

for critical reading of the manuscript and Plessey Research (Caswell) Ltd., where the samples were made. A SRC Case studentship is gratefully acknowledged.

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Received 18 August  
and accepted 10 October 1980

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## *The effect of substrate temperature on the electrical properties of thin chromium films*

It is generally known that, for films prepared by evaporation, the substrate temperature,  $T_s$ , is one of the most important parameters that influences both crystal properties and electrical properties of grown films. Higher substrate temperature usually leads to (a) larger grain sizes of the crystallites in the films [1] or (b) a greater tendency to form island structures in the films. Although considerable experimental work has been reported on the effect of  $T_s$  on the epitaxial, metallic and semiconductor films [2–4], there is, however, very little work reported on the polycrystalline films [5–7]. In this letter the effect of  $T_s$  on the *in situ* electrical resistivity,  $\rho$ , and the temperature coefficient of resistance, TCR, of evaporated chromium films in the thickness range 100 to 800 Å is reported. The electrical properties of thin chromium films [8] have already been reported.

Chromium of 99.999% purity obtained from Leico Industries, New York, USA, was evaporated from a tungsten helix at a pressure of  $3 \times 10^{-6}$  Torr onto a glass substrate held at different  $T_s$  i.e. 22, 100, 150 and 180°C. The substrate, prior to deposition, was subjected to both ultrasonic and ionic bombardment cleaning. The thickness of the film was monitored during deposition by a quartz crystal monitor. The films were grown at a uniform rate of  $3 \text{ Å sec}^{-1}$ . A constant current source was

used to maintain a current of 0.1 mA during TCR measurements. The TCR measurements have been performed *in situ* ( $\sim 5 \times 10^{-6}$  Torr) with the standard four-probe technique. During TCR measurements, the resistance of the film was measured after the temperature had equilibrated. The films were heated by a radiant heater at a uniform rate of  $7^\circ \text{ C min}^{-1}$  and were cooled at the same rate. The temperature of the film was measured by a chromel–alumel thermocouple held rigidly near the glass substrate. Resistance measurements were carried out both during heating and cooling cycles and were found to be reproducible within an experimental error of about 5%.

The electrical resistivity of thin films can be expressed as [9],

$$\rho = \rho_0 \left[ 1 + \frac{3}{8\lambda} (1-p) \right] \quad 0.1 < \lambda < 10 \quad (1)$$

where  $\rho$  is the resistivity of the film,  $\rho_0$  is the resistivity of the bulk metal,  $\lambda$  is the ratio of the film thickness,  $t$ , to the electron mean free path,  $l$ , in the bulk and  $p$  is the specularity parameter.

Fig. 1 shows the thickness dependence of the resistivity curves for thin chromium films over the thickness range 100 to 800 Å for four different  $T_s$  values (a) 22°C, (b) 100°C, (c) 150°C and (d) 180°C. It is evident from Fig. 1, that the resistivity is higher for all thicknesses at higher

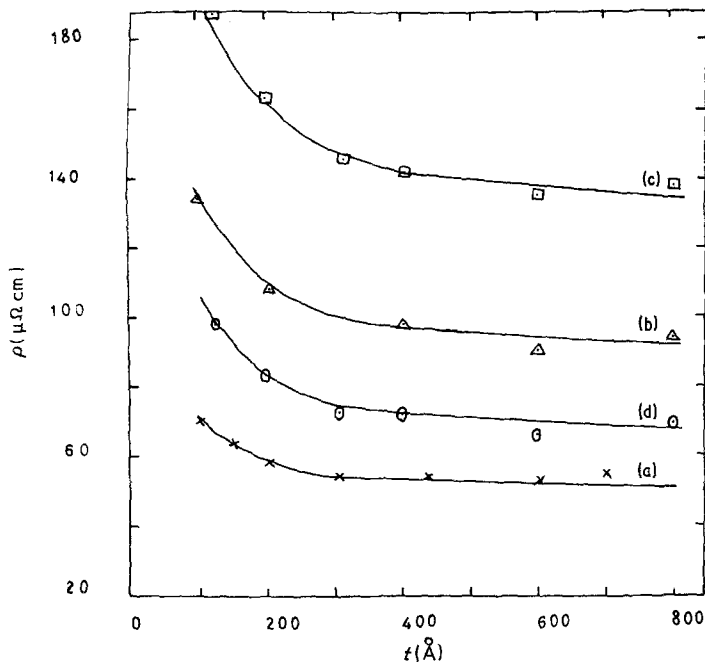


Figure 1 Thickness dependence of the resistivity curves for different substrate temperatures. The continuous lines are theoretical curves according to the Fuchs-Sondheimer theory. The experimental points are shown by  $\times$ ,  $\Delta$ ,  $\square$ ,  $\odot$  for  $T_s = 22, 100, 150$  and  $180^\circ\text{C}$ , respectively.

$T_s$  compared to that at room temperature ( $22^\circ\text{C}$ ). However, the resistivities of films deposited at  $180^\circ\text{C}$  are smaller than the resistivities at  $100$  and  $150^\circ\text{C}$  but greater than room temperature values. The resistivity values, at higher  $T_s$  are confirmed by repeating these *in situ* measurements several times. The initial increase in resistivities of films deposited at  $100$  and  $150^\circ\text{C}$  may be attributed to the increasing tendency to form island structures

[1]. The presence of island structures has been confirmed experimentally [8] by the fact that the TCR is negative for thin chromium films. However, at higher  $T_s$  ( $> 150^\circ\text{C}$ ) the islands grow in size, resulting in a semi-continuous film and hence the resistivity of the films at  $180^\circ\text{C}$  decreases. A similar decrease in the resistivity at higher  $T_s$  is also reported for thin ytterbium films [7] which also exhibited negative TCR for very

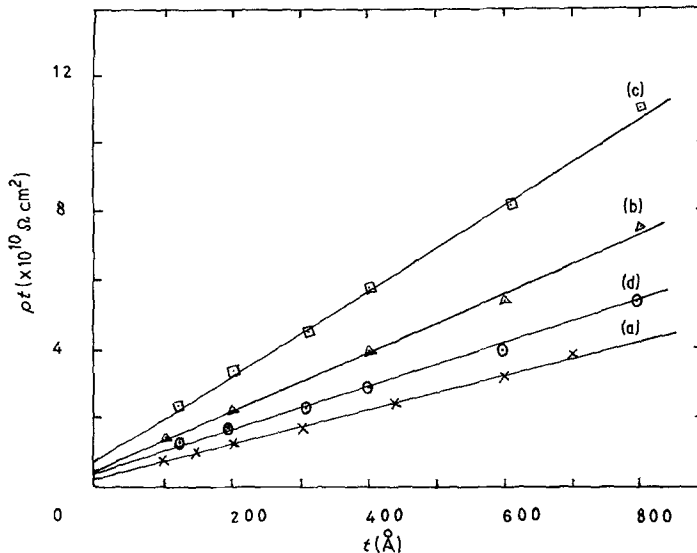


Figure 2  $\rho t$  against  $t$  graphs for four substrate temperatures (a)  $22^\circ\text{C}$ , (b)  $100^\circ\text{C}$ , (c)  $150^\circ\text{C}$  and (d)  $180^\circ\text{C}$ .

TABLE I

Substrate temperature, $T_s$ ( $^{\circ}$ C)	Electron mean free path, $l$ ( $\text{\AA}$ )	Specularity parameter, $p$	Infinitely thick film $\rho_0$ ( $\mu\Omega\text{cm}$ )	$l(1-p)$ ( $\text{\AA}$ )
22	153	0.3	50	107
100	200	0.2	85	160
150	178	0.1	125	160
180	240	0.25	63	180

low thicknesses [10]. Pal *et al.* [11] report a similar increase in the resistivity at higher  $T_s$  for copper films.

In Fig. 2,  $\rho t$  against  $t$  graphs have been plotted for films grown at four different  $T_s$  values: (a)  $22^{\circ}$  C, (b)  $100^{\circ}$  C, (c)  $150^{\circ}$  C and (d)  $180^{\circ}$  C. The slopes of these graphs, according to Equation 1, give  $\rho_0$ , the infinitely thick film resistivity. The intercepts of these graphs on the  $y$ -axis give the values of  $l(1-p)$  which are given in Table I. Using these values of  $\rho_0$  and  $l(1-p)$  in Equation 1, theoretical curves, for different values of  $p$ , ranging from 0 to 1 have been plotted, assuming an  $l$  value for total diffuse scattering. The best fits to the experimental data are obtained with  $p = 0.3, 0.2, 0.1$  and  $0.25$  for films grown at  $22, 100, 150$  and  $180^{\circ}$  C, respectively. Using these values of  $p$  and  $\rho_0$  values of  $l$  have been calculated for different  $T_s$  and these values are given in Table I.

Fig. 3 shows a graph of temperature coefficient of resistance against thickness. It is evident that the TCR is negative for all thicknesses. It becomes more negative at higher  $T_s$ , compared with that at room temperature. The TCR at

$T_s = 180^{\circ}$  C is less negative compared to that at  $100$  and  $150^{\circ}$  C.

In conclusion, for the first time, the effect of  $T_s$  on the electrical resistivity and TCR of thin chromium films has been reported. It is observed that the electrical resistivity increases at higher  $T_s$  i.e. upto  $150^{\circ}$  C and it decreases considerably at  $T_s = 180^{\circ}$  C. It is hoped that a better understanding of this anomaly will be obtained from structural studies of these films, which will be reported in a future communication.

#### Acknowledgements

We thank Mr S. M. Shivaprasad for many helpful discussions. One of the authors (LAU) is grateful to the University Grants Commission, New Delhi for the award of a Teacher Fellowship under the Faculty Improvement Programme.

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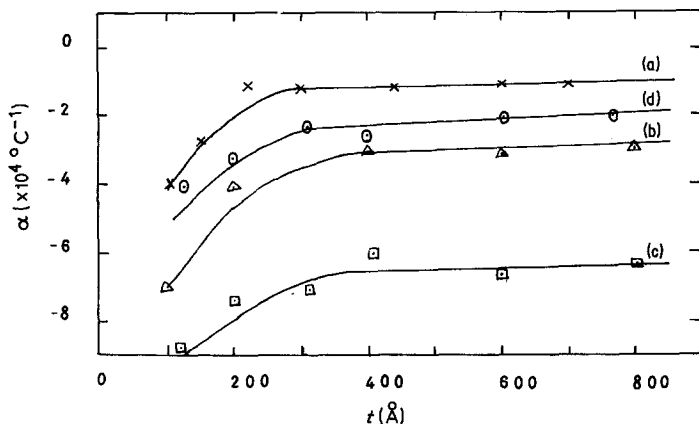


Figure 3 Temperature coefficient of resistance,  $\alpha$ , against film thickness,  $t$ , for different substrate temperatures (a)  $22^{\circ}$  C, (b)  $100^{\circ}$  C, (c)  $150^{\circ}$  C and (d)  $180^{\circ}$  C.

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*Received 13 August  
and accepted 15 September 1980*

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### *A rapid method for determining boron in metallic alloys*

Boron is an important alloying element in a number of commercial alloys. Small quantities of boron (< 0.1 wt%), when added to low alloy steels and cast irons, improve their mechanical properties, while large quantities of boron (> 1.0 wt%), when added to transition metal alloys with Ni, Fe, Co, etc., produce wear resistant hard facing alloys and promote the formation of amorphous alloys under certain processing conditions. Determination of boron concentration in these alloys by conventional analytical techniques, such as wet chemistry, atomic absorption and emission spectroscopy, is extremely difficult and time-consuming [1]. The problems are primarily associated with the slow rate of dissolution of boron-containing materials in chemical solutions which is required for these techniques. Very low solid solubility of boron in transition metals promotes the formation of stable boride phases in these alloys which are often responsible for the improved properties. These phases are highly stable and often remain as an undissolved residue in the solution, reducing the accuracy of the concentration analysis.

A method is reported herein for the non-destructive rapid determination of boron in metallic alloys. The method primarily employs

neutron activation analysis and can be used in alloys where the oxygen concentration is relatively low. A typical analysis consists of irradiating the boron-containing samples with high-energy neutron particles (14.5 MeV), a procedure which produces radioactive isotopes as reaction products. The characteristic emissions from these radioactive isotopes in the form of gamma- or beta-rays are then measured to obtain the chemical composition both qualitatively and quantitatively.

Selection of a specific energy band of radioactivity from the activated alloy, free from interference from other alloying constituents, is required for accurate compositional analysis. Table I gives the nuclear reactions of elements with 14.5 MeV neutrons which are of particular interest in the present investigation. The nominal composition of the boron containing alloys used in this study is given in Table II.

When boron is activated with 14.5 MeV neutrons, two measurable isotopes of  $^8\text{Li}$  and  $^{11}\text{Be}$  with half-lives,  $\tau$ , of 0.84 and 13.5 sec, respectively, are produced. The measurement of activity from the  $^8\text{Li}$  isotope is extremely difficult since it requires specialized *in situ* counting equipment. Moreover, although the measurement of the  $^{11}\text{Be}$  isotope activity can be easily achieved, its measurement at 2.1 MeV is not very sensitive and is subject to severe spectral interference from 1.78 MeV gamma rays arising from the  $^{28}\text{Al}$