

# Electrocomposite of Alumina in Nickel Matrix

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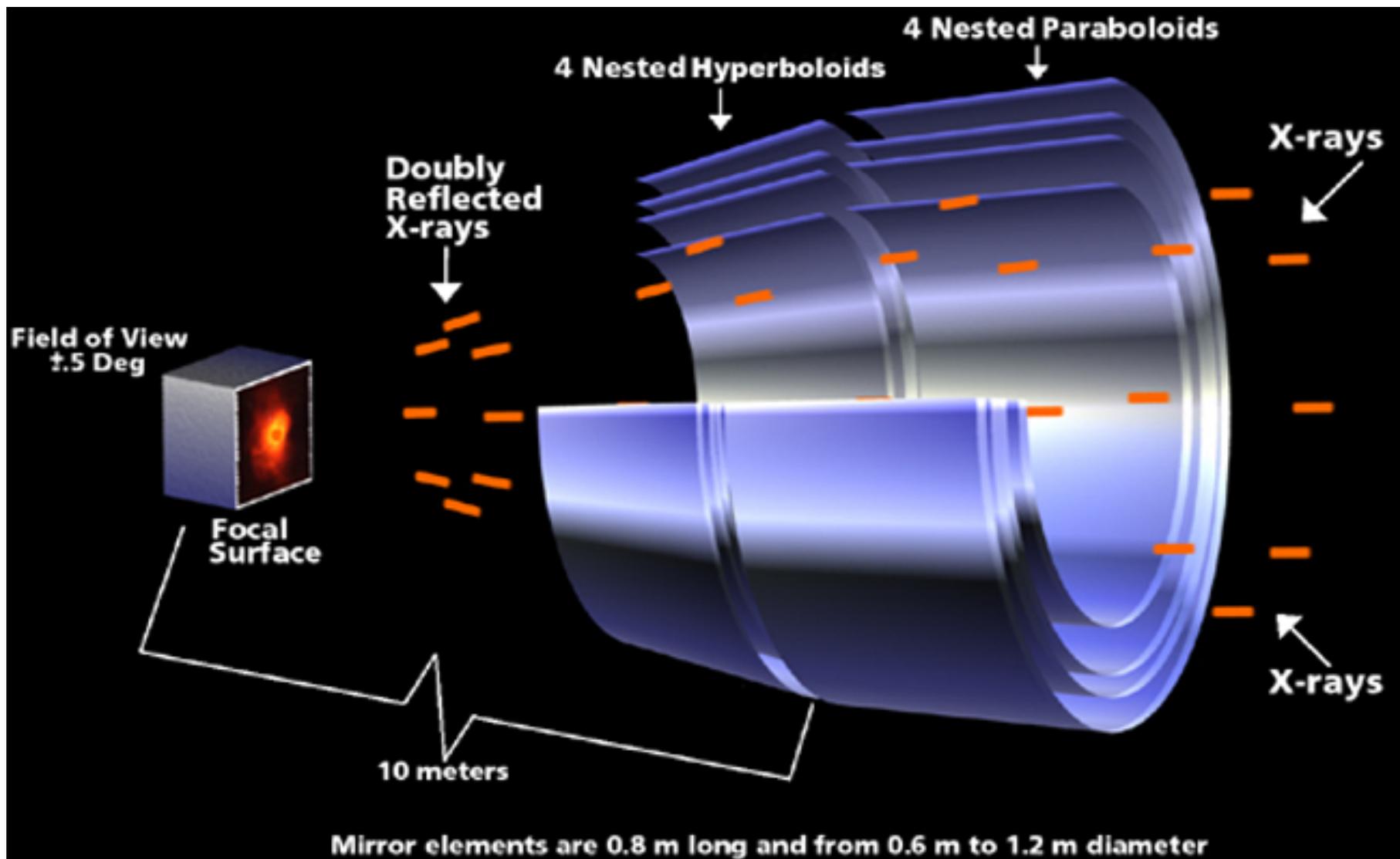
# *Introduction*

**Currently, the x-ray telescope mirrors used in balloon missions are electroformed nickel-cobalt alloy, in cylindrical shell shape. Due to strict weight limitations on the telescope, the mirror shells are only 4/1000 inch thick. At such shell thickness, maintaining their shape becomes problematic. The stiffness of a shell is approximately proportional to the third power of its thickness. Thus, increasing shell thickness and reducing mass density of the shell material becomes an attractive alternative. In this study, we concentrated on developing a nickel-alumina composite with low mass density and high strength.**



# Chandra X-ray Observatory

## Schematic of Grazing Incidence, X-ray Mirrors

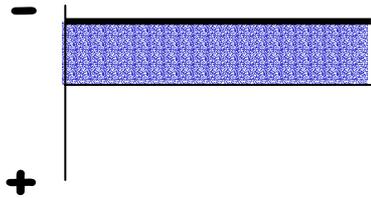


# *Plating Bath Properties*

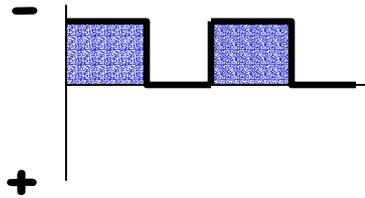
<b>Chemical composition</b>	Nickel metal as sulfamate	100 g/L
	Nickel bromide	2.288 g/L
	Boric acid	35 g/L
	Sodium dodecyl sulfate	0.2 g/L
	Alumina powder	50 g/L 0.05 $\mu\text{m}$ ( $\gamma$ -phase)
<b>Temperature</b>	47-52°C	
<b>pH</b>	3.57-4.15	
<b>Rotating cone rpm</b>	500	

# *Overview of Typical Waveforms Used*

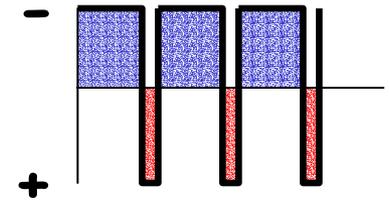
DC plating



Pulse Plating



Reverse -  
Pulse Plating



# *Particle Loading Effect - Dc Plating*

<b>Sample #</b>	<b>Particle Loading (gm/L)</b>	<b><math>\rho_{Com}</math> (gm/cm<sup>3</sup>)</b>	<b>n (1/cm<sup>3</sup>)</b>	<b>% Wt</b>
<b>1</b>	<b>0</b>	<b>8.8926</b>	<b>0</b>	<b>0</b>
<b>3</b>	<b>20</b>	<b>8.6601</b>	<b>8.2x10<sup>13</sup></b>	<b>1.6</b>
<b>5</b>	<b>50</b>	<b>8.6264</b>	<b>9.3x10<sup>13</sup></b>	<b>1.8</b>

**Samples were plated at 10 mA/cm<sup>2</sup> current density.**

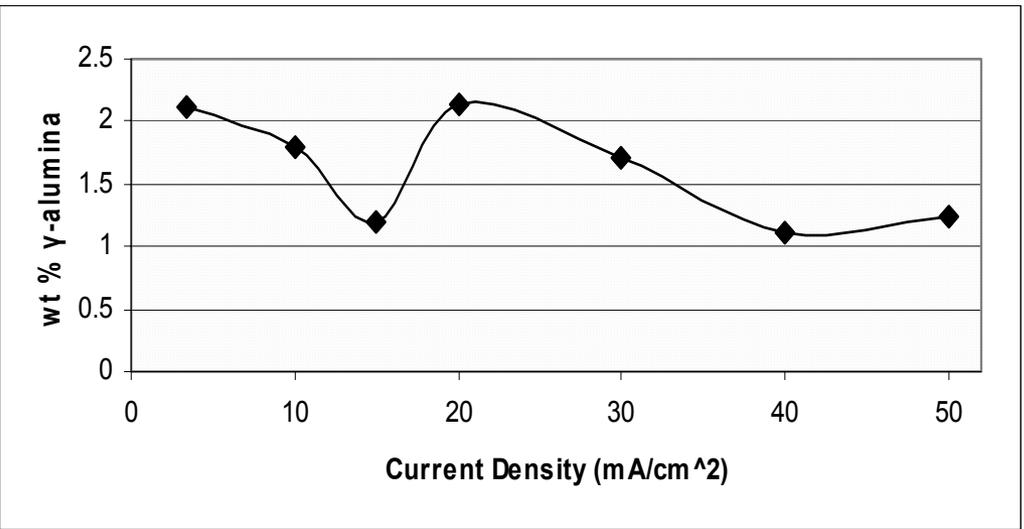
# *Estimated mass densities at Higher Particle Loadings*

$$\frac{dN}{dt} = \frac{\alpha S N_B}{\tau} \Rightarrow \%wt = \frac{\alpha C_B}{\rho_{Ni} D + \alpha C_B} \quad \alpha = 1.8 \times 10^{-5} \text{ cm}$$

<b>Particle Loading (gm/L)</b>	<b>% Wt</b>	<b><math>\rho</math> (gm/cm<sup>3</sup>)</b>
<b>20</b>	<b>0.8</b>	<b>8.8</b>
<b>50</b>	<b>2.0</b>	<b>8.6</b>
<b>100</b>	<b>3.9</b>	<b>8.4</b>
<b>150</b>	<b>5.8</b>	<b>8.1</b>
<b>200</b>	<b>7.5</b>	<b>7.9</b>

# Results from DC Plating

Current Density (mA/cm <sup>2</sup> )	Time (hrs)	m <sub>comp</sub> (g)	ρ <sub>comp</sub> (g/cm <sup>3</sup> )	Al <sub>2</sub> O <sub>3</sub> Wt %
3.4	64.5	11.49	8.583	2.1
10	20.92	11.37	8.6264	1.8
15	14	11.36	8.713	1.2
20	9	9.179	8.58	2.1
30	7.25	10.67	8.6391	1.7
40	5.25	11.38	8.7262	1.1
50	4.167	11.25	8.7075	1.2



$$\text{Al}_2\text{O}_3 \text{ Wt \%} = 100 * \frac{\rho_{\text{Al}_2\text{O}_3} \left( \frac{\rho_{\text{Ni}}}{\rho_{\text{comp}}} - 1 \right)}{\rho_{\text{Ni}} - \rho_{\text{Al}_2\text{O}_3}}$$

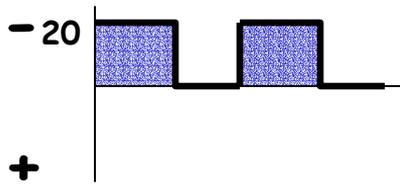
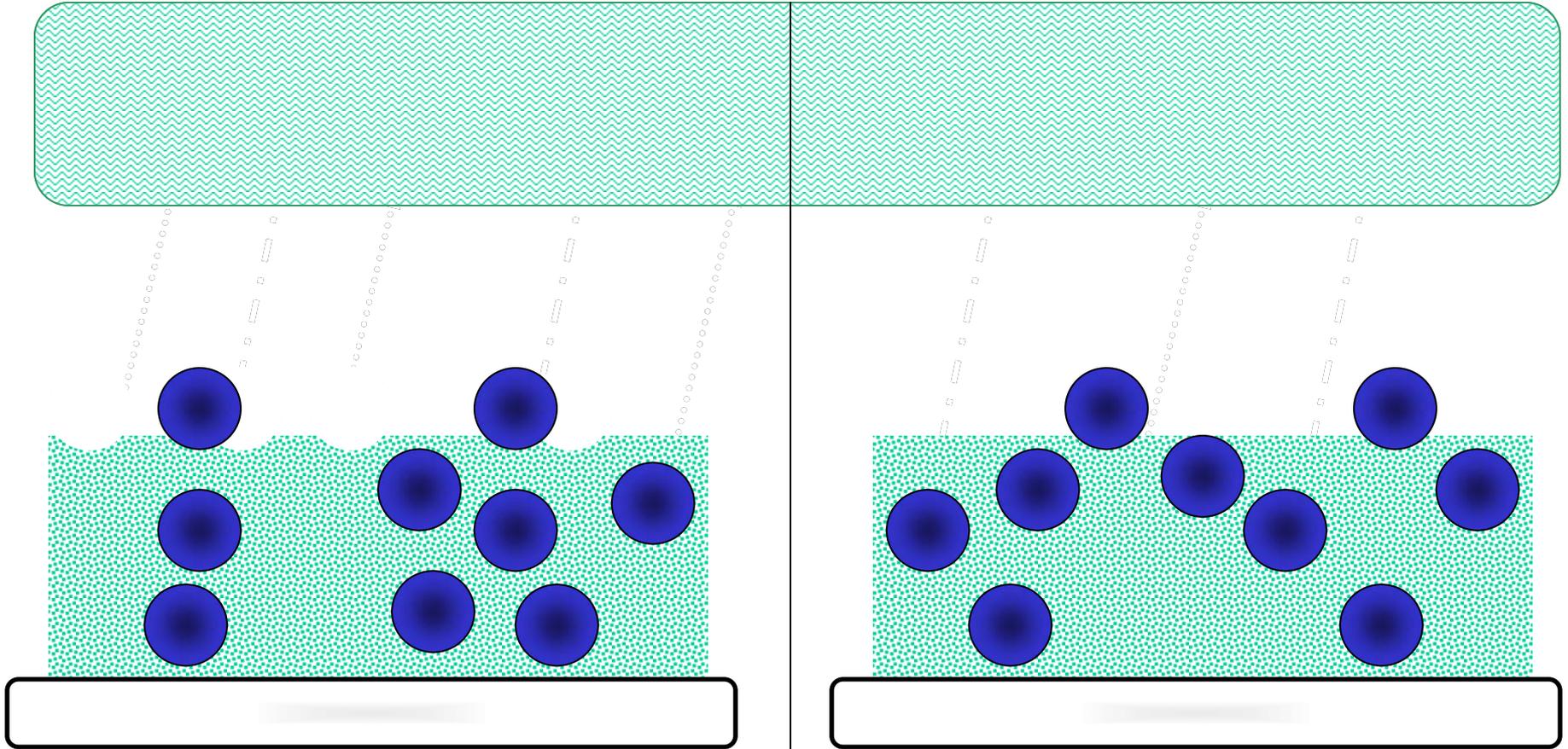
$$\rho_{\text{Al}_2\text{O}_3} = 3.3 \text{ g/cm}^3$$

$$\rho_{\text{Ni}} = 8.89 \text{ g/cm}^3$$

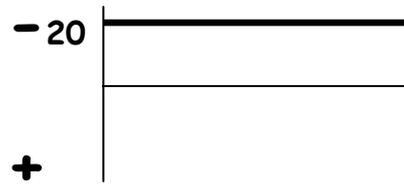
# *Pulse Plating*

<b><math>J_{on}</math> (mA/cm<sup>2</sup>)</b>	<b><math>t_{on}/t_{off}</math> (sec)</b>	<b><math>\rho_{com}</math> (gm/cm<sup>3</sup>)</b>	<b>% Wt</b>
<b>20</b>	<b>3.0/5.0</b>	<b>8.6964</b>	<b>1.3</b>
<b>40</b>	<b>3.0/5.0</b>	<b>8.6431</b>	<b>1.7</b>
<b>60</b>	<b>3.0/5.0</b>	<b>8.7202</b>	<b>1.2</b>

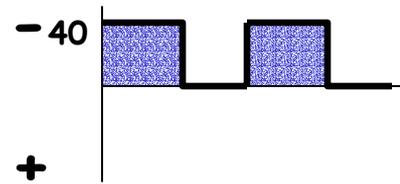
# Possible Pulse Plating Explanation



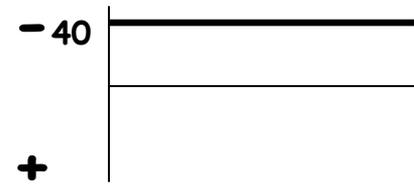
$$n = 6.93 \times 10^{13} / \text{cm}^3$$



$$n = 1.09 \times 10^{14} / \text{cm}^3$$



$$n = 8.77 \times 10^{13} / \text{cm}^3$$



$$n = 5.93 \times 10^{13} / \text{cm}^3$$

# *Reverse Pulse Plating - Short Pulses*

<b>Sample #</b>	<b><math>J_c</math> (mA/cm<sup>2</sup>)</b>	<b><math>t_c</math> (ms)</b>	<b><math>J_a</math> (mA/cm<sup>2</sup>)</b>	<b><math>t_a</math> (ms)</b>	<b><math>\rho_{com}</math> (gm/cm<sup>3</sup>)</b>
<b>16</b>	<b>40</b>	<b>500</b>	<b>- 40</b>	<b>100</b>	<b>8.6923</b>
<b>17</b>	<b>40</b>	<b>100</b>	<b>- 40</b>	<b>20</b>	<b>8.7767</b>
<b>18</b>	<b>40</b>	<b>100</b>	<b>-80</b>	<b>20</b>	<b>8.7443</b>

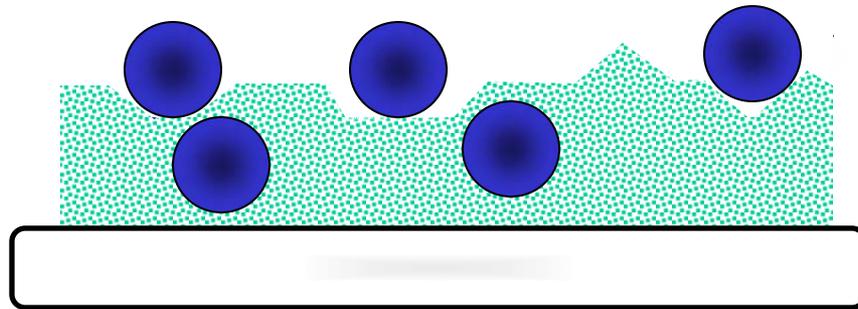
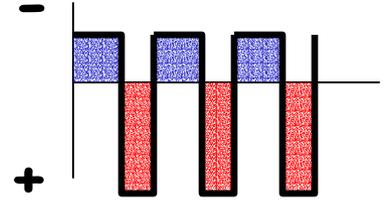
**Mass densities of the samples shown in the table are not significantly different than 8.7262 gm/cm<sup>3</sup>, the mass density of the composite plate by 40 mA/cm<sup>2</sup> direct current.**

# *Reverse Pulse Plating - Long Pulses*

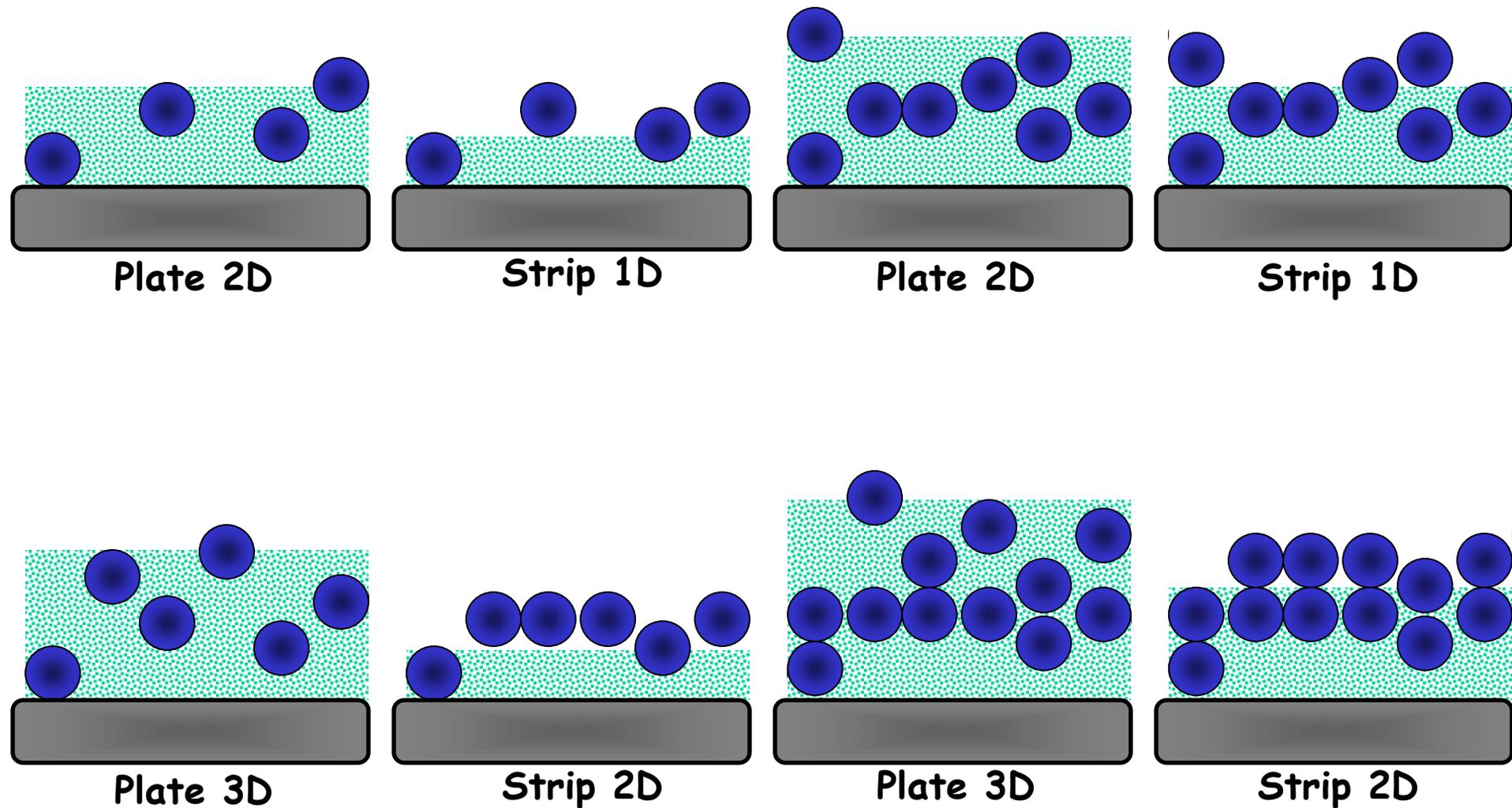
#	$J_c$ (mA/cm <sup>2</sup> )	$T_c$ (sec)	$J_a$ (mA/cm <sup>2</sup> )	$T_a$ (sec)	$\rho_{com}$ (gm/cm <sup>3</sup> )	% Wt
20	20	15.0	-40	3.0	7.072	13.9
21	20	22.0	-40	7.0	6.4	23.0

Both samples were first plated with 20 mA/cm<sup>2</sup> direct current for 30 minutes (about 0.2  $\mu$ m thick), then followed by reverse pulse plating as described in the table. At 20 mA/cm<sup>2</sup> current density, 7 seconds are required to grow 0.05  $\mu$ m thickness of the nickel composite, equivalent to the diameter of the alumina particles.

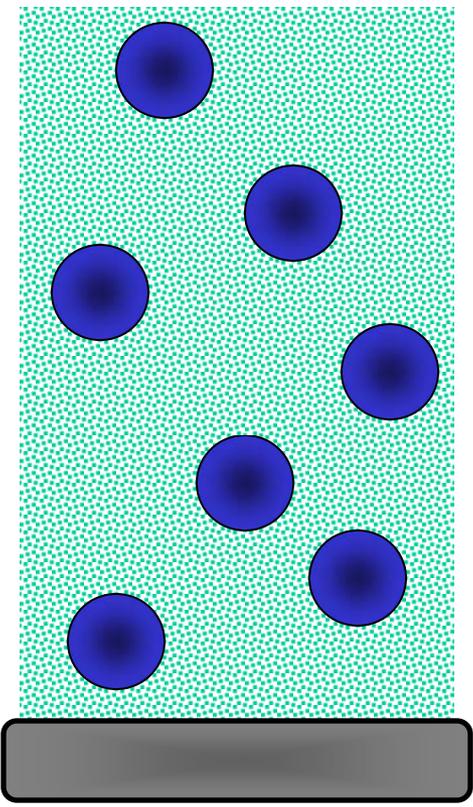
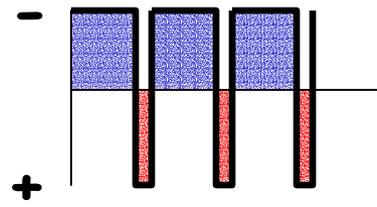
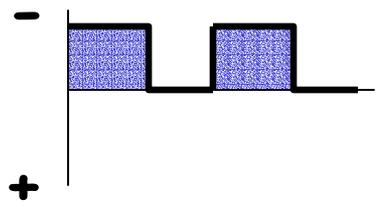
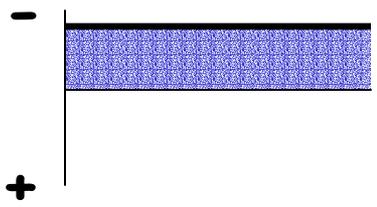
# *Reverse-Pulse Polarization*



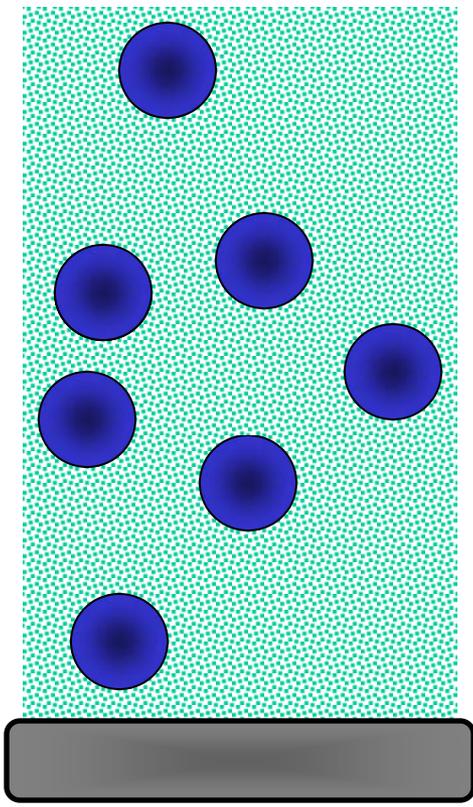
# *Comparison of Reverse-Pulse Plating Parameters*



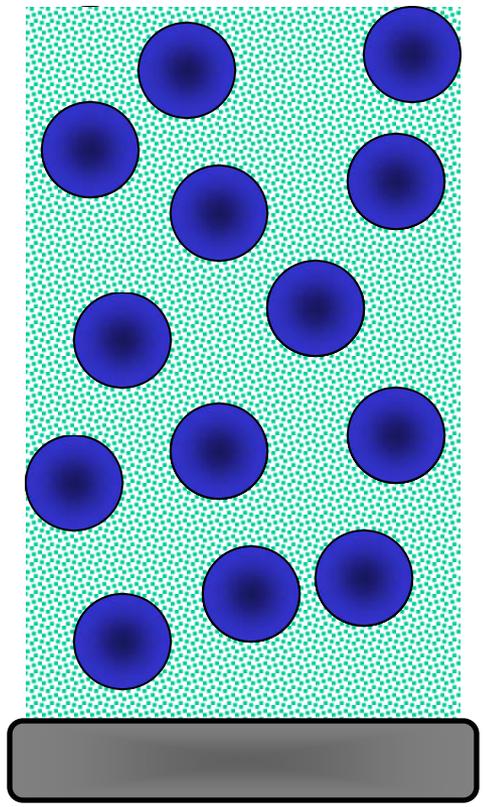
# Overview



DC

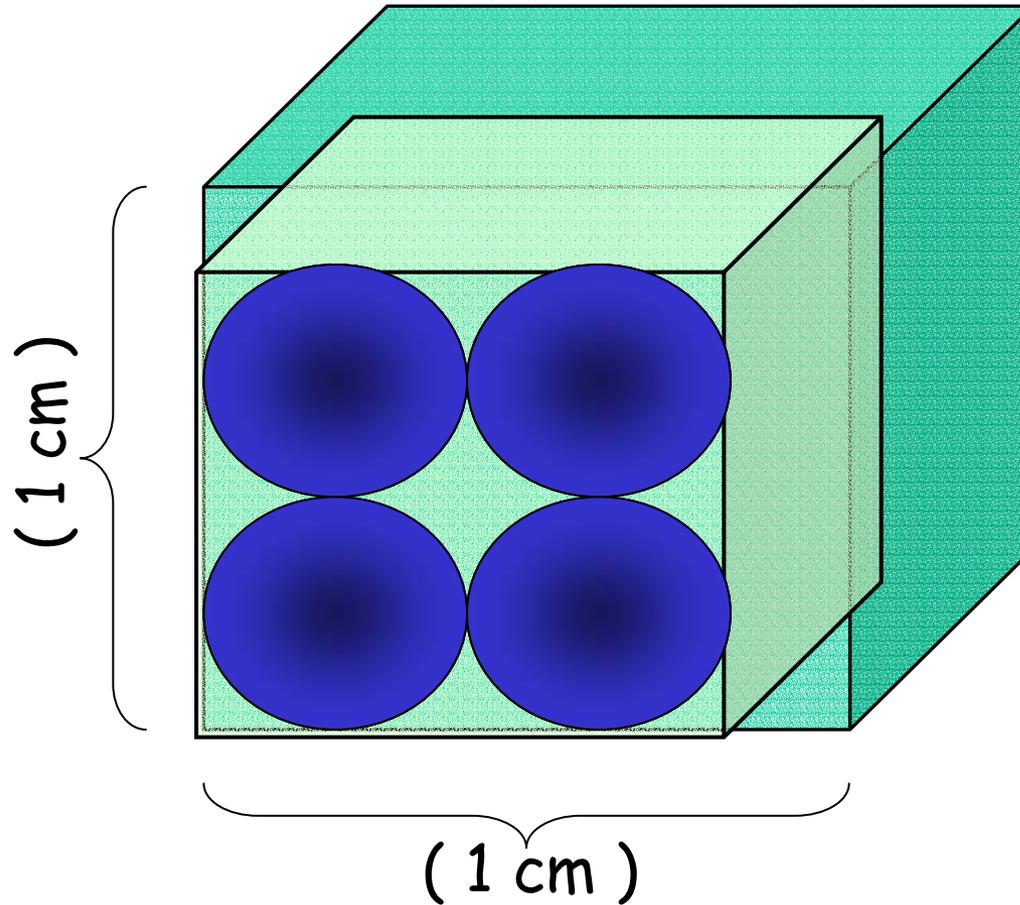


Regular Pulse



Reverse-Pulse

# Theoretical Minimum Density



$$\rho_{comp-min} = \frac{\pi}{6} (\rho_{Al_2O_3} - \rho_{Ni}) + \rho_{Ni} = 5.96 \text{ gm} / \text{cm}^3$$

# *Reverse Pulse Plating - Long Pulses with NTSA additive*

<b>Sample #</b>	<b>T<sub>c</sub> (s)</b>	<b>T<sub>a</sub> (s)</b>	<b>Plating</b>	<b>Stripping</b>	<b>ρ<sub>com</sub> (gm/cm<sup>3</sup>)</b>	<b>% Wt</b>
<b>25</b>		<b>0</b>	<b>DC Plating</b>		<b>8.7573</b>	<b>1.4</b>
<b>22*</b>	<b>15</b>	<b>3.0</b>	<b>2D</b>	<b>1D</b>	<b>7.9007</b>	<b>7.5</b>
<b>23*</b>	<b>23</b>	<b>7.0</b>	<b>3D</b>	<b>2D</b>	<b>7.0307</b>	<b>16</b>
<b>24*</b>	<b>29</b>	<b>10.0</b>	<b>4D</b>	<b>3D</b>	<b>6.4817</b>	<b>22</b>
<b>26*</b>	<b>37</b>	<b>14.0</b>	<b>5D</b>	<b>4D</b>	<b>6.7213</b>	<b>19</b>

**3.1 gm/L naphthalene trisulfonic acid (NTSA), a stress reducer, was added into the plating bath prior to these samples. All samples were first plated with 20 mA/cm<sup>2</sup> direct current for 30 minutes (about 0.2 μm thick), then followed by reverse pulse plating as described in the table.**

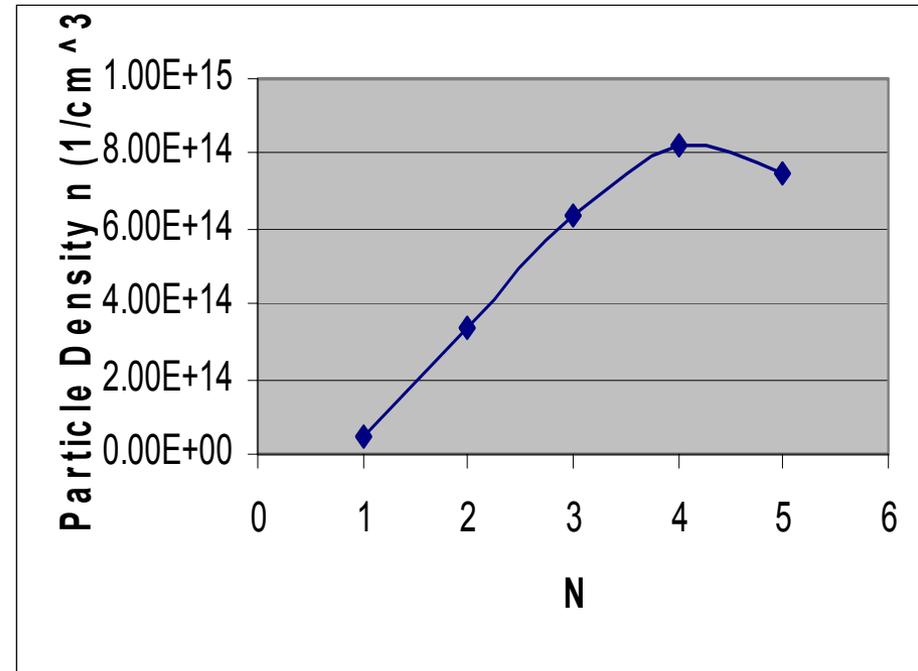
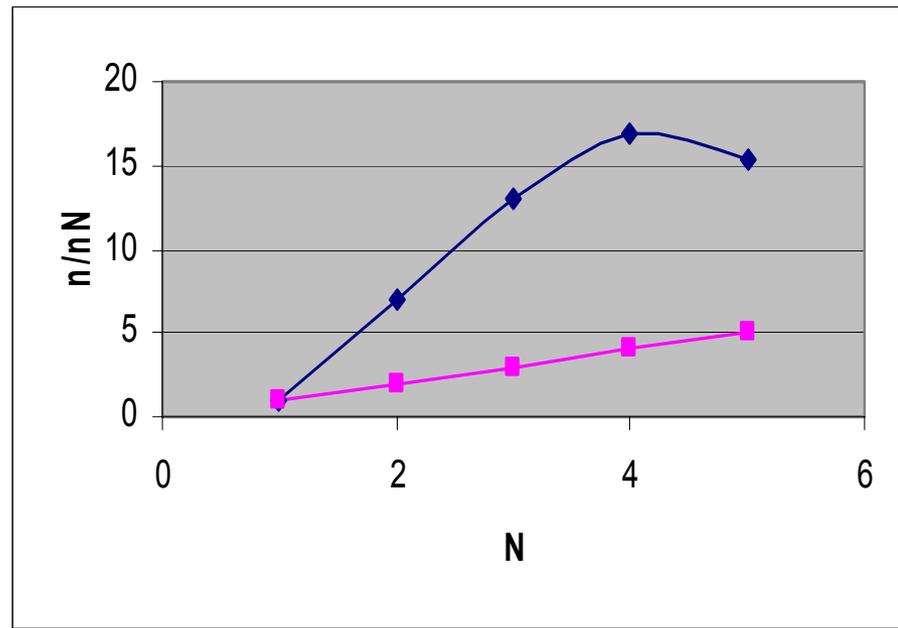
# *NTSA Additive Effect*

<b>Conditions</b>	<b><math>\rho</math> (gm/cm<sup>3</sup>) (without NTSA)</b>	<b><math>\rho</math> (gm/cm<sup>3</sup>) with NTSA</b>
<b>DC plating</b>	<b>8.58</b>	<b>8.75</b>
<b>Plating 2D, stripping 1D</b>	<b>7.07</b>	<b>7.90</b>
<b>Plating 3D, stripping 2D</b>	<b>6.4</b>	<b>7.03</b>

**Samples plated with NTSA are smoother and brighter than the samples without NTSA**

# *NTSA Samples*

<b>N</b>	<b><math>\rho</math> (gm/cm<sup>3</sup>)</b>	<b>n (1/cm<sup>3</sup>)</b>	<b><math>n/n_N</math></b>	<b>“Ideal” <math>n/n_N</math></b>
<b>1</b>	<b>8.7573</b>	<b><math>4.87 \times 10^{13}</math></b>	<b>1</b>	<b>1</b>
<b>2</b>	<b>7.9007</b>	<b><math>3.41 \times 10^{14}</math></b>	<b>7.00</b>	<b>2</b>
<b>3</b>	<b>7.0307</b>	<b><math>6.38 \times 10^{14}</math></b>	<b>13.1</b>	<b>3</b>
<b>4</b>	<b>6.4817</b>	<b><math>8.25 \times 10^{14}</math></b>	<b>16.9</b>	<b>4</b>
<b>5</b>	<b>6.7213</b>	<b><math>7.44 \times 10^{14}</math></b>	<b>15.3</b>	<b>5</b>



# *Conclusion*

- Alumina particle inclusion in the nickel matrix is about 2% wt using DC plating method.
- Periodic pulse or short reverse pulse plating does not significantly affect particle inclusion, compared with DC plating.
- Higher particle concentration can be found when the deposit thickness per cycle approaches the particle diameter in reverse pulse plating.
- The lowest mass density formed was 6.48 gm/cm<sup>3</sup>.

# *Acknowledgements*

**The authors would like to thank William Jones for creating this research opportunity and Jimmy Perkins for his assistance in composite density measurements. This work is funded by NASA/SFFP (2003) program.**