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RESISTIVITY-THICKNESS DEPENDENCE OF POLYCRYSTALLINE TITANIUM FILMS

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Transmission electron diffraction analysis showed that thin titanium films (thickness range from 10.9 to 113 nm) have polycrystalline structure. Films were deposited at a rate of 0.5—0.8 nm/s onto fresh cleaved mica substrates maintained at room temperature. Transmission electron microscopy indicated that the films are continuous, with needlelike structure. Fitting of resistivity-thickness relation has been carried out at different measuring temperatures. Agreement with Mayadas-Shatzkes theory was proved. The values of temperature coefficients of resistivity were positive. The mean free path of conduction electrons decreased from 44.19 to 21.43 nm with increasing temperature from 308 to 463 K.

1. Introduction

The electrical properties of titanium films have been studied by some workers[1-4]. Postnikov et al.[5] reported that monocrystalline films of titanium could be formed during condensation in vacuum. Ibrahim et al.[6] studied the dependence of electrical conduction mechanism of the titanium films on its structure. In this respect, they[6] prepared thin titanium films at a rate of 0.5—0.8 nm/s onto amorphous glass substrates maintained at room temperature. The aim of the present work

is to study the electrical properties of titanium films deposited onto fresh cleaved crystalline mica substrates. This might give an idea about the effect of the crystalline substrate on the resistivity-thickness dependence of thin titanium films.

2. *Experimental*

The titanium films were prepared by thermal evaporation technique under vacuum of 1.3×10^{-3} Pa. Spec pure titanium (Ti) of 99.999% was used. The film thickness ranged from 14 to 113 nm as measured by multiple beam Fizeau fringes method[7] and controlled by quartz crystal monitor. The rate of deposition was 0.5—0.8 nm/s and the deposition was carried out onto fresh cleaved mica substrates maintained at room temperature. The electrical measurements were done by Portable Potentiometer No. 7569 P (England) using aluminium electrodes. ELMID2 electron microscope (45 kV) was used for the investigation of titanium films by transmission electron microscopy and diffraction techniques.

3. *Results*

The electrical resistance R of thin Ti films of different thicknesses d was measured as a function of measuring temperature T. It was found that R increases gradually with increasing T from 308 to 463 K, while it decreases with increasing d from 14 to 113 nm as shown in Fig. 1. The increase of R with T is very remarkable in case of low film thickness. R-T dependence could be correlated with the polycrystalline structure of Ti films, because amorphous metallic films might lead to the decrease of R with T. Figs. 2 a, b show the electron diffraction patterns of Ti films of thicknesses 62.04 and 78.27 nm as representative examples for polycrystalline structure of Ti films.

In order to show whether the electrical results of Ti films relate to Fuchs-Sondheimer[8-10] theory (F-S) or Mayadas-Shatzkes[11] theory (M-S), it was necessary to determine the values of resistivity ϱ at different temperatures and plot relations between ϱ/ϱ_0 and d. The values of bulk resistivity ϱ_0 were determined by plotting a relation between ϱd and d at different temperatures (as shown in Figs. 3a to c at T 308 to 423 K), from which ϱ_0 and mean free path l of conduction electrons were determined according to the following equation of (F-S)[8-10]

$$\varrho = \varrho_0 \left[1 + \frac{3\,l\,(1-P)}{8d} \right], \tag{1}$$

where P is the specularity parameter, which might be taken as zero for total diffuse scattering when the grain size is comparable with the film thickness d and $d/l > 1$. By this way, values of l and ϱ_0 were determined as listed in Table 1:

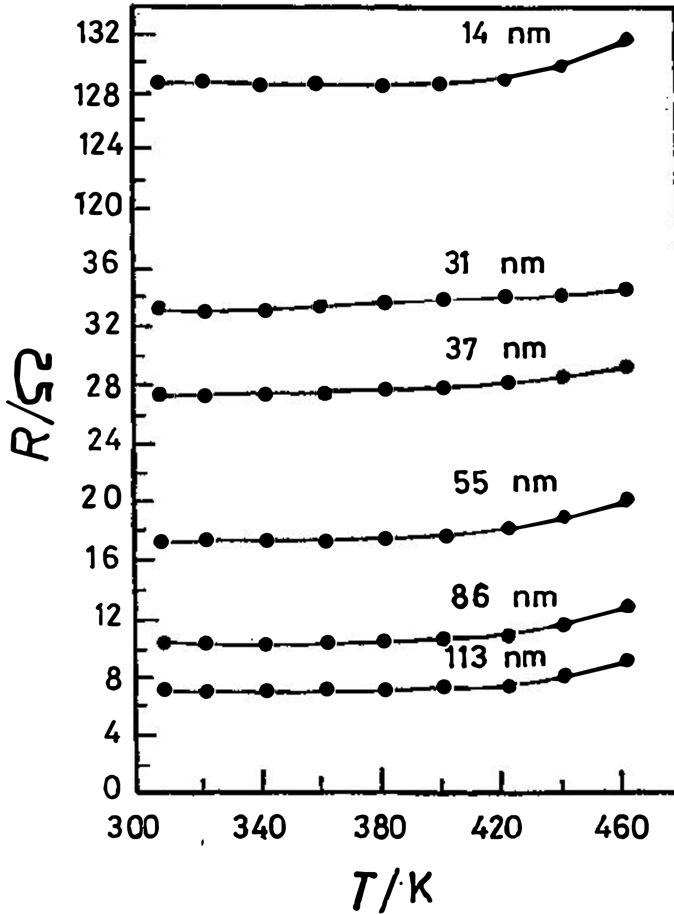
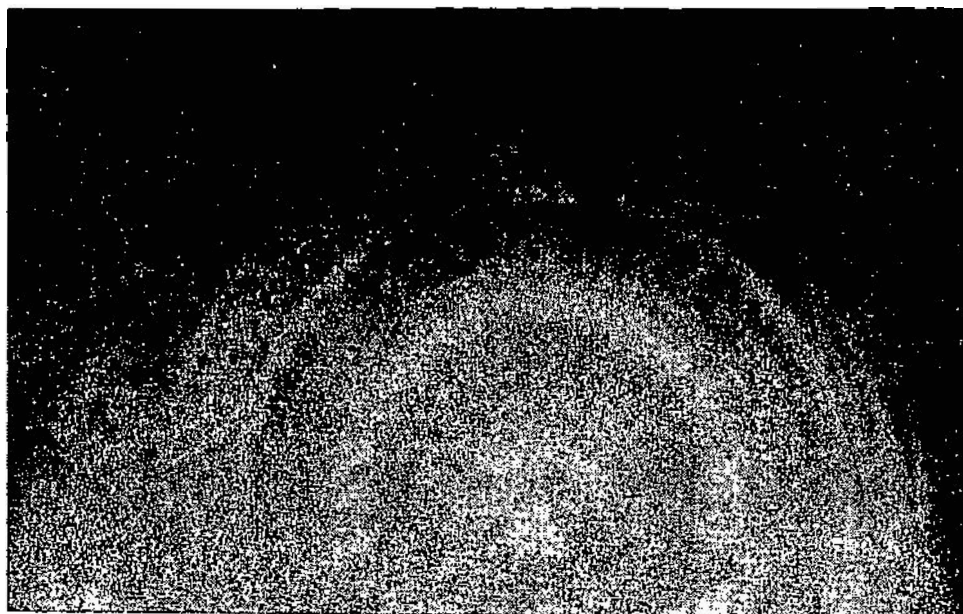


Fig. 1. Relation between resistence R and temperature T at different thicknesses.

TABLE 1

T/K	l/nm	$\varrho_0/\mu\Omega\cdot$cm
308	32.7	171
363	22.2	180
423	19.7	190

Values of l and ϱ_0 of thin Ti films at different T.



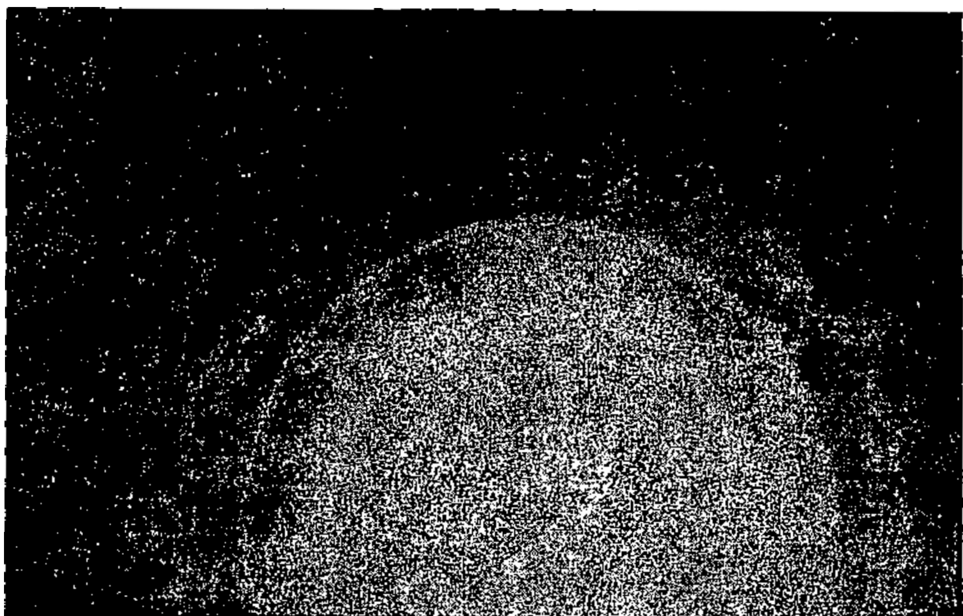


Fig. 2. Electron diffraction patterns of Ti films with thicknesses of: a) 62.04 nm, b) 78.27 nm.

Figs. 4a to c show the relations between ϱ/ϱ_0 and d at different measuring temperatures 308, 363 and 423 K, respectively. In these figures, block circles represent our experimental results, solid lines represent theoretical data using equation (1), whilst dashed lines represent theoretical data according to the following M-S[11] equation

$$\varrho = \varrho_0 \left[\frac{1}{f(a')} + \frac{3\,l\,(1-P)}{8d} \right], \tag{2}$$

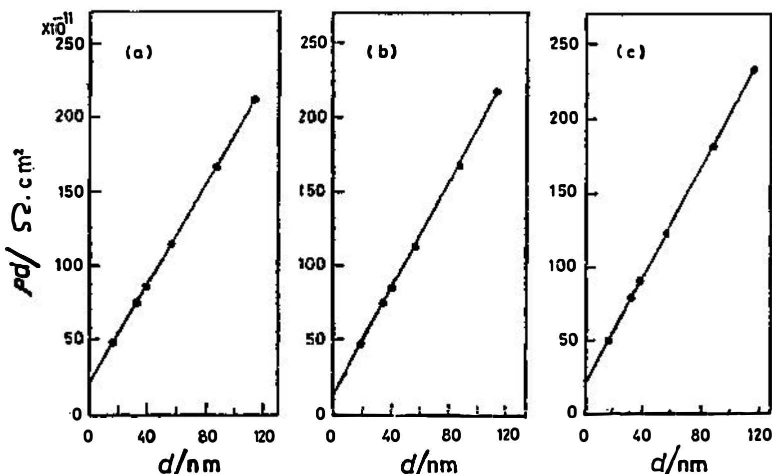


Fig. 3. Relation between ϱd and d at different temperatures: a) at 308 K, b) at 363 K, c) at 423 K.

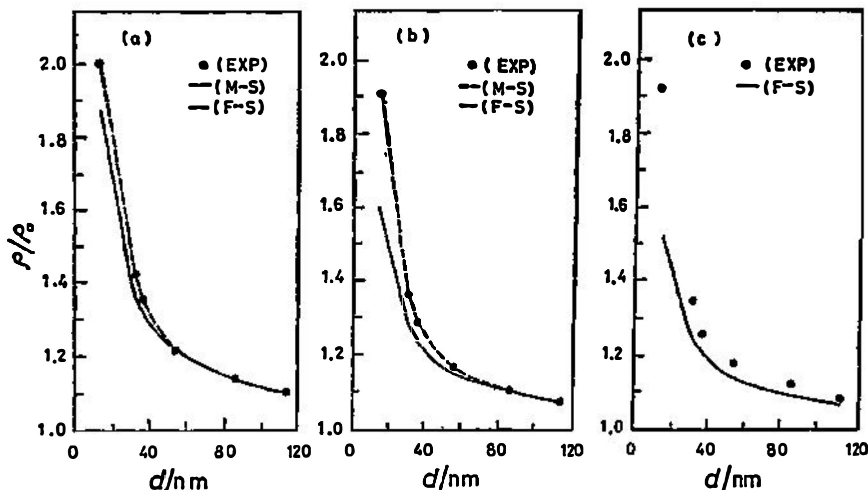
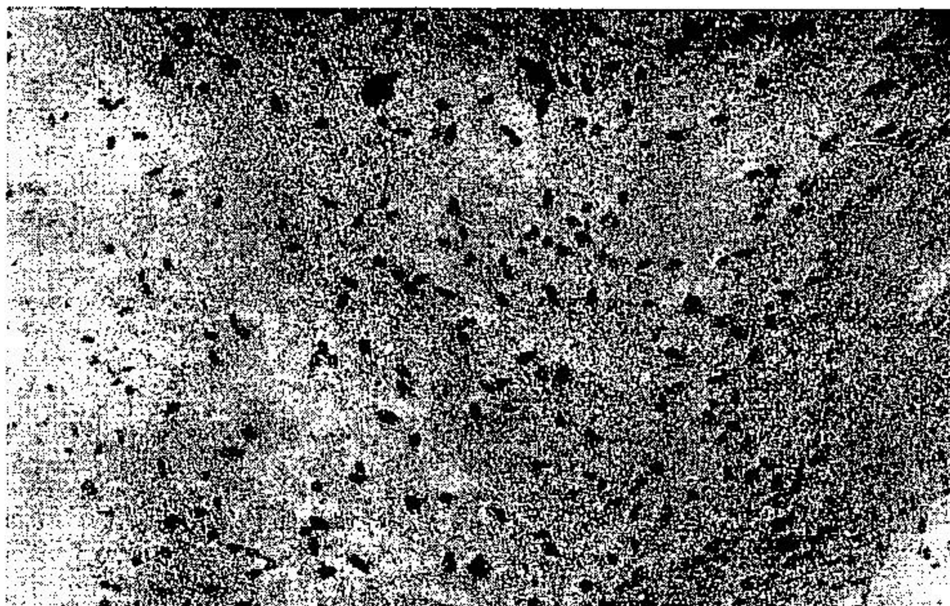


Fig. 4. Relation between resistivity ratio ϱ/ϱ_0 and thickness d: a) at 308 K, b) at 363 K, c) at 423 K.



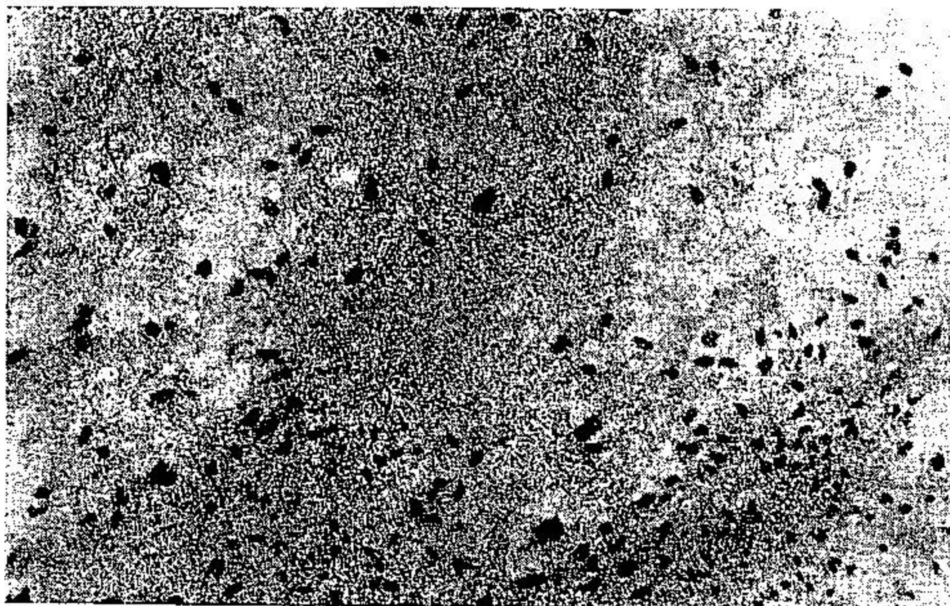


Fig. 5. Transmission micrograph of Ti films with thicknesses of: a) 10.9 nm, b) 44.3 nm.

where $f(\alpha') = 1 - \frac{3}{2}\alpha'$ and α' is known as physical quantity. It is clear that the experimental results are mainly related to M-S[11] theory where they deviate from F-S theoretical curves as in Figs. 4a and b. The accuracy of fitting between our results and M-S curve is about 98%. Moreover, the present experimental results have shown a marked discrepancy from the theoretical curve of F-S as shown in Fig. 4c. It was possible from the estimated values of α' to determine the values reflection coefficient of the scattered electrons at grain boundaries r according to the following formula

$$\alpha' = \frac{l}{a_g} \cdot \frac{r}{1 - r}, \tag{3}$$

where a_g is the grain size of Ti films, which was carefully determined from the observations by transmission electron microscopy (see e. g. Figs. 5a, b for Ti films of thickness 10 and 44.3 nm, respectively). It was found that the grain size of Ti films increased from 135 to 216 nm with increasing film thickness from 10.9 to 62.0 nm. Therefore, it was of interest to determine the values of α' and r at different measuring temperature and for different film thicknesses. Such values of α' and r are listed in Table 2.

TABLE 2

T/K	α'	r
308	0.08	0.12
363	0.2	0.45
423	—	—

Values of α' and r at different T of thin Ti films

Besides, the bulk temperature coefficient of resistance of Ti was determined from a relation between α and $\frac{1}{d}$ as shown in Fig. 6. By applying F-S[8-10] equation, α_0 was determined as follows:

$$\alpha = \alpha_0 \left[1 - \frac{3l(1-P)}{8d}\right]. \tag{4}$$

It was found that α_0 is 0.001 K^{-1}.

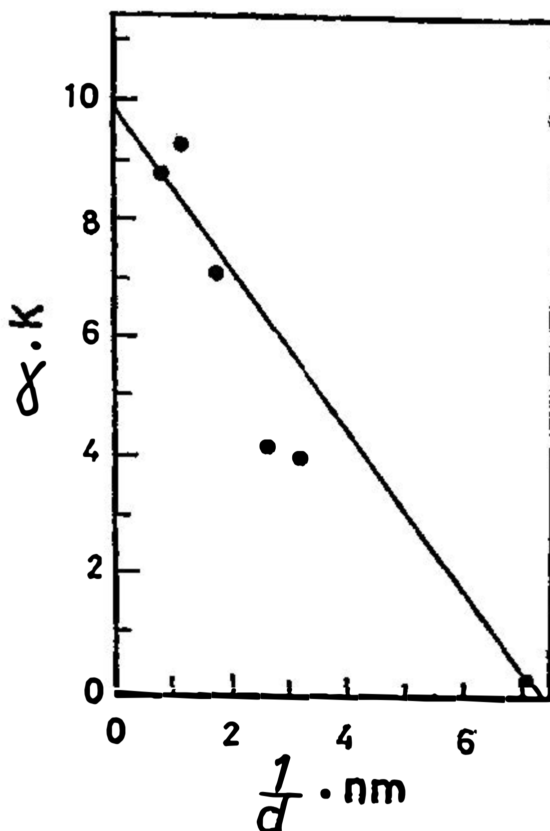


Fig. 6. Relation between temperature coefficient of resistance α and $\frac{1}{d}$.

4. *Discussion and conclusions*

From the present work, it was found that thin Ti films of different thicknesses deposited onto mica substrates have polycrystalline structure. This was not expected since the mica substrates have preferred sites for epitaxial growth of single crystalline state. However, it seems that results of present observation is in agreement with the work done by Gould et al.[4] who found that single crystal Ti films were prepared onto mica substrates heated to 420°C, while Grunbaumand Schwarz[12] found that epitaxial Ti films were found on mica at temperature of 500—600°C. This means that heating of mica substrate could lead to the formation of single crystalline Ti films.

The polycrystalline structure of Ti films is related to the behaviour of resistivity with temperature which gives rise to positive α. Moreover, the resistivity-thickness dependence of Ti films showed a reasonable agreement with *M-S*[11]

theory, because the agreement of polycrystalline metallic films with F-$S^{8-10)}$ theory is very difficult as has been found by El-Nahass et al.[13], Ibrahim et al.[6] and Abou-Saif et al.[14,15] during their work on thin films of Cr (glass substrate), Ti (glass substrate) as well as In and Sn (glass substrate). The agreement of present results with M-$S^{11)}$ theory was proved at lower measuring temperatures 308 and 363 K.

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OVISNOST OTPORA POLIKRISTALIČNIH TANKIH SLOJEVA TITANA O DEBLJINI

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Difrakcijom elektrona pri prolazu kroz tanke slojeve titana (debljine od 10,9 do 113 nm) utvrđeno je da slojevi imaju polikristaliničnu strukturu. Relacija koja opisuje ovisnost otpora o debljini je podešavana za mjerenja na nekoliko raznih temperatura, i nađeno je da vrijedi Mayadas-Shatzkesova teorija. Nađeno je da su termički koeficijenti otpornosti pozitivni, te da srednji slobodni put vodljivih elektrona pada sa 44,19 na 21,43 nm kada temperatura raste od 308 do 463 K.