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Production of Pyrex coated metal micro-wires from a preform and its applications

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Production of Pyrex Coated Metal Micro-Wires from a Preform and Its Applications

A thesis submitted in partial satisfaction of the
requirements for the degree Masters
in Mechanical Engineering

by

Suhrud Ghatpande

2020

ABSTRACT

The objective of this thesis research was to conduct a feasibility study on a new process for scalable production of Pyrex-coated metal microwires from a preform, namely Vacuum Stabilized Thermal Drawing (VSTD).

Existing methods for production of glass coated metal microwires suffer from lack of an effective method to control the flow rate of the molten metal during process, which results in poor diameter control and susceptibility to breakage due to capillary fluid instability. Chapter 1 gives introduction to existing methods and summarizes their drawbacks. VSTD process tackles fluid instability problem and helps in achieving flow rate control of the molten metal core via imposing volume conservation facilitated by introducing a negative pressure difference between the atmosphere and the induced vacuum inside the preform. For the purpose of feasibility study, process was carried out in two major steps. First step was to fabricate a pure consolidated preform by using principle of zone melting. A tin rod was inserted into a glass tube with one end sealed and vacuum attached to the other end, and passed through inductance furnace in multiple cycles to obtain a pure consolidated preform. Second step was to draw this preform into a Pyrex-coated metal microwire using a novel method called VSTD. For preform drawing, first a tower was built using aluminum extrusions and assembling drawing system, feeding system and furnace onto it. Later, the parameters such as the feeding speed, drawing speed, and furnace temperature range were first determined by experience, and then using these parameters preform was passed through the furnace with vacuum stabilization to control the molten metal flow rate. This resulted in

scalable preform drawing into microwire with uniform clad to core diameter ratio. Chapter 2 discusses about the experimental procedure for preform manufacturing and VSTD process and later gives mechanism summary in the sub section. The key results and characteristics of microwires are then revealed in Chapter 3, to show experimental validation of flow rate control, feasibility of the process, and importance of vacuum stabilization in microwire drawing.

In Chapter 4, niche applications of glass-coated metal microwire are demonstrated. For example, microwires derived from VSTD process can be used for producing standard-sized glass-coated nanoparticles. Another possibility of using this microwire is in transformers. VSTD process allows to manufacture of a wire which is equal to skin depth, which can result in high conductance in transformer wires. This method is also suitable for producing other multi-material microwires and has numerous benefits over existing methods. Chapter 5 concludes this research study and discusses future scope of this process.

ACKNOWLEDGEMENTS

I would like to start by thanking my advisor, Dr. Jingzhou Zhao for considering me as his research assistant as well as supporting and guiding me throughout my Master's degree. Without his constant encouragement and advice, this work would not have been possible.

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CHAPTER 1. INTRODUCTION

1.1 Background and Motivation

Glass coated metal microwires are very thin metal wires, coated with glass for extra protection and for obtaining other useful electrical, mechanical, and thermal properties. Glass coating helps dissipate excess heat generated by current flowing through the microwire, which increases the ampacity of the microwire. Pyrex-coated tin microwires are made here in Western New England's Advanced Manufacturing Laboratory using Tin as core material while Pyrex as coating material for this research study, however, they can be made using different core materials along with different coating materials depending on their coefficient of expansion, viscosity, and process parameters.

Glass coated metal microwires find numerous applications in electronics industries as well as in some niche medical industries. The small diameters and glass coating of these microwires make them desirable to use for high current and high frequency power transmission in harsh environments. Their magnetic properties also find applications in many sensory devices such as security tags, anti-theft badges, etc. Furthermore, recent laboratory study show promise of using these microwires as feedstock for the scalable production of surface functionalized inorganic nanoparticles (e.g. silica-coated metal nanoparticles) for targeted drug delivery and image enhancement agent. It can thus be said that these microwires have significance across multiple industries.

Preform drawing is a scalable manufacturing method which holds promise for industrial scale production of continuous ultra-thin glass-coated metal microwires. The process is performed in three sequential steps, namely preform feeding, melting under high temperature, and fiber pulling. Taylor-Ulitovski first introduced the thermal drawing method for metal fiber in 1924 [1], after which the process significantly evolved over the period of time and good quality low-cost microwires became possible to manufacture. Despite many advances in manufacturing techniques, there are many limitations such as achieving flow rate control, continuous microwire drawing, and industrial-scale production, that are yet to be addressed.

Figure 1.1 shows a thermal drawing tower. Assembly of thermal drawing tower is done using aluminum extrusions and a set of brackets. Brackets are bolted to the frame to give structural rigidity to the tower. Thermal drawing tower has three main components, namely a feeding system, high temperature furnace, and a tractor pulling system. These three components are assembled sequentially in the vertical direction with feeding system at the top, high-temperature furnace in the middle, and tractor pulling system at the bottom. The conventional preform drawing process involves attaching a preform to the chuck and feeding it into the furnace by using a feeding system assembly of support rods, a lead screw, stepper motor, and a base plate. Here, the stepper motor controls the motion of lead screw while furnace heats the glass cladded metal core preform to a sufficiently high temperature (>850°C), which is also above its glass transition temperature resulting in the necking of the preform. As the bottom portion of the preform softens, it starts flowing downwards due to gravity and re-solidify after exiting the furnace. In the magnified furnace section view, the necking of the preform can be seen. The preform is fed into the furnace with a predetermined speed V_p and the microwire or fiber coming out of the furnace is pulled with speed V_f using tractor pulling system. Furnace temperature can be controlled, and different temperature values can be set, depending upon materials passing through it. The fiber diameter can be controlled by varying feeding and pulling speed parameters.

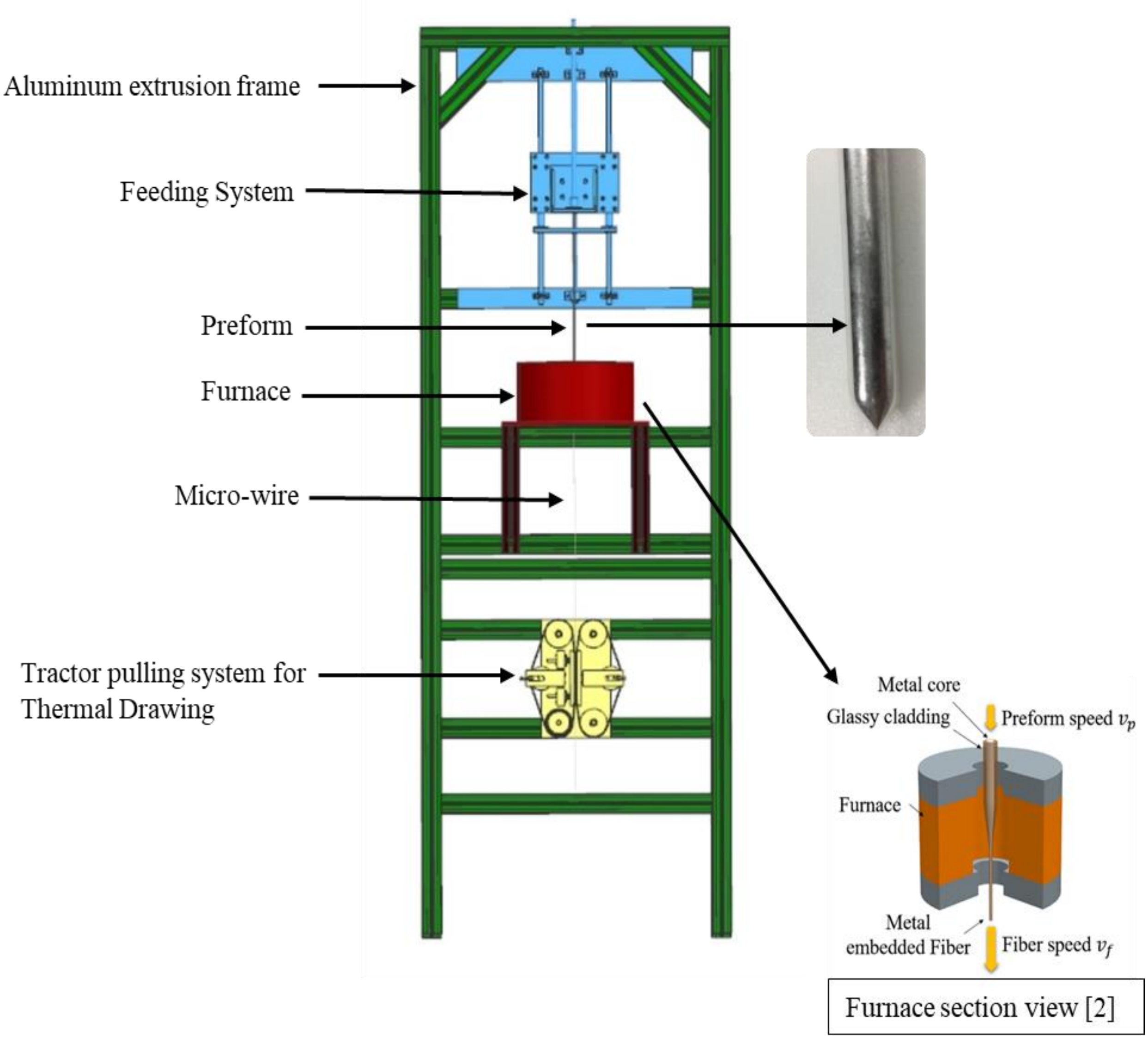

Figure 1.1 Thermal drawing tower

Fiber pulling speed is determined by using the law of mass conservation at steady state as per equation (1)

$$\frac{V_p}{V_f} = \frac{{D_f}^2}{{D_p}^2} \tag{1}$$

where,

D_f = Diameter of fiber D_p = Diameter of Preform

V_p = Preform feeding Speed V_f = Fiber Pulling speed

The main challenge of the conventional method for producing glass-coated metal fibers is the control of molten metal flow rate, which further causes difficulty in controlling wire diameter, core diameter, and cladding thickness. Moreover, there is no clear understanding regarding the mechanics behind the uniformity in microwire clad-core diameter ratio, solid-liquid boundary layer formation, and its breakage at the interface. So far, no one has studied the effect of pressure difference on core discontinuity, as well as the significance of stable pendant drop. Also, due to the inherent limitations of this method, there are restrictions over preform and metal core material combinations. For example, silica-coated copper microwire can be a very robust and highly conductive as silica can provide extra strength while copper has higher conductivity, but the manufacturing of such a microwire is challenging due to high temperature requirements. The current process allows the drawing of metal microwires only when metal core melting point is lower than its cladding (glass) softening temperature. Hence limited combinations of metal microwires like gold, silver, iron, copper, and platinum with borosilicate glass as cladding material are successfully drawn today. Diameter ranging from 1 to 120 micro-meter scale is successfully produced with the current method, but further reduction of diameter into nanoscale has not been successful.

This research aims to demonstrate the flow rate control over molten metal inside capillary using vacuum stabilization for continuous microwire production. Further, this research explains how the process is scalable and can be used in industrial-scale production. Current methods have limitations in achieving flow rate control with susceptibility to breakage; hence, these methods are challenging to be used for industrial-scale production. A novel approach using vacuum stabilization is introduced to show potential solution to the above problems. This method is discussed in section 2.2.

1.2 Literature Review on Different Flow Control Methods for Preform Drawing

Metal flow control during preform drawing plays a pivotal role in determining the overall characteristics of the microwire. Better control over the flow rate better is the control over microwire diameter and its continuity. Since the Taylor-Ulitovski method, numerous other methods were introduced for microwire drawing. This section summarizes how flow control is achieved during preform drawing by different methods.

1.2.1 Metal flow control by separation of core and cladding

This method is proposed by WMT machine technologies limited. It aims to separate the melting of core and glass materials in two separate crucibles, which enables metal and glass temperatures to be controlled independently hence allowing the manufacturing of more combinations of glass cladding and core materials. Here, two crucibles are vertically aligned with each other and used for heating core material separately by the first electromagnetic inductor in one crucible to its melting point and glass by the second electromagnetic inductor in a second crucible to its drawing temperature. This process allows molten core material to flow from the first crucible into the second crucible, where molten metal combines with softened glass, forming glass-coated wire. This wire can be drawn and processed for continuous production. In this way indirect control over metal flow is achieved. This method can be done in various configurations like continuous feeding of core and glass material in first and second crucible respectively or by feeding core material in limited quantity in the form of a rod, a bar or wire. If the melting point of the core material is close to the softening point of glass material, the core material may be melted in the second crucible. Later, core material and softened glass can be combined to form glass-coated wire. [3]

Core materials with melting points as low as 327 ºC (lead) to as high as 1769 ºC (platinum) can be fed into the crucibles. The device can be shielded with a heat resistant metal or a heat resistant ceramic. This device can accommodate different core materials like metal, alloy, a semiconductor, ceramic powder, etc. Glass material can be fed in different forms like glass powder, a glass ball or a glass tube. Glass material can consist of alkali silicate, borosilicate, aluminosilicate, quartz, silica, soda-lime, lead, etc. [3]

This method is versatile, where it can accept various forms of metal core and glass materials; hence, multi-material microwires can be produced. Metals with high melting points can be fed into the furnace hence enabling drawing of heat resistant microwires. The disadvantage of this method is no direct control over molten metal flow, as there is no formation of stable pendant drop, which consequently leads to unstable drawing. This phenomenon results in having difficulty in achieving diameter control. The process is also multistep and time-consuming.

Figure 1.2 shows the schematic of this process:

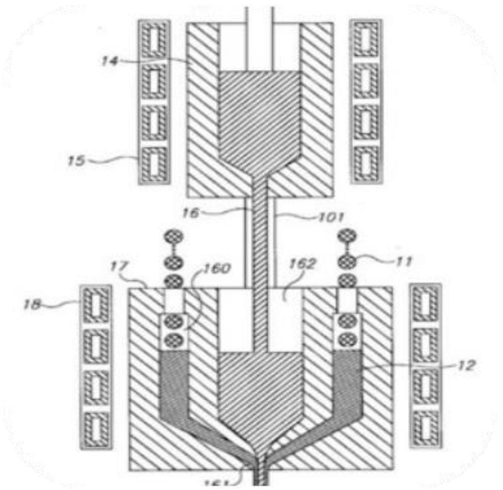

Figure 1.2 Metal flow control by separation of core and cladding [3]

1.2.2 Induction floated molten metal pool

This process uses the principle of induction heating to melt the metal core to a very high temperature. Induction heating only affects conductive materials, which also keeps glass unaffected. As a result, molten metal inside the preform floats at the bottom and gets stabilized. After the stabilized molten metal reaches sufficiently high temperature, glass starts necking from inside. A uniform fiber is obtained after this softened glass is drawn using a pulling system. Figure 1.3 shows the schematic of the process. There are various methods to control metal core and glass coating diameter. One method to control the metal core diameter is by varying the take-up speed of the microwire and cooling rate. The cooling rate can be increased by passing microwire through air or liquid jet. Also, glass thickness can be controlled by controlling the feeding rate of the glass tube in the furnace [4,5]. This method can be further made available for high dimensional precision commercial production of microwire. Electrical resistance can be monitored during the preform drawing process and if any change in resistance is noticed, it can be

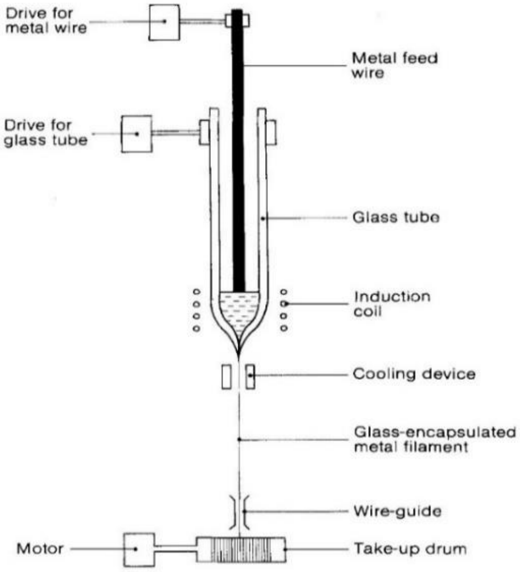

Figure 1.3 Induction floated molten metal pool [4]

fed back to alter process parameters using electrical circuity and hence, dimensional tolerancing can be achieved over microwire diameter. [4,6-8].

Some drawbacks of this process are a limitation to conductive materials, and ultrathin coating cannot be achieved on metal microwire. Also, only limited combinations of cladding and core materials can be drawn, as the difference between glass softening temperature and metal melting point cannot be too high.

1.2.3 Solid fiber coating

In this method, the metal core is coated by running a liquid stream of cladding through a die. Flow control can be easily achieved with the help of this method. Here, the polymer melt is poured vertically onto a bare wire and it is then extruded through a die. Uniform solid coated fiber is obtained after extrusion [9]. Figure 1.4 shows the schematic of the process. By this process, ultrathin coating cannot be achieved as the coating may become porous. For successful drawing, metal core's melting point needs to be higher than the coating. Also, this process is a multistep process hence time-consuming.

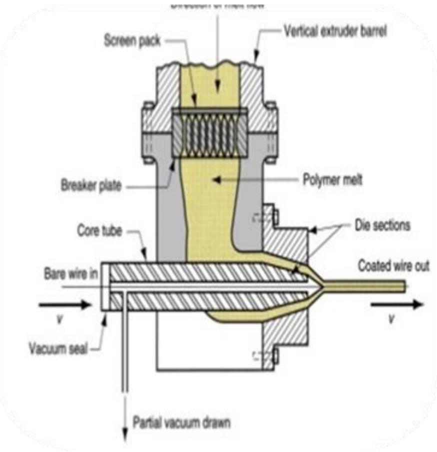

Figure 1.4 Solid fiber coating [9]

1.3 Literature Review on Different Methods of Preform Fabrication

A preform is a consolidated rod consisting of metal core and glass cladding. This rod is used as a feedstock material for the drawing of microwires. Depending on the preform manufacturing process, preform glass tubes can contain metal core in various shapes and forms. Preforms can also vary in length, which can drive the total drawing length of the continuous microwire. Microwire characteristics are based on the type of preform used as well as how well the preform is manufactured. This section aims to cover different Preform manufacturing methods.

1.3.1 Rod in tube method

The process is straightforward, a rod is inserted into a glass tube to obtain a preform. Metal can be inserted in glass tube in various forms such as powder, solid rod, semi-liquid, etc. Versatility of inserting material into glass tube enables a wide range of materials to be used as core materials. Silica-clad fiber with soft-glass core was successfully produced by this method [10,11]. Figure 1.5 shows the schematic of the process. The advantage of this method is that, various material combinations can be used to manufacture preform and this multi-material preform can be used to draw microwire. Hence, microwire can have different properties due to different core and cladding combinations. The disadvantage of this method is that the obtained preform is porous due to air trapping, inherent porosity, and impurity present in the core material, and hence, microwires drawn from this preform too are porous. Moreover, drawing microwires from this preform would not be continuous.

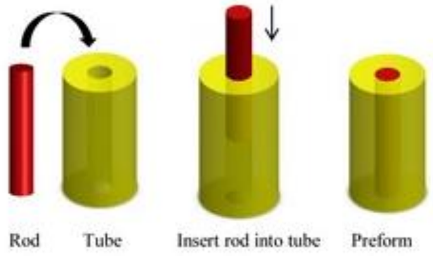

Figure 1.5 Rod in tube method [10]

1.3.2 Extrusion Method

Extrusion is a process used to create objects with a fixed cross-sectional profile by pushing a soft material through a die under pressure. Material in the form of a rod, typically called a billet, is placed inside a sleeve held in a furnace. The billet is heated to the softening temperature of the material, and pressure is applied to push the material through a die that imparts shape to the extruded preform rod, which is subsequently drawn into a fiber [10]. Figure 1.6 shows the schematic of the process.

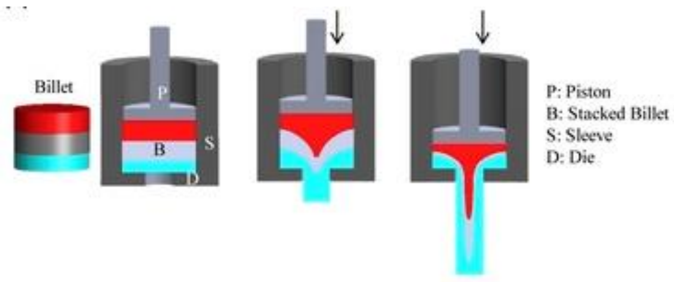

Figure 1.6 Extrusion method [10]

1.3.3 Stack and draw method

Rods, tubes, and or plates from a single or multiple material may be assembled into a preform with dimensions determined by the targeted fiber structure. Canes can be stacked together and assembled into a jacket to form a preform. Multiple stack-and-draw steps may be applied recursively to reach the required dimensions and attain complex transverse structures [10]. Figure 1.7 shows the schematic of the process.

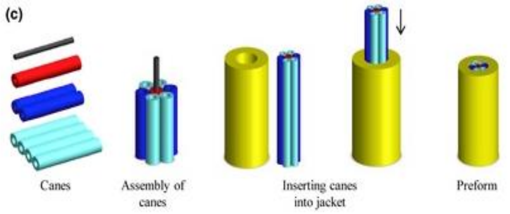

Figure 1.7 Stack and draw method [10]

1.3.4 Thin film rolling

This process incorporates a polymer in a preform through rolling a thin polymer film followed by thermal consolidation under vacuum above the glass transition temperature of the constituent materials until the individual films fuse. The schematic of the process is shown in the figure 1.8 [10]. Impurity, porosity, and discontinuity are the major drawbacks of this process.

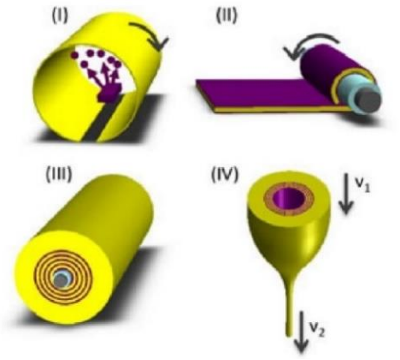

Figure 1.8 Thin film rolling method [10]

1.3.5 Problems with Existing Preform Fabrication Methods

Most of the current preform fabrication methods have drawbacks, which results in impure and porous preform. When such a porous preform is used for preform drawing, the microwires would also be porous and may result in discontinuity. This research aims to overcome these drawbacks by using preform consolidation under vacuum with zone melting. Defects such as porosity and air trapping are eliminated by a vacuum, while zone melting ensures impurity in metal is entierly separated from pure metal, and evenually a well-consolidated glass coated metal preform can be obtained via this process.

1.3.6 Preform consolidation using vacuum with zone melting (Used in this research study)

This method eliminates many of the drawbacks, which most of the other existing methods encounter. In this method, a vacuum is attached at the top of the preform which is steadily fed into the furnace. As preform traverses through the furnace, zone melting takes place, collecting all the impurity in the at the top side while leaving purer metal at the bottom of the tube for drawing. Vacuum pressure removes air pockets or gaps present in the preform. This method results in obtaining highly pure, non-porous, and homogeneous preforms.

1.4 Problems with Existing Thermal Drawing Methods

Existing preform drawing methods also possess various challenges for scalable production of crystalline core microwires. One of the common problems is to have better flow rate control over the molten metal. During the process, when the preform is heated in the furnace, the temperatures reach up to 870 °C. Such high temperatures render conventional flow systems like control valves, flow switches, and pumps useless. Moreover, molten metal has very low viscosity and low resistance between its core and cladding, making flow control even more difficult. Further, molten metal has very high surface tension, which causes a bubble-like shape to form at the tip of the preform called a pendant drop. Gravity causes this pendant drop to get pinched-off easily, making stable wire drawing improbable. All these factors result in great difficulty in having control over primary parameters of preform drawing such as wire diameter, core diameter, and coating thickness. Existing methods also have other challenges in achieving large scale production, continuous preform drawing, and scalability.

In this research, vacuum-stabilized thermal drawing is introduced and implemented to overcome the above challenges. By this method, control over molten metal flow can be achieved easily, which further enables control over microwire diameter, continuous preform drawing, and obtaining ultra-thin coating. Having continuity in preform drawing makes large-scale production and scalability a real possibility.

CHAPTER 2. EXPERIMENTAL PROCEDURES

2.1 Preform Fabrication Process

The method used in this study is called as preform consolidation using vacuum with zone melting.

Experimental procedure:

The goal was to fabricate a solid consolidated preform of required diameter, which later could be drawn into long continuous microwires. A glass tube of 6mm inner diameter and a tin rod of 1.5mm diameter were used in this experiment as cladding and core materials, respectively. First, the glass tube was cleaned from inside with isopropyl alcohol to dissolve any residual oil or other unwanted particles and made sure the surface was smooth for molten metal to flow freely. After that, one end of the glass tube was sealed using a micro-flame and a tin rod was inserted from the other end. Figure 2.1 shows the schematic of the process. This assembly was fed into the furnace with a vacuum attached at the top of the tube for fabricating a consolidated preform.

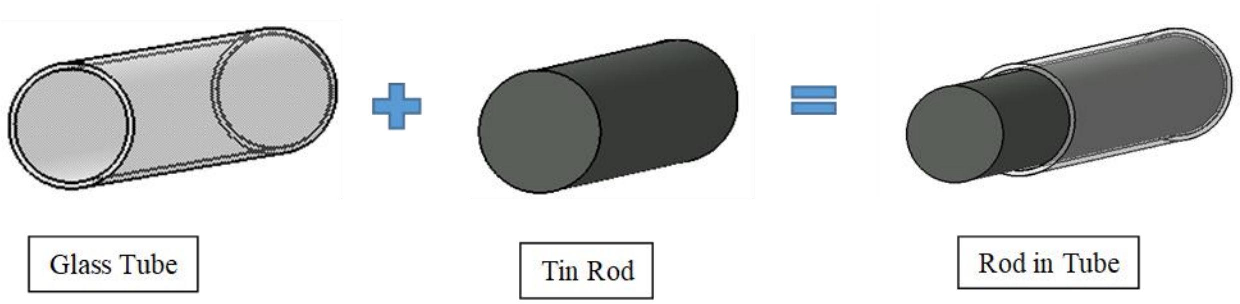

Figure 2.1 Preform fabrication process

Process Parameters:

It was necessary to determine optimal preform feeding speed to obtain the required diameter with continuous drawing. After many trials and errors, feeding speed was selected to be 50µm/s. Afterward, it was necessary to select furnace temperature, which would be just sufficient to melt tin rod but should also be less than glass transition temperature to avoid glass melting; hence, 300°C was selected as furnace temperature.

Zone melting:

Zone melting is a method for obtaining a highly purified form of metal by passing the metal rod through an inductance furnace. As the rod passes through the furnace the thin region around it starts melting. This molten region liquefies the impure metal at its forward edge leaving behind pure solidified metal as it moves through the glass tube [14].

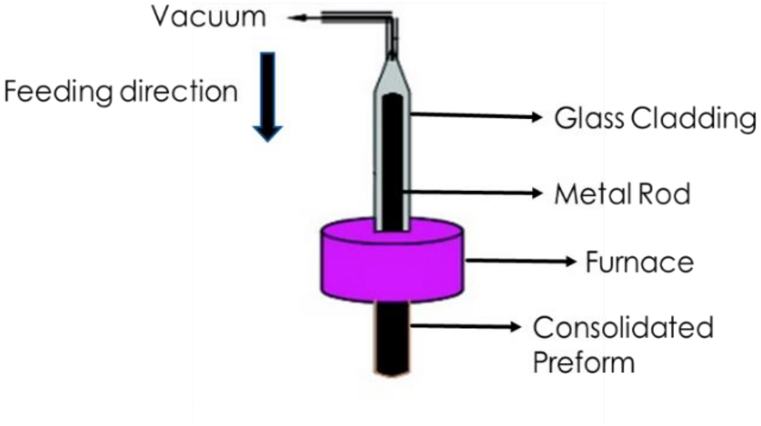

Figure 2.2 Schematic showing zone melting process [12]

This process is carried out by feeding the glass tube containing metal rod into the furnace in the downward direction, as shown in the figure 2.2. As it passes through the furnace, the metal rod starts melting and the impurities present in the metal rod float at the top side of the tube; the vacuum is introduced to minimize air pockets or gaps for obtaining pure metal at the forward edge.

After determining process parameters and setting up the thermal drawing tower, the zone melting process was carried out. The preform had to undergo four runs meaning the preform was heated along its length in four different cycles starting from bottom to the top. After the zone melting process was completed, a consolidated highly pure tin core preform was obtained. Figure 2.3 shows a consolidated preform.

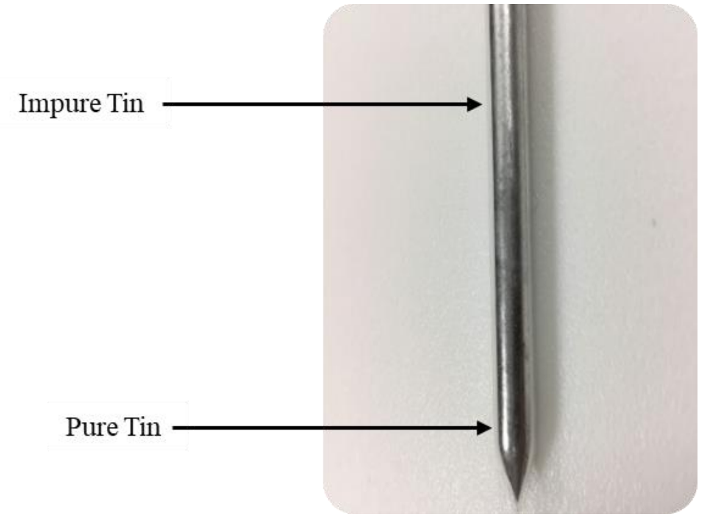

Figure 2.3 Consolidated preform

As can be seen from the figure 2.3, the pure tin is collected at the forward edge while the impure tin gets collected on the rear edge of the tube. Also, the metallic tin is completely adhered to the inner walls of the tube after the melting and cooling process is over, leaving no gap. This preform sample was considered ideal for microwire drawing and was later used for the same.

2.2 Vacuum Stabilized Thermal Drawing Process

The method of vacuum stabilized thermal drawing is introduced for the first time for producing microwires. Process involves creating resistance to the flow with the use of a vacuum. Figure 2.4. shows the process with a vacuum attached at the top.

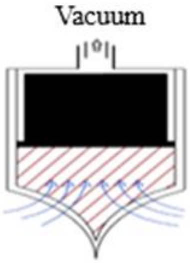

Figure 2.4 Vacuum stabilized molten metal pool

Vacuum helps to create pressure difference and in turn, controls the flow of molten metal. This method is used in this experiment as it outweighs other methods by having numerous advantages, this process is scalable, simple to control using mass conversion, it is not limited to conductive materials, has potential to achieve ultrathin coating, potential to achieve core to diameter ratio less than 1.2, which no technology has achieved yet as well as cladding material can have higher melting point than core material implying better protection against extreme conditions such as high temperature and corrosion.

The method is explained in details following figure 2.5.

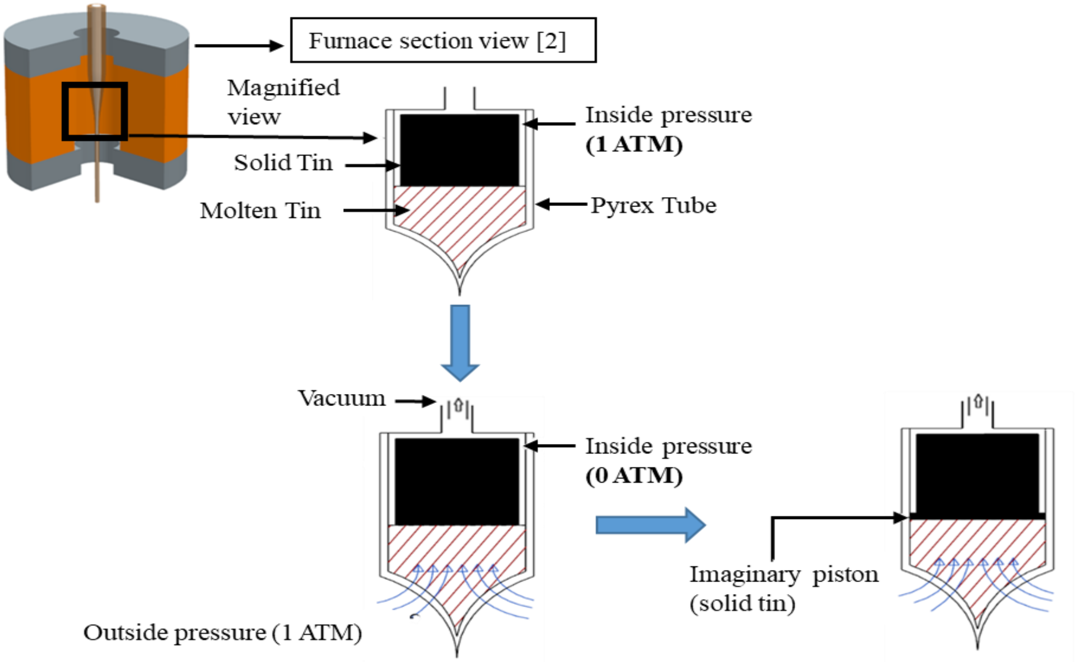

Figure 2.5 Schematic of VSTD process

VSTD is the modification of the conventional thermal drawing process. It is done by attaching a hose coming out of the vacuum pump to the top of the preform. A sealant was used to secure the connection, and other measures were taken to ensure there were no leakages. After securing connection, the preform was fed into the furnace using the feeding system at a constant speed. Vacuum enables to create a pressure difference between the preform and the atmosphere. This pressure difference plays a significant role in controlling the flow of the metal. Also, due to the difference in coefficient of expansion of core and cladding material, while cooling metal shrinks faster than glass forming a small gap inside the preform. A vacuum pressure is applied to this gap. As the preform is being fed into the furnace, metal core starts melting along with the glass. External pressure exerts an upward force on the molten metal, which in turn exerts counterbalancing force to balance the negative pressure, thus forming a thick solid layer of tin acting as an imaginary piston. This piston stabilizes the melt front and enables flow rate control. Self-terminated balance between negative pressures induces upward force and the counter forces. Solid layer thickness of the imaginary piston determines the amount of counter force needed to balance the negative pressure. The stabilized melt front was then pulled using the tractor pulling system at a constant rate to obtain long continuous microwire.

2.2.1 Mechanism Summary

Applying a vacuum creates a pressure difference between preform (inside pressure) and atmospheric pressure. This pressure difference enables the formation of an imaginary solid piston due to negative pressure exerted on molten metal. Solid piston stabilizes the melt front, which in turn prevents molten metal pool from flowing freely. This phenomenon allows the tractor pulling mechanism to have complete control over drawing of microwire from the molten metal pool.

By law of mass conservation as per equation (1) desired microwire diameter can be calculated.

Equation also implies higher the pulling speed thinner is the diameter and vice-a-versa.

Table 1.1 shows process parameters used for preform drawing (420 µm fiber diameter). Figures 2.6 and 2.7 show preform after drawing process.

Feeding Speed	20µm/s
Drawing Speed	7250µm/s
Temperature	870 °C

Table 2.1 Process parameters

Figure 2.6 Failed preform drawing

Figure 2.7 Successful preform drawing

Initially, the preform drawing was performed without vacuum stabilization. The result can be seen from figure 2.6, where the metal core gets completely separated from its front edge during preform drawing. Separation of the metal core made preform drawing discontinuous and unsuccessful. Lack of pressure difference made the pendant drop unstable and separated itself from the metal core during the drawing process. Flow rate control of molten metal was the key to stabilize pendant drop to have a continuous preform drawing. For this purpose, a vacuum was introduced at the top of the preform to create a pressure difference. Figure 2.7 shows preform after vacuum stabilized drawing. Here, the metal core remains intact during the drawing process due to the negative pressure difference between the induced vacuum and the atmosphere. Negative pressure difference facilitated flow rate control over molten metal, which stabilized pendant drop and prevented metal core separation during the process. Hence, applying vacuum made preform drawing consistent and enabled the production of long continuous microwires without any breakage.

Figure 2.8 (a) and (b) show microscopic images of 500µm and 200µm diameter microwire respectively after successful drawing. These images were captured under a digital compound microscope to examine the structure of the microwire. After examining, it was concluded that the microwire was continuous without any breakages and could carry current over its entire length.

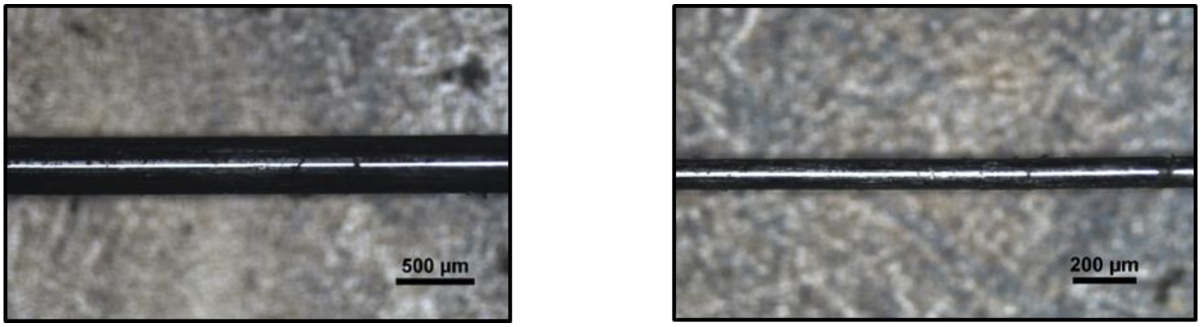

Figure 2.8 Microscopic images of microwires (a) 500µm wire (b) 200µm wire

CHAPTER 3. RESULTS

3.1 Characterization of microwires

Producing microwires with uniform cladding to core ratio using conventional thermal drawing methods was a challenge. Implementing vacuum stabilization made preform drawing consistent, and physical characteristic of uniform clad-core diameter ratio in microwires was immediately observed. Before preform drawing, initial measurements of preform were taken to calculate ratio between cladding and core. Preform had 8 mm clad diameter and 6 mm core diameter. The clad-core diameter ratio was calculated by dividing the clad diameter with core diameter and obtained value was 1.3. Figure 3.1 shows the preform with measured cladding and core diameter values.

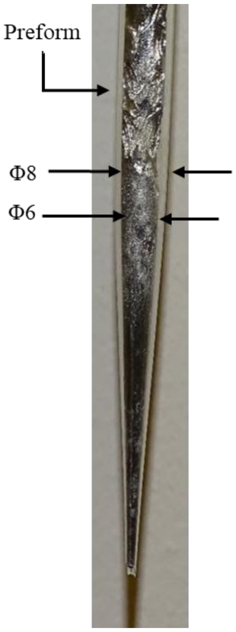

Figure 3.1 Actual consolidated preform

Later, using the VSTD process, 420 µm and 230 µm diameter wires were drawn through the same preform. The intent was to show the core-cladding ratio in both the microwires were the same as in the preform. At first, it was very difficult to measure core and cladding ratios in microwire. An elaborate process was involved in measuring core and cladding diameters under the microscope. First, the glass tube of 1 cm diameter was cut into two small sections using a glass cutter. Figure 3.2 show the glass tube section having 1 cm diameter.

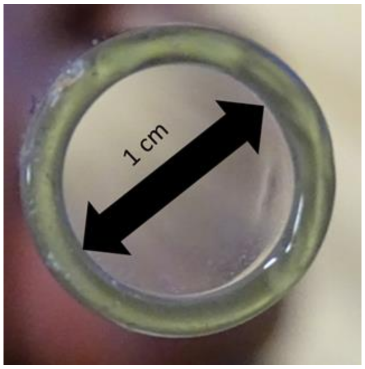

Figure 3.2 Glass tube section

A bundle of 420 µm and 230 µm diameter wires were placed separately in respective glass tube sections. After that, these two sections were immersed into an epoxy resin. This resin would become transparent and solidify over time, giving a clear view of the material inside. Figure 3.3 shows the hardened resin substrate containing microwires in the glass tube. Further, to get a clear

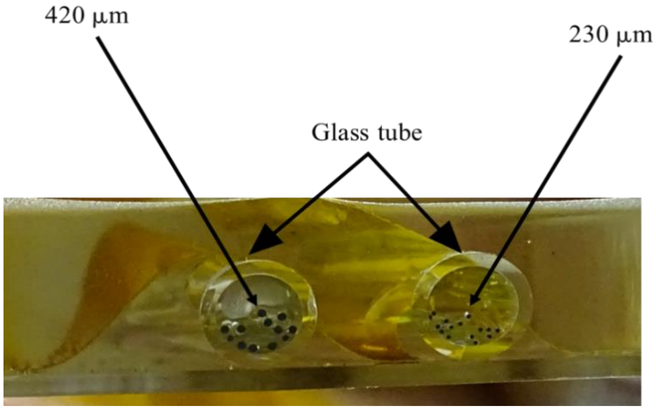

Figure 3.3 Microwire cross-section subsrate

view of the microwire cross-section, this hardened substrate was cut in between using table saw. Later, the cut surface was polished using a sanding instrument. Sandpapers ranging from 100-500 grit were used to obtain a very smooth and polished surface. This polished surface was then put under the microscope for measuring core and cladding diameters. In Fig 3.4 (a), the microwire section clearly shows the distinction between the core and cladding. The white region in the center is the metal wire, and it is surrounded by the dark circular region, which is the glass cladding. Fig 3.4 (b) and Fig 3.4 (c) show cross-sections of 230 µm and 420 µm, respectively. The clad-core diameter ratio for 230 µm diameter wire was calculated by dividing the clad radius with the core radius (i.e., 113.86/87.51), and the obtained value was 1.3. Similar calculations were done for 420 µm diameter wire, and same value for clad-core diameter ratio was obtained, which was 1.3.

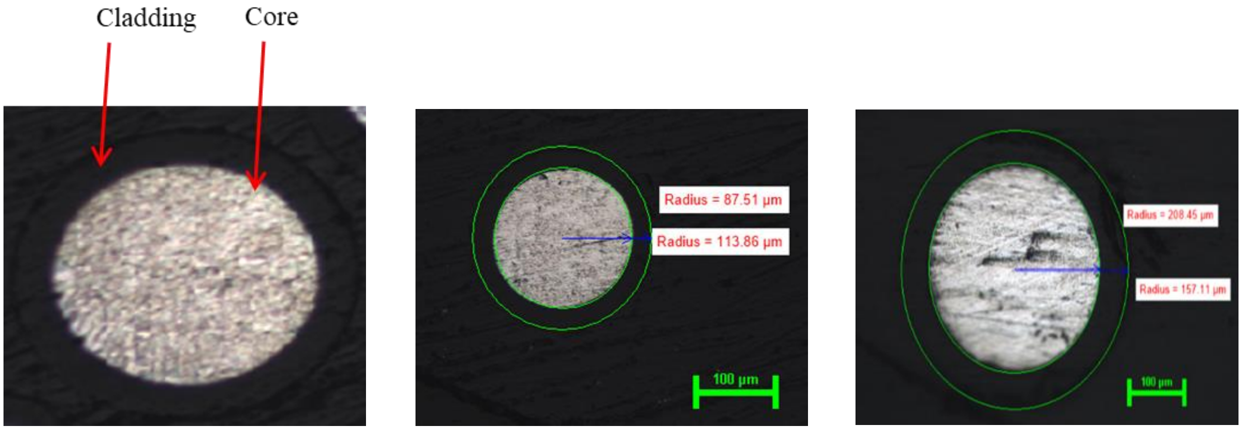

Figure 3.4 Experimantal validation showing physical characteristic (a) Microwire cross-section (b) 230µm wire cross-section (c) 420µm wire cross-section

It was observed that the clad-core diameter ratio for both diameter microwire and preform was 1.3. Hence, these observations gave conclusive evidence of microwire produced from the VSTD method has a physical characteristic of uniform cladding to core ratio throughout its entire length.

3.2 Drawing of long continuous microwire

This experiment started evolving gradually; initially, microwire drawing was done without using vacuum stabilization, which made microwires break during the drawing process. Discontinuity in microwires was due to this random breaking during the drawing process. The main reason behind breaking of microwires during the drawing process was capillary fluid instability due to lack of flow rate control over molten metal. Introduction of vacuum made possible to have flow rate control over molten metal. This resulted in formation of stable pendant drop, which then could be drawn into continuous microwires. These microwires could also be pinched off at any desired length during process, which enabled control over microwire length too. By using the law of mass conversation, masses going before and after drawing were equated for determining microwire diameter and other process parameters.

CHAPTER 4. APPLICATIONS

4.1 Nanomanufacturing

Microwires were successfully used in the laboratory for the production of glass coated nano-particles using the electro-spraying process.

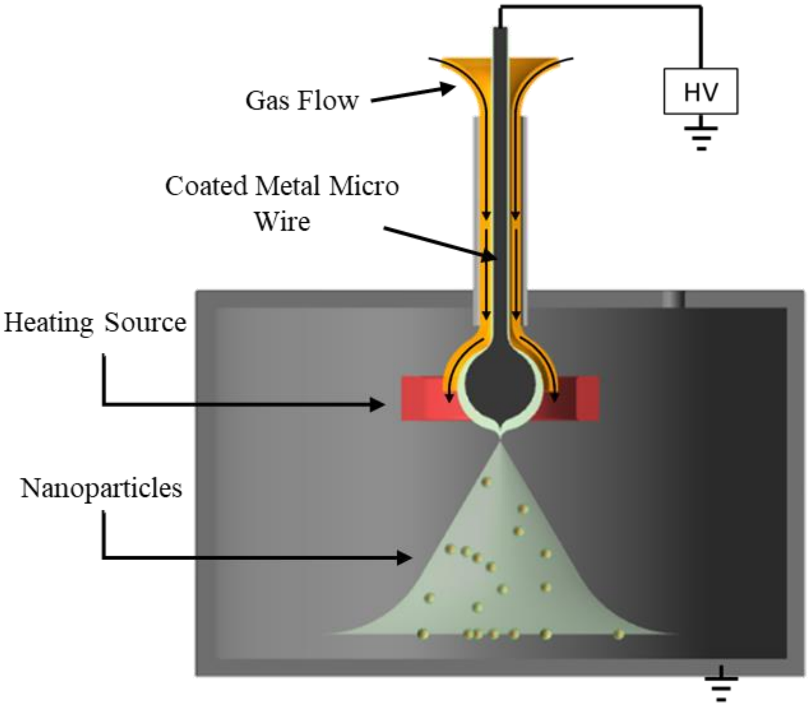

Figure 4.1 Schematic of electrospraying process

During typical electrospraying process, a liquid jet coming out of the nozzle is used to produce nanofibers by applying high voltage at the tip of the nozzle. The liquid reaching the diffuser tip overcomes its surface tension due to high voltage and forms a Taylor cone. The nanofiber jet coming out of the Taylor cone is attracted to the electrically charged collector plate due to electrostatic attraction. Depending upon initial conditions and materials, the shape, size, and form can be controlled. [15-20].

Three major processes were involved in manufacturing glass-coated nanoparticles. The first step was consolidating a glass-coated metal core preform and the second step was to draw glass-coated metal microwire out of the consolidated preform using the thermal drawing tower. The first two steps have been already covered in previous sections. The third step was to perform electro-spraying on the microwire to produce nanoparticles. The same thermal drawing tower was modified to accommodate the electro-spraying process. As can be seen from the figure 4.1, high voltage between 5 kV to 50 kV is applied between the tip of the microwire preform and the collector plate at the bottom, while a high temperature heating source was used to heat the microwire tip. Initially, three high temperature micro-flame guns spaced at an angle 120º were used as a heating source. During the process, it was observed that the high voltage was igniting the unburned hydrogen, which was leftover during micro-flame heating. This would completely burn out the microwire preform and eventually fail to produce nanoparticles. These micro-flame guns were then swapped with CO_2 laser to avoid generating hydrogen and prevent the burning out of the microwire preform. The CO_2 laser was directly focused at the microwire using a set of mirrors and then powered on for heating the tip of the microwire. Due to the high heat generated by the laser, the molten metal droplet was formed, and then it would drip into the Taylor cone. High voltage applied at the microwire tip would help overcome the surface tension on the droplet, which would then break into fine nanoparticles. These nanoparticles get collected on the negatively charged collection surface due to electrostatic attraction. The feeding system and collection plate has the ability to move relative to each other for maintaining the height difference. The diameter of nanoparticles could be controlled by varying this height difference [16,17].

A specific voltage is required to overcome the surface tension on the droplet of specific material for electrospinning. This critical voltage can be calculated by using equation (2).

$$Vc^2 = 4\frac{H^2}{L^2}\left(ln\frac{2L}{R} - \frac{3}{2}\right)(0.117\,\pi\gamma R) \tag{2}$$

where,

V_C = Critical Voltage H = Distance between capillary tip and collection surface

L = Length of capillary R = Radius of capillary

γ = Surface tension of liquid

If the applied voltage is higher than the calculated critical voltage, then a Taylor cone is formed at the bottom of the droplet, and ultimately nanoparticles would get sprayed or attracted to the collection plate due to electrostatic attraction. Figure 4.2 shows the electrospinning apparatus, and figure 4.3 (a) shows the actual image of Taylor cone formed at the bottom of the droplet, which was captured using a high-speed camera. Figure 4.3 (b) shows representation of a Taylor cone [16-20].

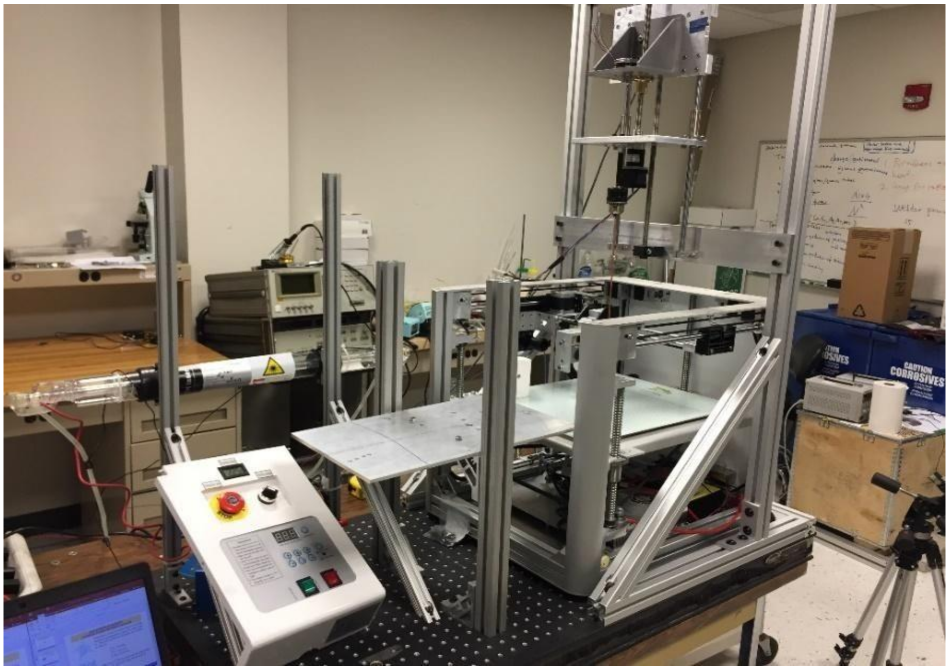

Figure 4.2 Electrospinning apparatus set up [16]

Figure 4.3 Formation of Taylor cone (a) Actual Taylor cone (b) Representation of Taylor cone [17]

4.1.1 Results and discussion

The microscopic images of collected nanoparticle samples are shown in Figure 4.4. In figure 4.4 (a), the dark black spot is pure tin or oxidized tin with the surrounding white area of glass (Pyrex) coating. Figure 4.4 (b) shows a microscopic image of nanoparticles at a 200 nm scale. These pictures prove that nonmanufacturing using the electrospraying process was successful [16].

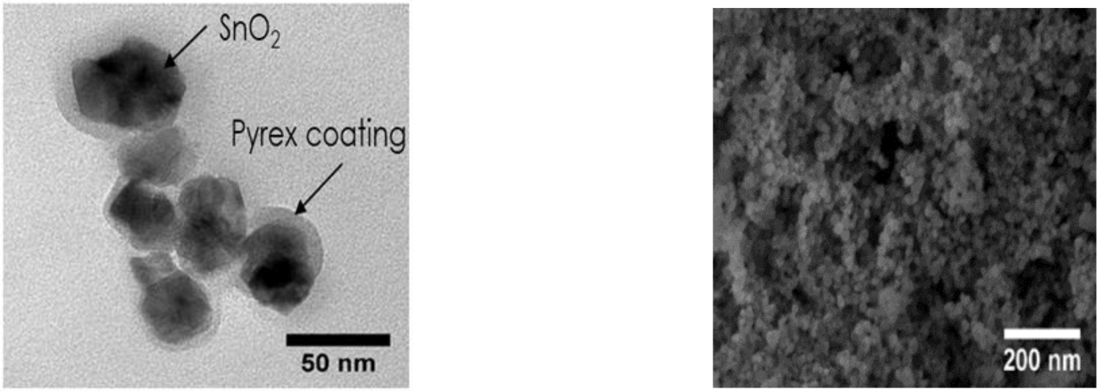

Figure 4.4 Microscopic images of nanoparticles (a) At 50nm scale (b) At 200nm scale

A Dynamic Light Scattering (DLS) test was also conducted to measure the mean diameter and polydispersity of produced nanoparticles. Figure 4.5 shows the result of the DLS test. The mean diameter was observed to be 141.63 nm, while polydispersity was 0.159, which showed that there is still room for improvements in the uniformity of the particle diameters [17].

Type	Start Date/Time	Sample ID	Eff. Diam. (nm)	Polydispersity	Baseline Index
DLS	4/17/2018 6:36:47 PM	pH 7.0_04172018_sample_1 - 1	143.11	0.171	6.9
DLS	4/17/2018 6:38:50 PM	pH 7.0_04172018_sample_1 - 2	141.85	0.144	9.9
DLS	4/17/2018 6:40:54 PM	pH 7.0_04172018_sample_1 - 3	139.92	0.161	8.4
		Mean	**141.63**	**0.159**	**8.4**
		Std Err	**0.93**	**0.008**	**0.9**
		Std Dev	**1.61**	**0.014**	**1.5**

Table 4.1 DLS Test Report [17]

4.1.2 Advantages of Electrospraying method

Electrospraying for producing glass-coated metal nanoparticles from microwires is a unique method, and it was used for the first time during this research study. Existing nanomanufacturing methods lack the ability to produce nanoparticles with a dense glass coating, which created the necessity to look for alternative method. Nanoparticles having different combinations of glass coating and metal core can be produced with electrospraying method. Glass coating the metal provides protection and solves the oxidation problem of metal during the heating process. These glass-coated particles find many applications in the medical industry, especially in medical diagnostics and targeted drug delivery.

4.2 High frequency applications

At high frequencies, wires tend to show high resistance due to skin effect. The solution to overcome this skin effect is to produce a thin wire having a diameter equal to its skin depth at a particular operating frequency. By using the VSTD method, microwires can be produced, which are thin enough to overcome skin effect at high frequencies. The skin depth of the wire at a given operating frequency can be calculated using Equation (3). After computing skin depth, a microwire having a diameter equal to skin depth value can be manufactured.

$$\delta = \sqrt{\frac{\rho}{\pi f \mu}} \tag{3}$$

where,

δ = Skin depth

ρ = Resistivity of the conductor

f = Operating frequency in hertz

μ = Permeability of the conductor [21]

4.2.1 Results and discussion

After drawing a microwire of different diameters, they were tested to validate the high-frequency application. The idea was to compute the equivalent resistivity of two different diameter microwires at high frequencies, and show how skin effect affects conductivity with change in size and frequency. For this study, a comparison was made between 420 μm and 230 μm diameter wires. This experiment was carried out in three steps. The first step was to fabricate a consolidated preform of a specific diameter for drawing required sized microwires. The second step was to draw preform into microwires using the VSTD method. After successfully drawing 420 μm and 230 μm diameter wires, the third step was to calculate their resistances on an LCR meter. For this purpose, the glass coating on two ends of the microwire was first removed to expose metal wire. An alligator clip was attached on both ends of metal wire and made sure it was firmly fixed so that the metal wire does not slip during the experiment. The other end of both the alligator clips was plugged into an LCR meter to complete the circuit. Frequency was slowly increased from 10 Hz to 1 Mhz at constant current, and several resistance data points were collected at specific intervals. Figure 4.6 (a) and (b) show the LCR instruments used for testing. The LCR instrument in figure 4.5 (a) has high frequency range up to 1 MHz. This instrument was used for collecting resistance data points in the frequency range beyond 20 Hz, while LCR meter in figure 4.5 (b) was used to collect data points in the frequency range of 10 to 20 Hz. Equivalent resistivity can be calculated using these data points.

Equivalent resistivity can be perceived as an apparent change in resistivity of the material due to skin effect at high frequencies. Equivalent resistivity was calculated using equation (4) for both microwires. A superimposed graph was plotted to compare change in equivalent resistivity with change in frequency.

$$\rho = \frac{R \cdot A}{L} \tag{4}$$

where,

ρ = Equivalent resistivity R = Resistance of wire

A = Surface area of the microwire L = Length of the microwire

Figure 4.5 LCR meter for resistance measurement (a) High frequency meter (b) Low frequency meter

As can be seen from figure 4.6, the scaled resistivity increases drastically for 420 µm wire at high frequencies when compared with the small 230 µm diameter wire. It shows that skin effect will be less for thinner wires. Hence it was proved that microwires having diameters equal to skin depth could be produced using the VSTD method, and they can be used in high-frequency applications like transformers. Conventional methods for Litz wire production lack the ability to produce a wire that can withstand harsh environments such as high temperature and corrosives. Litz wire produced through the VSTD process has the potential to carry currents at high frequencies and operate in harsh environments.

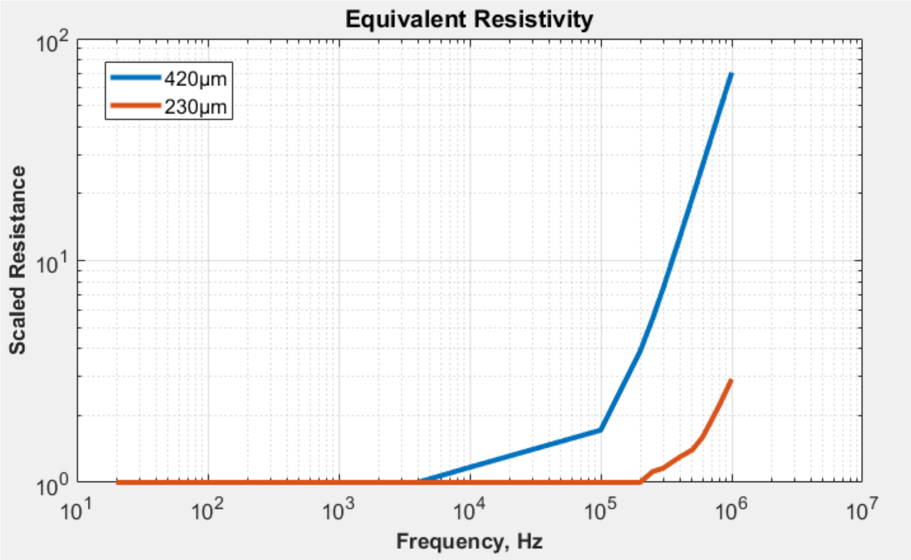

Figure 4.6 Equivalent resistivity graph for microwires

4.3 Benefits of VSTD over current methods

VSTD process offers a lot of benefits over existing methods. The methods discussed in section 1.2 especially lack the ability to have a flow rate control over molten metal and to form a stable pendant drop. Due to this, there is capillary fluid instability, which results in breakage and poor diameter control during preform drawing. The key advantage of VSTD is that it facilitates a stable preform drawing due to flow rate control over molten metal during the process. Flow rate control enables formation of a stable pendant drop, which allows to have the diameter control over the microwire. Moreover, with the VSTD process, ultra-thin coating can be achieved along with various combinations of core and clad materials. This method involves feeding and drawing in a single step, which makes it very convenient and less time consuming than the existing methods.

CHAPTER 5. CONCLUSIONS AND FUTURE WORK

This project started with the idea of making the manufacturing of microwires cheaper and scalable. The first step was to design and assemble the thermal drawing tower. It was essential to keep the assembly plain and cost-effective hence, procuring parts needed much research. Consolidating preform was the next challenge that required numerous trials and errors. Initially, fabricating preform without vacuum stabilization was carried out, which did not yield good results. Later, a method called rod in tube with zone melting under vacuum stabilization was tested and this method resulted in a consolidated preform. Subsequently, this method was adopted for preform drawing.

After successfully consolidating preform, obtaining continuous long fiber needed numerous trials. At first, preform drawing was inconsistent due to wrong process parameters. It was important to establish a relationship between fiber diameter, feeding and drawing speed. After establishing a relationship and performing necessary calculations, all process parameters were set to the correct derived values. The challenge was to draw continuous fiber; as fiber being delicate, it was susceptible to break at various locations. Vacuum was incorporated at the top of the preform to induce negative pressure and stabilize the pendant drop, which allowed to have the flow rate control over the molten metal. Having flow rate control finally resulted in the successful drawing of long continuous microwire.

This novel method of preform drawing also gave a fundamental explanation for the uniform clad-core diameter ratio in microwire. Large scale production is possible via VSTD method as this process is scalable. This method also has a multitude of potential applications and some of which were realized in the laboratory environment. Examples are successful production of nanoparticles by the electro-spraying process and high-frequency applications in transformers.

Future work:

Initially, the goal was to draw silica-coated copper microwires. Due to the temperature limitation of the available furnace, the goal of this research was set to the manufacturing of Pyrex coated tin microwires. A higher temperature furnace will need to be designed and built to generate the required amount of heat to melt silica-copper preform. Once the higher temperature furnace is ready, and once assembled, the same method can be used in the future for the production of silica-coated copper microwires. This microwire later can be used as Litz wire in transformers or in high-frequency applications for carrying current at very high frequencies with the least resistance. Electrospraying of silica-coated metal nanoparticles can be done similar to Pyrex-coated tin nanoparticles as well.

REFERENCES

[1] A Method of Drawing Metallic Filaments and a Discussion of their Properties and Uses G. F. TaylorPhys. Rev. **23**, 655 – Published 1 May 1924

[2] Scalable Manufacturing of Metal Micro/Nano Wires and Particles by Thermal Fiber Drawing from a Preform. Zhao, Jingzhou. University of California, Los Angeles, ProQuest Dissertations Publishing, 2017. 10637596.

[3] Glass-coated wires and methods for the production thereof, US patent US20110036123A1

[4] Production, properties and applications of microwire and related products, I.W Donald

[5] A. A. Baikov, French Patent 1361929 (1963)

[6] V. I. Zaborovsky, O. A. Ivanov and A. N. Savenkov, British Patent 1157 313 (1969)

[7] Idem, US Patent 3607201 (1971)

[8] Idem, British Patent 1120247 (1968)

[9] Extrusion Processing Shokoh Fatahi, Ph D.(Polymer Processing), P. Eng.

[10] Multimaterial Fibers Guangming Tao, Ayman F. Abouraddy, Alexander M. Stolyarov and Yoel Fink

[11] E. Snitzer and R. Tumminelli, “SiO2-clad Fibers with Selectively Volatilized Soft-glass Cores,” Opt. Lett., 14 [14] 757–759 (1989).

[12] **Fabrication of glass photonic crystal fibers with a die-cast process,** Zhou Guiyao, Hou Zhiyun, Li Shuguang, and Hou Lantian

[13] A Long Wavelength Model for Manufacturing of Continuous Metal Microwires by Thermal

Fiber Drawing From a Preform by Jingzhou Zhao and Xiaochun Li

[14] Wikipedia page, https://en.wikipedia.org/wiki/Zone_melting

[15] Wikipedia page, https://en.wikipedia.org/wiki/Electrospray

[16] Scalable Manufacturing of Continuous Metal Nanowires by Electrospinning, Yuri Gulak

[17] Scalable Manufacturing of Continuous Metal Nanowires by Electrospinning, Max Martel

[18] Thenmozhi, S., et al. “Electrospun Nanofibers: New Generation Materials for Advanced Applications.” *Materials Science and Engineering: B* , vol. 217, 2017, pp. 36-48., doi:10.1016/j.mseb.2017.01.001.

[19] Greiner, Andreas, and Joachim H. Wendroff. “Electrospinning: A Fascinating Method for the Preparation of Ultrathin Fibers.” 2007 *Wiley Online Library*, doi:10.1002/anie.200604646.

[20] Melt-electrospinning of Thermoplastic Polymers: An Experimental and Theoretical Analysis, Jason Michael Lyons, Drexel University, 2004.

[21] Transmission Lines and Networks by Walter C. Johnson, McGraw-Hill 1963 p58