

## Effect of Post-detachment Cleaning on the Hydrophilic Nature of Nickel Thin Film Nozzle Plates for Piezo Ink-jet Printer Heads

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A hydrophilic nickel nozzle plate used in piezo inkjet printer heads with a minimum 5° water contact angle was obtained by (1) primary cleaning of the detached nickel nozzle plate by immersion for 90 sec in a thoroughly pre-electrolyzed 20 wt% NaOH solution maintained at 97°C (207°F) and (2) secondary cleaning by 1 min of scrubbing with a wiper soaked with a newly formulated post-treatment cleaner to remove surface oxides and corrosion product films. Successfully obtaining a hydrophilic nickel nozzle plate surface was attributable to (1) thorough pre-electrolysis of the primary 20 wt% NaOH cleaner solution to remove dissolved oxygen and platable cations and (2) proper formulation of the post-treatment cleaner solution, especially in the selection of a non-ionic block copolymer type of surfactant. Pre-electrolysis must be carefully carried out to avoid possible aqueous caustic corrosion of the nickel nozzle plate as the stable region of the passivating NiO or Ni(OH)<sub>2</sub> films at 97°C (207°F) is smaller than that at room temperature. The effect of post-treatment cleaning was experimentally substantiated by measurement of the water contact angle on the post-treatment cleaned nickel nozzle plate surface.

### Introduction

Piezoelectric ink jet printers have been widely used as printing units for word processors or as output devices for personal computers.<sup>1</sup> Piezo ink jet head technology is favored for its semi-permanent service life and the wide range of inks that can be used because the ink chamber is not thermally heated. A piezo ink jet head consists of an

actuator, a chamber and a nozzle. A piezo ink jet printer cartridge contains an array of nozzles arranged such that an ink droplet is ejected from each nozzle channel through the action of a sudden pressure pulse produced by a piezoelectrically-induced instantaneous volume change of the ink chamber.<sup>1</sup> An overall cross section of a piezo ink jet head\* is shown schematically in Fig. 1.

The nickel thin film nozzle plate is fabricated by electroforming.<sup>2</sup> After mechanical detachment of the electroformed nozzle plate from the mandrel, a Ni / 3-wt% V cathode-coated silicone wafer, the front of the nozzle plate is always stained with photoresist residues as shown in Fig. 2. Since the front of the detached nozzle plate is to be subsequently electroplated with nickel, it must be free of any polymeric and organic residues, grease, particles or debris.<sup>3</sup> Therefore, the technical objectives of the present research were to: (1) devise the best cleaning method, suitable for mass production, for the front of the mechanically detached nickel nozzle plate, (2) determine the most efficient post-treatment of the cleaned surface and (3) establish the criteria for surface cleanliness by contact angle measurement of water drops. The cross-section of the mechanically detached nickel nozzle plate, cathode and substrate wafer is shown in Fig. 3.

### Background

#### *Primary cleaning method for mass-production*

Generally, electroplating processes demand the highest possible freedom from contaminants.<sup>4</sup> The front of the detached nickel nozzle plate must be free of contaminants such as: (a) photoresist residues, polymeric films, organic films, oils and greases, (b) inorganic fine particles of siliceous material, i.e., dirt suspended in (a), and (c) oxide films or other corrosion products that are present on the substrate.<sup>3</sup> Since extreme care was taken not to contaminate the detached nozzle plate with human oils, photoresist residues were the main contaminants to be removed in the post-detachment cleaning process.

### Nuts & Bolts: What This Paper Means to You

The piezo inkjet printer heads that we take for granted on our printers are quite a piece of technology. To the layman, the microscopically precise squirting of ink at the right place and the right time boggles the mind. Making these units poses its own challenges. By their very nature, they require rigorous cleaning procedures on working metal surfaces. Here, the development of a cleaning cycle, amenable to mass production, for a nickel nozzle plate is described. It's not your father's cleaner tank.

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\* S-model Piezo Ink Jet Head designed and developed by Samsung Electro-Mechanics Co., Ltd. (SEMCO), Suwon, Korea.

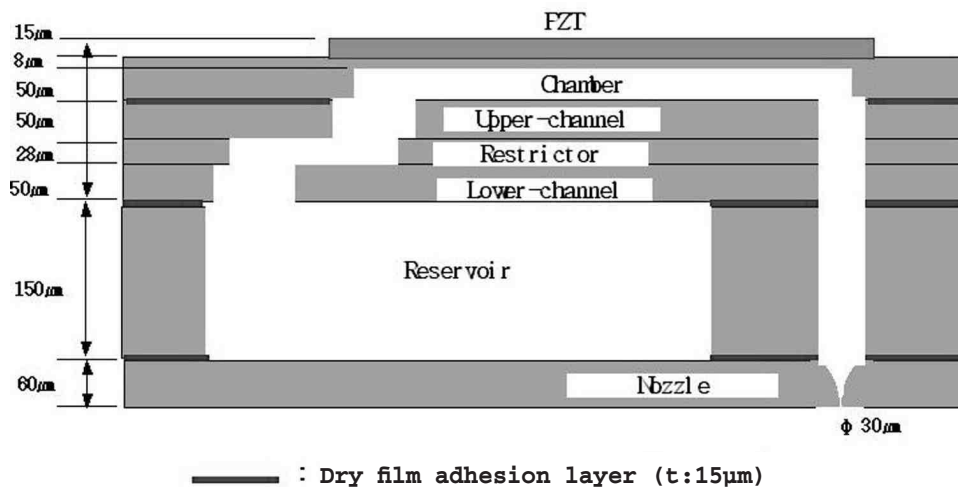


Figure 1—Cross-section of SEMCO S-model piezo inkjet head.

Alkaline cleaners are probably the most widely used of all cleaning media and can, unaided, produce surfaces sufficiently clean of polymeric and organic films, oils and grease for the electrodeposition of metals. Greases and oils are fatty acids, alcohols or esters, and are removed with hot aqueous solutions of high pH. These solutions are based on hydroxides, carbonates, phosphates, both simple and complex, and various silicates of sodium. The greases and oils present react with alkali to form water soluble soaps.<sup>3,4</sup> Synthetic surfactants, and complexing and chelating agents are used with these salts to assist the cleaning action.<sup>4</sup> Particulate dirt is usually removed together with the polymeric and organic films, oils and greases.<sup>3,4</sup>

In the present research, three aspects were carefully considered in selecting an alkaline cleaner amenable to mass production:

1. The possibility of using a concentrated aqueous solution of sodium hydroxide as the sole solute in the cleaner was investigated for simplicity in cleaner preparation. The alkalinity of the cleaner mixture to some extent governed its activity. The sodium hydroxide concentration should be at least 50 g/L (6.7 oz/gal).<sup>4</sup>

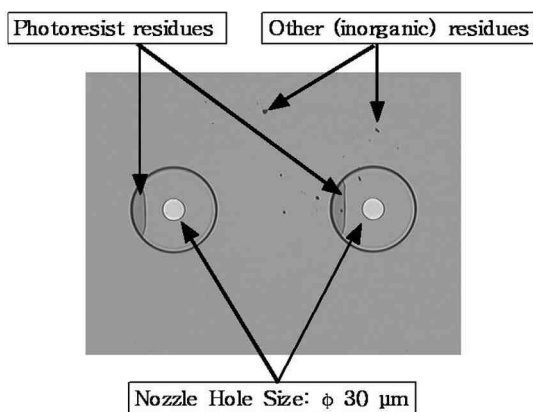


Figure 2—Front of as-detached nickel nozzle plate stained with photoresist and other (inorganic) residues as viewed in the direction of the arrow shown in Fig. 3.

2. Cleaning by immersion in the alkaline cleaner was adopted after consideration of the plant design and upkeep. Although spray-washing is more effective, it was excluded as an option because unnecessary losses would be caused by the ventilating system or overspray between zones.<sup>4</sup>

3. Aqueous sodium hydroxide solution was heated to near boiling temperature to accelerate immersion degreasing. This compensated for the reduced efficiency of immersion cleaning as compared to spray-washing.<sup>4</sup>

Nickel is quite resistant to a high pH environment over the entire range of 0 to 100% NaOH concentration in solution and because of its tendency to passivate.<sup>5-8</sup>

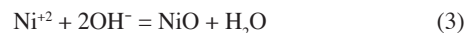
The effect of temperature on pH is such that the neutral point,

$$p\{[H_3O^+][OH^-]/2\} = -\log \{[H_3O^+][OH^-]/2\} \quad (1)$$

is 6.12 at 100°C (212°F) while at 25°C (77°F) it is 7.00. Further, the solubility product for nickel oxide,

$$K_s = [Ni^{+2}][OH^-]^2 \quad (2)$$

for a reaction such as



is reduced, albeit slightly to 15.26 at 100°C (212°F) from 15.96 at 20°C (68°F).<sup>9</sup> This is significant from a corrosion point of view in that the range of stability of NiO and Ni(OH)<sub>2</sub> as a potentially protective oxide film shifted from a pH range of 9 to 12 to a one of 5 to 9. Thus, with increasing temperature, a basic pH that could have been quite protective at low temperatures might give rise to a higher corrosion rate at high temperatures, especially if excess

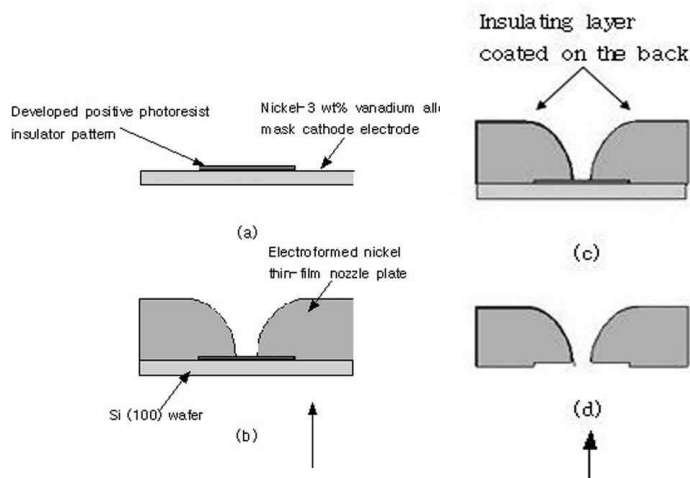


Figure 3—(a) Positive photoresist pattern on nickel – 3 wt% vanadium alloy mask cathode electrode sputter deposited onto Si(100) wafer. (b) Nickel electroformed on the alloy mask cathode electrode. (c) Insulating layer coated on the back of the electroformed nickel nozzle plate. (d) Mechanically detached nozzle plate. Fig. 2 shows this state. (Figs. 2, 5 and 6 are perpendicular to the bottom of the wafer in the direction of the arrows).

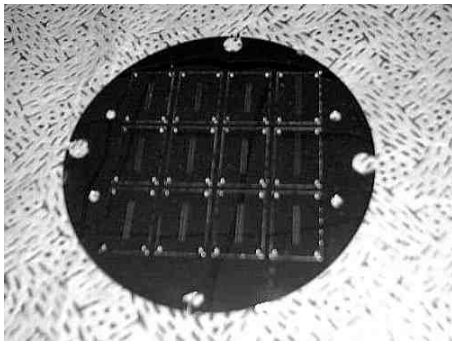


Figure 4—Twelve detached electroformed nozzle plates with screwed holes and placed on a static-resistant wipe.

oxygen were present to raise the potential substantially above the hydrogen equilibrium potential.<sup>10</sup> Therefore, the alkaline cleaning solution must be thoroughly deoxygenated. This, on the other hand, necessitates a thorough passivation during alkaline cleaning at 97°C (207°F).

Although particulate dirt is usually removed together with the oil, grease, organic and the polymeric films which bind it, the highly caustic composition and the high pH of the primary cleaning solution make removal of inorganics by rinsing difficult.<sup>3,4</sup> Therefore, a post-treatment of the cleaned surface was necessary to remove inorganics, particulate dirt and other debris efficiently.

#### Post-treatment of the cleaned surface

After removal from the cleaning bath, the nozzle plate is covered with a thin film of nickel oxide or other corrosion product.<sup>11-15</sup> These films are usually removed by dissolution in acid solutions. To check the efficacy of removing the photoresist residues and greases, the alkali solution was rinsed away or neutralized by dipping in dilute acid. Hydrochloric and/or sulfuric acids are the most common used and concentrations and temperatures are varied according to the substrate. Mixed acids containing wetting agents are supplied as proprietary mixtures; hydrochloric, sulfuric and phosphoric acids are common.<sup>3</sup> Inhibitors are generally added to acid solutions to protect the work being cleaned by retarding the chemical action of the acid on the basis metal.<sup>16-18</sup>

For efficient post-treatment of the cleaned surface and removal of any oxide, other scale, particulate dirt, oil, grease and any other contaminants, the following must be considered:

1. Sulfuric acid solution was selected as the post-treatment cleaner for its lower cost, reduced fuming and reduced handling volume when compared with hydrochloric acid.<sup>17</sup> This enables a wipe on/wipe off cleaning mode, greatly simplifying the manufacturing process.
2. Inhibitor must be added to the post-treatment solution to ensure dissolution of the nickel oxide or other scale without attacking the nickel nozzle plate itself. After much trial and error, m-nitrobenzene sulfonic acid, a granular sodium salt inhibitor\*\* was finally selected to reduce the acid fumes resulting from excessive reaction between the acid and nickel nozzle plate, thereby reducing acid consumption.<sup>18</sup>
3. Further reduction of acid fumes was required for easy and convenient indoor handling of nickel nozzle plate cleaning. Again, after trial and error, a nonionic block copolymer type of surfactant\*\*\* which had low-foaming characteristics was found to reduce acid fumes during wipe on/wipe off cleaning. In addition, a proper amount of demulsifying agent\*\*\*\* was added to the solution to address any environmental concerns.

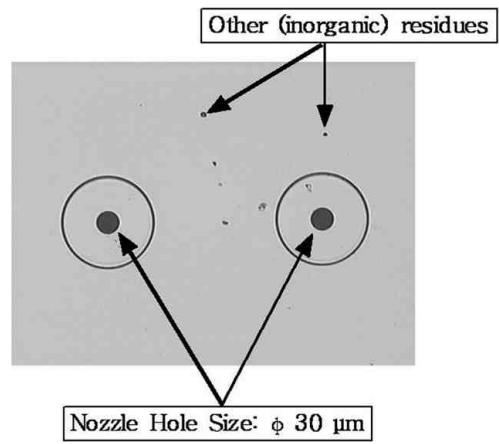


Figure 5—Front of primarily-cleaned nickel nozzle plate stained with other (inorganic) residues as viewed in the direction of the arrow shown in Fig. 3.

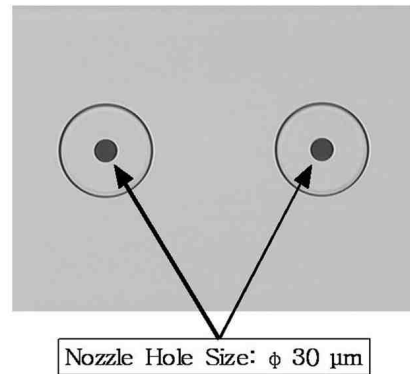


Figure 6—Front of post-treatment cleaned nickel nozzle plate as viewed in the direction of the arrow shown in Fig. 3.

#### Contact angle measurement

##### Of the cleaned and post-treated surface

Contaminants, especially grease and oil, can render a surface hydrophobic.<sup>3</sup> The presence of contamination is most often detected by behavioral techniques such as contact angle measurements (water drop test).<sup>19</sup> The efficacy of the cleaning process can be checked as follows: if, after removal from the acid and thorough rinsing, the draining metal surface remains wetted evenly all over for 30 to 60 sec (or until it dries by evaporation), hydrophobic contaminants have been removed. Traces of contaminants will cause the surface to de-wet, and surface tension draws the water into separate droplets, causing high contact angles. This is the water-break test. Traces of contaminants which remain when the nickel nozzle plate is subsequently plated do not actually prevent electrodeposition, but they are detrimental to adhesion and corrosion resistance.<sup>3</sup> The technical goal of the current research was to obtain a clean surface with a low contact angle on the nickel nozzle plate. Therefore, the final surface was examined by water contact angle measurements to check for residual hydrophobic contaminants.

## Experimental

### Alkaline cleaning

Cleaning was carried out in a Class 1000 clean room environment. For alkaline cleaning, a 20 wt% NaOH solution was prepared

\*\* Golpanol® MBS, BASF AG, Ludwigshafen, Germany

\*\*\* Pluronic® L35, BASF AG, Ludwigshafen, Germany

\*\*\*\* DeTeric ODP-LF, DeForest Enterprises, Boca Raton, FL.

in a 1-L volumetric flask from reagent grade NaOH and 18 M $\Omega$  deionized water. During preparation, a magnetic stirrer was used to vigorously stir the solution while it was heated to 40°C (104°F) to ensure complete dissolution. The solution was then boiled, bubbled with nitrogen gas (N<sub>2</sub>), cooled while passing N<sub>2</sub> over the solution and transferred to the pre-electrolysis cell as per the method of Lee and Staehle<sup>20</sup> to remove dissolved oxygen and platable cations.

After the pre-electrolyzed cleaning solution had been transferred to an acrylic cleaning tank [40(l)x23(w)x30(h) cm<sup>3</sup>; 15.7(l)x9.0(w)x11.8(h) in.<sup>3</sup>], the solution was heated and controlled to 97 ± 1.5°C (207 ± 2.7°F). To minimize exposure to the ambient atmosphere, the transfer was performed as quickly as possible. A heating element with a thermistor sensor was used to control the temperature of the solution in the bath. The heating element was covered with PTFE to protect it from the hot caustic environment.

The detached nickel nozzle plate was tightly screwed through a perforation, as shown in Fig. 4, against a PTFE cleaning jig and immersed in the cleaning solution, held at 97°C (207°F) for 90 sec. Research grade N<sub>2</sub> was continuously dispersed over the alkaline solution throughout the cleaning process to prevent further dissolution of ambient oxygen into solution. No agitation of the solution was used, however.

After alkaline cleaning, the nickel nozzle plate was removed from the bath and immediately rinsed to remove any chemical residues from the surface.<sup>4</sup> Each nozzle plate was first cleaned by immersing in acetone for 10 min in an ultrasonic bath. Further, the nozzle plate was ultrasonically cleaned in isopropyl alcohol and rinsed in running 18 M $\Omega$  deionized water. Each of these processes was done for 10 min. The nozzle plate was finally blown dry with a nitrogen jet to be ready for post-treatment.

### Post-treatment

For post-treatment of the cleaned nickel nozzle plate, a solution containing 25 wt% sulfuric acid, 0.3 wt% granular inhibitor, 9 wt% nonionic block copolymer type surfactant and 7 wt% demulsifying agent was prepared in a 1-L volumetric flask from reagent grade chemicals and 18 M $\Omega$  deionized water. Special care was taken to assure that each ingredient was completely dissolved before adding the next one. The pH was adjusted between 5 and 7 with 1N NaOH, while vigorously stirring with a magnetic stirrer. Unlike the alkaline cleaner makeup procedure, the post-treatment cleaning solution was prepared at ambient temperature. Finally, the solution was transferred to an acid-resistant Pyrex® beaker for wipe on/wipe off cleaning.

The alkaline-cleaned and rinsed nickel nozzle plate was carefully placed on a piece of static-resistant cloth as shown in Fig. 4. The cloth had been immersed in and thoroughly soaked with the secondary cleaner. Secondary cleaning was done by forcefully scrubbing the nickel nozzle plate with the cleaner-soaked wiper for 1 min. After secondary cleaning, the nickel nozzle plate was immediately rinsed to remove any chemical residue from the surface.

### Contact angle measurements

After each nickel nozzle plate was cleaned and rinsed, it was placed on a specimen viewing holder. Unless otherwise noted, the nickel nozzle specimen remained in the clean room environment of 40% relative humidity and 22°C (72°F). A small sessile drop of pure 18 M $\Omega$  deionized water allowed to contact the front surface of the nickel nozzle plate by means of a previously flame-cleaned platinum wire. The test water was prepared by following the method of Zisman, et al.<sup>21-23</sup>

The contact angle of the test water was measured with a goniometer. Its illuminator and microscope had been modified

so that they could be moved laterally and independently from the specimen viewing holder.

## Results and discussion

### Alkaline cleaning

The photoresist residues, polymeric and organic films, oils and greases were completely removed by alkaline cleaning, as shown in Fig. 5. They reacted with alkali to form water-soluble soaps. The higher the pH the more effective was the saponification.<sup>3</sup> Nickel was quite resistant to the high pH environment up to full concentration of the sodium hydroxide solution because of its tendency to passivate.<sup>5-8</sup>

The potential-pH diagram developed by Pourbaix, et al.<sup>8</sup> suggests the possible formation of all four oxides NiO, Ni<sub>3</sub>O<sub>4</sub>, Ni<sub>2</sub>O<sub>3</sub> and NiO<sub>2</sub>. It has also been suggested by experiments that the passivating film that forms on a nickel electrode in alkaline electrolytes is Ni(OH)<sub>2</sub> but others have attributed the phenomenon to the formation of Ni<sub>3</sub>O<sub>4</sub>, Ni<sub>2</sub>O<sub>3</sub> or NiOOH.<sup>24</sup> Ni(OH)<sub>2</sub> probably forms by a solid state process involving nucleation and growth. Under some alkaline conditions, particularly with the more concentrated solutions at high temperatures, films of 15 to 30 Å in thickness are formed at 90°C (194°F).<sup>7,20,25,26</sup>

Therefore, the resulting surface condition of the alkaline-cleaned nickel nozzle plate was not unexpected. Particulate dirt was usually removed together with the grease that bound it. However, high-pH solutions made removal of inorganics by rinsing rather difficult<sup>4</sup> as shown in Fig. 5.

### Post-treatment

The inorganic contaminants shown in Fig. 5 should have been removed along with the surface oxides and corrosion products because they were formed on the oxides and corrosion product films. The inorganic stains were removed by mechanically scrubbing the nickel nozzle plate with a static-free cloth that was thoroughly soaked with the post-treatment solution.

The post-treatment is an etching (pickling) process in that the surface oxide and corrosion product films are removed by dissolution in the cleaner solution soaked in the static-free cloth. This is also a very useful technique for removing surface layers (oxides and corrosion product films) and other contaminants thereon that are otherwise difficult to remove. The inorganic contaminants were completely removed by the post-treatment cleaning process as shown in Fig. 6. This enabled a consistent generation of the desired nickel nozzle plate surfaces for subsequent manufacturing processes.

### Contact angle measurements

Before post-treatment, the water contact angle on the nickel nozzle plate was about 53°. Metals and hard solids have high energy surfaces<sup>27</sup> and therefore it is highly likely that their surfaces are covered with organic and inorganic contaminants. Therefore, it is quite certain that the nickel nozzle surface was covered with hydrophobic contaminants.

The water contact angle however, was significantly reduced, to minimum of 5° after post-treatment cleaning. According to Zisman, et al.,<sup>21</sup> a noble metal surface can be hydrophilic in the absence of inorganic and organic contaminants. Also, White and Drobek<sup>28</sup> obtained contact angle values as low as 20° by changing the rinsing procedure. In view of these studies, the post-treatment-cleaned nickel nozzle surface was nearly free of inorganic and organic contaminants and subsequent manufacturing processes could be properly carried out.



## Summary

A highly efficient and mass-production-capable surface cleaning method for nickel nozzle plating of piezo inkjet printer heads consists of:

1. Primary cleaning by immersion for 90 sec in a thoroughly pre-electrolyzed solution containing 20 wt% NaOH maintained at 97°C (207°F) and
2. Secondary (post-treatment) cleaning by a 1-min scrubbing using a wiper soaked with the formulated post-treatment cleaner, discussed herein, to remove surface oxides and corrosion product films.

Thorough pre-electrolysis of the primary alkaline cleaner enabled nearly complete removal of dissolved oxygen and platable cations which could otherwise substantially raise the potential above the hydrogen equilibrium potential at 97°C (207°F). The pre-electrolysis procedure significantly lessened the possibility of aqueous caustic corrosion of the nickel nozzle plating during immersion cleaning at 97°C (207°F). Proper formulation of post-treatment cleaner solution, especially by the selection of a nonionic block copolymer type surfactant, greatly enhanced the cleaning efficiency. The effect of post-treatment cleaning was experimentally substantiated by measurement of the water contact angle before and after post-treatment. The water contact angle was reduced to a minimum of 5° from a value of 53° for the uncleaned surface.

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