Grain growth in Pt microheaters subjected to high current density under constant power

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Abstract
When 50 nm thick Pt microheaters of lateral dimensions $1 \times 10 \, \mu\text{m}^2$ are subjected to high electric power their resistance $R$ rises, as expected. Following an initial rise, however, there is a gradual decrement in $R$ while constant electric power dissipation is maintained. We find that this lowering in $R$ is accompanied by grain growth in the polycrystalline thin Pt film of our heaters. This is confirmed by XRD measurements and SEM imaging. Similar growth in grain size is observed in thin Pt films that are oven-annealed at high temperatures. Thus, we argue that maintaining high power dissipation in a microheater has the same effect on its material structure as post-annealing. We observe the in-plane grain size of a 50 nm thick as-grown Pt film/heater to be $D_\parallel = 15 \, \text{nm}$. When post-annealed at a temperature of $T = 600\, ^\circ\text{C}$ for 30 min, $D_\parallel = 30 \, \text{nm}$, compared with when electric current is run through a heater, we estimate the mean crystalline length to be $D_\parallel = 35 \, \text{nm}$.

Keywords: microheaters, grain growth, power regulation

(Some figures may appear in colour only in the online journal)
solely on the body’s temperature). Thus it is very important to control and regulate the individual heater’s temperature. The temperature of the microheater, $T$, is linearly dependent on the power dissipation in the heater, $P$, via the relation

$$T = T_0 + \frac{dT}{dP}P,$$  

(1)

where $T_0$ is the ambient (room) temperature (i.e. the value of $T$ at $P = 0$ mW), and $dT/dP$ is the thermal impedance of the microheater [21]. Thus by regulating the power dissipation in the heater one simultaneously regulates its temperature. The electrical resistance of the heaters $R$ varies with the temperature since

$$R = R_0(1 + \alpha \Delta T),$$  

(2)

where $\alpha$ is the temperature coefficient of resistance, $\Delta T = T - T_0$ and $R_0$ is the resistance at room temperature. We observe an increase in $R$ with higher $T$, just as expected.

When our heaters are subjected to high current density, which is done to achieve higher radiation temperatures and shorter wavelengths, they tend to fail due to electromigration. The time-to-failure $\tau$, of a given interconnection, or heater in our case, is given by Black’s equation [7] and $\tau \sim 1/J^2$. We use a value $n = 2$ for the current density exponent in accordance with [22]. In our work we deal with current densities up to $J \approx 7 \times 10^7$ A cm$^{-2}$.

In our research on electromigration and the time-to-failure of Pt microheaters, we have observed a gradual decrease with time in $R$, when microheaters are excited by electric current at constant power [22]. We know from previous work that such changes in $R$ do not affect the thermal impedance $dT/dP$ as this is governed by the thermal properties of the surroundings, most importantly the substrate’s thermal conductivity [21]. This slow drift in $R$ led us to further investigate the metal structure of our heaters by other means than just by measuring their resistance profiles. We hypothesize that subjecting the heaters to high power, and thus high temperature, has the same effect on the material structure as if they were annealed in an oven. The observed resistance drop seems similar to the effect of high current annealing reported in [20], but in our case the cause is different. Here we attribute the resistance change to a reduction in electron scattering at grain boundaries.

The outline of this paper will be as follows: at first we introduce our the experimental setup and samples. Then we focus on the distinct $R$-$t$ (resistance-time) and $R$-$P$ (resistance-power) profiles of our heaters. In the subsequent section we estimate the grain size of our heaters by two methods, by scanning electron microscopy (SEM) imaging and by grazing incidence x-ray diffraction (GIXRD) measurements.

2. Sample properties

The heaters used in our research are manufactured by e-beam lithography to ensure that their shape is well defined. A Si/SiO$_2$ (100 nm oxide) substrate is coated with a negative e-beam resist. The heaters are patterned in the resist and the part exposed to the beam is developed away.

The heaters, typically about 60 on each substrate chip, are deposited via sputtering where a 5 nm thick polycrystalline Cr adhesion layer is grown before the 50 nm polycrystalline Pt layer. The growth rate of Cr is 3.2 nm min$^{-1}$ and that of the Pt is 5.2 nm min$^{-1}$. For further details on the film growth we refer to [16]. A lift-off step in an acetone bath leaves us with the final structure. The chip is rinsed and then the heaters are ready for use. The dimensions of the microheaters used in this study are $1 \times 10 \mu$m$^2$, and their thickness is as detailed above. We have measured the surface roughness of our films by atomic force microscopy, and in all cases the rms roughness is below 1 nm. A typical microheater is depicted in figure 1.

We use four terminal electrical measurements to obtain the power dissipation in our heaters, $P$, as this enables us to have accurate control over the temperature of the heaters. We use a homemade circuit to both monitor and to regulate the power dissipation in the heater. This process is computer controlled via a programmable National Instruments Data Acquisition board (DAQ-board, NI-USB 6229). The setup and the circuit is thoroughly described in [23]. We subject our microheaters to a high current density, and wait until they eventually fail due to electromigration, in order to test their time-to-failure. By switching the current polarity at a frequency of $f = 20$ kHz (referred to as AC current stress in the article) we observed on the order of 10$^3$-fold increase in the time-to-failure of our heaters, compared to when they are biased at the same power level at DC [23]. Depending on the bias, our measurements can take quite some time, the longest measurement on a single heater lasted for over a week.

Three quantities describing the properties of our heaters are of special interest. These are their electrical resistance $R$ (in particular the cold resistance $R_0$), the thermal impedance $dT/dP$ and the temperature coefficient of resistance $\alpha$. Attempts were made to determine if any permanent changes occur in these quantities upon subjecting our heaters to high power. Our findings show that both $\alpha$ and $dT/dP$ appear to be essentially unchanged despite thermal cycling, whether it be by varying the current density or by external heating (on a hot-plate). However, we found that the heater resistance changes substantially in our samples and devote section 3 to discussion on those observations.

These properties, $\alpha$ and $dT/dP$, can be obtained by current–voltage ($I$–$V$) measurements, with the help of a hot plate to vary temperature, as follows: an as-grown heater is $I$–$V$ characterized. These measurements result in a cold resistivity of

![Figure 1. Heaters used in the research. The terminals are labelled with numbers 1–4 and each is about 200 $\times$ 180 $\mu$m$^2$. The dotted line in figure (a) demarcate the area pictured in figure (b). Images were obtained using an optical microscope.](image)
The effect of temperature on the resistance of a metal slab is well known and may be described by equation (4). When subjected to high power, the resistance of our heaters rose during the first few seconds as can be expected due to self-heating, but when the desired regulation power (90 mW) was reached we noted a decrease in $R$—steep at first but then with a gradually declining slope, as if the resistance follows an inverse power law. This can be seen in figure 2, that shows an $R$-$t$ (resistance-time) graph of an as-grown heater whose power was regulated at $P = 90$ mW under AC current stress.

$$\rho_t = 20 \mu\Omega \text{ cm.}$$  \hspace{1cm} (3)

This value is about 90% greater than the bulk resistivity of Pt [24], $\rho_0 = 10.4 \mu\Omega \text{ cm}$, but it should be kept in mind that this is a thin and narrow wire with large contribution to resistivity from surface scattering. Its $R$-$P$ profile is stored likewise. In the linear part of the $R$-$P$ graph the line is described by combining equations (1) and (2)

$$R = R_0 \left( 1 + \alpha \frac{dT}{dP} P \right)$$  \hspace{1cm} (4)

where $P$ is the power dissipated in the heater and $R_0$ is the resistance at $P = 0$ mW.

The heater is now measured on a hot plate, whose temperature is increased stepwise and changes in resistance are observed. We measured over a temperature range from room temperature up to about 110 °C, and found $\alpha$ from fitting equation 2 to be

$$\alpha = 2.20 \times 10^{-3} \text{ K}^{-1}.$$  \hspace{1cm} (5)

From the $R$-$P$ graph the value of $dT/dP$ can now be estimated. For our heaters we obtained a value of

$$\frac{dT}{dP} = 4.7 \text{ K mW}^{-1}.$$  \hspace{1cm} (6)

The heater is now subjected to constant high power (which is the quantity regulated) for a few minutes, a time long enough so an obvious decrease in the heaters resistance has been observed, see section 3. Following this procedure the heater’s $I$-$V$ profile is obtained again to measure the cold resistance and its $\alpha$ value is measured again to see if it had changed. We found no changes in $\alpha$ or $dT/dP$ as mentioned earlier. This is in agreement with results from Pt films annealed to temperatures of 1300 °C [25].

All the heaters in our study are manufactured in the exact same manner, therefore we assume they have the same physical characteristics. We chose to regulate heater power at a value of $P = 90$ mW in our measurements. This power value was obtained by trial and error, as heater failure is a statistical event. It was chosen such as to see a substantial decrease in $R$ in the first few minutes upon biasing and a ‘reasonable’ time-to-failure. At this high power we estimate the temperature in light of equations (1) and (6) as being $T_h = 710$ K corresponding to about 440 °C.

3. Monitoring resistance

In this particular measurement we ramped the heater power up slowly and then ramped it back down after 5 min of constant high power regulation. In [23] examples can be found of measurements where after the drop, the resistance reaches a minimum, followed by a rise in the resistance leading to the eventual break of the heater due to electromigration.

This early drop in $R$ is also observed in as-grown heaters subjected to DC current stress. If the bias is maintained for long enough time the resistance typically starts to rise again and eventually the heater breaks due to electromigration [23]. Further, it appears to reflect an irreversible change in the sample properties, i.e. the material structure, as far as we can see.

An interesting effect can be seen in figure 3(a), which is an $R$-$P$ graph of a heater where the power is ramped up at 1 mW s$^{-1}$. It compares the resistance during the first two ramp-ups, which are quite distinct. Later ramp-ups (the 3rd, 4th etc.) were almost identical to the second one, and thus omitted in the graph. During the first ramp-up there is a steep rise in the resistance at $P \approx 70$ mW (power is maintained constant in 1 mW steps). This power value corresponds to a temperature of about 370 °C, according to equation (1). We take this as a sign of permanent change in the heater’s material structure which seems to happen when its power is driven above a certain limit for the first time. A reduction in $R$ is caused by less electron scattering, which we attribute to growth in grain size of the polycrystalline Pt film [26, 27].

Figure 3(b) depicts an $R$-$t$ graph of the same heater, again with the power ramped up at a rate of 1 mW s$^{-1}$. When the desired power is reached it is maintained for several minutes, and then lowered again (corresponding to the dips in resistance). This is repeated at regular intervals. As can be seen in the figure, the resistance trace reaches the same value after each ramp-up as it had prior to the ramp down of the power, further supporting the hypothesis of irreversible change to the sample.

Changes in $R$, imply changes in the resistivity $\rho$ of the heater. Based on Matthiessen’s rule, we can simply add the contributions to the resistivity of a thin film $\rho_t$, such that

$$\rho_t = \rho_0 + \rho_{GB} + \rho_{SS} + \rho_{SR}.\hspace{1cm} (7)$$
Here, $\rho_0$ is the bulk resistivity, $\rho_{GB}$ a term caused by grain boundary scattering, $\rho_{SS}$ comes from surface scattering and $\rho_{SR}$ results from the surface roughness of the film [28]. Our films are smooth compared to the thickness, so we can safely say that $\rho_{SR}$ is very small and will not be considered here. According to the Fuchs–Sondheimer model [29, 30] $\rho_{SS}$ is a constant quantity for a constant value of the thickness $d$ of the metal film. We argue, based on measurements presented in the following section, that the main reason for the reduction in $\rho_f$ observed in figure 2, is due to grain growth in the Pt film, which yields a decrement in $\rho_{GB}$.

4. Grain size measurements

In order to test the grain growth hypothesis we conducted two kinds of measurements to measure the grain size of the heaters and of Pt thin films grown in an identical manner to the ones our heaters are patterned from. We employ GIXRD measurements and SEM which give different information about the sample. GIXRD measurements are useful for polycrystalline thin films where the grains are randomly oriented. One also typically obtains better signal strength than in ordinary $\theta$-2$\theta$ measurements. It should be kept in mind that during GIXRD the incident x-rays are kept at a fixed low angle while the detector is scanned through a range of angles. As a result, the different peaks in the GIXRD scan represent different plane orientations (or $k$-vector directions). While SEM imaging yields an estimate of $D_\parallel$, different GIXRD peaks can be used to obtain estimates of grain sizes in directions corresponding to the $k$-vector orientation. Further, we compare the effect of running large electric current (high current density) in our samples with that of annealing identical thin films in an oven at various temperatures. As mentioned earlier the self heating at high current density raises the heater temperature quite significantly (440 °C at 90 mW power dissipation).

Our heater structures are too small to measure them directly in an x-ray apparatus. Thus four Cr/Pt films were grown in the same manner as the heaters, and three of these were post-annealed for 30 min at 200 °C, 400 °C and 600 °C temperature, respectively. The results of subsequent GIXRD
measurements on all four films are presented in figure 4(a). Using the Scherrer equation, \( D_{GI} \) (crystalline coherence length), can be calculated in the following manner: \[ D_{GI} = \frac{K\lambda}{\beta \cos(\theta)} \] (8)

Here, \( \beta \) is the FWHM of the peaks in the GIXRD spectrum (three peaks were observed corresponding to crystalline planes [1 1 1], [2 0 0] and [2 2 0]), \( \theta \) is the peak position in the spectrum, \( \lambda \) is the wavelength of the x-rays used and \( K = 0.9 \) is the Scherrer correction factor. Scherrer’s equation is used to compare values of \( D_{GI} \) and observe trends, rather than for exact estimation of the grain size perpendicular to film plane. We graph the results in figure 4(b). Grain growth is apparent with increasing post-annealing temperature.

Similar measurements on Pt were not found in the literature, but in [28], GIXRD measurements on thin Ag films are discussed. Those results are in many respects identical to ours. They were conducted at a lower temperature range from about 90 K, up to 500 K (see chapter 3 of [27]). In figure 4(b) we see that the [1 1 1] and [2 0 0] peaks yield slowly increasing values of \( D_{GI} \), but appear not to undergo substantial changes. This is in accordance with [28], where those peaks give a constant value of \( D_{GI} \) for the same temperatures as ours, from 300 K and up. In addition we also measure the peak corresponding to the [2 2 0] crystalline plane. These planes appear to grow substantially in the temperature range of our measurements, just as the [1 1 1] and [2 0 0] planes did on lower temperatures in [27].

The [2 2 0] crystalline planes appears to dominate in the structure, since the intensity of the peak increases at the cost of the other two planes measured. One also sees on figure 4(a), that the intensity of the [2 2 0] diffraction peak starts to grow between 200 °C and 400 °C. We associate this change with the steep rise in electrical resistance occurring during the first ramp-up curve of figure 3(a), at a heater temperature of about 370 °C. This is an indication of changes in the material structure and at this temperature of 370 °C the activation energies for these processes have been reached.

We also estimate the in-plane grain size \( D_\parallel \) was also estimated in a SEM, see figures 5(a)–(f). Grain growth is apparent upon annealing. \( D_\parallel \) varies from about 15 nm for the unannealed film up to 30 nm for the one annealed at 600°C. In addition figures 5(e) and (f) display the grain size of microheaters before and after electrical measurement at 90 mW constant power.

![Figure 5](image-url)
The same grain growth happens when the heater is subjected to high current density which raises its temperature according to equation 1. The material is annealed, just not in an oven but by self-heating. In addition one notices that not only the size of the grains changes, but also the shape. The grains appear to take on a rounder shape in contrast to the more oblong shape of the grains in the unannealed films. Similar growth of post-annealed thin Pt films is reported in [32].

5. Conclusions

We investigated 50 nm thick Pt microheaters of lateral dimensions 1 × 10 μm², subjected to current density up to about \( J \approx 7 \times 10^7 \) A cm⁻². We regulate the electric power dissipated in the heater at a constant value of \( P_h = 90 \) mW, obtained by trial and error, chosen to see a substantial decrease in \( R_h \). At this high power we estimate the temperature in light of equations (1) and (6) to be \( T_h = 710 \) K, about 440 °C. As expected, by increasing the power dissipation in the microheater the resistance increases as described by equation (4). When the desired regulation power was reached, the heater’s resistance drops, steeply at first but then the process slows down. The decrement in the heater resistance is permanent, which indicates a modification of the heaters’ structural properties. To investigate the structure of these heaters, we used two different techniques: SEM and GIXRD. From those measurements we see that the grains in thin Pt films start to grow when heated to temperatures between 200 °C and 400 °C. Indeed we also see signs of these structural changes in the heater when current is run through it the first time, then at heater temperature of about 370 °C. We conclude that the observed decrease in the heaters resistance, is due to grain growth in the metal as it is subjected to high electrical power.

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