Mechanical Characterization of Ink-Jet Printed Ag Samples on Different Substrates

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Abstract. In this paper, the main activity was to investigate how different substrates, temperature of sintering and percentage of silver ink containing silver nanoparticles influence on Young's modulus and hardness of printed silver thin samples. Samples were prepared by low cost ink-jet printing technique using Dimatix Material Printer on polyimide flexible substrate and slide glass. Characterization of these samples was carried out by Nano Indenter using a three sided pyramidal (Berkovich) diamond tip. Measurement results show that the thickness of ink-jet printed silver layer varies for different percent of nanoparticles in silver ink. All measurements were done at same depth of indentation to avoid possibility of perforation of printed layer. The higher temperature of sintering and the higher percent of silver nanoparticles give the bigger Young's modulus and hardness of printed silver sample. This research provides very useful information about mechanical characterization of the silver layers on flexible substrates for printed-electronics.

Keywords: ink-jet printing, silver nanoparticle ink, nanoindentation.

1 Introduction

Ink-jet printing is a particularly attractive technique, for a direct write of patterns and the delivery of precise quantities of materials. It is desirable to fabricate onto polymeric or similar temperature sensitive substrates by solution-based printing process. Development of a solution-based process on a flexible substrate would allow roll-to-roll fabrication, which is an extremely inexpensive way to mass-produce circuits since it eliminates conventional photolithography and complex substrate processing including vapor phase deposition and etching. For these reasons, the ink-jet printing as a convenient and rapid processing technique to fabricate conductive lines has attracted great attention in recent years [1]. In the electronic industry, ink-jet printing tecnology has proved to be more flexible, low cost and more enviroment friendly [2] than the current manufacturing processes. This technology has shorted time of production and this result lower cost of production. The ink-jet printing technology has become an interesting aternative to the current manufacturing methods and has been integrated into the various phases of mass electronics production [3].

Nanomaterials have been of much benefit in improving process procedures in printable electronics, especially in connection with sintering process temperature and

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sintering process duration. Silver ink, based on nanoparticles makes it possible to lower the sintering temperature and to shorten sintering time. Evaluation of the mechanical reliability of silver ink-jet printed structures is crucial in reliable electrical design. Some of the analysis of mechanical performance of silver ink-jet printed layer are presented in [4].

For a mechanical characterization of such printed samples nanoindentation has been established as an important tool for characterization on the submicron scale. Such a test is usually conducted using instrumented machines with which indenter load and indenter displacement can be continuously and simultaneously recorded during indenter loading and unloading [5]. The two mechanical properties measured most frequently using load and displacement sensing indentation techniques are the Young’s modulus and the hardness [6]. The measured Young's modulus and hardness would be different, even for the same film, depending on the substrate materials because of the substrate effect on the measured properties [7].

In this paper, we present fabrication process of silver layer on different plastic/organic substrate and slide glass in ink-jet printing technique and their mechanical characterization. Relationship to internet of things and state of art/releated literature are shown in Section 2 and 3, respectively. In the Section 4 is present fabrication and characterization processes. Section 5 presents measurement results of mechanical characterization and the discussion. The reached conclusions of the obtained results follow finally in the Section 6.

2 Relationship to Internet of Nano-things

Over the internet, everything is connected through the online system to perform controlled devices and complex systems, for example power plants, industrial equipment, automotive and cellular industry, and even households. This leads to improvements in the quality of production and the saving of time, and all that is connected with the lower prices of products on commercial market.

The internet, there is a lot more data, all the time, growing at 50 percent a year, or more than doubling every two years, estimates IDC (International Data Corporation), a technology research firm. It’s not just more streams of data, but entirely new ones. For example, there are now countless digital sensors worldwide in industrial equipment, automobiles, electrical meters and shipping crates. They can measure and communicate location, movement, vibration, temperature, humidity, even chemical changes in the air. Link these communicating sensors to computing intelligence and you see the rise of what is called the Internet of Things or the Industrial Internet [8].

Nanotechnology promises new solutions for many applications in the biomedical, industrial and military fields as well as in consumer and industrial goods. The interconnection of nanoscale devices with existing communication networks and ultimately the Internet defines a new networking paradigm as the Internet of Nano-Things [9]. Many application in microelectronics involve combinations of plastic and metal constituents. Due to the different properties of the materials involved (e.g. Young’s modulus and hardness, thermal expansion coefficents, elestic constants yeild strenght) components can be subject to internal stresses during fabrication.

Ink-jet printing technology consist of complex micro- or nano-network of metal structures embedded within a plastic/organic substrate. The low sintering temperature (i.e. below 300°C) can be achieve using inks containing nanoparticles. This makes feasible the use of excelent electronic conductors such us silver or gold with different percentage participation in inks.

During the production of commponents, which is used in connecting things (the internet of things) could be used different materials. The choice of the materials depend on the purpose of the components and the conditions in which it will be used. For this reason, ti is very important to know all the characteristics of the used material. For example, if you want to make a RFID tag to be used in an environment which will significantly affect the material that we used in the preparation of tag, we need to know the materials' mechanical characteristics, like hardness, Young 's modulus, and scratch test. This leads to a better design of components, longer life and higher quality mode.

3 State of the Art / Related Literature

In a last twenty years, nanoindentation was introduced as a method for determining the modulus and hardness of materials by studying nanomechanical response as a function of penetration depth. During the years, this method has been constantly refined. Nanoscale characterization techniques are continuously challenged by the rapid progress in nanostructures and functional materials demanding higher resolutions and advanced measurement techniques for mechanical, chemical, electrical, and thermal characterization. This understanding will be valuable in supporting the impetus to harness multifunctionality of materials to realize nano- and micro-devices [10].

Nanoindentation is widely used to study the displacement of materials under specific applied loads to produce load–displacement curves. This will lead to the aim of presenting its contributions to progress in materials science [11]. One of the most fascinating things about nanoindentation is the number of different applications for which the technique is useful. One of these applications is: the utility of nanoindentation in the geological world, It relates some of the smallest experiments on mechanical properties that can be performed to some of the largest we know of: earthquakes. Other very important areas that have become popular recently are the accurate characterization of viscoelastic materials and polymers, polymers characterization as it relates to nano-imprinting and nano-forming, nanoindentation in the materials science of biomedicine [12].

4 Fabrication and Mechanical Characterization of Ink-Jet Printed Samples

In recent years, there has been tremendous interest in the development of printed electronics components as a means of achieving ultra-low-cost electronic circuits [13]. In the electronic industry, fabrication of conductive tracks is inevitable. Conventional photolithographic and electroless deposition techniques are widely adopted in the printing circuit board (PCB) for manufacturing its conductive circuits. However, this method is not only time consuming but also very complicated and expensive, because many processing steps are required to construct a layer of the circuit. Moreover, the electroplating and etching processes also produce large quantities of chemical waste. Therefore, there is an industrial need for direct digital printing to simplify the processes and to reduce manufacturing costs [1]. For good results it is very important to know all characteristic of materials which are used for ink-jet printing. Because of that we have tested mechanical characteristic of printed silver layers.

4.1 Fabrication of Ink-Jet Printed Sample

The samples for mechanical characterization were prepared by ink-jet printing technique by Dimatix Deposition Material Printer DMP-3000 [14], on polyimide flexible substrates with different thickness and slide glass, shown in Fig. 1.

Fig. 1. Dimatix Deposition Material Printer DMP-3000

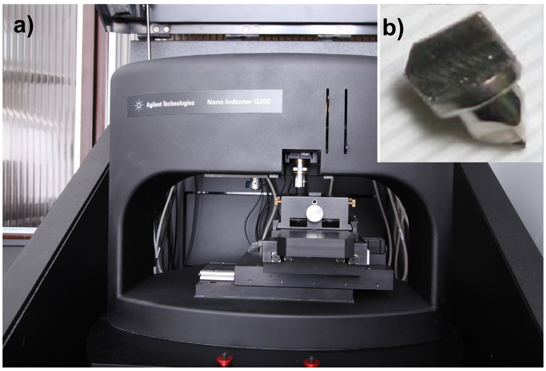

Fig. 2. a) Measurement system - Agilent Nano Indenter G200; b) Berkovich diamond tip

The thicknesses of polyimide film were 50 μm and 75 μm (manufacturer GTS Flexible Materials Ltd [15]) and thickness of slide glass was 1000 μm. As inks have used commercially available SunChemical silver nanoparticle inks with 20 wt% and

40 wt% of silver nanoparticles [16]. During the printing procedure has been used 16-nozzle piezzo cartridge with nozzle diameter of 25.4 µm. The distance between nozzles is 200 µm and drop spacing was 50 µm. After depositing silver nanoparticle ink on polyimide film and slide glass samples are sintered on air atmosphere at three different temperatures (200 ˚C, 225 ˚C and 250 ˚C) for 30 min. Printed samples, after sintering, are presented in Fig. 2.

4.2 Mechanical Characterization of Ink-Jet Printed Samples

After sintering process, characterization of this samples are carried out by measurement system with Agilent Nano Indenter G200 [17] using a three sided pyramidal (Berkovich) diamond tip. Measurement system and Bercovich tip are shown in Fig. 3. Berkovich tip is ideal for most testing purposes. It induces plasticity at very small loads which produces a meaningful measure of hardness. The Berkovich indenter tip has a large included angle which minimizes the influence of friction. Basic principle Nano Indenter G200 testing is employing a high-resolution actuator to force an indenter into a test surface and a high-resolution sensor to continuously measure the resulting penetration. As the indenter is driven into the material, both elastic and plastic deformation cause the formation of a hardness impression conforming to the shape of the indenter to some contact depth. After the indenter is withdrawn, only the elastic portion of the displacement is recovered; this recovery enables one to determine the elastic properties of a material [17]. The first step of measurement is preparing a sample by mounting it on a sample disk. For mounting samples double stick tape is used. Mounted samples are presented in Fig. 4.

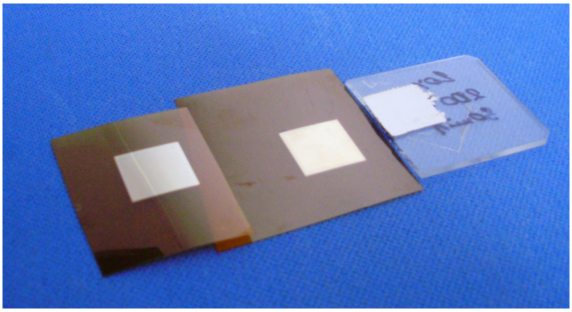

Fig. 3. Printed silver layers on different substrate after sintering

Middle position in sample tray is reserved for reference material (Corning 7980, fused silica or Pyrex 7740 [17]). One of these materials are used to indirectly verify the instrument accuracy according to the standards.

After placing sample of silver layer on plastic substrate on tray into the indenter, height of measuring sample must be adjusted to the height of the reference material which is fixed. Adjusting the height of the sample can be done with small thumbwheel for each sample holder; rolling of thumbwheel sets the height of sample. Finally, when samples are in line with reference material, it could be set measurement parameters. All measurements were done at same depth of indentation, around 200 nm to avoid possibility of perforation of printed layer. The Poisson's ratio for silver was set as 0.37. The ten indentations have been made for each sample.

Fig. 4. Samples mounted on holder and prepared for measurement

5 Measurement Results and Discussion

Fig. 5. presents load-displacement curves obtained by nanoindentation for ink-jet printed silver layers on 50 μm polyimide film sintered at 200 ˚C, 225 ˚C and 250 ˚C for 30 min. The observed curves are consistent with intuitive expectations; the largest load is required to silver layer sintered at highest temperature, at 250°C.

Table 1. summarize mean values of the 10 measurement data of Young's modulus, hardness, displacement into surface and load applied to test surface. As it can be seen from the Fig. 5. and Table 1, as it was expected higher temperature of sintering leads to higher hardness and Young's modulus of material.

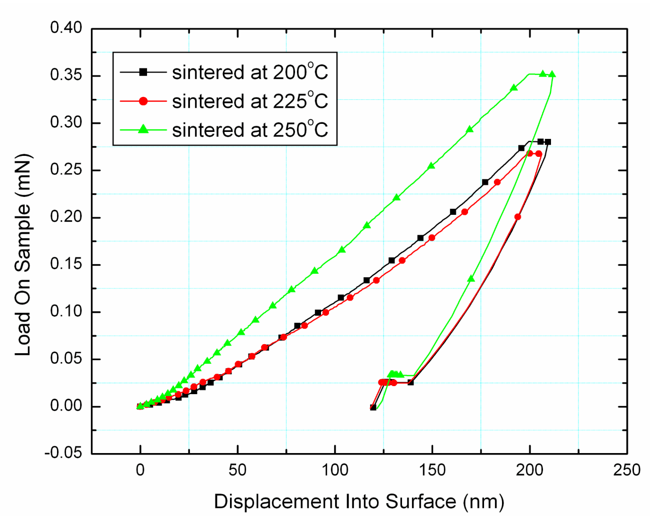

Fig. 5. Typical load-displacemet curves of ink-jet printed silver layers on 50 μm polyimide film at different temperature of sintering

Table 1. Mechanical properties of ink-jet printed silver layers determined by the nanoindentation

	Young's modulus at Max load (GPa)	Hardness at Max Load (GPa)	Displacement at Max Load (nm)	Load at Max Load (mN)
20 %wt on 50 µm polyimide at 200 °C	8.237	1.250	206.773	0.250
20 %wt on 50 µm polyimide at 225 °C	8.658	1.445	204.96	0.240
20 %wt on 50 µm polyimide at 250 °C	10.328	1.737	206.902	0.305
20 %wt on 75 µm polyimide at 200 °C	9.056	1.456	204.69	0.251
40 %wt on 50 µm polyimide at 200 °C	11.968	1.387	207.542	0.383
20 %wt on 1000 µm slide glass at 200 °C	95.506	1.627	219.182	1.697

The load-displacement curves obtained for silver layers printed with 20 wt% and 40 wt% silver nanoparticle ink on 50 µm polyimide substrate and sintered at 200 ºC for 30 min are presented in Fig. 6. It can be seen, silver layer printed with 40 wt% silver has larger load during approaching depth of 200 nm of indentation. According to that and to data from Table 1. it also visible that silver layer printed with 40 wt% has a higher value of Young's modulus and hardness that the layer printed with 20 wt% of silver nanoparticles.

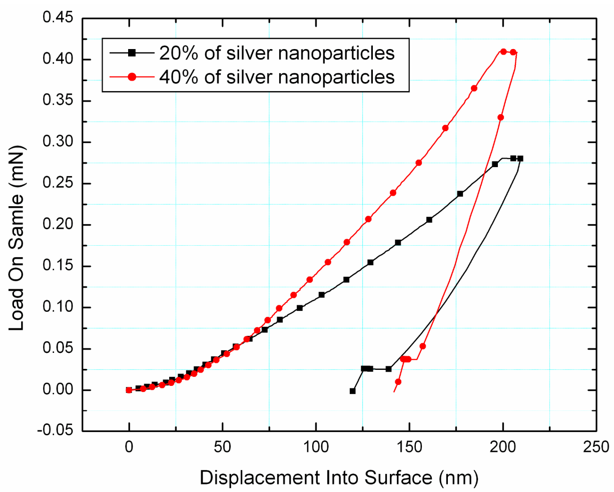

Fig. 6. Typical load-displacemet curves of ink-jet printed silver layers printed on 50 µm polyimide film with different percentage of silver nanoparticles in ink sintered at 200 ˚C

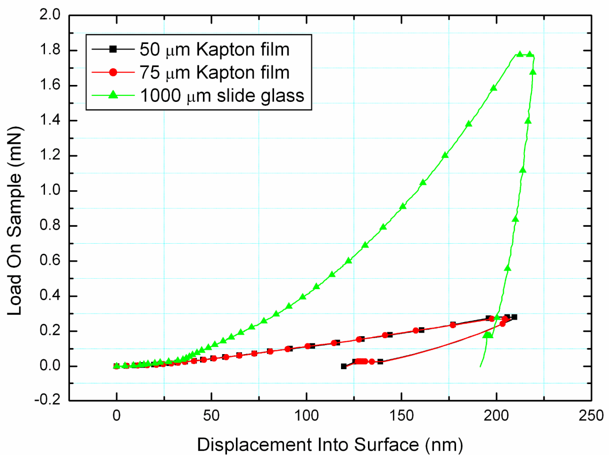

Fig. 7. Typical load-displacemet curves of ink-jet printed silver layers on different substrate sintered at 200 ˚C

The load-displacement curves for silver layer printed on polyimide film with thicknesses of 50 μm and 75 μm and on slide glass with thickness of 1000 μm are shown in Fig. 7. From Fig. 7. and data from Table 1, the visible is different type of substrate leads to different value of hardness. The silver layer printed on polyimide film with thickness of 50 μm has lowest and silver layer printed on slide glass has the highest value of Young's modulus and hardness. It is also visible that difference in load applied on layer printed on polyimide substrates is almost negligible in comparison with load applied on silver layer printed on slide glass.

6 Conclusion

In the last two decade, much attention has been paid to developing techniques for probing the mechanical properties of materials on the submicron scale. Developing instruments, which can continuously measure load and displacement during the indentation, could see the progress.

Mechanical performance of ink-jet silver layers was considered, suitable for electronic component production. Sample preparation and mechanical test setup were briefly described. Nanoindentation tests were performed to see how different parameters, such as thickness of substrate (50 and 75 μm), the type of substrate (polyimide and glass) and %wt of silver nanoparticles containing in silver ink, influence on Young's modulus and hardness of printed silver layers. According to average value of indentations silver layers, it was found that the higher temperature of sintering and the higher %wt of silver nanoparticles give the bigger Young's modulus and hardness of printed silver sample. It was also found that thickness and type of substrate have low influence on Young's modulus and hardness of ink-jet printed silver layer. This research provides very useful information about mechanical characterization of the silver layers on flexible substrates for different industrial applications on plastic/organic electronics.

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