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이티븀 광섬유 펄스 레이저 유도
화학액상 증착법을 통한
유리에 내장된 구리 도선 제조

Fabrication of Embedded Copper Wire on Glass
by Laser-Induced Chemical Liquid Phase Deposition
with Ytterbium Fiber Laser

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서재민

Abstract

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Glass possesses a lot of useful and attractive properties such as transparency, stability, and strength. For these properties, especially transmittance, glass is one of widely used materials in many industrial fields, such as mechanics, biology, medical science and engineering. However, glass is well-known as non-conductive and non-flexible material. Therefore, many attempts and researches have been done for making circuits on the glass. Since lots of deposition techniques have been widely studied such as electroless laser deposition, chemical deposition with hot plating and electronical method, those methods rarely contributed to increase usability of deposited glass due to durability, weak adhesion problems and so forth.

In this study, copper deposition has been processed inside of channel by laser to overcome existing disadvantages. LIBWE, which enables to machine glass easily regardless of glass' physical properties, has been conducted to make channels before deposition process. After the channels has been machined, the substrate is transferred to LCLD solution and laser beam initiate the deposition along the channels. Depth of 50 μm and various width (50 ~ 150 μm) of channels are filled with copper. With this process, disadvantages from previous studies such as lack of controllability, usability and durability are solved by generation of embedded copper wires. Furthermore, resistance can be controlled and further applications such as stacking and sealing are possible.

Keywords: Glass, Copper circuit, Laser induced phase chemical liquid phase deposition (LCLD), Laser-induced backside wet etching (LIBWE), Embedded copper circuits

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Chapter 1

Introduction

Interest of industrial use of glass has been widely spread due to its irrefutable characteristics. Micro fluidic devices, sensors and smart glasses are possible applications if glass could possess conductive by any method [1][2]. To give conductivity to glass, other conductive materials should be combined or attached to glass in various ways. However, glass' transparency should not be harmed while making conductive unit on the glass owe to transmittance is one of the most prominent properties of glass. Furthermore, applied conductive materials, such as copper and silver, are needed to be sufficient amount and should function properly. There are several previously researched methods to deposit conductive materials on the glass such as Indium tin oxide film (ITO) [3][4], femtosecond laser deposition [5][6], Electroless hot plating [7], and Laser induced chemical liquid phase deposition (LCLD) [8]. However, those techniques hardly improved usability of deposited glass due to some limitations such as durability.

To secure durability of deposited copper circuit, increase adhesion force between glass and deposited copper could be a solution. By fabricating micro structures on glass and embedding copper inside would offer more contacting surfaces those which can increase adhesion between glass and copper. Even though glass seems hard to micro structuring, many glass machining methods have been researched such as electro chemical discharge machining (ECDM) [9], ductile regime machining [10][11], CO₂ laser machining [12], and laser-induced backside wet etching (LIBWE) [13].

In this study, LIBWE and LCLD methods are chosen for generating copper wires inside of channels. Fabricating precisely controlled channels by LIBWE method and then filling each channel with copper particles by LCLD method. By combination of LIBWE and LCLD with 1064 nm Ytterbium laser, generated copper wires are affordable and robust enough to apply to many industrial applications in durability and many application perspectives. Since generated copper wires from the previous researches are exposed and protrusion over the glass surface, inserting copper wire inside of channel possibly more durable from outer circumstances. Durability differences between copper wires deposited on the surface of glass and inside of channel are comparably measured. Lastly, substantiate the possibilities of function as copper circuit by conducting conductivity test of produced copper circuits.

Chapter 2

Theoretical background

2.1 Laser-induced backside wet etching (LIBWE)

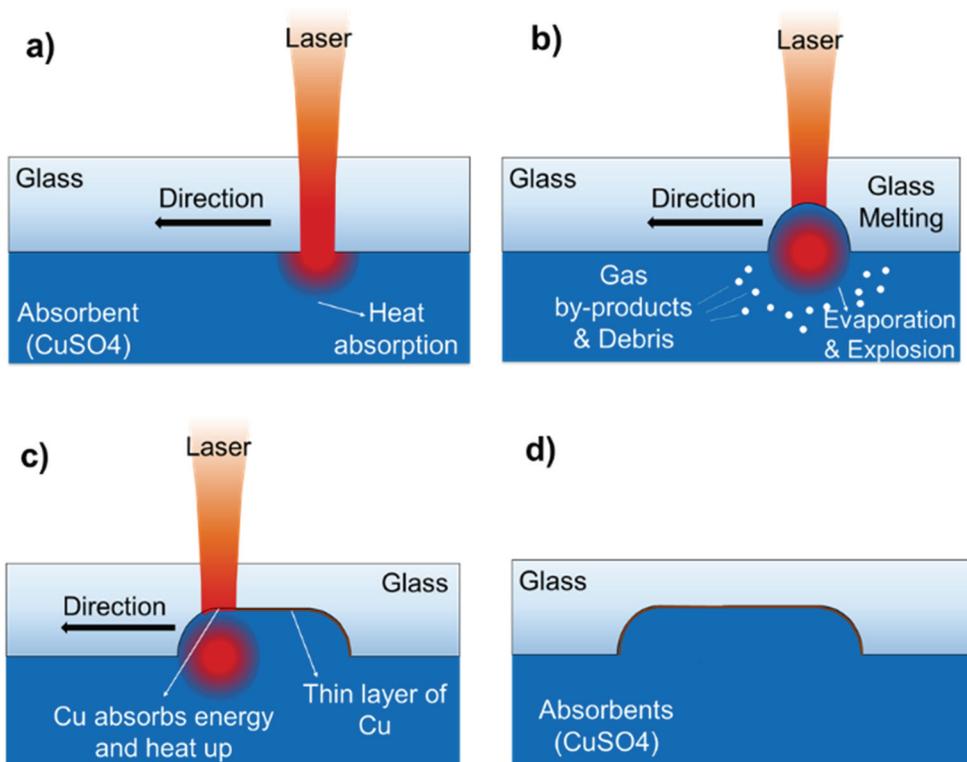


Fig. 1. Machining process of LIBWE with copper sulfate absorbent.

Laser-induced backside wet etching (LIBWE) is a technique that machining backside of transparent glass by laser. Absorbent is needed to be heated by laser beam and explode. To effectively heat up absorbent, the absorbent's absorbance is needed to match with the wavelength of laser that experimenters use. At the interface of the absorbent and the glass, the absorbent absorbs laser beam energy. The absorbent is heated and explode by laser beam (Fig. 1a) [14]. By explosion of absorbent, glass is melted and removed from the surface of glass with gas by-products and debris (Fig. 1b). Rough surface is generated by removal of glass particles and thin layer of copper metal is deposited (Fig. 1c). Since laser energy constantly heat up the absorbent and deposited copper, glass is sufficiently fabricated by repeating processes of above (Fig. 1d). LIBWE successfully eliminates tapered shape of fabricated channels [15] and recast layers are not formed.

Furthermore, LIBWE has some more advantages as becoming a precedent process of copper deposition process. First, LIBWE uses liquid phase of absorbents which suitable for 1064 nm Ytterbium laser [13]. Since copper deposition process is possible with the same laser device, whole processes of fabrication of channels and deposition can be simplified. Second, rough surface and metal deposited thin layer are generated by LIBWE process. Rough surface resulting from LIBWE offer anchor effect which offer better

adhesion for deposited copper. Additionally, deposited thin layer of metal function as initial seed layer for copper deposition [7].

2.2 Laser induced chemical liquid phase deposition (LCLD)

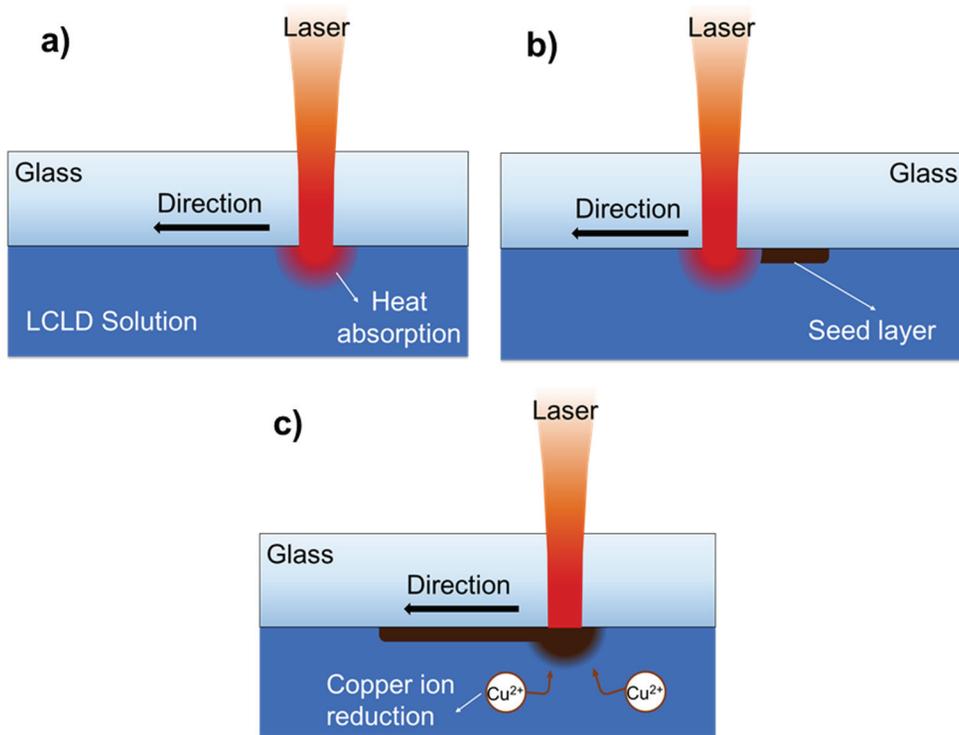
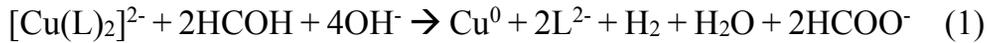


Fig. 2. Machining process of LCLD.



The chemical reaction of electroless copper deposition can be shown by formula (1). The solution is composed of Cu ion, ligand, reducing agent, and NaOH. Cu ions are supplied by copper compound such as CuCl₂ or CuSO₄. When dissolved copper ion in the water meets reducing agent, copper reduction occurs uncontrollably. So, ligands need to be added to the solution for stabilizes the copper ion. Furthermore, copper reduction could be occurred under high pH alkalinity surrounding. NaOH is well-known compound as pH controller. pH 11~12 is ideal for the experiment. [8]

To activate copper reduction to positive reaction direction, energy is needed to overcome activate energy. By experiments, laser beam is a sufficient energy source to activate this chemical reaction. Brief mechanism of the LCLD process is introduced Fig. 2. At the interface of the solution and the glass, the solution absorbs laser beam energy (Fig. 2a). Because of energy of the laser beam, copper starts to reduce and attaches to the surface of the glass as seed layer (Fig. 2b). Since laser continuously irradiated from the source, reduced copper from the solution grows from the seed layer (Fig. 2c).

Chapter 3

Experimental setup

3.1 Experiment and measurement device

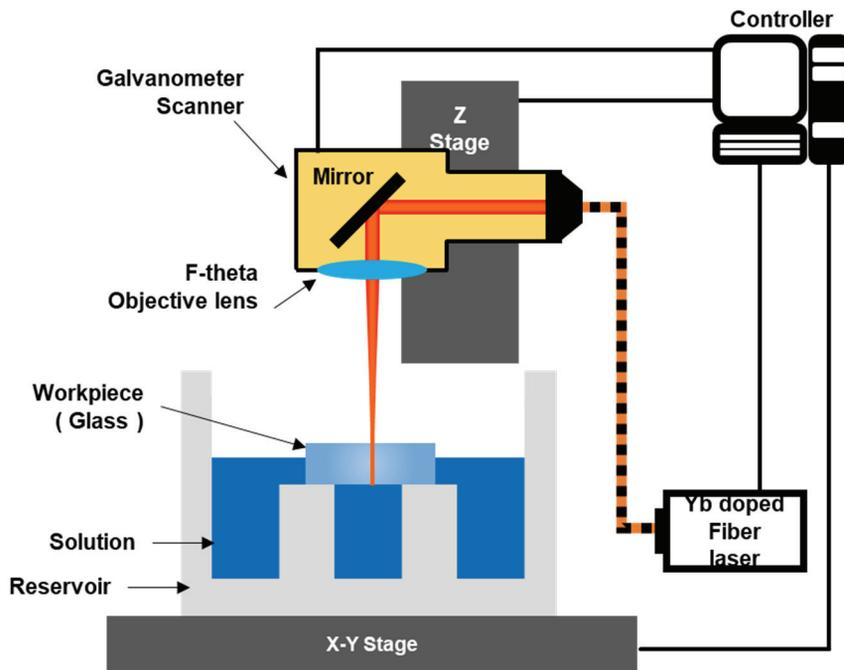


Fig. 3. Schematic diagram of LIBWE and LCLD experiments.

A schematic diagram of the LIBWE and LCLD system is shown in Fig. 3. 1064 nm wavelength of Ytterbium pulsed fiber laser (IPG, YLP-C-1-100-20-20, Germany) is used for the both LIBWE and LCLD processes. This laser device has 40 μm of spot size and 100 ns of pulse duration. The laser beam power can be varied up to 20 W by the source, and frequency can be adjusted 20 to 80 kHz.

Reservoir's and materials' positions can be controlled by X- and Y-axes stage due to reservoir is fixed to it. Furthermore, by moving Z-axis, laser focus can be adjusted, since a galvanometer (SCANcube® 10, Scanlab) is attached to the Z-axis stage. Lastly, the laser scan path is entered by controlling system and the galvanometer scanner actually make the path over the materials.

Polycarbonate (PC) is used for the reservoir due to its stability. PC does not react with neither LIBWE nor LCLD solutions that are needed for the experiments. The size of $15 \times 15 \times 1.9 \text{ mm}^3$ soda-lime glass (JMC glass, Korea) is used as a substrate. The glass is placed on a support and fixed to prevent moving.

Deposited copper wires on the normal surface are captured with the scanning electron microscope (SEM) (JSM-6360, JEOL, Japan) to observe those shape. Due to difficulties of measuring volume of deposited copper,

deposited height and width are addressed. Precise width and depth of channels are observed with the 3D surface profiler (μ Surf, NanoFocus, Germany). For the embedded copper wires, the cross-sections of the channels are prepared by cutting. For the elemental analysis of copper, the energy-dispersive X-ray spectroscopy (EDS) analysis was implemented. To test adhesion of copper, ultrasonic transducer (DH.WUC.A02H, Daihan-Sci, Korea) is used under condition of 50 W, and 28 kHz for 5 minutes. Multimeter is applied at both ends of wire for measuring resistance.

3.2 Experimental procedure

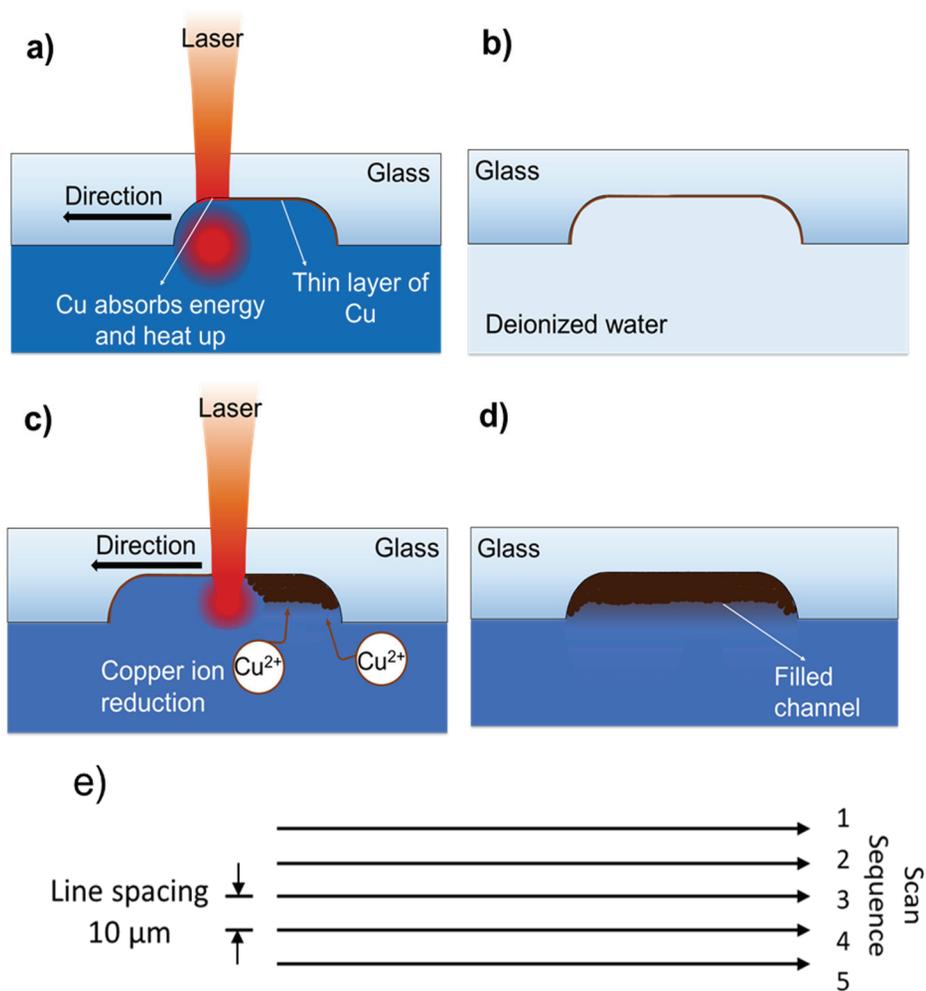


Fig. 4. (a) to (d) Experiment procedure and (e) definition of scan sequence.

Table 1. LIBWE Laser parameters

Variable	Value
Laser power [W]	15
Scan speed [mm/s]	150
Repetition rate of pulsed laser [kHz]	50
Scan length for milling process [mm]	2
Scan sequence	4, 8, 12
Number of laser scan	200, 300, 400, 500

Table 2. LCLD Laser parameters

Variable	Value
Laser power [W]	1, 2, 3
Scan speed [mm/s]	0.015
Repetition rate of pulsed laser [kHz]	80
Scan length for milling process [mm]	2
Number of laser scan	1, 2, 3

Table 3. LCLD Solution condition

Sort	Value
Copper (II) chloride concentration [g/L]	7
Rochelle salt [g/L]	30
Formaldehyde [g/L]	60
pH	12

After fixing the glass on the reservoir, pour LIBWE absorbent liquid into the reservoir until the absorbent contacts the glass' surface. 0.7 M Copper (II) sulfate pentahydrate is used as the absorbent due to having adequate absorbance at 1064 nm wavelength. Channel is generated along the laser path which shown as Fig. 4a. Channel's target depth is controlled by number of laser scan and channel's target width is controlled by laser scan sequence with 10 μm spacing as Fig. 4e.

Laser scan sequence, number of laser scan are changed under LIBWE process while laser power, scan speed, and frequency are fixed by a considerable amount of experiments for optimal results. For instance, laser power is fixed as 15 W owe to not only not sufficient depth is generated under lower power but also cracks are generated under higher power conditions.

After LIBWE process is over, empty the reservoir by removing the LIBWE absorbent. Pour deionized water to clean remaining LIBWE solution from the reservoir (Fig. 4b). After remove deionized water from reservoir, LCLD solution is filled until the solution contacts the surface of the glass. Use the same laser path which has been used during LIBWE process but with different laser parameters (Fig. 4c).

LCLD's laser parameters are different from LIBWE's and addressed Table 2. Laser power, number of laser scan are changed under LCLD process while scan speed, and frequency are fixed. Laser scan speed is fixed as 0.015 mm/s owe to not only not sufficient copper is deposited under faster scan speed but also too reckless reduction is caused under lower scan speed conditions.

For LCLD process, more complex solution is used than LIBWE's absorbent. The solution composition is addressed at Table 3. More specifically, for LCLD process, CuCl_2 is used as copper salt. According to Manshina et al, generated copper wires' resistance, topology and shape are differring based on what kind of copper compound is dissolved in the solution. Copper(II) chloride shows better topology of deposited results [8]. Furthermore, 0.05 M of Cu ion's concentration is appropriate while reckless reduction of copper could be occurred under higher concentration than 0.05 M [16]. Since potassium sodium tartrate tetrahydrate ($\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$, Rochelle salt) and Ethylenediaminetetraacetic acid (EDTA) are well-known ligands, Rochelle salt is chosen for further experiments. According to Kochemirovsky, Vladimir Alekseevich, et al and Kochemirovsky, V. A. Menchikov, L. G. et al, ligands are function as stabilizer of Cu ion that prevents uncontrolled reduction from the bulk of the solution [16], [17].

Formaldehyde is used among many other reducing agents. Lastly, pH is controlled by concentration of NaOH. pH 12 is maintained during whole process of LCLD experiments.

Due to channel's floor roughness and adhered metal ions, reduced copper attaches inside of channel during LCLD process (Fig. 4d).

Chapter 4

Results and discussion

4.1 Surface deposition

4.1.1 Shape & Controllability

Fig. 5 shows SEM images of deposited copper wires on normal surface of glass under 2 W of laser power. A few μm of copper wire is generated when the laser beam scanned once (Fig. 5a, 5b). Copper suddenly grows when laser scan applied twice and saturated or grow more at third scan (Fig. 5c, 5d). Since the number of laser scan has increased, the shape of deposited copper has deteriorated, especially when the number of laser scan is 3 (Fig. 5e, 5f).

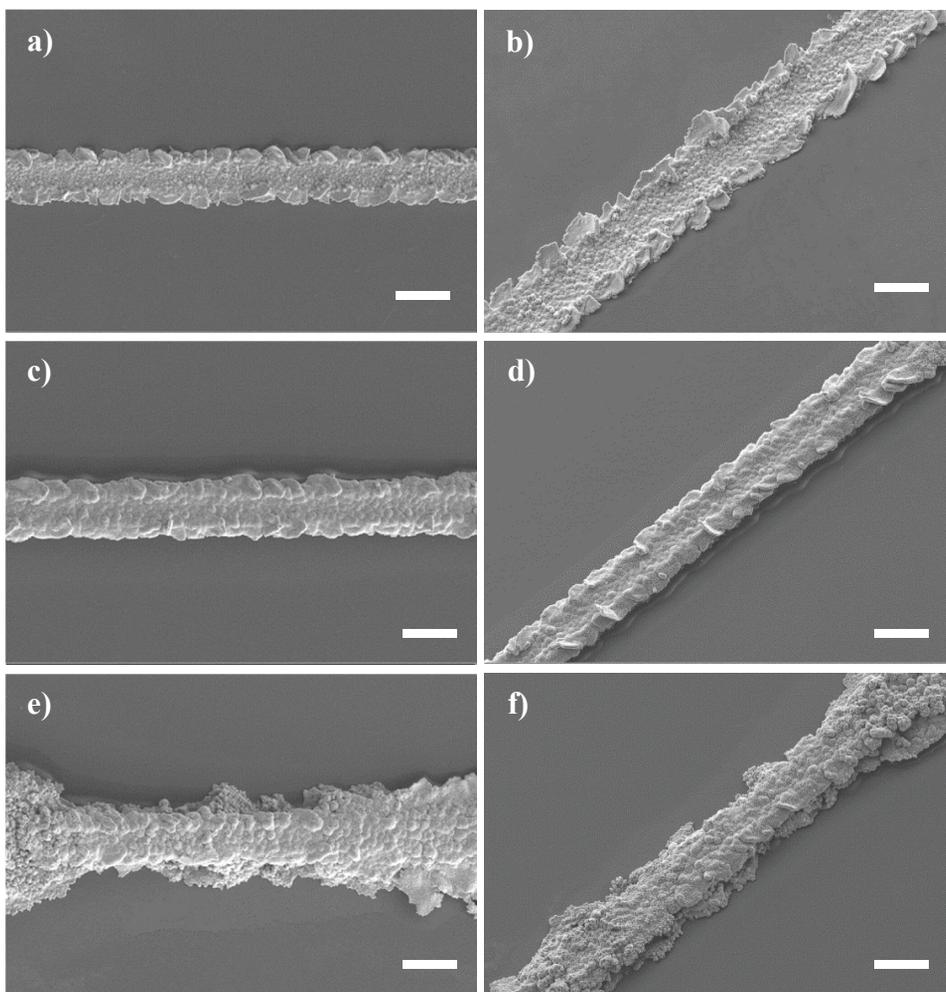
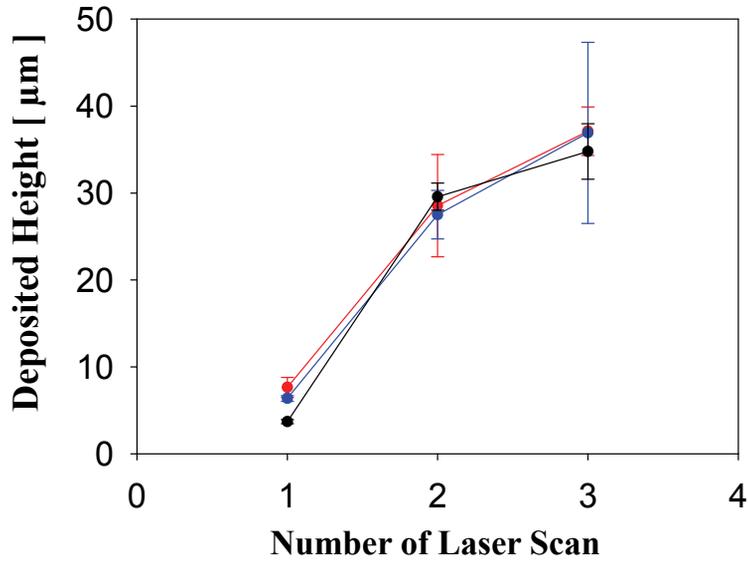


Fig. 5. SEM images of deposited copper on the surface of glass. The scale bars equal to 50 μm . Shape and dimension changes under 2 W of laser power and scan number of (a)-(b) 1, (c)-(d) 2, and (e)-(f) 3.

a)



b)

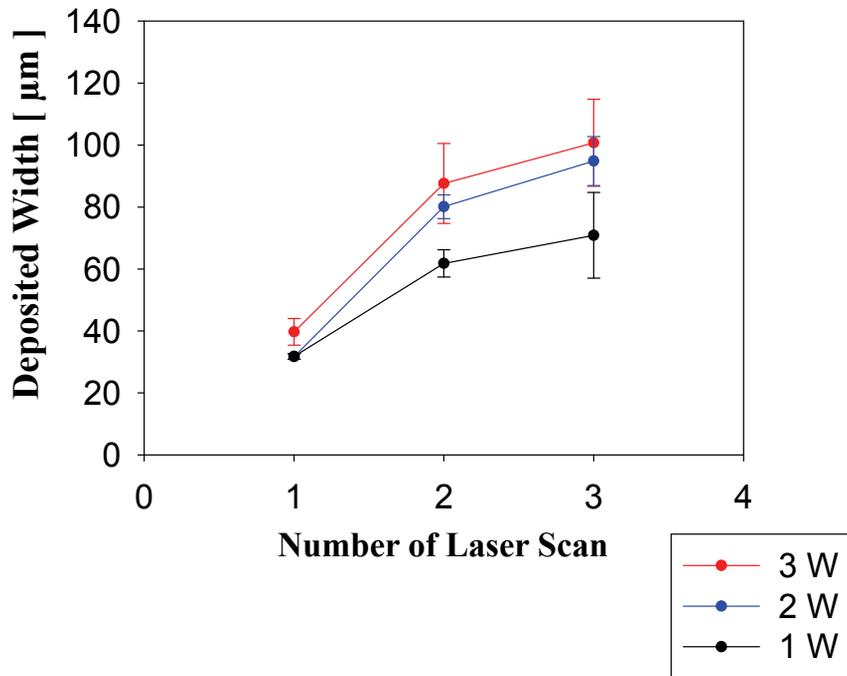


Fig. 6. Tendency of copper growth of (a) height and (b) width.

From the Fig. 6, even though copper wires' height and width grow since laser irradiated repeatedly, copper grows until 50 μm in its height (Fig. 6a) and until 100 μm in its width (Fig. 6b) but unpredictably. While copper wire's height is raising, width has changed spontaneously in its value. The height and width of copper wire are not independently separated. These results show that copper wires cannot be precisely controlled its dimension.

Copper deposition tendency those which are observed above can be overcome when deposition is applied inside of channel. This is because channel's shape and size can function as guidance while deposition is executed. For this expectancy, channels' dimensions are able to precisely controlled.

4.1.2 Durability

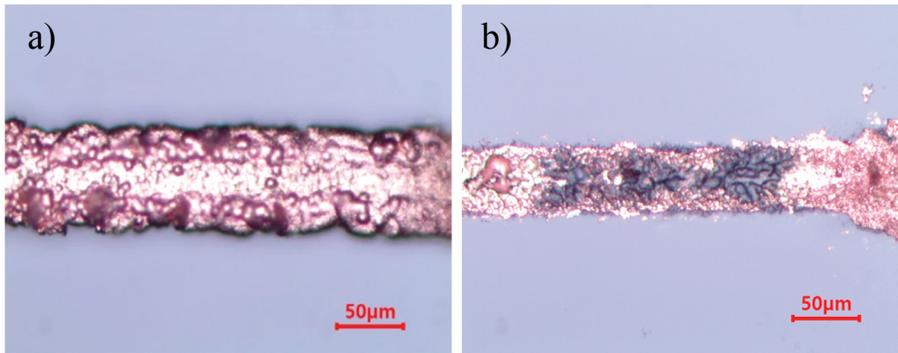


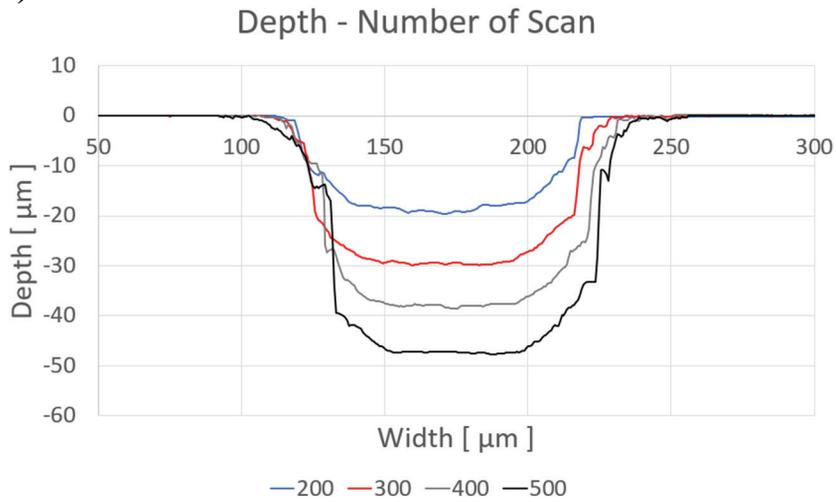
Fig. 7. Durability test. (a) Before and (b) after ultrasonic vibration.

Due to no previous treatments such as making rough surface for anchor effect [7] are implemented, the adhesion between glass surface and the deposited copper wire is not strong enough. From the Fig. 7a, 7b, normal copper wires are prone to ultrasonic vibration. The experiment is set under conditions of 50 W, and 28 kHz for 5 minutes. While copper wire is easily detached from the substrates, it will fail to be applied to various industrial applications. For better use of copper wire on glass material, securing robustness and adhesion seems to be needed.

4.2 Embedded deposition

4.2.1 Dimensions of channels - LIBWE

a)



b)

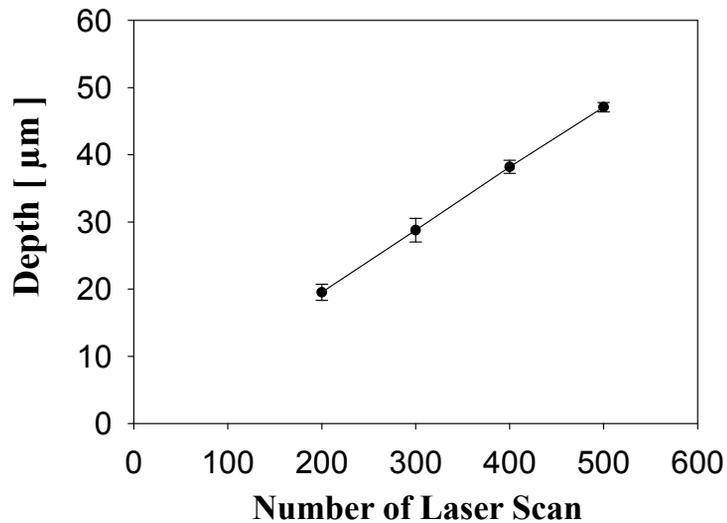


Fig. 8. LIBWE machining: Controlling depth of channel by number of laser scan.
(a) 3D profile data, (b) graph.

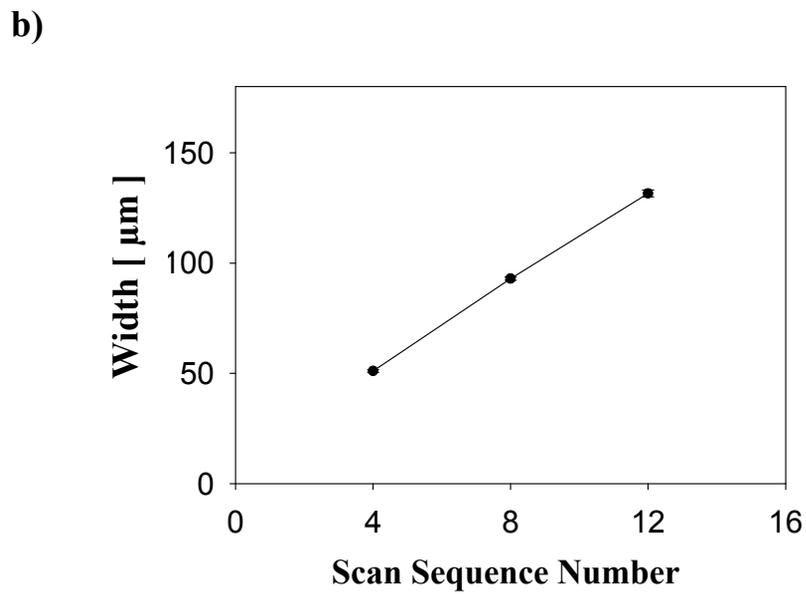
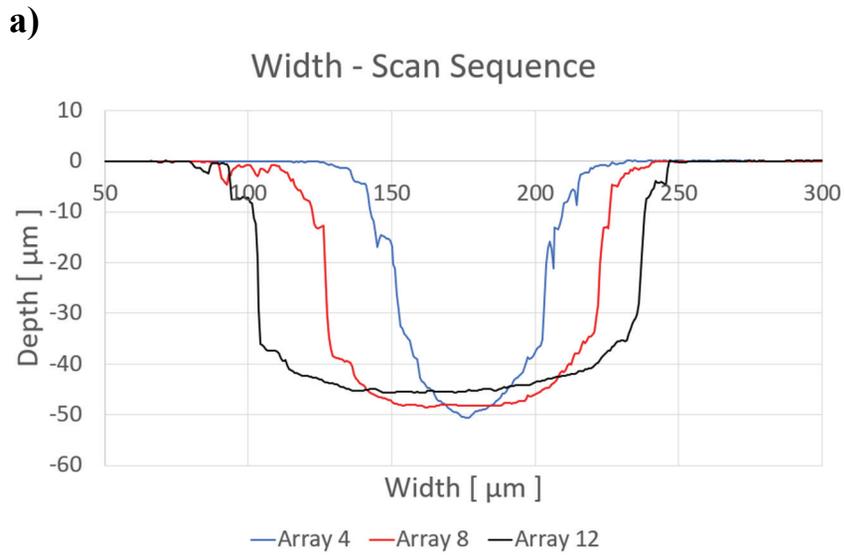


Fig. 9. LIBWE machining: Controlling width of channel by laser scan sequence. (a) 3D profile data, (b) graph.

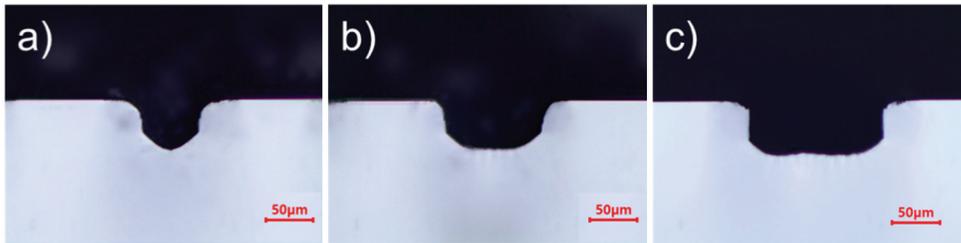


Fig. 10. Target channels' cross section views. Width of (a) 50 μm , (b) 100 μm , and (c) 150 μm . Depths are all 50 μm .

As mentioned above, LIBWE process is beneficial to generate seed layer for better results of LCLD. LIBWE process helps to generate seed layers due to metal ions are attached to fabricated surface [7]. In this case, 0.7 M of copper sulphate solution has been used as absorbents, thus copper ion is attached to surface of fabricated LIBWE channel. Because of previously attached copper ions, procedure for making seed layer can be omitted under LCLD process. LIBWE process offer both generating undercut of glass and the initiate seed layer of copper deposition.

Dimension of inserted copper wire is depending on channel's dimension. Hence, knowing channel's depth and width those which is produced by LIBWE process should be done prior to actual deposition. To adjust adequate width and depth of channels, scan sequence and repeating scan number are changed. Fabricated channels are observed and measured

with 3D profiler (Fig. 8a, 9a).

Channel has deepened while laser scan has repeated. 10 μm of depth has increased when every 100 times of laser scan has applied (Fig. 8b). Width of channel also well controlled by number of sequences. Channels which are used for copper deposition could be predictably fabricated before copper deposition applied. The goal depth of channel is 50 μm , and width varies 50 μm to 150 μm those which have interval of 50 μm (Fig. 9d). Target channels' sections are shown on Fig. 10.

4.2.2 Deposited shape & Controllability

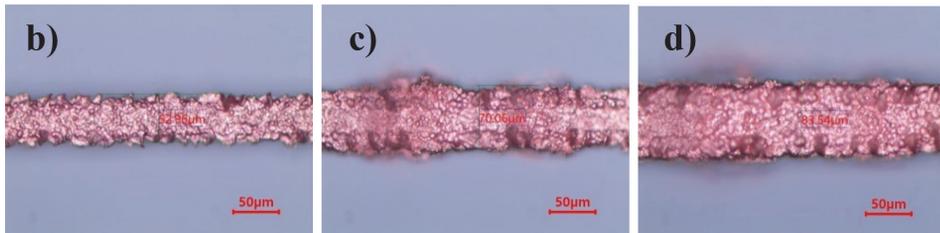
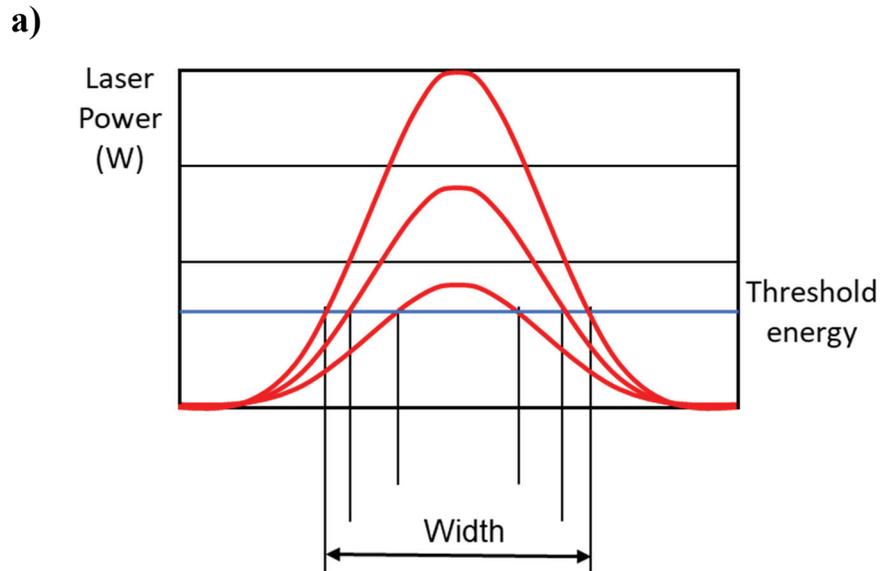


Fig. 11. Deposited width following laser powers.
(a) gaussian distribution of laser beam
laser power of (b) 1 W – 50 µm, (c) 2 W – 70 µm, and (d) 3 W – 85 µm.

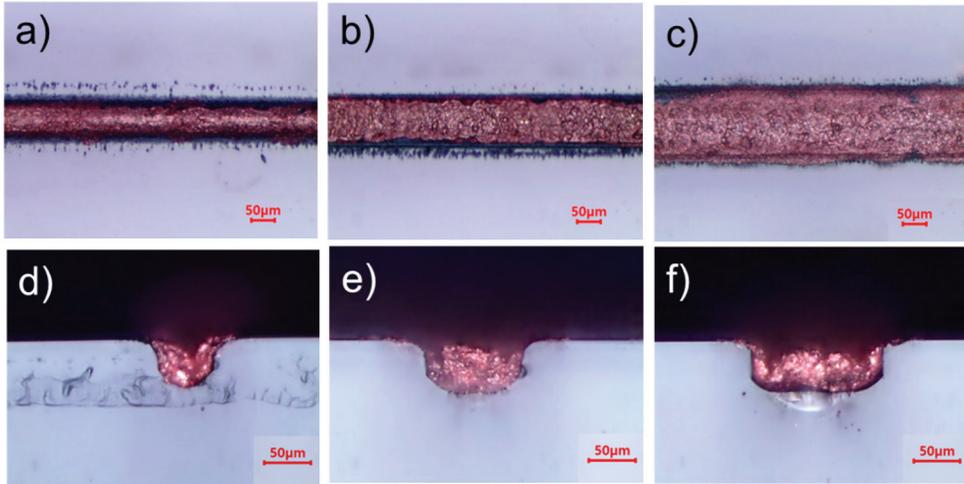


Fig. 13. Top view of embedded copper wires in width of (a) 50 μm , (b) 100 μm , and (c) 150 μm . Section views of (d) 50 μm , (e) 100 μm , and (f) 150 μm .

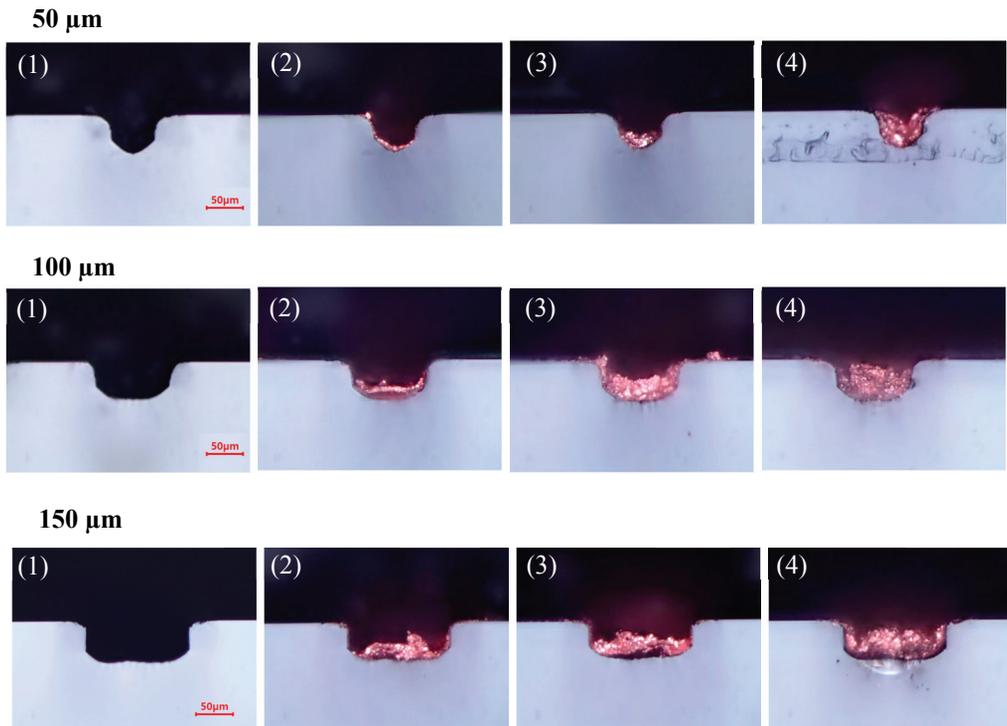


Fig. 12 Cross section views of the growing coppers that deposited inside of channels. Number of laser scans : (1) 0, (2) 1, (3) 2, (4) 3, respectively.

To deposit copper from channel's bottom, excessive reduction of copper needs to be prevented because excessive copper reduction occurs blocking of the entrance of channel. The blockage is a cause of deposition failure inside of channel. Due to above reason, laser power should be adjusted according to channel's width. The narrower channel is, the lower power of laser is needed (Fig. 11). In a production embedded copper wire to manufacture copper circuit, 1, 2, 3 W of laser powers were determined to be proper, correspond to each width of channels.

From the Fig. 12, deposited copper is well aligned and filled inside of various widths of channels. Since it is hard to recognize filling ratio of copper, cross section views are offered. Each widths of channels are fully filled with copper.

To verify how copper grows inside of channel, Fig. 13 is addressed. From empty channel to fully filled channel, as the number of laser scan increases, the height of copper grows inside of channel since width is fixed (Fig 13 (2) to (4)). While height was increased dramatically at the second scan of the laser when experiment is set on normal surface of glass, copper wire's thickness increases fairly constantly inside of channel. Amount of height growth variation changes depending on the width of channel.

4.2.3 Durability

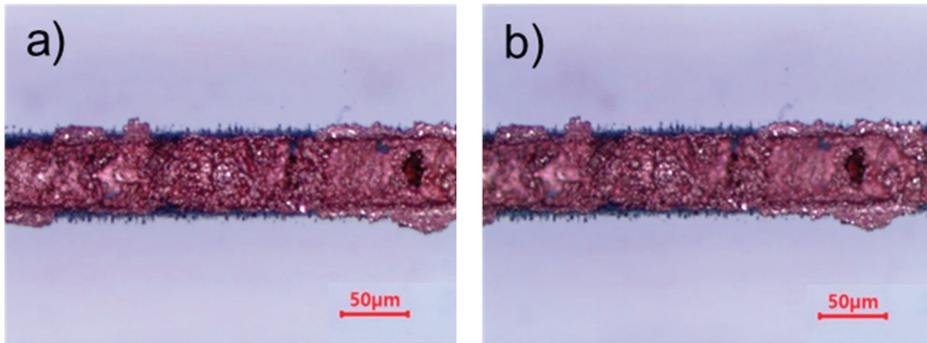


Fig. 14. Embedded copper wires durability test. (a) Before and (b) after ultrasonic vibration.

When copper wire is embedded beneath surface, more robust adhesion is obtained because porous and rough surface is generated from LIBWE process [18]. The result for embedded copper wire is shown on Fig. 14. Under the same ultrasonic vibration conditions (50 W, 28 kHz for 5 minutes) embedded copper wire robustly sustained.

4.2.4 Conductivity

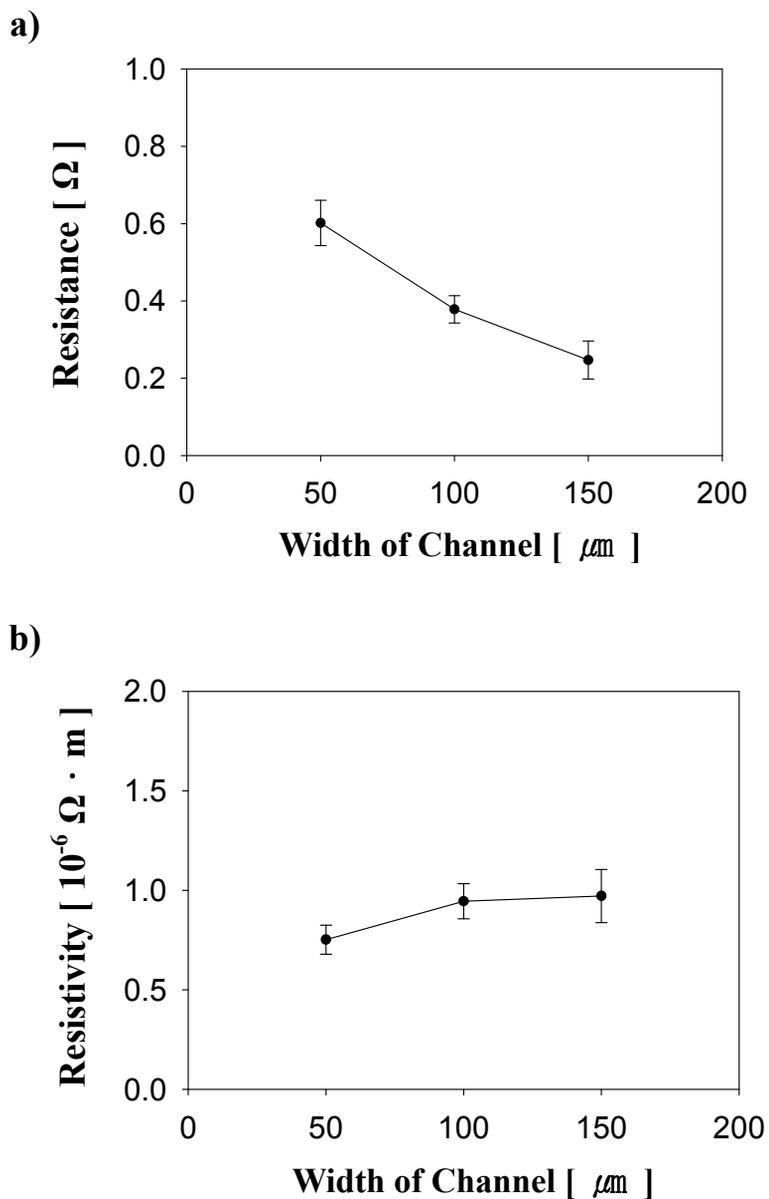


Fig. 15. Conductivity measurements. (a) Resistance (b) Resistivity

Resistance is decreased when copper wire is getting thicker and saturated. Controlling the resistance, height, and width through laser scan number on the normal surface of glass is hard due to uncertainty of copper deposition tendency. However, if copper inserted inside of channel, the amount of deposited copper could be controlled by channel's size. The resistance decreases while the size of channel has increased and filled with copper (Fig. 15a). Furthermore, by calculating resistivity from the results, $1.0 \times 10^{-6} \Omega \cdot m$ is obtained (Fig. 15b).

4.3 Applications

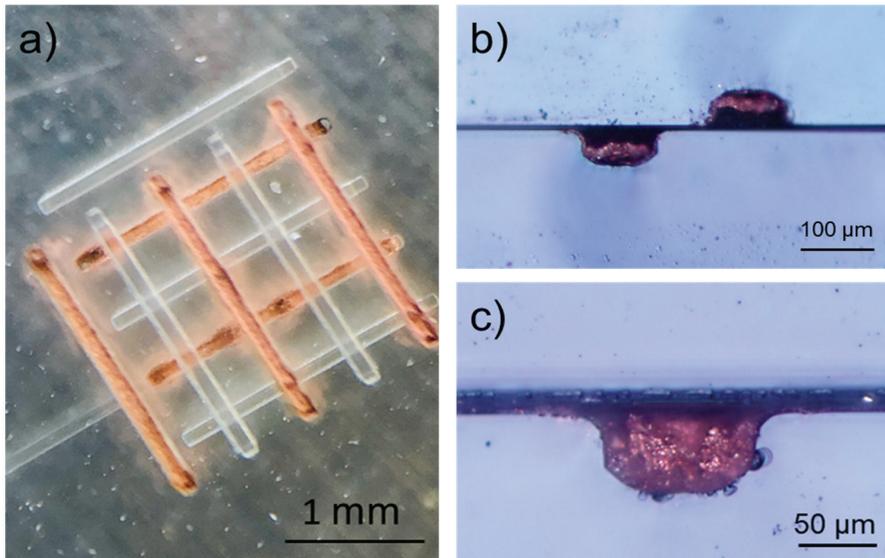


Fig. 16. Possibilities of applications. (a) Multilayers stacked wires, (b) microscope view of stacked structure, and (c) sealed copper wire by cover glass.

Since copper is wired inside of channel, some more applications can be considered. Copper wires can be organized in many layers without any further process or support (Fig. 16a, 16b). Furthermore, embedded copper can be sealed with other cover glass or materials to secure more usability and robustness (Fig. 16c).

Chapter 5

Conclusion

Many attempts for making practical and new generation of glasses with deposition techniques has been researched. Even though many glass deposition techniques are previously researched, deposited glass is not widely used. This is because durability and adhesion between deposited copper and glass is weak. If this problem can be improved, far more usage of deposited glass is possible to think of. In this study, copper wiring is tried inside of channels to protect deposited copper. Channels are precisely fabricated with LIBWE method and deposition has conducted with LCLD method. By combining those two methods, channels are function as precise guide of deposition. By doing so, embedded and robust copper wires are gained. Furthermore, controlling the resistances become easier due to controlling amount of deposited copper. Additionally, stacking and sealing, and far more applications are possible to apply for further use.

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국문 초록

일반적으로 유리는 다양한 특성을 가지고 있음에도 전도성을 가지고 있지 않아 활용에 제약이 있었다. 이에 전도성 물질을 도포하거나 증착하는 기술들은 많이 소개가 되어 있지만, 실사용에 있어서는 여러가지 이유로 잘 사용되지 못하였다. 이에 본 연구는 기존 유리에 대한 구리 증착 기술의 한계점인 내구성 및 접착성 대해서 지적하고 이를 극복하기 위한 조합공정을 제시하였다. 레이저 후면 식각 공정으로 유리를 미세가공한 후, 레이저 구리증착 공정을 통해, 깊이 50 μm 와 다양한 폭 (50 ~ 150 μm)의 미세가공물 내부에 구리를 증착하는 데에 성공하다. 그 과정에서 증착된 구리는 내구성 증대, 저항 조절성 확보 등을 특징으로 보였으며, 조합공정 효과에 대해 실험적으로 증명하였다. 더욱이 조합공정 결과물을 통해 유리 기반 회로, 다층 구조 회로 등의 가능성을 제시하였다.

주요어: 유리, 레이저 후면 식각 공정, 레이저 구리증착 공정, 구리 식립 공정, 구리 도선

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