherford cross section. The incident angle of the beam was 0°, while He^+ ions backscattered by target were

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detected at a scattering angle of 165°. All the non-RBS spectra were simulated by the SIMNRA 6.0 codes. Non-RBS measurements were performed at the NEC 9SDH-2 tandem accelerator at the Institute of Modern Physics of Fudan University, China.

2.3. X-ray diffraction (XRD) analysis

The crystal structure was investigated by XRD. The measurement was performed at a D/MAX 2550 V X-ray diffractometer made by Rigaknu, Japan. This diffractometer with Cu K_α source was used in a 2θ mode, 2θ varying from 20° to 68° with a 0.02° step.

2.4. Raman spectroscopy analysis

Raman spectroscopy is based on Raman effect, in which the incident photons are scattered inelastically by molecules or crystal lattices and collected. From the Raman spectra the details of the crystal structure can be determined. In this study the Raman spectra were measured over the range from 200 to 1800 cm^{-1} using a Jobin Yvon HR800 confocal Raman with the laser wavelength of 632.8 nm. The spectra were record with acquisition time of 100 s.

2.5. X-ray photoelectron spectroscopy (XPS) analysis

XPS was recorded using a Kratos AXIS ULTRA DLD instrument with the source of an Al K_α monochromatic X-ray. The gun was operated at 15 kV and 10 mA. Survey spectra were acquired from 0 to 1200 eV in steps of 1 eV for the pass energy of 160 eV. Region scans from selected ranges were obtained using the pass energy of 20 eV. The peaks were acquired from the selected elements Ti 2p, C 1s and O 1s. The base pressure during measurement is lower than 10^{-7} Pa. In order to avoid the effects of oxides, the samples were etched by Ar^+ for 3 min using the source of 2 kV and 30 mA over an area of 3×3 mm^2. The spectra were record for both unetched and etched samples.

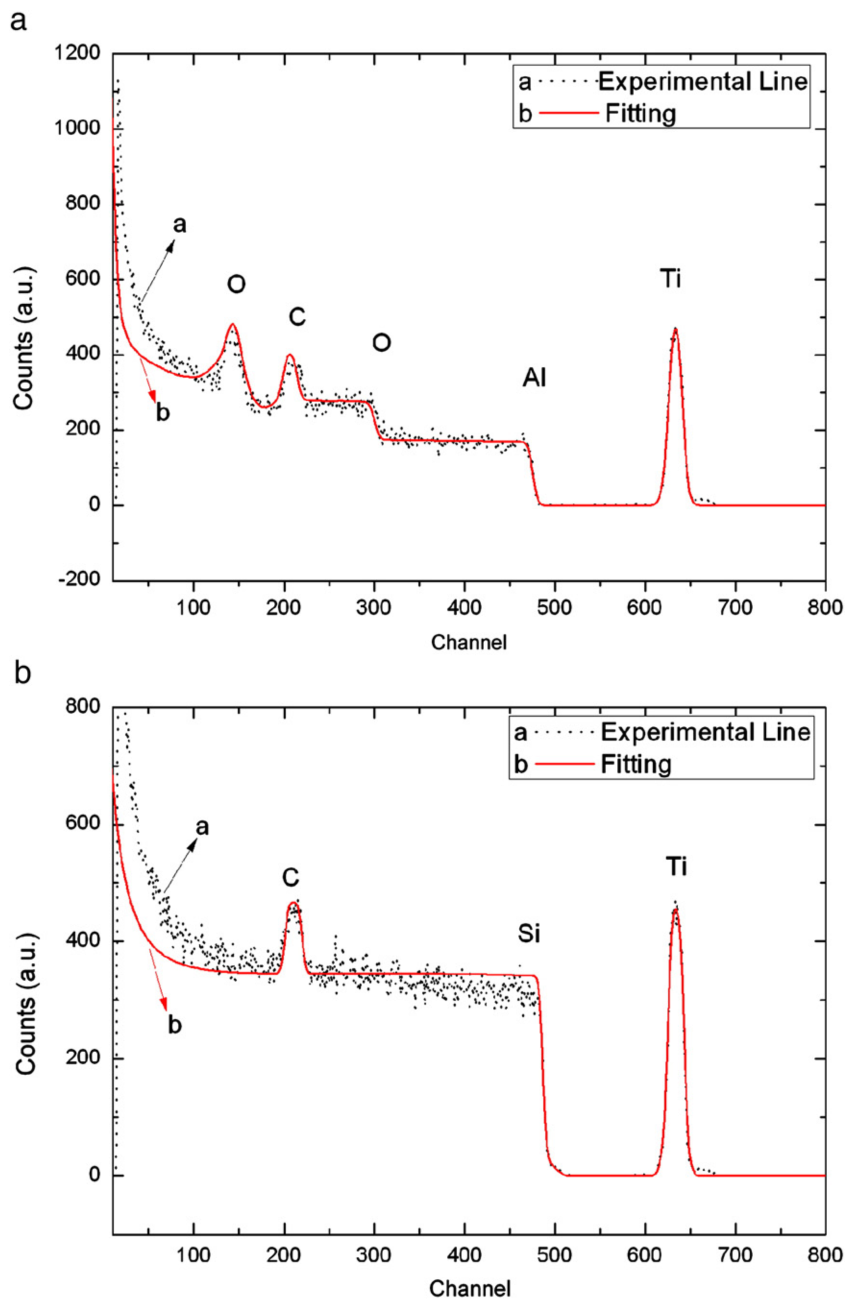

Fig. 1. RBS spectra of TiC films deposited on (a) Al_2O_3 substrate and (b) Si substrate with the deposition power of 100 W and bias voltage of −60 V.