Western Michigan University

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2016

Smart Ink for Flexo - 2

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Before formulating any kind of graphical or conductive inks, it is essential to understand how they flow or deform, and how they respond to changes in both shear rate and temperature.

Rheology helps generate a curve to analyze and characterize these changes, as well as changes in the concentration and type of pigments, binders, vehicles and additives.

Rheology curves of known commercial inks can be used as references for new formulations. Once a formulated ink displays a similar curve, it can be called successful. Similar to having a proof of a job before the real print production, a rheology trial can simulate how the ink will run in the press and if there is a need for alterations. These trials can help to save materials, time and money.

To recap the first half of the study, nano nickel pigments were used in the formulation of a conductive ink as an alternative to silver, gold and copper for certain printed electronics applications. The rheology of known flexo and screen inks was studied to compare with the formulation. A micron sized commercial nickel ink was screen printed on a paper and a PET to use as a reference for electrical characterization and its particle size and viscosity were optimized for flexo printing trials. Before purchasing the commercial nano nickel powder pigments for the formulation trials, a Turkevich based synthesis method was used to experience producing the nano nickel solution.

![Figure 1: Viscosity in response to shear rate](image-url)
RESULTS: PHASE 1

Figure 1 shows the viscosity behaviors of the inks in response to shear rate. Flexographic inks have an initial viscosity of roughly 100 Pa.s, while paste inks are about 1,000 Pa.s. All inks are viscous at lower shear rates and more fluid at higher shear rates. Although the viscosity values of the screen printing pastes and the flexographic inks look different at a low shear rate, they have overlaps after a certain point. This can be interpreted as adjusting viscosity may help to get a similar flow curve of flexographic inks. The high viscosity value of nickel ink at the beginning is due to its paste nature. The flow characteristics, especially that of silver ink, will be used as reference/control data.

Figure 2 shows the viscosity behaviors of the inks in response to temperature. Both regular ink and conductive silver ink temperature ramps are almost linear. At high temperature, the viscosity is slightly increasing. The reason for this is the evaporation of water. The screen printing paste has a phase change around 35 degrees Celsius. This behavior will be further analyzed by taking repeat measurements.

It is necessary to fit the experimental data to a model. Flow models are the best basic form of data processing in rheology. Because of this, we will next evaluate the rheology tests by finding a best fit model for the generated flow curves.

RESULTS: PHASE 2

The average particle size of the HPN-DEV nickel flake paste was determined before the size reduction through the FE-SEM images (Image 1) to be 4.3-µm. ±3.4. The shapes of the flakes were irregular.

The light absorption behavior of the nickel flake paste can be seen in Figure 2. When light passes through the sample, some portion is absorbed, and the rest reflects back and appears as a color to our eyes. Some substances absorb light, but the UV region is not visible to our eyes. UV-vis spectroscopy measures the absorption characteristics of the samples both in UV and the visible range of the spectrum. From this, we can assume the functional pigments used in the ink formulation can be activated during the sintering cycle. The NovaCentrix PulseForge has a 200-nm. to 1,500-nm. flash light output range. If the pigment particles have a peak within this range, it can be concluded they can efficiently absorb the flash light. From the data in Figure 3, it is clear nickel flake has a peak at 350-nm., which falls in the output range of the PulseForge.

The HPN-DEV nickel flake paste was screen printed on the cover board and PET for comparison purposes. The lowest sheet resistivity results were obtained for the paper samples at 1,700 microseconds and 2,000 microseconds, which correspond to 7,064 mJ/cm² and 7,857 mJ/cm², respectively. But the adhesion of the ink decreased, due to burning some of its binder content during the sintering process. In Image 2, the unbonded pigment particles can be seen around the print area. The paper samples were very good to withstand high pulse
length duration. The substrate did not get damaged. The photonic sintering procedure will be further optimized to be able to increase the adhesion.

The PET substrate had problems withstanding the higher pulse length durations. It started to shrink around 1,400-µs., which corresponds to 6,163 mJ/cm². The printed area also got bumps during the curing process due to moisture blistering (Image 3). Next, the sintering procedure for the PET will be changed to get optimum results.

Particle size images after the sonic disrupter and microfluidizer of the sample can be seen in Image 4 and Image 5. The average particle size obtained with the sonic disrupter was 2.5-µm., while the one from the microfluidizer was 7.6-µm. Some large particles and clumps of material were viewed in both samples, but overall dispersion appeared better in the sonic disrupter sample. Clumping was more obvious in the high viscosity sample that was run through the microfluidizer.

The lab equipment couldn’t reduce the nickel flake from micron to nano size. Instead, it caused the nickel particles to fuse together and form a larger cluster of particles.

HPN-DEV nickel paste originally had 62 percent solids loading. Based on the previous researchers’ methodology, the solid level reduced down to 30 percent. Then it was flexo printed on Harper’s QD flexography proofer as another control group for the experiments. The optimized ink did not perform well on the proofer (Image 6).

To further evaluate the plate quality, conductive flexo silver ink was printed on the proofer; it did perform well. The resistivity of the printed silver was 0.4 ohms/sq. Based on the print results, it was decided that the plate has the quality, but the surface tension of the adjusted ink will be optimized and the solid level of the ink will be readjusted.
RESULTS: PHASE 3

By using the Turkevich based method with careful control of the reaction parameters, two different trials were attempted to obtain nano nickel particles (Image 7). Even though the 8-nm. particle size was targeted at the beginning, test results showed that the average was a 157-nm. diameter for the first trial and 1.9-µ. for the second (Figure 4). Precipitation and agglomeration could be the reason for the bigger particle size from the second trial.

Both trial one and trial two have a peak in the PulseForge output range (Figure 5), which indicates that the nano nickel particles can be activated during the sintering cycle.

The solid content of the sample was measured using the thermogravimetry (TGA) test. Based on the results in Figure 6, the nano nickel level was less than 1 percent for the first trial and around 3 percent for the second trial. It was decided these nano nickel solutions should be further evaluated with inkjet printing instead of flexo printing, due to their very low solid content level.

RESULTS: PHASE 4

Surface tension of the CXT-0346 conductive silver (raw) ink was 30.34 mN/m (30.34 dyn/cm). Both Triton X-100 and T-DET N4 were not very effective in regard to lowering the surface tension of the conductive ink (Figure 7). The additions more or less made a difference on the raw ink. Out of curiosity, the surface tensions of the surfactants were measured (Figure 8). It was assumed that if the surface tension of the surfactant is higher than the raw ink, it is unable/limited to decreasing the tension of the ink.

Then, another surfactant was selected—Dylon 604—for the measurement. The tension of the Dylon 604 turned out to be 30.35 ± 0.11 and also was the most effective surfactant at lowering the surface tension of the raw ink (Figure 7). It was decided that Dylon 604 can be used in the next phases of the research. The effect of surfactant addition on sheet resistivity will also be evaluated.

Figure 9 shows the contact angle degrees of both DI and MI. The average angle for the DI water was 72 degrees ±1.3 and the average angle for the MI was 34 degrees ±0.4. From the Owens-Wendt formula, the surface energy of the cover paper was calculated as 44 mN/m.
dispersive component 36.89 mN/m; polar component 7.10 mN/m). The fundamental rule for ink to wet a substrate states its surface tension must be 10 mN/m lower than the substrate’s surface energy. Based on the surface energy of the cover paper, for the good wettability, the formulated inks’ surface tension must be below 34 mN/m.

Sheet resistivity results of the drawdowns obtained from binder #1, #2 and #3 were in the kiloohm and megaohm range. The calendering procedure was able to bring sheet resistivity results to the ohm range, but not lower than the results of binder #4. Figure 10 shows the resistivity results for both uncalendered and calendered samples of B#4. The lowest resistivity obtained was 1.6 ohms/sq. in. at 1,700-µm pulse length. The ink was evaluated on the flexo proofer (Image 8) but it did not perform well. The reason might be an incompatibility between the ink’s surface tension and the surface energy of the substrate. The ink tension will be measured to further evaluate the problem. The functional pigment level of the formulation will also be increased to get lower resistivity numbers.
Solid level, viscosity and binder type have high impact on both printability and resistivity. Surface tension of the ink and anilox parameters should be compatible both with the plate and surface energy of the substrate. Calendering has a high impact on reducing resistivity. Inks that perform well with drawdown may not perform identically on the proofer.

Printed electronics (PE) is one of the very important emerging fields in the industry and it is notably unavoidable. Even though there are remarkable advancements, there is still a high need for new ink formulations, new product designs and applications in the field of PE. Alternative, low cost materials will help to speed up technological innovations and the optimization of manufacturing processes. Among different types of printing methods, flexo has the advantage in terms of being the most dominant printing system in North and South America. This fact itself is enough of a reason to improve the flexo printability of printed electronics.

About the Author: Bilge Nazli Altay is a Ph.D. candidate in the paper and printing science program at Western Michigan University. Her research focuses on formulating conductive printing inks and conductive paper coatings for printed electronics applications. She is the lead teaching assistant for the school’s GPS1500 introduction to graphic and printing science course. Bilge enjoys assisting with instruction in the course (which is open to all university students), as it gives her great opportunities to interact with undergraduate students from different departments and nationalities.