In-situ resistivity measurements during growth of ultra-thin $Cr_{0.7}Mo_{0.3}$

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Introduction

- The growth of ultra-thin lattice matched $Cr_{0.7}Mo_{0.3}$ films on an MgO substrate, in a dc magnetron discharge, was investigated by *in-situ* resistivity measurements.
- We compare the resistivity of the films to a combination of the Fuchs-Sondheimer and the Maydas-Shatzkes model, assuming a thickness dependence of the grain size.
- Ultra-thin metallic conducting layers are used in nanoscale electronics as interconnects and diffusion barriers (Steinhoegl et al., 2005).
- The resistance of the signal pathway determines the operating frequency and/or sensitivity of the devices.
- As the thickness of the conductor approaches the mean free path of the conduction electrons λ_{bulk}, size effects start limiting the conductivity.
- Fuchs and Sondheimer (1952) showed that scattering of the conduction electrons, at the surface of the film, causes loss of conductivity. The scattering is characterized by a specularity parameter p, which represents the fraction of conduction electrons specularly scattered at the interface.
- If the thickness of the film is further reduced, the effects of interface roughness and grain boundaries lower the conductivity even more rapidly. Grain boundary effects have been studied by Mayadas and Shatzkes (1970).
- By choosing a film material, with a compatible crystal structure and lattice constant to that of the substrate, the odds of achieving layer by layer growth are increased.
- Chambers et al. (1995) suggested using an MgO substrate and a Cr_xMo_{1-x} alloy as film material, thereby achieving a better than 99% lattice match, for x in the range 0.56 to 0.80.
- *In-situ* measurements make it practical to vary growth parameters and optimize processes based on the resulting resistivity curves. Furthermore, the study of transient behavior of films after growth is possible (Barnat et al., 2002).

Experimental procedure

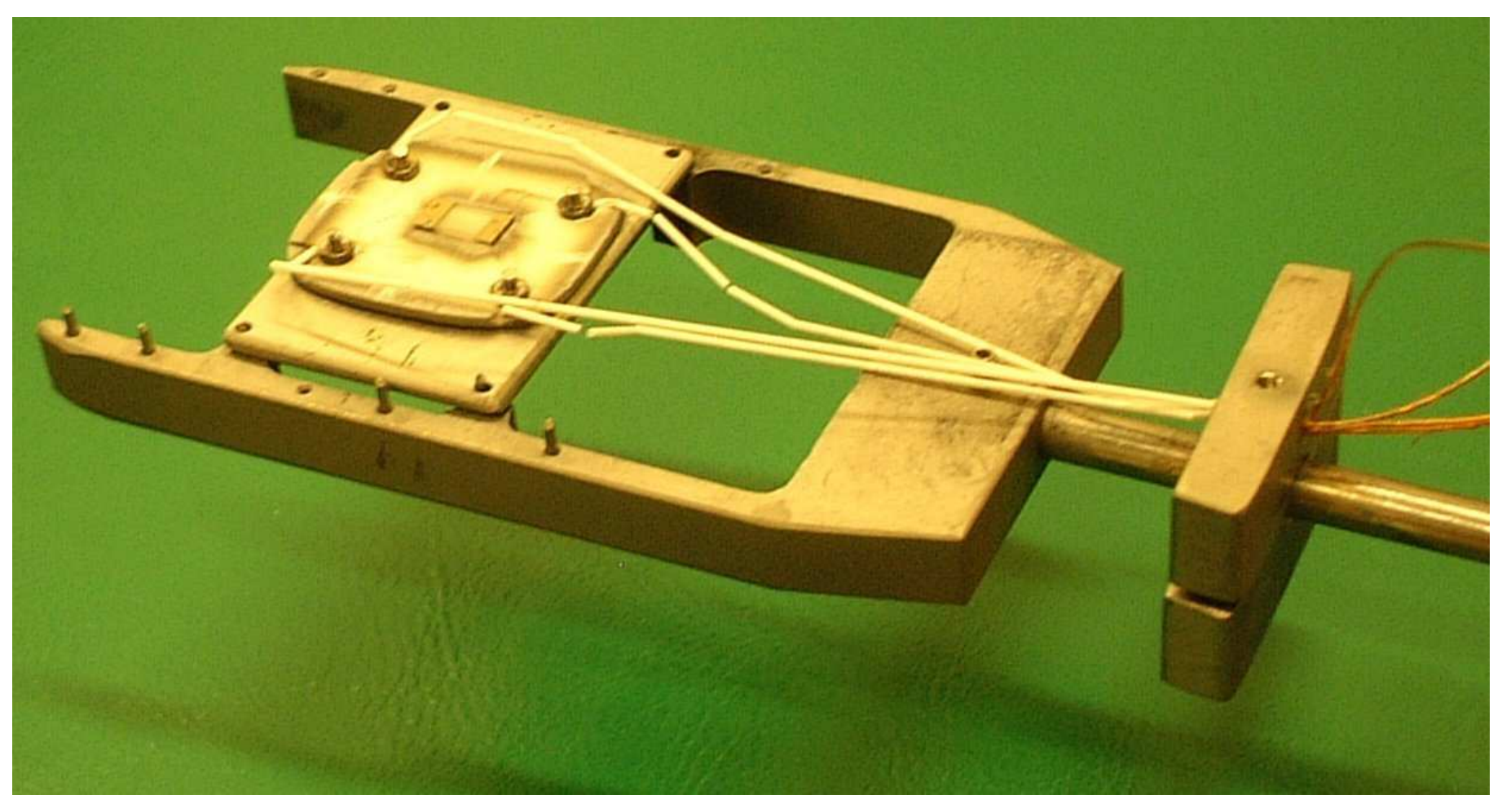

Figure 1: The four-point measurement setup is mounted on the sample loading fork. Leads are made from Macor insulated tungsten wire, that can withstand the growth temperature.

- Films of $Cr_{0.7}Mo_{0.3}$ alloy were grown on an MgO (100) substrate, at temperatures of 24°C, 200°C, and 400°C, in a direct current (dc) magnetron sputtering discharge.
- Contact pads of thickness $>$ 200 nm were grown from $Cr_{0.7}Mo_{0.3}$, leaving a square of (5×5) mm uncoated MgO in the middle of the sample.
- The substrate was electrically insulated from the chamber.

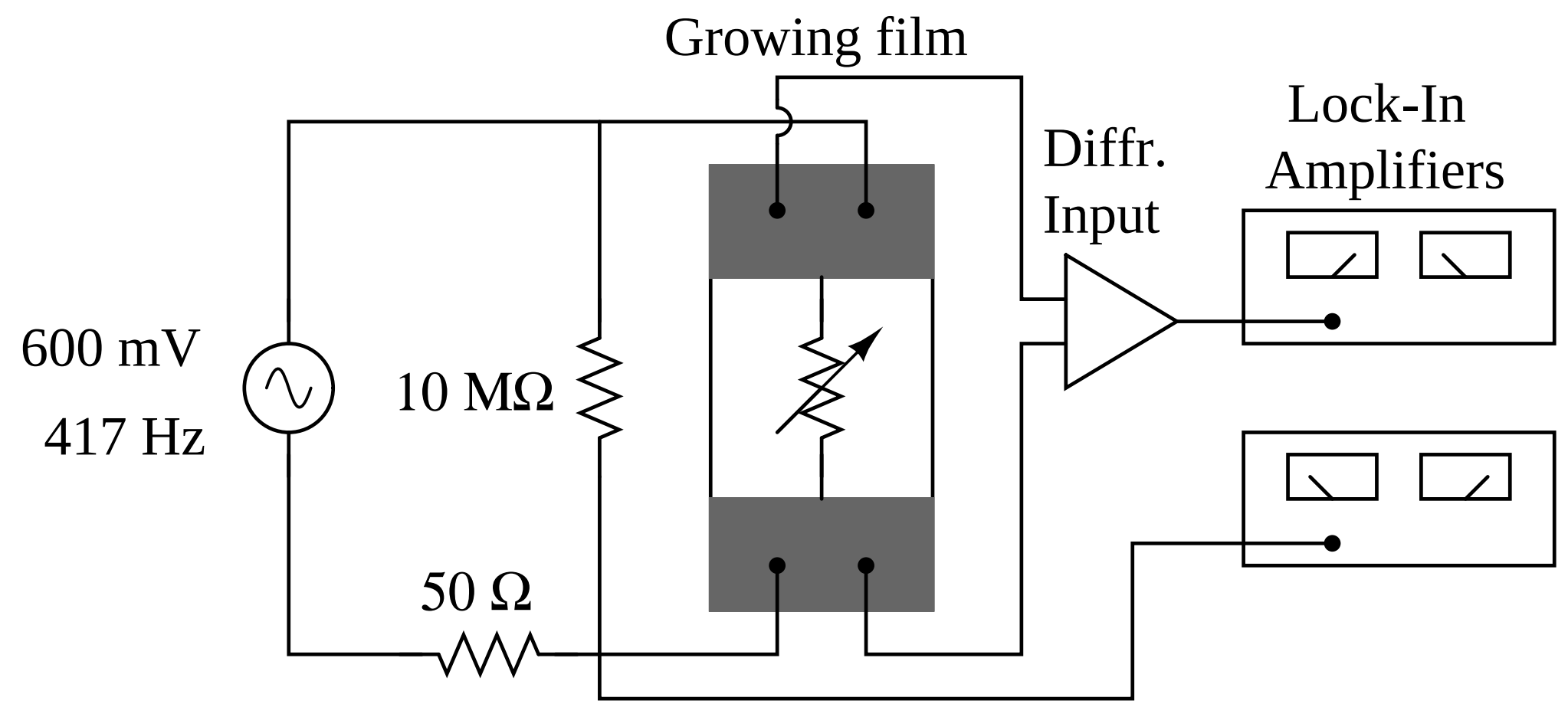

Figure 2: A schematic drawing of the *in-situ* measuring circuit.

- The resistance of the growing film was measured with a simplified version of the setup described by Barnat et al. (2003), suitable for measurements without a substrate bias.
- The lock-in technique eliminates any currents extracted from the plasma, which would otherwise erroneously lower the measured film resistance.
- The four-point method eliminates the effect of contact resistance.
- The voltage, over the film, is measured directly and the current, passing through the film, indirectly, by monitoring the voltage over a 50 Ω resistor, in series with the film.
- A function generator supplies a 600 mV RMS sinusoidal signal at 417 Hz.

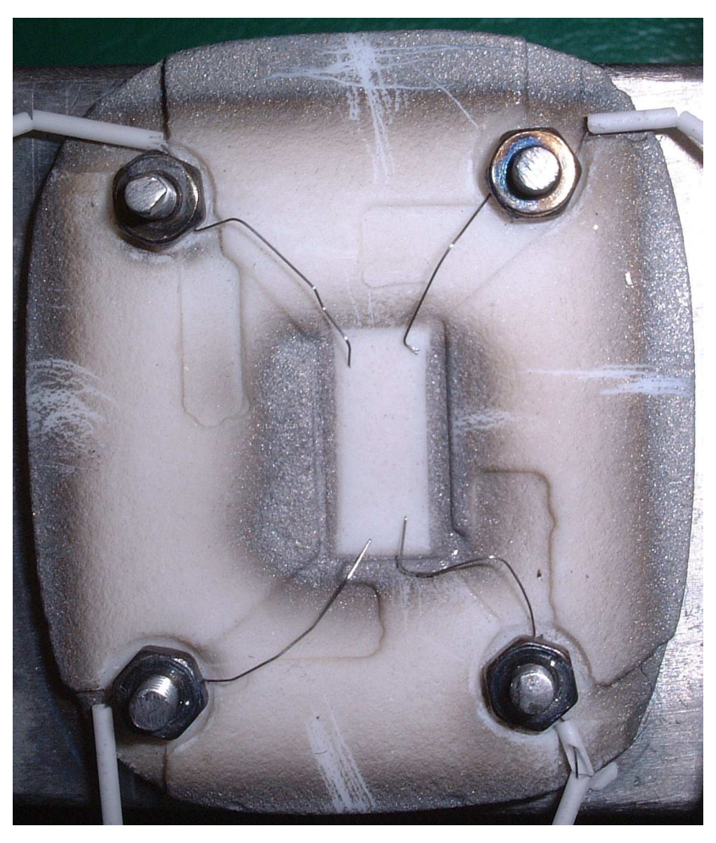

Figure 3: The substrate holder is made from Macor to insulate the four contacts.

- The signal was transmitted with a Capton insulated copper wire, except the last 10 cm, that were made from Macor insulated tungsten wire, which can withstand the 700°C outgassing temperature.
- The substrate holder is made from Macor to electrically insulate the four probe tips.
- The probes and connectors are shielded from the flux of sputtered particles with a thin mask of Macor, confining the growth to the rectangular exposed area of the substrate.
- The alloy composition was controlled by regulating the current to the magnetron targets.
- The composition was found with Vegard's law from the lattice constant of the CrMo peak at $\simeq 62.5°$ in the θ–2θ X-ray diffraction (XRD) scan of the films.
- Growth rate was found with low angle X-ray reflection measurements.

Results and discussion

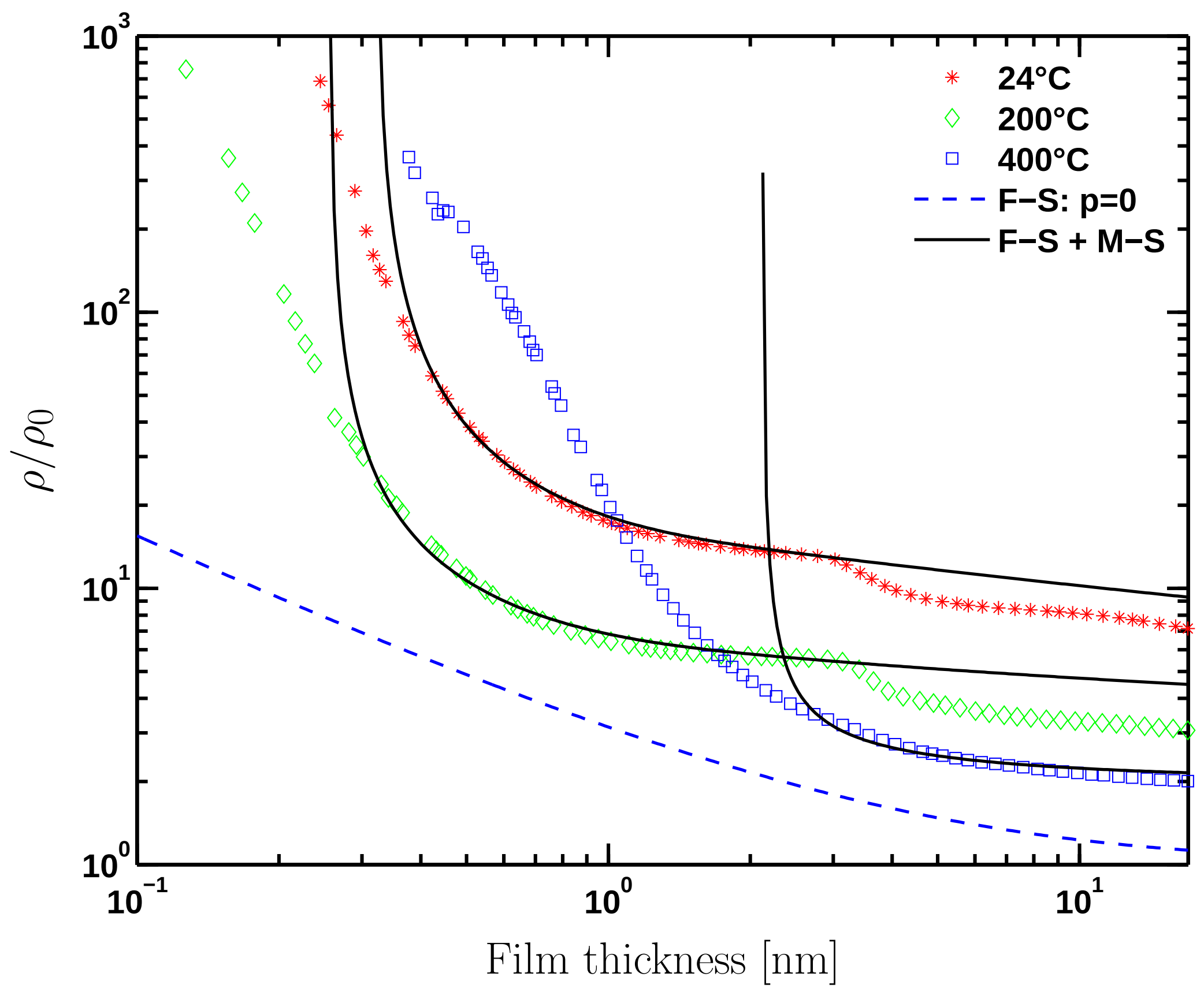

Figure 4: The electrical resistivity ρ, normalized by the bulk resistivity ρ_0, as a function of thickness of $Cr_{0.7}Mo_{0.3}$ films, grown at three different substrate temperatures. The results are compared to the Fuchs-Sondheimer model (F-S, dashed line), and to a combined F-S and Maydas-Shatzkes model (F-S + M-S, solid line), assuming a thickness dependent grain size.

- The coalescence thickness varies from less than 0.3 nm at 24°C and 200°C to 0.4 nm at 400°C, well below 2 monolayer nominal film thickness, suggesting layer by layer growth.
- Both the 24°C and the 200°C film show a kink between 3 and 4 nm.
- The Fuchs-Sondheimer model can not explain the observed conductivity, even assuming fully diffuse scattering ($p = 0$), since both the absolute resistivity and the increase in resistivity are too low.
- Mayadas and Shatzkes (1970) (M-S) showed that grains contribute a resistivity ρ_{g} according to

$$\frac{\rho_0}{\rho_{\mathrm{g}}} = 3\left[\frac{1}{3} - \frac{1}{2}\alpha + \alpha^2 - \alpha^3 \ln(1+\frac{1}{\alpha})\right], \quad \text{with} \quad \alpha = \frac{\lambda_{\mathrm{bulk}}}{G}\frac{R}{1-R}, \tag{1}$$

  where G is the average grain diameter and R represents the probability that electrons are reflected at the grain boundary.
- Following Barnat et al. (2002), we assume a grain size that follows a modified power law of the film thickness d

$$G(d) = G_1 d^n - G_2 e^{\frac{d}{\delta}} \tag{2}$$

  where G_1, G_2 and δ are dimensional scaling constants and n is an exponent dictating how the grain size scales with thickness.

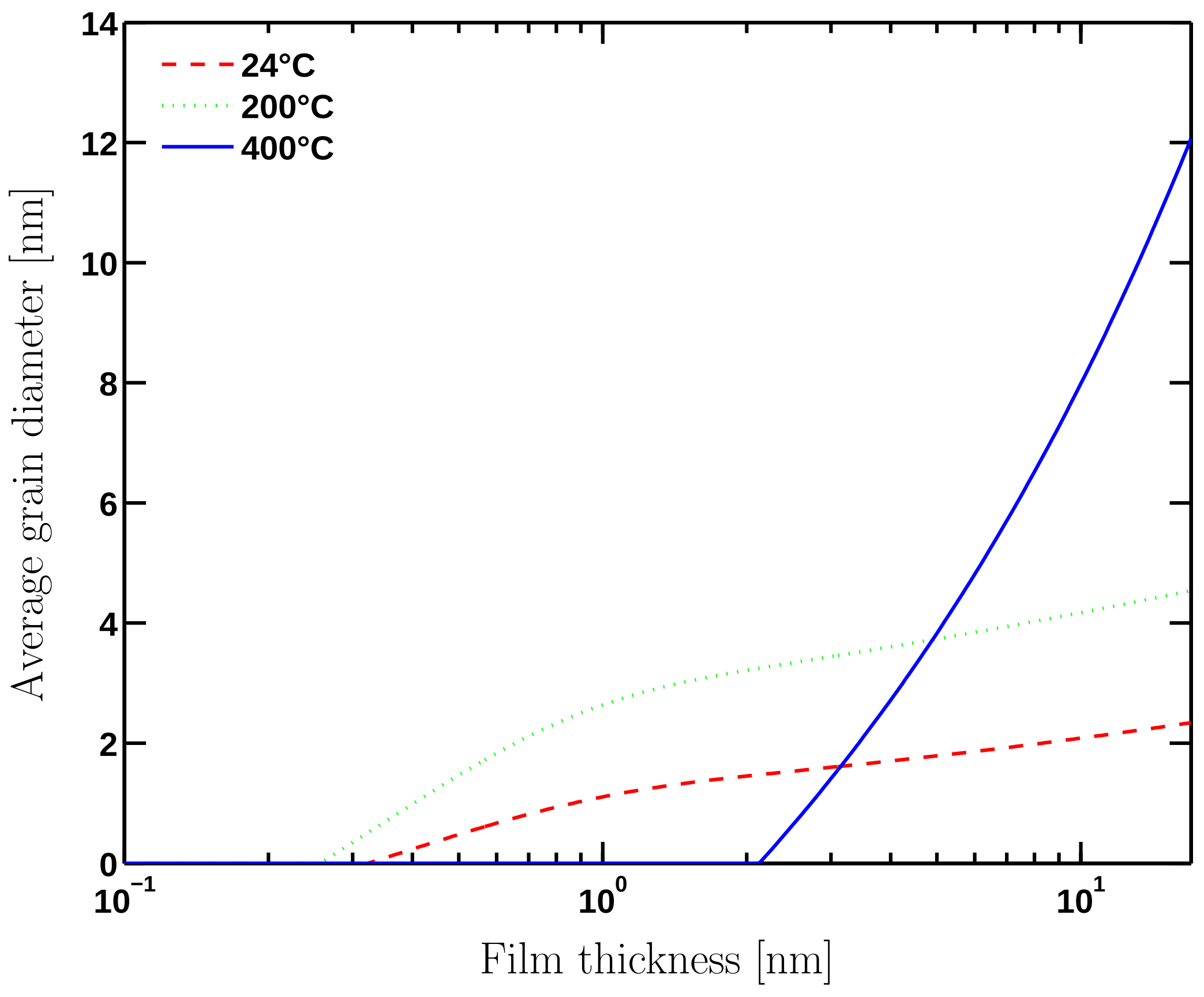

Figure 5: The grain size models used to fit the resistivity of the films in Fig. 4 to the combined F-S and M-S model.

- Above 2 nm the grain model curves order themselves according to temperature, with grain sizes of 2, 3, and 7 nm at 10 nm.
- At the lower temperatures, the metal particles landing on the surface have a lower surface mobility and thus the ordering of the growing layer is determined by the substrate.
- This results in a smooth epitaxial layer, as indicated by a low coalescence thickness, but a small grain size because of the strain.
- At a critical thickness (the kink in resistivity at 3 nm) the films relax to accommodate the increasing strain energy and growth proceeds with higher crystalline order resulting in a downwards step in resistivity.
- At higher temperatures, the sputtered particles have a higher surface mobility and are thus more free to cluster on the surface.
- This leads to a slower reduction of resistivity, but a larger grain size, and thus a lower resistivity than of the low temperature films above 2 nm.

Conclusions

- The resistivity of thin $Cr_{0.7}Mo_{0.3}$ films, deposited by magnetron sputtering on MgO, was examined *in-situ* at three different growth temperatures.
- A coalescence thickness of less than two monolayers suggests layer by layer growth of the films. The films grown at 24°C and 200°C coalesced at a lower thickness than that of the film grown at 400°C.
- The thickness dependence of the resistivity was compared to a combined Fuchs-Sondheimer and Maydas-Shatzkes model.
- By assuming a thickness dependence of the grain size, a reasonable fit was obtained.
- The model suggests that the grain size of the films increases with growth temperature.
- The growth curves give a good indication of the lower limit that film resistivity will put on interconnect dimensions in devices made with similar processing.

Acknowledgments

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