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Preparation and properties of titanium silicide coating glass by CVD

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Abstract

Titanium silicide thin films were prepared on glass substrates by chemical vapor deposition using SiH_4 and $TiCl_4$ as the precursors. The phase structure of the thin films was identified by XRD. The surface morphology of the thin films was observed by FESEM. The sheet resistance and optical behaviors of the thin films were measured by the four point resistivity test system and FTIR spectrometer, respectively. Titanium disilicide ($TiSi_2$) thin films with the face-centered orthorhombic structure are formed. The suitable formation temperature of the $TiSi_2$ crystalline phase is about 710 °C. The formation of $TiSi_2$ crystalline phase is dependent on the thickness of thin films and a quantity of the crystalline phase of $TiSi_2$ in the thin film is directly related to mole ratio of $SiH_4/TiCl_4$. The sheet resistance of the $TiSi_2$ thin films is dependent on the formation of the $TiSi_2$ crystalline phase. With the mole ratio of $SiH_4/TiCl_4$ of 3, the lowest sheet resistance (0.7 Ω/□) of titanium silicide thin film is formed at 710 °C. The maximum reflectance of the $TiSi_2$ thin films is about 0.95 on the broad IR heat radiation. A related reaction mechanism was proposed.
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1. Introduction

To maintain comfortable indoor condition, two kinds of coating glasses have been applied to the window areas of a building. One is solar controlled films; the other is low emission coating glass. Solar controlled film is used for controlling the solar energy entering through the window areas of a building in warm climates. More recently, reflecting and absorbing films have been applied to clear glass to achieve solar control. Reflection of unwanted radiation is more efficient than absorption, since reflection eliminates the radiation completely, whereas the part of the absorbed heat is eventually carried into the building [1]. Low emission (Low-E) coating glass is used for heat insulation where unhindered light transmission is required in cold climates. By reflecting in the IR it suppresses the exchange of heat radiation, which is often the most important energy leak in a system [2].

Window glass may not be changed frequently, and coating glass combined with the function of solar control and Low-E has attracted considerable attention in recent years. Titanium disilicide ($TiSi_2$) thin film has the properties of solar shielding and reflection of broad IR heat radiation. Due to low resistivity and good thermal stability of $TiSi_2$ [3–7], it has been widely applied to gate, contact, and interconnect metallization in ultra large scale integration (ULSI) technologies [4,8–11]. The low resistivity of $TiSi_2$ thin film has high reflectivity on the low-frequency electromagnetic wave, and it has color-neutral and can shield

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windows from excessive solar heat gain [1]. Therefore, a new type of coating glass will be exploited if $TiSi_2$ thin film is successfully prepared as a glass coating.

In this paper, we first report $TiSi_2$ thin film prepared on glass. Because of the demand of industrial production and large scale preparation in situ, $TiSi_2$ thin films were prepared on glass substrates by atmospheric pressure chemical vapor deposition (CVD). The relationship between the properties and the preparation of $TiSi_2$ thin films was investigated.

2. Experimental procedures

Titanium silicide thin films were deposited on glass substrates in a hot-wall quartz reaction chamber. SiH_4 was used as the silicon gas precursor. Nitrogen was used as a carrier gas, which went through two bubblers with liquid $TiCl_4$ to transport the Ti precursor to the reaction chamber. The deposition temperature and deposition time were controlled within the range between 650 °C and 750 °C and between 30 s and 180 s, respectively. The composition ratio of $SiH_4/TiCl_4$ was allowed from 1 to 5. The total gas flow rate and the concentration of ($SiH_4 + TiCl_4$) in total gases were used between 600 sccm and 1400 sccm, and between 1.9% and 4.4%, respectively.

The phase structure and the surface morphology of the thin films were identified by X-ray diffractometry (XRD) and field emission scanning electron microscopy (FESEM), respectively. The IR-reflectance of the thin films was observed by Fourier transfer infrared spectrometer. The sheet resistance of the titanium silicide thin films was measured using the four point probe method. Sputtered gold electrodes were deposited on the face of each sample. The distance between electrodes was 1.5 mm. Using this method, the relative error in the sheet resistance of $TiSi_2$ thin films was within ±5%.

3. Results

Fig. 1 is the XRD patterns of silicide thin films deposited for 120 s at different temperatures. It shows that crystalline phase of $TiSi_2$, which has face-centered orthorhombic structure do not form in the thin film at 670 °C and start to form when the thin film is deposited at a little bit higher temperature of 690 °C. With the deposition temperature increasing to 710 °C, the peak intensities of the $TiSi_2$ phase are increased to their maximum. With the deposition temperature increasing continuously from 710 °C to 750 °C, the peak intensities decrease gradually, although the phase of $TiSi_2$ still appeared in the thin film.

Fig. 2 shows the XRD patterns of the thin films deposited with different mole ratio of $SiH_4/TiCl_4$. For $SiH_4/TiCl_4 = 1$, there are only small peaks of Ti_5Si_3 phase formed. Increasing the ratio of $SiH_4/TiCl_4$ to 2, the $TiSi_2$ phase is formed while the Ti_5Si_3 phase is disappearing. The intensity of the $TiSi_2$ phase increases to the maximum with increasing the mole ratio of $SiH_4/TiCl_4$ to 3. After that, it begins to decrease with the increase in the mole ratio of $SiH_4/TiCl_4$ to 4, there is only the phase of $TiSi_2$, but the intensity decreases. The peaks of $TiSi_2$ phase finally disappear and two small peaks of Si phase appear when the mole ratio of $SiH_4/TiCl_4$ increases to 5.

Table 1 gives the sheet resistance (R_S) of the thin films deposited with different mole ratio of $SiH_4/TiCl_4$. The lowest sheet resistance was exhibited in the case with the mole ratio of $SiH_4/TiCl_4$ of 3. Their morphologies are shown in Fig. 3. In Fig. 3(a) ($SiH_4/TiCl_4 = 2$), some small crystalline particles are exhibited. In Fig. 3(b) ($SiH_4/TiCl_4 = 3$), the crystalline particles are evidently the largest and the

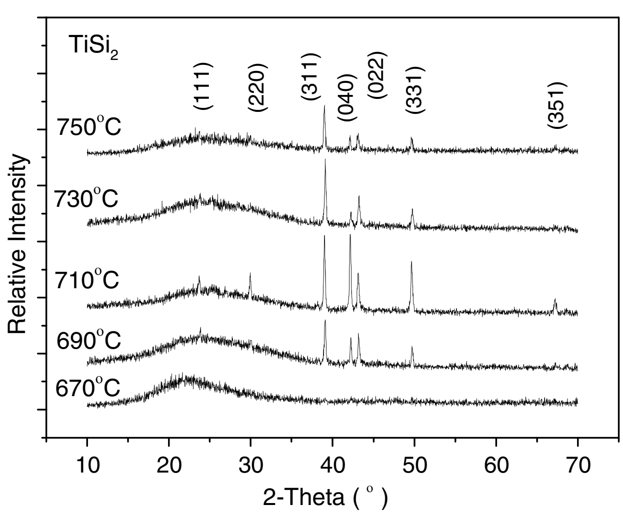

Fig. 1. XRD patterns of the thin films on glass substrates at different deposition temperatures for a total flow rate of 1200 sccm, a mole ratio of $SiH_4/TiCl_4$ of 3, a flow rate ($SiH_4 + TiCl_4$) of 26.6 sccm and a 2 min deposition condition.

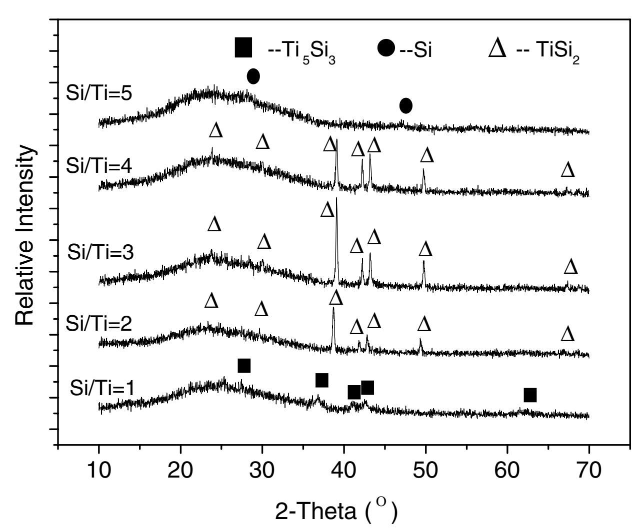

Fig. 2. XRD patterns of the thin films on glass substrates with varying mole ratio of $SiH_4/TiCl_4$ for a constant total flow rate of 1200 sccm, a flow rate ($SiH_4 + TiCl_4$) of 26.6 sccm, 710 °C and 2 min deposition condition.

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