Conductive Inks With Eproxy Resin Based Vehicles For Perovskite Screen Printing Metallization

INTRODUCTION

The object of this project have been working on developing new conductive ink formulas that use, on the one hand, epoxy resin glues and anhydrous solvents as vehicles and, on the other, conductive powders and flakes of different purities and sizes. The reason for using resin glues lies in that it allows to produce inks with high adhesion capabilities which can be cured at low temperatures. The use of anhydrous solvents enables the possibility of tweaking the ink in order to incorporate higher amounts of conductive materials, while ensuring the absence of water based components in the final mix.

The present poster summarizes the methodology and first results achieved in this ongoing project.

METHODOLOGY

Phase I: Mixing inks with conductive graphite powders, epoxy resin and different anhydrous solvents.

Phase II: Deposition by screen printing and doctor blade.

Phase III: Contact drying and curing at low temperatures.

Phase IV: Characterization

- Visual appearance by digital microscope.
- Resistance and resistivity by semiconductor characterization system.

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EXPERIMENTAL

Phase I: Mixing inks

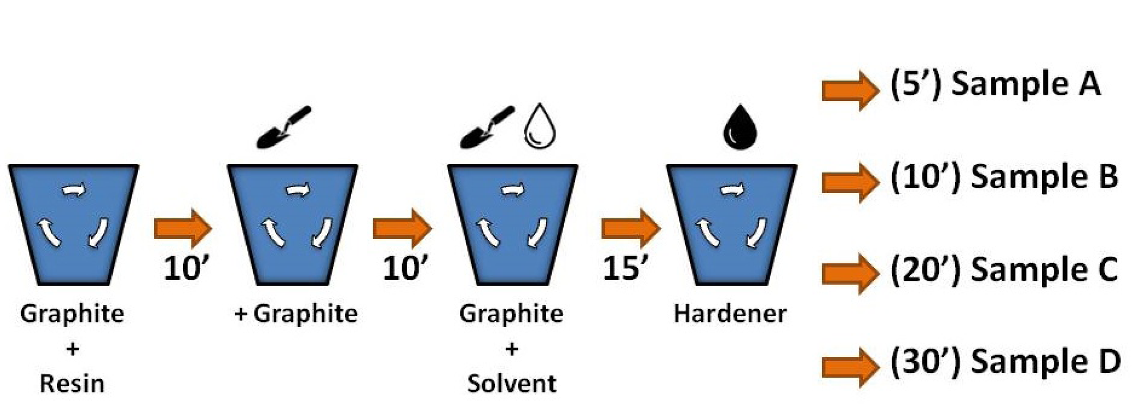

Figure 1: Fourth stages mixing procedure. Conducted with graphite powders, epoxy resin and different anhydrous solvents. Samples extracted at 5, 10, 20, 30 minutes after adding the hardener.

Phase II: Screen Printing

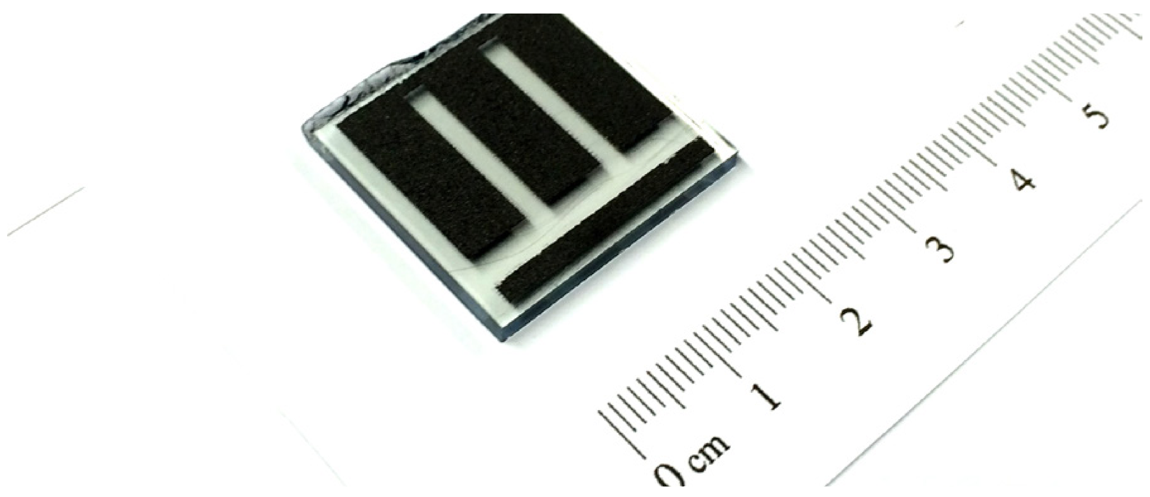

Figure 2: 10 µm thick contact example, deposited by screen printing (200 mesh, 0.040 D-Ø x 22.5 º, Squeegee blade: 70 durometer) over glass substrate (25mm x 25mm x 2mm). Cured 10 minutes in a drying oven at 100 ºC.

Phase III: Doctor Blade

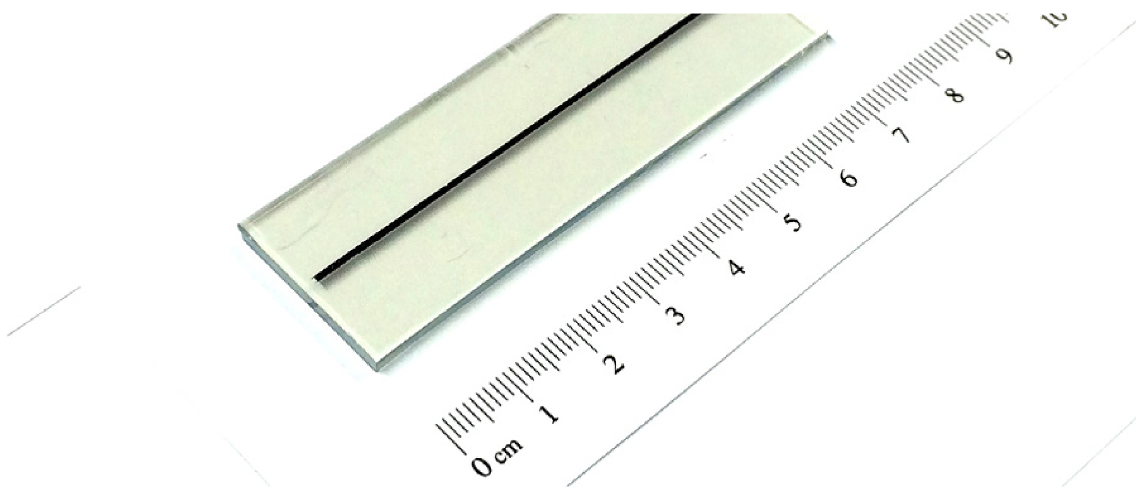

Figure 3: 63.5 µm thick contact example, deposited by doctor blade over glass substrate (25mm x 75mm) by extending the ink with a glass rod. Cured 10 minutes in a drying oven at 100 ºC.

RESULTS AND DISCUSSION

Basic ink recipe

> Target of the basic recipe:
- Suitable for screen printing.
- Conductive.

> Different mixtures to determine an optimal ratio graphite/ resin/ toluene.
> 32% / 58% / 10% = Best candidate.

Mix code	Graphite (wt%)	Resin (wt%)	Solvent (wt%)	Resistance (kΩ)	Resistivity (Ω·cm)
M19	25.97%	74.03%	0.00%	N/C	N/C
M20	35.03%	64.97%	0.00%	N/A	821.82
M21	31.30%	58.13%	10.57%	127.65	50.47
M22	28.76%	53.37%	17.87%	217.92	19.31
M23	33.33%	49.95%	16.72%	300.04	6.40

Table I: Basic ink recipe trials.
"N/C" stands for non conductive and "N/A" for non available.

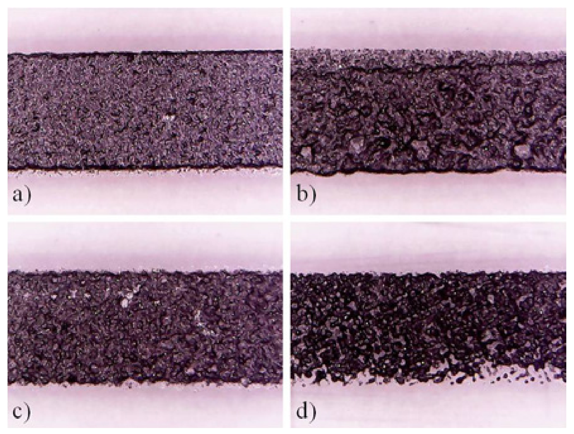

Figure 4: Microscope images showing contacts deposited by screen printing with inks M19 (a), M21 (b), M22 (c) and M23 (d).

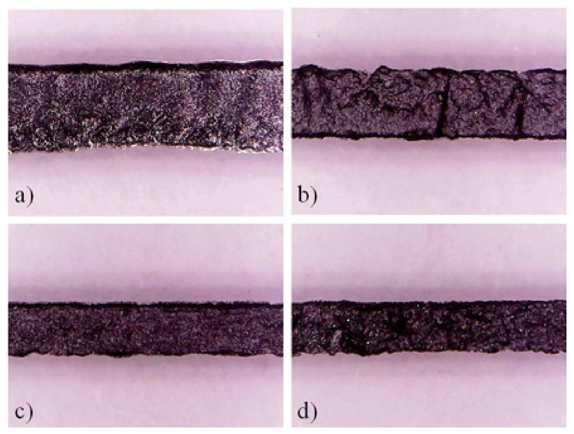

Figure 5: Microscope images showing contacts deposited by doctor blade with inks M19 (a), M21 (b), M22 (c) and M23 (d).

Ink trials with different solvents and printing times

> Study addition higher boiling point solvent.
- Effect on curing process.

> Assess printing suitability versus time.
- Optimized for screen printing purposes.

> Results:
- **Screen printing**: Acceleration on the resin bonding process leads to increase in the contact resistance.
- **Doctor blade**: Solvent density reduces the resin bonding speed during curing process.

Properties	Toluene	ClBz	DMF	NMP
Boiling point (oC)	110.6	132	153	202-204
Density (g/mL)	0.867	1.106	0.944	1.028
Vapor pressure @ 20oC (hPa	29	12	3.5	0.39
Surface tension @ 20 ºC (mN/m)	28.4	33.6	37.1	40.79

Table II: Relevant properties of the solvents used.

Time (min)	Toluene (kΩ)	Chlorobencene (kΩ)	DMF (kΩ)	NMP (kΩ)
5	142.79	201.01	120.21	153.10
10	395.73	459.83	166.93	165.75
20	447.89	380.88	298.32	366.27
30	1632.20	947.15	273.85	364.18

Table III: Resistance measured in the contacts deposited via screen printing.

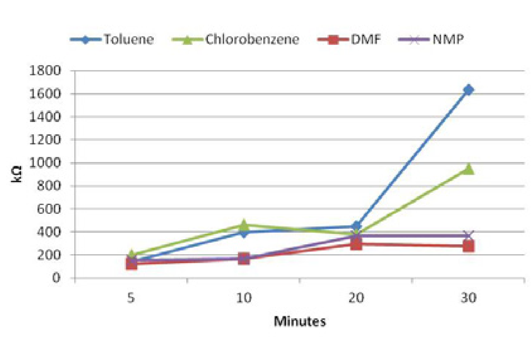

Figure 6: Resistance measured in the contacts deposited via screen printing.

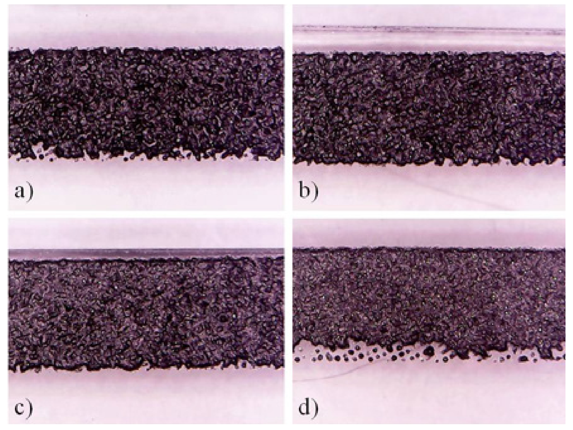

Figure 7: Microscope images showing contacts deposited by screen printing with Toluene (a), Chlorobencene (b), DMF (c) and NMP (d). All the contacts were deposited 20 minutes after adding the hardener.

Time (min)	Toluene (Ωcm)	Chlorobencene (Ωcm)	DMF(Ωcm)	NMP (Ωcm)
5	27.43	33.79	18.02	15.22
10	40.38	64.58	23.47	23.09
20	59.62	88.08	45.52	46.26
30	57.62	164.51	119.49	59.16

Table IV: Resistivity measured in the contacts deposited via Doctor blade.

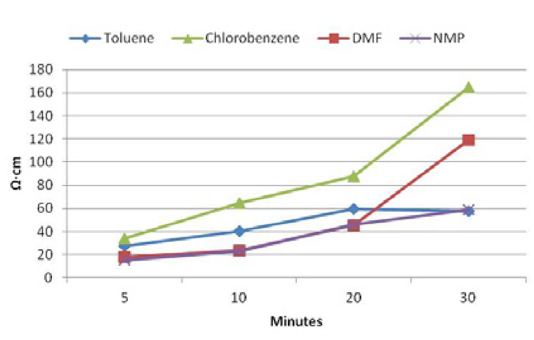

Figure 8: Resistivity measured in the contacts deposited via Doctor blade.

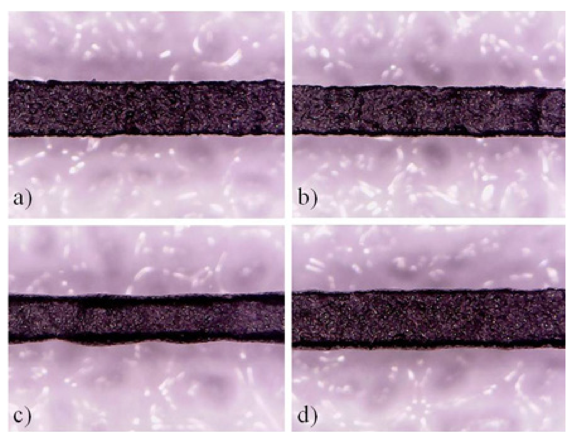

Figure 9: Microscope images showing contacts deposited by doctor blade with Toluene (a), Chlorobencene (b), DMF (c) and NMP (d). All the contacts were deposited 20 minutes after adding the hardener.

Ink trials with more graphite

> To achieve contacts with lowest resistance / resistivity.
> Solvents evaluated:
- N,N-dimethyl formamide (DMF).
- N-methyl-2-pyrrolidinone (NMP).

> Weight concentration: 35% / 58% / 7% (graphite / epoxy / solvent).
- Improvement reached but not significant.

Time (min)	DMF Resistance (kΩ)	NMP Resistance (kΩ)	DMF Resistivity (Ωcm)	NMP Resistivity (Ωcm)
5	116.28	84.59	10.88	12.24
10	154.75	123.42	28.57	22.98
20	231.94	188.04	22.98	30.12
30	257.37	267.09	22.53	30.04

Table V: Resistance and resistivity measured in the contacts deposited via screen printing and doctor blade, respectively.

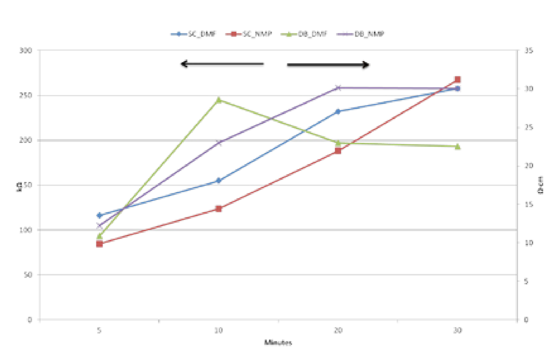

Figure 10: Resistance and resistivity measured in the contacts deposited via screen printing and doctor blade, respectively.

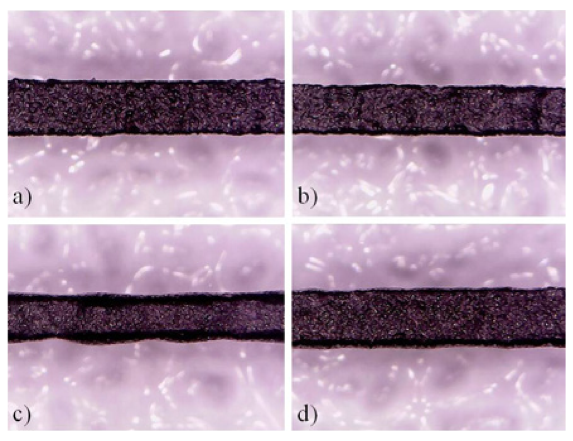

Figure 11: Microscope images showing contacts deposited by screen printing with DMF (a) and NMP (b) as well as doctor blade with DMF (c) and NMP (d). All the contacts were deposited 20 minutes after adding the hardener.

Conclusions

> A method for producing inks with epoxy resin/anhydrous solvents suitable for perovskite substrates has been developed.
> Graphite powders, as conducting material were used.
> Contacts were produced:
- Via screen printing to study their electric resistance.
- Via doctor blade to study their resistivity.

> Best screen printing contacts were achieved by recipes with DMF & NMP.
> Ways proposed for further improving the achieved results.

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