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Dependency of silver nanoparticles protective layers on sintering temperature of printed conductive structures

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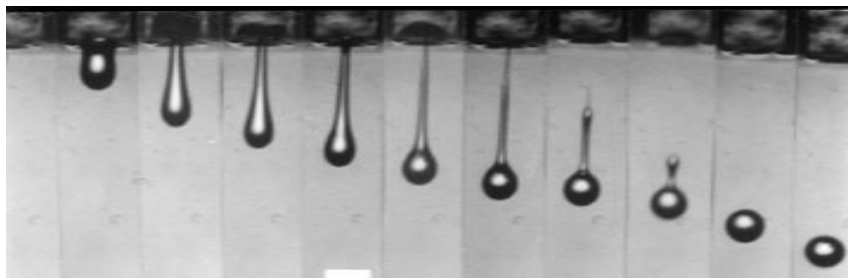
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General specification for Ink-Jet Printer

Nozzle diameter	10-100 μm
Droplet diameter	10 – 500 picoliters
Drop speed	1,5-2,5 m/s
Drop rate*	2000/s
Max. Throughput	0.5 $\mu\text{l/s}$
Viscosity range	0,5 – 20 mPas (unheated) 20-10000 mPas (heated)

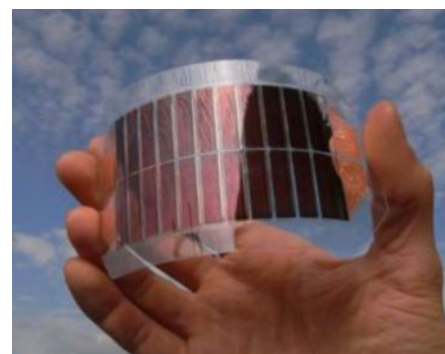
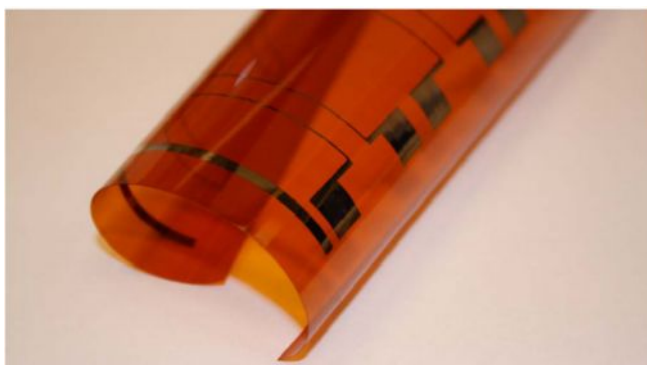
** depending on the liquid and capilar diameter*



Actual and future research

The commercially available ink \longrightarrow The ink challenge

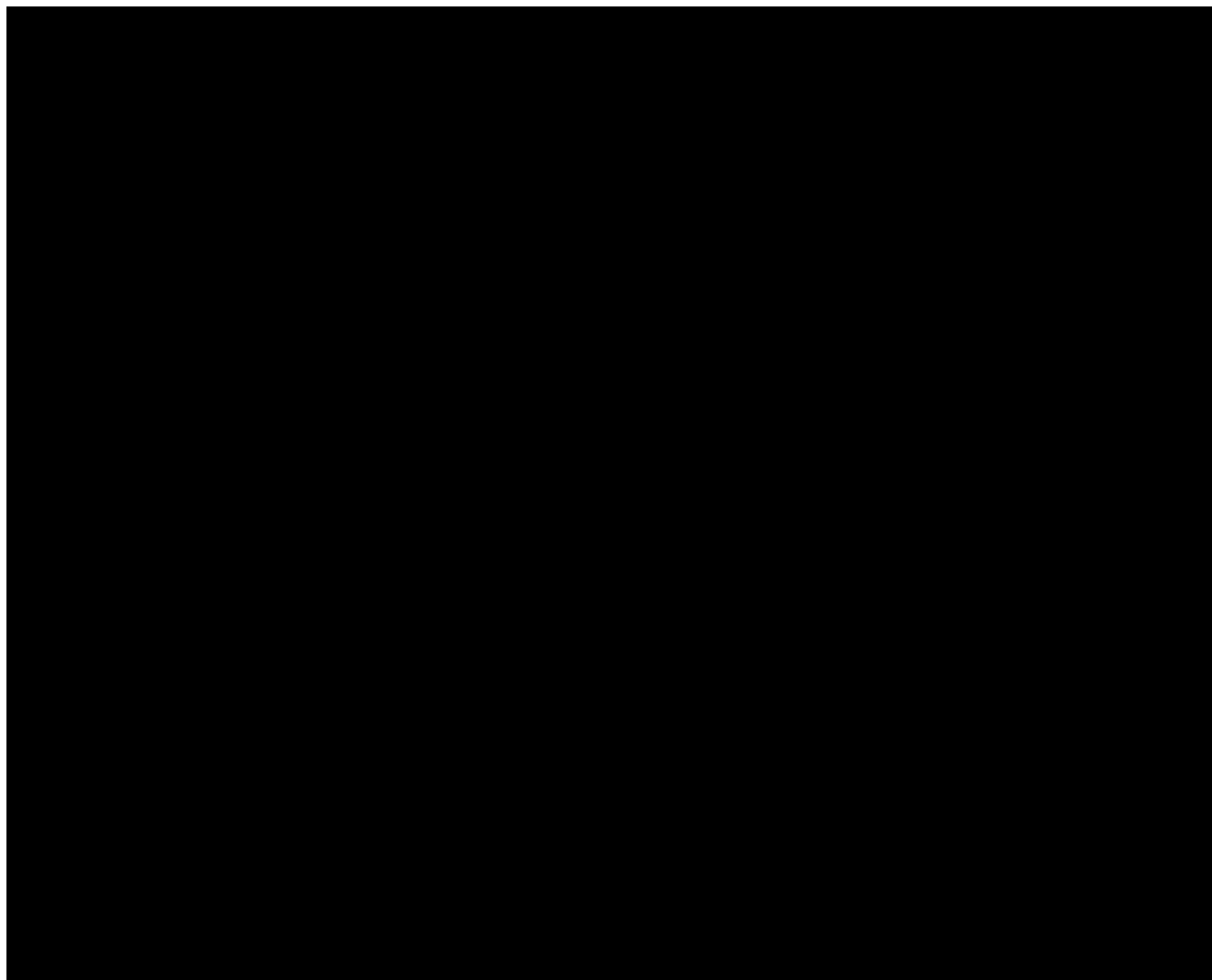
- Viscosity - 4 - 15,5 mPas
- Conductive filler - Ag
- Resistivity - 3 - 4 $\mu\Omega\text{cm}$
- Sizes of silver - 3 - 8 nm \longrightarrow 40 - 60 nm
- Content of silver - 40 - 50 % \longrightarrow 20 %
- Sintering temperature - 250 °C \longrightarrow 100 - 150 °C
- Printing possibility - non-flexible substrates \longrightarrow flexible substrates





Slow Motion Shoot NanoInk by Ink Jet Dispenser

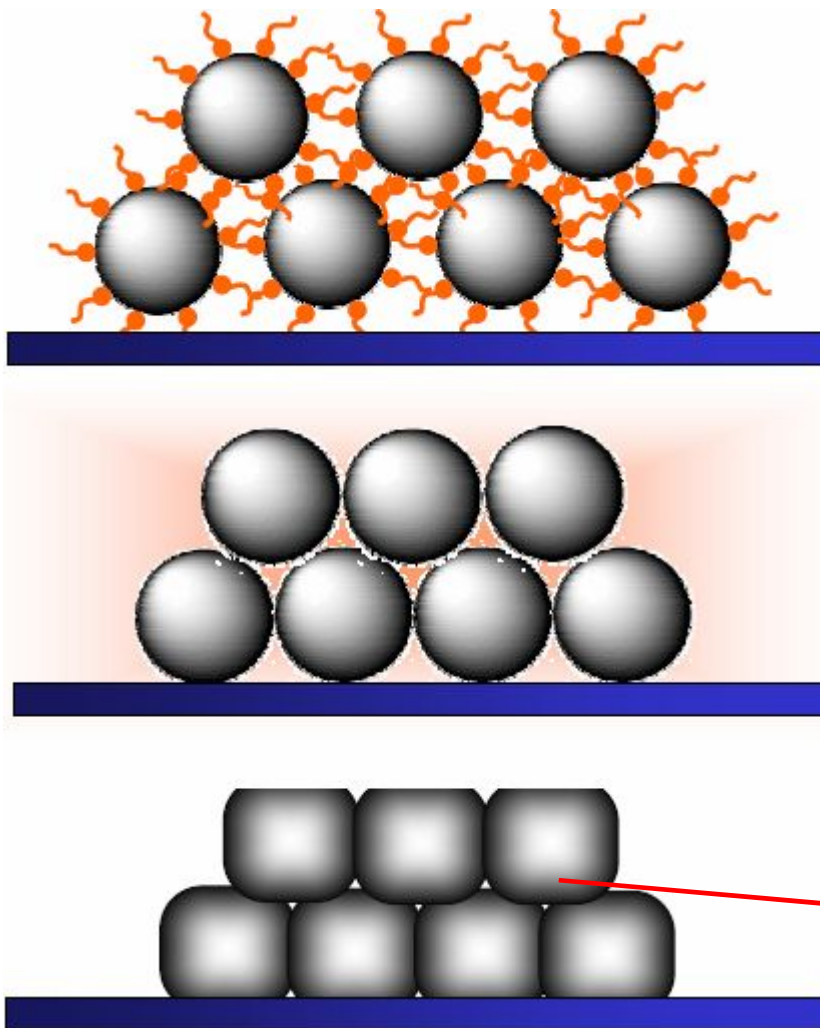
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MAY 11-15, 2011

Phenomena during sintering process

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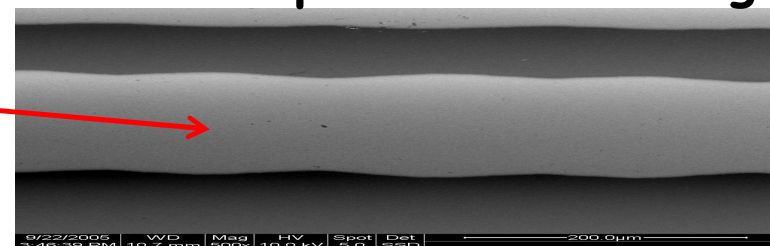
Evaporation of solvent



Removal of protection layers

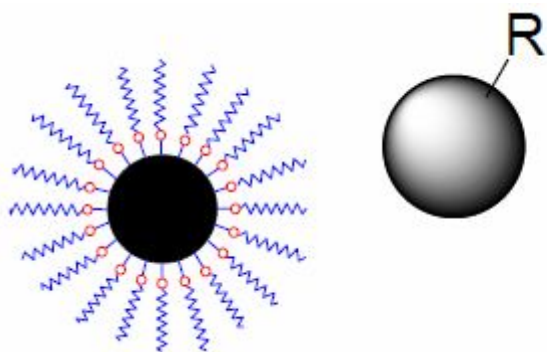


Low - temperature sintering



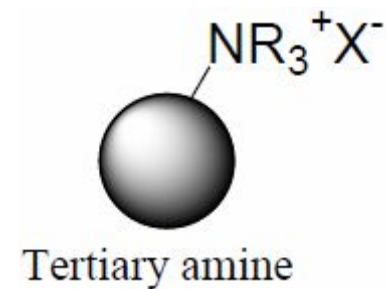
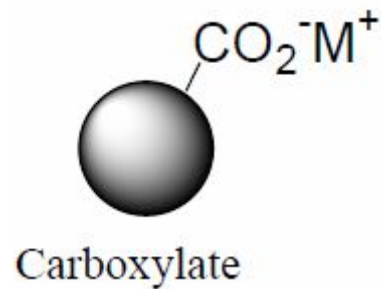
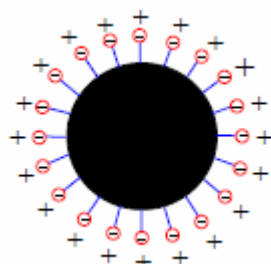
Protective coatings examples

Polymer (steric):



R: acrylic, polystyrene, PEO, PPO, etc

Charged (electrostatic):



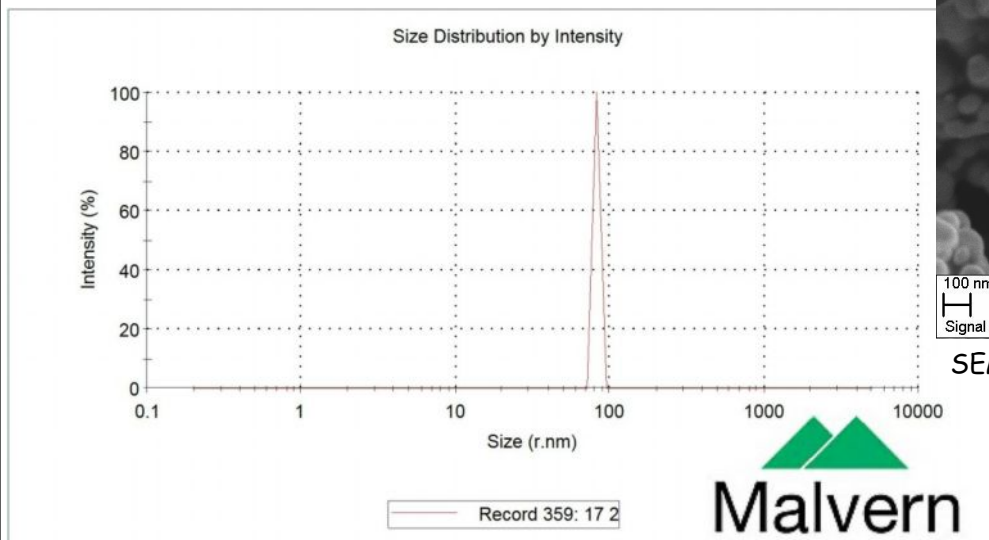
Type of studied nanosilver:

- nanosilver with **carboxylate** coating (Ag1),
- nanosilver with **amine** type coating (Ag2),
- nanosilver with **polymer** coating (Ag3).

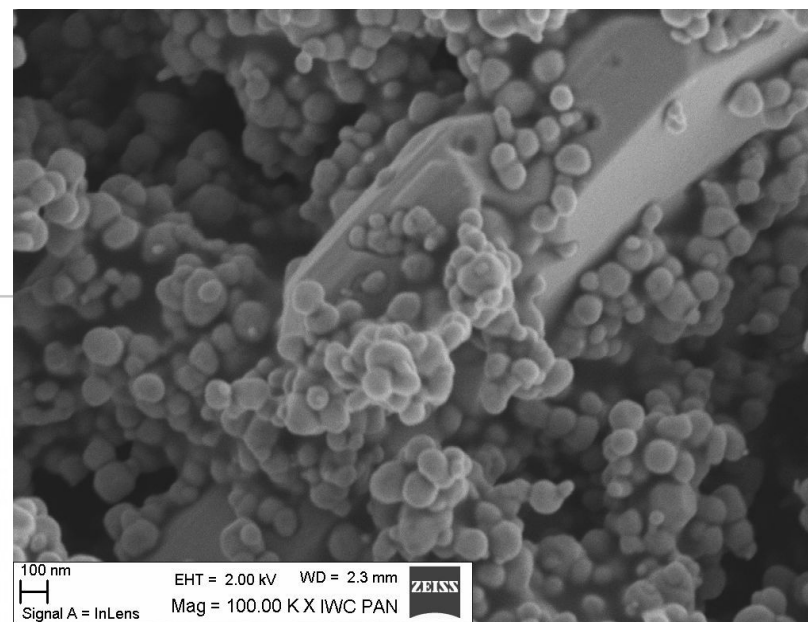
Metalic NanoSilver as a Filler for Ink Preparation

Nanosilver with
carboxylate protective
coating - Ag1

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Measurement of particle size „Malvern“ (Courtesy Polish Academy of Sciences)



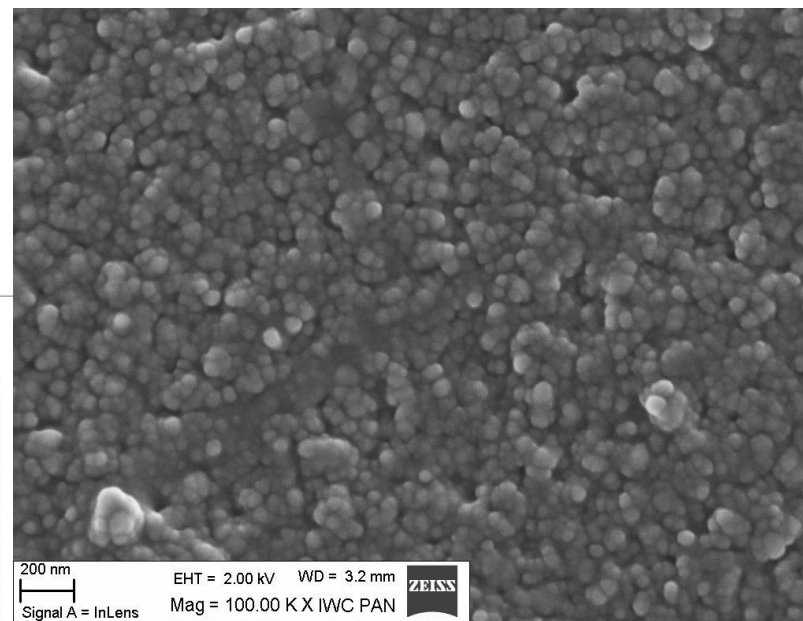
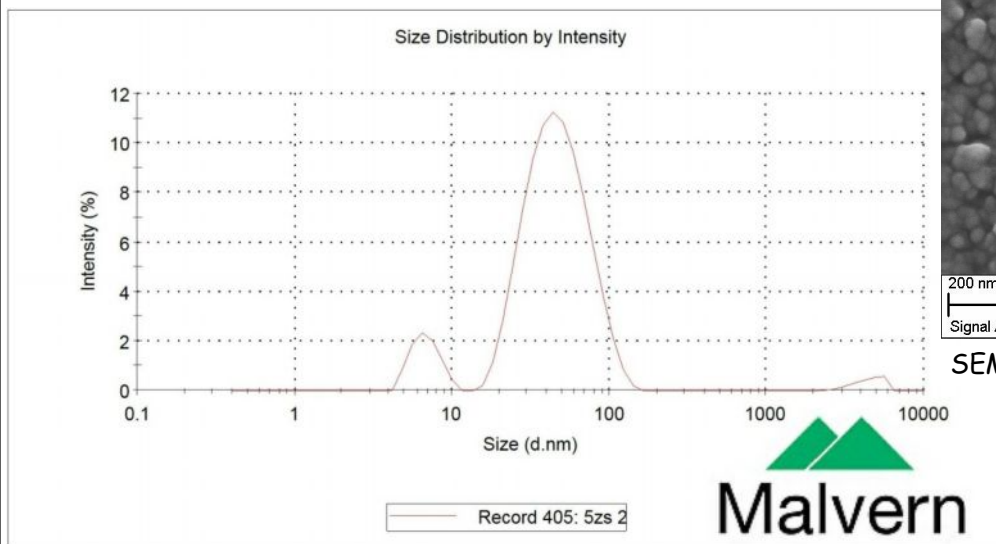
SEM picture nAg particles (Courtesy Polish Academy of Sciences)

Size - 80-100 nm

Metalic NanoSilver as a Filler for Ink Preparation

Nanosilver with
amine protective
 coating - Ag₂

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SEM picture nAg particles (Courtesy Polish Academy of Sciences)

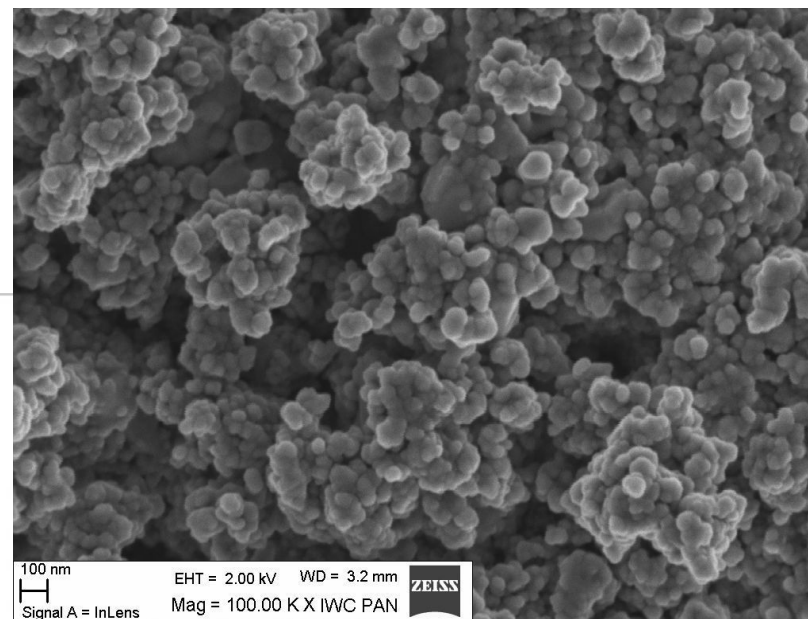
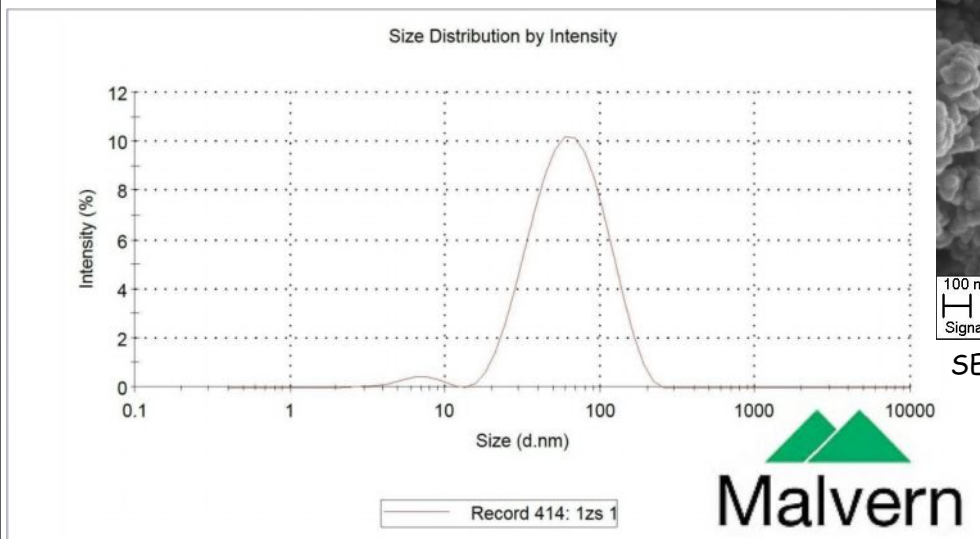
Size - ab. 50 nm

Measurement of particle size „Malvern“ (Courtesy Polish Academy of Sciences)

Metalic NanoSilver as a Filler for Ink Preparation

Nanosilver with
polymer protective
 coating - Ag₃

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SEM picture nAg particles (Courtesy Polish Academy of Sciences)

Size - 50-70 nm

Measurement of particle size „Malvern“ (Courtesy Polish Academy of Sciences)

Thermal studies:

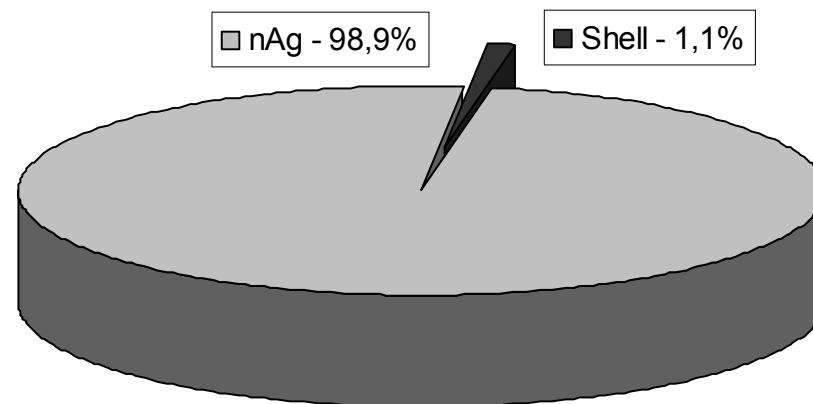
- the purity analysis for Ag1, Ag2 and Ag3 samples,
- the dynamics of removing the protective coating at 150 °C and 230 °C as a function of time for Ag1, Ag2 and Ag3 samples,
- the X-ray photoelectron spectroscopy (XPS) for nAg sample.

Sintering process

Purity analysis of nanosilver with **carboxylate** coating - Ag1:

- content of carboxylate coating - 1,1 %

Protective layer of silver nanoparticles at 500°C

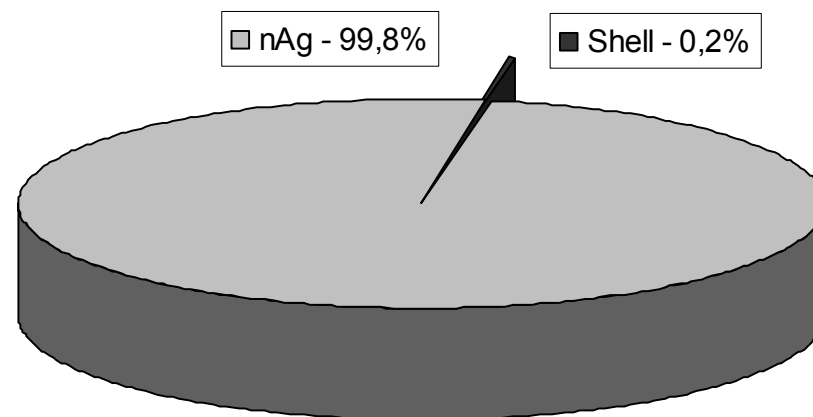


Sintering process

Purity analysis of nanosilver with **amine** coating - Ag₂:

- content of amine coating - 0,2 %

Protective layer of silver nanoparticles at 500°C

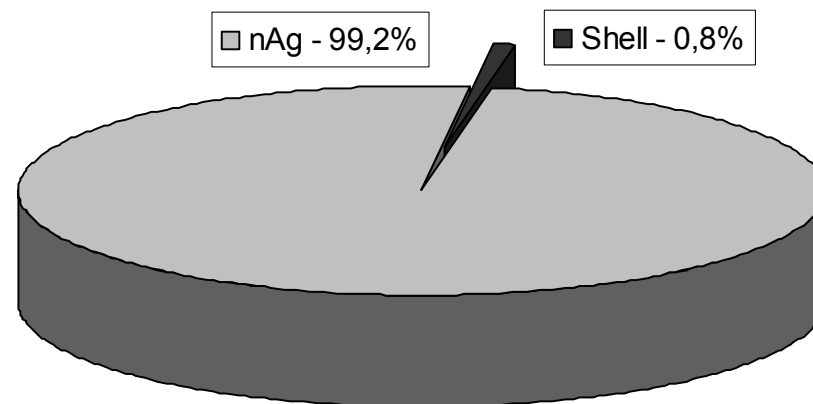


Sintering process

Purity analysis of nanosilver with polymer coating - Ag3:

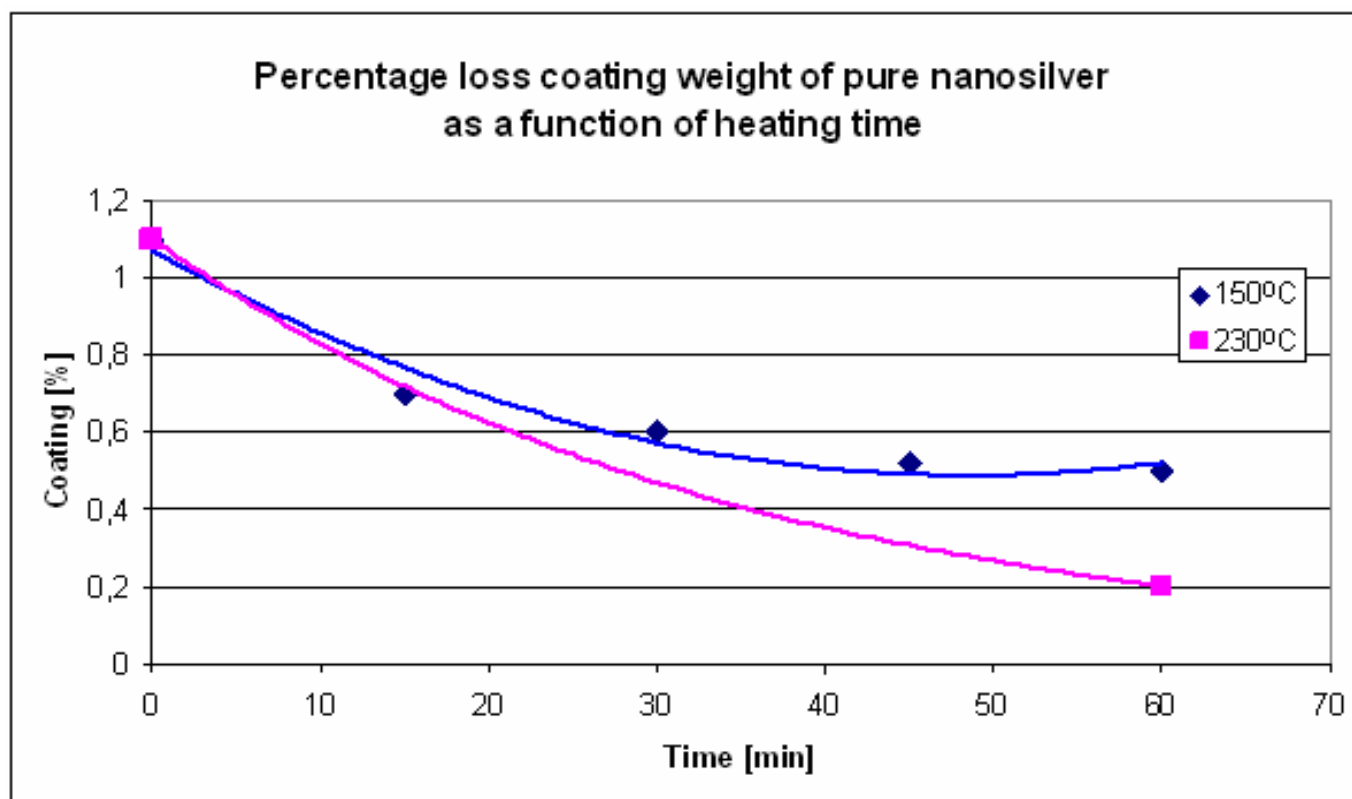
- content of polymer coating

Protective layer of silver nanoparticles at 500°C



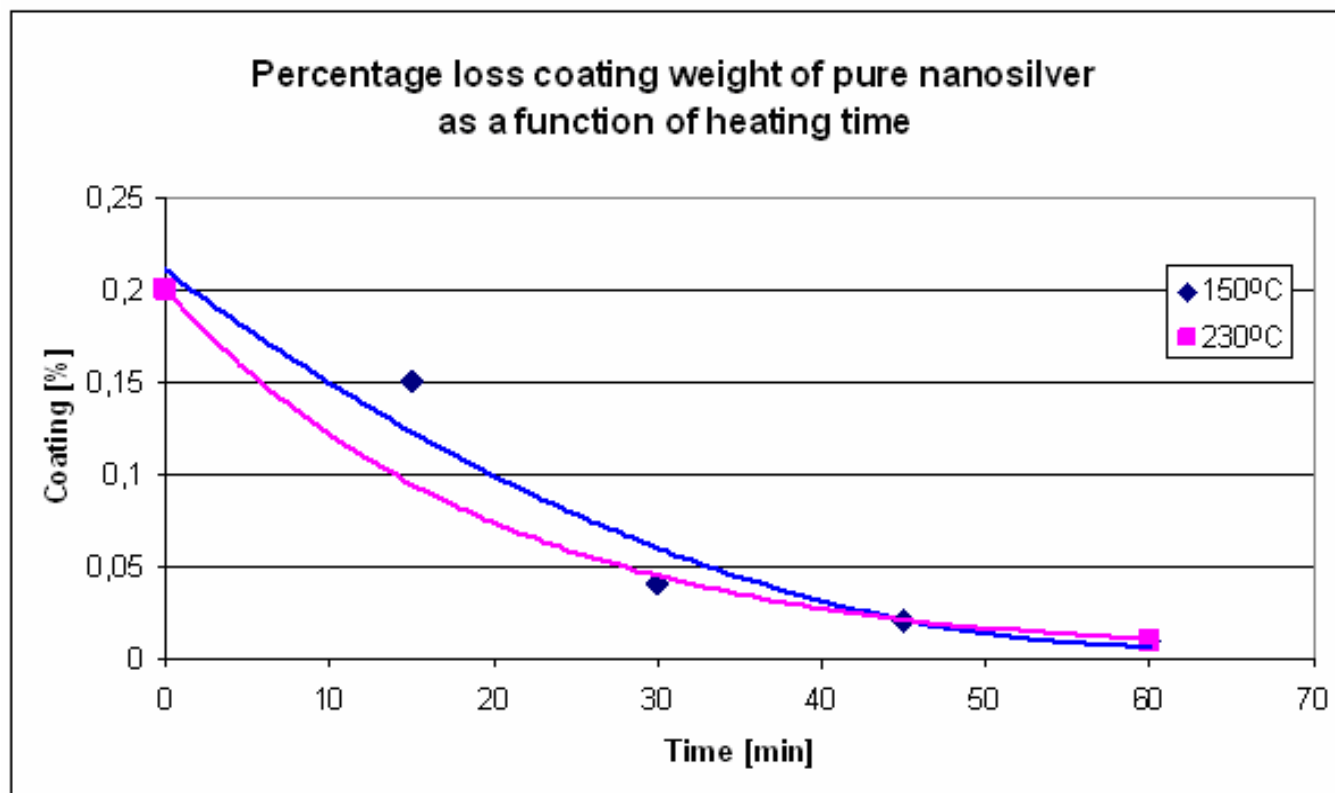
Sintering process

The dynamics of removing **carboxylate** coating at 150 °C and 230 °C as a function of time of Ag1



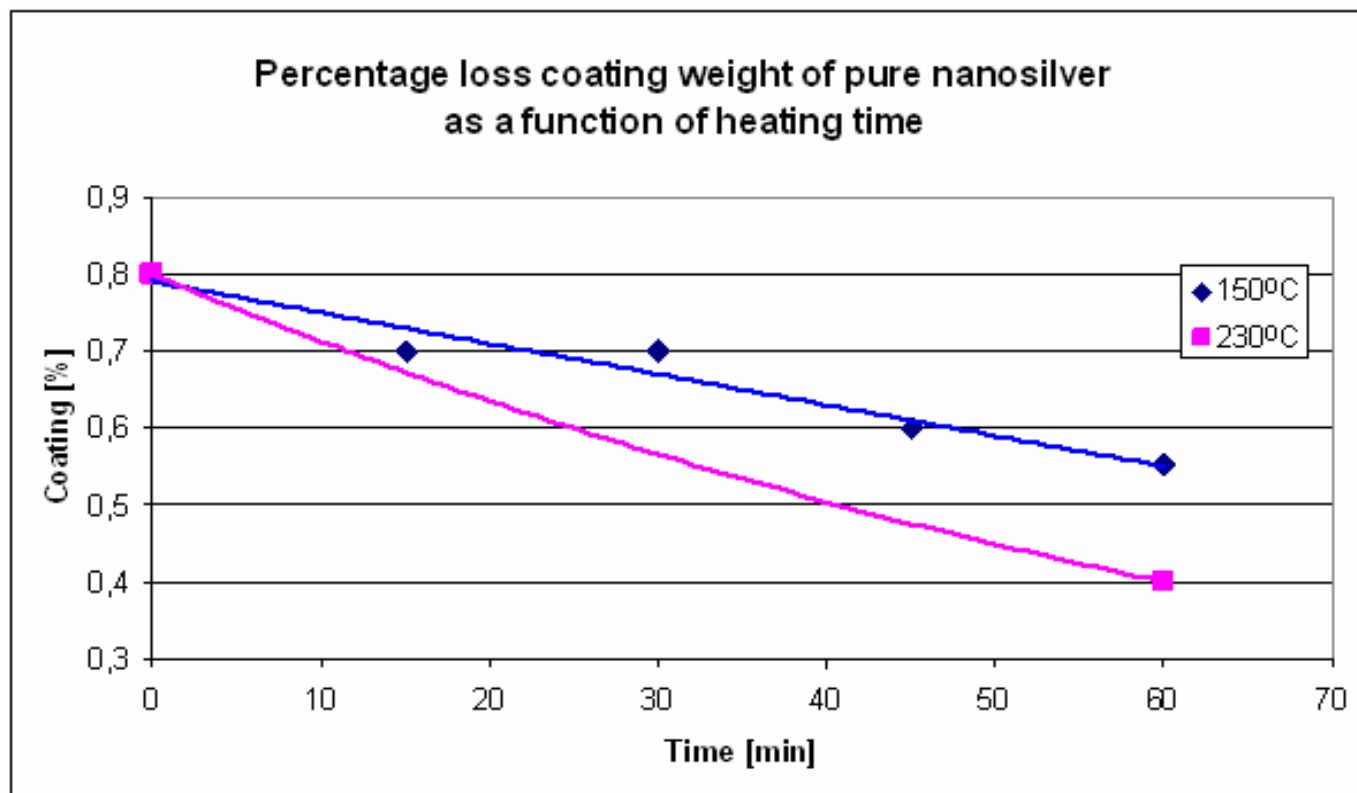
Sintering process

The dynamics of removing **amine** coating at 150 °C and 230 °C as a function of time of Ag₂



Sintering process

The dynamics of removing **polymer** coating at 150 °C and 230 °C as a function of time of Ag3

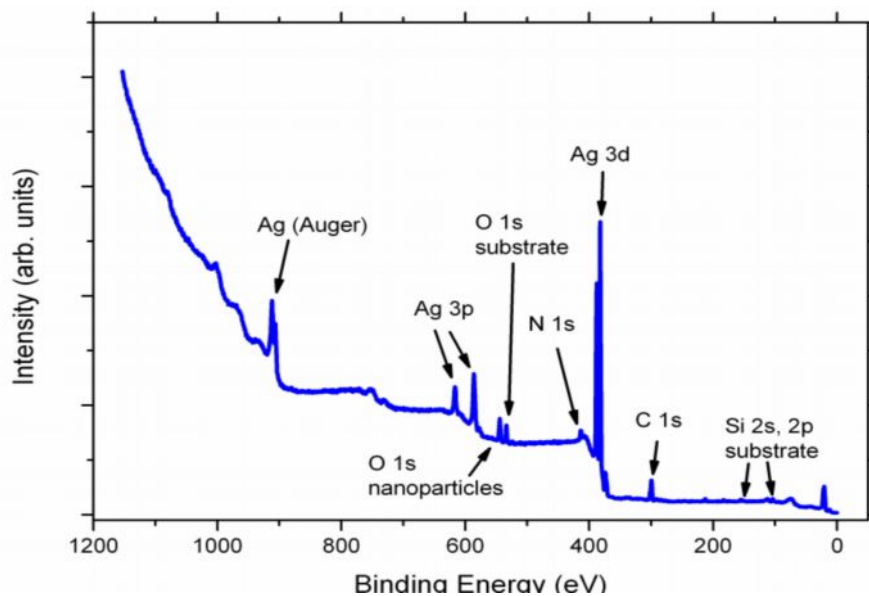


Sintering process

Example of XPS analysis for nAg before and after thermal process in ultra high vacuum up to 290 °C

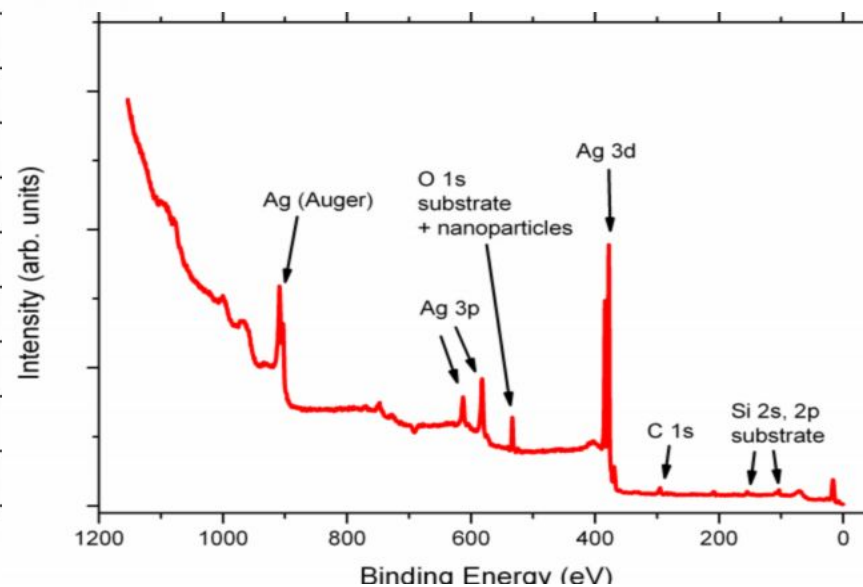
Before

- silver, carbon, oxygen & nitrogen;



After

- silver, small amount of carbon;



XPS analysis (Courtesy University of Łódź)

Summary:

- We have shown three types of protective layers as a representative from very wide group of materials.
- Different size of obtained nAg is connected with reaction conditions for different group of used materials.
- We have made the studies of % content of protective layers on surface each kind of nanosilver.
- The dynamics of removing protective coating at heating temperature as a function of time were carried out.
- The maximum protection material was about 1.1 % for nanosilver with carboxylate coating and in the best case 0.2 % for nanosilver with amine type coating.
- The remained amount of protective layers is necessary for improving adhesion of printed layer to substrates.

Thank you for your attention.

