# STUDY OF MULTILAYER STRUCTURE USING X-RAY DOUBLE CRYSTAL DIFFRACTION*

Wu Yunzhong , Xu Xueming , Wang Weiyuan
State Key Lab. of Transducer Technology
Shanghai Institute of Metallurgy, Chinese Academy of Sciences
Shanghai 200050 China

## ABSTRACT

By using X-ray double crystal diffraction technique the multilayer structure composed of glass membrane, platinum film and $\alpha$-$Al_2O_3$ substrate has been studied. It is found the stress is produced in the film by thermal mismatch within multilayer materials. The measuring results of thin film platinum resistors show that the stress were induce resistance change of device and different stress status will produce add resistance in different direction. Selecting proper glass material can make opposite stress in Pt film and opposite add resistance due to thermal mismatch. The reliability of Pt resistor has been improved with method of this stress compensation.

## 1. INTRODUCTION

Thin film platinum resistor is a new generation of temperature sensor. This device is a multilayer structure composed of glass membrane, Pt film and ceramics substrate. Results studied show that the failure of this sensor is caused by thermal mismatch stress within multilayer materials. By using X- ray double crystal diffraction technique the multilayer structure of glass/Pt/$\alpha$-$Al_2O_3$ that resembles to glass/Pt /ceramics has been studied in this paper. The purpose is to explore a method improving the reliability of thin film platinum resistor.

## 2. EXPERIMENT

When a prefect slice is effected by stress it will bend, using X- ray double crystal diffraction technique can measure indestructibly the curvature radius of the slice. The basis of the radius of curvature measurement is the noncoincidence of $K_{\alpha 1}$ and $K_{\alpha 2}$ double lines which occurs when both crystals in the double crystal spectrometer are not identical. For a pair of prefect crystals in the parallel,(1,-1) position, the spectrometer is dispersion free (Fig.1). So that, a double peak presents in the spectrometer for a bent crystal, the radius of curvature can be calculated according to the dispersion of both peaks[1]

$$r = \frac{1}{\cos\theta} \cdot \frac{\lambda}{\Delta\lambda} \cdot \frac{L}{\Delta\Phi} \qquad (1)$$

---

* This paper was supported by National Science Foundation of China.

where $\theta$ is the Bragg angle for the radiation, $\lambda$ is the wave length of X- ray, $\Delta\lambda$ is doublet separation, L is the geometrical length from focus of X-ray to second crystal. $\Delta\Phi$ is the angular resolution. Using cooper radiation on $\alpha$-$Al_2O_3$(4408), $\theta$=62.41°, $\lambda_1$=0.15405nm, $\lambda_2$=0.15443nm, L=575mm of study here leads to

$$r=635.4(m)/\Delta\Phi(s) \qquad (2)$$

The stress in film deposited on substrate is calculated from relationship[2]

$$\sigma = \frac{E}{6(1-\upsilon)}\left(\frac{D^2}{t}\right)\left(\frac{1}{r_f}-\frac{1}{r_0}\right) \qquad (3)$$

where D is the thickness of substrate, E and $\upsilon$ are the Yang's modulus and Paission's ratio of the substrate, respectively. $r_0$ and $r_f$ are the radius of curvature of the film/substrate combination before and after deposited, and t is the thickness of film. If thermal mismatch stress make the combination of film /substrate bend to down during heating or cooling than tension stress is induced in the film, we define the curvature radius to be minus. On the contrary, the sample bend to up, than, the stress in film is pressure stress, we define the radius as right. Because X-ray double crystal diffractometry is only applicable to single crystal, so the multilayer structure glass/Pt/ $\alpha$-$Al_2O_3$ were studied using (1102) $\alpha$-$Al_2O_3$ to substitute for ceramics in this work. Samples of the study here are treated as follows:

1. Six $\alpha$-$Al_2O_3$ wafers annealed at 1000℃,30', then cooled slowly to room temperature.
2. deposit Pt film on five wafers among which as mentioned above, then treat them at 1000℃,30' and number them 1#,2#,3#,4# and 5#, respectively. Another one of the six wafers is numbered O.
3. Make glass A to coat on samples 2# and 3# and treat them at 800℃,30'.
4. Again treat sample 3# at -80℃, 4h.
5. Glass B is coated on samples 4# and 5# and treat the samples at 800℃, 30'.
6. Again treat sample 5#at -80℃ ,4h.

## 3. RESULTS

The X-ray double crystal diffractometry of sample O is shown in Fig.2. It is a single peak that half high width of peak is equal to 7.6 " , it means that this $\alpha$-$Al_2O_3$ is a prefect crystal stress free. Diffractometry of sample 1# (Pt/$Al_2O_3$) is shown in Fig. 3. It presents obviously a double peak. Sample 2# to 5# also are double peak, however for 1# and 4# $K_{\alpha 2}$ peak occurs at the left of $K_{\alpha 1}$ peak and for 2#,3# and 5# $K_{\alpha 2}$ peak do at the right. It means that the Pt film on samples 1#and 4# are effected by tension stress and the Pt films on 2#,3# and 5# are effected by pressure stress. The measurement results of all six samples are shown in table 1.

**Table 1. measurement results of six samples**

| sample | structure and treated | radius of curvature(m) | stress status |
|---|---|---|---|
| 0 | $\alpha$-$Al_2O_3$ | $\infty$ | stress free |
| 1 | $Pt/Al_2O_3$ | -79.4 | tension |
| 2 | A/Pt/$\alpha$-$Al_2O_3$(800℃) | +22.7 | pressure |
| 3 | A/Pt/$\alpha$-$Al_2O_3$(-80℃) | +21.6 | pressure |
| 4 | B/Pt/$\alpha$-$Al_2O_3$(800℃) | $\approx\infty$ | tension |
| 5 | B/Pt/$\alpha$-$Al_2O_3$(-80℃) | +64.8 | pressure |

## 4. DISCUSSION

There are two kinds of stress in the film as-deposited on substrate. One is the stress caused by deposition process, which can be eliminated by proper heat treatment afterwards. Another is the thermal stress induced by the difference of thermal coefficient of expansion (TCE) between film and substrate, it is the stress that We do study. For the Pt/$\alpha$-$Al_2O_3$, sample 1#, TCE of $\alpha$-$Al_2O_3$ is smaller than that of Pt film, the contraction of Pt film is limited by the substrate when Pt/$\alpha$-$Al_2O_3$ combination is cooled from high to room temperature, tension stress is induced in Pt film. From formula(1), $\sigma = 3.5 \times 10^4 N/cm^2$ . In glass/Pt/$\alpha$-$Al_2O_3$ structure Pt film is effected from two sides, glass membrane and $\alpha$-$Al_2O_3$ substrate. Because TCE is different between glass A and B which We use, the stress in Pt film is also different for multilayer structures using glass A coating or glass B coating. Results We obtain are shown in Table 1.

It is well known that stress can induce resistance change of materials. Results we study indicate that tension stress make resistance increase and pressure stress makes it decrease. If selecting a proper glass material to make coating of Pt resistor, tension stress is produced in Pt film after high temperature treatment and pressure stress is induced after low temperature treatment, than the whole resistance change of this device can be limited to minimum. Glass B is the material that we hope as shown in Table 1. It is called stress compensation method. It is to use the method that the reliability of thin film platinum resistor has been improved.

## 5. CONCLUSION

1. Multilayer structure composed of glass membrane/Pt film/Pt/$\alpha$-$Al_2O_3$ has been studied using X-ray double crystal diffraction technique.
2. Thermal mismatch of three materials induces stress in Pt film.
3. Using different glass coating on device surface induce different stress in film.
4. Selecting proper glass material can make different stress in Pt film for identical structure. It is to use stress compensation method that the reliability of thin film platinum resistor has been improved.
5. How to calculate stress in multilayer structure had over two layers has to be investigated.

## ACKNOWLEDGE

The authors wish to thank Mr. J.Y.Chen for helpful suggestion in measurement of X-ray.

## REFERENCES

1. B.G.Coken and W.Focht, solid-states Electronics, 13(1970),105

2. D.S.Cambell, Handbook of Thin Film Technology, Chap.12, Ed by I. Maissel and R.Glang McGraw Will, New York,(1970)

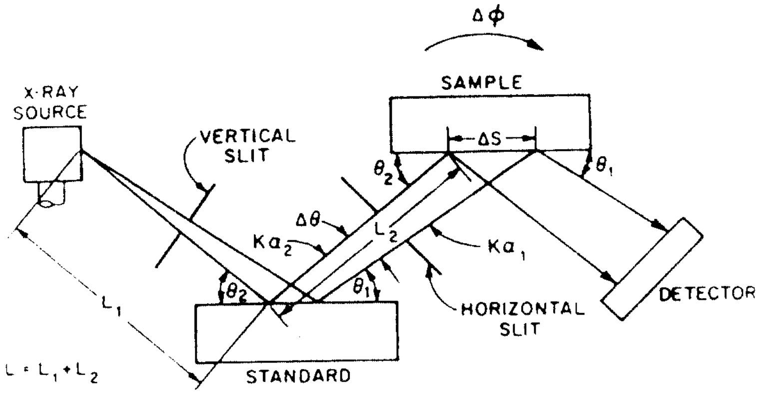

Fig.1. The parallel, (1, -1), nondispersive setting of a double crystal spectrometer.

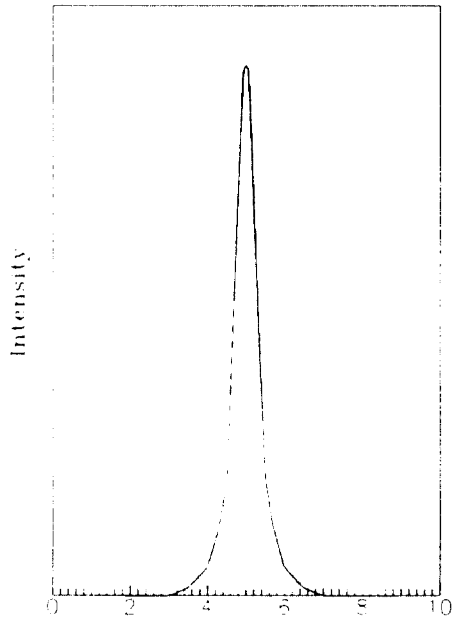

Fig.2. Diffractometry of sample 0#.

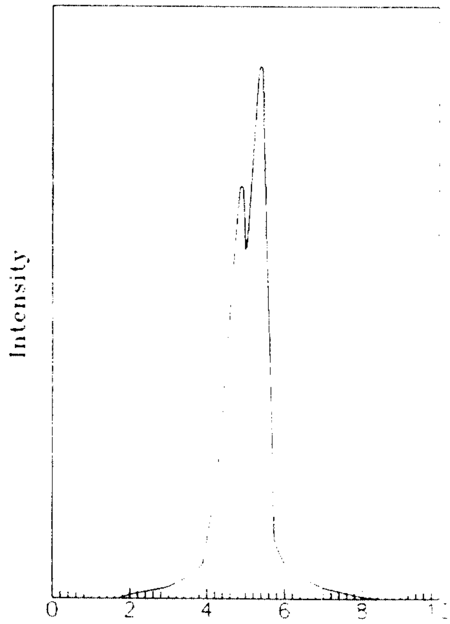

Fig. 3. Diffractometry of sample 1#.