CORRELATION BETWEEN CRYSTAL ORIENTATION AND NANO-GAP FORMED BY ELECTRO MIGRATION

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Abstract: Effect of electro migration on crystal structures of platinum nanowire (Nano bridge) during Nano-gap formation is investigated by means of Transmission Electron Microscopy (TEM). Selected area diffraction patterns as well as bright field images are used for this investigation. There were severely recensions in the polycrystalline Nano bridge and crystal structures around the nanogap changed completely during electro migration. Due to Joule heating, original small crystal with random orientation disappeared and newly crystals with a preferred orientation grew. They have [111] orientations (respect to beam direction) with slight misorientations. α and θ was defined to calculate the misorientation and used to represent Nano-gap formation mechanism. The calculation gives the breaking of Nano bridge occurred along grain boundaries in most of Nano bridges. The controlling system during electromigration may affect on the shapes of tips so that the shape of tips in Nano bridges, in which feedback control is applied, is more symmetric than others. The effect of temperature on atomic diffusivity might be the reason of the behaviour. [422] could be a preferred surface plane for mass transport in platinum Nano bridge in which atoms move along it.

Keywords: Electro migration, Nano-gap, platinum, crystal orientation, Transmission Electron Microscopy, misorientation

1. INTRODUCTION

An electrode is an electrical conductor used to make contact with a part of a circuit. Nanoelectrodes are tiny electrodes made of metals or semiconducting materials, the length scale of interest is in the range of 1–100 nm. Nanoelectrode could be made of CNTs [1,2], using different techniques including electron-beam-induced deposition (EBID) [3], electro deposition [4] di-electrophoresis technique [5] and electro migration [6]. Electro migration is one of the simple and inexpensive ways for nanoelectrode fabrication. It is considered to be gradual displacement of metal atoms of a conductor as a result of applied electric field. This effect is generally the main reliability issue in electronic industries which can trigger system failure. Recently, electro migration has been used as a tool for nanoelectrodes fabrication [7,8]; In fact, this failure mode could be exploited advantageously to break nanowires in a controllable and self-limiting fashion. Since the resolution limit for electron-beam lithography is roughly a few nanometres, it is difficult to fabricate Nano gaps below 3–4 nm precisely with a significant yield [9]. Therefore the best pairs of electrodes with nanometer nanogap are achieved through electro migrating. Recently many Nano gaps have been made by electro migrating platinum bridges [10]. The main challenge in this way is to control the fabrication process of nanoelectrodes. One of the motivations for development of Nano electrodes has been to fabricate electrodes with well defined shape. This subject is still in its infancy stage and needs more investigations on different parameters to determine shape of electrodes. By now few investigations have been done on the tip shape in fabrication of nanoelectrodes using electro migration, while it is a very important point in this field. Various investigations show critical role of structural factor in electro migration [11], so it is important to study parameters affecting electrodes’ tips shape. Fabrication of electrodes with defined shapes is necessary if they are supposed to be used in characterization of specimens with specific shapes and size.

In current study, a few parameters which may affect tip shape are investigated. Crystal orientations of grains on apex of nanoelectrode as well as structural changes of Nano bridge after
electro migration are studied. Then preferred orientation in grains at electromigrated nanoelectrodes is determined and bridge breaking mechanism is discussed. The effect of electro migration controlling system on tip shape is investigated. Finally, a set of preferred surface planes for mass transport in platinum is introduced.

2. EXPERIMENTAL PROCEDURE

2. 1. Specimen and Instruments

Transmission electron microscope (TEM) CM30UT-FEG and Monochromated Tecnai, at Delft University of Technology were used in this project.

The sample is 12 platinum bridges (Nanowires) located on silicon nitride membrane. The bridges (200 nm width 300 nm length 15 nm thick) had been fabricated by lithography. The chip composed of aluminium oxide as substrate and a thin film of silicon nitride (Si$_3$N$_4$) is used beneath platinum bridges. Fig. 1 shows schematic photo of the chip with Pt bridges (side view) and Fig. 2 is a real image of chip centre and an electromigrated bridge. By applying voltage on the chip, the bridges were opened due to electro migration and 24 pairs of nanoelectrodes formed on chip. Two different methods had been used for controlling electro migration process: feedback control and bias ramping. Table 1 gives the controlling systems applied on the bridges during electro migration. For bridges numbered 2 to 7 and 23 to 28 feedback control, for numbers 9 to 14 bias ramping and for bridge numbered 16 to 21 firstly bias ramping and then feedback control were applied. The aim of this work is to investigate effect of these methods on the shape of electrodes. Bias ramping is a passive control

<table>
<thead>
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<th>Bridge numbers</th>
<th>Controlling system</th>
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<tr>
<td>2 - 7</td>
<td>feedback control</td>
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<tr>
<td>9 - 14</td>
<td>bias ramping</td>
</tr>
<tr>
<td>16 - 21</td>
<td>bias ramping + feedback control</td>
</tr>
<tr>
<td>23 - 28</td>
<td>feedback control</td>
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Fig. 1. Scheme of the chip with Pt bridges (Side view)
process in which applied voltage is not regulated to real time conductance over the bridge. In feedback control, a voltage ramp (100 mV/s) is applied to the bridge while the current is measured for every point. In fact the conductance over the bridge is monitored so the bias across the bridge is adjusted.

2. 2. Methods

Before discussing about the method, it is necessary to define some expressions which will be used frequently in this paper. Bridge direction: Fig. 3A shows schematic image of a bridge before electro migration with the arrow giving bridge direction Electrode-electrode direction: Fig. 3B represents schematic image of a bridge (or Nano electrode) after electro migration. There are two sharp points indicating the nanoelectrodes. The arrow connecting them to each other is electrode-electrode direction: this arrow passes through the middle of each tip. Fig. 3C shows the image of both bridge and electrodes in the same place. Regions of interest are labelled by numbers. The crystal orientation at the apex of electrodes (regions 1 and 2 in Fig. 3C) will be distinguished. Then electrode–electrode direction (the arrow labelled 4 in Fig. 3C) will be studied. The sharp edges of the tips (edges labelled 5 and 6 in Fig. 3C) will be correlated to the crystallographic orientation by calibration of the images.

Fig. 2. A real image of chip centre where the bridges are located (left), bridge after electro migration (right)

Fig. 3 A) schematic image of bridge, the arrow represents bridge direction; B) schematic image of an electrode, the arrow represents electrode-electrode direction; C) schematic image of both bridge (before electro migration) and electrodes at the same place. Regions of interested, used for this investigation, are labelled by numbers. At the right, two axis, x and y, are shown. z axis is normal to the xy plane.
2.3. Calibration

Bridge direction is one of the image features used as a reference for calibration of diffraction patterns. To determine bridge direction in diffraction patterns, the following technique has been used. First of all, in TEM diffraction mode, electron beam was defocused on bridge to observe the apex on beam centre (Fig. 4A). Then, beam was focused on the specimen to reach diffraction pattern of tip, in which shape of the tip is observable in the beam center and reflections as well. Fig. 4B shows diffraction pattern from single crystalline tip in which tip image is detectable. The circle shows the tip in 220 reflection. The tip’s image is in the center of diffraction pattern, but it is so pale that is not observable in Fig. 4B. The tip shows the bridge direction on diffraction pattern (the arrow in Fig. 4B). In old TEM (not FEG), calibration between direct image and diffraction pattern is straightforward and diffraction pattern and direct image can be correlated for a given camera length. In the case of FEG, camera length allows only for a rough correlation, because the defocus of the beam on the sample can be compensated by the diffraction lens. So the image is rotated once the beam is focused. To find correct bridge direction on diffraction pattern, it is necessary to calculate the value of rotation ($\Phi$). So, after taking Fig. 4B, the beam was focused more and a diffraction pattern without tip image was taken (a full diffraction pattern) which is shown in Fig 5. Due to image rotation, reflections in Fig. 5 are not the same as those in Fig 4. In Fig 5, the dotted line shows (220) direction before focusing and the bold one is (220) direction after focusing. The angle between these lines, $\Phi$, gives amount of rotation during focusing (rotating angle). The angle was added to the bridge direction came from the image taken before focusing to find the bridge direction in full diffraction pattern:

$$\textit{Bridge direction in Fig. 4B + rotating angle (}\Phi\textit{) = bridge direction in full diffraction pattern}$$

Among all surface planes, probably a set of planes may have lowest energy for mass

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**Fig. 4** A) observation of electrode tip at beam centre in diffraction mode by defocusing, B) observation of diffraction pattern from single crystalline tip in which tip image is detectable. The circle shows the tip in 220 reflection. The arrow shows tip direction
transport. In fact, this surface plane has the least electro migration resistance. In order to find the surface, two images were compared (by calibration): bright field image (Fig. 6A) and diffraction pattern (Fig. 6B). Diffraction pattern taken from a grain at the electrode tip was overlaid on corresponding bright field image. Since the bridge direction on bright field image and diffraction pattern is known, the diffraction pattern on bright field image, was rotated to overlap the two directions (Fig. 6C). By such a calibration, it is possible to study the edges of the tip using diffraction pattern.

To analyze crystal orientations (with respect to beam direction) and study correlation between crystal orientations in the tip of nanoelectrode and its shape, three angles have to be determined: $\alpha$, $\beta$, $\theta$.

$\alpha$ is the angle between bridge direction and 220 reflection in the crystal diffraction pattern of the electrode tip. If we take $x, y$ and $z$ as three axis in space (in which each axis is normal to other axes and $z$ is parallel to the beam direction, Fig 3C), $\alpha$ gives the position of the crystal in $xy$ plane with respect to bridge direction. There are two values for $\alpha$ for each Nano bridge (a pair of nanoelectrodes): $\alpha_1$, $\alpha_2$.

$\beta$ is the angle between "bridge direction" and "electrode-electrode direction" for each bridge. There is just one value for $\beta$ in a Nano bridge.

$\theta$ gives the misorientation from an exact zone axis with respect to beam direction in a crystal.
To calculate $\theta$, the location of the Laue circle is used:

$$\tan \theta = \frac{r}{l} \quad (1)$$

Where $r$ and $l$ are the radius of the ring of bright spots measured on the negative of the diffraction pattern and the camera length, respectively.

Laue circle is the intersection of the zero order Laue plane with the Ewald sphere. Tilting the crystal slightly away from an exact zone axis with respect to the incident electron beam will make the diffraction spots different in intensity from those in the exact zone. The ring of brighter spots formed in this pattern, is Laue circle. The angle of the misorientation, $\theta$ can be calculated from Eq.1

Like $\alpha$, there are two values for $\theta$ in a Nano bridges: $\theta_1$, $\theta_2$.

3. RESULT AND DISCUSSION

3.1. Crystal Orientations in Nanoelectrodes

First, crystal dimensions and orientations in the bridges before electro migration are studied. Fig. 7 gives the bright field TEM image of a part of electromigrated chip. In Fig. 7, A and B are two different sections of base (The "base" is all parts of sample except the bridge) and C is the electromigrated bridge (a pair of nanoelectrodes). B is an area on the "base" which is more than 500 nm far from the nanogap. Grain sizes in region B are bigger than those in region A. It implies that the region of the base close to the bridge was influenced by electro migration and grains growth has occurred, while region A is apparently intact and grain sizes and crystal orientation are the same as those of the bridge before electro migration. In fact, electrical field has changed the

Fig. 7. The bright field TEM image of a Nano bridge after electromigration. A, B and C show three regions with different grain sizes.
grain sizes and orientations in the bridges as well as region B, whereas region A has remained intact (or the change is low and might not be detectable). The main reason for this phenomenon is heating caused by current density. In fact, increasing of local temperature as a result of high current density, leads to recrystallization and therefore original small grains are replaced by new big ones. The dimensions of the bridge are not comparable to those of base (bridge dimensions: 200 nm width 300 length 15 thick, base dimensions: 2 μm width 3 μm length 15 nm thick) and so current density in the bridge is much higher than that in the base. As a result, the current density in the base is not be strong enough to alter the base dramatically; however there are an observable changes in region B in the base. Heat transferring from the bridge to the base is the cause of these changing. Since region A is far from the bridge, it could not be heated by the current. Therefore, the region A can be employed for studying grain sizes and crystal orientations of the bridge before electro migration.

As long as the current density is above a threshold level, electro migration takes place [12]. The current density heats up the metal locally and the heat increases atomic diffusivities, resulting in mass transport [13,14]. If the current density is below the level, the atomic diffusivity is not strong enough and compressive stress hinders atomic transportation [15]. The origin of this stress is chemical bonds among atoms. In the bridge, current density is above the threshold level and electro migration takes place. So there are severe changes in crystal orientation.

Fig. 8 represents selected area electron diffraction patterns (DP) from region A, B and C of Fig. 7. The diffraction pattern of region A contains many rings indicating that the region has a polycrystalline structure. The position of the rings shows random orientation of the grains. So, the bridge could have such grain sizes and orientations before electro migration (small grains with random orientations). In the case of region B, such rings are recognized, demonstrating that the region B has polycrystalline structure too. But the position of

Fig. 8. Selected area electron diffraction patterns (DP) taken from region A, B, C in Fig. 7 shows 220 direction on Fig.4B and the bold line is 220 direction in this image (full diffraction pattern). $\gamma$ is angle between these lines.
the rings shows preferred orientation in this area. The rings belonged to (220), (311), (420), (422), (440) and (624) planes are observable in the figure. Since these reflections belong to crystals with [111] orientations with respect to beam direction, the crystal orientations in region B could be [111]. Thus recrystallization took place in region B and there are perceivable changes on the base around the bridge (region B). It is the neighborhood effect of the bridge on the base in which the heat of bridge has been transferred to the base and recrystallization took place. Consequently, before electro migration the bridge has small grains with random orientation. Once an electrical field is applied and the voltage passes a threshold (minimum current density for heating and increasing diffusivity for starting electro migration), some grains grow in the bridge while others are disappeared. In fact, grain coarsening occurs during electro migration in the bridge as well as the base near the bridge (region B in Fig.7). Such grain growth have been reported by other researchers [16,17]

Now, question is which grains grow during electro migration and what are their crystal orientations? To answer the question, three pairs of electrodes were taken and diffraction pattern of the grains in the electrodes were taken by TEM. Fig.9 represents the three pairs of electrodes. Many voids are observed in these images. Voids (vacancy crystals) are formed during electro migration and this phenomenon has been reported previously [18,19]. Selected area diffraction pattern from labeled grains were taken. They revealed that all grains have [111] orientations with respect to beam direction; However it is not exactly [111] and actually there is a misorientation observed in all grains (further, it will be discussed). In Fig. 8, region C represents diffraction pattern from the grain labeled "1" in Fig. 9b and shows that crystal orientation in the bridge is close to [111]. The fade Kikuchi lines are observed in the diffraction pattern showing the zone axis. Joule heating raises temperature of the bridge and as a result recrystallization happens during electro migration. Origin of the joule heating is high current density in the bridge due to its low area compared to the base. Since crystals with [111]
orientation have relatively low surface energy [20], new crystals form with [111] orientations. In fact, to minimize surface area, interface energies of the new crystals grow with [111] orientations. This finding fits with previous studies indicating that there is close correlation between stronger <111> texture and more EM resistance. wires containing more <111> textures exhibit better resistance to electro migration, therefore <111> texture is the strongest texture against electro migration [21,22]. In other words, the time to failure of <111> texture is longer than that of other textures so that they are desirable for longer electro migration lifetime. The arrows in Fig. 9A and 9B reveal the original place of bridges before electro migration. There could be seen recessions in the bridge and base. In the case of the base, joule heating (comes from the bridge to the base) could be the main cause of grain coarsening as well as the recession. As mentioned before, the joule heating is transferred from bridge to the base near the bridge. So, the heating triggers grain coarsening, leading to these recessions in the edges of the base.

### 3. 2. Misorientation and Tip Shape

Table 2 gives $\alpha$, $\theta$, $\beta$ for all bridges. $\alpha_1$ and $\theta_1$ are misorientation angles for the first electrode and $\alpha_2$ and $\theta_2$ are these angles for the second one in a pair of nanoelectrodes. Minus sign for $\alpha$ means the angle between [220] direction in crystal at tip of electrode and bridge direction is counter-clockwise (where zero is bridge

<table>
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<tr>
<th>Bridge</th>
<th>$\alpha_1$</th>
<th>$\theta_1$</th>
<th>$\alpha_2$</th>
<th>$\theta_2$</th>
<th>$\beta$</th>
<th>$\alpha_1$</th>
<th>$\theta_1$</th>
<th>$\alpha_2$</th>
<th>$\theta_2$</th>
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<td>1.89</td>
<td>-28.5</td>
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<td>8</td>
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<td>5</td>
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<td>37</td>
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<td>2.25</td>
<td>-17.5</td>
<td>3.07</td>
<td>3</td>
<td>Bridge 19</td>
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<td>13.5</td>
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direction), while plus sign denotes the angle is clockwise. Minus sign for $\beta$ show that the angle between electrode – electrode direction and bridge direction is counter-clockwise where zero is bridge direction.

As discussed early, the crystal orientation for the crystals in bridges is about [111] with respect to beam direction. The misorientations ($\theta$) from [111] (with respect to beam direction) are between 1.5 and 5 degrees (Table 2). The average misorientations are 2.7 and 3.1 for $\theta_1$ and $\theta_2$, respectively. The orientation of crystals in the bridge is close to [111].

$\beta$ is different from bridge to bridge where in eleven bridges the angle is counter-clockwise and in twelve, it is clockwise. There is a bridge with $\beta=0$. The average angles for $\beta$ are 5 and -8.8 degrees, $\beta$ is minus for about half of the bridges in which the controlling system is feedback control. For those with bias ramping, roughly in half of bridges $\beta$ is minus. So the controlling system does not effect on the electrode – electrode direction in the bridges.

$\alpha_1$ and $\alpha_2$ are the same only for bridge number 6 and for the rest of the bridges, they have different values. When $\alpha_1$ and $\alpha_2$ value are not the same in a pair of electrodes, it demonstrates that breaking of Nano bridge taken place along a grain boundary, because there are two different grains on the tips are. If $\alpha_1$ and $\alpha_2$ have the same value, there are two possibilities:

1- $\theta_1$ and $\theta_2$ have the same value; it implies that the grains on both tips could be identical and breaking has happen in a single grain in the bridge. In other words, at the late stage of electro migration, atomic movements was not responsible for nanogap formation in the bridge and a single grain was physically broken into two pieces (one part is the first electrode and the other part is second) therefore misorientations are equal. Keep in mind that if $\theta_1$ and $\theta_2$ was the same, the direction of misorientation must be determined, because they might have the same value but different directions. In this case the origins of grains are not the same.

2- $\theta_1$ and $\theta_2$ do not have the same value, it denotes that the grains in both tip are two completely different grains.

In the case of bridge number 6, $\theta_1$ and $\theta_2$ are 3.5 and 4.4, respectively. While $\alpha_1$ and $\alpha_2$ have the same values, the misorientations are different. So the grains may not be same and breaking of bridge probably took place along a grain boundary. Since the difference between $\theta_1$ and $\theta_2$ is small (0.9 degree), there is another possibility. The grain may be same and the origin of such small misorientation could be the grain tilting (occurred during breaking). Consequently, the breaking of Nano bridge has occurred along grain boundaries in most of bridges.

3. 3. Effect of Controlling System on Tip Shapes

Tip shape in the bridges in which feed back control was applied was more symmetric than others. The reason might be related to the temperature. When bias ramping is applied, the local temperature in a bridge is higher than the case in which feedback control is applied. That is because of passive control and also constant voltage ascending in bias ramping. As a result, current density and temperature increase over the time. On the other hand in feedback control there is an active control in which voltage does not rise continuously. Thus in feedback control current density and temperature are lower than that of bias ramping. Activation energy and atomic diffusivity could be main parameters to determine electro migration mechanism [23, 24]. This means that these parameters determine which trajectory is the most favorite pathway for electro migration. Activation energy for surface electro migration in some metals such as Al [23] and bamboo-like structure of Cu[25,26] is lower than that of other pathways. There is not similare information for platinum. Hu et al [25] reported that in polycrystalline films a mixture of surface and grain boundary would be preferred paths.

The following equations show the correlation between diffusivity and temperature:

$$D_{gb} = 0.3 \exp(-17.8 \frac{T_n}{RT})$$ (2)
\[ D_s = 0.014 \exp\left(-13 \frac{T_m}{RT}\right) \] (3)

Where \(D_{gb}\) and \(D_s\) are grain boundary diffusivity and surface diffusivity respectively and \(T_m\) is the melting point. When temperature increases, the value of diffusivity goes up, but the grain boundary diffusivity rises faster than surface diffusivity. Since electro migration activation energy for platinum is unknown, it is not easy to predict its mechanism. Nanoelectrodes made by feedback control have more symmetric apex than others. If electron migration in the bridge occurs through surface diffusion, the speed and amount of atomic motions would be similar in both edges of the bridge and more symmetric tip shape is expected. On the other hand in feedback control system, bridge temperature is lower than bias ramping. Therefore, in low temperature and feedback control system surface diffusion might be dominant pathway and its activation energy determines the mechanism. With temperature increase, the value of grain boundary diffusivity increases more rapidly than that of surface diffusivity. Since the tip shapes are asymmetric in these cases, grain boundary diffusion might be predominant way for electron migration. So values of diffusivities, and not activation energy determine the mechanism.

3. 4. Low Energy Surface Plane

If surface diffusion was the dominating pathway for some bridges, there might be surface plane with lowest energy along which atomic movements mainly happen. The surface plane is easiest direction for mass transport during electro migration which may affect the tip shape. Such planes have been seen previously in metals. The lowest energy face in copper lines at room temperature has been reported [27]. Assuming that there is such a low energy surface planes in the platinum Nano bridges, it is expected to observe the planes on the tips (after electro migration). In fact atoms must move along the surface plane during electro migration and so, it is expected a recession along the surface planes in Nano bridges. After nanogap formation, these surface planes could be detected in the tip of the electrode from its edges. The bright field TEM image and diffraction pattern can be exploited to find the surface planes. The results demonstrate the presence of at least one \(\{422\}\) surface plane in each electrode. It infers that \(\{422\}\) are the surface planes with lowest energy for electron migration in platinum. Low energy of \(\{422\}\) surface planes might be related to resistivity of platinum. Previously it has been discovered that one direction has the least resistivity among others during electro migration in face-centered-cubic (fcc) crystals [28]. Electrons in the different surfaces of platinum have many routes to pass; each surface can be a way for electrons, but the electrons choose the surface having low resistivity. \(\{422\}\) surface planes might have lowest resistivity among all planes.

4. CONCLUSION

Platinum Nano bridges have a polycrystalline structure before electro migration. The crystals have small size with random orientations (with respect to beam direction). During electro migration, there were severe recessions in the polycrystalline Nano bridge and crystal structures around nanogap changed completely. Original grains disappear and new grains grow; voids were formed in those areas where grain growth occurred.

In fact, the original platinum grains are replaced by a new set of grains that grow until the original grains are entirely consumed. The newly grown crystals have \(\{111\}\) orientations with slight misorientations. When the width of a bridge reaches a few nanometers during electro migration, there are two possible ways for breaking of the bridge: a single grain breaks physically into parts or breaking occurs along grains boundaries. This study showed that the breaking of bridge predominantly takes place along grains boundaries.

Since the high current density is the main reason for grains growth, it is expected to be no considerable change in the base of the sample because of very low current density in this area compared to the bridge. However, there was an
observable change in the crystal size of the base near the bridge which must be due to heat transfer from the bridge to this region. Therefore, recrystallization takes place in the base near the bridge and new crystals have [111] orientations. On the other hand, the base far from the bridge remains intact during electro migration because bridge can not heat up the region (it is a heat sink). So, its crystal sizes and orientations are the same as that of fabricated platinum before electro migration.

Local temperature effects on atomic diffusivity and determines electro migration mechanism. Surface or grain boundary diffusion could be main pathways for electro migration. In low temperature surface diffusion and in high temperature, grain boundary diffusion could be the main pathway for electro migration. There is a preferred surface plane for mass transport in platinum along which atoms move along it. \{422\} were observed as preferred surface planes in the edge of the tips.

The shapes of the tips might be influenced by electro migration controlling system. If feedback control is applied, the shapes of the tips in bridges would be more symmetric than bridges made by bias ramping. The effect of temperature on surface and grain boundary diffusivity is the reason of this behavior. At high local temperature, grain boundary diffusivity is predominant way for electro migration and shape of the electrodes will be dependent on grain boundary, whereas in low local temperature surface diffusion occurs in the bridge making electrodes with more symmetric shape.

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