STUDIES ON DYNAMIC ELECTROLESS NICKEL PLATING ON THE INTERIOR OF INCONEL

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SUMMARY

A nonmetallic flow system was used to "nickel-plate" the inside of straight and bent sections of $\frac{1}{4}$-inch Inconel (15 Cr, 5 Fe, Balance Ni) tubing by an electroless plating process. In this process nickelous chloride is catalytically decomposed under controlled conditions without the use of an external emf. The as-plated coating is an amorphous nickel-phosphorous alloy which, upon heat treatment, changes to a two-phase crystalline structure of nickel and nickel phosphide.

Previous work had established the feasibility of this general method. The purpose of this investigation was to determine the type of bath and conditions under which a better coating could be obtained and to apply this to a bent section.

Of the three plating baths studied the following bath under the conditions indicated showed the best results:

Nickel Chloride ($NiCl_2 \cdot 6H_2O$)	21 g./l.
Sodium Hypophosphite ($NaH_2PO_2 \cdot H_2O$)	24 g./l.
Sodium Acetate ($NaC_2H_3O_2$)	10 g./l.
Flow Rate	30 - 50 cc/min
Temperature	99°C
P H	5

This bath exhibited a rate of deposition of 0.00067 inches per hour giving a sound, coherent coating on the interior of the

12-inch long tube. The plate was uniform and non-porous.

A tube bent 180° using a $\frac{1}{2}$-inch bending radius was plated. A continuously thin, uniform plate was produced around the bent area.

Aging curves were plotted from heat treated sections of the plated tube. The sections were treated at five temperatures between 250° and 600°C at times ranging from one-half to forty-eight hours. The curves showed a precipitation hardening effect with maximum hardness obtained after two hours at 400°C.

Metallographic studies were made on the as-plated and heat treated samples and selected photomicrographs were taken of the plate.

I. INTRODUCTION

Interest in electroless "nickel plating" has been growing in recent years. The process is a unique method of chemical plating which differs from the normal electrolytic displacement process in that thick, non-directional coatings can be built up in the absence of an external emf.

Experience gained from over three years in commercial plating shops has proved the success of this new method of nickel plating. Results of previous research show that electroless nickel has very good corrosion-resistant properties in certain environments and responds to precipitation-hardening heat treatment.

A method for nickel plating heat exchanger tubes electrolessly giving a coherent and heat treatable coating would be very desirable. The purpose of this investigation was to study the plating of various plating baths on the interior of ¼-inch Inconel (15 Cr, 5 Fe, Balance Ni) heat exchanger tubing using a dynamic system, and to study metallographically and by hardness determinations the heat treatment characteristics of the nickel-phosphorous alloy coating.

II. REVIEW OF THE LITERATURE

Although theoretical concepts of electroless plating have been known since 1846, Brenner and Riddel (1)*, in 1946, developed the first practical method for the deposition of nickel without the use of electricity. This controllable catalytic reduction process is theoretically based on the following chemical equations:

$$n(H_2PO_2)^- \xrightarrow[\text{catalyst}]{-2nH} n(PO_2)^-$$

$$2H^+ \longrightarrow H_2$$

$$(PO_2)^- + H_2O \longrightarrow H(HPO_3)^-$$

$$Ni^{++} + 2H \xrightarrow[\text{catalyst}]{} Ni^o + 2H^+$$

$$(H_2PO_2)^- + 3Ni^{++} + 7H \xrightarrow[\text{catalyst}]{} Ni_3P + 5H^+ + 2H_2O$$

Temperature and concentration conditions were determined by Brenner and Riddel which inhibited the spontaneous and uncontrollable reduction of a nickel salt in solution to "black" nickel. The reaction did not occur spontaneously in all systems, it was found, under controlled conditions of temperature, pH, flow rate, and filter arrangement. Only such catalytic surfaces as steel, iron, gold, cobalt, aluminum, nickel, or "activated" non-catalytic surfaces were found to cause the deposition of the nickel-phosphorous alloy.

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* Numbers in parenthesis refer to bibliography appended to this report.

General Physical Properties of the electroless Plate

Nickel deposits of this type are not pure nickel but contain considerable amounts of phosphorous. Qualitative tests indicate that the coating contains at least three percent phosphorous with a maximum of 11.4% before losing its metallic appearance (2).

The adhesion of the deposits to the base metal is such that the deposits will not flake off, but in some cases will detach on flexing. Steel, aluminum, brass, copper, nickel, gold,cobalt,silver, and palladium exhibit good adherence. The degree of adhesion depends upon the surface treatment given the metal prior to plating (3).

The plating rate under usual operating conditions is between 0.0002 and 0.0005 inches per hour but is dependent upon the bath pH. There does not appear to be any limit to the potential thickness which is proportional to the amount of hypophosphite present. The thickness can be controlled easily, and can be held to within ten percent of the average thickness regardless of the surface contour. Electroplated coatings often have tolerances of over 100 percent (4).

The hardness of electroless nickel is greater than that of electroplated nickel. The plate hardness increases with the percent phosphorous, but long-time heat treatments at elevated temperatures (over aging) causes a decrease in hardness (3).

The corrosion resistance of electroless nickel approaches that of wrought nickel. The porosity of electroless nickel can be decreased by heat treatment, but even without heat treatment, the porosity is less than that of electroplated nickel (3). For adequate protection the

thickness requirements are as follows (4):

Indoor	0.0005 inch
Mild Outdoor exposure	0.0010 inch
Severe Outdoor exposure	0.0015-0.0020 inch
Abrasion resistance	0.0010-0.0030 inch

Plating Operations

Although additional preparation is sometime necessary, metal surface treatment for electroless plating is essentially the same as for electroplating. Sulfuric acid and zincate dips are used for steels and aluminum, respectively, while one-minute bright dip of palladium chloride (0.02 g./l.) and hydrochloric acid (200 g./l.) is required for copper and its alloys (3).

For continuous plating a pump, heat exchangers, and a filter is required if plating is carried out in a tank of glass, porcelain, enamel, or ceramics. Uniform external tank heating is required and can be obtained by using a steam or direct gas heated, hot-water jacketed vessel.

Nickel plate is rapidly acquired soon after immersion in the bath for catalytic metals; non-catalytic metals require "activation" through contact with a piece of catalytic metal such as steel or aluminum. The maximum area of plating surface is recommended to not exceed 100 square inches per gallon of plating solution. It is not necessary to continuously filter the bath, but is is recommended that this be done at least every four days. The solution should be kept at room temperature when not in use (5).

Electroless nickel is reported to have been plated on glass and phenolics although this phase of electroless plating is still being developed. There are limitations to this application - the principal ones being (6):

1) the softening of the plastic at the plating bath temperature,

2) the necessity of sensitizing the non-catalytic surface and,

3) the possibility of organic contamination.

Typical Formulations

Practical electroless nickel plating baths are classed as either acid or alkaline.

ACID BATH: The acid bath developed by Brenner and Riddell produced semibright nickel coatings. The bath's composition and plating characteristics are (7):

Nickel Chloride ($NiCl_2 \cdot 6H_2O$)	4.0 oz. per gal.
Sodium hypophosphite ($NaH_2PO_2 \cdot H_2O$)	1.3 oz. per gal.
Sodium citrate ($Na_3C_6H_5O_7 \cdot 5\frac{1}{2}H_2O$)	1.3 oz. per gal.
Temperature	194°F
pH	4 to 6
Deposition rate	0.0002 inches per hour

Excellent results were reported by H. J. West in his investigation of barrel plating. Barrels made of polystyrene or Lucite can

be used satisfactorily at the plating temperature. The conditions and compositions of West's plating bath for an 80*gallon tank are (8):

Nickel chloride	800 fl. oz. of a 5-lb. per gal.soln.
Sodium hypophosphite	100 oz.
Sodium citrate	100 oz.
Temperature	180°F
pH	4
Deposition rate	0.00015 inches in 30 min.

ALKALINE BATH: From the alkaline bath, Brenner and Riddell obtained brighter plates. A possible disadvantage of the alkaline bath is the evolution of ammonia fumes, since ammonium hydroxide is used to adjust the pH. Most plating shops are equipped with suitable exhaust units, however; and ammonia fumes would not be a problem. The following plating bath was recommended by Brenner and Riddell (7):

Nickel chloride	4.0 oz. per gal.
Ammonium chloride	6.7 oz. per gal.
Sodium hypophosphite	1.3 oz. per gal.
Sodium citrate	13.4 oz. per gal.
Temperature	194°F
pH	8 to 10
Deposition rate	0.0002 to 0.0003 in. per hr.

ACETATE BATH: G. Gutzeit and A. Krieg developed an acid electroless solution buffered with acetate. A typical acetate bath having about the optimum composition, according to Gutzeit and Krieg, is as follows (7):

Sodium acetate ($NaC_2H_3O_2$)	10 g./l.
Sodium hypophospite ($NaH_2PO_2 \cdot H_2O$)	24 g./l.
Nickel chloride ($NiCl_2 \cdot 6H_2O$)	21 g./l.
Temperature	99°C
pH	5
Deposition rate	0.001 in. per hr.

SUCCINATE BATH: E. J. Ramirez and G. Gutzeit produced a plating bath using a succinate as a buffer. This bath exhibited the distingushing characteristic of a high rate of deposition. This bath had the following typical composition (7):

Sodium hypophosphite ($NaH_2PO_2 \cdot H_2O$)	30 g./l.
Nickel chloride ($NiCl_3 \cdot 6H_2O$)	22 g./l.
Sodium succinate ($Na_2C_4H_4 \cdot 6H_2O$)	15 g./l.
Temperature	99°C
pH	5
Deposition rate	0.0013 in. per hr.

The rate of deposition depends upon the composition of the solutions and is approximately proportional to the concentration of the hypophosphite. The temperature of the bath is most critical for deposition initiation, and rates are dependent upon this condition. Other factors, such as the concentration of organic salt, affect the rate of deposition.

Since the process is an immersion or electroless type, uniform deposits can be obtained on all flat surfaces, corners, edges, recesses,

bores, and diameters, internal or external. Therefore, it offers an excellent method of plating for salvage or protection of precision parts or tools. Thieving or shielding problems such as would be expected with electrolytic deposits can be eliminated.

The chief advantages of the electroless nickel plating process are:

1. No electricity is required.
2. Complex parts can be coated uniformly.
3. Continuous coatings, limited only by cost and processing time, can be built up.
4. Good resistance to humidity and salt spray is achieved as compared to electroplated nickel and chromium.
5. Coatings can be welded.

The disadvantages of the electroless process which limit its application are:

1. As-plated-coatings are brittle.
2. The process is costly from the material and control standpoint.
3. Operating temperature may reduce the mechanical strength of the base metals which have been hardened by heat treatment or cold working.
4. Constant attention is required in continuous processing is operated manually.
5. Coatings are affected by severe outdoor weathering conditions.
6. Coatings have higher coefficient of friction than chromium.

III. MATERIALS AND EQUIPMENT

The Pump - An electrically-driven, multi-speed "finger" pump was chosen for use in the dynamic system. This pump was chosen for its unique characteristic of positive displacement action without being in direct contact with the plating solution.

Heat Exchangers - Four glass, double-piped heat exchangers were used in the system. Two of the thirty-inch long heat exchangers were used to heat the flowing solution to about 180° - 200°F utilizing steam. The other two exchangers used tap water to cool the bath. A twelve-inch heat exchanger provided a steam jacket around the Inconel test section. The flow of solution to steam or water was counter current in all the heat exchangers.

The Filter - A porcelain funnel-type filter with a four-inch-diameter perforated plate supporting a piece of filter paper was used in the system. Flow through the filter was by gravity.

Miscellaneous Equipment - Mercury-in-glass thermometers were used for temperature measurement and a Macbeth pH meter was used to continuously record the solution pH. Connections between parts of the flow system were made using tygon tubing. Glass and plexiglass tanks were employed throughout the system. Flow rates were measured using a rotameter whose range was between zero and fifty cubic centimeters per minute.

The diagram of Figure 1 shows the system schematically, and a photograph of the apparatus appears in Figure 2. The system was

designed to pump the solution to an elevated tank "J" (not shown in Fig. 2) from which the solution flowed by gravity through the heating heat exchangers "B", into the plexiglass tank "C", for temperature measurement and flow stabilization; through the Inconel test section which is steam jacketed by the heat exchanger "E"; into the tank "D" for temperature measurement; through the cooling heat exchangers "F"; through the rotameter "G" for flow measurement; through the filter"H"; into the tank "I" (not shown in Fig. 2) for pH measurement by the Macbeth pH meter "L", from which it is pumped to the elevated tank "J" by pump "A", completing the cycle.

The tubing plated in the experiment was wrought-annealed seamless Inconel (nom. comp. 80 Ni, 15Cr, 5Fe) tube of approximately one-fourth inch inside diameter, with a wall thickness of 0.025 inches.

The plating baths studied were the acid, the succinate, and the acetate which are described in Table I.

Tube and muffle-type electrical resistance laboratory furnaces were used to heat treat the samples in the experiment. The Tukon mirro-hardness tester was used to obtain hardness data for the heat treated coatings.

One of the Inconel tubes mentioned above was bent 180° and plated. A 1½-inch, glass tube, stoppered on one end, with inlet and outlet ports for steam was used for the heat exchanger jacket around the bent tube. This apparatus is shown schematically in Figures 3 and 3a.

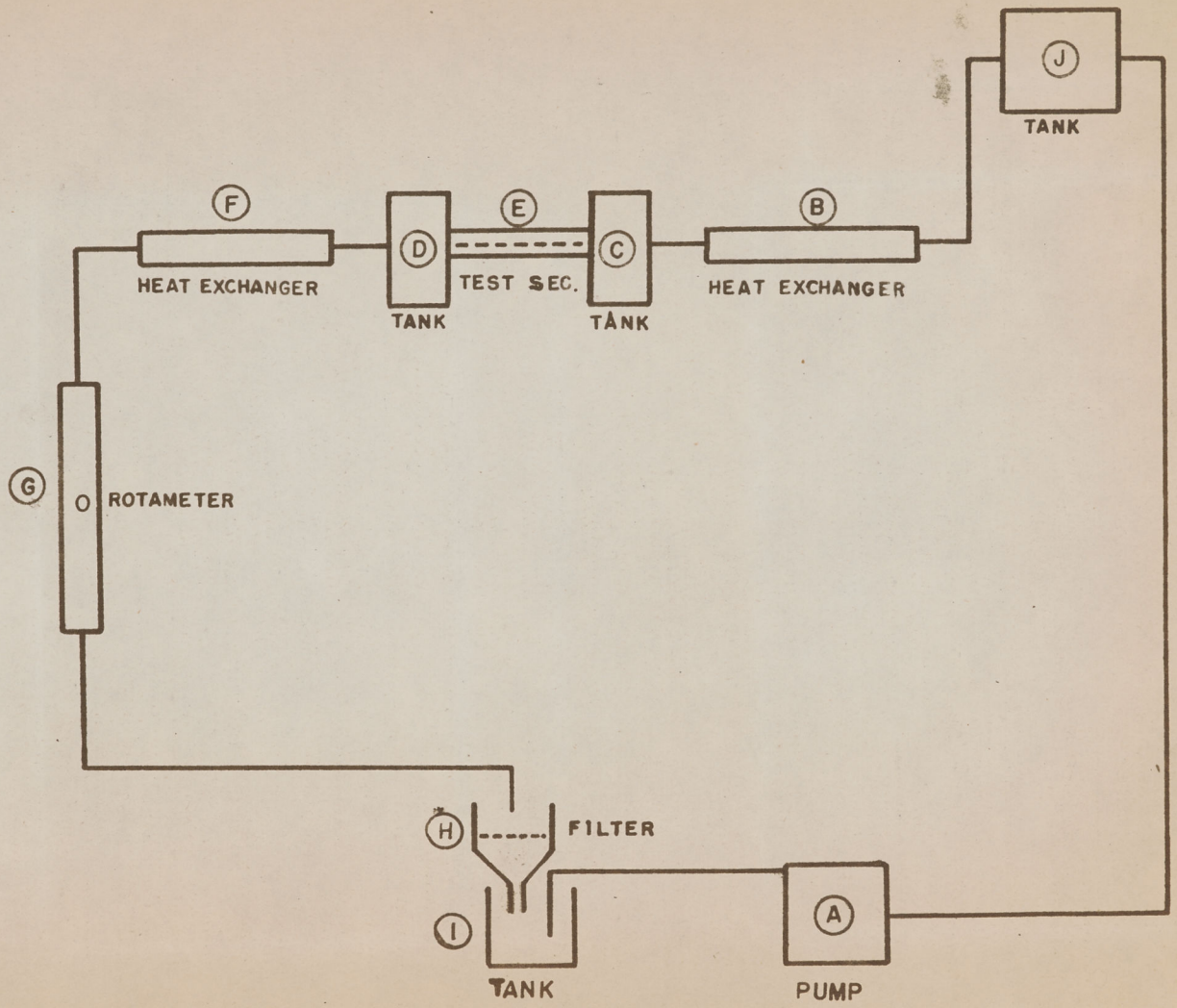


FIGURE 1

SCHEMATIC DIAGRAM OF DYNAMIC PLATING APPARATUS

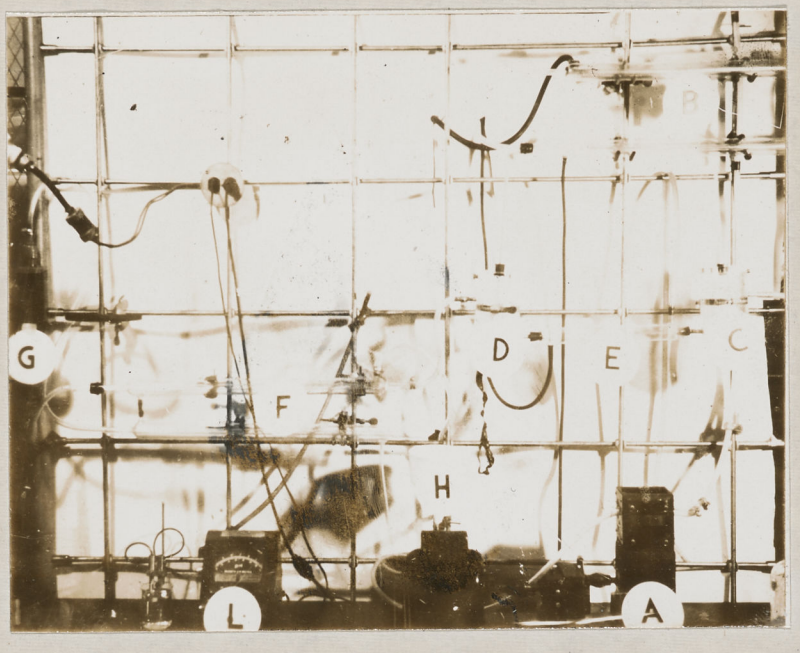


Figure 2: Flow system for electroless plating operation
A. Pump B. Heat exchanger C. Tank D. Tank
E. Steam jacket for Inconel tube F. Heat exchanger
G. Rotameter H. Filter L. pH meter



Figure 3: Photograph of Inconel tube bent 180° used in the system for the bent tube plating test

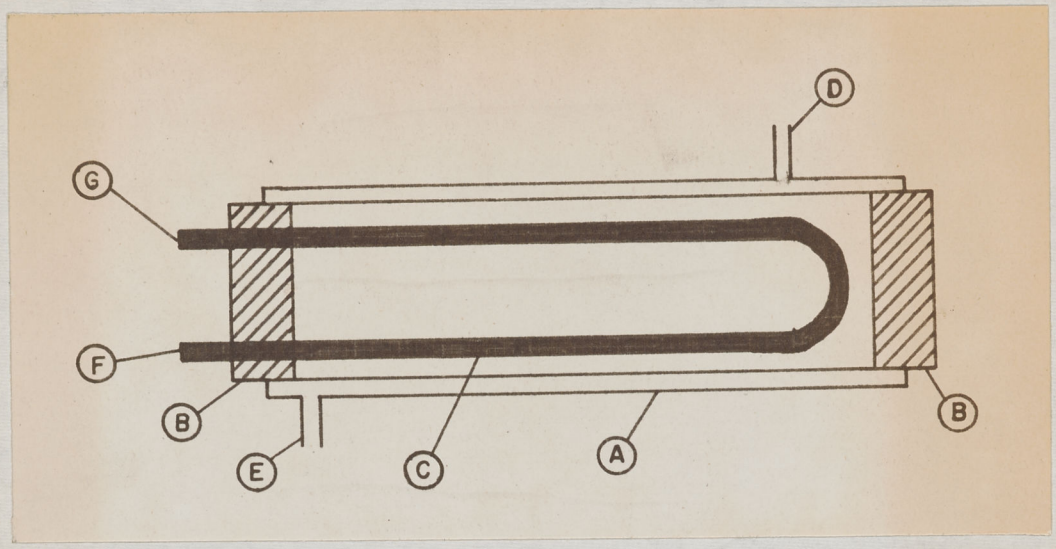


Figure 3a: Steam jacket apparatus used in bent tube test - A. $1\frac{1}{2}$-inch glass tube B. Rubber plugs C. Bent Inconel tube D. Steam inlet E. Steam outlet F. Plating solution inlet G. Plating solution outlet



Figure 4: Photograph showing Ni-P plate taken from walls of hot heat exchanger walls

TABLE 1 - 7

Characteristics and Compositions of Plating Baths Studied

Bath	Acid - g./l.	Acetate - g./l.	Succinate - g./l.
Nickel Chloride ($NiCl_2 \cdot 6H_2O$)	30	21	22
Sodium hypophosphite ($NaH_2PO_2 \cdot H_2O$)	10	24	30
Sodium hydroxyacetate ($NaC_2H_3O_3$)	50	--	--
Sodium acetate ($NaC_2H_3O_2$)	--	10	--
Sodium succinate ($Na_2C_4H_4 \cdot \underline{6}H_2O$)	--	--	15
Temperature	90°C	99°C	99°C
pH	4-6	5	5
Rate of deposition (in./hr.)	0.0006	0.0010	0.0013

IV. EXPERIMENTAL PROCEDURE

Description of Plating Operation

The plating baths studied in the work were prepared as follows:

Acid Bath: A six-liter solution was made by first dissolving 180 grams of nickel chloride (30 b/1) and 60 grams of sodium hypophosphite (10 g/1) each in a liter of distilled water. Sodium hydroxyacetate was made by adjusting the pH of glycolic acid to 6 with sodium hydroxide. The dissolved nickel chloride and sodium hypophosphite were then added to the glycolic acid containing 300 grams of sodium hydroxyacetate (50 g/1) and the volume adjusted to six liters.

Succinate Bath: One hundred and thirty-two grams of nickel chloride (22 g/1), 180 grams of sodium hypophosphite (30 g/1), and 90 grams of sodium succinate (15 g/1) were each dissolved in a liter of distilled water and mixed in a large container. The volume was adjusted to six liters by the addition of distilled water, and the pH was brought to 5 by the addition of hydrochloric acid.

Acetate Bath: A six-liter solution was made as above by the addition of 126 grams of nickel choride (21 g/1), 144 grams of sodium hypophosphite (24 g/1), and 60 grams of sodium acetate (10 g/1) to distilled water. The solution was adjusted to a pH of 5 by the addition of hydrochloric acid.

Tube Preparation

The tube was prepared for plating by the following method:

1. Immersed in alkaline cleaning solution until free from surface water-break.
2. Water rinsed.
3. Immersed in concentrated hydrochloric acid.
4. Water rinsed.
5. One minute immersion in sulfuric acid.
6. Water rinsed.

Plating Procedure

The tube was then placed in the plating apparatus, and the plating bath was pumped into the system. The steam and water was turned on and the flow was started by the gravity feed from the elevated tank. The flow was regulated by an adjustable "pinch stop" located on the tygon tubing at the up-stream side of the test section.

An Inconel tube was bent 180° with a ½-inch radium and plated using the acetate bath to determine the plating possibilities of two-pass heat exchanger tubes. This test section is shown in Figure 3.

The presence of aluminum at the ends of the test section promoted the initiation of plate deposition.

Description of the Heat Treatment Operation

Samples varying in length from 1/8 to 1/4 inches were out on the cut-off wheel in preparation for heat treatment. About forty of these samples were sealed in evacuated vycor capsules and placed in furnaces at different temperatures. The tube-type furnaces were used to heat treat at temperatures of 250° and 300°C while the muffle-type laboratory furnaces were used at 400°, 500°, and 600°C. One sample

from each furnace was quenched from each furnace after ½ hour, 1 hour, 2 hours, 3 hours, 4 hours, 24 hours, and 48 hours had elapsed. One sample was heat treated at 800°C for 2 hours in one of the muffle furnaces.

After heat treatment each sample was studied metallographically by mounting in bakelite and polishing. The diamond pyramid hardness number of each sample was taken on the Tukon micro-hardness tester using a load of 0.1 Kg. The samples were etched and photomicrographs were made of selected samples.

V. RESULTS

The results of the plating operation are as follows:

Acid Bath: The solution produced a thin, porous plate as shown in Figure 5a. The pH of the acid bath remained between 4 and 6 throughout a ten hour operation but evidences of spontaneous decomposition were noticed near the end of the operation. (Spontaneous decomposition of the bath is identified by the formation of a black precipitate in the solution.) (3)

Succinate Bath: The succinate bath plated rapidly giving a thick, however locally non-uniform coating, as seen in Figure 5b. The optimium temperature of 99°C was never attained because the solution boiled in the heat exchangers. This caused plating on the walls of the heaters and temperature tanks of the system. The coating could easily be removed after the apparatus cooled and a sample of this coating is shown in Figure 4.

Acetate Bath: This solution produced a sound, non-porous coating at a high deposition rate. The optimium temperature was not attained but a thick, uniform plate was produced as shown in Figure 5c. The bath produced no boiling in the heat exchangers or plating on the walls of the apparatus. This solution was the best tested.

The U-tube specimen gave a thin but uniform coating around the curved portion

Results of the heat treatment are as follows:

As Plated: Amorphous solid in layers parallel to plated surface

was obtained, as shown in Figure 5c. The as-plated hardness was 430 DPH.

250°C: The hardness ranged from approximately 450 DPH after one-half hour to a maximum of 1000 DPH occurring after 3 hours as shown in Figure 7. There was no apparent change in microstructure.

300°C: Maximum hardness of approximately 925 DPH occurred after 2 hours and an aging effect of hardness of 650 DPH after 48 hours, as shown in Figure 8. Figure 6 shows this heat treatment after four hours, and the appearance of a slight precipitation.

400°C: A maximum hardness of approximetely 1030 DPH occurred after about 2 hours and an aging to 400 DPH seen in Figure 9. There was a steady increase of precipitation throughout the range of remaining heat treat temperatures.

500°C: A maximum hardness of 525 DPH was observed after one hour and a minimum aged hardness of approximately 425 DPH after 4 hours-Figure 10.

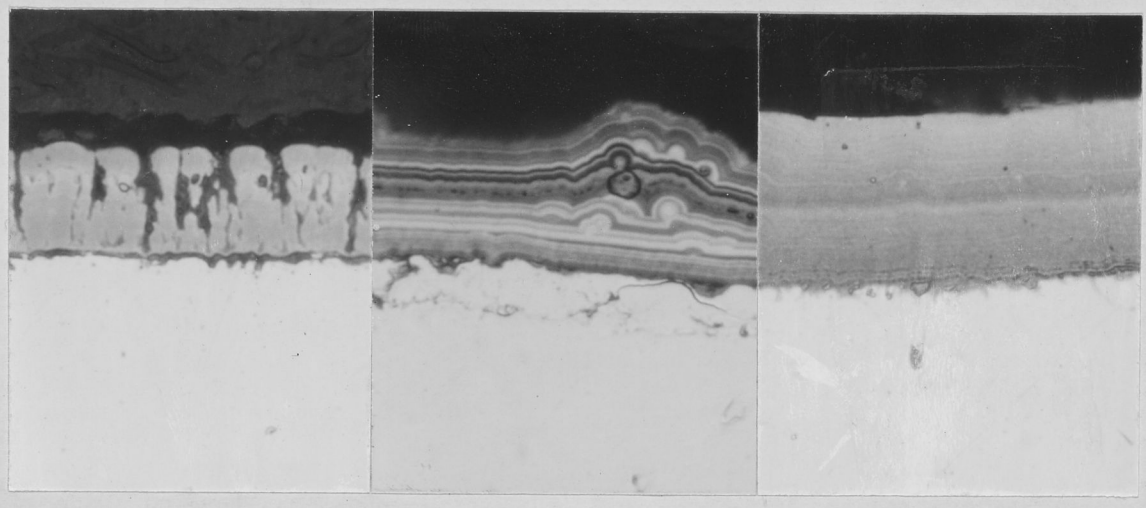
600°C: An aged hardness of approximately 400 DPH occurred after 3 hours, but Figure 11 also shows a maximum hardness occurring after one hour of about 610 DPH.

After precipitation a globular growth occurred along the coating and is exhibited in Figure 6.

The sample heat treated for two hours at 800°C exhibited globular transformation products of the intermetallic compound, Ni_3P, as seen

in Figure 12. The hardness of this specimen was 260 DPH.

All hardness data are presented in Table II.



(a) (b) (c)
Figure 5 750 X Etchant: Glyceria Regia

As-plated samples of Ni-P plate of the Acid Bath (a), Succinate Bath (b), and Acetate Bath (c).

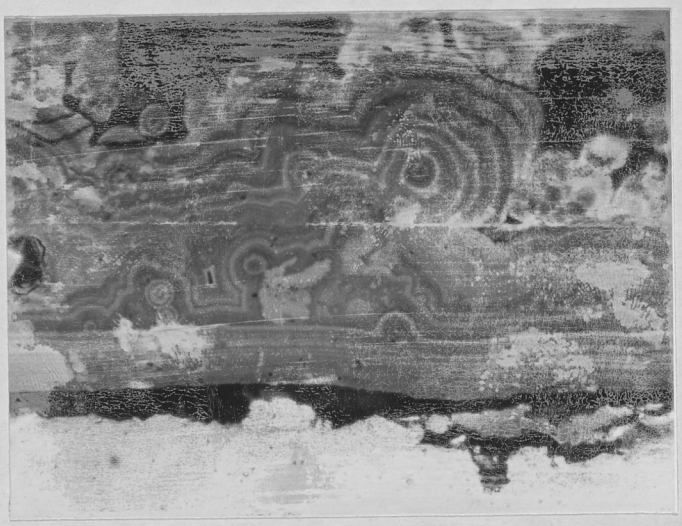


Figure 6 750 X Etchant: Glyceria Regia

Heat treated sample of Ni-P plate produced by the Succinate Bath held at 300°C for 4 hours showing nonuniform surface contour and globular formation

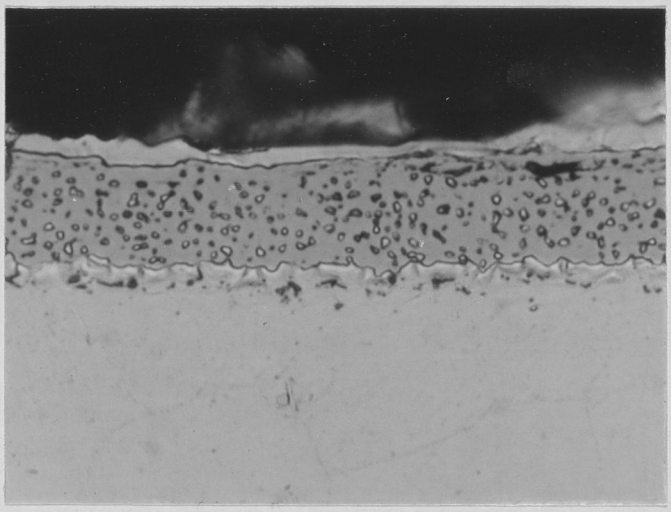


Figure 12 750 X Etchant: Glyceria Regia

Ni-P plate produced by the Acetate Bath heat treated for two hours at 800°C showing dispersion of Ni_3P precipitate in a nickel matrix.

TABLE II

HARDNESS DATA FOR HEAT TREATED ELECTROLESS NICKEL

Temperature (°C)	Time (hr.)	Hardness (DPH)
250°C	½	455
	1	782
	2	762
	3	1003
	4	894
	24	642
	48	536
300°C	½	743
	1	803
	2	960
	3	782
	4	792
	24	749
	48	642

TABLE II

HARDNESS DATA FOR HEAT TREATED ELECTROLESS NICKEL (CONTINUED)

Temperature (°C)	Time (hr.)	Hardness (DPH)
400°C	½	933
	1	907
	2	1033
	3	642
	4	599
	24	488
	48	397
500°C	½	525
	1	473
	2	442
	3	442
	4	429
	24	508
	48	442

TABLE II

HARDNESS DATA FOR HEAT TREATED ELECTROLESS NICKEL (CONTINUED)

Temperature (°C)	Time (hr.)	Hardness (DPH)
600°C	½	606
	1	498
	2	442
	3	397
	4	429
	24	508
	48	442
800°C	2	260

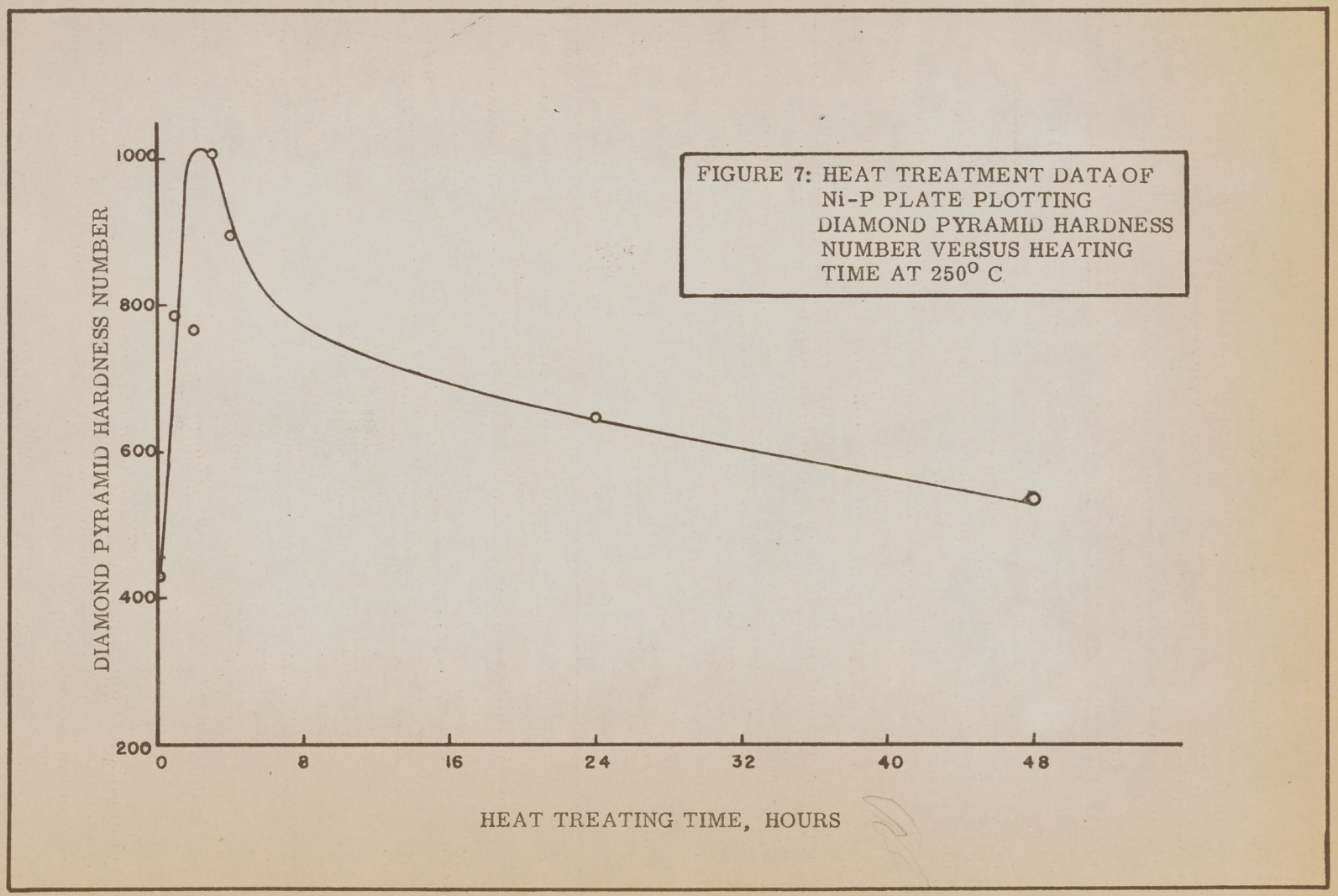


FIGURE 7: HEAT TREATMENT DATA OF Ni-P PLATE PLOTTING DIAMOND PYRAMID HARDNESS NUMBER VERSUS HEATING TIME AT 250° C

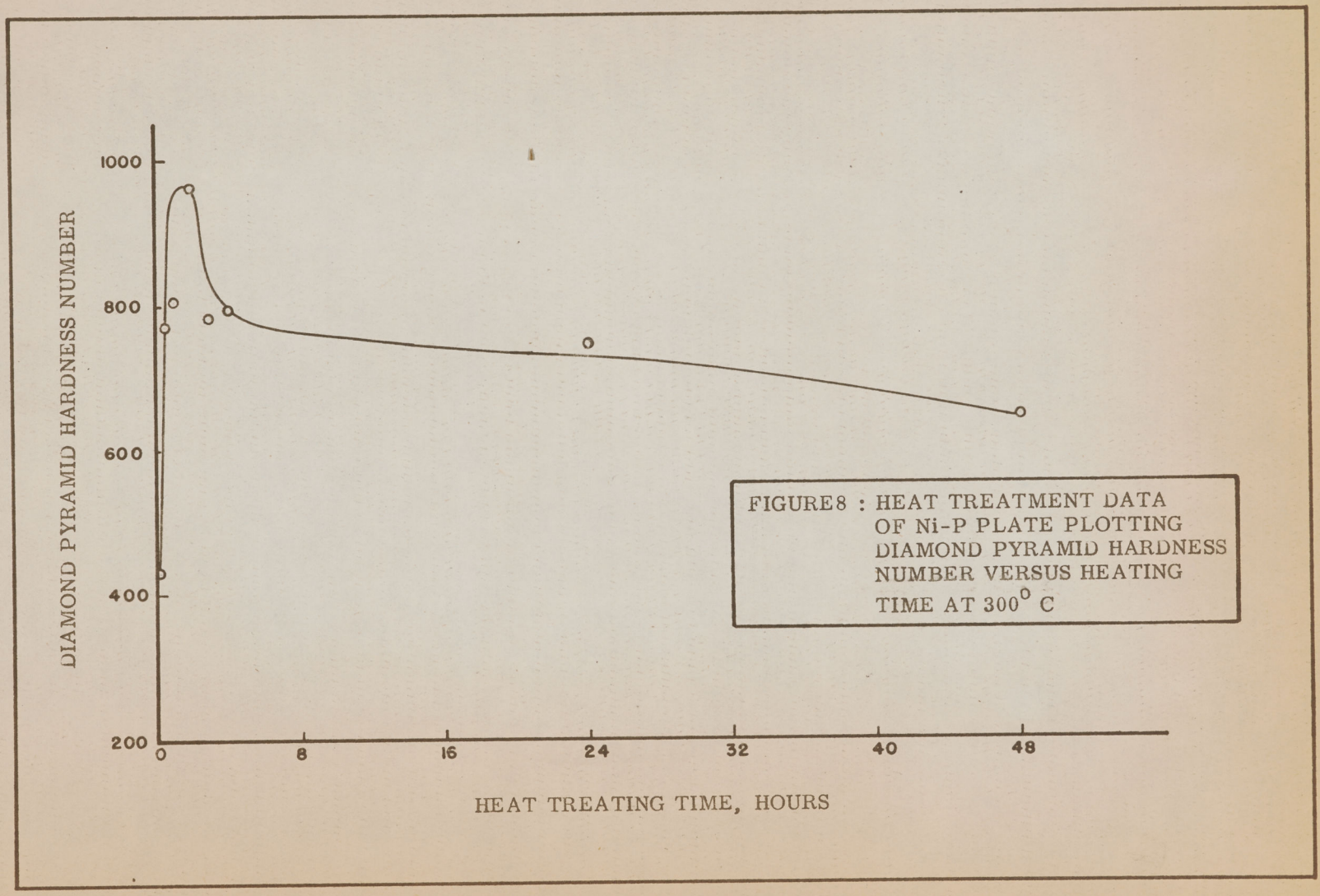


FIGURE8 : HEAT TREATMENT DATA OF Ni-P PLATE PLOTTING DIAMOND PYRAMID HARDNESS NUMBER VERSUS HEATING TIME AT 300^{0} C

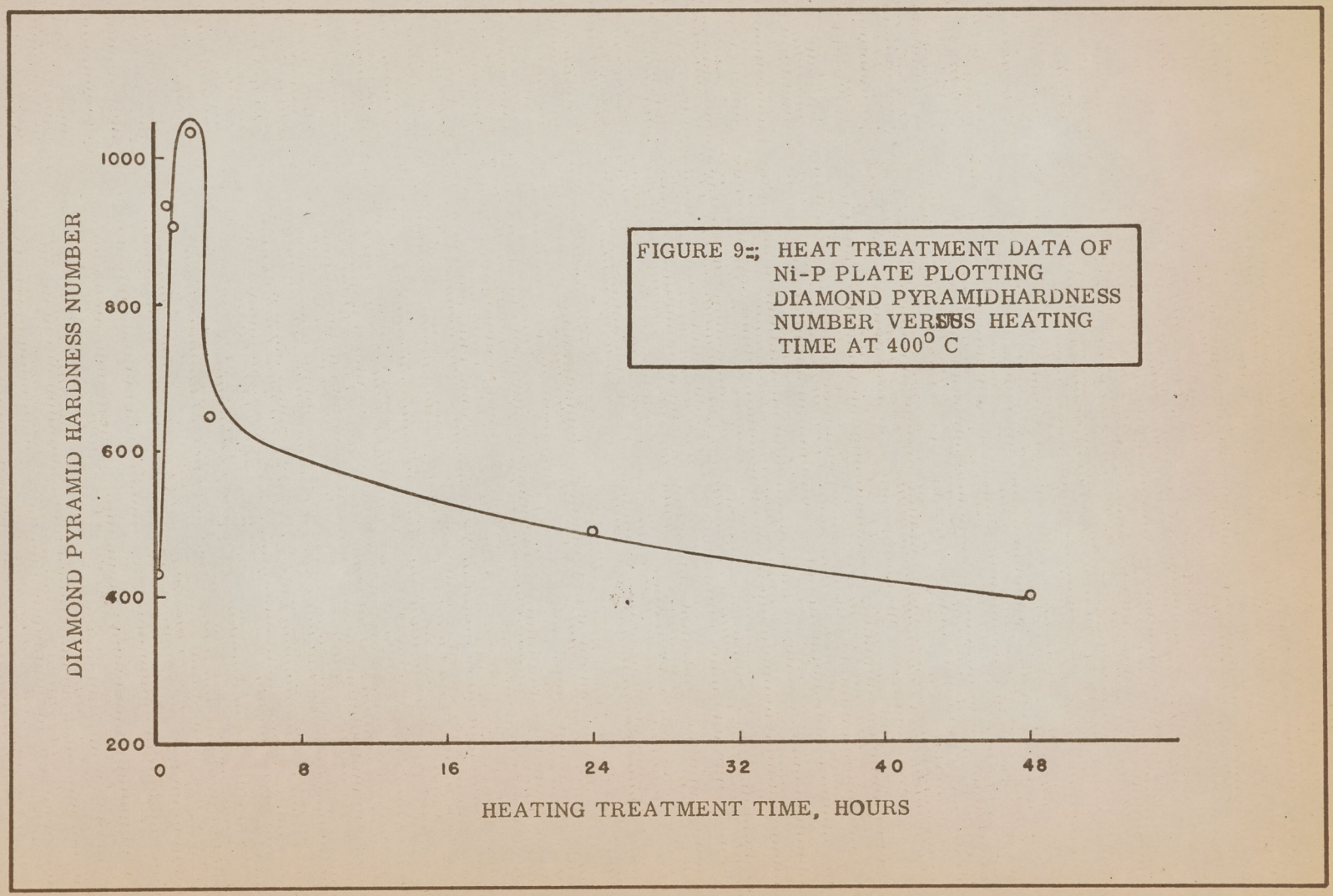


FIGURE 9:; HEAT TREATMENT DATA OF Ni-P PLATE PLOTTING DIAMOND PYRAMIDHARDNESS NUMBER VERSUS HEATING TIME AT 400° C

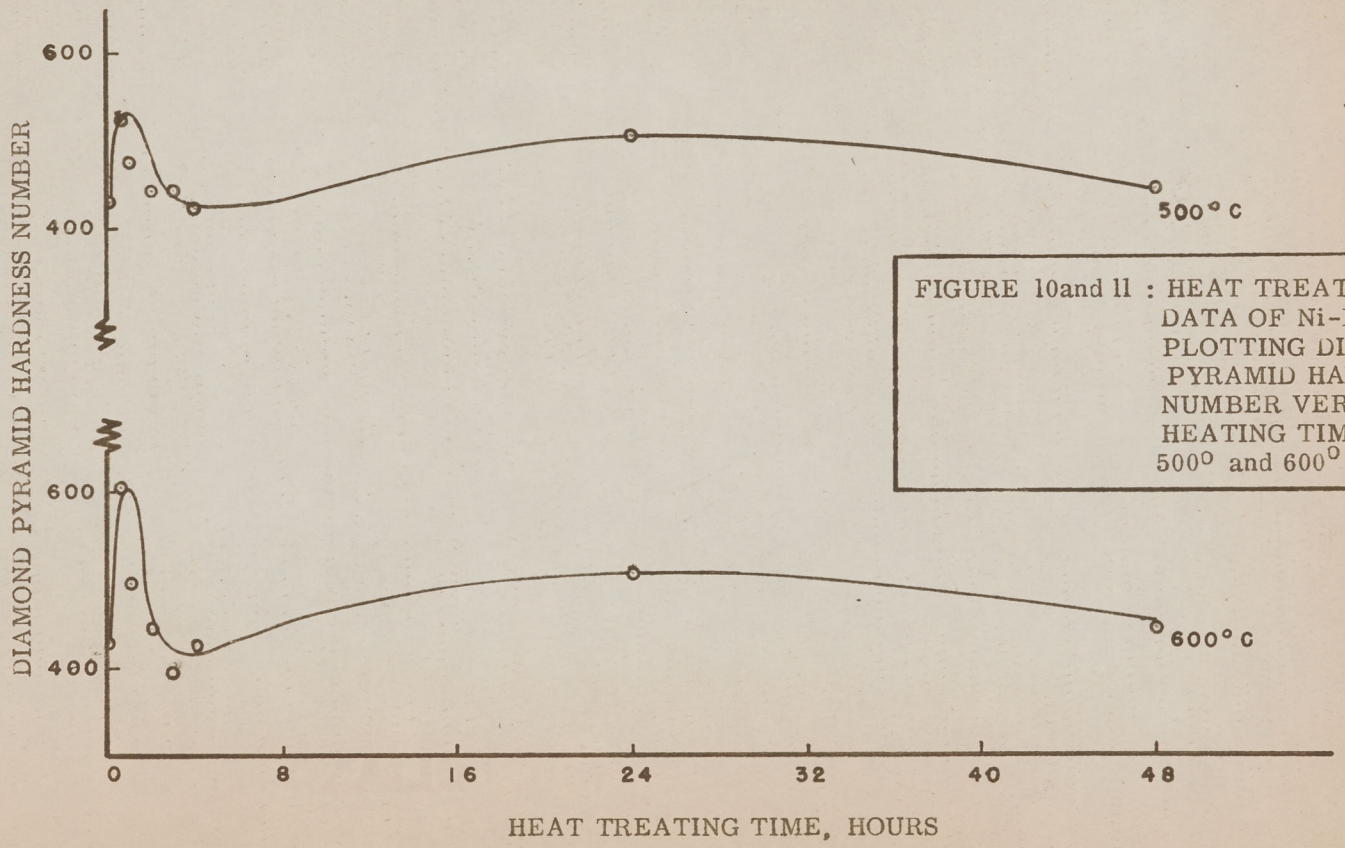


FIGURE 10and 11 : HEAT TREATMENT DATA OF Ni-P PLATE PLOTTING DIAMOND PYRAMID HARDNESS NUMBER VERSUS HEATING TIME AT 500^{o} and 600^{o}

VI. DISCUSSION OF RESULTS

The acetate plating bath yielded the best results of the three solutions studied. It produced approximately one and one-third mils of plate for a two hour operation at 185°F. The literature states that this solution should plate a mil per hour if the bath temperature is 99°C. The plate was very sound and adherent.

The bent tube test was not as satisfactory as was desired. It was shown that it is possible to plate around the curved area at this dimension but a better flow system should be designed to improve the conditions and thus produce a thicker coating in a given time. This improvement might be accomplished by designing the heating system such that the heat be gradually increased until the hottest area would be at the plating section. This would cut down on boiling in the apparatus and thus yield a higher plating temperature.

Figures 5b, 5c, and 6 exhibit striations parallel to the surface being plated. These striations are characteristic of periodic variations of compositions (Presumably phosphorous) rather than alternate lamellal of different phases. Growth appears to originate at point centers or nuclei and develops at an equal rate in all directions, hence, the concentric circle pattern (Figures 5b and 6). (2)

Higher hardnesses were obtained after aging in the temperature range of 250° to 400°C than temperatures higher than 400°C. It is known that electroless nickel deposits as an amorphous material and then at 300°C a transformation to a two-phase crystalline material takes place. This transformation is said to be one occurring by the

nucleation and growth process whose rate is increased at increasing temperatures above 300°C and decreased at temperatures below 300°C. Thus some transformation might have occurred during heat treatment at 250°C permitting age - hardening. However, since presumably only a part of the material crystallized at this temperature, the hardness was not as high as that of the more completely transformed material, heat treated at temperatures above 300°C.

As the temperature increased the maximum hardness was reached in a shorter time. The heat treatment at 400°C seemed to reach maximum hardness in less than four hours while the 500°and 600° C heat treatment seemed to reach maximum hardness in less than one-half an hour (the lowest time interval studied).

The maximum hardness of 1025 DPH after two hours at 400°C compared favorably with the data given in the literature of the Kouigen plate. (10) The maximum hardness for this coating occurred after a one hour heat treatment at 400°C and was about 1030 DPH.

There is a good possibility of localized hardening producing the scattered data in Figures 7, 8, 9, 10, and 11. The transformation process is an exothermic one and the temperature rise due to nuclei formation might be extended enough to cause some regions to be at a higher temperature for a longer time than other areas, thus leading to localized over-aging. Under the light loading used for micro-hardness reading (0.1 kg.) the local effects are undoubtably reflected in the data.

The reason for the local nonuniformities in surface contour of coatings produced in the succinate bath (all Figures 5b and 6) is not known and could profitably be the subject of further study.

The sample heat treated at 800°C for two hours is shown in Figure 12. Here a transformation has occurred which might help explain the transformation precipitation at lower temperatures. Here the transformation preceeded toward equilibrium and produced a dispersion of the intermetallic compound, Ni_3P in a Ni matrix. The size of the Ni_3P particles is temperature dependent, i.e., the lower the heat treatment temperature the finer the dispersion. This leads to the deduction that the precipitation at the low heat treated temperatures is Ni_3P particles. (2)

VII. CONCLUSIONS AND RECOMMENDATIONS

The following conclusions can be made from these studies:

1. Of three baths studied an acetate plating gives the best plate and plating conditions using a flow system.
2. A thin but uniform, continuous plate can be obtained on a tube bent 180° with a $\frac{1}{2}$ inch radius of bend.
3. The coating responds to precipitation-hardening type heat treatments and upon heat treatment at 800°C for two hours a dispersion of Ni_3P can be observed in a Ni matrix.
4. Striations are present in the as-plated coatings parallel to the plated surface.
5. A maximum hardness of 1025 DPH was obtained after a heat treatment of two hours at 400°C.

For any future work on this subject, it is recommended that:

1. The bent tube test be performed using an improved flow system and lower flow rates at different bending radii.
2. Using something like a salt bath for a heat source, study the effect of very short time heat treatments on the coating characteristics.
3. Using the baths here studied or others obtain a thicker coating of about four mils for heat treatment so that 1 Kg. loading hardness readings can be made.

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APPROVAL

The foregoing thesis is hereby approved as a creditable study of an engineering subject, carried out and presented in a manner sufficiently satisfactory to warrant its acceptance as a prerequisite to the degree for which it has been submitted. It is to be understood that by this approval the undersigned does not necessarily endorse or approve any statement made, opinions expressed, or conclusions drawn therein but approves the thesis only for the purpose for which it is submitted.

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