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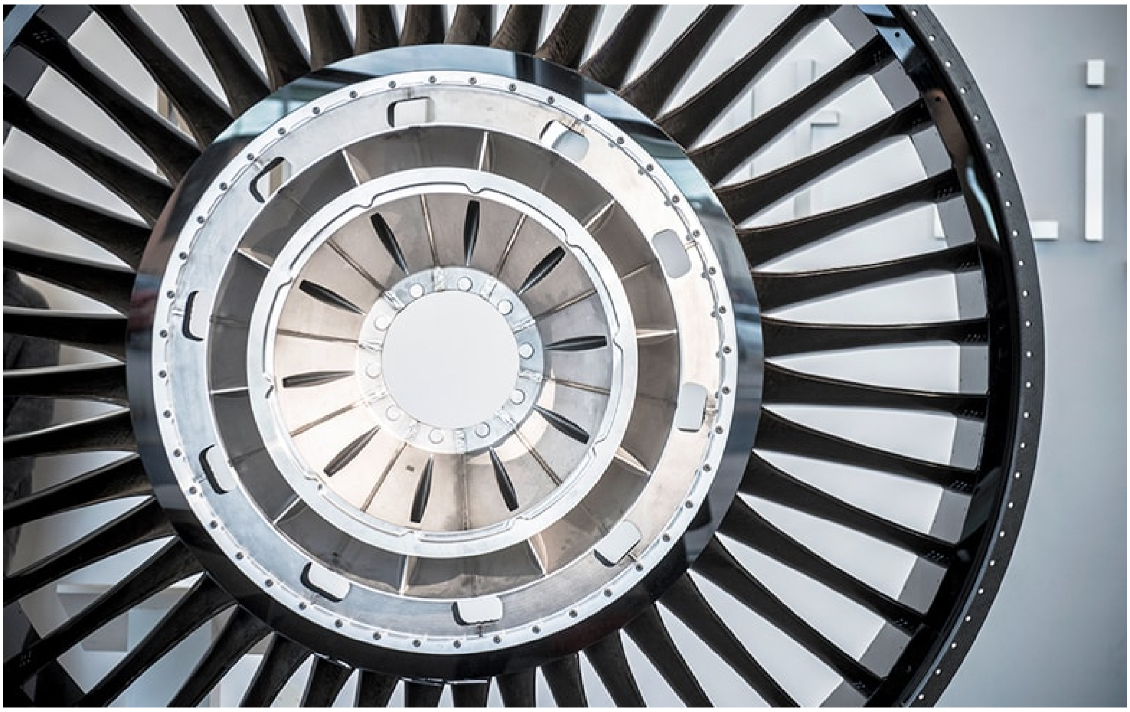

Thermal treatment of nickel plated coatings

Bachelor Thesis Chemical Engineering

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Department of Chemistry and Chemical Engineering
CHALMERS UNIVERSITY OF TECHNOLOGY
Gothenburg, Sweden 2019

Bachelor thesis 2019

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Cover: A photo of a component from an engine in an airplane.

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Abstract

GKN Aerospace is a global company manufacturing aircraft and aero-engine components. Electroless nickel plating is a commonly used technique to harden surfaces in e.g. aerospace industry. Electroless plating is more expensive than electroplating, but is often preferred due to its excellent uniformity and is often applied on products that are difficult to plate. To further harden the coated layer, a heat treatment can be done. During the heat treatment, the structure of the nickel plated layer is crystallized. The heat treatment can be disrupted in the process, which requires a chemical stripping process to remove the unfinished nickel layer. Due to that time-consuming process, additional heat treatment was investigated in this thesis work, as an alternative process to reach acceptable values of hardness. Wear ability is also of interest, but that would require more time than available to investigate.

13 plates were electrolessly nickel plated and the deposit contained mid phosphorus. The test plates were then heat treated by a program with associated temperature and time depending on plate number. Plates 1-4 were heat treated complete cycles, plates 5-9 were disrupted in the heating phase and then heat treated completely. Plates 10-13 were disrupted in the heat treatment and then heat treated completely. The results indicated that regardless of where the first heat treatment was disrupted, the additional heat treatment resulted in acceptable hardness values. Plate 4 was heat treated below the limits for this process but still achieved the required value of hardness, 900 $HV_{0.1}$. This indicated that the temperature tolerance could be extended to benefit the process. This would result in a more efficient and less time-consuming process, but first the effect of an additional heat treatment on wear resistance is recommended to be further investigated by GKN Aerospace. Then it will be possible to determine if the chemical stripping process can be replaced by an additional heat treatment.

Acknowledgements

I would like to express my sincere appreciation to GKN Aerospace, in Trollhättan, for having me as their thesis worker. First, I would like to thank Karin Ericson for being helpful to find a thesis work of interest and for introducing me. Secondly, a special thank you to my supervisor Göran Johansson who has supported me during my work. Finally, I wish to express my thankfulness to my examinor Jan-Erik Svensson.

Julia Wikman, Gothenburg, June 2019

Contents

1
Introduction

Plating is a commonly used technique in industries to form an adherent layer of metal, by a redox reaction, on surfaces. The deposited coating is applied to protect or harden the product, make the product corrosion resistant, or to improve the wear resistance etc. Wear can be explained as the loss of material during a mechanical action against a surface. Electroplating and electroless plating are two examples of methods used for plating. Electroless plating, also called autocatalytic plating, can be defined as;

> "Deposition of a metallic coating by controlled chemical reduction that is catalyzed by the metal or alloy being deposited" [1].

Electroless plating has low labor costs but is more expensive because of higher chemical cost compared to electroplating. The excellent uniformity, as seen in Figure 1.1, makes the electroless plating easier to apply, if the product is difficult to plate due to size, shape or location [2], [3]. The uniformity makes the method suitable for many different applications, e.g. plastics, printing and aerospace [4].

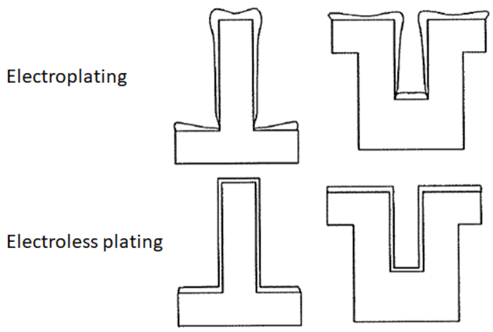

Figure 1.1: The figure shows a schematic picture of the difference in the buildup of a nickel layer in electroplating and electroless plating. The electroless nickel layer grows with equal deposition rate on all areas.

Electroless plating was invented in 1946 by Brenner and Ridell when they were investigating how addition of chemicals affected nickel plating [1]. It is difficult to produce pure nickel during electroless nickel plating therefore two common coatings produced are nickel phosphorus and nickel boron. Nickel boron usually has a higher hardness and better wear resistance than nickel phosphorous but is more expensive. Nickel phosphorous is a coating that can be used with advantages on products that require corrosion resistance and wear resistance [5]. The coatings can be divided into low, mid and high phosphorous depended on the content of phosphorus [1].

GKN Aerospace is a global company that manufactures aircraft and aero-engine components. Electroless nickel plating is a commonly used method for components, made of titanium, in the front of the aero-engine, shown in Figure 1.2, which results in higher hardness. Heat treatment is then used to fix the structure to achieve even higher hardness. At GKN Aerospace the products are required to have high hardness and wear resistance but there is no specific requirements on how resistant the layer should be to corrosion. Some factors, e.g. power failure or inaccuracies in the oven control system, can disrupt the heat treatment. This results in an incomplete heat treatment and the plated layer has to be removed by chemical stripping. It requires chemicals, e.g. nitric acid, to dissolve the layer and is a time-consuming process.

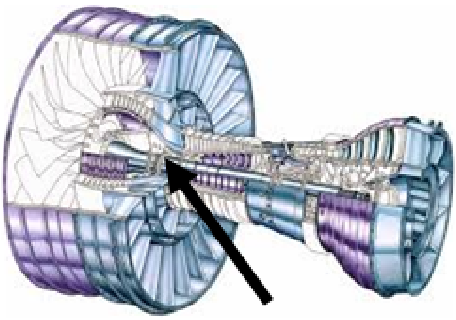

Figure 1.2: A schematic picture of an aero-engine. The arrow in the picture shows where the electroless nickel plated components occur in the aero-engine.

1.1 Aim

The aim of this report is to evaluate how additional heat treatment may affect the hardness of electroless nickel coatings containing mid phosphorus. To examine if additional heat treatment is a suitable process to avoid chemical stripping.

1.2 Limitations

Electroless nickel plating containing phosphorous will be the only plating method discussed and evaluated in this report. Hardness is the only test that will be studied in the laboratory. Wear test is commonly performed on electroless nickel-plated products to compare resistance to a specific type of wear. Literature shows that there is a correlation between hardness and wear ability [6]. The wear ability is of interest, but the test can only be performed in laboratories outside GKN Aerospace and require more time than available. This resulted in the decision of only measuring harness during this thesis work.

2

Theory

2.1 Electroless Nickel Plating

To perform electroless nickel plating, a source of nickel ions, a reducing agent, suitable complexing agent, stabilizer and energy are needed [7]. The product needs to be cleaned before plating so that the nickel ions can adhere on the surface as optimally as possible. The cleaning can be executed by different solutions in one or several steps, before entering the tank that contain the components required for electroless nickel plating [1]. The plating process continues until the catalytic surface, the surface being plated, lack contact with fresh solution. Therefore it is very important to maintain a powerful agitation in the tank and to filter the solution continuously to constantly have a fresh solution [2]. To obtain nickel ions a nickel salt is added. Nickel sulfate is the most commonly used salt but nickel chloride can also be used [8]. It is very important that no deposit is formed on the tank's walls, the filter or the pumps and therefore tanks made of e.g. plastic or passivated, addition of an outer protective layer on the material, stainless steel are preferred. In addition to passivation of stainless steel tanks, protective current is added to further protect the tank from being coated with nickel phosphorus layer [1].

The nickel layer can be classified in three groups, low (~1-4%), mid (~5-9%) and high (~10-13%) regarding the content of phosphorous. Mid-phosphorous contains 50% nickel ions, Ni^{2+}, and 50% nickel phosphide, Ni_3P. High-phosphorus have higher content of Ni_3P in a Ni^{2+} matrix and low-phosphorus has reversed content [1]. The phosphorus content and how it is included in the coating is important for its properties. Low-deposit have a higher hardness, as plated, compared to high-deposit, as shown in Table 2.1, due to a high number of grains. A grain is the area in the deposit where the face centered cubic, FCC, structure is able to be maintained. If the deposit contains many small areas of grains, it is considered to have a crystalline structure [7]. Low-deposit, as plated, has often a porous structure which is an amorphous structure with cracks and holes. A phosphorous content over 10% and under 0.05% can be assumed to be completely amorphous [2].

Table 2.1: The table explain how the different phosphorus content in the nickel coatings, as plated and after heat treatment effects the hardness. Vickers hardness is a method that measures hardness and H$V_{0.1}$ is when the test has been executed with a 100 gram weight [8].

P content	**H$V_{0.1}$, as plated**	**H$V_{0.1}$, HT 400°C for 1h**
1.5-3%	550-700	750-1000
4-9%	550-580	900-1000
10-14%	480-500	900-950

2.1.1 Reducing Agent

The reducing agent is the component that is controlling the properties of the nickel plated layer. Sodium hypophosphite is usually used as reducing agent due to lower costs, easier to control and higher corrosion resistance compared to the reducing agents containing boron [2]. Energy is added to the plating solution by heating up the solution to an optimal temperature within the limit, 85-95°C, to activate the reactions. If the temperature of the process solution is high enough the phosphite ion reacts with water, as shown in the following base equation:

$$H_2PO_2{}^- + H_2O \rightarrow H(HPO)_3{}^- + 2H^\circ \text{ [8].} \tag{2.1}$$

Equation 2.1 produces orthophosphite ions, a byproduct, and atomic hydrogen that can react in three different ways. The first reaction, Equation 2.2, produces hydrogen gas, as a byproduct from the nickel plating process. The nickel ions can be reduced as the reducing agent releases electrons, or by the atomic hydrogen, shown in Equation 2.3, respectively. Sodium hypophosiphite and atomic hydrogen can produce phosphorous, as shown in Equation 2.4 [8].

$$2H^\circ \rightarrow H_2 \tag{2.2}$$

$$2H^\circ + Ni^{2+} \rightarrow Ni^\circ + 2H^+ \tag{2.3}$$

$$H_2PO_2{}^- + H^\circ \rightarrow P^\circ + H_2O + OH^- \tag{2.4}$$

The tank containing sodium hypophosphite, can be either acidic or alkaline. An acidic solution has a higher deposition rate, is easier to control and is more stable compared to an alkaline solution [2]. Decreased pH in the solution increases the phosphorus content in the deposit [1].

2.1.2 Additives

The usage of complexing agents control the reaction, the deposition rate and keep the nickel ions in the solution. The complexing agent can also be used as a buffer to prevent the pH to descent, as hydrogen is formed [8]. A stabilizer can be used to lower the spontaneous precipitation by unwanted particles, e.g. dust, in the solution [2]. The stabilizer can contribute to a decreased deposition rate and it can be required to add accelerators, usually organic additives, to increase the rate. To avoid pitting, holes in the surface of the deposit caused by the bubbles of the hydrogen gas, wetting agents are used [8]. Byproducts from the reaction like orthophosphite, hydrogen ions and dissolved metals, can affect the tank characteristics, as pH and deposition rate [2].

2.2 Thermal Treatment

To further improve the hardness of the coating, heat treatment can be done. The hardness of the nickel layer is depending on the content of phosphorous, the time and temperature of the heat treatment, as shown in Figure 2.1, [7]. During the heat treatment, structural changes in the deposited layer starts to occur at the temperature between 220°C-260°C [2]. At 300°C the amorphous structure starts to crystallize. At 375°C the crystallization is completed. If the deposit is heated during a longer time, a risk of overaging occurs. Overaging occurs after the hardness value peaks then descends due to constitution of Ni_3P, as shown after the marked line in Figure 2.1 [7].

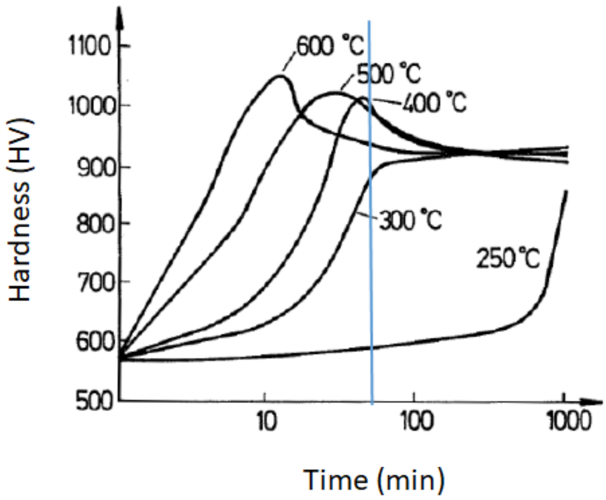

Figure 2.1: A modified graph that shows the correlation between time, hardness and temperature. The marked line indicates that overaging occurs after approximately 70 minutes for a heat treated deposit at 400°C [8].

To obtain the highest hardness, a temperature over 400°C is necessary, as shown in the marked area in Figure 2.2. As described earlier in this report coating containing low phosphorus has a higher hardness, as plated, but after heat treatment it is reversed. It is the phosphorous content that makes it possible to affect the crystallization that has direct impact on the hardness, as shown in Table 1.1 [8].

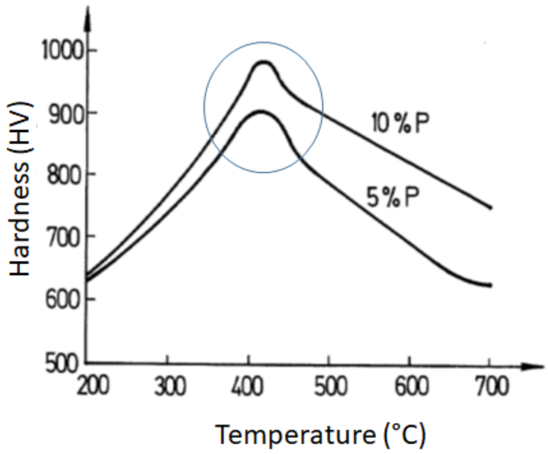

Figure 2.2: A modified graph that shows the correlation between hardness and temperature for a coatings containing of 5% and 10% phosphorous respectively. In the marked area the highest hardness is accomplished at approximately 400°C [8].

2.3 Vickers Hardness

Vickers Hardness is a method used to measure hardness of different materials e.g. coatings. Vickers Hardness can be divided as micro and macro Vickers. Micro Vickers is used for thin materials with limited area with a test load of 1-1000g. Macro Vickers is used for thicker and more rough materials with a test load of 1-120kg. Equation 2.5 is used to calculate the Vickers hardness value where the d is mean value of the diagonal (mm), L is load (kg) and the top angle of the impression body is 136° [9]. To be able to measure Vickers hardness a thickness of 100 μm is required on the deposited layer.

$$\frac{1.8544 \cdot L}{d^2} \tag{2.5}$$

As mentioned previous the hardness increases if the deposit is heat treated due to crystallization. The Figure 2.3 confirm that a deposit that has been heat treated (4) has a higher value of hardness than a deposit that has not been heat treated (3) [7].

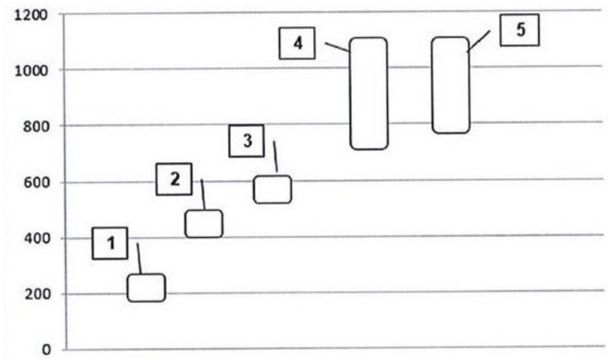

Figure 2.3: The figure is developed at GKN Aerospace and shows a schematic picture of hardness ($HV_{0.1}$) value of 1: Ductile nickel plating, 2: Hard nickel plating, 3: Electroless nickel phosphorus layer, as plated, 4: Heat treated electroless nickel phosphorus plating and 5: Hard chromium plating.

2.4 Process

The electroless nickel plating process at GKN Aerospace consist of the main steps shown in Figure 2.4. The first step, cleaning, has the main purpose to clean the product with alkaline solution so that the nickel plating is easier attached to the surface. The product is cleaned from chemicals and dried, then masked with e.g. durable tape to protect the area of the product were the nickel plating should not occur. Pretreatment can be done in one or several steps were the oxides are removed with e.g. blasting or acid. The oxides are not electrically conductive and works as an isolating layer and inhibits the nickel ions ability to attach to the surface.

Metal strike is a step were a thin layer, usually copper or nickel, is plated on the product to form a ground layer were it will be easier for the main plating to be attached later in the process. The plating step is described in section 2.1 and how long the product are supposed to be in the bath depends on the age of the bath. An older bath has a slower plating rate and a control plate is used to periodically measure the thickness of the deposition to control the deposition rate. If the deposition rate is to low there can be problem with some parameters in the tank, e.g. pH or temperature, and actions needs to be done. At GKN Aerospace, mid phosphorus is used to plate products made of titanium, which is expensive and hard to plate. For this thesis work only the deposit layer should be evaluated and therefore, test plates made of steel is used. This is because titanium is more expensive and harder to plate. Between all the steps, there is usually a cleaning step to remove the remaining chemicals and to prepare the product for the next step.

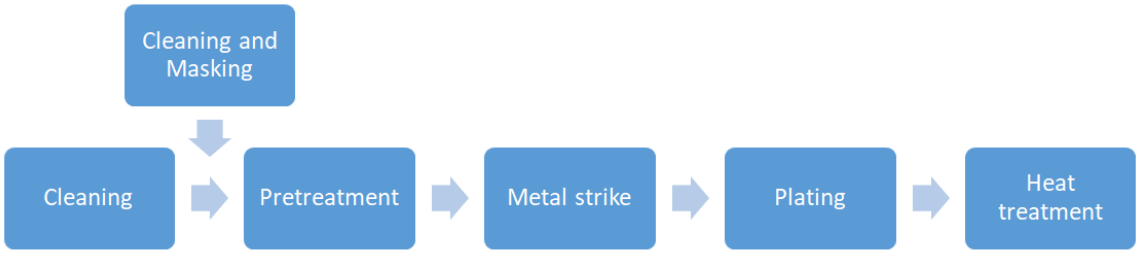

Figure 2.4: A simplified schematic picture, developed at GKN Aerospace, of the electroless nickel plating process at GKN Aerospace is shown in the figure.

The first tank, in the cleaning and masking step, contains water at room temperature, approximately 23°C, and the two other tanks have a temperature of 50°C. The nickel plating tank requires a temperature of approximately 90°C and a pH value in the range of 4.7-5.2. The solution in the tank needs to reach correct parameters before the plating process can be started. Laboratory tests are made to control the concentration of sodium hypophosphite and nickel ions and a measuring device is used to control the pH. A nickel meter is used to measure the nickel concentration and if a difference occurs, compared to the calculated value, a signal is sent to the pumps that will correct the chemicals to get a value within the limits. In order for electroless nickel plating to occur, a specific bath load is required. If the product does not fit in the range a metal roll is used in the bath to fulfill an accepted value of bath load. An electrical current is used to help activate the reaction in the tank. The positive pole is immersed in the solution and it is important that it does not touch the product or the walls of the tank. After the plating process is done, the temperature in the plating tank is lowered to below 70°C so that the chemicals do not decompose.

When the test plates are plated, they are washed in a tank containing hot water and then dried by blowing on them with clean compressed air. Then they are heat treated in an oven, explained in section 2.2. The heat treatment curve is explained in Figure 2.5 and three phases are distinguished. In the first phase, heating phase, the temperature is programmed to increase from the start temperature in the oven to the temperature of the heat treatment as rapid as possible. In the next phase the heat treatment is maintained at 400°C ± 6°C for one hour. During the last phase, the cooling phase, the temperature will decrease. GKN Aerospace has a requirement to achieve a hardness of at least 900 $HV_{0.1}$ for the finished product.

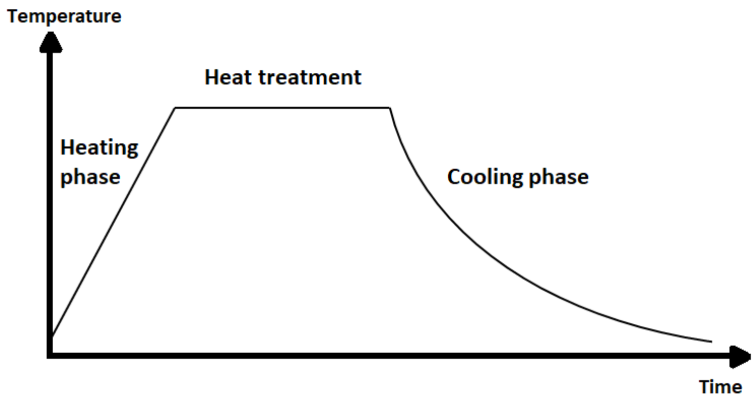

Figure 2.5: A schematic graph of the heat treatment curve with temperature on the y-axis and time on the x-axis. The graph is developed at GKN Aerospace.

3

Methods

3.1 Electroless Nickel Plating

A titration of the solution in the plating tank is performed to control the concentration of both nickel ions and sodium hypophosphite. The calculated laboratory value of nickel was used to adjust the nickel meter. The tests are performed at the middle of the plating as well for precaution because the plating tank has not been used for a long time.

Place one test plate in the cleaning tank containing concentrated hydrochloric acid for five seconds. Dip the testing plate in the cleaning series of three tanks. Let the prepared plates be in the last cleaning tank until all the plates and the control plate are done. Pretreatment and metal strike is not required because of plates made of steel is used instead of titanium. Put all plates in the nickel plating tank, see Figure 3.1, at the same time and write down the time. Mid phosphorus nickel deposit increases until the limit of approximately 130 μm is reached and then the plates are removed from the solution.

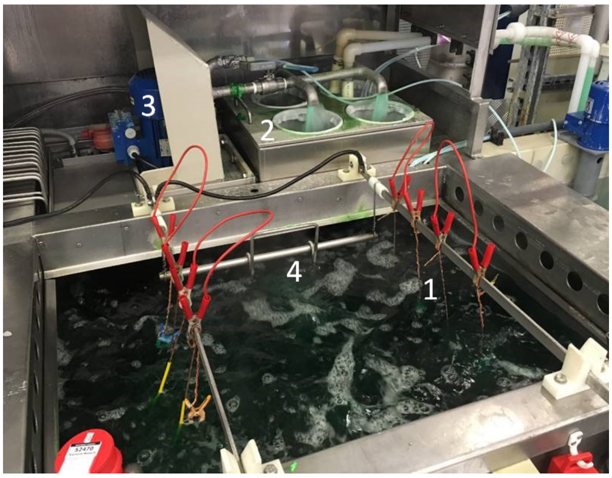

Figure 3.1: A picture showing the test plates being plated with electroless nickel (1). The filters (2), the pump (3) and the bath load (4) are also showed in the picture.

3.2 Thermal Treatment

Clean all the plated test plates with acetone, mark them with a vibro-pen and put them into plastic zip-bags. Drill a hole at one edge of all the plates and use a metallic hook in the oven to attach the plate during the heat treatment. Depending on which plate number that is heat treated, the program is programmed with the associated temperature and time. Heat treatment A, plate 1-4, is programmed for complete programs at different temperature and sets of heat treatment, shown in Table 3.1. Heat treatment B, plate 5-9, is programmed for a complete 400°C program for 1 hour but is disrupted in the heating phase at the associated temperature for each plate, shown in Table 3.2. The temperatures are chosen with continuous interval from 300-400°C. Before 300°C the oven is not calibrated and therefore this interval was chosen. Heat treatment C, plate 10-13, is also programmed for a complete 400°C program for associated time for each plate, shown in Table 3.3. Plate 12 and 13 are used to see if the hardness is accepted outside the time interval of 60min ± 10 min.

Table 3.1: A table explaining how the thermal treatment of the heat treatment A will be executed. C stands for complete and HT stands for heat treatment.

Plate	HT 1	HT 2	HT 3	Comments
1	C 400°C	-	-	Reference
2	C 400°C	C 400°C	-	Reference
3	C 400°C	C 400°C	C 400°C	
4	C 380°C	-	-	To test the limits

Table 3.2: A table explaining how the thermal treatment of the heat treatment B will be executed. I stands for incomplete, stop T is the temperature when the heat treatment was stopped and raised T is the temperature the oven reached after the heat treatment was stopped.

Plate	HT 1	HT 2	Stop T	Raised T
5	I 280-320°C	C 400°C	302.1°C	304.1°C
6	I 320-340°C	C 400°C	325.1°C	327.1°C
7	I 340-360°C	C 400°C	350.1°C	351.9°C
8	I 360-380°C	C 400°C	370.4°C	372.0°C
9	I 380-400°C	C 400°C	390.1°C	391.8°C

Table 3.3: A table explaining how the thermal treatment of the heat treatment C will be executed.

Plate	HT 1	HT 2
10	I 15 min	C 400°C
11	I 35 min	C 400°C
12	I 49 min	C 400°C
13	I 71 min	C 400°C

3.3 Vickers Hardness Test

Use a cutter to cut a piece, 3cm from each side, from the test plate. Use a protecting rubber material under the plate and a load to avoid vibrations during the cut. The cutting process takes approximately 30 minutes. Clean the plate and put it in an oven holding 70°C. Use the embedment machine and place the plate in the pipe. Use a plastic holder to help the plate standing up. Add bakelite powder so that the plate is covered. Press start and wait for 15 minutes. The machine melts the bakelite and then cool it to get the form, shown in Figure 3.2. Polish the form until the plate is shown in the top of the form. Repeat the steps for all test plates.

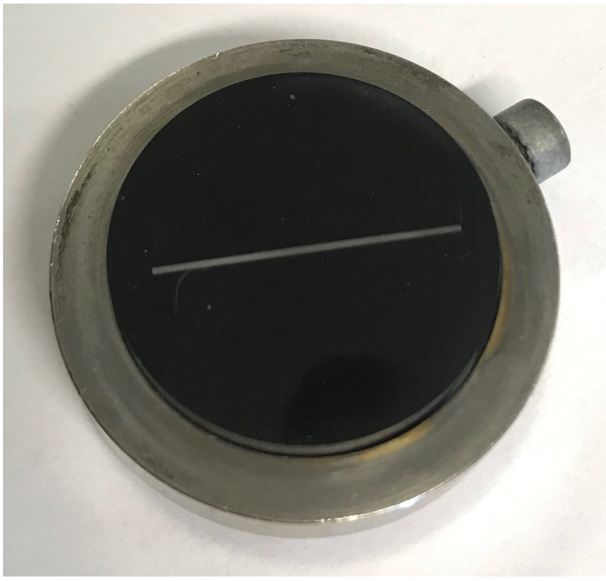

Figure 3.2: The picture shows the finished form after polishing the embedded form. The metal ring around the form is used to hold the form in place during polishing. The metal plate in the middle is the cross-section where the diamond pyramid, in the Vickers hardness test, will be pressed.

Use micro Vickers hardness test (100g) to test the cross-section of the plates. Use a microscope to see the nickel plated layer and place the marker in the middle, shown in Figure 3.3 A. Make a mark on five different even location on the top and bottom layer. Mark the edges on the pressed area, shown in Figure 3.3 B, and calculate the average diagonal. Use Equation 2.5 to calculate the hardness. Ten values of hardness will be given from the test. Write down the average, minimum and maximum values.

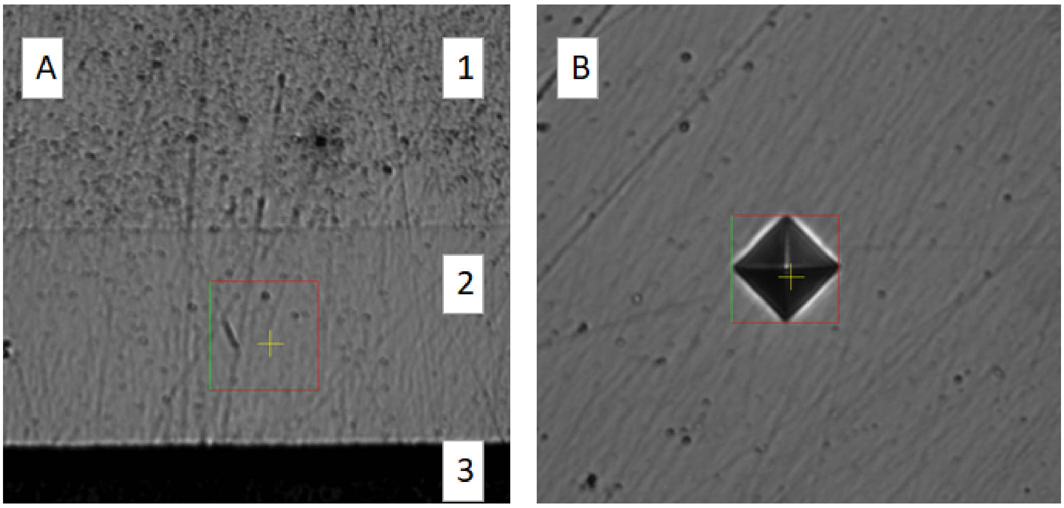

Figure 3.3: A: The picture shows the test plate (1), the nickel-plated bottom layer (2) and the bakelite layer (3) in the form. B: The picture shows the pressed area from the diamond pyramid.

4

Results/Discussion

The results from the Vickers hardness test, from heat treatment A, B and C, are presented in Table 4.1. Heat treatment A was heat treated with complete cycles. Heat treatment B was interrupted during the heating phase, then heat treated with a complete cycle. Heat treatment C was interrupted during the heat treatment, then heat treated with a complete cycle. The results indicate that an additional heat treatment can reach acceptable values of hardness without overaging occurring and regardless of where on the heat treatment curve the first heat treatment was interrupted. At GKN Aerospace a hardness of at least 900 $HV_{0.1}$ is necessary and though the results from the hardness measurement differs, all values are over the limit.

Table 4.1: The table presents the hardness values, from the Vickers hardness test ($HV_{0.1}$), of the thirteen test plates. The hardness (avg) is an average value from the 10 marks that were made during the test.

Heat treatment	Plate	Hardness (avg)	Min value	Max value
A	1	1028.1	1001	1059
A	2	1003.6	983	1020
A	3	1001.2	983	1020
A	4	1006.6	989	1020
B	5	1012.1	1001	1026
B	6	1004.6	995	1013
B	7	997.2	971	1020
B	8	1005.9	989	1026
B	9	1003	971	1026
C	10	999.2	989	1007
C	11	1004.7	995	1020
C	12	1001.6	989	1013
C	13	998	977	1013

The program of the heat treatment had no set start temperature and the temperature of the oven was different when the test plates were taken out. When the temperature was below 100°C, the oven was either opened, so the temperature dropped faster, or not opened until a temperature of approximately 50°C was reached. How the cooling phase was executed in the oven depends on whether a new heat treatment was planned to start directly after or the next day. Due to no reaction in the coated layer under 220°C this should not affect the results significantly and the results should be comparable. A difference in the colors of the test plate in heat treatment A, B and C are shown in Figure 4.1. The difference in the colors is depending on the oxides that are produced during heat treatment, but the difference in color has no effect on the result of the hardness test.

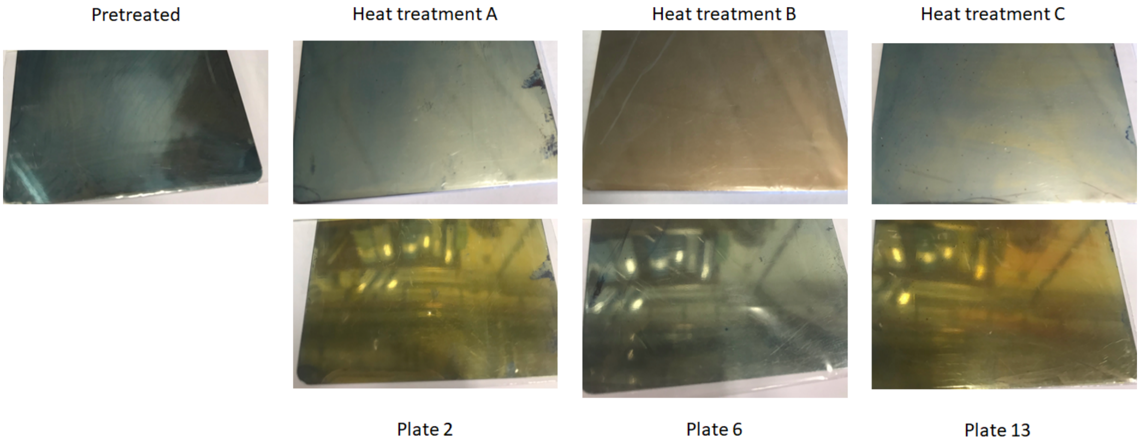

Figure 4.1: The figure shows pictures of the color of the test plates at different stages. The color of the pretreated test plate is silver. The color of the test plates, in the HT A and C, are silver after HT 1 and gold after HT 2. The color in HT B are silver/brown after HT 1 and silver/blue after HT2.

The electrolessly nickel plated layer is not homogeneous and can have cracks and scratches which can result in differences in the hardness test. This test was executed on thin test plates made of steel. In the process this should be performed on larger, more massive products made of titanium. This could mislead the result because it takes longer time to heat up a larger product. If the heat treatment was interrupted at 300°C, the test plate would probably have the same temperature. The product would have a lower temperature than the oven and this could be avoided if the temperature of the product is measured as a reference temperature.

The limits of the temperature of the oven is $400°C$ ±6°C but literature value imply that the crystallization is finished at $375°C$. The result of plate 4 imply that the temperature tolerance might be able to be extended, assuming that the wear resistance is not affected. A more extended limit makes it easier to maintain the correct temperature of the oven. The result from a previous Vickers hardness test, shown in Appendix A.1, confirms that the limit is reached with a temperature in the interval between 300-$350°C$.

To further investigate the impact of additional heat treatment on electroless nickel plated coatings, the effect of variation in time and temperature on the additional heat treatment, could be examined. Also, the effect of additional heat treatment on wear resistance should be examined by GKN Aerospace.

5 Summary

Additional heat treatment was investigated, in this thesis work, to see if it is possible to replace the stripping process at GKN Aerospace. Thirteen test plates were electrolessly nickel plated and then heat treated to achieve a higher hardness. The plates in heat treatment A were heat treated for complete cycles. The plates in heat treatment B were interrupted in the heating phase and in heat treatment C the plates were interrupted in the heat treatment. The plates in heat treatment B and C were then heat treated with a complete cycle. The Vickers hardness ($HV_{0.1}$) values of the thirteen test plates indicate that an additional heat treatment satisfy the limits of hardness at GKN Aerospace. Irrespective of where on the heat treatment curve the first heat treatment is interrupted, the hardness value reached above the required limit of 900 $HV_{0.1}$. Plate 4 was heat treated for a complete cycle of 380°C and the result, together with a previous Vickers hardness test, Appendix A.1, implies that an extension of the temperature tolerance of the oven is possible, assuming that the wear resistance is not affected by the additional heat treatment. This would result in a more efficient and less time-consuming process. To confirm that an extension of the temperature tolerance is possible and to replace the chemical stripping process with an additional heat treatment, the effect of an additional heat treatment on the wear resistance is recommended to be further investigated by GKN Aerospace.

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A
Appendix 1

Table A.1: Values of hardness (H$V_{0.1}$) from a previous test at GKN Aerospace. The test showed the values of hardness during an one hour heat treatment at different temperatures.

Test	Hardness (avg)	Min value	Max value
250°C, 1h	721	701	736
300°C, 1h	813	731	899
350°C, 1h	997	989	1006
450°C, 1h	945	928	966