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Metallic Powders for Composite Inks: Formulation, Application, and Characterization

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ABSTRACT

In this study, the suitability of four metal powders and one alloy powder for use in composite inks was investigated on the basis of the contribution by each metallic powder to the formulation, printability, hardness, rheology, conductivity and thermal properties of the inks. The effects of the binder chemistry, the composition of the inks and the application conditions on the rheological, electrical, thermal properties of the inks were investigated. The results show controlled film deposition could be achieved by using a screen printing technique. The relationship between the viscosities of the inks with an increase in temperature depends on themetal powder used in formulation and on the polymeric binder chosen. Those inks that were based on the poly(vinylpyrrolidine)-co-stypre (PVS-co-S) have shown an increase in viscosity as the flow temperature was increased above 60 °C. The resistance properties of these inks depend on the temperature at which the inks were dried. This can allow for a control on the flow of electrons in the ink prints and a balance between print hardnessand electrical properties to be achieved. Of the three substrates that were used in the studies, the inks applied onto the tinplated steel and onto the PET substrate showed signs of ink-delamination, after drying. However, no such delamination was encountered from the inks that were applied onto the paperboard substrate.

Keywords: *Metal powder; ink; conductivity; hardness; rheology; analysis*

INTRODUCTION

In recent years, composite ink formulations containing metallic powders have found applications in several technologies including the field of printed electronics, allowing a significant revolution in electronic technology to take place. Many of the substrates for such kinds of inks fall into a wide range of surface types, such as paper, plastics and fabric [1], glass, card, metal, ceramic [2] and textiles[3]. Depending on rheological properties, the ink formulations can be applied to the relevant substrates using printing processes, especially screen printing[4], ink jet printing [5] and gravure printing[6]. These three technologies in particular reduce the costs of the manufacture, the number of processing steps and the contamination of the environment dramatically, compared with traditional electronic fabrication processes [7]. The application areas of metal powder-based inks include mobile devices, e-readers, sports apparatus and sportswear, healthcare technologies, toys, games and security [8, 9]. Hence, the combination of an efficient printing process and improved electronic technology should facilitate an increase in productivity and the value in use.

Some of the common properties of composite inks, that relate to their performance, include thermal stability, hardness, tensile strength [10], flexibility and compatibility with the underlying substrate[1]. Hardness testing often involves using a pointed or spherical, hard object, brought into contact with the surface of the ink for a specified time period[11]. Thermal properties of inks can be tested using thermo gravimetric analytical methods and can help understand the drying or curing of the inks, their thermal stability and their decomposition patterns[12]. On the other hand, appropriate rheological studies can help understand the viscosity and flow characteristics of composite inks [13].

Owing to the fact that the application of metallic powder-based inks is still relatively new, there is the need to understand the precise influence of each ink component and the effect of any interactions between the ink ingredients, on the performance of the inks. Thus, the formulation and study of inks on the basis of different metallic powders and different polymeric binders will be a point to help understand more thoroughly the electrical, rheological, mechanical and thermal properties of the inks. The aim of this study is therefore to investigate and to develop composite inks that can be printed using screen printing techniques, possessing good electrical conductivity, good rheological characteristics and good thermal/mechanical properties. This will help establish an understanding of relevant mechanisms of operation of metallic powder-based composite inks.

MATERIALS AND METHODS

Materials

Paperboard that was used as substrate was supplied by Incada Exel, Proctor Paper and Board Ltd. This paperboard has a gammage of 240 g/m2, a thickness of 400 µm and a calliper value of 15.7 pt. The poly (ethylene) terephthalate (PET) based inter digitated electrode substrate was supplied by Peratech Ltd and has been pre-printed with carbon and with silver to give electrode characteristics. The tinplated steel substrates (TH415) were manufactured and supplied by Arcelor Mittal and have a thickness of 0.21mm. Relevant information on the different metallic powder samples is shown in

Table1. *Information on the different metallic powders used in formulation*

Powder	Supplier	Nature/Characteristic
Ni	Vale Inco Europe Ltd, Swansea, West Glamorgan	This contains near-spherical aggregates with a granular surface morphology. The aggregates range in size from 2.5µm to 8µm.
Fe	Sigma Aldrich Ltd, Gillingham, Dorset, UK	This iron powder contains irregular shaped particles with an approximate size range from 25µm to 100µm.
Cu	Echart UK Ltd, Unit C The Sidings Station Road Ampthill, Bedfordshirem, UK	This is a cornflake shaped material that was developed to be resistant to oxidation.
Bronze	Echart UK Ltd, Unit C The Sidings Station Road Ampthill, Bedfordshirem, UK	This is a cornflake powder which has been heat treated and is resistant to oxidation.
Zn	Obtained from Colour Science Analytical, Leeds University	This is comprised of spherical particles that range in size from 1µm to 10µm.

The polymeric binders poly (vinylpyrrolidine) (PVP) and poly (vinylpyrrolidine)-co-styrene (PVP-co-S) were purchased from Sigma Aldrich. The PVP binder is a linear non-ionic polymer that is soluble in water, having polymer chains ranging from low to high molecular weight. The PVP-co-S polymeric binder is chemically composed of 2-Pyrrolidone, 1-ethenyl-, and polymer with ethenyl benzene. Both materials in the PVS-co-S binder have an average solids contribution of ≥38 to ≤42 wt%, with water contributing ≥58 to ≤62 wt%, to the overall solids of the binder.

Doubly-distilled, deionised water, discharged from an Elga, 18 Ω, pure lab-flex-Velolia distiller, was used where relevant in the preparation of the wet ink samples and in adjusting the solids content of the various ink formulations. This grade of deionised water was also used for the contact angle measurements that were carried out.

Analysis of Metallic Powders and Polymeric Binders

Electron micrographs were recorded using a Scanning Electron Microscope (Jeol JSM-6610LV model). The sample preparation involved the deposition of a representative amount of the sample powder, onto a standard SEM stub using carbon-conductive tape. Each sample was then gold-coated using a Bio-Rad SC500 diode sputter coating unit. The sample was examined under the electron microscope over the magnification range of x 200 to x 15000, using an accelerating voltage range from 5 kV through to 30 kV.

The thermal decomposition profiles (TGA) of the ink materials were recorded using a TA l Instruments Q50 model over a temperature range of 25 − 500^{o}C, with a heating rate of 10^{o}C /min. The N2 gas balance purge flow rate was 40 mL/min and the sample purge flow rate was 60 mL/min. A small quantity of the sample, (approximately 10 mg), was used for each analysis. Differential scanning calorimetric measurement (DSC) were carried out using a TA Instruments Q20 model over a temperature range 25^{o}C – 500^{o}C, in order to monitor the transition temperatures of the polymeric binder samples.

Rheological evaluations were carried out using a rheometer, TA−Instruments (ARES-G2). The evaluations were run under a continuous shear stress ramp mode at 20^{o}C. A steel parallel plate, with a 40mm diameter was used with a truncation gap of 50 µm. The rheometer was also used for temperature ramp studies using the same plate geometry. In these, the ink samples were evaluated at a constant shear stress rate of 10 s-1, over a controlled range of temperatures between 25^{o}C and 80^{o}C.

Formulation, Application and Drying of the Composite Inks

All of the inks used in this study were formulated containing 50% (wt%) of the specified metallic powder and 50% (wt%) of the specified polymeric binder. Each binder contains approximately 50% water by weight. Each ink was formulated through a mixing process. 5g of each metal powder was weighed and added into a marked glass container followed by the addition of 5g of one option of the polymer binder (PVP or PVC-co-S). The addition of the binder to the container in each case was done under agitation, using an IKA-Werke Eurostar overhead stirrer. The mixing was continued at a speed of 50 rpm, for 10 min. This low stirrer speed was used in order not to destroy the shape and texture of the metallic powder particles, thus avoiding the loss of electrical properties.

In the application of the inks on to the substrate using screen printing, a Roku Print SD05 screen printing unit was used. Each ink was forced through the open areas of the stainless stencil mesh surface using a squeegee under 4 bars pressure. The squeegee setup was mechanically adjustable. The print setup was controlled electronically via the control panel. Each ink was screen printed onto a PET substrate, a paperboard substrate, a silver inter-digitated electrode sheet, and a tinplate steel substrate. The squeegee has a shore “A hardness” value of 80 and was supplied by Steve Wood Ltd, having an angle of 60° to the horizontal in the print direction. During the ink application, the off-contact gap between screen and print bed was set at 3 mm.

Testing Electrical Properties of the Ink Prints

Printed inter-digitated electrodes were cut in half and overlaid so that the printed experimental ink was sandwiched between two carbon electrodes. The design means that electric current must pass through the pressure sensitive ink layer. It was ensured that the electrodes and conductive tracks from each half were aligned before each group of electrodes was crimped by the gold-plated contacts. An electrical circuit was established in order to measure the relationship between the electrical properties of each ink print at a known applied force. The dried printed sample was placed on a hard flat table, while a hard cover with mass 0.2035 g and diameter 0.8 cm, was used as a base for other known weights to sit on when measuring the electrical resistance of the printed ink film. This was used to keep a constant contact area when different weights were applied. The resistance measurements were taken using a Caltek Instrument AG 1021 digital multimeter (supplied by Caltek Industrial Ltd, Hong Kong). As illustrated in Figure1, the electrode area was compressed using a set of cylindrical weights, which included 5 g, 10 g, 20 g, 50 g, 100 g, 200 g and 500 g. The multimeter’s electrodes were placed in contact with the two gold-plated contacts and the readings were recorded.

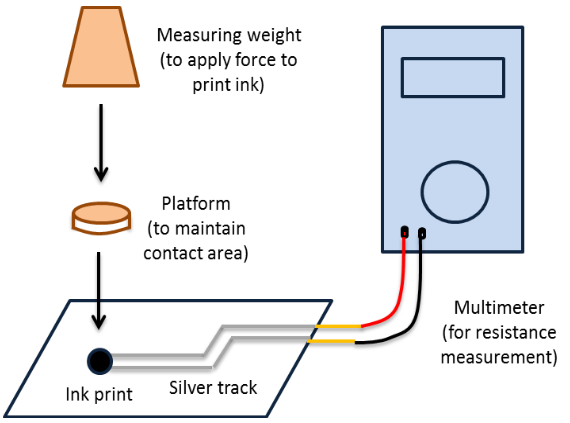


Figure1. *Schematic diagram of the setup used for measuring the electrical resistance of the ink print samples*

Testing for Hardness of the Ink Prints

The hardness (resistance to penetration) of the dried ink print surface was measured using a Sheen Instrument’s pendulum hardness rocker, type 7262. Typically, an ink print of 100 cm^2 area was used. The panel was mounted on the base that was attached to the cross bar. The counter was set to zero and the rocker was released. The hardness of the surface or the degree of dampening was measured in terms of the number of times the light beam was broken by a projecting plate that was allowed to swing to and fro through an angle of 6^{o}. With each complete rock, a value of 1 was recorded. During this procedure, the shield “door” was closed in order to protect the measurement system from interference, from air currents. In all cases, the final reading was taken when the counter finally stopped. The hardness value of each sample was measured in duplicate and an average value determined on the basis that the repeat values all fell within acceptable consistency limits.

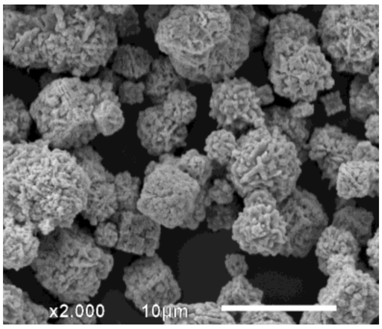
Contact Angle Measurements

Double distilled deionised water which was the solvent contained in all of the inks was used to measure the contact angle of the three substrates that were used. In the procedure, sessile drops of the deionised water were applied to the surface of each substrate material and the contact angle, that the fluid made on the surface, was recorded. Each measurement was repeated three times and an average value was adopted in each case, on the basis that the range was within acceptable limits[14].

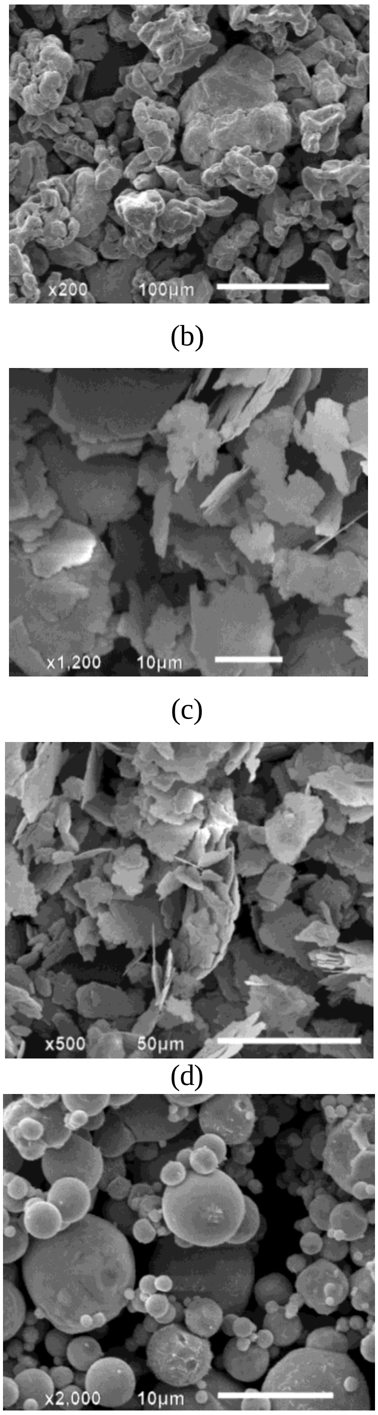
RESULTS AND DISCUSSION

Morphology of the Metal Powders

The morphology for the metallic powder particles are shown in Figure2. Obviously, the nature of the powder particles and the particle size distribution is different for each individual sample. The Ni powder can be seen to have near-spherical particle aggregates that have a granular morphology. The Fe powder can be considered to contain irregular shaped particles. Clearly, the Cu powder and the bronze powder are composed of cornflake shaped particles. In contrast to all the other metallic powders, the particles in the Zn powder are fully spherical. With these major particle shape differences between the powders, it can therefore be expected that the flow characteristics of the various inks will be dominated by shape effects and particle morphologies. Thus, for each ink, the rotation, transition, movement and alignment of the particles can govern its rheology. Results arising from rheological measurement are discussed in the next section.



(a)



(b)

(c)

(d)

(e)

Figure2. *Morphologies of (a) Ni powder and (b) Fe powder (c) Cu powder, (d) Bronze powder and (e) Zn powder, under scanning electron microscope*

Rheological Characteristics of the Ink

Ideal viscosity values and good rheological characteristics are required by the inks. Thus, it is important to monitor the flow pattern of the ink materials under highly controlled conditions relating to the applied shear stress, shear strain rate and temperature. Both Figure3 and Figure4 show that the various ink formulations studied exhibit a shear thinning flow character at 25^{o}C. The viscosities of the inks decrease with an increase in the shear rate. The shear thinning behaviour of the inks was more pronounced between shear rate values of 1 s-^{1} and 1000 s-^{1}.The value of the viscosity and the extent of shear thinning of one ink formulation, at a particular value of shear rate, are different from another ink because of differences in morphology and distribution of the particulate metallic materials in the ink samples.

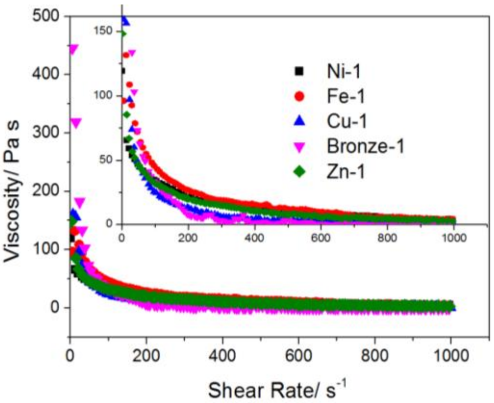


Figure3. *Viscosity/shear rate rheological behaviour of ink formulations containing the PVP binder*

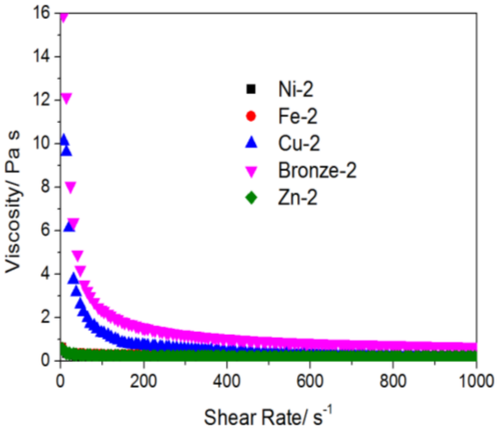


Figure4. *Viscosity/shear rate rheological behaviour of ink formulations containing the PVP-co-S binder*

The results of the study of the viscosity vs temperature relationships, Figure5 and Figure6, indicate that the viscosity of the inks depend on temperature. The viscosity decreases with an increase in the heating temperature, as a result of polymer section chains gliding past one another more easily as the thermal energy is increased. It is possible that the bronze powder-based ink shows a distinct viscosity vs temperature behaviour because as the temperature was increased, due to the water component being rapidly allowing the binder polymer to build structure, leading to an increase in viscosity.

Figure5 shows a different pattern of viscosity vs temperature compared with Figure 6. Clearly, this difference arises because the two sets of inks contained different polymeric binders. In the inks that contain the PvP-co-Styrene, as the heating temperature reached 60°C, curing of the inks commenced. Also, it is possible that as the temperature of the ink formulations were increased up to 60 °C, the formation of metallic powder aggregates was favoured. This therefore increases the resistance of the inks to flow and leads to a significant increase in viscosity. The overall data shows that during application of the inks, careful consideration needs to be given to the temperature at which the process is being conducted. If an undesirable change in viscosity occurs during application, the ease of application and the overall properties of the inks can significantly be affected.

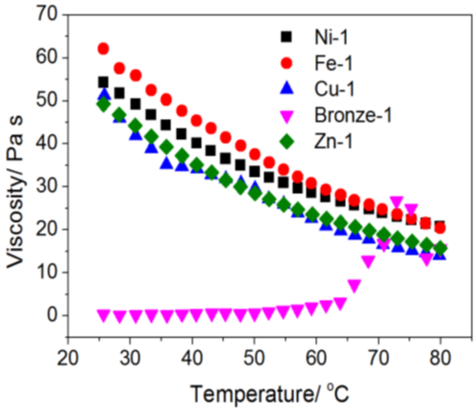


Figure5. *Viscosity/temperature profile of the different ink formulations, each containing the PvP polymeric binder*

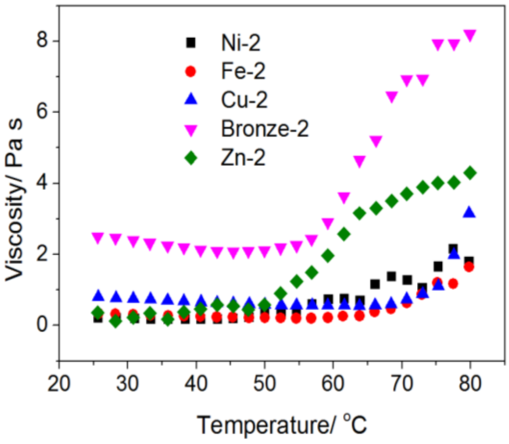


Figure6. *Viscosity/temperature profile of the different ink formulations, each containing the PvP-co-S polymeric binder*

Electrical Resistivity/Conductivity Properties of the Inks

All of the ink prints that were formulated containing Cu powder, Bronze powder and Zn powder were found to possess electrically insulating behaviour. Following a maximum pressure of 2 Kg being applied to the prints does not result in any conductivity in the materials. On the basis of the insulating property of these ink prints, only the electrical properties of Ni powder-based prints and Fe powder-based prints were further investigated.

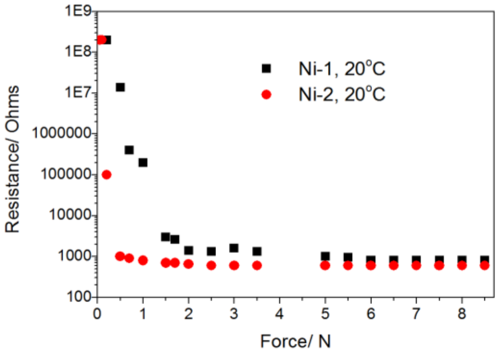


Figure7. *Electrical properties of Ni powder-based prints, (1) containing PVP binder and (2) containing PVP-co-S binder*

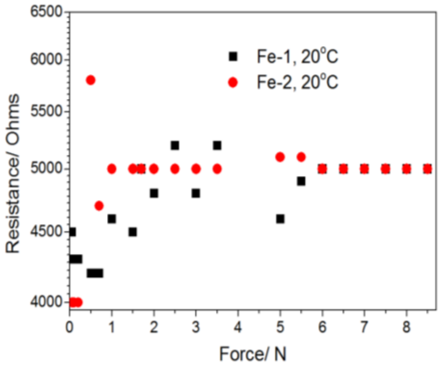


Figure8. *Electrical properties of Fe powder-based prints, (1) containing PVP binder and (2) containing PVP-co-S binder*

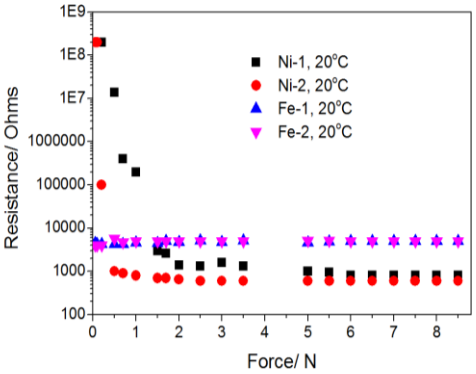


Figure9. *Overlay of electrical properties of Nipowder-based prints and the Fe powder-based prints, (1) containing PVP binder and (2) containing PVP-co-S binder*

Both the Ni powder-based ink prints and the Fe powder-based inks exhibited pressure electrically sensitive properties, as shown in Figure7 to Figure9. As a result, the electrical resistance of the ink prints changes exponentially with increasing applied forces. However, both ink prints were electrically insulating when no pressure was applied to their respective surfaces. Following the increase in applied pressure, the prints changed from an insulating behaviour to a conductive nature. Ni-1 based ink prints showed controlled electrically sensing behaviour and Ni-2 based inks behave more like electrically switching materials. Electrical sensing behaviour means that the resistance gradually changes with an increasing force. Electrical switching behaviour means that the resistance rapidly changes with increasing force. The contrasting behaviour shown by two Ni powder-based ink prints arises because of the nature of the polymeric binders that the two inks contain, i.e. PVP and PVP-co-S. The characteristics of the two different polymeric binders (PVP and PVP-co-S)are discussed in the next section.

In Figure7 to Figure9, both Fe powder-based ink prints have shown that the electrical properties of the prints change with the applied force. The prints have behaved electrically conductive when no pressure is applied. However, the resistance values have fluctuated with changing applied forces. This is because of the characteristics nature of the metallic Fe powder in comparison to the Ni powder material, as shown and discussed with respect to Figure2. In Figure10 and Figure11, it can be seen that the presence of either of the metallic powder in both polymeric binder options did not result in any chemical interaction between the metallic particles and the host polymer chains. When these data were compared with the FT-IR spectra of both binders, there were no noticeable differences in the peaks and therefore this indicates that there are no chemical interactions between the metallic particles and the polymeric materials.

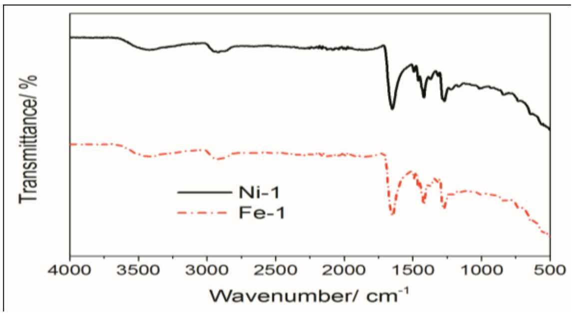


Figure10. *FT-IR data from ink prints that were formulated (and then dried at 40°C) containing either with Ni powder or with Fe powder. Both inks contain the PVP polymeric binder*

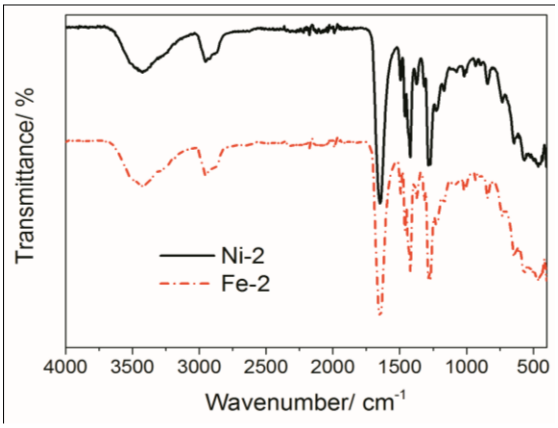


Figure11. *FT-IR data from ink prints that were formulated (and then dried at 40oC) containing either with Ni powder or with Fe powder. Both inks contain the PVP-co-S binder*

Effect of Drying/Curing Temperature on Electrical Resistivity/Conductivity Properties of the Inks

With respect to Figure2 and Figure3, the temperature at which the ink prints were dried has an effect on the electrical characteristics of the inks. Drying the prints at 100°C makes them less conductive (highest values of resistivity) compared to the conductivity of the ink prints after drying at 20°C. In particular, after drying at 100°C, the Ni-1 ink print has lost its switch characteristic as can be seen by the linearity of the Ni-1 ink print plot, in Figure2. It can be said that the higher the dying temperature, the less flexible are the ink prints and as such the electrons in the print have less ability to move and generate current.

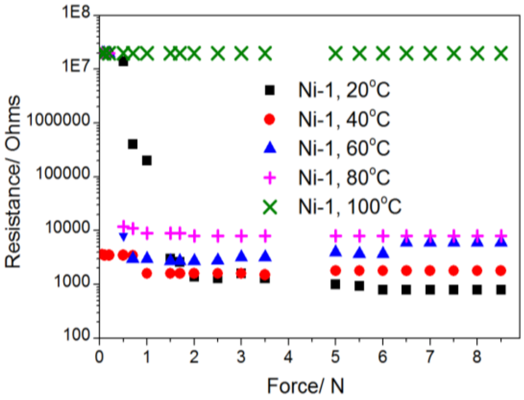


Figure22. *Plots of resistance vs force, for the ink prints that contained Ni powder and that were dried at different temperatures. Each contains the PVP polymeric binder*

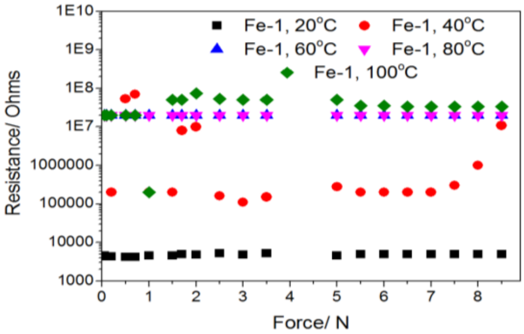


Figure33. *Plots of resistance vs force, for the ink prints that contained Fe powder and that were dried at different temperatures. Each contains the PVP polymeric binder*

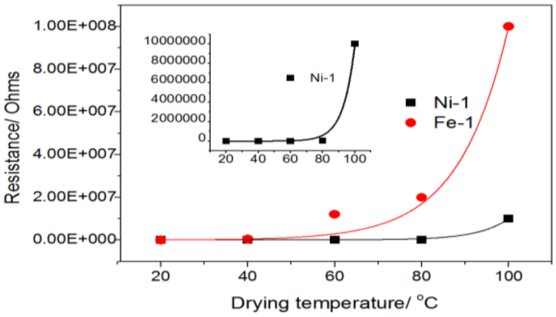


Figure14. *Plot of the relationship between resistance and drying temperature, from ink prints that were formulated containing either with Ni powder or with Fe powder. Both inks contain the PVP polymeric binder*

As shown in Figure14, the resistivity of the ink prints increases exponentially with an increase in the drying temperature. This is associated with an increase in the extent of drying/curing in the inks as the temperature was increased. Therefore, as more structure was formed in the inks, following the temperature increase, the ability/mobility of the electrons is affected. The results show that for any intended application, the temperature at which the inks are dried is of paramount importance. This choice of temperature will not only affect the conductivity of the inks, but will also affect their mechanical properties, such as hardness and flexibility. Also, producing ink prints with too much hardness and reduced flexibility might cause cracks/gaps in the ink film which will in turn restrict the movement of the electrons in such inks. Examples of delamination in some Fe powder based inks are shown and discussed in the next section, in connection with Figure17.

Properties of the Binders and their Influence on Hardness and Print Film Formation

Decomposition arising during the TGA induced breakdown of the polymeric binders is shown in Figure43. Differences in thermal stability can be observed between the two polymeric binders. The extent to which the PVP-co-S binder holds onto water is greater than that shown by the PVP polymeric binder. The overall thermal stability/reactivity of the binders however is similar. In the TGA profile of the binders, each thermo gram indicates a curve which can be correlated to loss of water, as the temperature is increased up to around 100°C and 150°C for the PVP binder and for the PVP-co-S binder, respectively. This is then followed by any self-cross linking reactions that take place up to approximately 400 °C. Comparison of the self-crosslinking curve in Figure4, for each polymeric binder with Figur, suggests that the properties of each ink can vary with the temperature of drying, or curing.

It was discussed with respect to Figure5 and Figure6, that the rheological properties of the ink formulations can be affected by the temperature of flow, or the temperature of application. Considering the fact that the PVP-co-S polymeric binder has shown greater affinity to solvent components (Figure4) and has a less intense endothermic peak (Figur, 80°C to 200°C), this can allow for a greater extent of structure formation in those inks containing the PVP-co-S binder, in comparison to those containing the PVP binder. This resulted in differences in viscosity vs temperature trends between the two sets of inks. This point can be explored further where application temperatures on such inks are to be studied in more detail.

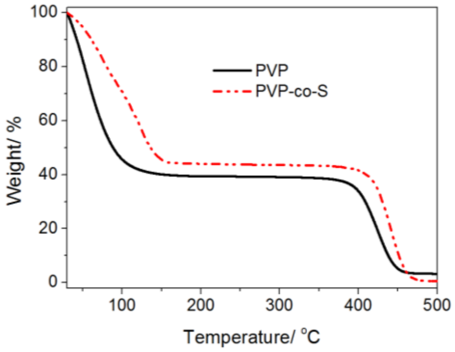


Figure45. *TGA, 10°C/min plots of the polymeric binders that were used in formulating the inks, PVP binder and PVP-co-S binder*

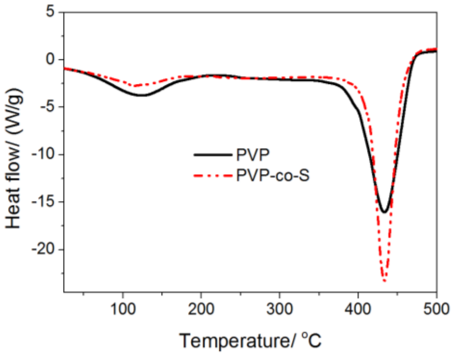


Figure16. *DSC, 10°C/min plots of the polymeric binders that were used in formulating the inks, PVP binder and PVP-co-S binder*

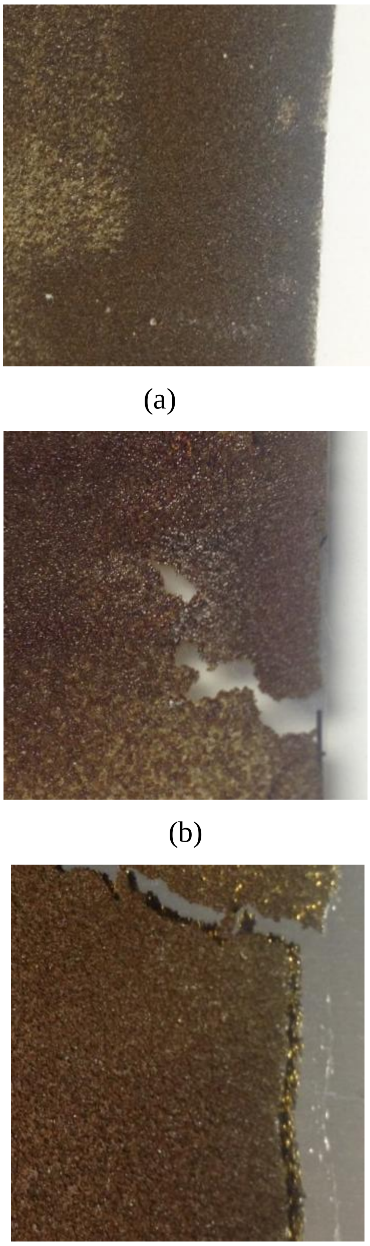
Table2. *Hardness value of ink prints that were applied to different substrates*

Sample Ink	Binder	Metal Substrate	PET Substrate	Paper Board Substrate
Ni-1	PVP	42 ± 2	45 ± 3	38 ± 5
Fe-1	PVP	41 ± 3	48 ± 2	38 ± 2
Ni-2	PVP-co-S	54 ± 4	68 ± 3	18 ± 1
Fe-2	PVP-co-S	36 ± 4	43 ± 2	22 ± 1

Table2 shows the effect of using different substrates on the hardness of the ink prints. The overall order of hardness of the ink prints is: ink prints on PET substrate > ink prints on metal substrate > ink prints on paper board substrate. Thus, the choice of the substrate would be important in determining the overall electrical properties of the ink prints. This observation is valid since Figure12 to Figure14 inclusive suggest that the extent of drying, the hardness/flexibility, and the conductivity of the inks, can be related.

The problems that can be encountered when using the inks on PET substrate, or on tinplated steel substrates, include spreading problems and blistering of thick films. With respect to some of the inks that were formulated with the Fe powder, applied onto the two substrates, delamination of the prints was encountered. Since the inks were formulated containing water as the solvent, the aspect of dried ink delamination can be related to the wetting character of the ink on the surface of the substrate. Water has a surface tension value of 72 mNm-^{1} and PET substrates are known to have low surface energy values. However, the wett ability of an ink on a PET substrate can be enhanced by employing appropriate surface energy pre-treatment procedures[15].

From the drop test measurements, the contact angle of water on PET was found to be 73.5°.The contact angle of water on the tinplated substrate was 80^{o}. In contrast, the contact angle of the same solvent on the paperboard substrate was 50^{o}. A proportion of the ink on the paperboard substrate must be absorbed during the wetting process and this must have enhanced the overall adhesion of the inks to the substrate.


(a)

(b)

(c)

Figure57. *Ink prints based on PVP, dried at 20 ^{o}C: (a) on paperboard substrate, (b) on PET substrate and (c) on tinplated steel substrate*

CONCLUSIONS

Ink formulations and ink prints were successfully developed based on the use of different options of metallic powder particles. The choice of polymeric binder has a significant effect on the rheological properties of the inks but had limited effect on the electrical properties of the inks. The choice of polymeric binder in formulation and the choice of substrate in application also affect the hardness of the ink prints. The electrical properties of the Ni powder containing ink prints and the Fe powder containing prints depend upon the temperature at which the inks are dried. Conductivity of the inks generally decreased with an increase in the drying temperature. None of the inks that were formulated on the basis of the Cu, the bronze and the Zn metallic powders, showed any desirable electrical conductivity properties. Such inks electrically insulated and were not found to have any potential for use in pressure sensitive electrical devices, in contrast to those inks that were formulated and applied, based on the Ni metallic powder and the Fe metallic powder.

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