UNIVERSITY OF COPENHAGEN

Bachelor Thesis

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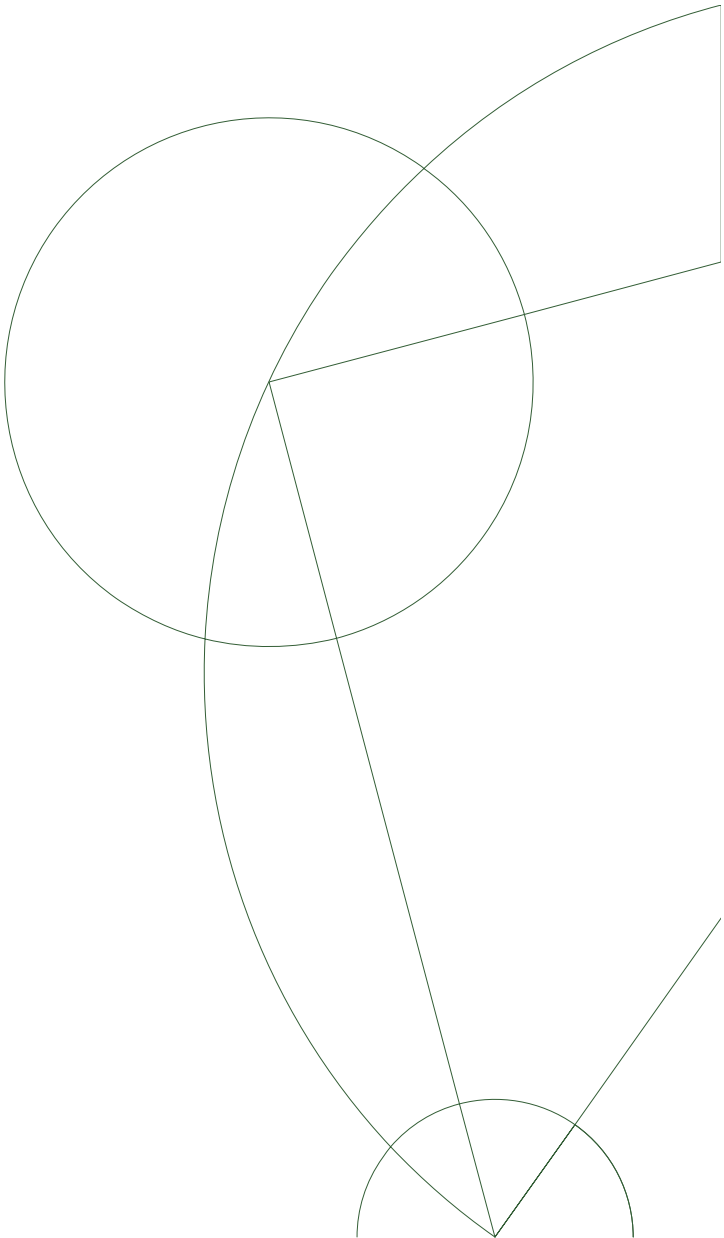
Ag Catalysed InAs Nanowire Growth
and Scanning Electron Microscopy



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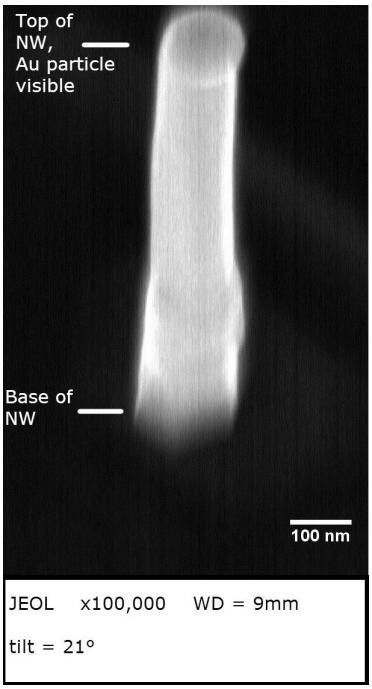
Content

Abstract

This project studies the possibilities of Ag as catalyst for growth of InAs Nanowires. Two experiments are done. One investigating the development of a 10 nm Ag film during annealing at varying time scales and the other exploring epitaxial growth of InAs Nanowires on (001) and (111)B substrates with a 2 nm Ag film where the growth temperature is varied. Characterization is done in a scanning electron microscope. The Ag film was deposited by electron beam evaporation and the growth was conducted inside a molecular beam epitaxy system. It is discovered that Ag particles appear already at room temperature and that the mean particle area increases with annealing time. Nanowire growth was present at 420°C growth temperature, but it was not possible to prove that the Ag had catalysed it. Surface structure at this temperature was made of triangular islands for the (111)B substrate and square holes for the (001). The former is similar to results from a study done with Au on GaAs (111)B where Nanowire growth was achieved indicating that this is the preferable growth temperature for future experimentation.

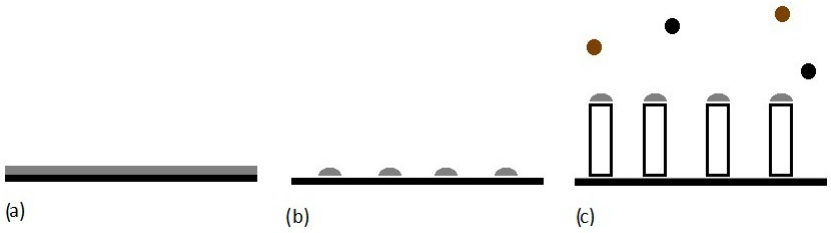
1 Introduction

This project is focused on the growth of Semiconductor III-V Nanowires (NW's), a field in condensed matter physics that shows enormous potential for future developments in electronics, optics and biology [1] [2]. My primary role in the project was to use Scanning Electron Microscopy to take images of the samples and analyse the results. I was also set to help with a step in the fabrication process of the NW's, which was evaporating a thin film of Ag on the substrates. A simple way to define a NW is as a 1-dimensional semiconductor rod often grown from III-V materials like GaAs or InAs. The diameter of the rod will range up to 100 nm while the length of the rod is much larger than this, often several μm, figure 1.



Figur 1: SEM image of a Au catalysed InAs NW. The Au particle is clearly visible at the top of the NW.

Such a system has the benefits of a semiconductor, such as possibilities for advanced band-structure engineering and high carrier mobility while also having pronounced quantum confinement effects [1]. The growth process of NW's is a complicated procedure governed by thermodynamics and kinetics [3]. A way to control the growth has been discovered through the use of a catalyst particle that acts as a seed for the growth of the wires. The size and shape of the catalyst particle will determine the characteristics of the NW that will grow from it. There exists different methods for deploying these catalyst particles for example lithography, aerosol deposition or thin film evaporation [4]. For this project the thin film evaporation was used. Here a thin film of the catalyst material is evaporated on the sample followed by a heat treatment that turns the film into particles. The complete growth process can be seen in figure 2. First a thin film is evaporated on the sample. It is then heated causing particles to form. Finally semiconductor III-V materials is sprayed over the sample resulting in NW growth.



Figur 2: (a) A substrate is covered with a thin film. (b) The substrate is heated causing the film to condense into small particles. (c) Semiconductor material is sprayed upon the substrate causing wires to grow under the particles.

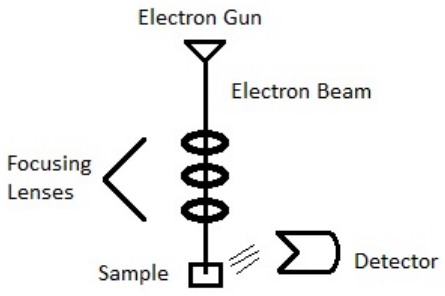
The most used catalyst material is Au which is chosen because of good properties regarding crystal structure and resistance to oxidation and its impact on NW growth has been studied extensively [4], [5]. The problem with using Au is that it degrades performance in Si devices, which make up a large amount of the electronics industries, and it is a very expensive material [1]. Therefore it is interesting to study the possibility of using a different material than Au. This project is a part of a larger study trying to examine Ag as a substitute catalyst. Ag holds many of the same good properties as Au while being cheaper [1]. Ag has already been proven to be able to catalyse NW growth in a study done by A. T. Vogel, *et al.* [6] where Ag catalysed the growth of InSb NW's and in a study done by Dong Pan, *et al.* where InAs NW's was grown on a Si(111) substrate [7]. This project will be dedicated to investigating the behaviour of Ag particles forming from a thin film and using this to guide a NW growth experiment.

2 Instruments

2.1 Scanning electron microscopy

The small size of a NW on the nanometer-scale requires more advanced imaging microscopes than conventional optical microscopes. Two frequently used devices are the Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM). These microscopes utilize signals in the form of electrons or X-rays to study surface, interior, composition and crystal struc-

ture of a sample. The SEM records the signals that are reflected back from the sample but if the sample is thin enough some signals may be transmitted. These transmitted signals are recorded in the TEM. As an example the transmitted electrons are affected by the lattice and they can thus give information about the crystal structure of the sample. The SEM studies a sample by scanning it with an electron beam (e-beam) which generates signals that are recorded. The entire system is kept under a vacuum so the electrons do not interact with particles before they hit the sample. The electrons are created inside an electron gun. Inside this gun free electrons are either created as thermal electrons from a heated filament or by using a sharp tip of tungsten as an cathode which, supplied with a voltage difference to an anode, will strip electrons of the tip. These electrons are accelerated and focused through a series of electromagnetic lenses onto the sample and the generated signals are observed through a detector [8]. A schematic view of the microscope is seen in figure 3.



Figur 3: A simple schematic view of a SEM. An electron gun produces an electron beam that are focused onto a sample through a series of electromagnetic lenses. Interactions between sample and e-beam are recorded by the detector.

When the electron beam hits the sample different types of interactions will occur. Some of the electrons will inelastically scatter inside the sample releasing free electrons in the sample. If these electrons are created at the surface or up to 50 Å inside the sample they can escape [3]. The escaped electrons are called Secondary Electrons (SE) and are the electrons used to study the surface of a sample. By comparing the recorded SE's with the position of the scanning e-beam a surface image of the sample can be constructed. As the incident angle between the beam and the sample increases it becomes easier

for SE's to escape from the sample. This effect makes steep surfaces light up more than the surrounding area creating a 3-dimensional effect which is very suitable for study of NW's since they tend to be inclined or vertical. Apart from condensed matter physics SE's are also used in electronics and biology, where they can be used to image small scale electronic systems, insects or bacteria [8]. Other electrons will scatter more or less elastically inside the sample. These are called Backscattered Electrons (BE). BE's have higher energy than SE's and can escape from further inside the sample. They are used to study the inner composition of the sample. The sample will also radiate characteristic X-rays due to excitations in the sample. These X-rays can be recorded and used to determine which elements the sample contains. The SEM is a versatile tool for NW study and characterisation. It allows for images to be taken in a range from x 10 to x 1 000 000 magnification [3] which is good for viewing NW's. Additionally the sample can be tilted inside the SEM so it can be viewed at an angle.

2.2 Thin film evaporation

The thin film evaporations for this experiment was done by e-beam evaporation. In e-beam evaporation a beam of electrons is steered with magnetic and electric fields onto a source material. This process adds energy to the source material which will start to evaporate particles from its surface. The evaporated particles will travel onto a sample. This will create a layer of the source material on top of the sample. A quartz crystal monitor is used to measure the rate of film deposition in Å/s. The quartz crystal monitor uses a resonator of quartz that is disturbed as mass is added or removed. A shutter in front of the sample allows for thickness to be controlled down to 0.1 nm. As in the SEM the system is placed in a high vacuum so the electrons do not interact with other particles. Additionally the energy of the free source particles is low, less than 1 eV [9]. Because of this the mean free path must be increased to make sure that the particles does not interact with other particles. Generally the mean free path must be brought to a value above the distance between the source material and the sample, which results in a pressure of $3.0 \cdot 10^{-4}$ Torr or lower [9]. On this specific system evaporations is carried out in a vacuum of 10^{-7} Torr or lower. The samples are held inside the system by a little metal arm and a small area of the sample will therefore not be covered with the thin film, figure 4. This area can be used to see the

Figur 4: Picture taken of the sample holder for the e-beam evaporation system.

impact of the source material on the sample.

2.3 Molecular Beam Epitaxy

For this project the Molecular Beam Epitaxy (MBE) technique was used to grow NW's. Epitaxial growth refers to growth of crystal layers on top of a substrate. The deposited layers will order its crystal structure with respect to the structure of the substrate. In MBE the epitaxial growth is initiated by atoms or clusters of atoms that are generated by heating up a source material. These particles will travel to the substrate surface through an Ultra High Vacuum (UHV) environment forming layers of the source material on the surface [10]. The system is build up of three chambers that a trolley loaded with samples can pass through. The first two chambers are designed for a process called degassing. In the first chamber the samples are heated to 200°C and kept there for 2 hours. In the second chamber the samples are heated to 250°C and kept there for 1 hour [2]. The process of degassing removes loosely bound materials from the sample surface such as water, CO_2 and organic materials. The last chamber serves two purposes. First the substrates are put through a heat treatment called annealing. The substrates are heated to a high temperature, for InAs substrates 550°C is common [2]. This lifts the oxide layer on the surface. However, at this temperature the As in the substrate is desorbing so a backup pressure of As is started to counter this. Secondly NW growth takes place here. The growth of the NW's is done in a UHV of 10^{-11} Torr, to reduce impurities formed in the sample [2]. Cells containing the source materials are heated and particles will be released at a measurable rate and travel to the substrate. The As cell is outfitted with a valve for pressure regulation. Since the epitaxial growth is done by building

upon a substrate the crystal structure of the substrate is very important. A typical choice is the (111)B crystal structure where vertical growth is easy to acquire. This comes at the cost of these NW's often having a lot of growth defects. Alternatively the (001) structure is used. This structure has been shown to grow NW's with less defects [11] and it is a cheaper substrate while it is not as easy to get vertical growth. Because of this it is of interest to study growth of NW's using both of these structures.

3 Experiment

Two series of experiments were conducted for this project. The first studied the annealing process of a thin Ag film on an InAs surface. Results from this experiment was used to guide the parameters of the second experiment where InAs NW growth using Ag catalyst particles was investigated.

3.1 Annealing experiment

The size and shape of the Ag particles determines the characteristics of the NW's which underlines why it is important to first investigate the behaviour of Ag alone on InAs before growth is conducted. In this experiment series 5 samples were studied. All samples had a 10 nm Ag film evaporated in an AJA-International e-beam evaporation system by colleagues. 10 nm was chosen so that it would be easier to see the Ag particles formed on the substrates in the SEM. Annealing and degassing was conducted in a MBE system by colleagues. Annealing temperature was set to 550°C. The temperature was kept at 550°C for 0, 5 and 15 minutes to investigate the development of the Ag with time. The parameters for each sample can be seen in table 1. The first sample had no other treatment than the metal deposition. The next 4 were all degassed and sample 2 was taken out after this. The 3 remaining were annealed with a As backup pressure of $1.2 \cdot 10^{-5}$ Torr.

Tabel 1: Parameters for the annealing experiment series.

Sample nr.	Film Thickness	Degassing	Annealing with As backup pressure	Substrate
1	10 nm	-	-	(001)
2	10 nm	250°C for 1 hour	-	(001)
3	10 nm	250°C for 1 hour	550°C for 0 minutes	(111)B
4	10 nm	250°C for 1 hour	550°C for 5 minutes	(111)B
5	10 nm	250°C for 1 hour	550°C for 15 minutes	(111)B

After processing the samples were analysed primarily in the JEOL JSM-6320F SEM and a few times in the Raith E-line SEM. Images was taken at different areas of the sample to give an overview of the entire sample and to discover possible differences in the shape, size and density of the particles. The non coated area was studied to discover the impact of the Ag film. Two different magnification ranges were used in the SEM. Images with large particles was taken in a range from x 7000 - x 9000 while images of small particles was taken at about x 40 000 magnification. Acceleration voltages between 10 and 20 kV were used for the imaging. Images were primarily taken with a top view over the sample, but a few images was taken with the sample placed in a vertical position so the 3-dimensional shape of the particles could be examined. The top view images taken in the JEOL SEM was taken into the program ImageJ. ImageJ comes with a particle analysis tool that was used to determine density and mean area of the particles. From the mean area an approximate mean diameter could be extracted assuming that the particles was circular.

3.2 Nanowire growth experiment

For the NW growth experiment 3 different sets of parameters was investigated. For every set of parameters a (001) and a (111)B InAs substrate were glued, using Ga, onto a GaAs quarter wafer that fit into the MBE. I evaporated a 2 nm Ag film on all 6 samples in the AJA-International e-beam evaporation system. Based on results from previous studies successful in growing InAs NW's with Ag the growth parameters were tailored. Dong Pan, *et al.* [7] discovered that InAs NW's can be grown in a temperature ran-

Tabel 2: Parameters for the growth experiment series

Sample nr.	Growth T	Film thickness	As pressure	T of In cell	Substrate
1A	420°C	2 nm	1.5 $\cdot 10^{-5}$ Torr	860°C	(001)
1B	420°C	2 nm	1.5 $\cdot 10^{-5}$ Torr	860°C	(111)B
2A	470°C	2 nm	1.5 $\cdot 10^{-5}$ Torr	860°C	(001)
2B	470°C	2 nm	1.5 $\cdot 10^{-5}$ Torr	860°C	(111)B
3A	520°C	2 nm	1.5 $\cdot 10^{-5}$ Torr	860°C	(001)
3B	520°C	2 nm	1.5 $\cdot 10^{-5}$ Torr	860°C	(111)B

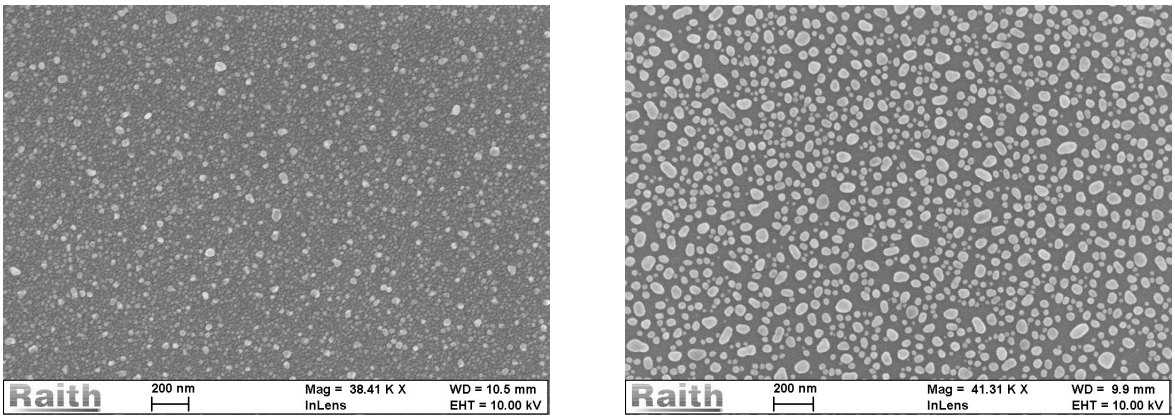
ge from 380°C to 530°C. As a result of this 420°, 470°and 520°C was chosen as growth temperatures, table 2. A Ag film thickness of around 2 nm was used in the previous study so this was also chosen for this experiment. The annealing time was set to 2 minutes. This was guided by the results from the annealing experiment. The As pressure and In cell temperature was fixed for all the samples as seen in table 2.

SEM analysis was carried out after growth both on the JEOL JSM-6320F SEM and the Raith E-line SEM. Images was taken with a top view to get an overview over the samples and discover similarities and differences. The structure of the surface and NW's were imaged with the sample placed in a 45°angle to get a 3-dimensional view. The analysis was carried out in 2 steps. First an image was taken with a magnification of x 700 to get an overview of the area. After this zoomed images was taken of objects discovered in the overview image. These images was taken in a large magnification range from x 7000 to x 40 000 depending on the size of the object discovered. Analysis was also done on the non coated area to discover if NW's had grown there.

4 Results

4.1 Annealing experiment

Ag particles appeared already on the first sample. During degassing the film changed to a high density layer of silver particles. After annealing larger particles was formed and as the annealing time was increased the mean area of particles increased from roughly 250 nm to 380 nm, but the density of



(a) (b)

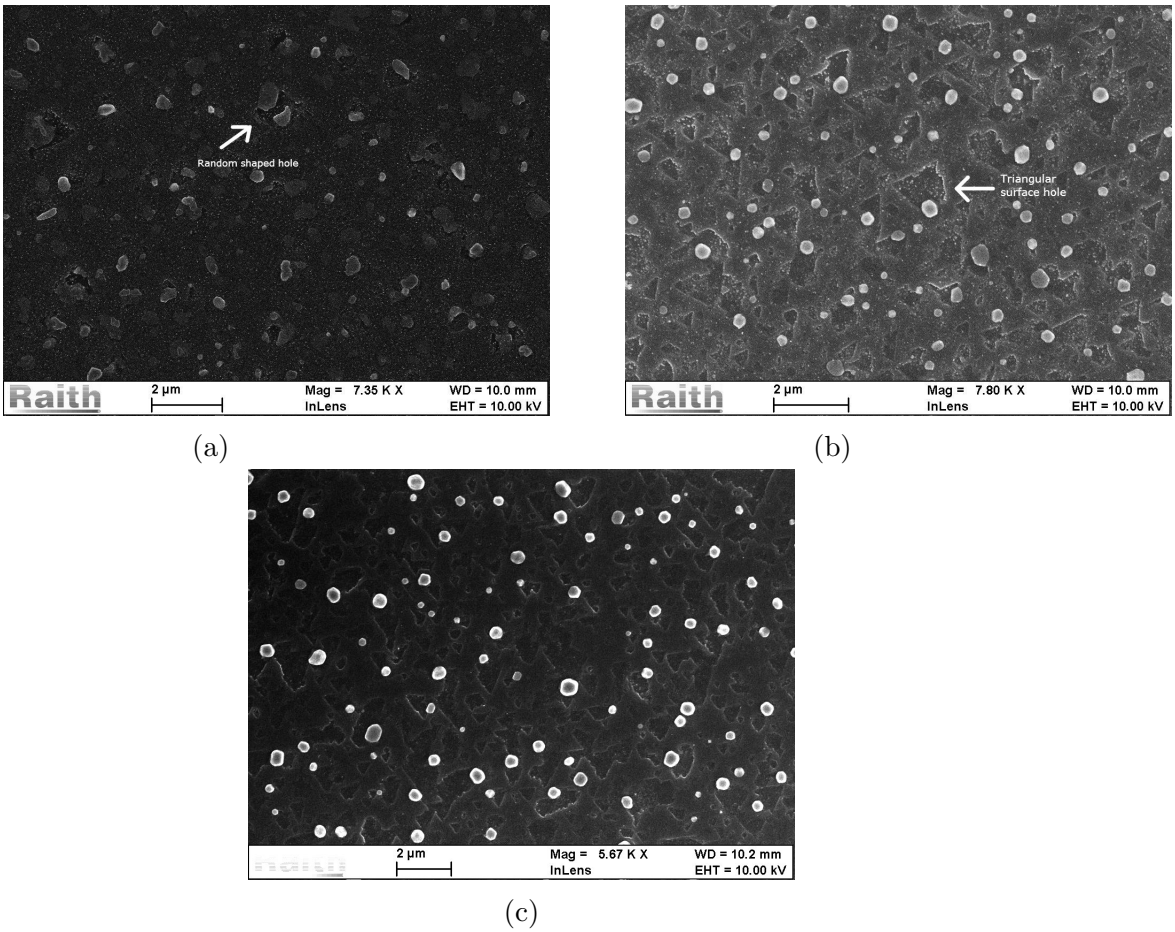
Figur 5: (a) Sample 1, only metal evaporation. Some particles but mostly surface. (b) Sample 2, degassed at 250°for 1 hour. High particle density of sub 100 nm size.

particles decreased from 0.86 $/\mu m^2$ to 0.27 $/\mu m^2$, see table 3. Annealing also affected the surface of the samples. Surface holes changed from random shapes to triangular with increase in annealing time, figure 6.

Sample 1, only Ag evaporation, had particles present, figure 5a. The density of the particles was the second highest at 4 particles per μm^2. The surface contained no surface holes. Sample 2, degassed for 1 hour at 250°C, figure 5b, showed particle growth with the highest density in the experiment, but with the lowest mean area at $7 \cdot 10^{-4}$ μm^2. Sample 3, 550°C brought down immediately, was the first sample to show growth of larger particles up to 500 nm in diameter and with a mean diameter of 250 nm. A large amount of smaller particles were also present on the sample. Their sizes were sub

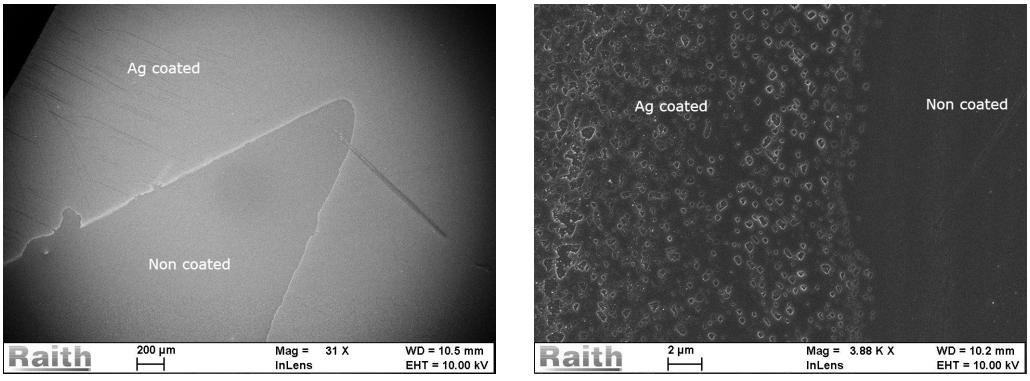
Tabel 3: Table containing results from data analysis of SEM images in ImageJ

Sample	Treatment	Density [$/\mu m^2$]	Mean area [μm^2]	Mean diameter [μm]
1	Metal Deposition	4.09	0.0041	0.0723
2	250°C, 1 hour	396.12	0.0007	0.0312
3	550°C, 0 minutes	0.8631	0.0510	0.2548
4	550°C, 5 minutes	0.5454	0.0731	0.3051
5	550°C, 15 minutes	0.2741	0.1162	0.3846



Figur 6: (a) Sample 3, 550°C and brought immediately down. Both large particles and small particles. (b) Sample 4, 550°C for 5 min. More large particles than Sample 3 and fewer small. (c) Sample 5, 550°C for 15 min. Few small particles remain. Mean area at maximum.

100 nm. The large particles occupied random shaped surface holes while the smaller ones formed on the surface and in the holes, figure 6a. Differentiating between the smaller particles and the inclined surface holes in the ImageJ program was difficult. Because of this the density measured is made up of particles large enough to be recognized as particles. Sample 4, 550°C for 5 minutes, showed the same general result as sample 3. Large particles become more frequent and the small ones less frequent as seen on figure 6b. The larger particles also grew to a mean diameter of 300 nm. The surface holes on sample 4 began to show a triangular shape. Sample 5, 550°C for 15 minutes, has even larger particles with a mean diameter of 380 nm. Here very few of the small sub 100 nm particles remained, figure 6c. Only around the non coated area of the sample were they still present. The surface holes of sample 5 were also triangular. Looking at the non coated area a change in the surface of the samples is observed, figure 7. Inside the non coated area the surface has few holes and no particles. Moving towards the coated area holes starts to form on the surface. Further in particles appear. First it is small particles, but as distance to the non coated area grows the particles become larger.

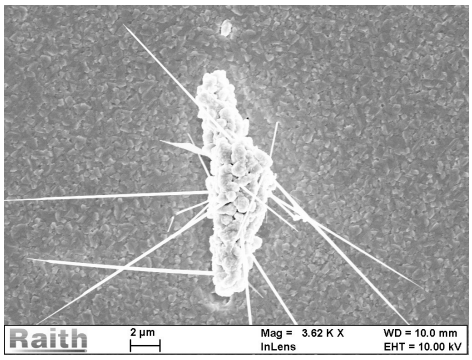


(a) (b)

Figur 7: (a) SEM image of the non coated area of a sample from a low magnification. (b) closer look at sample 5, annealed for 15 min. Inside the non coated area there are some surface holes. Moving away holes cover the surface and particles begin to appear.

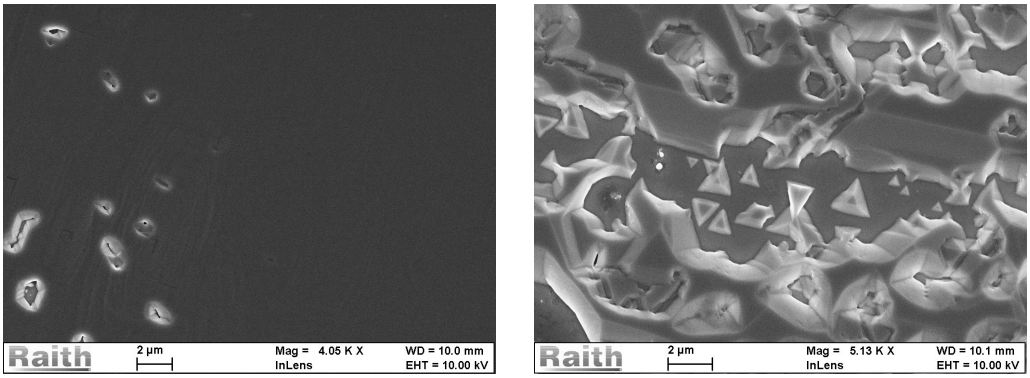
4.2 Nanowire growth experiment

Contamination particles were present on all the growth samples and had in many cases caused NW growth, figure 8.



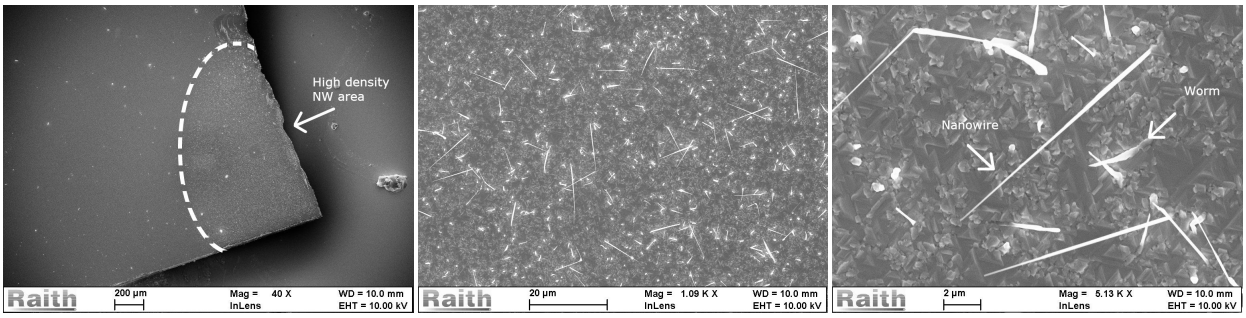
Figur 8: Contamination particle found on sample 1A, (001) substrate grown at 420°C. NW growth is present, but it is not Ag seeded.

As the growth temperature was reduced the surface structure of the samples evolved. The highest growth temperature yielded surfaces that were dominated by randomly shaped holes, figure 9. The small triangular particles seen in figure 9b was present on samples 2B and 3B, both (111)B substrates.



(a) (b)

Figur 9: (a) SEM image of the surface of sample 3A, (001) substrate grown at 520°C. (b) SEM image of the surface of 3B, (111)B substrate grown at 520°C.

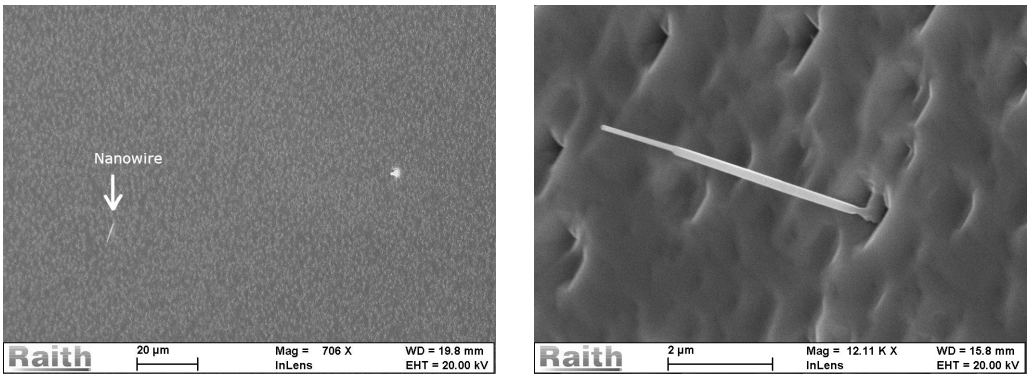


(a) (b) (c)

Figur 10: (a) sample 1B, (111)B substrate grown at 420°C. Overview image of the area with high density NW growth. (b) sample 1B, image of the NW's from 1k magnification displaying the high density. (c) sample 1B, close up of the NW's in 5k magnification

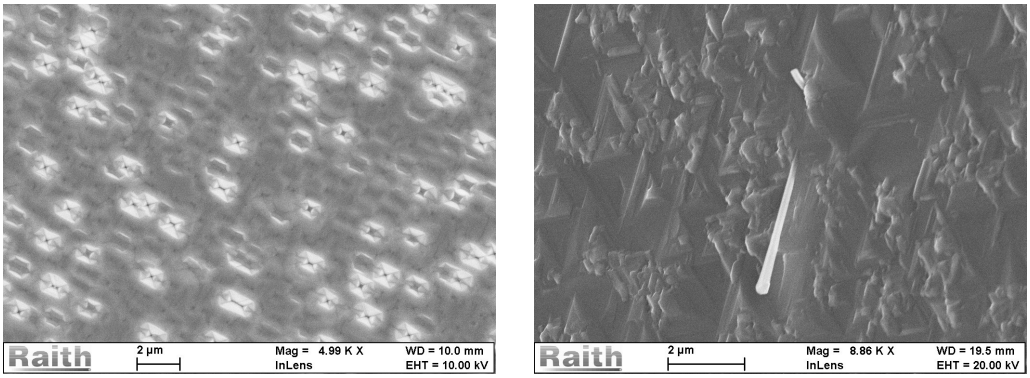
The samples grown at the lowest temperature, 420°C, showed a different surface structure and had NW's grown on them. Two types of NW growth was present on the samples. The first was an area with a high density of NW's, figure 10. The areas were very small, only covering roughly 5% of the total area, and were in both cases placed at a corner of the sample. The area also contained a large amount of random uncontrolled growth called worms. The second type had very low density of NW's present all over the sample, figure 11. A x 700 magnified SEM image would typically contain 1 or 2 NW's if any. It was not possible to find any particles at the tip of these NW's during the SEM analysis.

Analysis of the non coated area of both samples from growth series 1 revealed that no NW's had grown on the non coated area on sample 1A, (001) substrate, while on sample 1B, (111)B substrate, it had. The NW's in the non coated area were found far from the edges. Combining tilted images and top view images the surface structure of samples 1A and 1B was examined. Sample 1A had square shaped holes in the surface while sample 1B had grown triangular shaped islands, figure 12. The surface on the non Ag coated area were randomly structured in both cases.



(a) (b)

Figur 11: (a) sample 1B, (111)B substrate grown at 420°C, overview from x 700 magnification showing one of the scattered NW's. (b) sample 1A, (001) substrate grown at 420°C, showing a close-up image of a NW



(a) (b)

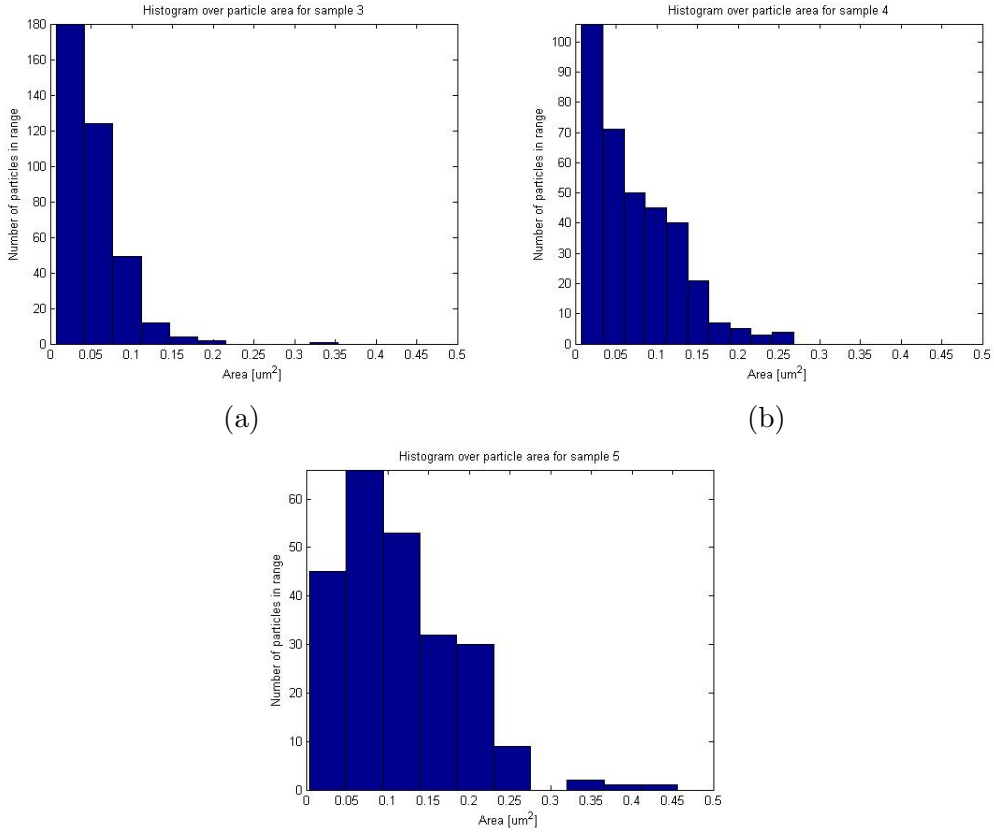
Figur 12: (a) Image of the surface of sample 1B, (111)B substrate grown at 420°C. Fig b) Image of the surface of sample 1A, (001) substrate grown at 420°C.

5 Discussion

The annealing experiment was conducted to give an understanding of how the Ag reacted with the InAs substrate during the annealing phase. It was observed that particles had already formed on sample 1 that only had Ag deposited. This might be an indication that the Ag is mobile on InAs already at room temperature which would allow it to form particles. Another possibility would be contamination on the sample, but this seems unlikely

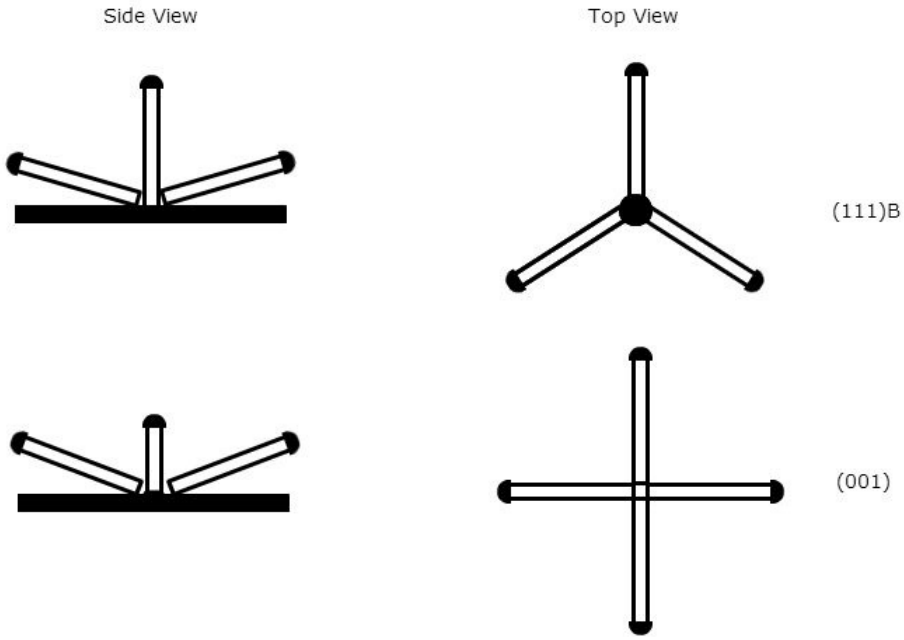
given the amount of particles and their continuity over the sample. From the data in table 3 it can be seen that the average diameter of the particles increase as annealing time goes up. This point can be further emphasized by looking at the raw data used to make table 3 in a histogram, figure 13. It is clearly visible that the bins start to fill more to the right, meaning that the average diameter increases, from sample 3 through 5. This also underlines the fact that the smaller particles vanishes as annealing time increases. This process of smaller particles disappearing around larger particles is called *Ostwald Ripening*. This process is governed by thermodynamics and describes how smaller particles on a surface will dissolve and redeposit upon a larger particle because larger particles are energetically favoured on a surface [12]. *Ostwald Ripening* in this experiment is more evident for longer annealing times. This is most likely because longer annealing times allows the Ag to move around on the surface for longer. Other than *Ostwald Ripening* there is a possibility that the smaller Ag particles simply evaporate inside the MBE during annealing. To test that a simple analysis can be conducted. With 10 nm Ag deposited the samples had 0.01 μm^3 Ag for every μm^2 prior to treatment. Assuming that the particles on sample 3 through 5 is shaped as half spheres the volume of Ag after annealing can be calculated from the data in table 3. A volume of 0.0028 μm^3 Ag per μm^2 for sample 3, 0.0047 μm^3 Ag per μm^2 for sample 4 and 0.0095 μm^3 Ag per μm^2 for sample 5 was found. The volume for sample 3 and 4 is much lower than the pre annealing, but some of the Ag is contained in the small particles that were not analysed in the ImageJ tool which could explain why they are so far from the original volume. Sample 5, which had the fewest of the very small particles, are very close to the 0.01 μm^3 Ag per μm^2 from pre annealing indicating that only a low volume of the Ag have evaporated.

In experiment series 2 NW growth was present on sample 1A and 1B, grown at 420°C. It was however not possible to definitely conclude that the NW's were catalysed by the Ag particles based on the SEM images since particles could not be observed on the ends of the NW's. The areas on samples 1A and 1B that had a high density of NW's was promising, but ultimately the small size of the area and its placement at the corner of the samples, where it is easier to get contamination, led to the conclusion that the NW's were not Ag catalysed. The NW's found all over the sample in lower density shows more promise for not being a product of contamination. It is however possible that they are grown by another growth process called self seeded growth



Figur 13: Histogram figures over mean area of sample 3-5, made from data gathered with the ImageJ program.

where the group III metallic element, here In, is used as catalyst [1]. Through the growth experiment it was observed that samples 1A and 1B developed a distinctly shaped surface structure with square holes and triangular islands respectively. The rest of the samples were dominated by random structured surface holes. The structures found on samples 1A and 1B are a result of which growth directions is most energetically favourable for the substrate. For the (001) substrate, sample 1A, there exists 4 inclined growth directions with 90° between them which results in the square hole shape. For the (111)B substrate, sample 1B, there are 3 inclined growth directions with 180° between them and 1 vertical growth direction which results in the triangular islands [11], figure 14.



Figur 14: Schematic view of the favoured growth directions for both (111)B and (001) substrates.

In the work done by Zhi-Ming Liao *et al.* [5] the triangular islands are also observed with Au particles on top. In this work it is investigated how (111)B GaAs substrate with Au as catalyst behaves under growth. It is concluded that GaAs NW growth happens on top of the triangular islands which are created during annealing. Assuming that the growth process using Ag is similar to Au the presences of these islands on the 1B sample indicates that the lowest growth temperature are closest to starting NW growth. The only thing that is missing is the Ag particles on top of the islands which could be achieved by increasing the thickness of the deposited film.

6 Conclusion

The experiments explored the behaviour of a Ag film on an InAs substrate but Ag catalysed NW growth was not proven. It was discovered that Ag particles was present on a sample already at room temperature and that the average particle area went down as annealing time went up, consistent with the *Ostwald Ripening* process. NW growth was observed on the growth samples but it could not be proven that the growth was not due to contamination or self seeded growth. Additionally comparisons between this experiment and work done by Zhi-Ming Liao *et al.* [5] showed that the triangular shaped islands found at the lowest growth temperature, 420°C, could be an indication of close-to NW growth conditions where only Ag particles were missing. This conclusion could guide parameters for experiments further studying this subject. If more time had been available for this project it would have been interesting to study the forming of Ag particles already at room temperature as this was an unexpected result. It would also have been interesting to conduct a more thorough examination of the NW's to determine if some were catalysed by Ag particles.

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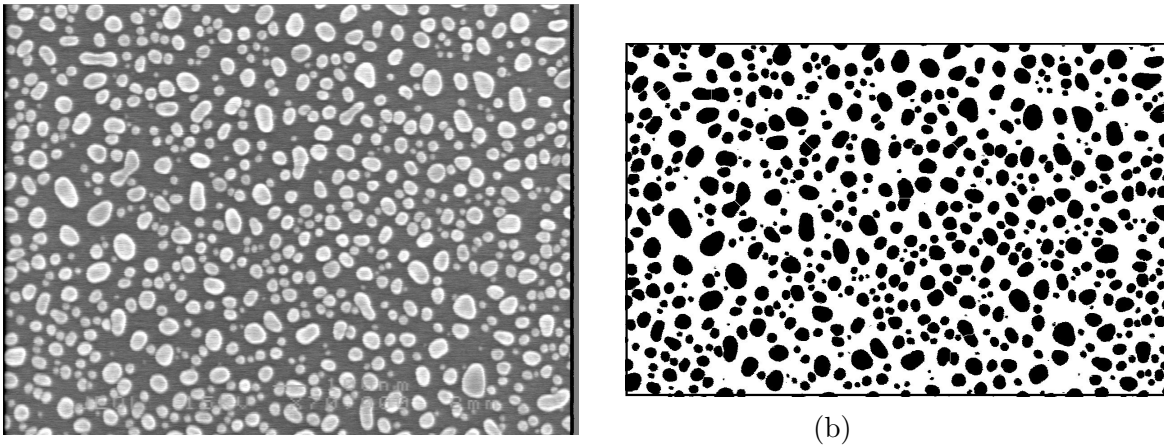
8 Appendix

8.1 Appendix A - ImageJ work procedure

To analyse the SEM images taken in the annealing experiment series the ImageJ program was used. ImageJ comes with a practical counting and analysing tool that made it possible to count many particles and gain information about each particle. The first thing one must do after having loaded the image into ImageJ is to define a scale from the scale bar which comes with all the SEM images. After this one must change the Threshold of the image. The Threshold lets you set a range on a gray scale, pixels with a colour inside the range will be turned white and the ones outside will turn black. Because of this images with good contrast between what is to be analysed and everything else is preferable, figure 15.

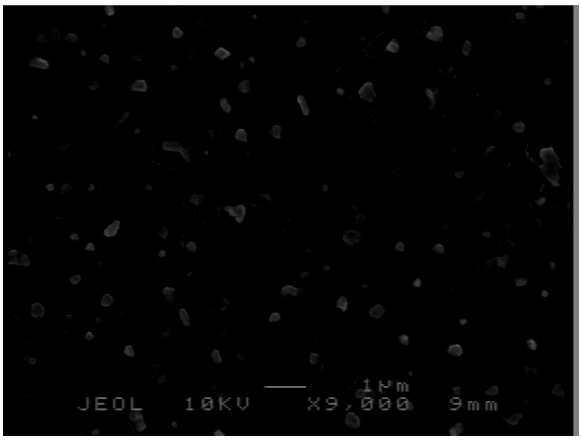


(a) (b)

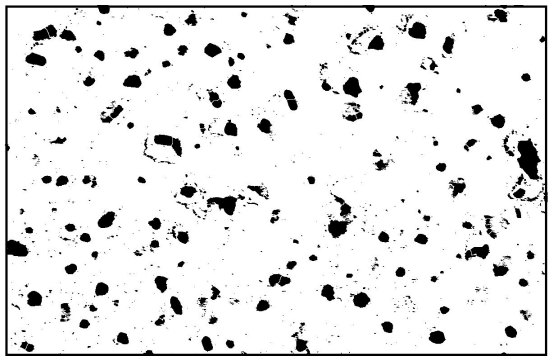
Figur 15: (a) A SEM image of sample 2, degassed at 250°C for 1 hour, from the annealing experiment. (b) The image after having been processed in ImageJ. The particles are nicely present and no surface background is present.

The program does come with a brightness/contrast tool that allows for tweaking of these parameters to achieve better results. If the contrast between interesting and uninteresting objects are to low the uninteresting objects will be counted as well and the analysis will be worsened, figure 16. ImageJ comes with a set of tools that can be used after threshold has been changed to improve the images. Since particle objects in the SEM will light up on

the images because they have inclined surfaces they will turn white after Threshold has been changed. This is a problem because ImageJ counts black objects, so the image was inverted using the *Convert to mask* tool. After this the *Fill holes* and *Watershed* tools were used. They fill any holes in black objects and splits oddly shaped objects. To analyse the particles the *Analyse particles* tool is used. Two parameters can be changed to define what objects are to be counted. *Size* lets you define a range from 0 to infinity. Particles with a size outside this range will not be counted. *Circ* lets you define a range from 0 - 1. This reflects how closely the objects should resemble a circle to be counted. After counting different information can be displayed and a distribution can be made in the program. For this project area was the primary parameter of interest and the distributions where done outside of ImageJ.



(a)



(b)

Figur 16: (a) A SEM image of sample 3, annealed at 550°C and immediatly down again, from the annealing experiment. (b) The image after having been processed in ImageJ. The particles are visible, but a lot of surface background are present.