Medical instruments identification via a luminescent micro-particle coating as an alternative for existing identification methods

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Thesis Report

Medical instruments identification via a luminescent micro-particle coating as an alternative for existing identification methods

by

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This thesis is subject to confidentiality until 31 December 2018

TU Delft

Abbreviations

The following abbreviations are used in this thesis:

Abbreviations	
BAM:Eu	Barium Magnesium Aluminate, Europium doped
CDS	Cleaning, Disinfecting and Sterilizing
CSSU	Central Sterile Supply Unit
EDS	Energy Dispersive X-Ray Spectroscopy
EMI	Electro Magnetic Interference
ENP	Electroless Nickel Plating
FDA	Food and Drug Administration
IMDRF	International Medical Device Regulators Forum
MAM	Medical Asset Management
RFID	Radio Frequency Identification
RTLS	Real Time Location System
SEM	Scanning Electron Microscopy
UDI	Unique Device Identification
YO:Eu	Yttrium-oxide, Europium doped

Preface

'And then we found out the instrument was over 30 years old!' This sentence was the starting point of my graduation project. During the course *Surgery for Engineers*, we discussed a case about an instrument (small tongs) which broke down during a small intervention. Fortunately, this happens at the ENT clinic, so the medical professional could easily retrieve all the small metal parts. The ENT doctor wanted to know how this was possible, and via a serial number and some old advertisements they found out that the instrument was over 30 years old. After hearing this story I immediately thought; they could/should have know this was bound to happen! How is it possible that for the instruments that save our lives this information is not available?

This was the starting point for my graduation project and the result of this lays in front of you. This was not possible without the help of John van den Dobbelsteen. John helped me to shift from RFID as a tracking technique to the alternative technique which I'll explain in this thesis. This alternative technique was the result of a creative moment between John and Julian Apachitei. I have to thank them both for introducing this technique to me. I have to thank Julian again for his patience and help with the chemical part of this research. As he clearly told me at the beginning; This project involves a lot of chemistry and you are not a chemistry student. I gladly accepted this challenge and I actually really enjoyed the chemistry part. This research could also not have been successful without the help of Matty Wijnen. Not only did he provided the chemical components for the electroless nickel plating, but he also helped me with the nickel strike. I have to thank Rob Luttjeboer, for helping me in the chemistry lab and Sander Leeflang for helping me with the Scanning Electron Microscope.

Next to that, I want to thank my friends and family. My dad Bert, whom I always called when something went right or completely wrong with my experiments. My mother Jet, whom provided everything so I could completely relax when I went home for the weekend. Marloes & Jan Jaap and Annelore & Bas who both had beautiful babies during my graduation project. Anne-Jetske and Tijn, no stack of literature or failed experiments can bring me down when we are dancing the 'Disco Pogo'. My girlfriend, Evalyn, whom was the one that give me a 'schop onder mijn kont' when needed. And finaly, my grandpa Pake, for being the source of my preference for technology, by infusing me with the 'Pake make' genes. Thank you all for the support, not just during my graduation project but during the entire 7,5 years of study.

J. H. Sanders
Delft, December 2017

Contents

1

Abstract

Medical Asset Management (MAM), track and tracing of medical instruments throughout a hospital, is very comprehensive and complicated. To implement such a system on individual instrument level, a technique that can create Unique Device Identification (UDI) codes is needed. Current UDI systems, such as RFID and barcode scanning, do not suffice. Adding a RFID tag to a large instrument is not a problem, but adding the tag to a small scissor can be problematic. Adding a barcode to an instrument can be done in multiple different ways such as; laser engraving, adding a sticker or adding a tag with a barcode on it. The tags tend to be removed by medical professionals and stickers can get loose. Laser engraving seems to be a good alternative, if no human material would stay behind in the slots after sterilizing.
Both techniques discussed above are not specific designed for Medical Asset Management. The goal of this thesis is to research if a different technique can be used for MAM; medical instruments identification via luminescent micro-particles coating. The main question is:
Is it possible to coat individual medical instruments with a coating via electroless nickel plating, with embedded luminescent micro-particles and identify these instruments?
To answer this question, this thesis is divided into 4 parts; Electroless nickel plating of stainless steel; Physicochemical characterization of the coatings; Identification of the substrates and Implementation. During the 8 experiments, 316L medical grade stainless steel substrates are coated and characterized. The identification part consists out of building a setup for a UV-C light and creating a Matlab file for automatically identification of the substrates. Medical instrument experts are consulted for the implementation part.
With the use of a nickel strike as a pre-treatment, a successful coating was embedded into the substrates. The Barium Magnesium Aluminate (BAM:Eu) particles were visible via the SEM analysis and was identified via a EDS analysis. Exciting the substrate with the UV-C light resulted in a clear blue substrate. Trying the same experiments with different particles, Yttrium-Oxide (YO:Eu), was not successful. The dispersion of the YO:Eu particles was different in comparison to the BAM:Eu particles. The YO:Eu particles eventually sank to the bottom or agglomerated on the top of the solution. The created Matlab script was successful in identifying both colors. During the discussion of the technique with medical instruments experts, questions arose about the change in material properties. Some properties do change, but the impact of this needs to be further researched.
This research proved the concept of identifying individual medical instruments via a luminescent micro particle coating. It is possible to coat and identify a substrate with this technique.

2

Introduction

Hospitals have a lot of assets inside their building. All these assets (e.g. beds, apparatus, instruments etc.) need to have a storage place so they can be easily found when needed. Medical Asset Management (MAM) is the term that describes the systems that are used to manage (track and trace) all the medical assets in a hospital. These assets can include anything that can be found in a hospital. The focus of this research will be on the medical asset management of instruments.

Supply costs account for more than one-third of the average operating budget, and constitute the second largest expenditure in hospitals[7] [3]. The supply chain of a hospital is very complex, some of the unique challenges stated in Coustasse et al.[7] are; high costs due to the large amount of expensive inventory, and the lack of visibility in the supply chain can result in a lot of lost, stolen or misplaced equipment. Furthermore a reason for the high costs is the complexity of inventory tracking. This is because of the urgency of medical procedures and the unpredictability due to the diversity of the patients' characteristics.

Healthcare operation failures are the effects of common phenomena that are identified in five problems by Yao et al[31]. These five common phenomena are:

1. Inefficient workflow
2. Increased costs
3. Medical mistakes
4. Theft loss
5. Drug counterfeiting

Decreasing the inefficient workflow, which is linked to the increasing costs, could be cost saving. In 2015, the Netherlands spend 95,3€ billion, which is over 14% of the GDP[4] and these costs are increasing ever more. In addition, as stated above, supply costs account for more than one-third of the average operating budget and constitute the second largest expenditure in hospitals[7]. Furthermore, achieving high operational efficiency is essential for the organizational performance evaluation[31]. A way to reduce hospitals' expenses is to address supply chain management inefficiencies. This can be achieved by using automatic data capture technologies and supply chain automation solutions[3]. A process of decreasing the inefficient workflow is called *lean thinking*. This principle tries to reduce the waste, where waste is defined as any activity that consumes resources without creating value, while increasing the percentage of value[3].

Traceability is described by Bendavid et al. as; the ability to track forward the movement of products through specified stages of the supply chain and trace backward the history, application or location of products[3]. Bendavid et al. also discussed why it is necessary to track and trace individual instruments:

1. To comply with regulatory requirements and guidance on product recall and withdrawal
2. Increase efficiency of operations
3. Ensure product authentication
4. Support patient safety

2.1. What to measure

At the moment, there is not a system that can easily, without extra acts by medical personnel, manage these instruments. Techniques which can do parts of this, are discussed in section 2.2. The big data that can be derived from tracking all the movements of the instruments through a hospital can be very interesting and can result in many cost-effective modifications. Different kinds of data which can be collected are discussed below as well as the benefits of this data.

2.1.1. Device data

The device data can store all the information about the device, such as; type of instrument, year of origin, manufacturer, supplier, serial number etc.

2.1.2. User data

The user data can store the information about usage. This can track the amount of times used [6]. This allows for better insight about the quality of the instruments and can indicate when an instrument needs to be replaced, repaired or discarded. This can also include what kind of maintenance and cleaning is done and by who and when[6]. Other user data can be on which patient the instrument was used, for what kind of surgery operation and by which medical professional.

2.1.3. Real time location data

The real time location data can store the data about the location of the data and can give insight about in which sterilization phase the instrument is. This data can also be used to see which instrument is used during a surgery operation and how many times this instrument was available but not used.

2.1.4. Benefits of the data

Gathering all the data stated above gives great insight in the life cycle of medical instruments. Examples of the benefits of this data are discussed it the next part.

Optimizing the kits

Mhlaba et al.[17] conducted a data analysis about the percentage of used instruments by kit. They observed 20 surgical cases, performed by 7 different surgeons and performing 10 different procedures. The utilization of the instruments was on average 22% (SD=±10%, range 9%-43%). They also calculated the average price for reprocessing an individual instrument, which is between $0,34-$0,47 in a tray and $0,81-$0,84 for individually wrapped instruments[17]. This shows that decreasing the amount of instruments in a kit can also lead to more savings. Farrokh et al. did a similar research and found that they could reduce the instruments by 70% for a minimally invasive spine surgery. This resulted also in a decreased set up time of 37%[11].

Reduce amount of expensive devices

Another way to safe money is to reduce the expensive devices inside a hospital. It is sometimes hard to find the desired mobile device, since medical personnel tend to 'hide' the device, discussed in the article of Kamel Boulos et al[16]. 'Hiding' certain mobile devices only enlarges the problem. This results in over-purchasing of 10 to 20% of expensive mobile medical devices such as ICU ventilators and intravenous (IV) pumps. Reducing the amount of these expensive devices will not only save money on capital expenses but also on operational expenses (storage and maintenance)[16].

Reduce inventory analysis time

Bendavid et al.[3] did a research at Centre hospitalier de l'Université de Montréal (1200 beds). They recorded the time necessary for the replenishment process and forcasted the time saved on logistics processes due to productivity gains during the replenishment process for traceable items. Using traceable items reduced the time needed for these processes significantly.

Anticipate repair

If number of usage could be recorded by the system, surgical instruments could be taken care at the appropriate timing before being beyond repair, and the number of disposed instruments would decrease[29]. This can result in a longer life span of the instruments which will reduce the costs.

All this data proves there is a opportunity present to enhance the efficiency of the usage of instruments. Coustasse et al.[7] stated that hospital personnel fails to locate a mobile asset anywhere from 15 to 20 percent of the time because of misplacement. By automating manual tasks, the chance of an error is reduced and work can be done faster. The challenge for optimizing the kits is to predict which instruments are necessary for each surgery operation. By track and tracing individual instruments, a database can be created to give in-

sight about the usage and the location. This database can be used to create better kits whit lesser instruments which reduces the costs of cleaning and enlarges the life-span. Furthermore, it gives insight on the location of instruments and their stock keeping unit.

2.2. Existing UDI codes techniques

To track and trace medical instrument throughout the hospital, the instruments need to be given a UDI code. To add a UDI code to a medical device, different techniques are available. The most commonly used technique in hospitals for identifying devices is applying a barcode to the device. An alternative for barcode scanning is the use of RFID tags and readers. The advantages and disadvantages are discussed in the next part.

2.2.1. Advantages and disadvantages of barcode scanning

The biggest advantage of barcodes is that it can be printed on a surface. This can be done with ink or can be laser engraved on an asset. The disadvantage is that a barcode can be hard to read when the line of sight (LOS) is not free. The laser must be in line with the code and free of any blockage. This makes it hard to scan a barcode since instruments are often covered in human material. Another disadvantage of barcode scanning is that each individual barcode needs to be manually scanned. This is time consuming and prone to result in a miss scan or can be forgotten[21]. Since there is no information stored in the code, a barcode is very safe. The only information stored on the device is the product number which is only useful with the information stored in the software. When a product is lost, there will be no loss of information.

2.2.2. Advantages and disadvantages of RFID

Studies have shown that applying RFID to medical equipment has resulted in: 1) an increase in efficiency; 2) lower supply costs and 3) an increase service quality[7]. An advantage of RFID technology above other RTLS is that multiple tags can be read at once when in the proximity of a reader, while e.g. bar-codes need to be read one by one and need to be in line of sight of the scanner. The range of the reader can be controlled, which makes it possible to create different zones (e.g. a readerzone around the patient, a readerzone around the mayo stand etc.). According to surgeons' comments who used surgical instruments with RFID tags, the attached tags did not interfere with the normal surgery procedure[29]. However, not every instrument can be provided with a RFID-tag. For example, small clips used for brain surgery or a scissor used for eye surgery are too small to add a RFID-tag[29].

Another disadvantage is the possibility that RFID-signals can lead to electromagnetic interference (EMI) on critical care equipment. This was tested by Van der Togt et al. (2008). on 41 medical devices (in 17 categories, 22 different manufacturers). Van der Togt used an active 125 kHz tag and a passive 868 MHz tag. They tested each device three times which resulted in 34 EMI incidents of which 22 were classified as hazardous, 2 as significant and 10 as light[27]. In another article from Van der Togt et al. (2011), he states that the performance of RFID technology to current industry and ISO standards does not fully guarantee that an RFID system will likewise show good performance within the specific health care settings[28]. More testing is necessary to see if this new (for the hospital) technology does not bring new risks to patients' health. On the other hand, research done by Guédon et al. (2014) showed no interference of 94 active RFID tags (869.3 MHz)[12]. These tags where placed on the devices for 2 minutes and resulted in no interference with all the devices.

2.3. Laws & Regulations

On 21 June 2017, the minister at that time of Health, E. Schippers, sent a letter to the chairman of the Lower Chamber about the agreements of unambiguous coding of medical devices, see Appendix A (figure A.1). Applying a unique device identification (UDI) code to each instrument to facility a track and trace system has many benefits. It will not only improve patient safety, but will also reduce the costs of the supply chain in a hospital. The Food and Drug Administration (FDA) and the International Medical Device Regulators Forum (IMDRF) are also pushing for the application of UDI's to medical devices. The FDA wants to have the UDI completely introduced by 2020. According to the IMDRF, UDI should be introduced because of:
'A globally harmonized and consistent approach to UDI is expected to increase patient safety and help optimize patient care by facilitating the[2];

1. Traceability of medical devices, especially for field safety corrective actions
2. Adequate identification of medical devices through distribution and use
3. Identification of medical devices in adverse events
4. Reduction of medical errors
5. Documenting and longitudinal capture of data on medical devices.'

This proves there is a demand of a system which can create UDI codes and apply them to medical instruments to be used for a track and trace system.

2.4. Conclusion introduction

As discussed above, the existing techniques have their advantages and disadvantages. Besides, other reasons for the slow adoption as discussed in the literature research are:

1. Unclear Return on Investment
2. Unclear Patient Advantages
3. Privacy concerns
4. No standardization in RFID technique for hospitals

Standardizing a technique for hospitals to use for tracking and tracing purposes can be very important and should be the next step, as discussed in the letter of E. Schippers (see Appendix A). Hospitals are holding back on investing in one particular technique, since the technique that will become the standard is as of today unknown. A conclusion can be drawn that RFID technology and barcode scanning are not the solution for tracking and tracing medical instruments.

An alternative and less researched about tracking method is adding a coating of luminescent microparticles to the medical instruments. This coating is very durable and will not be affected by the cleaning or sterilization process. The particles will emit light when excited by a UV-light. This could make it possible to create different color-codes to identify medical instruments without seeing any change to the instruments.

2.5. Aim and scope

The aim of this research is to develop a track and trace system for individual medical instruments via electroless nickel plating coating bearing luminescent micro-particles, which are automatically readable with a camera when excited by UV light. The thesis is divided into four parts:

1. Electroless nickel plating of stainless steel
2. Physicochemical characterization of the coating
3. Identification of substrates
4. Implementation of the technique

The scope of this research is to assess if the luminescent micro-particle coating has the potential to be an alternative for the existing identification techniques. It is limited to only this technique and two luminescent particles.

2.6. Research questions

The main question of this thesis is to research the possibility to provide individual medical instruments with a coating via electroless nickel plating, with embedded luminescent micro-particles, and identify these instruments.

Sub questions

1. Is it possible to coat 316L stainless steel with luminescent micro particles?
   (a) Do the particles emit light when excited by UV-light?
   (b) Do the particles mix with the electroless nickel solution?
   (c) What kind of pre-treatment is necessary for 316L stainless steel for electroless nickel plating?
   (d) Is it possible to use BAM:Eu and YO:Eu as particles for this experiment?
2. Is it possible to characterize the properties of the nickel plated substrates?
   (a) Are the particles visible via SEM?
   (b) Are the particles detectable via EDS?
   (c) Does the presence of the micro particles influence the thickness of the nickel layer?
3. Is it possible to identify the difference in color of the excited luminescent micro particles using a Matlab file?
   (a) Is it possible to identify the different particles when excited using a Matlab file when dry?
   (b) Is it possible to identify the different particles when excited using a Matlab file when embedded in the coating?
4. Wat is the opinion of medical instrument experts about using this technique for individual medical instruments identification in a hospital?

2.7. Experiments

To answer the research question as stated, multiple experiments were performed:

Exp. 1: Check particles with UV-Lamp when dry
Exp. 2: Mix particles in nickel bath at 20°C
Exp. 3: Nickel plating of 2 substrates of stainless steel 316L
Exp. 4: Nickel plating of 1 substrate of steel and 1 substrate of stainless steel 316L after nickel strike
Exp. 5: Nickel plating after nickel strike of 2 substrates of stainless steel 316L
Exp. 6: Nickel plating after nickel strike of 2 substrates of stainless steel 316L with BAM:Eu particles
Exp. 7: Nickel plating after nickel strike of 2 substrates of stainless steel 316L with YO:Eu particles
Exp. 8: Nickel plating after nickel strike of 2 substrates of stainless steel 316L with BAM:Eu particles at the top half and YO:Eu particles at the bottom half

To validate the experiments and to check if the coating is successfully applied the following characterizations are performed:

Char. A: Physical
Char. B: Morphology - Scanning Electron Microscopy (SEM)
Char. C: Chemical composition - Energy Dispersive X-Ray Spectroscopy (EDS)

The identification part will focus on exciting the substrate with a UV-C light and the identification of the substrates. This is done with a Matlab file. To get feedback from the end-user, the medical instrument professionals from LUMC, the technique will be discussed with them.

3

Method

The methods used for the different parts of this research are discussed in this chapter. As stated in the introduction, the parts of the thesis are;

1. Electroless nickel plating of stainless steel
2. Physical & chemical characterization of the coating
3. Identification of substrates
4. Implementation of the technique

3.1. Electroless nickel plating

Electroless plating is the autocatalytic reduction of aqueous metal ions absorbed to a substrate without passage of external current.[24]. The advantage of electroless over electrolytic is that the nickel coating will follow the contours of the original substrates equally and creates a uniform thickness[20]. This is not the case when electrolytic nickel plating is used. The process of electroless nickel plating is explained in the document provided by AHC-Benelux [1] and called DNC-520. The DNC-520 is a process used for industrial nickel plating. This process needed to be adapted to be used for the experiments. A new document was created for easy, step by step, documentation of the performed experiments. This document is adapted for each experiment. An example of this document is added in Appendix E, figure E.1.

3.1.1. Pretreatment

Nickel strike

Stainless steel is covered by an oxide layer to protect the materials. This will also protect the material inside the nickel bath. To enhance the possibility of a successful nickel plating, a nickel strike is necessary. This is done in a electrolytic nickel bath, which consist of nickel chloride and 30% HCl. The nickel strike replaces the oxide layer with a thin layer of nickel (approx. 500nm). This new, thin nickel layer oxidizes not as fast as the original layer after being activated in HCl. The nickel strike was performed by AHC-Benelux [1].

Degreasing

Before the substrate can be used for electroless nickel plating, the substrates needed to be degreased and activated. This was done by placing six beakers in a row and filling them with 100ml of the following:

Beaker 1: Acetone
Beaker 2: Demi Water
Beaker 3: Ethanol
Beaker 4: Demi Water
Beaker 5: 30% HCl
Beaker 6: Demi Water

The substrates were placed in beaker 1, 3 and 5 for 1 minute and were rinsed after each minute in beaker 2, 4 and 6 and hung to dry.

3.1.2. Electroless nickel plating process

Nickel bath

To create a 0,5 liter nickel bath, the two DNC-520 solutions are needed and demi water in the following concentrations:

75 vol.-% Demi water (389ml)
18 vol.-% Solution-1 (Badensaltz, 90ml)
4.2 vol.-% Replenisher-1 (21ml)

This leaves 2.8 vol.-% for adding a acid or base to control the pH level. The pH level must be between 4.4 and 4.8 at 20°C. If the pH level needed to be lowered, a sulphuric acid was added. If the pH level needed to be increased, ammonia was added. If the pH level was stable, the remaining 2.8 vol.-% (14 ml) was filled with demi water.

Bath loading

As stated in document DNC-520 the bath loading (substrates) must be between 0.2 and 1 dm^2/l. For the experiment, a bath of 0,5 liter was used. This allowed for a bath loading between 1000mm^2 and 5000mm^2 per 0.5 liter.

Bath maintenance

During the nickel plating process, the nickel bath is heated up to between 88 and 94 °C for 60 minutes. At this temperature, water will evaporate. To keep the correct concentrations during these 60 minutes, demi-water needs to be added to keep the nickel bath at the 0,5 liter level and to keep the substrates submerged in the solution.

3.2. Characterization of nickel-phosphor coating

The substrates needed be to analyzed to see if the coating was embedded into the substrates. This analyses consisted out of physical characterization, morphology analysis and chemical composition analysis.

3.2.1. Physical characterization

The characterization of the physical properties was done in the following ways:

Weight Analysis

The substrates were weighted before and after each nickel plating process. The weight of the substrates is a good indicator of a successful nickel plating process, since the weight should be increased after the nickel plating process. It is also possible to calculate the thickness of the nickel layer with this data via formula 3.1[23].

$$T = \frac{b-a}{\rho} \cdot \frac{1}{A} \tag{3.1}$$

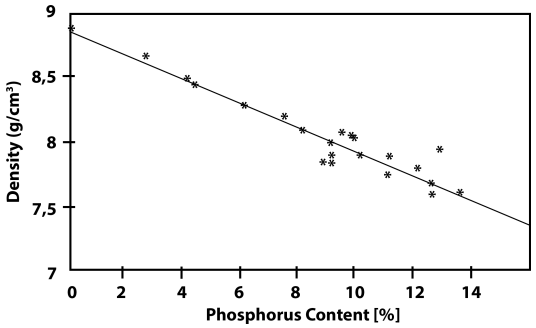
With b being the weight of substrate after nickel plating (in mg), a the weight of substrate before nickel plating (in mg), ρ the density of the electroless nickel (in mg/cm^3) and A the surface area of the substrate (in cm^2). The surface area of the substrate is 18,25cm^2, as discussed in Chapter 4. The density of the electroless nickel depends on percentage of phosphor as can be seen in figure 3.1 which approx. has the formula stated in equation 3.2, with x being the phosphorus content percentage. In the document '*DNC-520*' provided by AHC Benelux [1], a phosphor content percentage of 9 - 13 % should be achieved. This document also stated that the deposition rate should be around 10 - 14 μm/h.

$$\rho = 8,875 - 0,09375 \cdot x \tag{3.2}$$

Color analysis

The color of the substrate should change after nickel plating the substrates. Before nickel plating the substrates, they should be more yellowish. After nickel plating the substrates, they shall have a uniform, metallic appearance without visible defects [23]. Imperfections in the coating caused by imperfections in the substrate are possible, since the nickel coating coats the substrate uniformly. The coated substrates were compared to a provided piece of nickel plated substrate.

Figure 3.1: Effect of composition on deposit density[20].



3.2.2. Morphology analysis

The morphology of the substrates are characterized via Scanning Electron Microscopy (SEM). This microscope is used to make pictures at magnifications of 100, 1000 and 2000 times. These pictures showed the structure of the coating present on the substrate. Before the substrate were be placed in the SEM, the substrate needed to be prepared, as presented in Appendix F fig. F.1. The substrates were cut to create a cross-section which exposed the layers on the side of the substrate. This can be seen in F.2. The gray area is the substrate which is analyzed during this step. Ten substrates were mounted in PolyFast, which is a conductive resin (fig. F.3) for SEM and EDS analyses. This was done in the Mounting & Sanding and Polishing room at 3ME. Cleaning the substrates is important to only analyze the elements of the substrates and not the elements of a contamination. This was done by placing four beakers in a row and filling them with the following liquids and adding the substrates to it for 1 minute:

Beaker 1: Acetone
Beaker 2: Demi Water
Beaker 3: 2 iso-Propanol
Beaker 4: Demi Water

The substrates went in beaker 1 and 3 for 1 minute and were rinsed between the beakers in 2 and 4.

3.2.3. Chemical composition analysis

The characterization of the chemical components of the coating were evaluated via Energy Dispersive X-Ray Spectroscopy (EDS). After a magnification of 2000 times, multiple points were selected to be analyzed. The EDS showed the different atom-percentages of the elements present at the selected points. These percentages were compared to the chemical formulas of the luminescent particles. The formula for BAM:Eu is shown in (3.3) and the formula for YO:Eu is shown in (3.4).

$$Ba_{0,86}Mg_1Al_{10}O_{16} : Eu_{0,14} \tag{3.3}$$

$$Y_{1,92}O_3 : Eu^{3+}_{0,08} \tag{3.4}$$

3.3. Identification

During the identification part of this research the substrates were excited by a UV-light to test if the substrates emit color. The substrates were excited with a UV-light of 254nm (UV-C). Since UV-C light can be harmful to humans when directly exposed to it, the UV-light was placed inside a box. A Matlab file was created to automatically identify blue (BAM:Eu) from red (YO:Eu).

3.4. Implementation

For feedback from the user, medical instrument experts were consulted. The electroless nickel plating with luminescent micro-particles technique was explained and the (dis)advantages are discussed. The focus of this discussion was on the possible hazards for this technique in the CSSU. This was done with Dr. Ir. Van der Eijk[26] and Drs. Jungblut[15] from the LUMC.

4

Materials

The materials used for the four parts of this research are explained in this chapter.

4.1. Electroless nickel plating

The materials used for the electroless nickel plating process are explained in this part. In subsection 4.1.1 the experimental setup is explained and in subsection 4.1.2 the materials used for this setup are explained.

4.1.1. Experimental setup

The experimental set-up improved over time during this thesis project. The final version of the set-up is added in Appendix D. The schematic set-up for this experiment is presented in figure D.1 and pictures of the set-up are presented in figure D.2.

4.1.2. Materials used for electroless nickel plating

Particles

The particles used for this research are chosen from the provided list added in Appendix B. For the electroless nickel plating process, two particles are selected, *Barium Magnesium Aluminate, Europium doped (BAM:Eu,* and *Yttrium-oxide, Europium doped (YO:Eu).* A blue-emitting and a red-emitting particle are chosen since the difference in their wavelengths are the biggest, which makes it easier to distinguish between them (figure B.1). The particles are acquired via *Sigma-Aldrich*[18]

Barium Magnesium Aluminate, Europium doped (BAM:Eu)

BAM:Eu is considered to be a very good blue phosphor, since it has high chemical stability, a high luminance efficiency and good chromaticity under ultraviolet light[14]. The emission peak of BAM:Eu is between 440nm and 460nm. The characteristics of BAM:Eu are in appendix C (figure C.1).

Yttrium-oxide, Europium doped (YO:Eu)

YO:Eu is the most frequently used material to provide red light emission for electronic devices. Yttrium-oxide is very chemically stable which makes it perfect to be a host material for Europium[8]. The emission peak of YO:Eu is between 609nm and 613nm. The characteristics of YO:Eu are in appendix C (figure C.2).

Substrate

The material used for the substrates is 316L medical grade stainless steel, since most of the medical instruments are made from this material. The plate is cut in substrates of 42.5mm x 20mm x 1mm. A hole of 2mm is drilled in the substrate. The surface area of the strip is $1825 mm^2$. To validate the nickel bath (exp 4.), another steel substrate (not stainless steel) is used. These strips are 50mm x 20mm x 1mm, thus a surface area of $2140 mm^2$. This makes it possible to put a maximum of 2 strips (1 of each or two the same) in a 0,5 liter solution since the maximum bath loading is $5000 mm^2$, see chapter 3.

Chemical materials
DNC-520 is the commercial name of the process used for creating the nickel bath. DNC-520 consists of 2 chemicals, Solution-1 and Replenisher-1. These chemicals are provided by AHC Benelux [1]. Other necessary chemicals are ammonia, to control pH level, and acetone, ethanol and HCl to clean the substrate. These chemicals are available in the PME-Lab. All the experiments needed to be performed in a chemical cabinet with a fume hood. This was also available in the PME-Lab, as well as all the necessary glassware, pH-meter and personal protection equipment (lab-coat, gloves and glasses). A magnetic stirrer/heater was available from the Misit-Lab (IKA - RET Control-Visc).

4.2. Characterization

Weighing the substrates before and after the nickel plating process was done with scale 'Scaltec SBC 33'. For analyzing the cross-section of the substrates, a SEM mount is created in the Predopress with PolyFast. PolyFast is a conductive resin which makes it suitable for usage with the SEM. LaboPol-21 was used for sanding the mount with P80, P180, P320, P800 and P1200 sanding paper. Polishing is done with the LaboPol-5, with polishing substance of $3\mu m$ and $1\mu m$. The used Scanning Electron Microscope is the 'JEOL JSM-IT100 InTouchSchope'™with the extended EDS functions. This microscope was provided by the faculty 3ME of the TU Delft. To keep the substrates in place in the Predopress, a plastic clip was used which can hold 5 substrates. For surface analysis, conductive double-sided carbon tape was used to keep the substrates in place in the vacuum chamber of the SEM.

4.3. Identification

The materials used for the identification of the substrates were;

1. UV-light and holder
2. Matlab file
3. Photo camera

A UV-light with a wavelength of 254nm was used to excite the particles. This wavelength is often used in literature to excite these specific particles[5][13][22][32]. The UV-light used for the identification was: Philips TUV TL-D 15W UV-C. This light is installed in a 'Norton Montagebalk SMV 115 IND'. The top of the set-up is covered, to make sure the UV-C light does only go to the particle and protects the user from the UV-C radiation, since UV-C radiation is harmful for humans[19]. After being excited by the UV-C light, a picture was taken with a photo camera (iPhone 7). The used Matlab file was a file that can detect the color of a selected area of a picture and translate this color to a specific code/instrument.

4.4. Implementation

To explain the electroless nickel plating with luminescent micro-particles technique to the medical instrument professionals, a short presentation about the project was created. This presentation is the starting point of the discussion about the possible hazards of such a system.

5
Results

This chapter shows the results of the four parts of this research. The results of the experiments and the characterization are explained in section 5.1. In section 5.2 the results of the identification part are shown and finally in section 5.3 the results of the meeting with the medical instrument experts are shown.

5.1. Experiments

The results of the experimental part of this research are explained in this section. The experiments are analyzed according to the method explained for the characterization part of this research.

5.1.1. Experiment 0

Exp. 0 Analyze substrates before being nickel-plated
Goal: Set a baseline for the SEM/EDS analysis
To compare the substrates, a SEM and EDS analysis was done of a clean, 316L stainless steel substrate and a 316L stainless steel substrate after nickel strike to use as a baseline. The results are shown in Appendix G. The black parts of the pictures is the epoxy in which the substrates are embedded for analysis. The light parts are the cross-sections of the substrates.

Morphology/Chemical composition - Clean Substrate
As can be seen in Appendix G, figure G.1 number 1, 2 and 3, the substrate is very even and there are no lines near the side of the substrate. In table G.1 the elements and their present [%] are shown. The locations of the analysis are shown in picture 4 of figure G.1.

Morphology/Chemical composition - After Nickel Strike
In figure G.2 picture 1, 2, and 3, the results of the SEM analysis of the substrate after being pre-treated with a nickel strike are presented. A very small, thin layer on the side of substrate can be seen. The EDS analysis, shown in table G.2, showed that the nickel concentration in this thin layer is much higher in comparison to the data shown in table G.1. This proves that the thin layer visible in the SEM pictures is the nickel strike layer.

Conclusion Experiment 0
It is possible to identify the difference between a clean substrate and a substrate pre-treated with a nickel strike. This is of importance for the analysis of future experiments.

5.1.2. Experiment 1

Exp. 1 Check particles with UV-Lamp when dry
Goal: To see if the particles emit light when excited by UV-Light
As can be seen in Appendix H figure H.1 and H.2, the blue and red color are very clear. This proves that these particles do emit light when excited by UV-light. Furthermore, this experiment shows the obvious difference between the two particle colors. This is of importance when analyzing the different color substrates.

5.1.3. Experiment 2

Exp. 2 Mix particles in nickel bath at 20°C
*Goal:*To check if the micro particles (BAM:Eu and YO:Eu) mix with the nickel bath.
To simulate the nickel bath used for electroless nickel plating, the same concentrations of solutions are used, see Appendix I figure I.1. Both type of particles, BAM:Eu and YO:Eu, dissolved well in the nickel bath. This can be seen in figure I.2. The solution did not turn black, which would indicate a chemical unstable solution. This experiment proves that the particles are suitable for blending in to the nickel bath and therefore can be used for electroless nickel plating.

5.1.4. Experiment 3

Exp. 3 Nickel plating of 2 substrates of stainless steel 316L
Goal: To determine if a nickel strike is necessary as a pre-treatment.
Since electroless nickel plating is an auto-catalytic process, a hefty reaction should take place immediately after placing the substrates in the nickel bath. This gas formation is hydrogen evolution. This did not happen during experiment 3, as can be seen in Appendix J figure J.1 and J.2. These pictures are taken just after adding the substrates to the nickel bath. Just above and around the substrates no gas formation (H_2 evolution) can be seen.

Morphology/Chemical composition
As presented in figure J.3, number 1, 2 and 3, the SEM analysis looks very similar to the analysis of Experiment 0. The small, bright line on the side of the substrate is an effect of the SEM, this is not a different layer. The EDS analysis confirmed this, as can be seen in in table J.1. The available elements and their present percentages are similar to Exp. 0.

Conclusion Experiment 3
After performing this experiment multiple times, the conclusion was drawn that the nickel plating was not successful. This is based on the data from the SEM/EDS analysis but also on the fact that no gas formation happened (H_2 evolution) and it did not have the correct color afterwards. A solution for this can be to pre-treat the substrates with a nickel strike.

5.1.5. Experiment 4

Exp. 4 Nickel plating of 1 substrate of steel and 1 substrate of stainless steel 316L after nickel strike
Goal: To verify the composition of the nickel bath and the pre-treatment process.
Two different substrates are used for this experiment. The first substrate used for this experiment is the 316L medical grade stainless steel. The other substrate used, is a regular steel substrate, provided by AHC Benelux[1]. This substrate should react immediately, without any pre-treatment, when placed in the nickel bath. This is because steel does not have an oxide layer to protect the material. If no reaction happens to the steel plate, the conclusion can be drawn that the nickel bath composition is not correct. If a reaction happens to the steel substrate, but not to the 316L medical grade stainless steel substrate, the conclusion can be drawn that the pre-treatment, the nickel strike, is not sufficient. As can be seen in Appendix K figure K.1, there is clearly gas formation (H_2 evolution) around the substrates. This is a good indicator of a successful nickel plating.

Physical Characterization

Weight Analysis

As stated in chapter 3, the substrates are weighted before and after being nickel plated. The added weight is another good indicator of a successful nickel plating. In table 5.1 the results are shown. With the use of formulas 3.1 and 3.2 stated in Chapter 3, the theoretical thickness of the nickel coating on the 316L substrate can be calculated. During the EDS analysis it was found that the phosphorus content percentage is approx. 7% which gives a ρ of $8{,}22 g/cm^3$. Combining this with the information from table 5.1 and formula 3.1 gives a theoretical thickness of $3{,}50 \cdot e^{-6} m$ ($3{,}50 \mu m$). The calculations are in Appendix K equation K.1.

Table 5.1: Weight of substrates of exp. 4 before and after nickel plating

Substrate	Weight before NP [kg]	Weight after NP [kg]	Added Weight [kg]	Added Weight [%]
Steel	7,5681 e^{-3}	7,6235 e^{-3}	0,0554 e^{-3}	0,73202
316L Stainless steel	6,8151 e^{-3}	6,8676 e^{-3}	0,0525 e^{-3}	0,77035

Color Analysis

In figure K.2 a picture is shown with the different substrates. In this picture, the change in color is clearly visible. The color of substrate 2 and 3 are almost equal to the color of the nickel substrate in the background. This is in line with the findings from the weight analysis and the H^+ evolution during the the plating process.

Morphology/Chemical composition
As can be seen in figure K.3, numbers 1, 2 and 3, a clear new layer can be seen. This was as expected, since the substrate gained weight and there was clear gas formation in the nickel bath. It is also possible to see the nickel strike line between the nickel-phosphor layer and the original 316L SS. From picture 3, the thickness of the layer was derived and compared to the theoretical thickness, which is $3{,}50 \mu m$. The measured thickness from the SEM analysis is approx. $3{,}90 \mu m$. In tab. K.1 the elements found at the selected points of picture 4 are shown. It clearly shows the presence of the nickel-phosphor layer on the side and the iron of the 316L stainless steel substrate. The results of both points in the nickel-phosphor layer are approximately the same which indicates a uniform layer.

Conclusion Experiment 4
Since both, the steel substrate and the 316L stainless steel substrate, reacted to the nickel bath, the conclusion can be drawn that this experiment was successful. Both gained approx. the same weight percentage and the color clearly changed and matched to the provided nickel plated substrate. Also, the SEM and EDS analysis showed a clear new layer on the side which consists of the correct elements, nickel and phosphor. Although stated in chapter 3 the phosphorus content should be between 9 and 13%, this was a bit lower for this experiment (approx. 7.00%). The deposition rate should be around 10 - 14 $\mu m/h$ which actually is $3{,}90 \mu m/h$. This can be explained by the fact that the nickel-phosphor concentration declines fast in a 500ml bath. This resulted in a gradually declining reaction between the nickel bath and the substrate.

5.1.6. Experiment 5

Exp. 5 Nickel plating of 2 substrates of stainless steel 316L after nickel strike
Goal: This experiment is performed to see if the 2 substrates can be nickel plated after nickel strike
After confirming the proper nickel-bath was created, two 316L stainless steel substrates were nickel-plated. This experiment set a baseline to which experiment 6 and 7 were compared. Appendix L figure L.1 picture 1 and 2 shows the amount of H_2 evolution at the beginning and at the end. As can be seen, a hefty H_2 evolution is taking place at the beginning, but after 60 minutes, no reaction is happening anymore.

Physical Characterization

Weight Analysis

As can be seen in table 5.2, the substrates gained approx. the same weight. The EDS (table L.1) showed a phosphorus content of 13%. Using the added weight from 316L stainless steel 1 (0,0561g) and the known formulas gives a theoretical thickness of $4,01 \cdot e^{-6} m$ ($4,01 \mu m$). The calculations are in equation L.1.

Table 5.2: Weight of substrates of exp. 5 before and after nickel plating

Substrate	Weight before NP [kg]	Weight after NP [kg]	Added Weight [kg]	Added Weight [%]
316L Stainless steel 1	6,9055 e^{-3}	6,9616 e^{-3}	0,0561 e^{-3}	0,81240
316L Stainless steel 2	6,8843 e^{-3}	6,9418 e^{-3}	0,0575 e^{-3}	0,83523
Average			*0,0568* e^{-3}	*0,82382*

Color Analysis

During this experiment, the color of the substrates changed in a very uniform, metallic color. This is clearly visible in figure L.1, picture 3. The first and third substrate are the nickel-plated substrates and the second en fourth substrate are the substrates before nickel-plating. Comparing the substrates to the provided nickel-plated substrate shows also the similarity, visible in figure L.2.

Morphology/Chemical composition
The results of the Morphology and the Chemical characterization are shown in figure L.3 and table L.1. These results are very similar to the results of experiment 4, with the exemption of a higher phosphor content. According to the formulas used for calculating the theoretical thickness of the layer, a higher phosphor content should lead to a thicker layer. The calculated theoretical thickness is $4,01 \mu m$, which is already higher in comparison to experiment 4. Deriving the thickness of the layer from the SEM, results in a thickness of $4,27 \mu m$. Point 2 of the EDS analysis has different values since this point is placed approx. on the nickel strike line.

Conclusion Experiment 5
Experiment 5 started with a hefty H_2 evolution, which already indicated a successful nickel-plating process. The second indicator of a successful nickel-plating process was the gained weight and the change of color into a uniform, metallic color. The successful coating was confirmed by the SEM and EDS analysis. However, despite all these positive indicators, experiment 5 showed that even with a high phosphor content the layer does not get to the deposition rate of 10 - 14 $\mu m/h$ but stayed at approx. $4,27 \mu m/h$.

5.1.7. Experiment 6

Exp. 6 Nickel plating of 2 substrates of stainless steel 316L after nickel strike with BAM:Eu particles
Goal: To see if it is possible to add luminescent micro particles (BAM:Eu) to an electroless nickel plating process.
The pictures in Appendix M figure M.1 and figure M.2 show the set-up of experiment 6. As can be seen in picture 1 and 2 of both figures, the dispersion of the BAM:Eu particles in the nickel-bath was very even. The gas formation started immediately after adding the substrates to the nickel-bath and the intensity gradually decreased over time.

Physical Characterization

Weight Analysis

In table 5.3 the weight difference of 6 substrates are shown. The difference between these 6 is the bathing time; substrate 1 & 2 60 min; substrate 3 & 4 45 min; substrate 5 & 6 75 min. As expected substrate 3 & 4 gained less weight and substrate 5 & 6 gained more weight. The SEM and EDS analysis shown in figure M.4 and table M.1 are of substrate 5. The phosphor content of this substrate is approx. 13%, the added weight is 0,0798 $e^{-3} kg$. Adding this data to equation 3.2 and 3.1 gives a theoretical thickness of $5,64 \mu m$ in 75 min, as can be seen in equation M.1.

Table 5.3: Weight of substrates of exp. 6 before and after nickel plating

Substrate	Weight before NP [kg]	Weight after NP [kg]	Added Weight [kg]	Added Weight [%]
316L Stainless steel 1	6,9007 e^{-3}	6,9603 e^{-3}	0,0596 e^{-3}	0,86368
316L Stainless steel 2	6,8908 e^{-3}	6,9515 e^{-3}	0,0607 e^{-3}	0,88088
316L Stainless steel 3	6,8219 e^{-3}	6,8613 e^{-3}	0,0394 e^{-3}	0,57755
316L Stainless steel 4	6,8621 e^{-3}	6,9016 e^{-3}	0,0395 e^{-3}	0,57763
316L Stainless steel 5	6,8275 e^{-3}	6,9037 e^{-3}	0,0798 e^{-3}	1,16880
316L Stainless steel 6	6,7877 e^{-3}	6,8680 e^{-3}	0,0803 e^{-3}	1,18302
Average			*0,0597* e^{-3}	*0,87526*

Color Analysis

As presented in figure M.1 picture 3 & 4 and figure M.2 pictures 3 & 4 the color changed to the uniform, metallic color as expected. Unlike experiment 5, this color change only was visible on one side. The other side was uniformly colored, but instead of a metallic color it was matte, as can be seen in figure M.3.

Morphology/Chemical composition

Since both sides have a different color, the SEM/EDS analysis was performed on both sides and on the surface of the substrate. The atom-percentages discovered via the EDS analysis were compared to equation 5.1. The ratio of the atom-percentages of the BAM:Eu particles were derived from this equation and were added to the table.

$$Ba_{0,86}Mg_1Al_{10}O_{16} : Eu_{0,14} \tag{5.1}$$

Particle Side

In Appendix M figure M.4 and table M.1 the pictures of the SEM analysis and the EDS results are presented. The derived thickness from the SEM analysis was $6,20 \mu m$ in 75min. The BAM:Eu particles were clearly visible in the nickel-phosphor layer. The selected points for EDS analysis validated this result. Point 1, 2, 3 and 4 are the points on the BAM:Eu particles, as can be seen in table M.1. The ratio of the elements of the BAM:Eu particles were approx. the same as the ratio derived from equation 5.1.

Surface

The pictures of the SEM analysis of the surface of the 316L substrate coated with BAM:Eu particles are shown in figure M.5. In these pictures the particles are clearly visible and were identified via EDS (table M.3). Points 2, 3 and 5 are identified as the BAM:Eu particles and in point 1 and 4 are the nickel-phosphor layer visible. Some of the particles are protruding outside of the nickel-phosphor layer as can be seen in the pictures. The reason for this could be the thickness of the nickel-phosphor layer ($6,20 \mu m$) which is smaller than the particle size ($5-9 \mu m$ figure C.1).

No Particle Side

Since the color was different on each side of the substrate, the other side of the cross-section was analyzed as well. The SEM and EDS analysis are shown in figure M.6 and table M.4. As can be seen in the SEM pictures, a clear nickel-phosphor layer and nickel-strike line is visible. Meanwhile, no BAM:Eu particles are visible. This was confirmed by the EDS analysis which only detected nickel and phosphor elements on the outside layer.

Conclusion Experiment 6

Experiment 6 proved that it was possible to coat a 316L stainless steel substrate with a nickel coating with embedded BAM:Eu particles. The deposition rate calculated from the thickness derived from the SEM analysis was 4,96 $\mu m/h$ ($6,20 \cdot \frac{4}{5}$), which was still less than 10-14 $\mu m/h$, despite of the phosphor content being around 13%. The fact that only one side of the substrate was coated can be because of the rotation of the stirrer with respect to the orientation of the substrates in the nickel bath. As a result of this, the shear stress between the particles and the substrate was different on each side. The side with the lower shear stress was coated with BAM:Eu particles since it has more time to 'grasp' onto the nickel-phosphor coating.

5.1.8. Experiment 7

Exp. 7 Nickel plating of 2 substrates of stainless steel 316L after nickel strike with YO:Eu particles
Goal: To see if it is possible to add luminescent micro particles (YO:Eu) to an electroless nickel plating process.
In Appendix N figure N.1 and figure N.2 pictures of experiment 7 are shown. The first observation of experiment 7 was after adding the YO:Eu particles to the nickel-bath. The nickel-bath was more cloudy compared to the nickel-bath with the BAM:Eu particles. During the 60 minutes of experiment 7 the cloudy nickel-bath changed. At the end of the experiment the top of the nickel bath was almost clear and the bottom got even more cloudy. This is visible when comparing picture 1 of figure (N.1 to picture 2 of figure N.2. In picture 1, the substrate is completely invisible, in contrast to picture 2 the substrate is almost completely visible. At the end of experiment 7, some particles agglomerated together and floated on top of the nickel-bath. Besides these observations, after adding the substrates to the nickel-bath less gas formation was visible in comparison to the previous experiments.

Physical Characterization

Weight Analysis

In table 5.4 the weight results of experiment 7 are presented. All the substrates stayed in the nickel bath for 60min. As expected, the substrates gained less weight compared to experiment 6, since less gas evolution was observed during the experiment. The SEM and EDS shown in figure N.4 and table N.1 are of substrate 4. The phosphor content of this substrate was approx. 12%, the added weight 0,0214 $e^{-3}kg$. This results in a theoretical thickness of 1,51 μm (see equation N.1), which is indeed a thinner layer compared to the previous experiments.

Table 5.4: Weight of substrates of exp. 7 before and after nickel plating

Substrate	Weight before NP [kg]	Weight after NP [kg]	Added Weight [kg]	Added Weight [%]
316L Stainless steel 1	6,8200 e^{-3}	6,8360 e^{-3}	0,0160 e^{-3}	0,23460
316L Stainless steel 2	6,8824 e^{-3}	6,8983 e^{-3}	0,0159 e^{-3}	0,23102
316L Stainless steel 3	6,8009 e^{-3}	6,8219 e^{-3}	0,0210 e^{-3}	0,30878
316L Stainless steel 4	6,9120 e^{-3}	6,9334 e^{-3}	0,0214 e^{-3}	0,30961
316L Stainless steel 5	6,8036 e^{-3}	6,8204 e^{-3}	0,0168 e^{-3}	0,24693
316L Stainless steel 6	6,7954 e^{-3}	6,8129 e^{-3}	0,0175 e^{-3}	0,25753
Average			*0,0181* e^{-3}	*0,26475*

Color Analysis

Comparing the change in color of the substrates after being taken out of the nickel bath, the nickel-phosphor coating is clearly visible. The substrates had a very uniform, metallic appearance when compared to the substrate with the nickel-strike (figure N.1 and figure N.2). The difference between experiment 6 and 7 was clearly visible when comparing the substrate to the provided nickel sample. As can be seen in figure N.3, the substrates have the uniform, metallic appearance on both sides and is not matte on 1 side.

Morphology/Chemical composition
In figure N.4 and table N.1 the SEM and EDS results are shown. A clearly thinner nickel-phosphor layer is visible without the presence of any YO:Eu particles. This is the case on both sides of the substrates. The thickness derived from the SEM analysis is 1,66 μm. The EDS analysis confirmed the absence of the YO:Eu particles in the nickel-phosphor layer since it only detected nickel, phosphor and iron. The ratio of the elements of the YO:Eu particles should have been the following:

$$Y_{1,92}O_3 : Eu^{3+}_{0,08} \tag{5.2}$$

Conclusion Experiment 7
Using YO:Eu particles to coat 316L stainless steel substrates via electroless nickel plating was not successful. No particles were embedded inside the nickel-phosphor coating. Furthermore, the deposition rate of this experiment was only 1,51 $\mu m/h$. A conclusion can be drawn that YO:Eu is not suitable for this technique. A reason for this can be that $Y_2O_3 : Eu$ is a hygroscopic powder[9]. This means it can easily absorb water. When the particle absorbs water, they gain in size. This makes it harder for the particle to be embedded into

the nickel-phosphor coating. Another reason for a non-successful coating is the fact that the YO:Eu particles agglomerated on the top of the nickel-bath as can be seen in figure N.2.

5.1.9. Experiment 8

Exp. 8 Nickel plating of 2 substrates of stainless steel 316L after nickel strike with BAM:Eu particles at the top half and YO:Eu particles at the bottom half
Goal: To see if it is possible to add 2 luminescent micro particles (BAM:Eu & YO:Eu) to the substrate (316L stainless steel after nickel strike) creating a multi color code (top blue, bottom red)
As can be seen in Appendix O figure O.1, the multi color code was created by masking. Firstly the bottom half was masked with Teflon tape and the top was coated with BAM:Eu particles (picture 1). After removing the substrate from the BAM:Eu nickel-bath, the substrate was masked on the top half and added to the YO:Eu nickel bath (picture 2). The results of this experiment were similar to the results of Exp. 6 and Exp. 7. Hefty H_2 evolution was observed during the BAM:Eu nickel plating part, and barely any H_2 evolution was observed during the YO:Eu nickel plating part.

Physical Characterization

Weight Analysis

Similar results as in Exp. 6 and Exp. 7 can be seen in table 5.5. During the first nickel-plating process with the BAM:Eu particles, the substrate gained 0,8604% weight. This is a similar number found during Exp. 6. The second nickel plating process with the YO:Eu was less successful and resulted in an added weight percentage of 0,1068%. In figure O.3 and table O.1 the results of the SEM and EDS of substrate 12 are shown. The phosphor content of this experiment was approx. 9% and the A of this experiment was $\frac{18,25}{2}$, since only half the substrate was coated each time. The theoretical thickness of the BAM:Eu side was 4,04μm (equation O.1) and the theoretical thickness of the YO:Eu side was 0,48μm (equation O.3).

Table 5.5: Weight of substrates exp.8 before and after nickel plating

	316 Stainless Steel 1	316 Stainless Steel 2
Weight before NP [kg]	6,9039 e^{-3}	6,8447 e^{-3}
Weight after NP with BAM [kg] *Including holderstick*	12,8172 e^{-3}	13,2876 e^{-3}
Weight after NP with YO [kg] *Including holderstick*	12,8209 e^{-3}	13,2911 e^{-3}
Weight after NP with YO [kg] *Excluding holderstick*	6,9373 e^{-3}	6,8778 e^{-3}
Holderstick	5,8836 e^{-3}	6,4133 e^{-3}
Weight after NP with BAM [kg] *Excluding holderstick*	6,9336 e^{-3}	6,8743 e^{-3}
Added weight after BAM [kg]	0,0297 e^{-3}	0,0296 e^{-3}
Added weight after YO [kg]	0,0037 e^{-3}	0,0035 e^{-3}
Added weight BAM [%]	0,8604	0,8650
Added weight YO [%]	0,1068	0,1018

Color Analysis

The color analysis of Exp. 8 was similar to Exp. 6 and Exp. 7 as well. As can be seen in figure O.2 the top half (BAM:Eu particles) has a more matte finish in comparison to the bottom half (YO:Eu particles) which is more metallic. The line which divides the BAM:Eu part and the YO:Eu part is clearly visible.

Morphology/Chemical composition

The SEM results are presented in figure O.3. The presence of the BAM:Eu particles are clearly visible in picture 2 & 3 as well as the nickel-phosphor coating. This is not the case for the YO:Eu particles. Similar to Exp. 7, the particles were not embedded into to nickel-phosphor coating. The nickel-phosphor coating was also much thinner in comparison to the BAM:Eu part. The thickness derived from the SEM analysis for the BAM:Eu side was 3,87μm and from the YO:Eu side was 0,59μm. The presence of the BAM:Eu particles and the absence of the YO:Eu particles is also confirmed by the EDS analysis shown in table O.1.

Conclusion Experiment 8

The results of Exp. 8 are a summation of Exp. 6 and Exp. 7. Coating the 316L stainless steel substrate with BAM:Eu particles can be done, but the YO:Eu particles do not embed in the nickel-phosphor layer. The deposition rate of the BAM:Eu particle coating is 3,87$\mu m/h$ and of the YO:Eu particle coating is 0,59$\mu m/h$. Both

are much lower than the theoretical deposition rate of 10-14$\mu m/h$. This experiment showed that it is possible to mask the substrate to only coat certain parts of the substrates, but the fact that YO:Eu is not suitable for this coating made it not possible to create a multi color code.

5.2. Identification

This section shows the results of the identification part of the results. This is the part in which the coated substrates are analyzed via the UV-C light and are being identified via a Matlab file.

5.2.1. Setup

As stated in Chapter 3 the setup for the identification consist out of the UV-light, a camera and a Matlab file. The setup of the identification part is presented in Appendix P figure P.1. Since excessive exposure to UV-C light is harmful, a semi-closed setup is built. After placing the substrates inside the setup, the front plate can be lowered. This prevents direct exposure of the UV-light but still makes it possible to evaluate the substrates. Appendix C figure C.1 states a *UV Excitation Source* between 440-460 nm for the BAM:Eu particle. This converts to a 'RGB' code of $0;0;255-0;102;255$. For the YO:Eu particle the *Emission Peak* is between 609 - 6013 nm, which is in RGB $255;141;0-255;125;0$. These RGB codes are needed for the Matlab file.

5.2.2. Matlab file

The Matlab file used for the identification of the substrates consists out of the following parts:

1. Select picture of substrate when excited by UV-light
2. Select part of picture to use for identification
3. Acknowledge selection
4. Create mean of color of selection
5. Check if new selection is Red, Green or Blue
6. Convert color to name of substrate

The used Matlab code is added in Appendix P.2.

5.2.3. Results

BAM:Eu Coating

In Appendix P figure P.2 the pictures of the excited substrates are shown. The blue coating is clearly visible in pictures 1, 3, 5 and 7. The non-excited substrates are shown in pictures 2, 4, 6 and 8. When observing the excited substrates, some differences can be noticed. The substrates of picture 3 are not as 'blue' as the substrates shown in picture 1 and 5. This can be explained by looking at the thickness of the layers (table 5.6). The substrates in picture 3 have a much thinner layer in comparison to the substrates of picture 1 and 5, which explains the less 'blue' coating.

Table 5.6: Thickness Substrates Exp. 6

Picture	Thickness Left Substrate [μm]	Thickness Right Substrate [μm]
1	4,29	4,14
3	2,66	2,66
5	6,20	5,82

YO:Eu Coating

During the analysis of Exp. 7, the SEM and EDS showed no presence of the YO:Eu particles. Analyzing these substrates with the UV-light gave a similar result. No change in color occurred when excited by UV-light, as can be seen in figure P.3. The color of excited YO:Eu particles is shown in picture 4 of figure P.3.

5.3. Implementation

The hazards of this technique are discussed with two medical instruments experts. The results are shown in this section. During the meeting with Dr. Ir. Van der Eijk and Drs. Jungblut multiple subjects are discussed. The subjects are divided into 2 categories;

1. Change in material properties
2. Cleaning, disinfecting and sterilizing

5.3.1. Change in material properties

The subjects discussed in this category are focused on the change in material properties. The material properties of the nickel-phosphor coating and the 316L medical grade stainless steel are summed up in table 5.7. A property with a big difference is the hardness. The nickel-phosphor coating is harder in comparison to the 316L, this means it is more prone to damage other instruments. The most important difference is the presence of other elements. The elements present in the coating need to be *'biocompatible'*. This means that the coating much have the quality of not having toxic or injurious effects on the biological system[10]. Since some of the material properties are changed after being coated, the durability of the coating needs to be analyzed. The current lifespan of instruments can be up to 30 years, the durability of this coating needs have a similar lifespan.

Table 5.7: Properties of nickel-phosphor coating and 316L

Property	Value Coating	Value 316L	
Hardness	570 - 1000	222	$[HV]$
E-Modulus	170 - 200	170	$[kN/mm^2]$
Density	7900 - 8200	8000	$[kg/m^3]$
Melting point	865 - 900	1375 - 1400	$[K]$
Heat Conductance	0,04	0,016	$[W/cm \cdot °C]$
Phosphorous Content	9 - 13	0,045	[%]
Nickel Content	87 - 91	10 - 14,0	[%]

5.3.2. Cleaning, disinfecting and sterilizing (CDS)

Discussed in this category are subjects related to the CDS phase in the CSSU. The pictures of the SEM analysis shows that it seems that the particles stick out of the coating. This can test the durability of the coating, especially when the coating goes through the CDS phase regularly and is exposed to steam and hydrogen peroxide. Another concern from the expert was the multi angular shape of the particles (as can be seen in the SEM images). The experts are concerned about the ability to clean these particles. For example, a norovirus is 27-35nm [30], while the BAM:Eu particles can be up to 9μm (9000nm) big. The experts fear that the gaps between the particles are hard to sterilize.

6

Discussion

In this thesis the technique of identifying medical instruments via a luminescent micro-particle coating as an alternative for the existing identification techniques is researched. The coatings were added to the 316L substrates by electroless nickel plating. The performed experiments build up to the final result, which is a successfully coated substrate. This answers the main question; it is possible to coat individual medical instruments with a coating via electroless nickel plating, which includes luminescent micro-particles and identify these instruments. At least, it is possible to coat a 316L medical grade stainless steel substrate with BAM:Eu particles on 1 side and excite and identify this substrate. This result proves the feasibility of using this technique for identifying medical instruments.

Next to the answer of the main question, additional insights are found. Specific pre-treatment (nickel strike) is necessary for 316L medical grade stainless steel. Furthermore, BAM:Eu is a good particle to use for this technique, when excited by a UV-C light, it is clearly different from a uncoated substrate. YO:Eu particle did not work for this experiment, reasons for this are explained in the results (chapter 5). A successful YO:Eu coating might be created after adapting the electroless nickel plating process.

In the introduction the disadvantages of the use of RFID and barcode scanning as a medical instruments identification technique are stated. For RFID the biggest disadvantage is the fact that a tag needs to be added onto the instrument. This tag could disturb the medical professional with their work. An instrument coated with the luminescent micro-particle coating is only 10 μm thicker and gains approx. 1% in weight. This will be unnoticeable for the medical professional. Furthermore, the entire instrument can be coated, which makes it possible to identify the instrument at any random point. This is a big advantage over barcode scanning, which requires a clear line of sight to one specif point. Furthermore, barcodes are added (sticker) or engraved into the instrument. As stated in the introduction, barcode stickers can be removed and human material can stay in the engraved barcode slots. The luminescent particle code will not be removed by the sterilizing process and there are no engraved slots for human material to stay behind. On the other hand, the SEM analysis showed the possibility that the particle sticks out of the coating. The possibility that bacteria could stay behind on the coating should be further researched.

Another disadvantage of RFID is the electro magnetic interference (EMI). Different opinions are found in literature about this subject. The luminescent micro-particle coating does not use this technique, so no EMI can occur. On the other hand, this technique uses a nickel based coating. Nickel allergy has a prevalence of 17% for women and 3% for men[25]. Nickel allergy is a type-4 allergy, a delayed-type-hypersensitivity to nickel, which means long exposure times are necessary to the nickel to set off an allergic reaction. Besides, there is also nickel in 316L medical grade stainless steel, about 10-14%. However, this should still be further researched what the influence of the nickel would be when used during a surgery operation.

To excite the luminescent micro-particles, a UV-C light is needed. UV-C light is harmful to the human as discussed in 3. This limit the excitation locations of the instruments, since it is not possible to identify instruments near the human body. This is a weakness of this technique which limits the possibilities of this technique in comparison to RFID and barcode scanning. Similar to barcode scanning, the particles need to be excited by a light source with a clear line of sight. But the difference and the advantage of the luminescent particle coating is that it can be scanned anywhere on the instrument in comparison to on just 1 barcode location. Moreover, barcode scanning and the luminescent particle coating needs to be read one by one, whereas RFID tags can be read simultaneously. For example, all the instruments in a surgical instrument kit

can be read at once with the use of RFID tags and a reader to see if all the instruments are in the kit, and for barcode scanning and the luminescent particle coating the instruments need to be scanned one by one.

To identify multiple instruments, different UDI codes need to be created. This can be done in multiple ways. A color coded barcode could be created by masking the instruments. Masking is a very labor-intensive method but makes it possible to put the instrument in different color baths. An alternative is to create different gradients of luminescent colors. This can be achieved by adding different percentages colors to the nickel-bath. A consequence of this technique is that for every code a new nickel-bath is necessary. Another disadvantage of this technique is that the color needs to be uniformly distributed in the coating. This is necessary to make the instrument readably at any point of the instrument. The advantage of this technique is that every instrument can be put in a nickel-bath without the use of masking. A combination of both techniques is also possible, creating a not uniformly distributed gradient of color nickel bath, and only coating a specific surface area with this coating. This will generate a unique combination of colors which can be used as a UDI code.

This research was limited by the quality of the coating. The deposition rate of 10 - 14 $\mu m/h$ was never obtained. Improving this can result in a better and more evenly dispersion of the particles and the nickel-phosphor coating. It is possible that the addition of the particles to the nickel-bath could have limited the deposition rate. The electroless nickel plating process is not developed for the purpose to add particles to the substrate. In line with this, another limitation of this research is the use of only 2 particles (small sample size). BAM:Eu and YO:Eu particles showed a complete different result. Performing the same experiment with different luminescent micro particles can influence the result enormously. Another part that could have been of influence on the deposition rate is the size of the nickel-bath. The concentration of the nickel and phosphor in the nickel-bath decreases during the process. Although the maximum bath loading was not exceeded, it was close to the maximum. Another factor which could have limited the nickel-phosphor deposition rate is the nickel-strike. It was not possible to perform the nickel-strike at the TU Delft and had to be done by experts at AHC Benelux at Eindhoven. The best nickel-phosphor deposition rate can be obtained immediately after the nickel-strike. Furthermore, the modification of the DNC-520 process to the process used for the experiments could be optimized. This is discussed in chapter 8.

The starting point of this research was to develop an alternative for the existing techniques available to use as a medical asset management system. The results of this thesis proves the ability of this technique to be used for a medical instruments identification system. Combining electroless nickel plating with luminescent micro-particles is not an established technique and should be further developed. To be worthwhile continuing researching this disruptive technique, it must have the potential to be a superior system in comparison to the current systems, RFID and barcode scanning. Right now, it is hard to compare the techniques since identifying medical instruments via a luminescent particles coating is in a different phase comparing to RFID and barcode scanning. The fact that, with the limitation stated above, it still was possible to coat, characterize, excite and identify the substrates makes me believe that this technique can be used for identifying medical instruments. The particles used for such a coating does not have to be luminescent micro-particles. Alternative particles need to be further researched.

7

Conclusion

Tracking and tracing medical instruments throughout a hospital can gain very useful information. That is way agencies such as FDA and IMDRF are pushing for such a system. The data generated by such a system can improve efficiency and patient safety. An even more important reason for such a system is that it can help lowering the costs of healthcare by optimizing the use of medical instruments. The current available techniques (RFID and Barcode scanning) for tracking and tracing instruments throughout a hospital do not suffice. These techniques are not specifically designed for medical instruments. A new technique needs to be designed to create track and trace system for individual medical instruments.

This research proved that it is possible to create a luminescent coating with the use of micro-particles. It is also possible to excite these particles and automatically identify them. The selected luminescent micro-particles (BAM:Eu and YO:Eu) do emit light when excited by a UV-C light. The nickel-bath remained chemically stable after the micro-particles were added to it. As expected, it was not possible to directly coat the substrates with the nickel-phosphor coating. The substrates needed to be pre-treated with a nickel strike and needed to be activated with HCl. After this pre-treatment it was possible to embed a luminescent coating into the substrate with the use of BAM:Eu particles. On the contrary, this did not work with the YO:Eu particles. YO:Eu particles are hygroscopic, which means they attract and hold water molecules. During the characterization of the substrates, it was possible to identify the nickel-strike layer, the nickel-phosphor layer and the BAM:Eu particles. The visual identification of these layers was confirmed by the EDS analysis. Comparing the thickness of the nickel-phosphor layer of a substrate with and without the BAM:Eu particles showed no clear difference. The substrate coated with the BAM:Eu particle clearly changed color when excited with a UV-light. It was possible to identify the difference between the particles with the created Matlab code. The substrate which supposed to be coated with YO:Eu was replaced with pure YO:Eu powder to evaluate the Matlab code. If this technique would be implemented in a hospital, an analysis of the changed properties of the material need to be performed to see if any harmful material is present in the coating. It should also be analyzed what the lifespan is of the coating when the instruments are going through the sterilization process regularly. More recommendations for future research are discussed in chapter 8.

8

Recommendations

In this chapter, recommendations for future research are explained.

1. Optimizing the nickel-plating setup
   The nickel-plating setup is derived from the DNC-520 instructions. It was decided to use a 500ml bath with 2 substrates, to minimize the chemical waste. According to the DNC-520 a surface area of a maximum of 2 substrates could be added to the nickel-bath. Future research should use higher nickel-bath to substrate ratio. This could increase the thickness of the coating. Next to the nickel-bath, a change to the setup could also improve the results. By rotation the substrate (approx. 3 rpm) through the nickel-bath, an equal sheer stress could be created on both sides of the substrate. This could help with adding the coating to both sides of the substrate. Another adjustment to the setup could help the dispersion of the micro-particles throughout the nickel-bath. Different sizes of the stirrer bar could be researched in combination with different speeds. An alternative for this is to create a setup which transports the particles from the bottom of the nickel-bath to the top of the nickel-bath and let gravity bring them back to the bottom.

2. Different kind of luminescent micro particles
   Since the results of this researched showed the difference between BAM:Eu and YO:Eu particles, it is possible that when using different luminescent particles (see Appendix B, they react differently to the nickel bath. Future research could also include combinations of particles in one nickel-bath.

3. Change in material properties
   The effect of the nickel-coating on medical instruments needs to be further research. How well is the nickel-coating resistant to the sterilization process. How does this influence the life-span of the instrument.

4. Creating different UDI code (gradient, UDI on entire instrument, color barcode, masking)
   Future research could include a research about different techniques for creating UDI codes. This research only tried to coat the entire substrate into one color, but different techniques are needed to create multiple UDI codes. Possible techniques to do this are discussed in the discussion (chapter 6).

A

Letter of minister of health

Figure A.1: Letter of minister of health

Ministerie van Volksgezondheid,
Welzijn en Sport

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www.rijksoverheid.nl

Kenmerk
1153699-165420-GMT

Bijlage(n)
1

Uw brief
-

Correspondentie uitsluitend richten aan het retouradres met vermelding van de datum en het kenmerk van deze brief.

Datum
Betreft Afspraken eenduidige codering medische hulpmiddelen

Geachte voorzitter,

Hierbij stuur ik u ter informatie het afsprakendocument eenduidige codering medische hulpmiddelen (ADC). Dit document is vandaag ondertekend door verschillende partijen in de zorg: Nederlandse Vereniging van Ziekenhuizen (NVZ), Nederlandse Federatie van Universitair Medische Centra (Nfu), Zelfstandige klinieken Nederland (ZKN), Federatie van Technologiebranches (FHI), Ondernemersorganisatie voor de technologische industrie (FME) en de belangenorganisatie van producenten, importeurs en handelaren van medische hulpmiddelen, Nefemed. Daarmee is de hele keten betrokken van productie tot gebruik bij de patiënt.
Eerder heb ik partijen gevraagd om deze afspraken te maken. Ik ben verheugd dat het is gelukt . Dat is goed voor de patiënt en veiligheid in de zorg in Nederland. Deze codering maakt elektronisch scannen mogelijk zodat altijd duidelijk is bij welke patiënt welk medisch hulpmiddel is gebruikt. Dat is belangrijk omdat daarmee de patiëntveiligheid verbetert.
Nu komt het nog voor dat er geen gestandaardiseerde code, of helemaal geen code op een medisch hulpmiddel staat, daardoor kan er niet goed gescand worden. De nieuwe afspraken sluiten goed aan op de registratie in het Landelijk Implantaten Register en lopen vooruit op de nieuwe Europese regels voor medische hulpmiddelen.
De invoering van de eenduidige codering gebeurt gefaseerd, uitgangspunt is de lijst van implantaten die ook gehanteerd wordt voor het Landelijk Implantaten Register. De ingangsdatum is 1 juli 2018; vanaf september 2018 wordt de lijst verder uitgebreid. Het afsprakendocument met voorbeelden uit de praktijk wordt gepubliceerd op www.vmszorg.nl.

Hoogachtend,

de minister van Volksgezondheid,
Welzijn en Sport,

mw. drs. E.I. Schippers

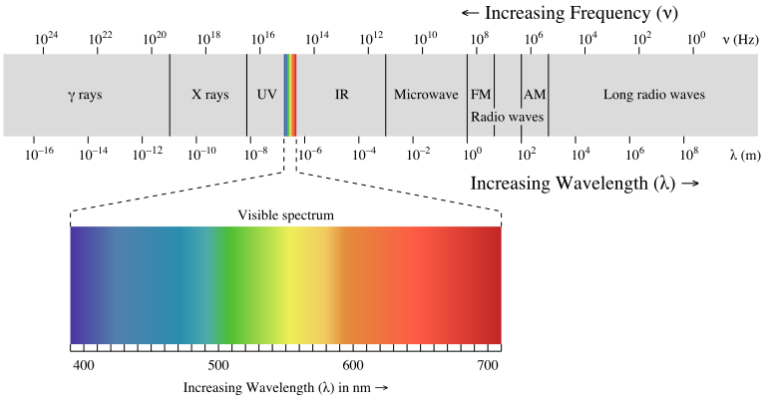
B

List of particles

Table B.1: List of luminescent particles

Name	Chemical Code	Color
Calcium-Halophosphate	$CaPo_4$	White-emitting
Calcium Tungstate	$CaWO_4$	Blue-emitting
Calcium Tungstate, Europium doped	$CaWO_4 : Eu^{3+}$	Blue-emitting
Barium Magnesium Aluminate, Europium doped	$BaMgAl_{10}O_{16} : Eu^{2+}$	Blue-emitting
Europium-oxide	Eu_2O_3	Red-emitting
Yttrium-oxide, Europium doped	$Y_2O_3 : Eu^{3+}$	Red-emitting
Gadolinium Oxysulfide, Terbium doped	$Gd_2O_2S : Tb^{3+}$	Green-emitting
Yttrium Aluminium Garnet, Cerium doped	$Y_3Al_5O_12 : Ce^{3+}$	Yellow-emitting

Figure B.1: Color spectrum



C

Characteristics of particles

Figure C.1: BAM:Eu characteristics

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Website: www.sigmaaldrich.com
Email USA: techserv@sial.com
Outside USA: eurtechserv@sial.com

Product Specification

Product Name:
Barium magnesium aluminate, europium doped - 99% trace metals basis

Product Number: **756512**
CAS Number: 724792-82-9

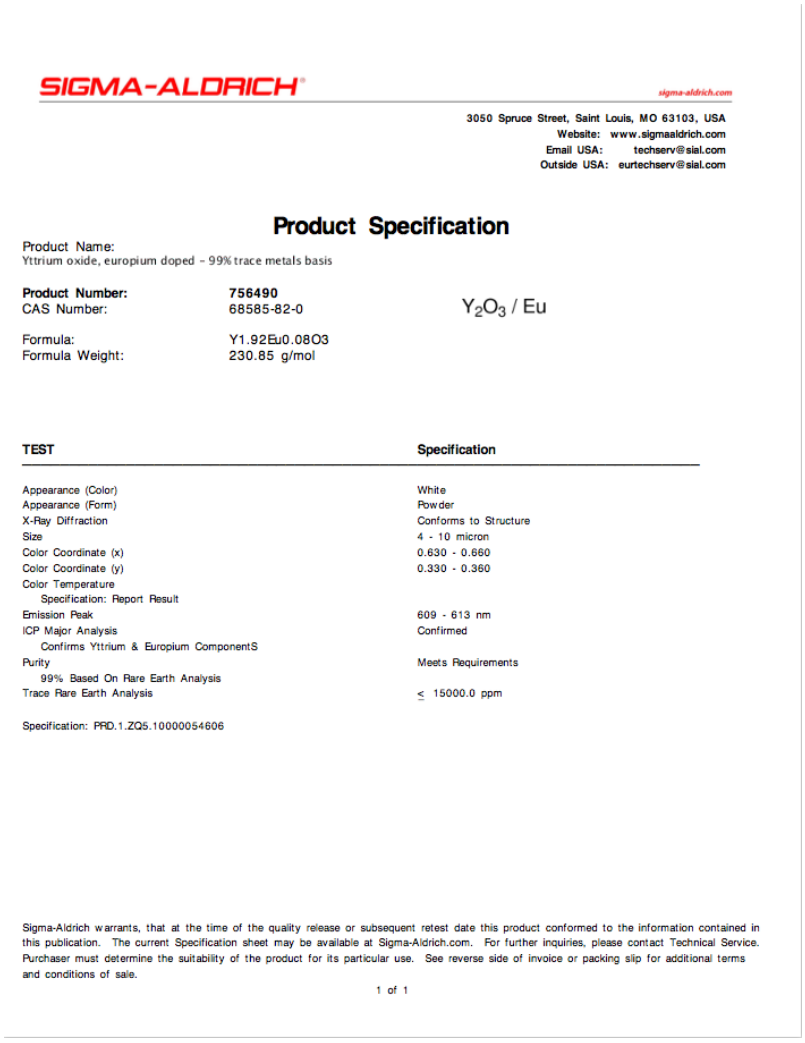
$BaMgAl_{10}O_{17}:Eu$

Formula: Ba0.86Eu0.14MgAl10O17
Formula Weight: 705.49 g/mol

TEST	Specification
Appearance (Color)	White to Light Yellow
Appearance (Form)	Powder
X-Ray Diffraction	Conforms to Structure
Size (d50)	5 - 9 micron
Color Coordinate (x)	0.125 - 0.155
Color Coordinate (y)	0.040 - 0.060
UV Excitation Source	440 - 460 nm
ICP Major Analysis Confirms Ba, Mg, Al, Eu Components	Confirmed
Purity 99% Based On Trace Metals Analysis	Meets Requirements
Trace Metal Analysis	≤ 20000.0 ppm

Specification: PRD.0.ZQ5.10000050304

Figure C.2: YO:Eu characteristics



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3050 Spruce Street, Saint Louis, MO 63103, USA
Website: www.sigmaaldrich.com
Email USA: techserv@sial.com
Outside USA: eurtechserv@sial.com

Product Specification

Product Name:
Yttrium oxide, europium doped - 99% trace metals basis

Product Number: **756490**
CAS Number: 68585-82-0

Y_2O_3 / Eu

Formula: Y1.92Eu0.08O3
Formula Weight: 230.85 g/mol

TEST	Specification
Appearance (Color)	White
Appearance (Form)	Powder
X-Ray Diffraction	Conforms to Structure
Size	4 - 10 micron
Color Coordinate (x)	0.630 - 0.660
Color Coordinate (y)	0.330 - 0.360
Color Temperature	
Specification: Report Result	
Emission Peak	609 - 613 nm
ICP Major Analysis	Confirmed
Confirms Yttrium & Europium ComponentS	
Purity	Meets Requirements
99% Based On Rare Earth Analysis	
Trace Rare Earth Analysis	≤ 15000.0 ppm

Specification: PRD.1.ZQ5.10000054606

D

Setup

Figure D.1: Experimental set-up - schematic

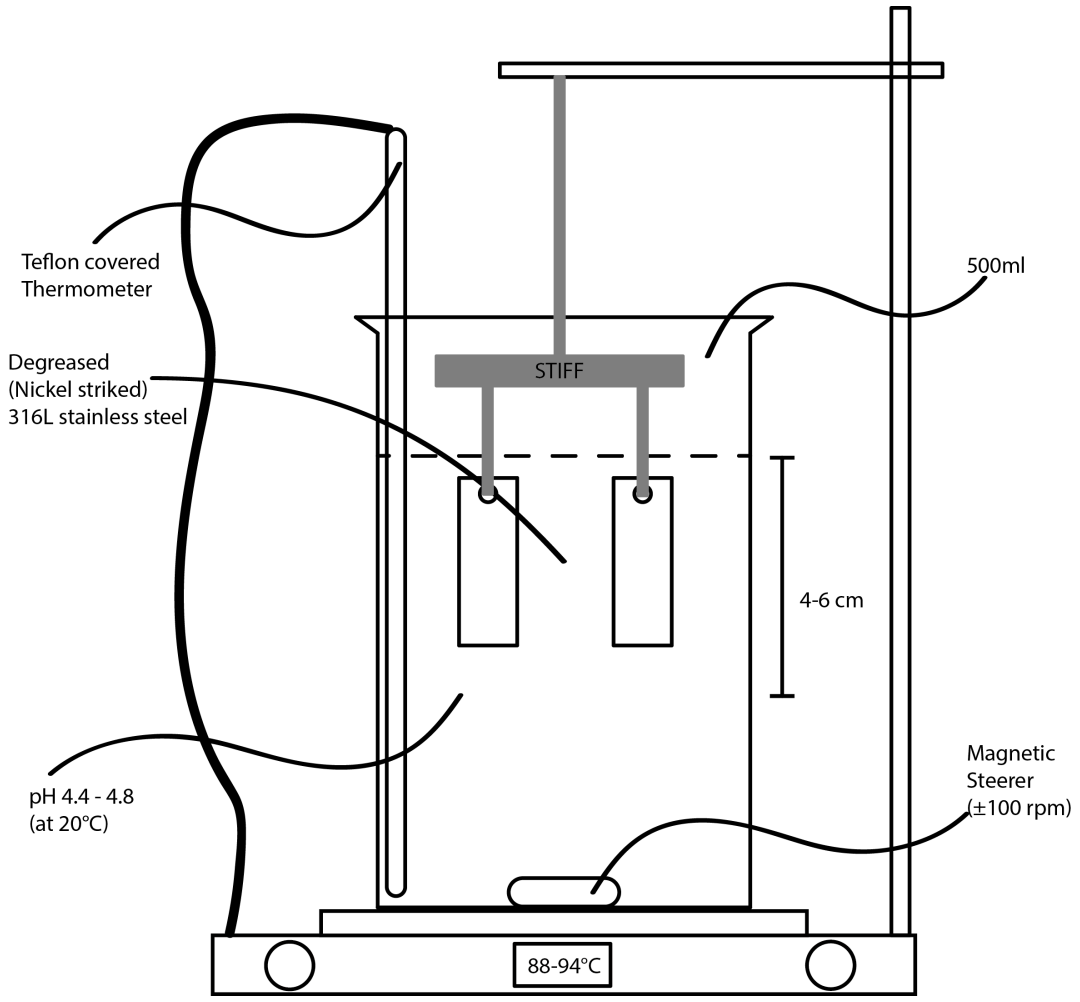
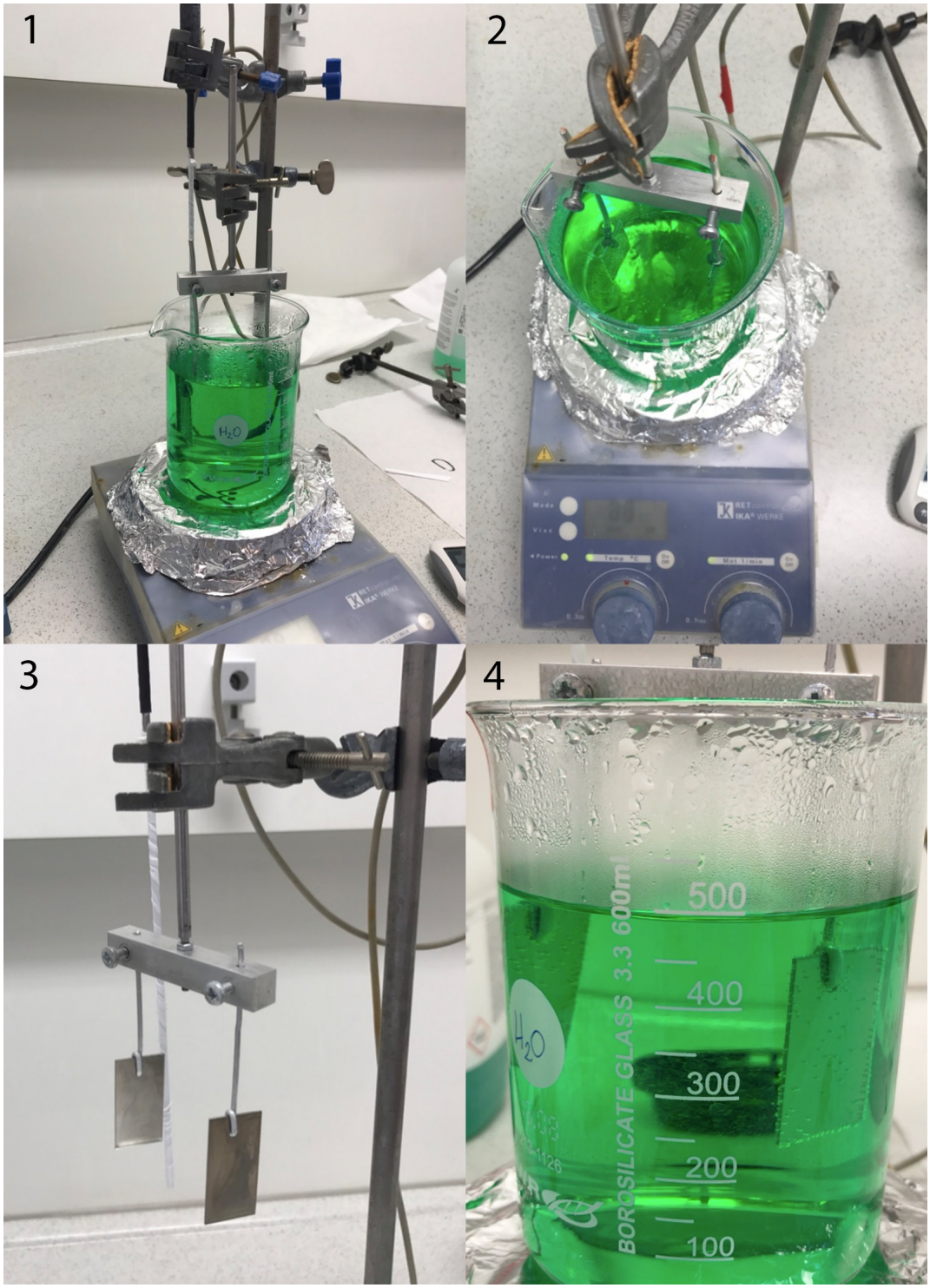


Figure D.2: Pictures of set-up:
1) Front view from setup, with heater/stirrer, teflon covered probe, substrate holder;
2) Top view from set-up;
3) Substrate holder with 2 substrates;
4) Front view of substrate in nickel bath



E

Documentation nickel plating

Figure E.1: Datasheet experiment 6

Exp. 6: Electroless nickel plating of 2 substrates of SS316L after nickel strike with BAM:Eu particles

Goal

To see if it is possible to add luminescent micro particles (BAM:Eu) to an electroless nickel plating process.

Materials – Non chemical

- Heater/MS
- Stirrer
- Beaker 500 ml 2x
- Beaker 200 ml 3x
- Graduated cylinder 50 ml 3x
- Beaker 100 ml 5x
- Syringe 20 ml 1x
- Teflon
- pH meter
- Scale
- Substrate (SS316L after nickel strike)
- Holder for substrate
- Jerrycan for disposal
- De-ionized water in spraybottle

Materials – Chemical

- BAM:Eu
- De-ionized water
- Soultion-1
- Replenisher-1
- Ammonia (solution 10)
- Acetone
- Ethanol
- 30% HCl

Preparation

- Wear gloves/glasses
- Clean beakers
- Cover probe in Teflon
- Weigh substrates
- Create label for jerrycan
  Write label with:
  - Nickel 5g/L
  - H_2PO_3 10g/L
  - pH 4-5
  - $BaMgAl_{10}O_{17}$:EU 2g/L
- Clean substrate
  - Wipe substrate clean and dry with paper
  - Place 5 beakers of 100ml in a row
  - Fill beaker 1 with: Acetone
  - Fill beaker 2 with: Demi water
  - Fill beaker 3 with: Ethanol
  - Fill beaker 4 with: Demi water
  - Fill beaker 5 with: 30% HCl
  - Fill beaker 6 with Demi water
  - Dip substrate 1 min in beaker 1
  - Rinse in beaker 2
  - Dip substrate 1 min in beaker 3
  - Rinse in beaker 4
  - Dip substrate 1 min in beaker 5
  - Rinse in beaker 6
  - Let dry in holder
- Measure volumes
  - 200ml de-ionized water
  - 90ml Solution-1
  - 21ml Replenisher-1
  - 100ml de-ionized water
  - ±50ml Ammonia (Solution-10) (after check pH)
  - x ml de-ionized water (x = 500 - 411 - solution-10)

Process

1. Add 200ml de-ionized water
2. Heat up and hold at 20°C
3. Let it stir for 1 minute 200 RPM, when vortex appears, lower RPM
4. Add in ±10s to 90ml Solution-1
5. Let it stir for 1 minute
6. Add 21ml Replenisher-1
7. Let it stir for 1 minute
8. Add 100ml de-ionized water
9. Track pH for 5 minutes
10. Check if pH is stable for 5min between 4.4 and 4.8 (rather 4.8)
11. If pH is stable go to step 14 if is not stable go to step 12
12. Add Ammonia by syringe to increase pH level or add acid to decrease pH level
13. Redo step 9, 10 & 11
14. Calculate x (x = 500 - 411 – added ammonia/acid)
15. Add x ml de-ionized water to solution
16. Add mark at height of solution
17. Stir for 30 minutes and heat up till 88°C and hold between 88 - 94°C
18. Add slowly 1 or 2 grams of luminescent particles
19. Stir for 5 minutes (if solution turns black → destabilized)
20. Add substrate to solution, set timer 60 min, make sure the plates don't move
21. Check height of solution every 10 min, if necessary add de-ionized water
22. Remove substrate from solution
23. Hang to dry on holder
24. Remove salt attached to plate under the tap (optional use soft sponge)

Notes

_____
_____
_____
_____
_____
_____

Documentation

1. Amount of water added: _____ml
2. Hold at temperature: _____ °C
3. *Let it stir for 1 minute* _____ *RPM* (Vortex Yes / No)
4. Amount of solution-1 added: _____ml
5. *Let it stir for 1 minute (showed* _____ *RPM)* (Vortex Yes / No)
6. Amount of Replenisher-1 added: _____ml
7. *Let it stir for 1 minute (showed* _____ *RPM)* (Vortex Yes / No)
8. Amount of de-ionized water added: _____ml
9. pH checked for _____ sec, highest value ____, lowest value____
10. pH after 5 min ____
11. *pH level to high / low, add Ammonia / Acid*
12. Ammonia / Acid added ____ ml, new pH _____
13. *Ammonia / Acid added* ____ *ml (2nd time) new pH* _____
14. Calculate x (x = 500 - 411 – added ammonia/acid)

    500 – *(1)*____ – *(4)*____ – *(6)*____ – *(8)*____ – *(12+13)*____ = *(15)* _____
15. Amount of de-ionized water added: _____ml
16. *Mark added at height of solution* (Vortex Yes / No)
17. Stirred for ____ min and heated up till ____°C, display showed ____°C
18. Amount of particles BAM:Eu / YO:Eu added: _____ gram
19. Stirred for ____ min (solution turned black Yes / No)
20. *Added plate to solution, timer set 60 min, substrates did not move*
21. 10 min, added ____ ml de-ionized water

    20 min, added ____ ml de-ionized water

    30 min, added ____ ml de-ionized water

    40 min, added ____ ml de-ionized water

    50 min, added ____ ml de-ionized water
22. *Plate removed from solution*
23. *Plates hung to dry*
24. *Salt layer on top Yes / No, if yes removed Yes / No*

F

Documentation characterization B&C

F.1. Datasheet characterization B&C

Figure F.1: Datasheet characterization B&C

Exp. B & C: SEM and EDS analysis

Goal

A SEM analysis is done to see the structure of the coating on the substrate. A EDS analysis is performed to quantify the amount of coating on the substrates.

Materials – Non chemical

- Substrates
- SEM machine *'JEOL JSM-IT100 InTouchSchope'*
- Holder for substrates
- Carbon stickers
- USB-stick
- Sanding machines (LaboPol-21)
- Polishing machines (LaboPol-5)
- Mounting machine (Predopress)
- Sanding paper (P80 – P180 – P320 – P800 – P1200)
- Polishing Cloths

Materials – Chemical

- Acetone
- Ethanol/2-iso propanol
- PolyFast
- Non-Stick powder
- Polishing Wax (3μm – 1μm)

Preparation

- Cut substrate in pieces of approx. 15mm * 15mm
- Create SEM Mount (for cross-section analysis)
  - Use instructions in Mounting/Sanding and Polishing room
  - Take picture and write down the order the samples are placed in mount
- Clean substrates in mount
  - Wipe substrates and mount clean and dry with paper
  - Place 4 beakers of 100ml in a row
  - Fill beaker 1 with: Acetone
  - Fill beaker 2 with: Demi water
  - Fill beaker 3 with: 2-iso propanol
  - Fill beaker 4 with: Demi water
  - Dip substrate 1 min in beaker 1
  - Rinse in beaker 2
  - Dip substrate 1 min in beaker 3
  - Rinse in beaker 4
  - Dip substrate 1 min in beaker 5
  - Rinse in beaker 6
  - Let dry in holder/use blow-dryer
- Prepare holder (for surface analysis)
  - Put metal holder on clean sheet
  - Put carbon stickers in the metal holder
  - Stick the substrates flat on the carbon stickers
  - Make sure substrates touch the carbon sticker
- Create file structure
  - Sample 1&2&3&4&5 – Analysis 1&2&3 – SEM & EDS

Process

1. Add (max 5) substrates in holder
   \1/2\3/4\5/

   1 =_____

   2 =_____

   3 =_____

   4 =_____

   5 =_____

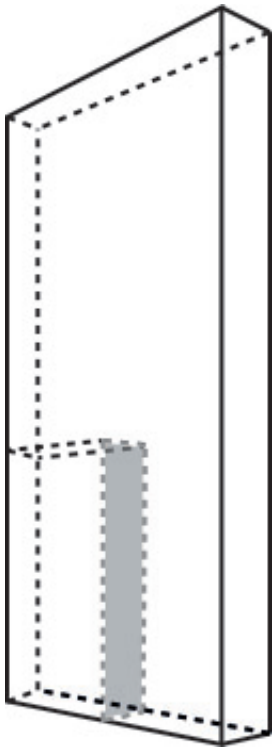
2. Place holder and start software (camera and program)
3. Find substrate *1*
4. *Find good spot, take picture at 100x*
5. *Find good spot, take picture at 1000x*
6. *Find good spot, take picture at 2000x*
7. Open EDS and perform analysis on multiple points (3+)
8. Copy the quantified analysis to file
9. Select all points and export them

Notes

_____
_____
_____
_____
_____
_____

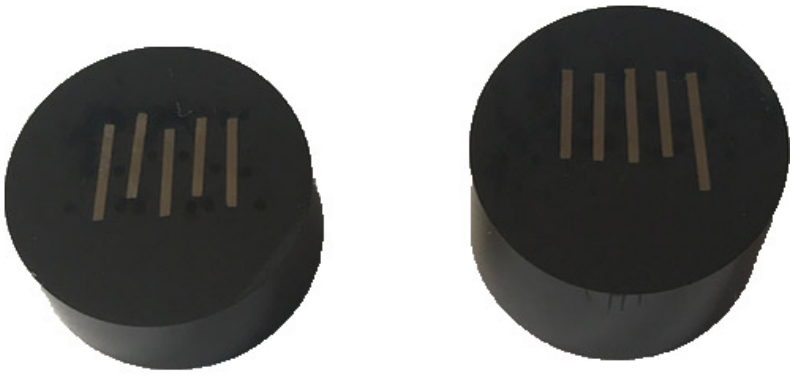
F.2. Cross-section

Figure F.2: Analyzed part of substrate



F.3. Substrates in epoxy mount

Figure F.3: Substrates in epoxy mount



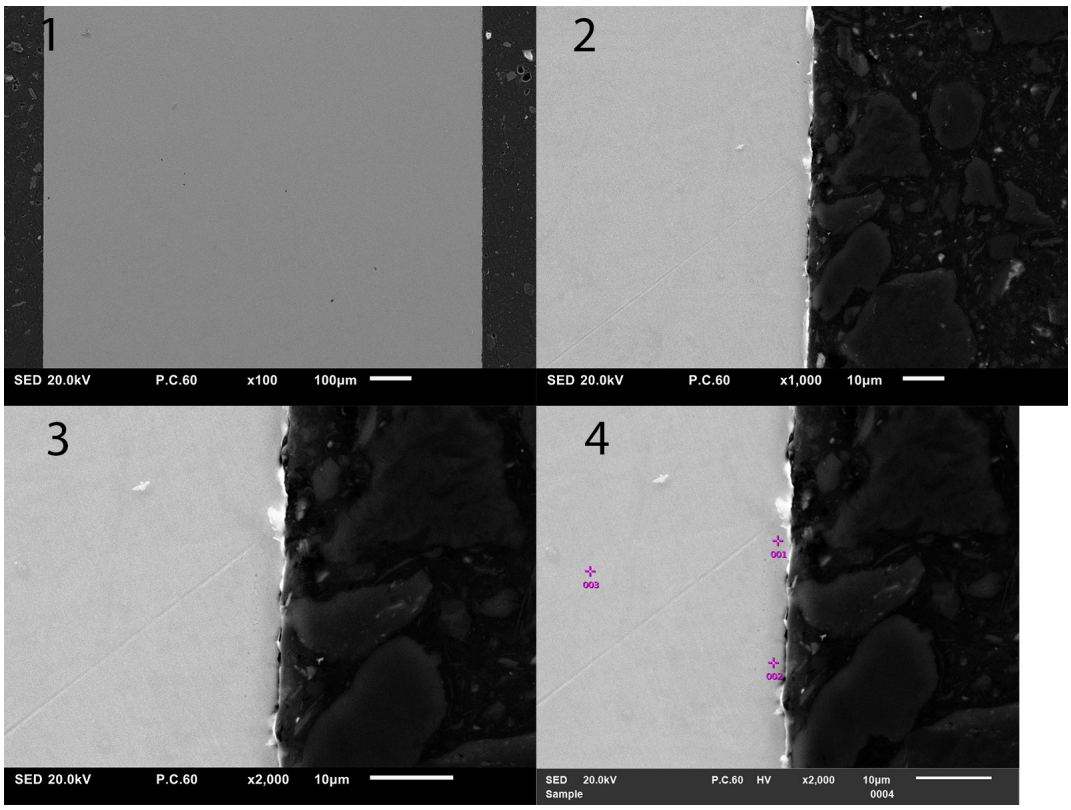
G

Experiment 0

G.1. SEM & EDS results (clean)

G.1.1. Microscope pictures

Figure G.1: SEM & EDS pictures of clean 316L
1) Cross-section of 316L x100;
2) Cross-section of 316L x1000;
3) Cross-section of 316L x2000;
4) Points analyzed by EDS



G.1.2. EDS results

Table G.1: Exp.0 clean - EDS results

1

Formula	Mass[%]	Atom[%]
Cr	18.91	20.17
Mn	1.59	1.61
Fe	64.02	63.59
Ni	15.48	14.63
Total	100.00	100.00

2

Formula	Mass[%]	Atom[%]
Cr	18.92	20.19
Fe	66.31	65.86
Ni	14.77	13.95
Total	100.00	100.00

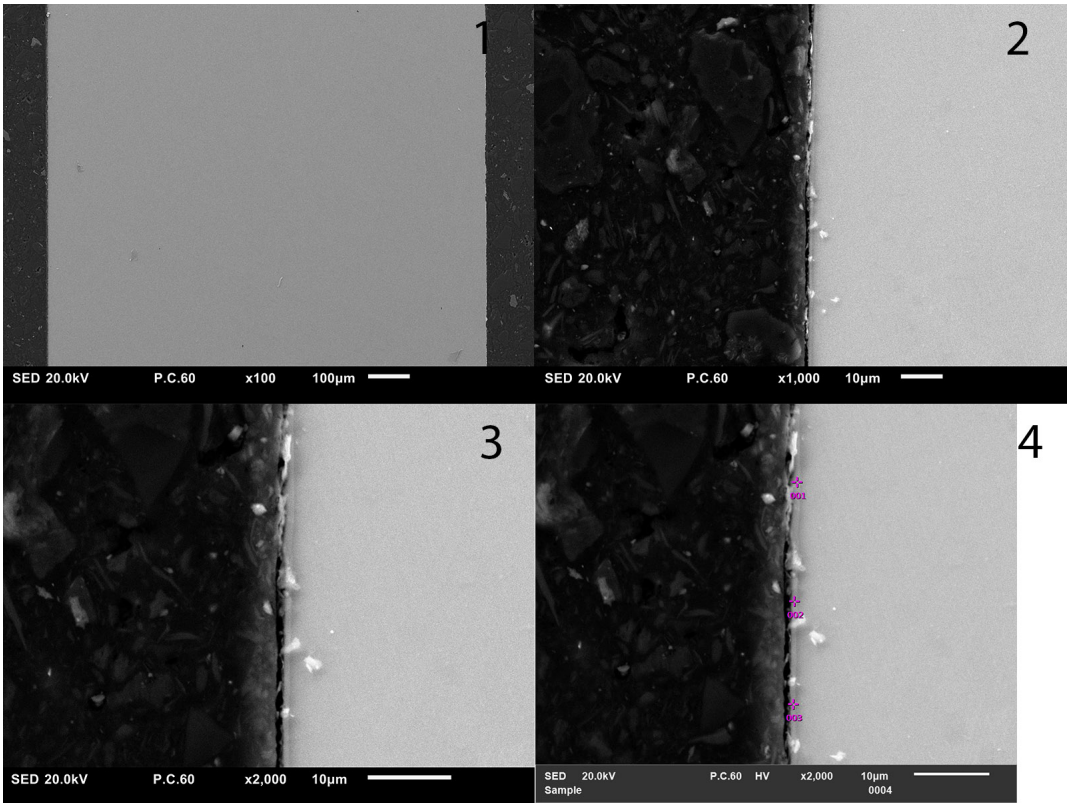
3

Formula	Mass[%]	Atom[%]
Cr	19.08	20.35
Mn	2.79	2.82
Fe	63.26	62.80
Ni	14.86	14.03
Total	100.00	100.00

G.2. SEM & EDS results (nickel strike)

G.2.1. Microscope pictures

Figure G.2: SEM & EDS pictures of 316L after nickel strike
1) Cross-section of 316L x100;
2) Cross-section of 316L x1000;
3) Cross-section of 316L x2000;
4) Points analyzed by EDS



G.2.2. EDS results

Table G.2: Exp. 0 after nickel strike - EDS Results

1

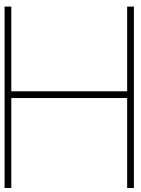
Formula	Mass[%]	Atom[%]
Cr	10.14	11.09
Mn	1.25	1.29
Fe	35.59	36.25
Ni	53.02	51.37
Total	100.00	100.00

2

Formula	Mass[%]	Atom[%]
Cr	8.43	9.25
Mn	0.66	0.69
Fe	33.34	34.08
Ni	57.57	55.98
Total	100.00	100.00

3

Formula	Mass[%]	Atom[%]
Cr	14.65	16.00
Mn	1.72	1.78
Fe	48.39	49.19
Ni	32.47	31.40
Mo	2.76	1.63
Total	100.00	100.00



Experiment 1

H.1. Datasheet experiment 1

Figure H.1: Datasheet experiment 1

Exp. 1 – Check particles with UV-Lamp when dry

Goal

To see if particles emit light when excited by UV-Lamp

Materials

- UV-Lamp
- BAM:Eu
- YO:Eu

Process

1. Open BAM:Eu flacon
2. Shine UV-Lamp directly into flacon
3. See if Blue light emits from particles
4. Take a small amount of BAM:Eu out of flacon
5. Shine UV-Lamp on particles
6. See if Blue light emits from particles
7. Close BAM:Eu flacon

8. Open YO:Eu flacon
9. Shine UV-Lamp directly into flacon
10. See if Red light emits from particles
11. Take a small amount of YO:Eu out of flacon
12. Shine UV-Lamp on particles
13. See if Red light emits from particles
14. Close YO:Eu flacon

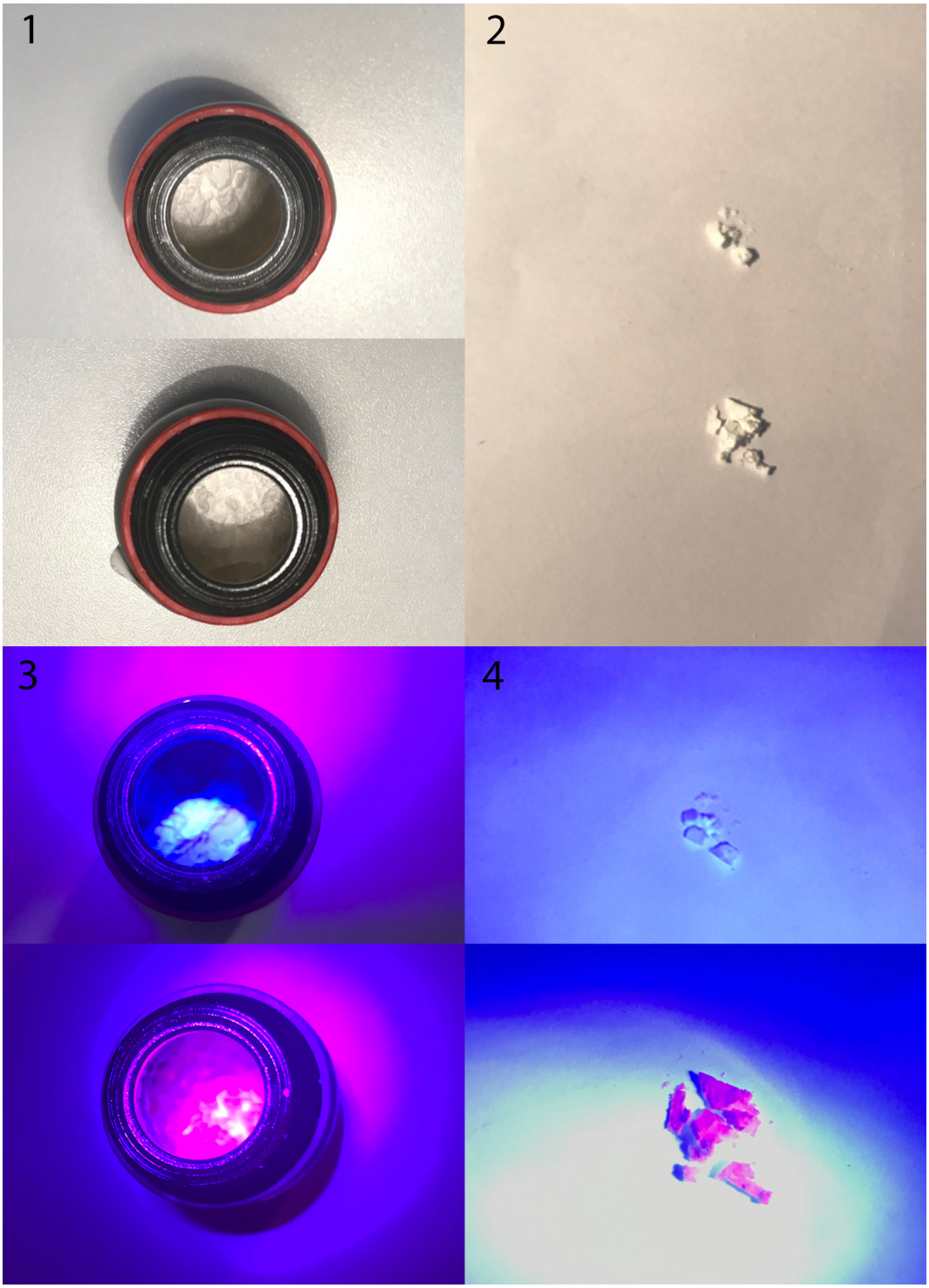
Documentation

1. *Open BAM:Eu flacon*
2. *Shine UV-Lamp directly into flacon*
3. Blue light emits from particles; YES / NO
4. *Take a small amount of BAM:Eu out of flacon*
5. *Shine UV-Lamp on particles*
6. Blue light emits from particles; YES / NO
7. *Close BAM:Eu flacon*

8. *Open YO:Eu flacon*
9. *Shine UV-Lamp directly into flacon*
10. RED light emits from particles; YES / NO
11. Take a small amount of YO:Eu out of flacon
12. Shine UV-Lamp on particles
13. Red light emits from particles; YES / NO
14. *Close YO:Eu flacon*

H.2. Pictures experiment 1

Figure H.2: Pictures experiment 1:
1) Top: Flacon BAM:Eu, Bottom: Flacon YO:Eu;
2) Top: BAM:Eu particles, Bottom: YO:Eu particles;
3) Top: Flacon BAM:Eu under UV-light, Bottom: Flacon YO:Eu under UV-light;
4) Top: BAM:Eu particles under UV-light, Bottom: YO:Eu particles under UV-light



Experiment 2

I.1. Datasheet experiment 2

Figure I.1: Datasheet experiment 2

Exp. 2 – Mix particles in nickel bath at 20°C

Goal

To see if particles float or mix and if the nickel bath stays stable

Materials

- 1L de-ionized water
- 180ml Solution-1
- 42ml Replenisher-1
- 2x 500+ml beaker
- Magnetic stirrer
- Jerrycan for disposal
- 1-2 gram particles (BAM:Eu & YO:Eu)

Process

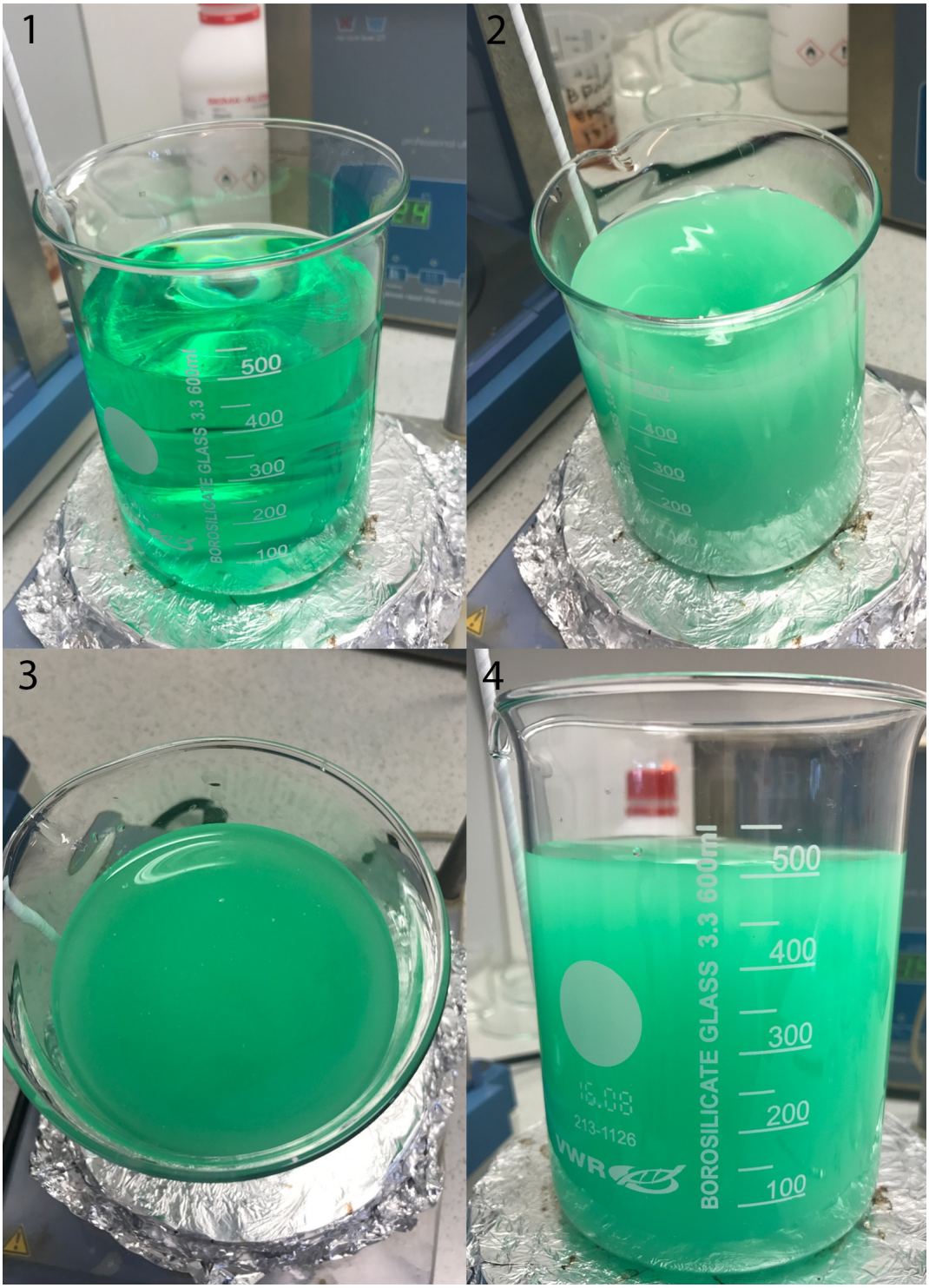
1. Add 200ml de-ionized water
2. Let it stir for 1 minute
3. Add in ±10s to 90ml Solution-1
4. Let it stir for 1 minute
5. Add 21ml Replenisher-1
6. Let it stir for 1 minute
7. Add 100ml de-ionized water
8. Fill till 500ml
9. Let it stir for 1 minute
10. Add slowly 1 or 2 grams of luminescent particles
11. Stir for 5 minutes (if solution turns black → destabilized)
12. Check if particles mix with solution or float.
13. Dispose in jerrycan with label

Documentation

1. Amount of water added: _____ml
2. *Let it stir for 1 minute*
3. Amount of solution-1 added: _____ml
4. *Let it stir for 1 minute*
5. Amount of Replenisher-1 added: _____ml
6. *Let it stir for 1 minute*
7. Amount of de-ionized water added: _____ml
8. Amount of de-ionized water added: _____ml
9. *Let it stir for 1 minute*
10. Amount of particles BAM:Eu / YO:Eu added: _____ gram
11. Stirred for ____ min (solution turned black Yes / No)
12. Particles Mix / Float

I.2. Pictures experiment 2

Figure I.2: Pictures experiment 2:
1) Nickel bath without particles added;
2) Stirring BAM:Eu particles in nickel bath;
3) BAM:Eu particles in nickel bath after 10 min;
4) YO:Eu particles in nickel bath after 10 min



J

Experiment 3

J.1. Pictures experiment 3

Figure J.1: Pictures experiment 3.1:
1) Side view of nickel bath with substrates;
2) Top view of nickel bath with substrates;
3) Enlarged image of substrate in nickel bath;
4) Enlarged image of top of nickel bath

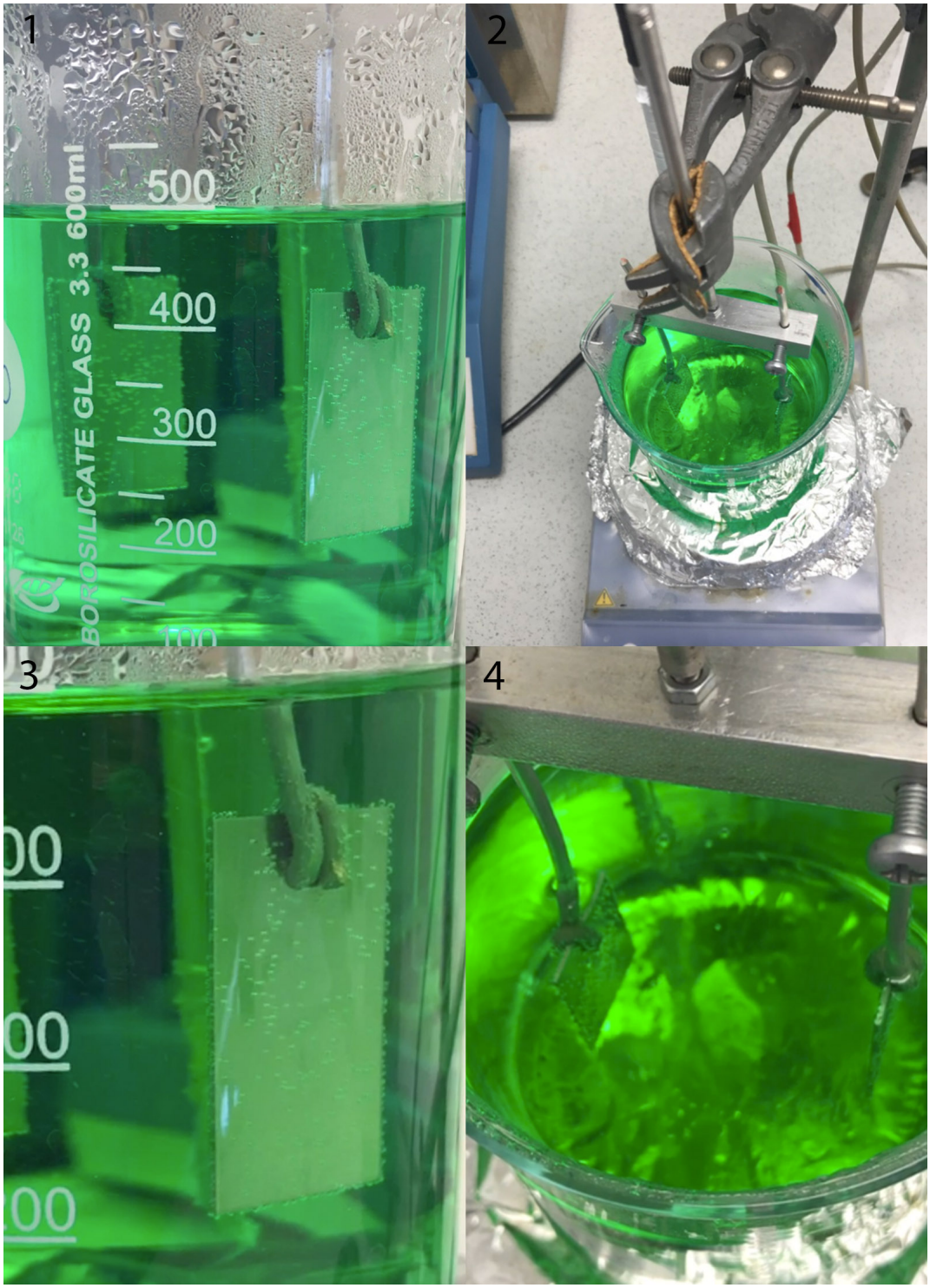
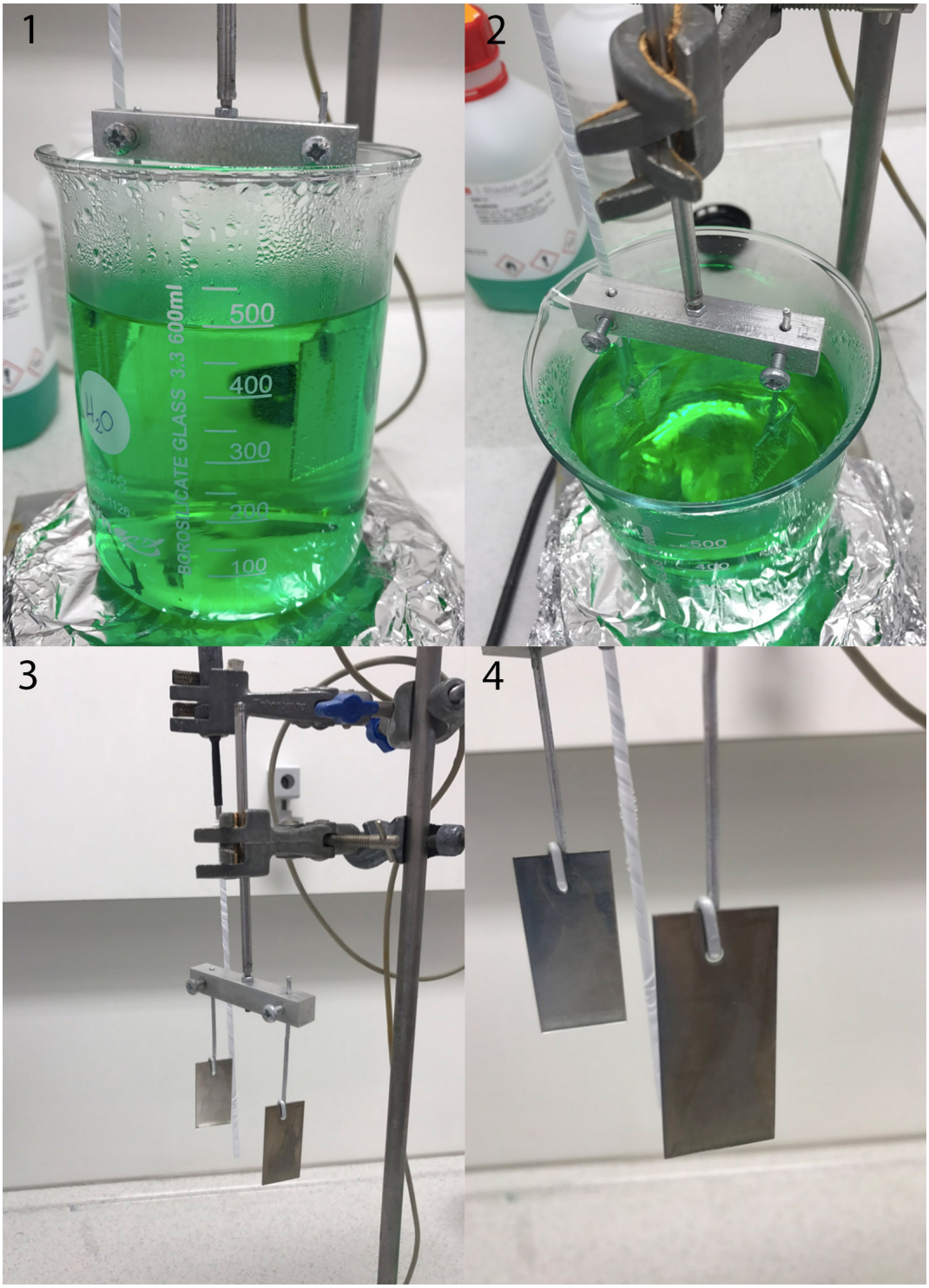


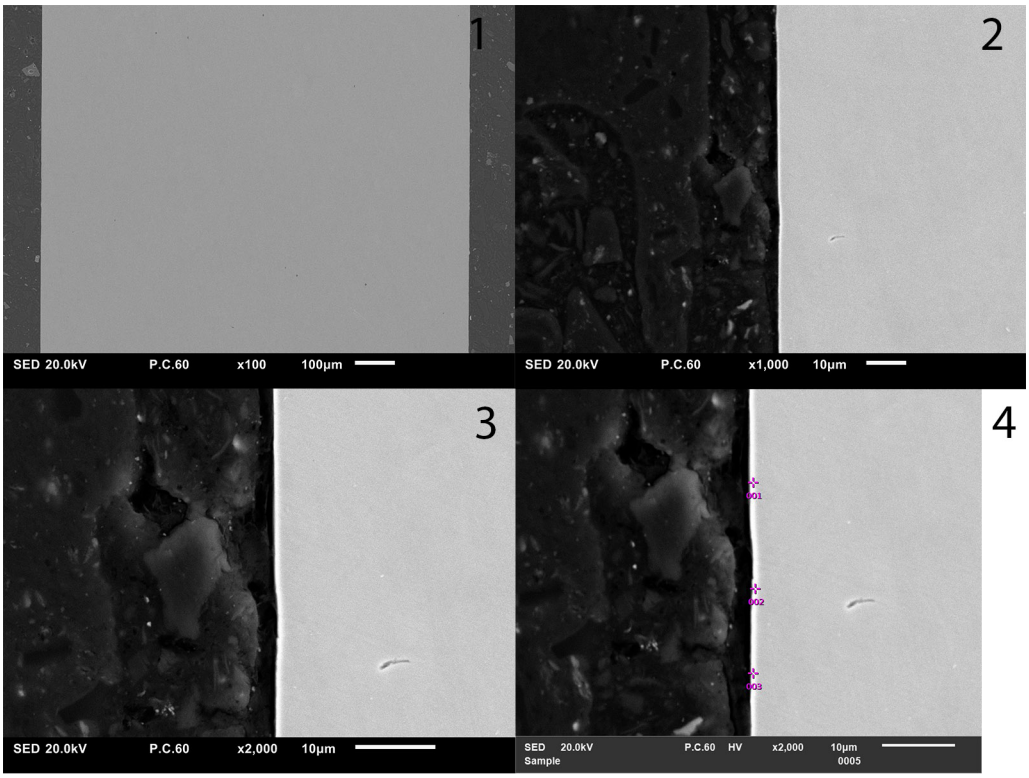
Figure J.2: Pictures experiment 3.2:
1) Side view of nickel bath with substrates;
2) Top view of nickel bath with substrates;
3) Substrates after nickel bath;
4) Enlarged image of substrates after nickel bath



J.2. SEM & EDS results

J.2.1. Microscope pictures

Figure J.3: SEM & EDS pictures of experiment 3
1) Cross-section of 316L after nickel plating x100;
2) Cross-section of 316L after nickel plating x1000;
3) Cross-section of 316L after nickel plating x2000;
4) Points analyzed by EDS



J.2.2. EDS results

Table J.1: Exp. 3: EDS results

1

Formula	Mass[%]	Atom[%]
Cr	19.40	20.92
Mn	2.28	2.33
Fe	61.47	61.71
Ni	14.04	13.41
Mo	2.80	1.63
Total	100.00	100.00

2

Formula	Mass[%]	Atom[%]
Cr	18.63	20.15
Mn	2.14	2.19
Fe	61.06	61.49
Ni	14.85	14.22
Mo	3.33	1.95
Total	100.00	100.00

3

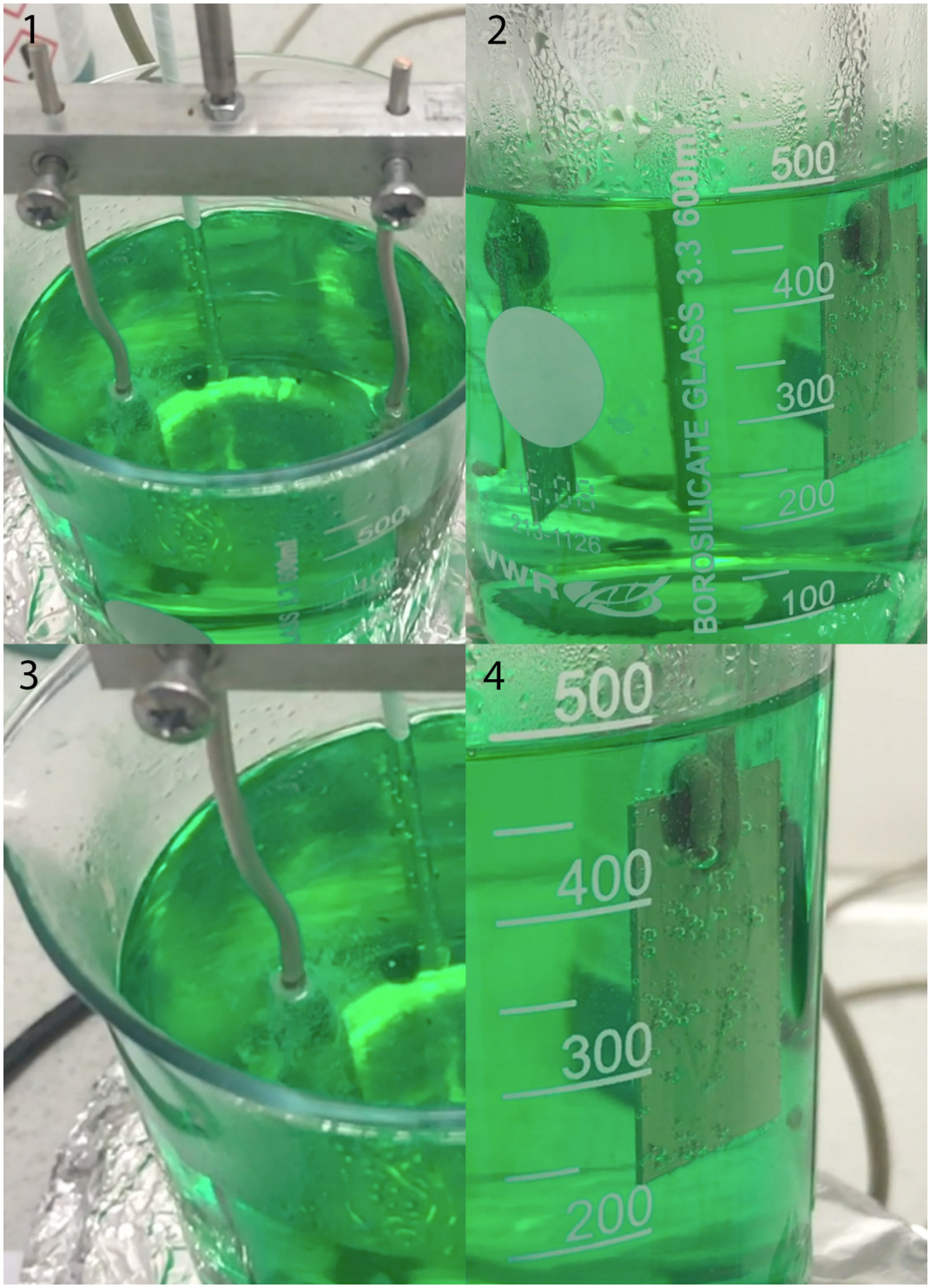
Formula	Mass[%]	Atom[%]
Cr	19.18	20.45
Mn	2.06	2.08
Fe	63.99	63.52
Ni	14.77	13.95
Total	100.00	100.00

K

Experiment 4

K.1. Pictures experiment 4

Figure K.1: Pictures experiment 4, left steel, right 316L:
1) Top view of substrates in nickel bath;
2) Side view of substrates in nickel bath;
3) Enlarged image of steel substrate;
4) Enlarged image of 316L substrate



K.2. Coating thickness calculation

$$
\begin{aligned}
b - a &= 0,0525 \\
\rho &= 8,875 - 0,09375 \cdot 7 \\
&= 8,22 \\
A &= 18,25
\end{aligned}
\tag{K.1}
$$

$$
\begin{aligned}
T &= \frac{0,0525}{8,22} \cdot \frac{1}{18,25} \\
&= 3,50
\end{aligned}
$$

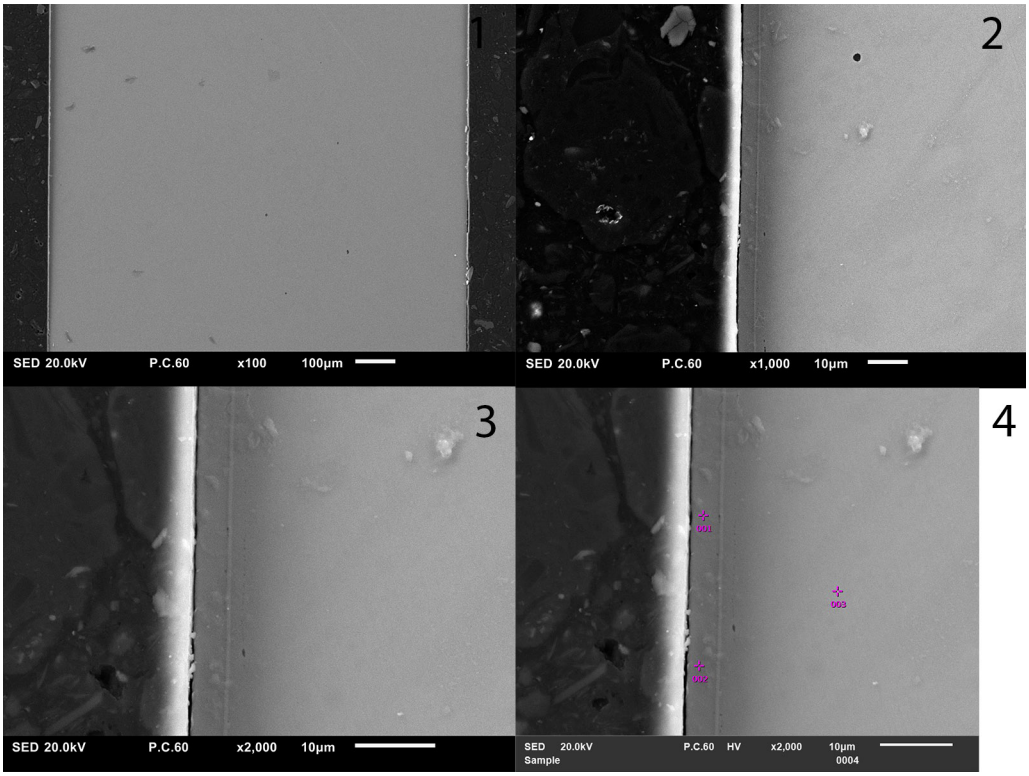
K.3. Color analysis

Figure K.2: Experiment 4 substrates on a nickel plate
1) 316L stainless steel after nickel strike;
2) 316L stainless steel after nickel plating;
3) Steel after nickel plating;
4) Steel before nickel plating

K.4. SEM & EDS results

K.4.1. Microscope pictures

Figure K.3: SEM & EDS pictures of experiment 4
1) Cross-section of 316L after nickel plating x100;
2) Cross-section of 316L after nickel plating x1000;
3) Cross-section of 316L after nickel plating x2000;
4) Points analyzed by EDS



K.4.2. EDS results

Table K.1: Exp. 4: EDS Results

1		
Formula	Mass[%]	Atom[%]
P	6.98	12.45
Ni	93.02	87.55
Total	100.00	100.00

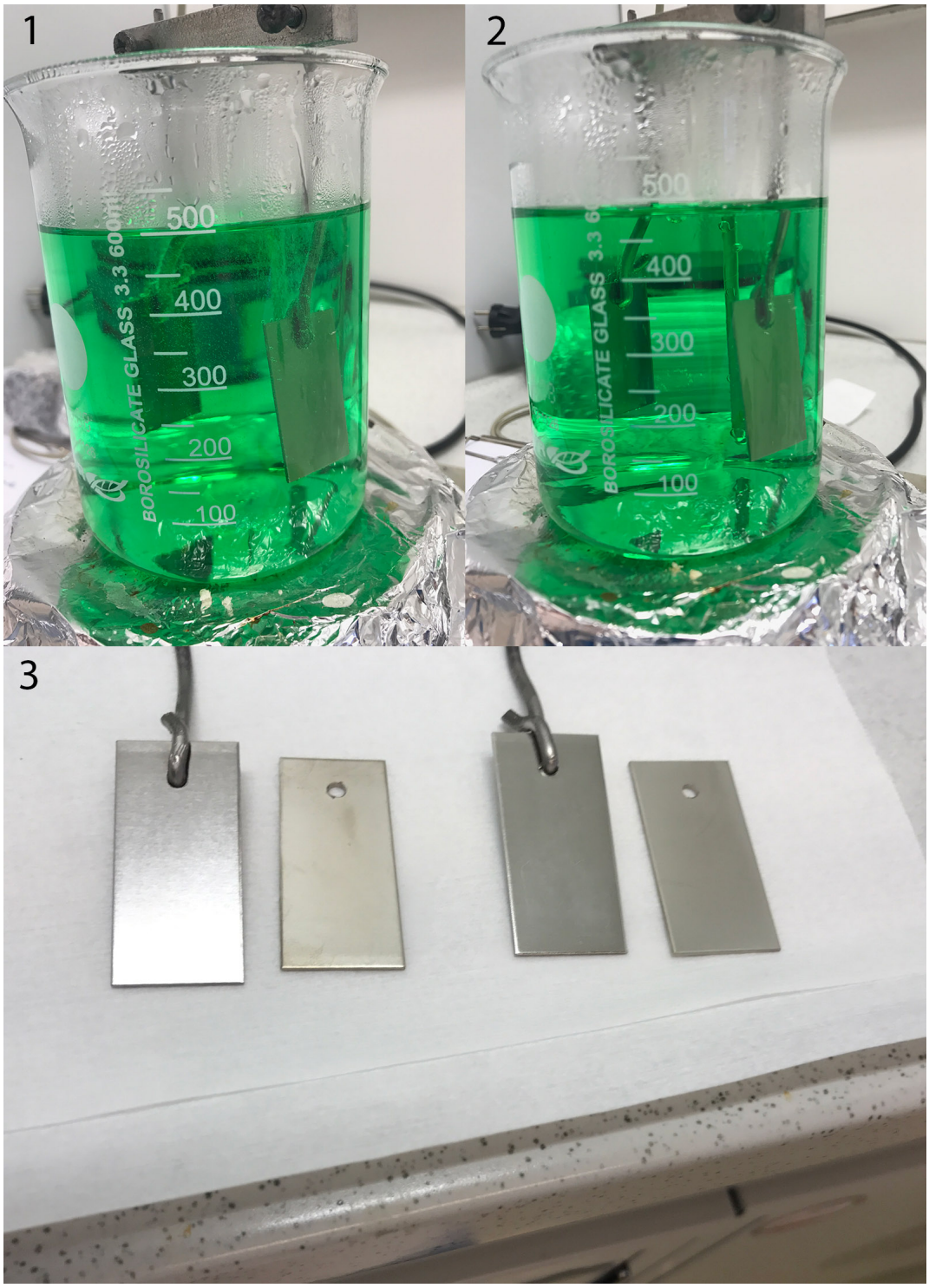
2		
Formula	Mass[%]	Atom[%]
P	6.55	11.72
Fe	2.70	2.67
Ni	90.75	85.61
Total	100.00	100.00

3		
Formula	Mass[%]	Atom[%]
Fe	100.00	100.00
Total	100.00	100.00

Experiment 5

L.1. Pictures experiment 5

Figure L.1: Pictures experiment 5:
1) Side view of nickel bath at beginning;
2) Side view of nickel bath at end;
3) Substrates before and after nickel plating



L.2. Coating thickness calculation

$$
\begin{aligned}
b - a &= 0,0561 \\
\rho &= 8,875 - 0,09375 \cdot 13 \\
&= 7,66 \\
A &= 18,25
\end{aligned}
\tag{L.1}
$$

$$
\begin{aligned}
T &= \frac{0,0561}{7,66} \cdot \frac{1}{18,25} \\
&= 4,01
\end{aligned}
$$

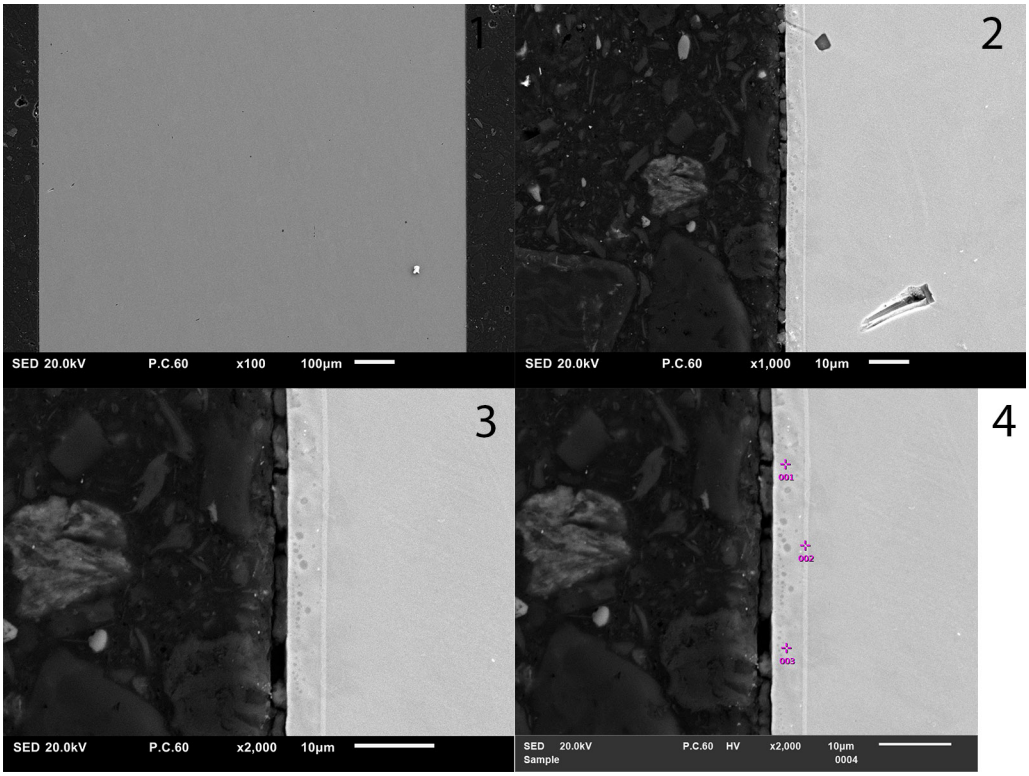
L.3. Color analysis

Figure L.2: Experiment 5 substrates on a nickel plate
1) 316L stainless steel after nickel plating;
2) 316L stainless steel after nickel strike;
3) 316L stainless steel after nickel plating

L.4. SEM & EDS results

L.4.1. Microscope pictures

Figure L.3: SEM & EDS pictures of experiment 5
1) Cross-section of 316L after nickel plating x100;
2) Cross-section of 316L after nickel plating x1000;
3) Cross-section of 316L after nickel plating x2000;
4) Points analyzed by EDS



L.4.2. EDS results

Table L.1: Exp. 5: EDS results

1

Formula	Mass[%]	Atom[%]
P	13.14	22.29
Ni	86.86	77.71
Total	100.00	100.00

2

Formula	Mass[%]	Atom[%]
Cr	10.64	11.61
Mn	1.45	1.49
Fe	39.00	39.62
Ni	48.92	47.28
Total	100.00	100.00

3

Formula	Mass[%]	Atom[%]
P	13.34	22.59
Ni	86.66	77.41
Total	100.00	100.00

M

Experiment 6

M.1. Pictures experiment 6

Figure M.1: Pictures experiment 6:
1) Side view nickel bath with BAM:Eu particles;
2) Top view nickel bath with BAM:Eu particles;
3) Substrate 3 before and after;
4) Substrate 4 before and after

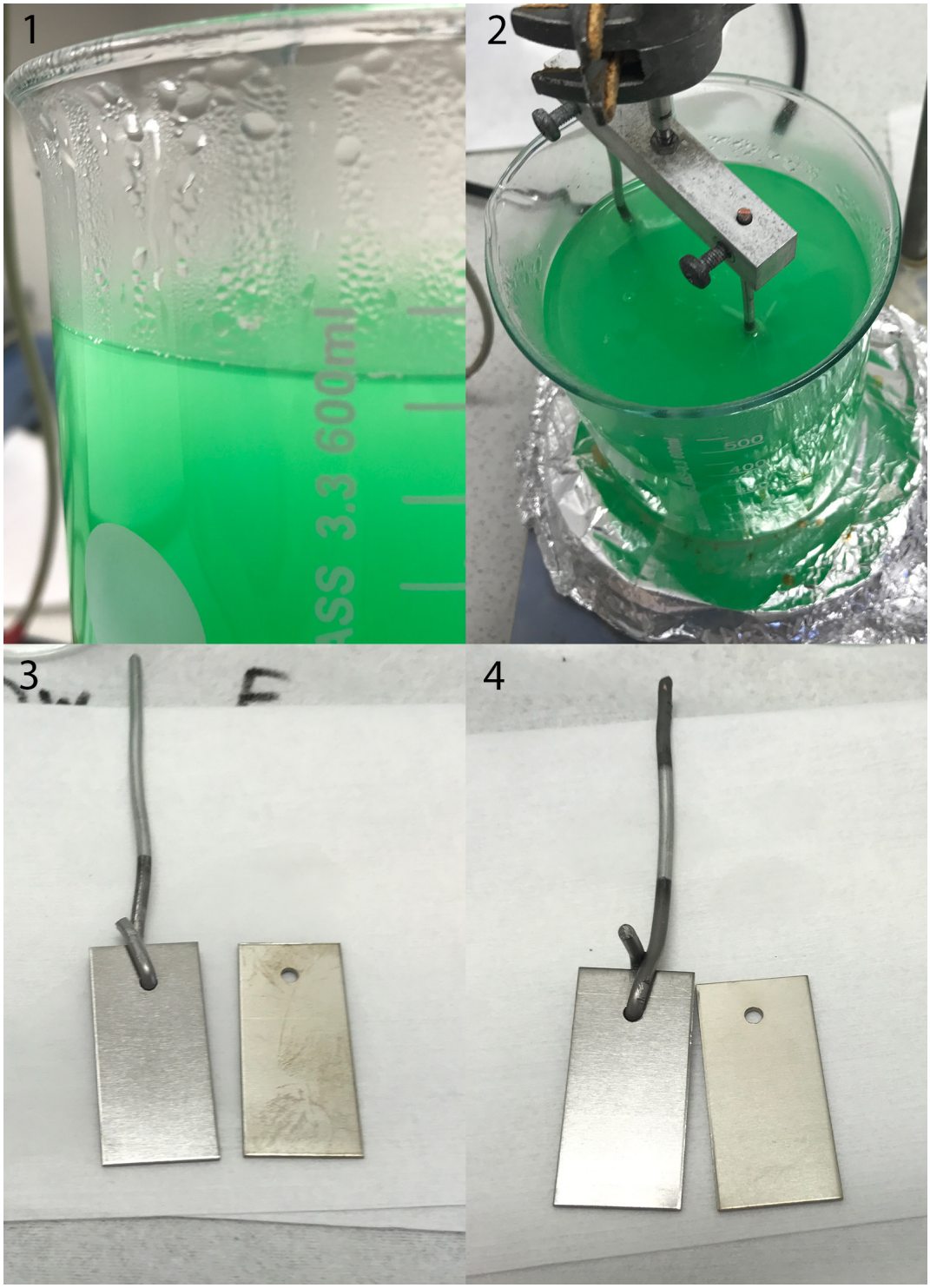
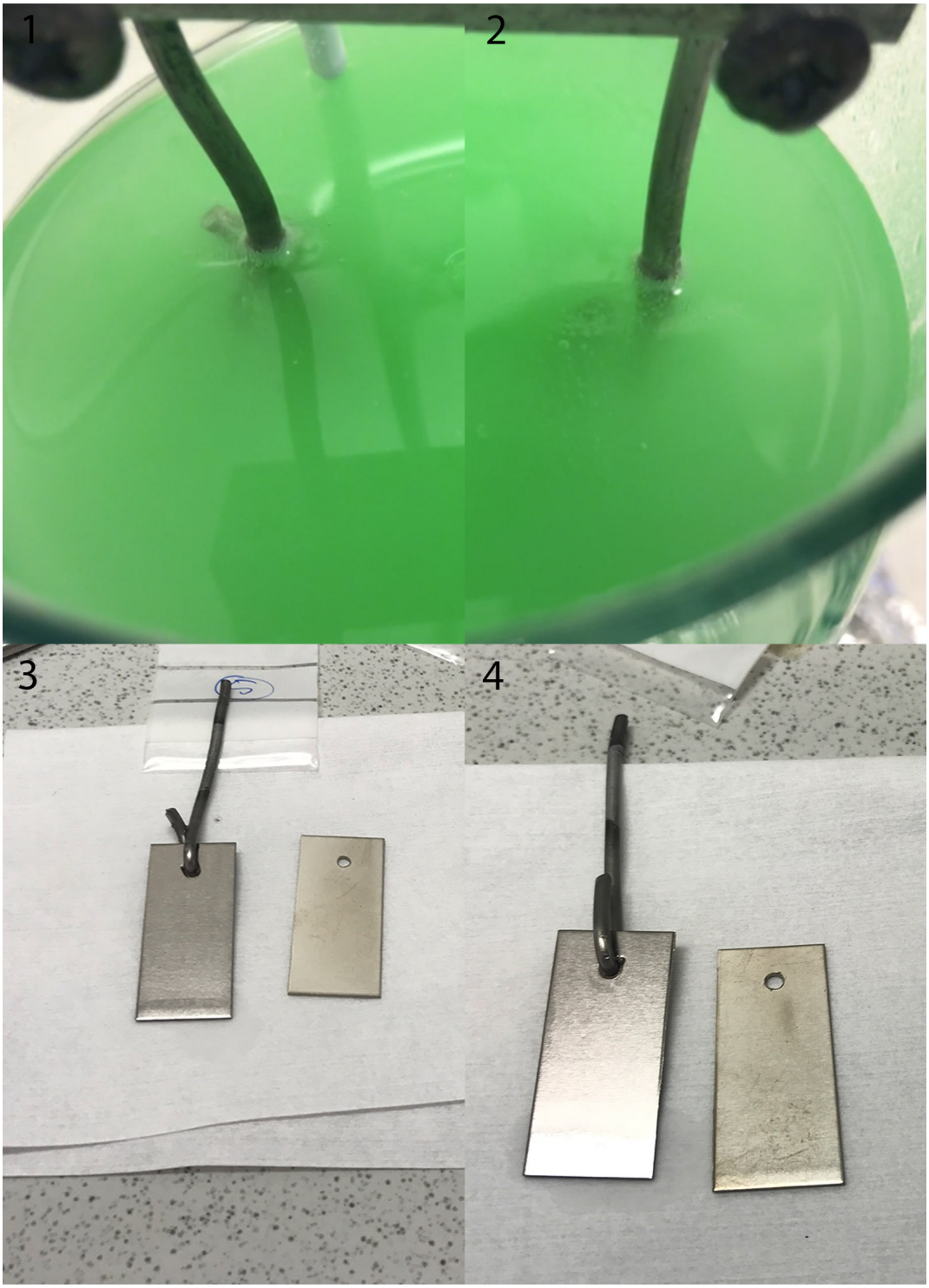


Figure M.2: Pictures experiment 6:
1) Gas evolution at substrate 5;
2) Gas evolution at substrate 6;
3) Substrate 5 before and after;
4) Substrate 6 before and after



M.2. Coating thickness calculation

$$b - a = 0,0798 \tag{M.1}$$

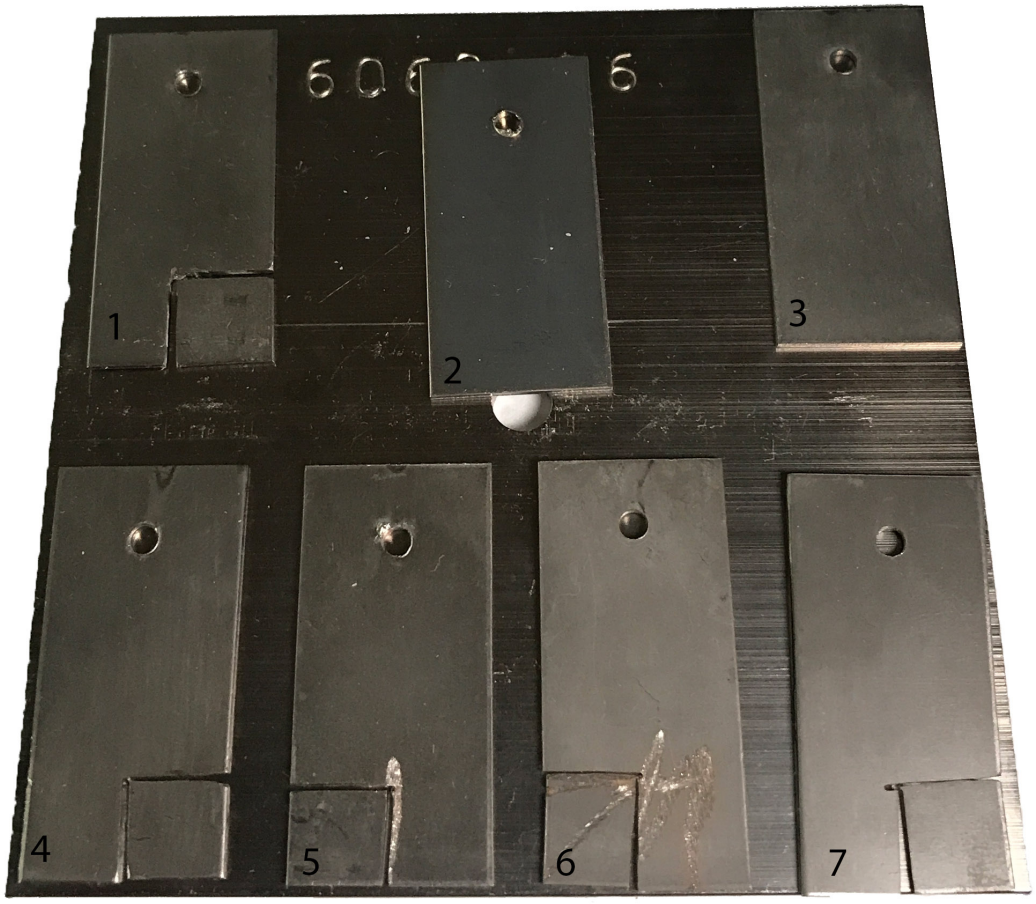
$$\begin{aligned} \rho &= 8,875 - 0,09375 \cdot 13 \\ &= 7,66 \\ A &= 18,25 \end{aligned}$$

(M.2)

$$\begin{aligned} T &= \frac{0,0798}{7,66} \cdot \frac{1}{18,25} \\ &= 5,64 \end{aligned}$$

M.3. Color analysis

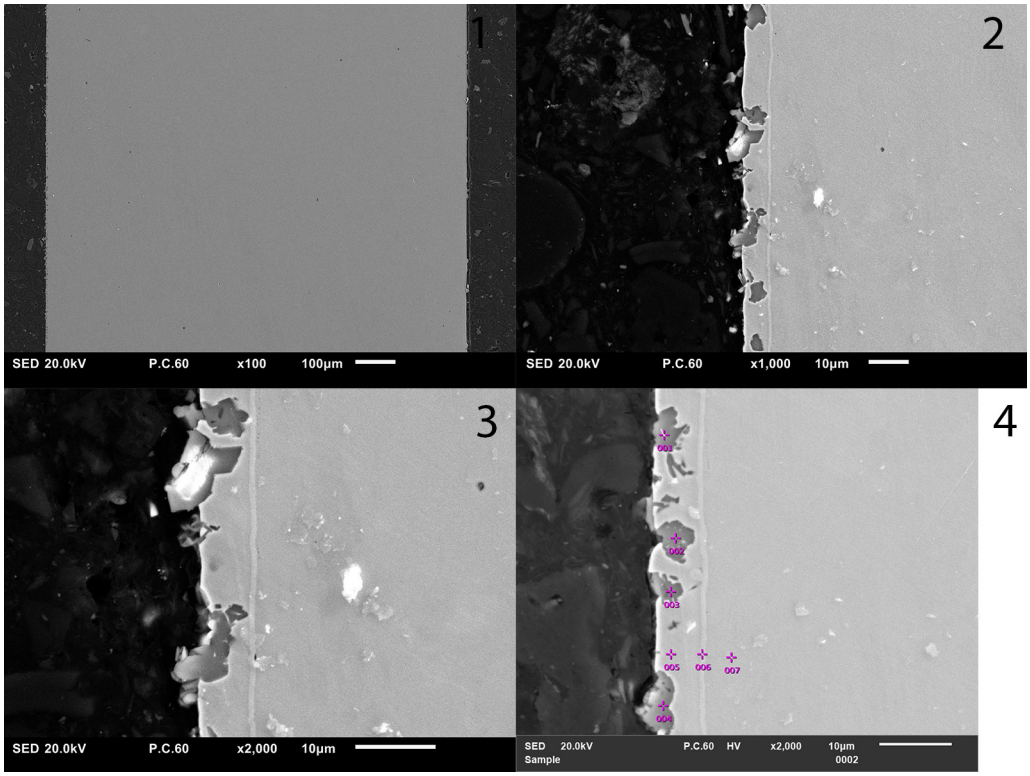
Figure M.3: Experiment 6 substrates on a nickel late
1, 3-7) 316L stainless steel after nickel plating with BAM particles;
2) 316L stainless steel after nickel strike



M.4. SEM & EDS results

M.4.1. Microscope pictures particle side

Figure M.4: SEM & EDS pictures of experiment 6 (particle side)
1) Cross-section of 316L after nickel plating with BAM [x100];
2) Cross-section of 316L after nickel plating with BAM [x1000];
3) Cross-section of 316L after nickel plating with BAM [x2000];
4) Points analyzed by EDS



M.4.2. EDS results

Table M.1: Exp. 6: EDS results particle side

1

Formula	Mass[%]	Atom[%]
O	33.25	53.48
Mg	3.75	3.97
Al	32.56	31.04
P	2.28	1.90
Ni	17.39	7.62
Ba	9.07	1.70
Eu*	1.70	0.29
Total	100.00	100.00

2

Formula	Mass[%]	Atom[%]
O	37.30	58.67
Mg	3.30	3.41
Al	34.76	32.42
Ni	4.14	1.78
Ba	18.46	3.38
Eu*	2.05	0.34
Total	100.00	100.00

3

Formula	Mass[%]	Atom[%]
O	35.15	56.92
Mg	3.28	3.50
Al	32.20	30.92
P	1.32	1.10
Ni	9.17	4.05
Ba	16.08	3.03
Eu*	2.79	0.48
Total	100.00	100.00

4

Formula	Mass[%]	Atom[%]
O	35.69	56.96
Mg	3.27	3.43
Al	32.47	30.73
P	1.44	1.19
Ni	10.77	4.68
Ba	14.56	2.71
Eu*	1.80	0.30
Total	100.00	100.00

5

Formula	Mass[%]	Atom[%]
P	12.80	21.77
Ni	87.20	78.23
Total	100.00	100.00

6

Formula	Mass[%]	Atom[%]
Cr	7.24	8.00
Fe	25.41	26.13
Ni	67.35	65.87
Total	100.00	100.00

7

Formula	Mass[%]	Atom[%]
Cr	18.98	20.52
Mn	1.90	1.94
Fe	61.73	62.13
Ni	14.06	13.46
Mo	3.33	1.95
Total	100.00	100.00

Table M.2: Exp. 6: EDS results BAM ratio

1

Formula	Atom[%]	Ratio
O	53,48	13,471
Mg	3,97	1,000
Al	31,04	7,819
P	1,9	0,479
Ni	7,62	1,919
Ba	1,7	0,428
Eu*	0,29	0,073

BAM Ratio
16
1
10
N/A
N/A
0,86
0,14

3

Formula	Atom[%]	Ratio
O	56,92	16,263
Mg	3,5	1,000
Al	30,92	8,834
P	1,1	0,314
Ni	4,05	1,157
Ba	3,03	0,866
Eu*	0,48	0,137

2

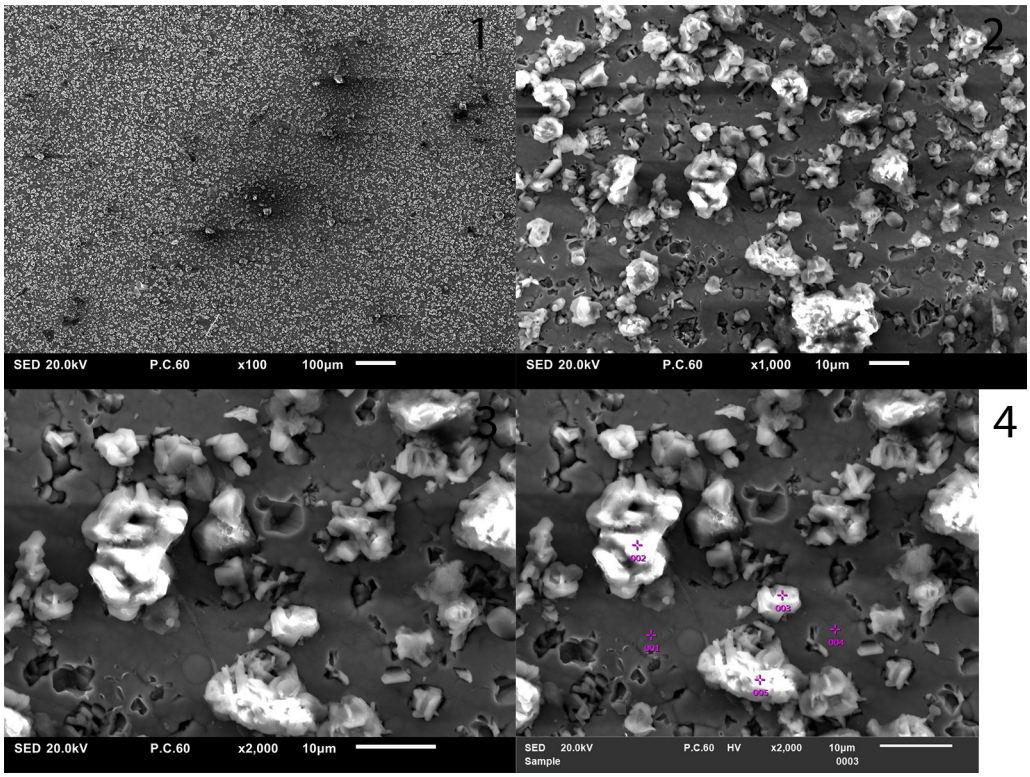
Formula	Atom[%]	Ratio
O	58,67	17,205
Mg	3,41	1,000
Al	32,42	9,507
Ni	1,78	0,522
Ba	3,38	0,991
Eu*	0,34	0,100

4

Formula	Atom[%]	Ratio
O	56,96	16,606
Mg	3,43	1,000
Al	30,73	8,959
P	1,19	0,347
Ni	4,68	1,364
Ba	2,71	0,790
Eu	0,3	0,087

M.4.3. Microscope pictures surface

Figure M.5: SEM & EDS pictures of experiment 6 (surface)
1) Surface of 316L after nickel plating with BAM [x100];
2) Surface of 316L after nickel plating with BAM [x1000];
3) Surface of 316L after nickel plating with BAM [x2000];
4) Points analyzed by EDS



M.4.4. EDS results surface

Table M.3: Exp. 6: EDS results surface

1

Formula	Mass[%]	Atom[%]
P	13.65	23.06
Ni	86.35	76.94
Total	100.00	100.00

2

Formula	Mass[%]	Atom[%]
O	44.34	63.04
Mg	4.17	3.91
Al	36.25	30.56
Ba	13.79	2.28
Eu*	1.44	0.22
Total	100.00	100.00

3

Formula	Mass[%]	Atom[%]
O*	40.83	60.65
Mg	3.72	3.64
Al	36.17	31.86
Ni	2.36	0.95
Ba	15.12	2.62
Eu*	1.80	0.28
Total	100.00	100.00

4

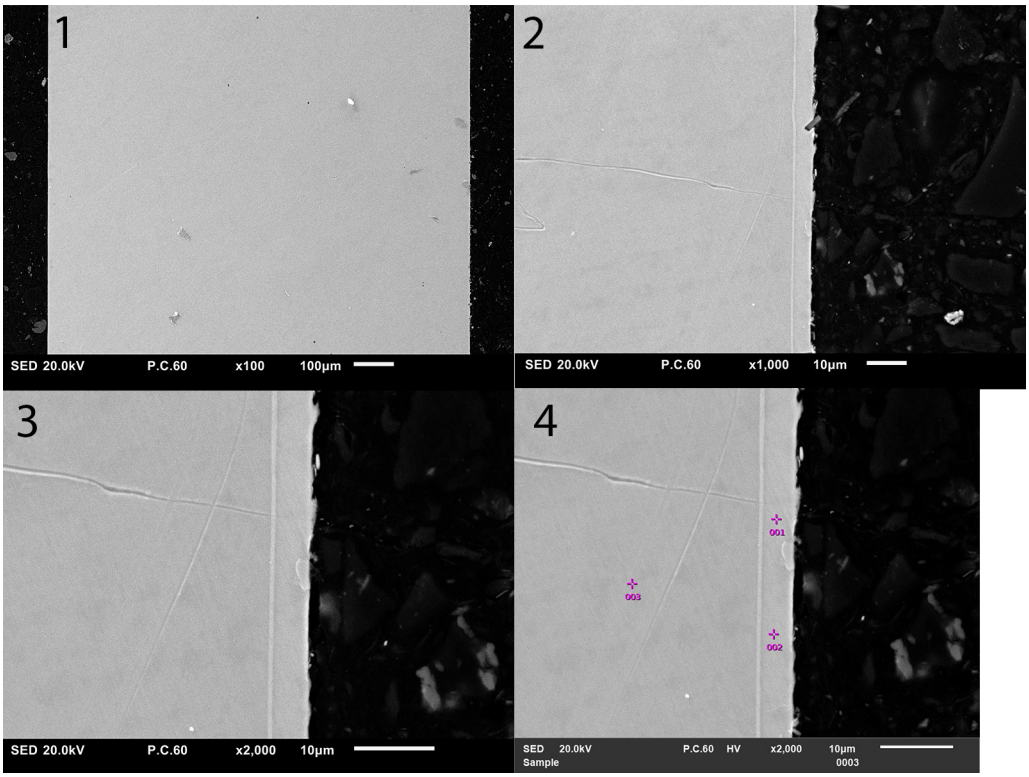
Formula	Mass[%]	Atom[%]
O	1.29	4.06
Al	1.09	2.05
P	12.81	20.90
Ni	84.81	72.99
Total	100.00	100.00

5

Formula	Mass[%]	Atom[%]
O	33.52	54.37
Mg	3.55	3.79
Al	38.17	36.71
Ni	1.93	0.85
Ba	20.36	3.85
Eu*	2.46	0.42
Total	100.00	100.00

M.4.5. Microscope pictures no particle side

Figure M.6: SEM & EDS pictures of experiment 6 (no particle side)
1) Cross-section of 316L after nickel plating with BAM [x100];
2) Cross-section of 316L after nickel plating with BAM [x1000];
3) Cross-section of 316L after nickel plating with BAM [x2000];
4) Points analyzed by EDS



M.4.6. EDS results no particle side

Table M.4: Exp. 6: EDS results no particle side

1		
Formula	Mass[%]	Atom[%]
P	12.57	21.41
Ni	87.43	78.59
Total	100.00	100.00

2		
Formula	Mass[%]	Atom[%]
P	13.32	22.56
Ni	86.68	77.44
Total	100.00	100.00

3		
Formula	Mass[%]	Atom[%]
S	0.91	1.57
Cr	20.13	21.29
Mn	1.40	1.40
Fe	63.59	62.65
Ni	13.97	13.09
Total	100.00	100.00

Experiment 7

N.1. Pictures experiment 7

Figure N.1: Pictures experiment 7:
1) H_2 evolution at substrate 7;
2) YO:Eu particles residue;
3) Substrate 7 before and after;
4) Substrate 8 before and after

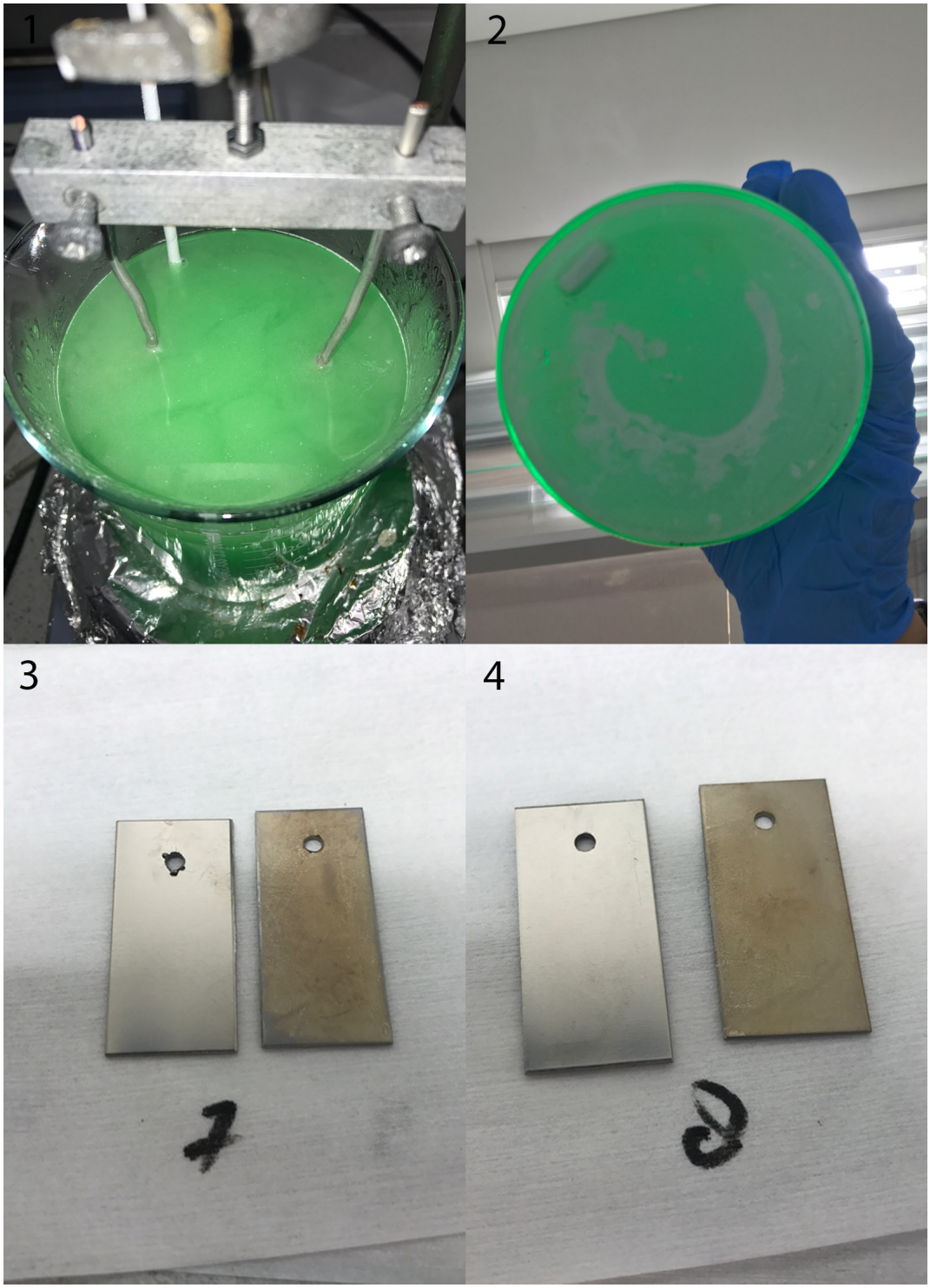
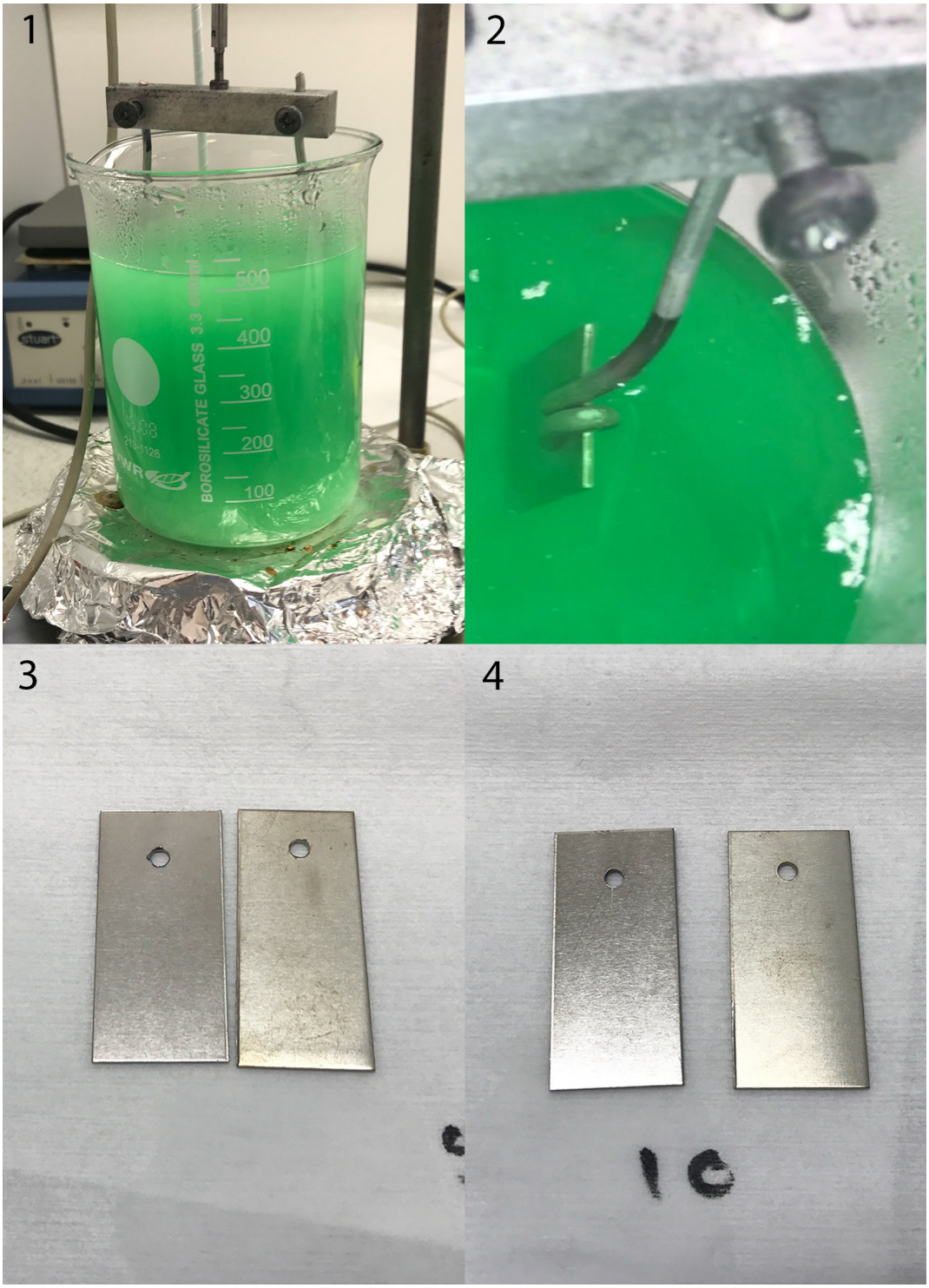


Figure N.2: Pictures experiment 7:
1) Side view nickel bath with YO:Eu particles;
2) Agglomeration of YO:Eu particles in nickel bath;
3) Substrate 9 before and after;
4) Substrate 10 before and after



N.2. Coating thickness calculation

$$b - a = 0,0214 \tag{N.1}$$

$$\begin{aligned} \rho &= 8,875 - 0,09375 \cdot 12 \\ &= 7,75 \\ A &= 18,25 \end{aligned}$$

$$T = \frac{0,0214}{7,75} \cdot \frac{1}{18,25} \tag{N.2}$$
$$= 1,51$$

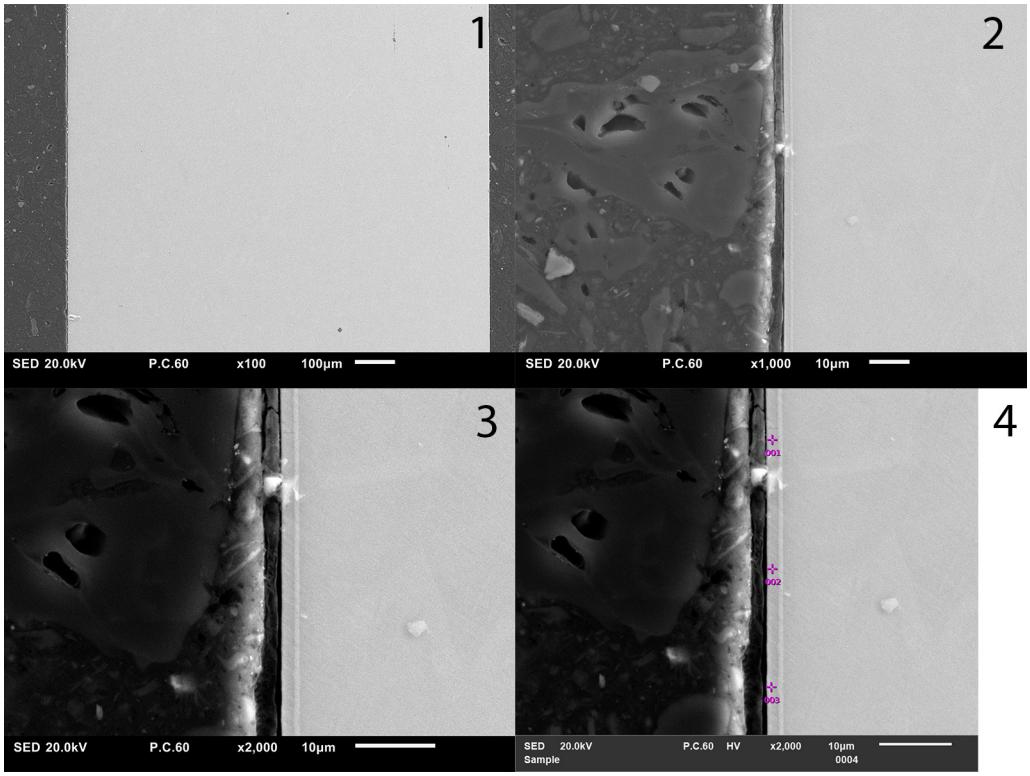
N.3. Color analysis

Figure N.3: Experiment 7 substrates on a nickel plate
1, 3-7) 316L stainless steel after nickel plating with YO particles;
2) 316L stainless steel after nickel strike

N.4. SEM & EDS results

N.4.1. Microscope pictures

Figure N.4: SEM & EDS pictures of experiment 7
1) Cross-section of 316L after nickel plating x100;
2) Cross-section of 316L after nickel plating x1000;
3) Cross-section of 316L after nickel plating x2000;
4) Points analyzed by EDS



N.4.2. EDS results

Table N.1: Exp. 7: EDS results

1

Formula	Mass[%]	Atom[%]
P	11.81	20.22
Fe	2.97	2.82
Ni	85.22	76.96
Total	100.00	100.00

2

Formula	Mass[%]	Atom[%]
P	12.27	20.93
Fe	2.78	2.63
Ni	84.95	76.45
Total	100.00	100.00

3

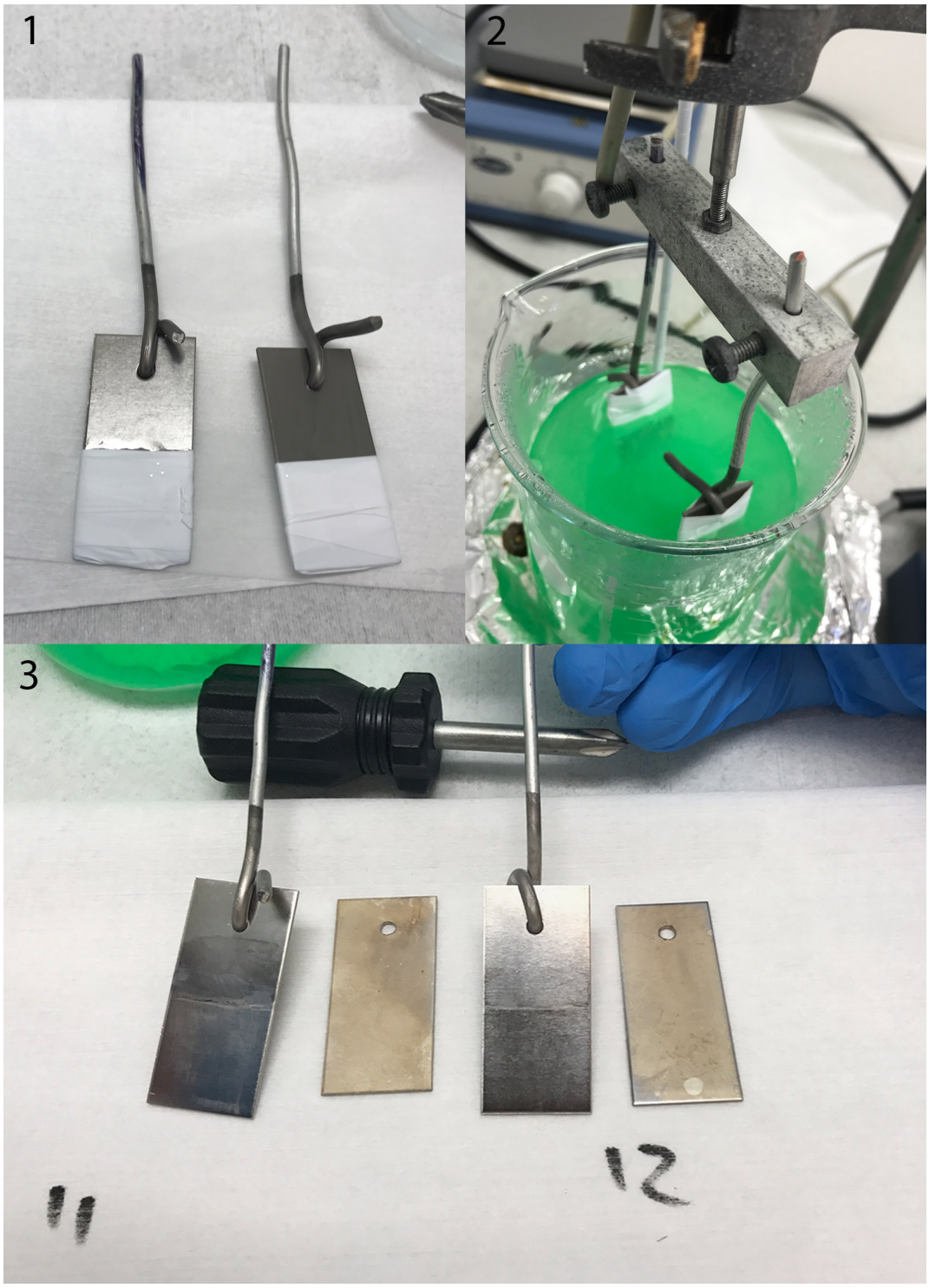
Formula	Mass[%]	Atom[%]
P	12.33	21.02
Fe	2.95	2.79
Ni	84.72	76.20
Total	100.00	100.00

0

Experiment 8

O.1. Pictures experiment 8

Figure O.1: Pictures experiment 8:
1) Masking bottom;
2) Masking top in nickel-bath;
3) Experiment 8 before and after



O.2. Coating thickness calculation

BAM:Eu side

$$b - a = 0,0296 \tag{O.1}$$
$$\begin{aligned} \rho &= 8,875 - 0,09375 \cdot 9 \\ &= 8,03 \end{aligned}$$
$$A = 9,125$$

$$\begin{aligned} T &= \frac{0,0296}{8,03} \cdot \frac{1}{9,125} \\ &= 4,04 \end{aligned} \tag{O.2}$$

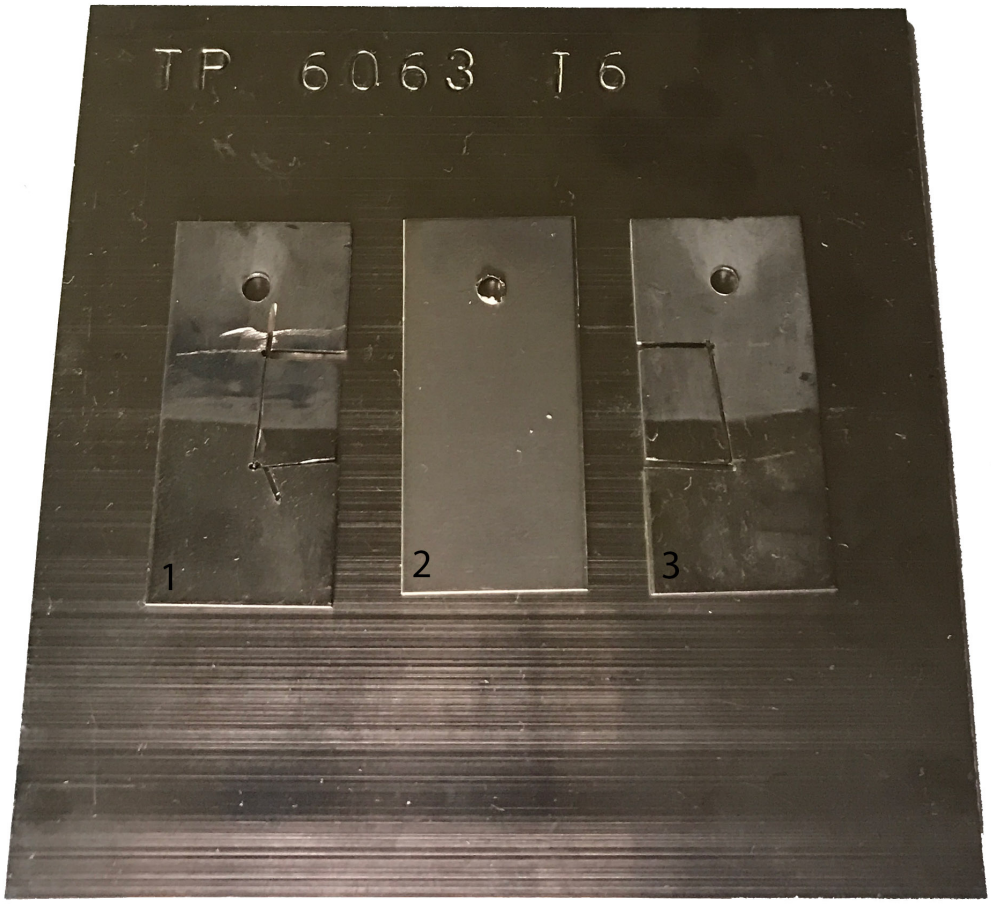
YO:Eu side

$$b - a = 0,0035 \tag{O.3}$$
$$\begin{aligned} \rho &= 8,875 - 0,09375 \cdot 9 \\ &= 8,03 \end{aligned}$$
$$A = 9,125$$

$$\begin{aligned} T &= \frac{0,0035}{8,03} \cdot \frac{1}{9,125} \\ &= 0,48 \end{aligned} \tag{O.4}$$

O.3. Color analysis

Figure O.2: Experiment 8 substrates on a nickel plate
1, 3) 316L stainless steel after nickel plating with BAM (top) and YO (bottom) particles;
2) 316L stainless steel after nickel strike



O.4. SEM & EDS results

Figure O.3: SEM & EDS pictures of experiment 8
1) Cross-section of 316L after nickel plating (BAM/YO) x100;
2) Cross-section of 316L after nickel plating (BAM side) x1000;
3) Cross-section of 316L after nickel plating (BAM side) x2000;
4) Cross-section of 316L after nickel plating (YO side) x2000;
5) Points analyzed by EDS (BAM side);
6) Points analyzed by EDS (YO side)

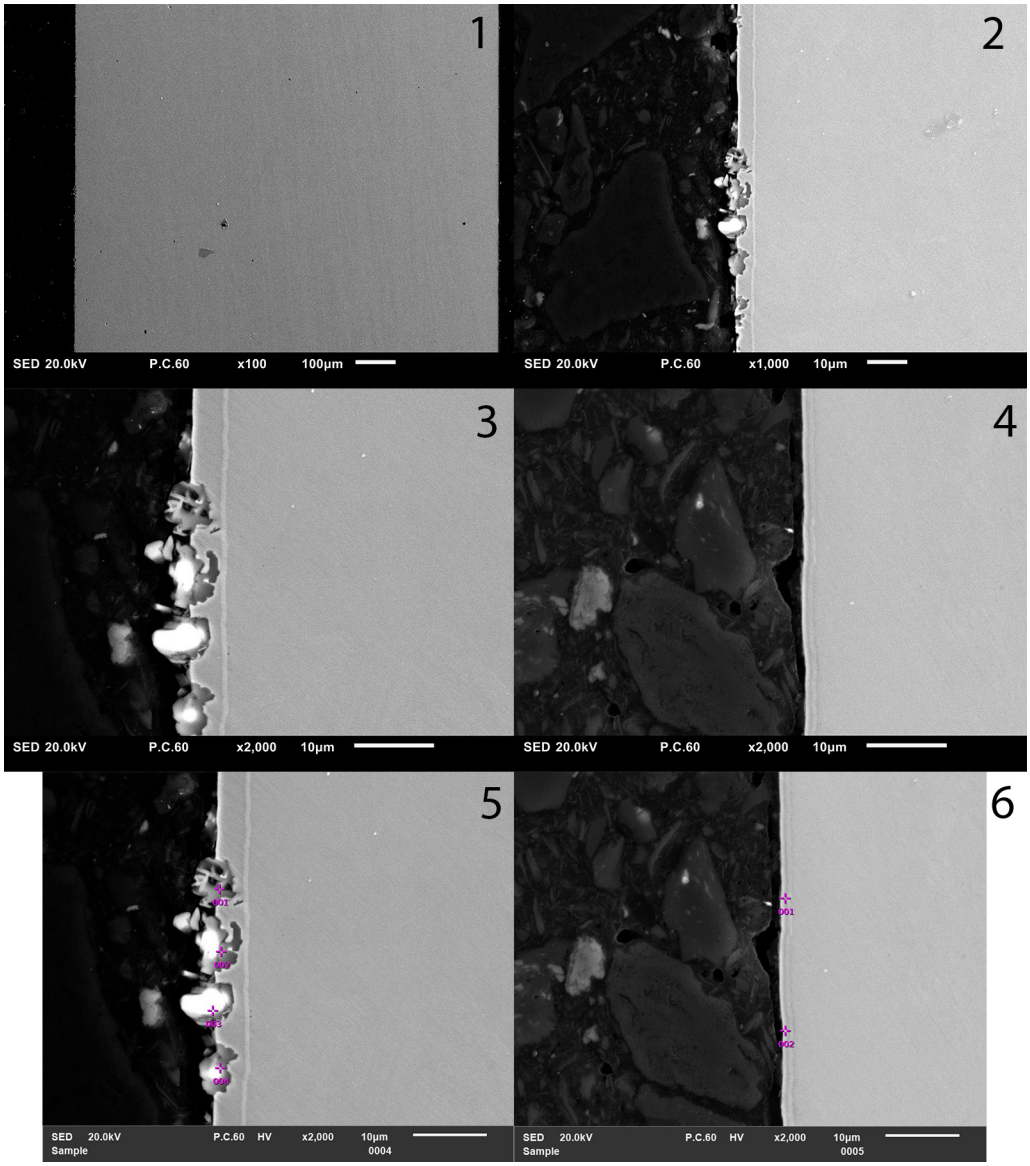


Table O.1: Exp. 8: EDS results (left BAM side; right YO side)

1

Formula	Mass[%]	Atom[%]
O	37.73	58.01
Mg	3.35	3.39
Al	35.25	32.14
Ni	9.33	3.91
Ba	13.06	2.34
Eu*	1.28	0.21
Total	100.00	100.00

2

Formula	Mass[%]	Atom[%]
O	37.63	58.57
Mg	3.64	3.73
Al	32.94	30.41
P	0.93	0.75
Ni	8.51	3.61
Ba	14.51	2.63
Eu*	1.85	0.30
Total	100.00	100.00

3

Formula	Mass[%]	Atom[%]
O	40.23	60.52
Mg	3.33	3.30
Al	36.71	32.75
Ba	18.46	3.23
Eu*	1.27	0.20
Total	100.00	100.00

4

Formula	Mass[%]	Atom[%]
O	38.93	59.37
Mg	3.32	3.33
Al	36.13	32.67
Ni	3.41	1.42
Ba	16.66	2.96
Eu*	1.54	0.25
Total	100.00	100.00

1

Formula	Mass[%]	Atom[%]
P	2.21	4.07
Cr	2.59	2.84
Mn	0.66	0.69
Fe	10.71	10.94
Ni	83.82	81.45
Total	100.00	100.00

2

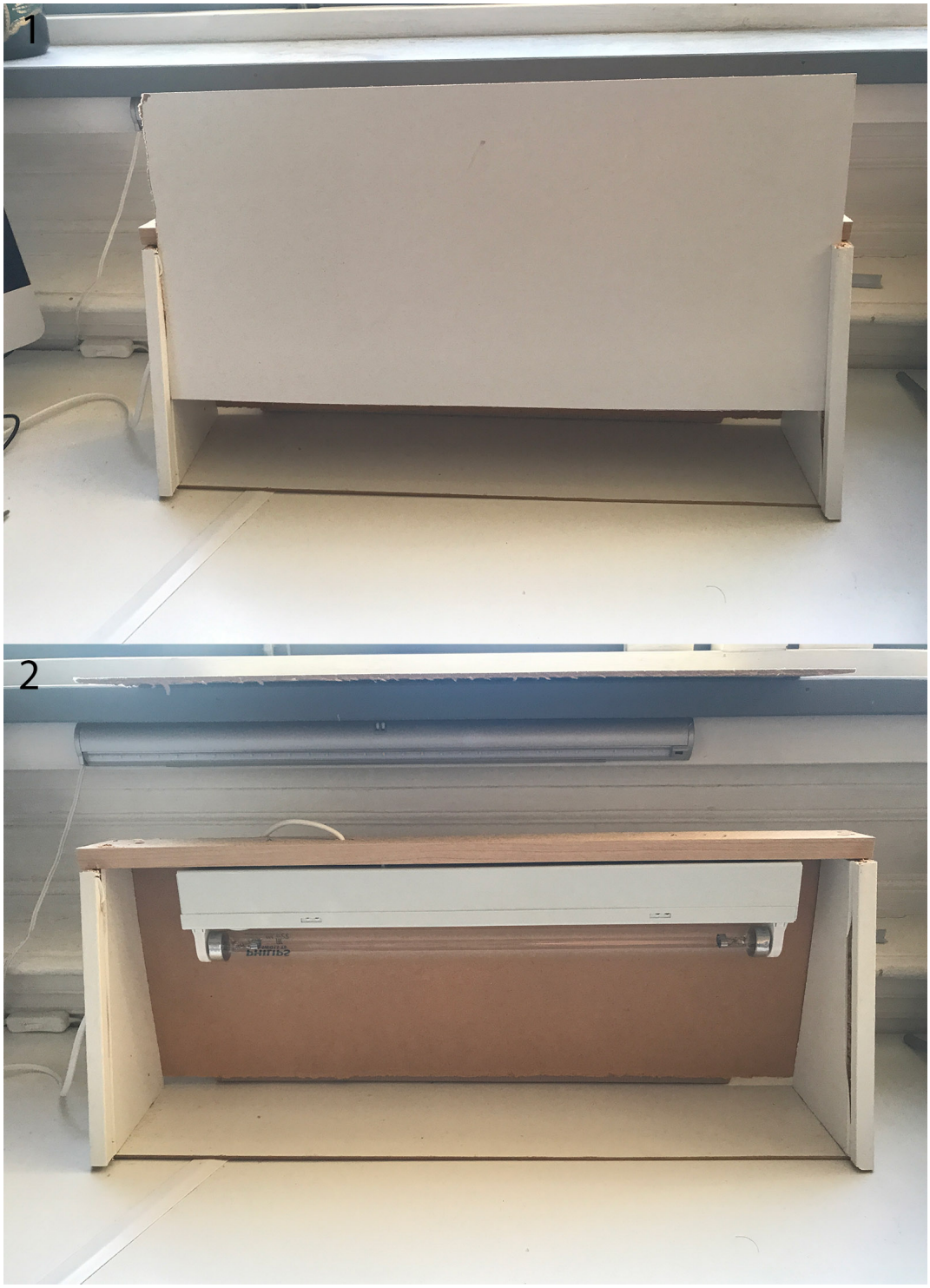
Formula	Mass[%]	Atom[%]
P	9.11	15.94
Fe	4.46	4.33
Ni	86.43	79.74
Total	100.00	100.00

P

Identification

P.1. Setup

Figure P.1: Identification setup



P.2. Matlab code

```
clear all
close all
warning('off')

filename = uigetfile({'*.jpg;*.tif;*.png;*.gif','All Image Files';...
          '*.*','All Files' });

fig = imread(filename);
s = size(fig);


low = 60;
mid = 120;
high = 180;


handles.H = figure(1);
shower = imshow(filename);
rect = getrect;
if rect(1)+rect(3) > s(2)
    rect(3) = s(1)-rect(1);
end
if rect(2) + rect(4) > s(1)
    rect(4) = s(2) - rect(2);
end
close(handles.H);


fig = imread(filename);
new_fig = fig( rect(2):rect(2)+rect(4),rect(1):rect(1)+rect(3),:);
handles.H = figure(1);
imshow(new_fig);
dlgTitle    = 'User Question';
dlgQuestion = 'Is the selection correct?';
choice = questdlg(dlgQuestion,dlgTitle,'Yes','No, reselect', 'Yes');
close(handles.H);


r = mean(mean(new_fig(:,:,1)));
g = mean(mean(new_fig(:,:,2)));
b = mean(mean(new_fig(:,:,3)));

new_fig(:,:,1) = r;
new_fig(:,:,2) = g;
new_fig(:,:,3) = b;

RGB = [r, g, b];
imshow(new_fig)

if RGB(1)> high && RGB(2) < mid && RGB(3) > high
    keuze = 'Tongs_1'
    %Blue

elseif RGB(1)< low && RGB(2) > low && RGB(3) < high
    keuze = 'groen'
```

```
elseif RGB(1)> high && RGB(2) > low && RGB(3) < high
    keuze = 'Scissors_1'
    %Red
else
    keuze = 'Unclear...'

end
```

P.3. Pictures excited substrates

Figure P.2: Pictures excited BAM:Eu substrates - left side UV-light; right side normal light

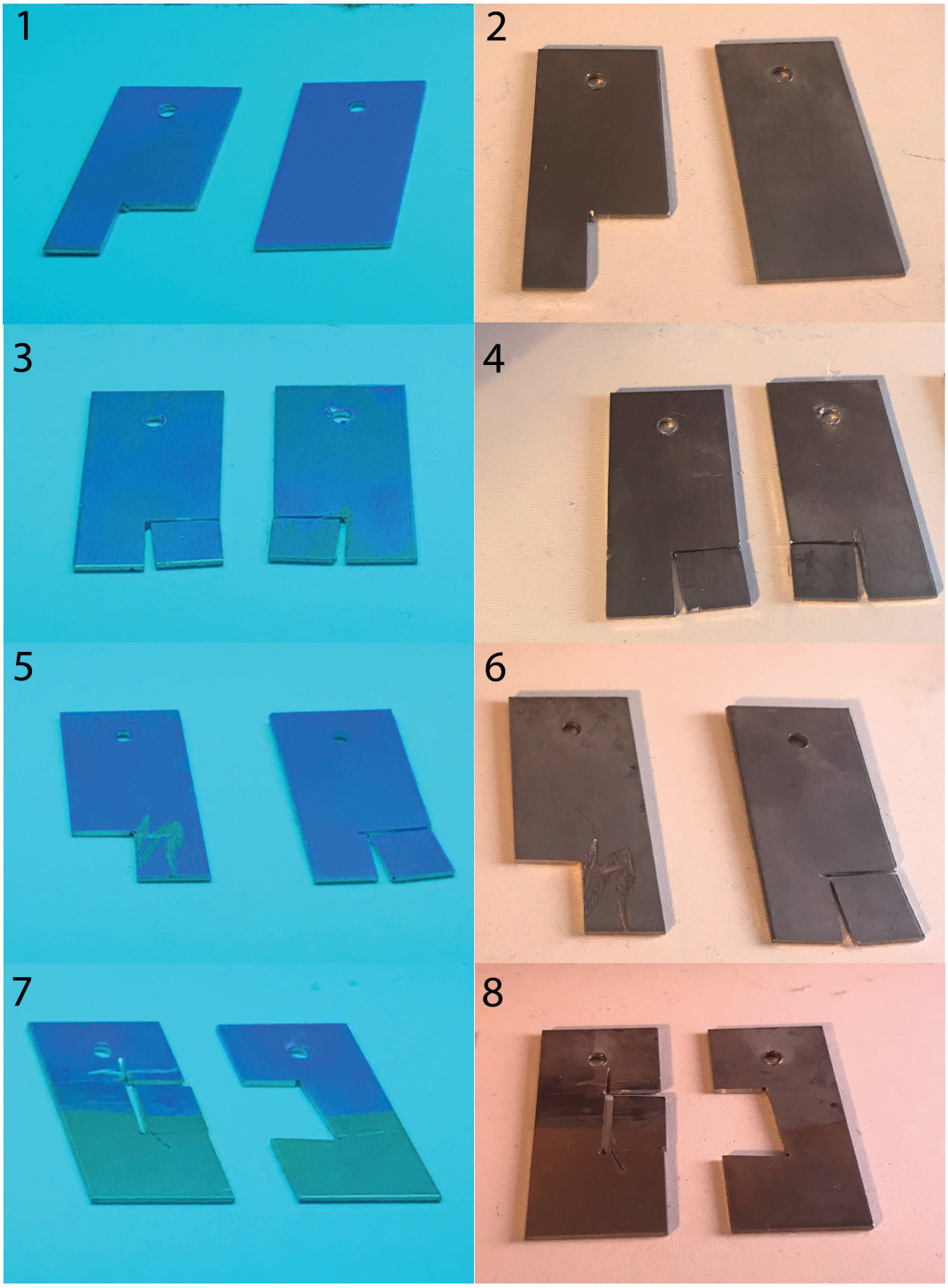
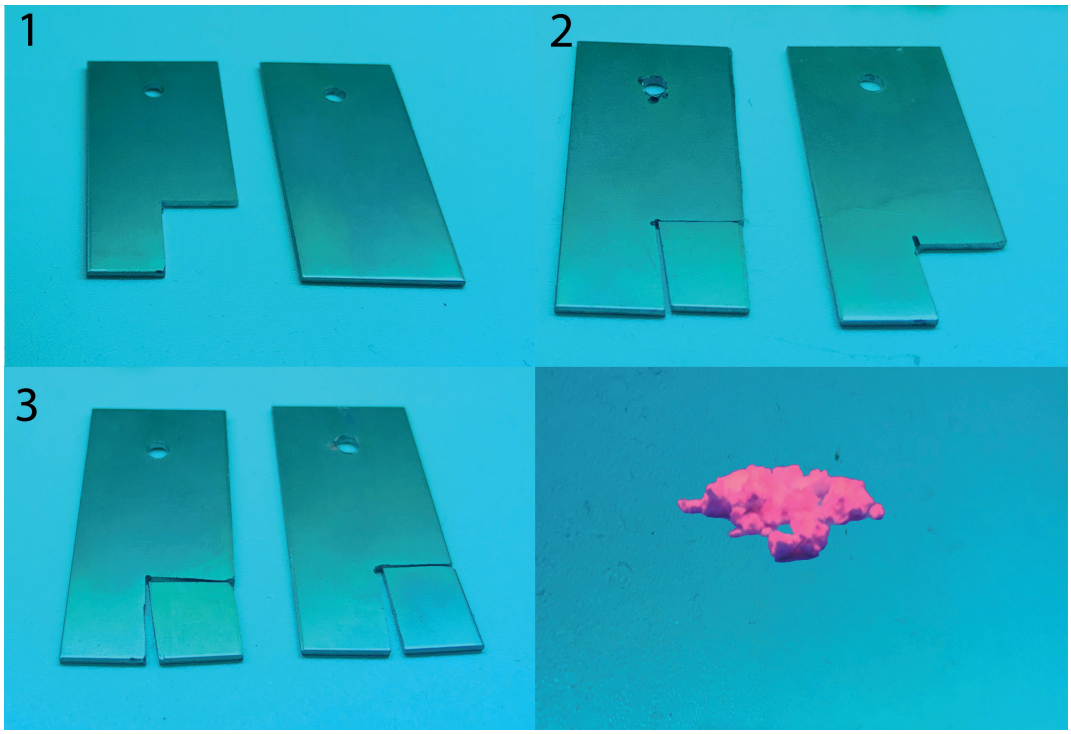


Figure P.3: Pictures excited YO:Eu substrates



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