Research Article

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Feasibility Study of Microneedle Fabrication from a thin Nitinol Wire Using a CW Single-Mode Fiber Laser

<https://doi.org/10.1515/eng-2019-0023>

Received Mar 29, 2019; accepted Jun 04, 2019

Abstract: In this paper, feasibility studies are presented on microneedle fabrication by using a multiple-pulse laser microhole drilling technique to drill an axial hole into a thin nitinol wire of 150 μm in diameter. Nitinol is chosen for its biocompatibility and excellent super-elasticity to eliminate breakage risk. One potential use of this microneedle is for drawing a small amount of blood for glucose monitoring. The critical factor for drilling microholes into a thin nitinol wire axially is the restoration of the semi-infinite material condition, which is the key to prevent the thin wire from being melt away by the laser due to excessive heat transfer in the radial direction. The results show that holes, up to 607 μm in depth, can be drilled into a thin nitinol wire of 150 μm in diameter using 18 repetitions of a 3-pulse group with 13 μs pluses. However, hole quality is poor. The challenges for improving hole qualities, such as centering, hole blockage, through holes, and process parameters, are discussed.

Keywords: Microhole Drilling, laser drilling Laser Ablation, Microsecond Pulse, Fiber Laser, Nitinol, Super-elasticity, Microneedle Drilling

1 Introduction

Laser drilling is an important industrial process for critical applications, such as fabricating cooling holes in turbine components. The development of high power, single-mode fiber laser established a new level of beam quality. In comparison with ns-, ps-, and fs-lasers, the peak power of this

modulated fiber laser pulse is low but its excellent beam quality allows the laser beam to be focused to a very small spot for reaching very high power densities, suitable for microhole drilling.

The laser beam produced by a 300 W, CW, Yb-doped single-mode fiber laser (YLR-300, IPG) has a near perfect beam quality $M^2 = 1.04$ at 1075 nm wavelength. In addition, this laser has an initial spike, which is five times of the steady state power. Due to its excellent beam quality, this laser beam can be focused down to 10 μm with a 100 mm lens, achieving a peak power density of 1.9 GW/cm^2 . This power density is comparable with that of a typical ns short pulse laser. However, unlike ns-lasers, this continuous wave laser can also be modulated to produce pulses from 1 μs to any length of pulse duration. Using this fiber laser, Tu *et al.* (2013, 2014) [1, 2] presented a single pulse drilling process of blind holes on a stainless steel plate with a pulse duration from 1 to 8 μs without assist gas. With a single 1- μs pulse, it was possible to produce a blind hole 167 μm in depth and 19 μm in the opening diameter on a 0.8 mm stainless steel plate. Note that the hole depth drilled by a single ns laser pulse is about 1 to 10 μm .

The drilling mechanisms were established by determining the contributions of hole drilling by evaporation and melt ejection theoretically and experimentally. It was found that evaporation contributed approximately 1/3 of the hole drilling, while melt ejection accounts for the remaining removal. A series of diagrams, denoted as process anatomy, were presented to illustrate the transition of the hole in this drilling process [1].

Tu *et al.* (2016) explored how this short, micro-second pulse drilling process could be extended to drill through holes on stainless steel plates using multiple pulses. It was established that there is a synergistic effect if a subsequent pulse is irradiated at the target within 100 μs of the previous pulse before the melt solidifies. Another contributing factor of the synergistic effect is related to the melt ejection efficiency. As the hole deepens, the melt ejection becomes less effective to eject the melt completely out of the hole, resulting in a partially blocked hole. A subsequent laser pulse needs to reopen the hole before the hole can be

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deepened further. The total drilling times through an 800 μm plate were found to be 634 ms and 21.9 ms at 13 kHz and 20 kHz, respectively. The drilling efficiency at the 20 kHz repetition rate is drastically higher, needing only 428 shots, compared with 8240 shots at the 13 kHz, an improvement of nearly 200 times. It is confirmed that this multiple-pulse drilling technique with microsecond pulses using a 300 W single mode fiber laser is a viable technique to produce high aspect ratio through holes with a simple and robust setup for the production environment.

In this paper, feasibility studies are presented on the use of the above reviewed microhole drilling technique to drill an axial hole into a thin nitinol wire of 150 μm in diameter. Nitinol is chosen for its biocompatibility and excellent super-elasticity to eliminate breakage risk. One potential use of this microneedle is for drawing a small amount of blood for glucose monitoring.

This paper is organized as follows. First, a brief review of micro-needle fabrication is provided. Important process parameters of micro-hole drilling with a 300 W, CW, Yb-doped single-mode fiber laser are then presented. Difficulties in drilling a microhole into a thin nitinol wire are discussed and solutions to overcome these difficulties are proposed. Finally, experimental procedures and results are presented, followed by discussions and conclusions.

2 Review of Micro-Needle Fabrication

2.1 Conventional Hypodermic Needles and Fabrication Limits

The industry behind the conventional hypodermic needle is large with many companies and manufacturers but the manufacturing techniques are similar. To create the needle itself, molten steel is first drawn through a die specific for each gauge of needle. As the steel is drawn, a continuous hollow wire is formed and rolled before it is cut into the specific length of each needle. This is the most common form of mass needle production. Needle geometries that are too complex to be easily produced with drawn steel are manufactured using die casting. The syringe barrel and plunger are typically made using extrusion molding or injection molding of plastics, glass and rubber and may be fitted to the needle during assembly and packaging [4].

Although the conventional hypodermic needle has greatly expanded the abilities of modern pharmaceuticals, it does have its limitations. The smallest currently available conventional needles are 30 gauge or 305 μm in outer

diameter. Pin injectors are available as small as 31 gauge or 254 μm . These smaller needles are typically used for insulin monitoring and administration [5]. Since the force required for a needle to puncture the skin is directly dependent on the needle tip diameter, smaller needles require less force, resulting in less pain and skin damage to the patient [6, 7].

2.2 Microneedle Applications

Modern medicine would benefit greatly from a reliable method of transporting drugs through the skin. Unfortunately, the outermost layer of human skin, the stratum corneum, is amazingly effective as a barrier to the outside environment despite being only 10 to 15 μm deep [8]. Of the many attempts to increase skin permeability for drug and fluid transport, the creation of microscale needles has become the most promising and active approach [9]. Microneedles have been manufactured in a variety of lengths from 30 μm to 6 mm [5, 10] and offer extremely small tip diameters as small as 5 μm [5].

Hollow microneedles also give promise for the painless removal of blood and other fluids from around the epidermis. More efficient methods of testing can now monitor glucose levels in blood with as little as 200 nL samples [11]. This has the potential to be removed using microneedle arrays or a single microneedle [7, 11]. In addition to monitoring glucose levels, hollow microneedles could replace catheters in delivering precisely controlled amounts of insulin [6].

2.3 Development of microneedles

A majority of the literature on microneedles indicates that the most common material used is silicon. Henry *et al.* (1998) created the first solid microneedles in 1998 with silicon using the microfabrication process of integrated circuits (IC). Lin and Pisano (1999) published a method employing IC manufacturing techniques to produce a silicon microneedle. This approach used a larger hollow needle instead of an array of solid needles and would allow the use of bubble-powered pumps on the micron scale to electrically control delivery rates. Silicon dioxide was used to fabricate glass hollow microneedle arrays machined from bulk silicon [5]. Silicon hollow microneedle arrays produced using micromachining and etching were developed [7]. Despite the success of silicon microneedle fabrication, the risk of needle breakage due to the brittle nature of silicon cannot be overlooked.

Metallic microneedles were first created using seed layers machined from bulk silicon and the plating of a layer of metal such as palladium, gold and nickel [5, 6]. Metallic hollow microneedle arrays were created with silicon molds using laser fabrication that featured tapered geometries and varying wall thicknesses [12]. Laser cutting was used to create a range of geometry profiles in stainless steel plates. These profiles were then bent out of plane to create a solid microneedle array on the stainless steel substrate [13]. Another approach to creating solid metallic microneedle arrays was demonstrated by solidifying laser-induced ablation of tantalum plate. This method produced solid microneedles with extremely small tip radii and aspect ratios as high as six [14]. Microneedles with metal plating would need a wall thickness of $18\text{ }\mu\text{m}$ to sustain a bending moment up to 0.71 Nm to ensure necessary strength and flexibility of the microneedle [5].

Although the use of titanium and steel is more desirable than nickel, silicon, or silicon dioxide for microneedles because of their ductility and biocompatibility, fabrication of microneedles using these materials is still lacking. Nitinol, as an alloy of titanium and nickel, is the material used in vascular surgery and other applications due to its super-elasticity, shape memory capability, and biocompatibility. Titanium is nearly entirely inert, while nickel is a known allergen. However, Nickel and Titanium form an extremely strong intermetallic bond, preventing the release of nickel ions to raise the risk of adverse reaction [15].

2.4 Proposed nitinol hollow microneedles

Figure 1 is an illustration of the envisioned microneedle. A microneedle is fabricated by drilling an axial hole with a diameter from $50\text{ to }75\text{ }\mu\text{m}$ and a depth of $300\text{ to }700\text{ }\mu\text{m}$ into a thin nitinol wire of a diameter from $80\text{ to }150\text{ }\mu\text{m}$. For comparison, a typical acupuncture solid needle has a diameter typically at $80\text{ }\mu\text{m}$, and it is known for painless insertion into human's tissue. In this study, nitinol wires of $150\text{ }\mu\text{m}$ meter were chosen for experiments because it is the thinnest wire available. A second hole is drilled from the side so that a blood flow passage can be formed to draw blood into a collection device for glucose monitoring. In this paper, the feasibility of drilling an axial hole into the thin nitinol wire is explored. The feasibility of drilling the radial hole involves likely totally different mechanisms, to be explored in the future, and is beyond the scope of this paper. No microneedles reviewed in Section 2.3 featured such a radial hole.

Figure 1: An envisioned microneedle, made of a thin nitinol wire, combined with a glucose monitoring system.

3 Microhole Drilling with CW Fiber Laser

3.1 Profiles of Modulated Fiber Laser Pulses

As reviewed above, the laser beam of a continuous-wave, single-mode, Yb-doped fiber laser (YLR-300, IPG) is modulated to produce short laser pulses for drilling microholes. An external circuit was designed to modulate laser pulses with durations from $1\text{ }\mu\text{s}$ to 1 s . This laser modulation control is different from the Q-switching control because the modulated laser power remains constant while the deposited energy is determined by the pulse duration.

Figure 2 illustrates the measured laser beam profiles of the $1\text{-}\mu\text{s}$ and $15\text{-}\mu\text{s}$ laser pulses. For the $1\text{-}\mu\text{s}$ pulse, the laser power initially rises to about 1500 W at 500 ns and then falls to the steady state value of 300 W after $1\text{ }\mu\text{s}$. The overall pulse gradually decays to zero after $10\text{ }\mu\text{s}$. For the $15\text{-}\mu\text{s}$ pulse, the same initial spike and the decaying pattern at the end of the pulse are observed. However, the laser power rebounded to about 500 W after $1\text{ }\mu\text{s}$ and then settles to the steady state power of 300 W for an additional $14\text{ }\mu\text{s}$. As highlighted in Figure 2, the energy contained in the initial spike is deposited to the material at a much higher power density; therefore, this energy can ablate the material. On the other hand, after the initial spike, the energy deposited is at a much lower power density, only sufficient to melt the material for advancing the microhole depth. Detailed discussion of the temporal profile of this laser beam can be found in Tu *et al.* (2013).

Figure 2: Profiles of 1- μ s and 15- μ s laser pulses. Both pulses have an identical initial spike at 1,450 W which is approximately five times of the steady state power of 300 W. This is an improved diagram from Tu *et al.* (2016).

3.2 Process Anatomy of Single Pulse Drilling

Based on the experimental and simulation results presented in Tu *et al.* (2013), a temporal process anatomy diagram of the microsecond laser drilling, using the single-mode fiber laser, was compiled. Four stages of the drilling process are depicted in Figure 3 to depict the laser/material interaction mechanisms, hole formation, material removal mechanisms, and vapor/plasma properties. This process anatomy diagram is also color-coded to provide the temperature values during the process. According to Figure 3, in the first three stages ($t < 600$ ns), the hole drilling is mainly due to evaporation by the energies from the initial spike of the laser beam and those radiated from the induced plasma. Evaporation accounts for 1/3 of the final hole depth. Between stages 3 and 4, the hole is further deepened by the steady state laser power at 300 W. Finally, at the end of stage #4 ($t = 5$ μ s), the vapor pressure has decreased, which allows the melt to explode away as droplets seen in Figure 3. The melt ejection in stage 4 accounts for 2/3 of the overall hole drilling.

3.3 Process Anatomy of Multiple-Pulse Drilling with Microsecond Pulses

The modulation controller also allows for producing multiple pulses at precise time intervals. The profile of a group of ten-pulse laser beam is shown in Figure 4. It was found that if the subsequent laser pulses are fired within 100 μ s, the drilling process is more efficient. Detailed discussion on multiple-pulse microhole drilling can be found in Tu *et al.* (2016).

Figure 3: Process anatomy of the rapid drilling process using a one micro-second laser pulse generated by a single-mode CW fiber laser. This is an improved diagram from Tu *et al.* (2013).

Figure 4: Ten shots pulse profiles and plasma radiation measurements.

The process anatomy of a multiple-pulse drilling group is illustrated in Figure 5 for the 2nd pulse, 3rd pulse, 4th pulse, and beyond. The process anatomy of the first pulse is the same as Figure 3.

Second Pulse Stage

The second pulse starts 61 μ s after the first when a hole is formed. It is important to keep this delay interval less than 100 μ s so that the hole wall has not completely solidified. In this way, the second pulse does not have to re-melt the material and the hole can be deepened substantially to nearly 300 μ m, as shown in first image of Figure 5. However, when the hole becomes deeper, the ejection process becomes less efficient. As the melt begins to move up the cavity, some of it can solidify near the top of the hole, causing hole blockage.

Subsequent Pulse Stages

The third pulse needs to reopen the hole blockage by evaporation, producing high plasma radiation, as shown in the second image of Figure 5. With the hole re-opened, the fourth pulse can again reach the bottom of the cavity to further widen and deepen the hole (third image). As the

Figure 5: Subsequent process anatomy with multiple pulse drilling. This is an improved image based on Tu *et al.* (2016).

hole gets deeper, again, the melt ejection becomes more difficult. The melt from the bottom of the hole can now re-solidify in the middle and near the opening of the hole (fourth image). The hole must be re-opened before it can be deepened. The deepening and re-opening are repeated during the multiple-pulse drilling process.

The decrease in laser peak power from the first pulse to all following pulses causes a diminishing return effect in hole depth formation as additional pulses are added to a group. A group of three pulses was chosen as the basis for further multiple-pulse experiments in stainless steel and nitinol. This configuration often demonstrated significant increase in total hole depth without increasing molten material to a point that there was frequent solidification and hole blockage. Experimental results discussed below were achieved using repetitions of this three-pulse group over a millisecond time scale. Using this method, the first or primary pulse of each group repeated the 1,500 W peak power output to keep drilling efficiency high as hole depth increased.

As envisioned in Figure 1, the microhole depth needs to be deeper than 300 μm ; therefore, the multiple-pulse drilling technique will be used for the fabrication of the proposed microneedles.

4 Experimental Setup and Sample Polishing

The laser beam of the fiber laser was delivered via an optical fiber, a collimator, an isolator, an expander and a laser head which has a 100 mm focusing lens. The laser beam was then focused down to a spot size approximately 10 μm in diameter at the sample surface. The power densities pro-

duced by the pulse were approximately 1.9 GW/cm^2 at the peak power of 1,500 W and 380 MW/cm^2 at the steady state power of 300 W.

Pulses were fired at a 0.8 mm thick plate of SS316 stainless steel and nitinol wires. The samples were cleaned with acetone prior to the tests, but no other preparation was performed. The samples were examined using an optical microscope. Cross sectioning was then performed to measure the depth and the geometry of the hole. Note that because the hole size is very small, approximately 50 μm in the opening, the sample polishing process is very time consuming and great attention is needed to determine the hole center by removing a few microns each time. Even with an advanced polishing machine, often the center of the hole was missed. In average, it took approximately 4 hours to polish a hole and about 20 images were taken to determine the likely maximum hole depth.

5 Results

5.1 Single Pulse Drilling

As reviewed above, our previous research work on laser microhole drilling had focused on drilling holes on stainless steel plates of various thickness. For the microneedle fabrication, nitinol was chosen for its excellent super-elasticity property and biocompatibility. Two nitinol wires of $\varnothing 350\mu\text{m}$ and $\varnothing 150\mu\text{m}$ were acquired. No nitinol wires thinner than $\varnothing 150\mu\text{m}$ were available at the time of the experiment. The first task is to compare the hole characteristics between samples of a stainless steel plate and a $\varnothing 350\mu\text{m}$ nitinol wire. Two holes, each drilled by a single 10 μs pulse, are shown in Figure 6. Note that the hole drilled in the stainless plate is substantially deeper (175 μm vs 118 μm) with a narrower opening (37 μm vs 52 μm). This result is in fact not expected because nitinol has a lower thermal conductivity than that of the stainless steel. Typically, materials with higher thermal conductivity will produce lower aspect ratio microholes. The material properties of stainless steel and nitinol are listed in Table 1.

In order to determine the reason for this discrepancy, the heat transfer conditions during laser drilling via an idealized laser drilling model is examined.

When the laser beam is first irradiated at the surface of a sample, the heat transfer mechanism can be described by the classical heat equation in a cylindrical coordinate

Figure 6: Comparison of microholes drilled by a 10 upmus pulse on a stainless plate (a) and a nitinol wire (b).

Table 1: Material Properties of Stainless Steel and Nitinol

	Stainless Steel	Nitinol
Density, ρ	7500 kg/m ³	6450 kg/m ³
Thermal Conductivity, k_s	29 W/m K	18 W/m K
Heat Capacity, c	630 J/kg K	620 J/kg K
Melting Temperature, T_m	1400 K	1310 K
Vaporization Temperature, T_v	3134 K	2760 K
Latent Heat, Melting, L_m	0.2×10^6 J/kg	0.2×10^6 J/kg
Latent Heat, Evaporation, L_v	7.6×10^6 J/kg	7.6×10^6 J/kg

system (r, θ, z) , centered on the laser [16],

$$\rho c \frac{\partial T}{\partial t} = k_s \left[\frac{1}{r} \frac{\partial}{\partial r} \left(r \frac{\partial T}{\partial r} \right) + \frac{\partial^2 T}{\partial z^2} \right] \quad (1)$$

$r > 0, z > 0, t > 0$

where T is the temperature, ρ , c , and k_s are respectively the density, heat capacity, and thermal conductivity of the solid material. The boundary condition is, with W as the laser power flux irradiated at the surface,

$$k_s \frac{\partial T}{\partial z} = -W \quad \text{at } z = 0 \quad (2)$$

If non-dimensionalizing Equation (1) is conducted by defining,

$$r^* = R_b, \quad z^* = \frac{k_s (T_v - T_m)}{W}, \quad (3)$$

$$t^* = \frac{\rho c k_s (T_v - T_m)^2}{W^2}$$

where R_b is the radius of the laser beam, and T_v and T_m are respectively the vaporization and melting temperature. The non-dimensional variables are defined as,

$$\tilde{r} = \frac{r}{R_b}, \quad \tilde{z} = \frac{z}{z^*}, \quad \tilde{t} = \frac{t}{t^*}, \quad \tilde{T} = \frac{T - T_m}{T_v - T_m} \quad (4)$$

Equation (1) can then be non-dimensionalized as

$$\frac{\partial \tilde{T}}{\partial \tilde{t}} = \epsilon \left[\frac{1}{\tilde{r}} \frac{\partial}{\partial \tilde{r}} \left(\tilde{r} \frac{\partial \tilde{T}}{\partial \tilde{r}} \right) + \frac{\partial^2 \tilde{T}}{\partial \tilde{z}^2} \right] \quad (5)$$

where

$$\epsilon = k_s^2 \frac{(T_v - T_m)^2}{R_b^2 W^2}$$

The value of ϵ is the ratio of laser energy transfer in the r direction to the z direction. For stainless steel, the value of ϵ is in the order of 10^{-8} , which indicates that the laser energy is almost entirely in the z direction. It can be found that the value of ϵ for stainless steel is 4.5 times that of nitinol. In other words, there is much less laser energy being transmitted in the radial direction for the nitinol sample. As a result, the opening diameter of a microhole drilled in nitinol should be smaller than that of stainless steel. However, the result of Figure 5 is the contrary. Closer examination of the drilling condition reveals that this contrary result is due to the difference in boundary conditions. Equation (1) assumes that the sample is a semi-infinite material and temperature is at the room temperature at infinity. This assumption is true for the stainless steel plate sample. However, for the nitinol wire with a diameter of 350 μm , though 35 times of the beam diameter, it is not a semi-infinite material. The temperature at the outside boundary of the nitinol wire cannot stay at the room temperature, allowing the melt front to advance radially to enlarge the hole opening.

Figure 5 indicates a serious problem for drilling a microhole into a thin nitinol wire. As the wire diameter gets smaller, the hole opening could be as large as the entire wire diameter. As a result, a hole cannot be formed without the tip of the wire melting away. This observation was confirmed when the nitinol wire of 150 μm was used for microhole drilling

5.2 New Drilling Setup for Thin Nitinol Wires

In order to drill a microhole into a nitinol wire with a diameter at or less than $150\text{ }\mu\text{m}$, a new setup for drilling thin nitinol wires was proposed. The idea is to restore the semi-infinite material condition for a thin wire so that the heat transfer in the radial direction is limited for achieving high aspect ratio microholes axially into the thin nitinol wire. For this objective, it is proposed to mount the thin nitinol wire to a stainless steel plate so that the wire and the plate together constitute a semi-infinite material. This new setup is depicted in Figure 7.

Figure 7: Proposed setup for drilling thin nitinol wires to restore semi-infinite material condition.

As shown in Figure 7, a hole slightly larger than the nitinol wire is drilled through a stainless steel plate with a $700\text{ }\mu\text{m}$ thickness. This hole is created through mechanical drilling using micro-drill bits. This plate is held by a fixture to be perpendicular to the laser beam. A nitinol wire is then inserted into the hole and flush with the top surface of the stainless steel plate. Note that even the wire is positioned to be flush with the plate surface, it is possible that the tip of the wire, for example, $50\text{ }\mu\text{m}$, could be above the surface. No practical method to measure the protrusion in the actual setup (Figure 8) is available. Before insertion, heat conductive paste is applied inside the hole to fill the gap between the nitinol wire and the inner wall of the hole. The paste helps to reduce the thermal resistance between the wire and the stainless steel plate. As a result, the stainless steel plate and the nitinol wire now form a solid piece as a semi-infinite material. The actual setup is shown in Figure 8.

For the $\varnothing 150\text{ }\mu\text{m}$ nitinol wire, $\varnothing 200\text{ }\mu\text{m}$ drill bits are used to drill through the stainless plate. The actual hole created has a diameter of $244\text{ }\mu\text{m}$. Therefore, the gap between the wire and the inside wall of the hole is $47\text{ }\mu\text{m}$, which is filled with the heat conductive paste. Two types of heat conductive pastes were used for experiments. The silicon paste has a thermal conductivity of 149 W/mK , while

Figure 8: Actual setup for drilling thin nitinol wires to restore semi-infinite material condition.

the diamond paste has 3000 W/mK . Both pastes have a much higher thermal conductivity than those of nitinol and stainless steel.

The microholes drilled on a stainless plate is used as the upper bound in drilling performance and compare them with the microholes drilled on thin nitinol wires. This comparison provides a measure if the setup of Figures 7 and 8 restores the semi-infinite material condition.

5.3 Micro-Needle Fabrication with $\varnothing 150\text{ }\mu\text{m}$ Nitinol wires

Based on Figure 6, it is clear that one single laser pulse is not capable of creating a microhole with sufficient depth into a nitinol wire, as envisioned in Figure 1. Therefore, the use of three shots of the $10\text{ }\mu\text{s}$ pulse for microneedle fabrication using different heat conductive pastes was explored. These three pulses are the same as the first three pulses shown in Figure 4, with the first one at 1500 W and the second and third pulses at about 900 W . These three pulses is designated as one group. Note that the interval between the pulses within the group is kept to be less than $100\text{ }\mu\text{s}$ for synergistic effect. The entire group can be repeated at a lower repetition rate so that the first pulse will be at 1500 W . The results are shown in Figure 9. According to Figure 9, it appears that the heat conductive paste does not have much effect because the shapes of the microholes in all three microneedles are very similar with the hole diameter in the range of $\varnothing 20\text{ }\mu\text{m}$.

These results indicated that as long as the nitinol wire is mounted inside a stainless steel plate, the semi-infinite material condition is restored. The hole drilled in the nitinol wire now has an opening diameter less than that of a stainless sample, as predicted by the heat transfer model. The hole diameter in all three conditions are simi-

Figure 9: Microneedles with laser-drilled microholes using one group of three 10 μ s pulses, with different heat conductive pastes.

lar. Regarding the hole depths, the one with silicon paste is 190 μ m, vs 286 and 262 μ m for the other two conditions, respectively. One possible explanation is that the tip of the wire was melted away.

As discussed above, when a wire was inserted into the plate, attempts were made to make it flushed with the top surface of the plate. However, it could happen that the tip of the wire could be sticking out of the plate by, for example, 50 μ m. This exposed tip of the wire does not satisfy the semi-infinite material condition and it could easily be melted away by the laser beam, as discussed in Section 5.1. Melting at the tip of the wire is apparent in the results presented in Figure 9, which is a supporting evidence for the need of the semi-infinite material requirement.

As discussed above, the average gap between the nitinol wire of $\varnothing 150$ μ m and the inside hole wall of the stainless plate (Figure 7) is 47 μ m. However, it is difficult to know if the wire is completely centered inside the stainless steel plate hole. The wire might be in contact with the inside wall. The silicon paste was chosen for all the subsequent experiments. An indirect effect of using the silicon paste was the ability to work with higher flow rates of assist gas from the laser head, possibly increasing the efficiency of removing molten material from the hole. Without the use of paste, almost any use of the assist gas would blow the nitinol wire out of the stainless steel plate.

5.4 Microneedles with Deeper Holes

In order to achieve higher hole depth, seven repetitions of the three 10 μ s pulse group are used to drill a microhole axially into $\varnothing 150$ μ m nitinol wires to form a microneedle as envisioned in Figure 1. The group repetition rate is at 100 Hz. Two of such microneedles are shown in Figure 10. In the left of Figure 10, the microhole has an opening of $\varnothing 97$ μ m and a depth of 200 μ m. However, the actual hole penetration is 365 μ m, but the hole was blocked by melt, as

Figure 10: Two Microneedles with laser drilled microholes using seven repetitions of the three 10 μ s pulse group. Needle (a) shows more blockage than needle (b).

illustrated in Figure 5. The microneedle shown in the right of Figure 10 has a depth of 92 μ m and an opening of $\varnothing 79$ μ m. The actual penetration is 360 μ m.

From Figure 10, it is also apparent that melting of the wire tip, as pointed out in Section 5.1, might have occurred. The technical challenges of centering will be discussed in the next section. It is also clear that centering is very critical in the drilling process. Centering, however, is not a trivial technical challenge and it is very time consuming to achieve correct centering alignment. As discussed above, the microhole drilling on a stainless steel plate can be used as a performance upper bound for microneedle fabrication. Drilling on a stainless plate is much easier to perform because no centering is required.

5.5 Process Parameters based on Microholes in Stainless Steel Plates

As shown in Figure 10, the microneedle fabrication is achieved by drilling axial holes into $\varnothing 150$ μ m nitinol wires. However, the hole blockage by re-solidified melt is a major issue. In this section, microholes drilled on stainless plate are presented for exploring better process parameters.

First, the effects of repetition numbers of the three-pulse group is explored. As indicated by Figure 5, additional laser pulses can reopen the blockage and deepen the hole. In Figure 11, microholes drilled with 7, 13, 18, and 24 repetitions of the 10- μ s pulse group are presented. The results of seven groups into stainless steel shown in Figure 11 may be compared to those into the $\varnothing 150$ μ m nitinol wire of Figure 10.

Figure 11: Microholes on stainless steel plates drilled by 7, 13, 18, and 24 repetitions of the three 10 μ s pulse group.

At 7 pulse groups, a hole was formed into stainless steel with an opening of $\varnothing 33 \mu\text{m}$ and a depth of 301 μm (Figure 11), as compared with $\varnothing 97 \mu\text{m}$ and a 365 μm , respectively on a $\varnothing 150 \mu\text{m}$ nitinol wire, with the semi-infinite material condition setup (Figure 10). The hole depth for this case is not conclusive because it is difficult to tell the actual depth due to the blockage. Both have severe blockages, but unlike Figure 6, the microholes in the thin nitinol wire is now bigger and deeper. This is a clear indication that the semi-infinite material condition is at least partially restored with the proposed setup.

As shown in (b) of Figure 11, with 13 pulse groups, the melt blockage was opened and the hole becomes deeper. At 18 pulse groups (Figure 8c), the hole is greatly deepened to 418 μm but the blockage becomes severe again. At 24 pulse groups (Figure 8d), the hole is reopened and deepened to 442 μm .

The results of Figure 11 only indicate that the blocked hole can be re-opened with different numbers of repetitions of the three-pulse group. It is difficult to reliably predict if a specific number of group can re-open the hole blockage. This issue will be further address in Section 6.

5.6 Microneedle Fabrication with Different Number of Pulses and Pulse Durations

Based on Figure 11, experiments were conducted to fabricate microneedles from $\varnothing 150 \mu\text{m}$ nitinol wires at 7 and 18 laser pulse groups with durations of 10 and 13 μs . The results are shown Figures 12 and 13.

Figure 12 presents two microneedles fabricated by 7 repetitions of the 13 μs pulse group. It is apparent that a

Figure 12: Microneedles drilled by 7 repetitions of the three 13 μ s pulse group.

Figure 13: Microneedles drilled by 18 repetitions of the three 10 μ s pulse group and the three 13 μ s pulse group.

longer laser pulse promotes more melting, as analyzed in Tu *et al.* (2013; 2014), without deepening the hole. The hole depth is shortened to 320 and 271 μm , respectively. The largest hole open is $\varnothing 127 \mu\text{m}$, almost the same of the nitinol wire diameter ($\varnothing 150 \mu\text{m}$). Due to centering error, both microneedles have partially melted wall. The thinnest section of the wall is 24 and 10 μm , respectively.

Figure 13 presents two microneedles fabricated by 18 repetitions of the 10 and 13 μs three-pulse groups, respectively. The hole depth with 18 groups of the 10 μs pulse is 486 μm with an opening diameter about $\varnothing 92 \mu\text{m}$. However, due to centering error, this is a partial hole. The microneedle fabricated by 18 groups of the 13 μs pulse has a hole

depth of 607 μm . There is also centering error to produce an incomplete hole.

6 Discussions, Conclusion, and Future Work

The results presented in Section 5 supports the feasibility to fabricate microneedles using the microhole drilling technique with a modulated CW single-mode fiber laser, pioneered in Tu *et al.* (2013; 2014; 2016). However, before such technique can be adopted for actual microneedle fabrication, several technical challenges must be addressed.

6.1 Centering Errors

From Figures 9, 12 and 13, it is evident that the laser beam must be placed precisely at the center of the nitinol wire. Although there is no theoretical barrier to achieve such precision requirements, it is not a trivial task in our current laboratory setup. The control resolution of the x-y stage for positioning of the laser beam is $\pm 1 \mu\text{m}$, which is more adequate for centering the laser beam; however, the actual beam center is very difficult to determine because the laser beam is infrared, not visible to human eyes. In addition, because there is a 47 μm gap between the holding hole drilled in the stainless steel plate and the $\varnothing 150 \mu\text{m}$ nitinol wire, the inherent positioning error could be as large as 47 μm every time a new wire is mounted. To reduce this positioning error, the holding hole drilled by mechanical drill bit should be closer to the actual wire diameter. For actual production, a holding hole could be drilled by wire EDM for more precise dimensions.

6.2 Laser Pulse Parameters

From the results of Section 5, it is clear that melting could have a positive or negative effect on microneedle fabrication. Melt ejection contributes rapid and deep hole depth generation but melt ejection could also block the microhole. Shorter laser pulses could reduce melting but with lower hole deepening rates. For quality control, it might be preferred to limit the pulse duration to be 10 μs or less. The number of pulses is also important in the microhole drilling but it is very hard to predict if there is hole blockage. However, sensors can be used, such as photodiodes, to detect the blockage during drilling, as discussed in Tu *et al.* (2016).

6.3 Microneedles with Through Holes

As discussed in Tu *et al.* (2016), a through hole can be drilled through a 800 μm with 428 shots of 1 μs at a repetition rate of 20 kHz. Therefore, it is likely a through hole can be drilled through a nitinol wire of 700 μm length, inserted in the hole of the stainless steel plate. A hole of 607 μm is already achieved with 18 shots of the 13 μs pulse (Figure 13).

In a through hole drilling, the hole blockage due to re-solidified melt is no longer an issue. Although through hole drilling experiment was not conducted in this study, it is expected that a through hole through a nitinol wire with 700 μm in length is achievable.

6.4 Nitinol wires thinner than $\varnothing 150 \mu\text{m}$

Microhole drilling for nitinol wires thinner than $\varnothing 150 \mu\text{m}$ was not conducted because such wires were not available to us. It is envisioned that with shorter laser pulses, microhole needles with a diameter about $\varnothing 100 \mu\text{m}$ should be possible because the hole diameters of the microneedles presented in Figure 9, 10, 12, and 13 are typically less than $\varnothing 80 \mu\text{m}$. The needle wall thickness as low as 10 μm is observed (Figure 12).

In conclusion, the results presented in this paper have presented solid evidence on the feasibility of microneedle fabrication using the rapid microhole drilling technique presented in Tu *et al.* (2016). The critical factor for drilling microholes into a thin nitinol wire is the restoration of the semi-infinite material condition.

Acknowledgement: This investigation is supported by the Department of Mechanical and Aerospace Engineering at North Carolina State University, NSF Grants CMS-0402857, DMI-0355481, DMI-0355214, and DMI- 0944509.

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