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Silicon micro pyramidal syringe array for electrospinning spiral shape single fiber

Mahsa Madadi Masouleh¹, Reza Askari Moghadam¹@ and Javad Koohsorkhi¹@

¹ Advanced Micro and Nano Devices Lab., Faculty of New Sciences and Technologies, University of Tehran, Tehran, Iran
² Sensors and Actuators Lab., Faculty of New Sciences and Technologies, University of Tehran, Tehran, Iran
E-mail: r.askari@ur.ac.ir

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Abstract

In this study, a new method is presented to fabricate spiral shape single fiber. The micro-needle array (40 μm hole diameter, 80 μm outer diameter, and 100 μm height) is utilized instead of the needle to reduce the diameter of fibers which are electrospun from 23Wt% PVP concentration. In order to have fine and bead-free fibers, the structural parameters of the micro-needle array and space which close microneedles act as an individual one are simulated. The Wet and Dry Etching techniques are used for fabrication of micro-needles. The experimental setup consists of the stepper motor and micrometer head as the pump, distance controller, and voltage generator. The single fiber can be electrospun by applying 1–3 kV bias within 1 mm gap between micro-needle and collector. Using nonconductive collector causes the formation of spiral type single fiber instead of agglomerate fiber. Elastic and expulsion forces in charged fibers seem to be the main reasons of fiber separation and spiral shape formation. The spiral shape fiber is made without traditional lithography techniques like direct patterning or contact exposure which are more expensive and time-consuming. The alteration in the fiber pattern can be seen by changing the applied voltage and spinneret, microneedle and needle. After various experiments, spirality pattern electrospun by microneedle with 10–15 μm and 15–35 μm distribution area of first and second circles and 500–570 nm and 570–660 nm diameter of first and second circles is gained as the structure with minimum distribution area and fiber width. This structure is created when the applied voltage and distance between microneedle and collector are 1kV and 1 mm.

1. Introduction

Electrospinning is a beneficial process to fabricate fibers with diameters ranging from a few hundreds of nanometers to a few micrometer from polymer solutions at circumstance temperature and atmospheric pressure (Zheng et al 2010). Fibers have great characteristics such as smaller diameter, high surface-to-volume ratio and long length which are useful in various industrial and scientific fields such as filtration membranes, optical tools, bio-scaffolds and MEMS devices (Yang et al 2004, Lee et al 2007, Meechaisue et al 2007, Heikkilä et al 2008, Haider et al 2015). Conventional electrospinning process acts at high voltages above the tens of kilovolts and provides randomly coiled fibers because of the turning instability of fiber jet (Reneker et al 2000). Many parameters such as polymer concentration, solution viscosity and conductivity, the applied voltage, needle to collector distance, solution flow rate, needle diameter and temperature and humidity of environment affect on the electrospinning process (Haider et al 2015).

The smaller diameter of fibers is created by the small diameter capillaries (Andrady (2008)). Also, a smaller needle diameter ends up to the bead-free fibers and the beads are produced due to instability of the jet initiation (Mo et al 2004). So, small diameter needles are beneficial to electrospin fibers. Micro-electro-mechanical systems (MEMS) technology has been applied in many research fields. Micro-needle array has been fabricated using this technology. Micro-needles have found some applications in cancer treatment, transfer of DNA...
solution to the core, blood extraction, three-dimensional measurement and stimulation nervous signals (Aten et al 2008, Chuang et al 2010, Tsai et al 2011, Wang et al 2015, Kim et al 2016). Spinneret which is fabricated by MEMS technology is used in electrospinning process to form the fibers (Liivak et al 1998). Hollow needles are fabricated using different methods such as the combination of deep etching and rotating small angle deposition (Sanaee, Mohajerzadeh 2010).

The distribution area of the fibers is dependent on the collector material. For example, silicon surface has smaller distribution area in comparison to the SiO2 surface (Zheng et al 2008). Near-Field ElectroSpinning (NFES) has been used to achieve single fiber instead of randomly fibers by decreasing spinneret to collector distance and voltage in electrospinning (Hellmann et al 2009).

The rheological behaviour of polymers injected over fabrics has characterized by spiral moulds. Spiral bending capabilities can be proposed for fabricating microscale soft-robots. Energy harvesting can be done by spiral-like electrodes. Micro-pyro-electrospinning (μPES) method made fiber into spiral pattern for the first time. Also, spiral shape fiber was created without mask, lithography or direct printing which are expensive and time consuming. Spiral shape fiber was created in single step process for various applications such as cell patterning (Martinez et al 2011, Chen et al 2015, Paek et al 2015, Mecozzi et al 2016).

In this study, spinneret characteristics are defined based on electric field and electric potential simulation to get the soft and thin fibers. According to these results, microneedle with the pyramidal reservoir is introduced and fabricated using MEMS technology. Finally, the single fiber with spiral shape is produced by using electrohydrodynamic direct writing setup and controlling the parameters such as spinneret, distance, voltage and collector material. The proposed mechanism to achieve these ordered spiral structures is combination of dip-pen lithography, inkjet printing, and electrospinning without substrate movement.

2. Silicon micro pyramidal syringe

Figure 1 shows schematic of the experimental setup for positioning of spiral shape fiber by micro pyramidal syringe (consists of the pyramidal reservoir and micro-needle).

To optimize parameters for obtaining thin and bead-free fiber, the micro pyramidal syringe structure was simulated by Comsol® Ver 5.2 add-on AC/DC module. Here, all the structure supposed to be in the air sphere. At first, the geometries of the setup (such as micro-needle, collector and the electrode) were simulated by software and then the experimental setup dimensions, positions, and substance were selected from simulation results.

3. Simulation

In this section electrical field and electric potential are simulated for micro needle structure.

3.1. Electric field and electric potential simulation

The electric field and the electric potential of the electrospinning system at short distance and low applied voltage were examined using the finite element method (FEM). The configuration of a micro-needle spinneret system is shown in figure 2(a) getting suitable distance between close microneedles is important. As exhibited in
Figures 2(b) and (c) electric field y component (2D simulation) is shown on contour in the $1 \times 4$ microneedle array. It shows that if the space between adjacent microneedles is almost 1.5 mm, the microneedle operates as an independent one (figure 2(b)). However, if the distance between nozzles is 1 mm, the effect of adjacent microneedles will be seen (figure 2(c)). So, 1.5 mm considered as a suitable space between adjacent microneedles. Besides, as shown in figure 2(b) electric field at the microneedle tip is maximum and electrospinning occurs because of voltage difference between micro-needle tip and collector. The processing parameters are summarized in table 1.
Figure 3 shows the distribution of the electric potential on the surface. The maximum electric potential at the micro-needle is located on the micro-needle tip end because of micro-needle’s extruded structure as shown in figure 3(a).

The electric potential on the surface at the micro-needle tip versus height is displayed in the inset of figure 3(a) that a concentrated point of voltage is observed at center. The center region of the collector has smaller field intensity in contrast to the corners of the collector as shown in figure 3(b). The electric potential at the collector surface versus height in the micro-needle system exhibits in the inset of figure 3(b).

The electric potential (V) was applied to an aluminum sheet that micro-needle array was mounted on it. The surface that micro-needle was mounted on it and micro-needle tip were coated by 30 nm gold layer. Zero potential is applied to the copper sheet which holds the glass plate. The glass plate is the fiber collector. Taylor

Table 1. Processing parameters and micro-needle characterization for micro-needle spinneret electrospinning system and simulation.

<table>
<thead>
<tr>
<th>experimental setup</th>
<th>micro-needle spinneret</th>
<th>parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Applied voltage (V)</td>
<td>1000</td>
<td>v</td>
</tr>
<tr>
<td>The distance between micro-needle and collector (mm)</td>
<td>1</td>
<td>d</td>
</tr>
<tr>
<td>Length of micro-needle (μm)</td>
<td>100</td>
<td>l</td>
</tr>
<tr>
<td>Micro-needle hole outer radius (μm)</td>
<td>50</td>
<td>b</td>
</tr>
<tr>
<td>Micro-needle hole inner radius (μm)</td>
<td>20</td>
<td>a</td>
</tr>
</tbody>
</table>

Figure 3 shows the distribution of the electric potential on the surface. The maximum electric potential at the micro-needle is located on the micro-needle tip end because of micro-needle’s extruded structure as shown in figure 3(a).
cone is formed when the electric field force overcomes the surface tension and viscous force of polymer solution. So, a jet of polymer solution toward glass plate is created. As a result, fibers are put on the collector.

The electric field \( E \) can be calculated by the gradient of electric potential \( V \), as shown in (1).

\[
E = -\nabla V
\]  

(1)

The electric field has been simulated at different horizontal line which is parallel with micro-needle tip surface at various distance 0 (on the micro-needle surface), 10, 30, 50 \( \mu m \) (figure 4(a)) and also horizontal line at 70, 100, 300, 500, 700 and 1000 \( \mu m \) (on the collector) in the XY plane (figure 4(b)).

As exhibited in figure 4(a), the maximum electric field is observed at the edges of the micro-needle and the minimum value occurs at the hole of micro-needle along \( z = 0 \). Micro-needle tip is out of the surface and there is maximum electric field intensity around the tip. So, the polymer solution is stretched out from micro-needle to collector. As the distance between micro-needle and horizontal line increases, the electric field decreases slowly as shown in figure 4(b). The electric field value is constant and low on the collector. The fine and uniform fibers are created due to the equal distribution of electric field on the collector. So, distance causes different field strength.

3.2. Electric field and electric potential simulation for micro-needle design

Figure 5 displays the electric field along the \( z \)-axis in the central line considering various micro-needle hole radius, outer radius, and length. They are assumed 10, 20, 30 and 40 \( \mu m \) for micro-needle hole radius, 50, 100, 150 and 200 \( \mu m \) for micro-needle radius from the nozzle center to the outer nozzle and 25, 50, 75 and 100 \( \mu m \) for micro-needle length. When the distance from micro-needle increases, the results show that the field intensity decreases slowly but this value increases for the positions besides the micro-needle apex. In the end, fixed and low value electric field occurs on the collector. In the higher electric field in the regions near to micro-needle, better Taylor cone in the spinning path is occurred. Therefore, long jet direction and thinner fiber are displayed in the higher electric field. It is clear that the micro-needle which has the smaller hole, the smaller outer radius, and the larger length makes the larger electric field in the regions near to micro-needle as shown in figure 5.
produces a better Taylor cone in the spinning path. Also, it provides long jet direction and thinner fiber. So, it is better to use micro-needle with the smaller hole and outer radius and longer length. Due to the limitations of the fabrication process, a micro-needle with 20 \(\mu\)m hole radius, 40 \(\mu\)m outer radius, and the 100 \(\mu\)m length is used. So, effective parameters such as voltage, the distance between micro-needle and collector, length of the micro-needle, inner hole and outer radius of the micro-needle affect on the electric field and electric potential. In the end, the suitable conditions for fabricating micro-needle, as mentioned above, are followed to achieve the thin fiber without beads.

4. Micro-needle fabrication

In this study, hollow micro-needles are fabricated using the combination of Wet Etching and Deep Reactive Ion Etching (DRIE) of \(\langle 100 \rangle\) oriented, 510 \(\mu\)m thickness silicon wafer.
Metal micro-needles array requires complicated and expensive fabrication methods. The mechanical properties of silicon micro-needles, like high Young’s Modulus, and indentation hardness, are comparable to metal micro-needles. Since the fabrication process of silicon has been achieved precisely, silicon is selected as the microneedle material. In addition, silicon has a relatively high hardness value (Li et al 2019, Ma, Wu (2017), Lhernould 2013). The fabrication process of the micro-needle array is shown in figure 6. A solution of (NH₄OH: H₂O₂: H₂O, 1:1:5) at 75 °C temperature for 10 min is used for cleaning of the silicon wafer. Plasma Enhanced Chemical Vapor Deposition (PECVD) was applied to deposit a silicon nitride layer on to both sides of silicon with a thickness of 225 nm to protect silicon against wet etching. Chromium was deposited on to both sides of the wafer to a thickness of 160 nm by electron beam evaporation system to keep silicon nitride against Reactive Ion Etching (RIE) and DRIE. Then it is lithographically patterned (using Shipley photoresist) to get a 4 × 4 array of squares each gaging with 668 μm in width, where the center to center spacing between squares in each row and column is 1.5 mm. The photoresist is coated onto the other side of the wafer to protect chromium. Next, the exposed chromium layer and silicon nitride are removed by chromium etchant and Reactive Ion Etching, respectively. The acetone was used to remove photoresist.

Pyramid shaped reservoirs are fabricated on the wafer by KOH (30Wt%) heated to 80 °C. 54.7° tapered walls are built by wet etching along crystal plane. Whenever 410 μm of bulk silicon is etched, the wet etching process is stopped. As the silicon microneedle was fabricated using bulk micromachining process, its fragility was reduced. Pyramid shapes reservoirs cause the polymer solution to move as a stream flow toward the micro-needle tip. SEM image after wet etching is shown in figure 7. The photoresist is spun on the other side of the wafer. A second mask is aligned with silicon backside to pattern the micro-needles tips and output holes. This mask has the same 4 × 4 array of micro-needle with 40 μm inner diameter, 20 μm wall thickness, and 100 μm height for each one and 1.5 mm center to center distance of micro-needles.

The photoresist was developed with KOH. When exposed chromium layer was removed, silicon nitride and the silicon substrate are etched directly by Deep Reactive Ion Etching until silicon membranes are disappeared. So, hollow micro-needles with good surfaces are fabricated as shown in figure 8. The micro-needle reservoir and tip are fabricated in thick bulk silicon in order to provide tolerant and fixed structure. As the silicon microneedle

**Figure 6.** Schematic of the process to fabricate hollow micro-needles. (a) Silicon substrate. (b) Si₃N₄ deposition. (c) Cr deposition. (d) coating of Shipley photoresist. (e) UV exposure and development. (f) Cr etching, RIE, and Wet Etching. (g) Second mask lithographic patterning (backside alignment). (h) Turning over, Cr etching, and DRIE.
is fabricated using bulk micromachining process, its fragility is reduced. Also, it is possible to control its height, hole diameter, and outer diameter precisely. Silicon provides fabrication of small orifice microneedle.

The features of the outer walls can be controlled by the gases used during DRIE. The rise in the flow of SF₆ gas is the reason for slightly isotropic etching instead of anisotropic (Jiang 2006, Mehran et al 2011). The hole at the base of the microneedle is bigger than the hole of its tip due to the increase in fluoride ions. Therefore, the polymer solution movement toward the tip of the microneedle is more similar to laminar stream flow. Laminar flow would improve the uniformity of fibers and enhance repeatability of fiber production. Also, the microneedle wall thickness is assumed sufficiently large to avoid damaging cause of undercut during etching process. The microneedle wall specifications can be completely controlled by etchant gas concentration, RF power and time. It is possible to etch microneedle wall vertically through adjusting the gases utilized in DRIE.

In the development of high aspect ratio structures, one of the undesirable side effects of DRIE is the formation of grass. However, it can be reduced by controlling the effective parameters in the vertical etching process such as the gases used during etching and passivation (Mehran et al 2011). Since the grass on the silicon surface is homogeneous, the impact of them on the electric field uniformity is negligible (Steglich et al 2014). In addition, large length of micro needle can decrease the effects of surface roughness on electric field. Because of large microneedle height, 100 μm, the effect of surface roughness can be ignored. Due to the Cr deposition on the surface of the microneedle tip, roughness is eliminated on the tip which is the most important part for electric field generation.

5. Results and discussion

Different patterns such as spiral shape can be made by changing the effective parameters of the electrospinning process. Spiral shape fibers can be used at different applications. This pattern is achieved at low voltage and distance between spinneret and collector. Different Polyvinylpyrrolidone (PVP) concentrations have been investigated and the fibers without beads were achieved by 23 Wt% concentration of PVP. So, this concentration is used in all tests, in this paper.
5.1. Preparation of materials
Polyvinylpyrrolidone (PVP) (MW = 1,300,000, Merck) solution was provided by dissolving PVP powder in methanol to prepare a solution with a 23 Wt% concentration pursued by stirring for 5 min at about 30 °C. The polymer solution should have proper concentration and viscosity. The acceptable concentration is achieved whenever the micro-needles do not clog and fibers are spun well.

5.2. Experimental setup
Figure 9 shows the experimental setup. It consists of a glass that mounted on a copper sheet as the collector, 0.5 ml plastic syringe, micro-needle array (micro-needle with inner diameter 40 μm and outer diameter 80 μm), stepper motor, micrometer head, distance controller and the voltage generator. The whole setup was placed perpendicular to the collector.

The micro-needle array and needle as spinneret were used in this setup. As mentioned before, the gold has been deposited on micro-needle array. So, gold is deposited on the tip of the microneedle, the outer wall of the microneedle, and the silicon surface. The positive charge of the power supply can be transferred to the tip of the micro-needles. In addition, gold makes the microneedle stronger and more resistant to aging. The micro-needle array is attached to an aluminum sheet (an electrode to be connected to the voltage generator). A glass mounted on the copper plane is used to collect fibers. The aluminum sheet is straddled on a needle as a polymer solution source. The polymer solution is forced out by micrometer head which is attached to a stepper motor. The flow rate is controlled by stepper motor shaft rotation. Applied force by stepper motor causes the drops formation of the polymer solution at the spinneret tip. Taylor cone is formed by applying a voltage between the spinneret and the copper surface. A jet of polymer solution toward collector is made when the electric field overcomes the surface tension of the solution.

At last, fiber samples are gold deposited to decrease the charging effect. Then, the gathered fibers are characterized using a scanning electron microscope (SEM). The spinneret characteristics, the collector conductivity, the distance between spinneret and collector, and applied voltage are the important parameters which affect on the electric field, the distance between fibers, and the diameter and shape of fibers.

5.3. Results
Micro-needle array was fabricated based on simulated results. Micro-needle with 40 μm hole diameter is used instead of the needle to create smaller droplet in order to achieve the thin fiber without beads. The polymer solution was conducted with 0.1 ml hr⁻¹ flow rate toward the spinneret tip. The ordered single fiber instead of buckling fibers was made by shortening voltage and the distance between spinneret and collector based on near-field electrospinning (NFES) method. In this study, the micro-needle array or needle unlike the NFES method are used instead of the probe for electrospinning. The base of Taylor cone is dependent to the spinneret hole radius. Therefore diameter, softness and uniformity of fibers are improved when micro-needle is used instead of needle or probe. So, the thinner single fiber without beads in the low distance would be resulted by the micro-needle.

The electrical field will be changed when the glass is placed on the copper sheet as the collector.
Therefore, the behavior of electrospinning changes because of the abnormal ground at the collector. If the collector is not conductive, the charged fiber can not move rapidly and the expulsion force causes the fibers to be collected as spirally pattern on the collector. Expulsion force of charges and elongation of surface tension affect on the electrospinning process and the fiber patterns. The first point of this pattern is the junction between spinneret tip axis and glass substrate and then the continuous single fiber turns around the initial point spiral. Spiral shape single fiber on glass is observed when fibers are placed away from previously spun fibers. So, the distance between fibers on the insulative collector is more than the conductive collector. Therefore, the width of the distribution area is raised because of the expulsion force of charges on fibers and the elastic force. Also, charged fibers are dragged more because of the repulsion force on the dielectric collector. So, the diameter of fibers is diminished on the glass. Diameter and deviation of fibers are impressed by the electric field and spinneret specifications. Figure 10 shows the definition of first and second circles and their diameters in spiral shape.

5.3.1. Effect of the applied voltage on the electrospun structures by microneedle
As shown in figure 11 spiral shapes electrospun by a micro-needle array don’t effect on each other because the distance between two close micro-needles is defined based on simulation results.

Table 2 shows that various structures by microneedle can be deposited by changing the applied voltage at the distance of 1 mm. When the voltage is 5 kV, the fibers on the collector twist together disorderly. When the applied voltage is decreased to 4 kV, the twisted fibers decrease and single fiber begins to appear, but fibers still have irregular shapes. Table 2 demonstrates single wavy spiral structures that wavy shape disappear when continuously decreasing the voltage to 2 kV. Finally, when the voltage is 1 kV, the single nanofiber with the decreased distance between fibers and fiber width is electrospun. In addition,
Figure 11. Spiral shapes produced by a micro-needle array. Applied voltage is 1 kV. Distance between collector and spinneret and concentration are 1 mm and 23Wt%.

Table 2. Structures produced by micro-needle. Under the same conditions (distance between collector and spinneret = 1 mm, concentration = 23Wt%).

<table>
<thead>
<tr>
<th>Structures</th>
<th>Voltage (kV)</th>
<th>5</th>
<th>4</th>
<th>3</th>
<th>2</th>
<th>1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distribution area of first circle (μm)</td>
<td>—</td>
<td>—</td>
<td>90–200</td>
<td>20–30</td>
<td>10–15</td>
<td></td>
</tr>
<tr>
<td>Distribution area of second circle (μm)</td>
<td>—</td>
<td>—</td>
<td>200–310</td>
<td>30–60</td>
<td>15–35</td>
<td></td>
</tr>
<tr>
<td>Diameter of first circle (nm)</td>
<td>—</td>
<td>—</td>
<td>820–2800</td>
<td>650–860</td>
<td>500–570</td>
<td></td>
</tr>
<tr>
<td>Diameter of second circle (nm)</td>
<td>—</td>
<td>—</td>
<td>2800–3500</td>
<td>860–1600</td>
<td>570–660</td>
<td></td>
</tr>
</tbody>
</table>

Table 3. Structures produced by needle. Under the same conditions (distance between collector and spinneret = 1 mm, concentration = 23Wt%).

<table>
<thead>
<tr>
<th>Structures</th>
<th>Voltage (kV)</th>
<th>2</th>
<th>1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distribution area of first circle (μm)</td>
<td>240–300</td>
<td>60–100</td>
<td></td>
</tr>
<tr>
<td>Distribution area of second circle (μm)</td>
<td>300–440</td>
<td>100–220</td>
<td></td>
</tr>
<tr>
<td>Diameter of first circle (nm)</td>
<td>2400–3000</td>
<td>2300–2700</td>
<td></td>
</tr>
<tr>
<td>Diameter of second circle (nm)</td>
<td>3000–3600</td>
<td>2700–3300</td>
<td></td>
</tr>
</tbody>
</table>

since the speed of the exhaust jet grows up when the voltage between spinneret and collector is increased, distance between fibers and the fiber width are increased. The voltage increment causes a reduction of the stretch time.
5.3.2. Effect of the hole radius of spinneret on the spiral shapes
Minimum required voltage to have Taylor cone at the distance of 1 mm is 1 kV. As exhibited in tables 2 and 3, since the hole radius of micro-needle (40 μm hole diameter, 80 μm outer diameter and 100 μm height) is less than the needle (133 μm hole diameter and 360 μm outer diameter), distance between fibers and the fiber width are decreased by microneedle. According to our results, by using microneedle, the spiral shape with 10–15 μm and 15–35 μm distribution area of first and second circles and 500–570 nm and 570–660 nm diameter of first and second circles is achieved. By using the needle, these values are in the range of 60–100 μm, 100–220 μm, 2300–2700 nm, and 2700–3300 nm respectively. These fibers are achieved when the spinneret is located 1 mm far from the collector and the voltage between spinneret and substrate is 1 kV. Also, the fiber width and distance between fibers are grown from the initial point of the spiral shape toward the end.

6. Conclusion
In this paper, a new method is introduced to produce spiral shape fibers by changing effective parameters on the electrospinning. The different spiral shapes are achieved by the mask free and single step approach. Better fibers and Taylor cone are achieved by smaller inner and outer radius of the micro-needle hole and taller length of the micro-needle fiber. The inner and outer radius of the micro-needle and the length of the micro-needle are chosen 20, 40 and 100 μm, respectively. In presented experimental setup, solution flow rate, the spinneret, voltage and distance between the spinneret and collector are adjusted to produce small diameter and different shape fibers. The spiral templates of the single fiber from 23Wt% concentration PVP solution are formed because of the nonconductive fixed collector, elastic force and expulsion force of charges on fibers. The distance between fibers and the fiber width of the spiral shapes are decreased by reducing voltage and using the micro-needle instead of the needle. In the applied voltage 1 kV and distance between spinneret and collector 1 mm, the micro-needle created the spiral pattern which the distribution area of first and second circles and diameter of first and second circles are in the range of 10–15 μm, 15–35 μm, 500–570 nm, and 570–660 nm, while these values are in the range of 60–100 μm, 100–220 μm, 2300–2700 nm, and 2700–3300 nm, if the needle is used at the same conditions.

ORCID iDs
Reza Askari Moghadam @ https://orcid.org/0000-0001-8394-7256
Javad Koohsorkhi @ https://orcid.org/0000-0001-6527-0800

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