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NAVAL POSTGRADUATE SCHOOL

Monterey, California

SUBMINIATURE HOT-WIRE SENSOR CONSTRUCTION

by

P. M. Ligrani

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The detailed procedure for constructing subminiature hot-wire sensors is given. The wire used is 0.625 μm diameter, 90 per cent platinum/10 per cent rhodium, plated with 30-40 μm diameter of silver. For construction, the plated wire is first bent into the appropriate shape, and then soldered onto stainless steel prongs. Portions of the silver are then etched, removing it entirely from a 200 mm-400 mm length of platinum/rhodium wire. Small portions

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of copper plating are then added to give the desired platinum/rhodium wire sensing length, and to insure that good mechanical and electrical connections exist to the sensor. With this procedure, subminiature hot wire sensors have been constructed which operate with minimal drift, and are mechanically robust for long periods of time. The sensors have been demonstrated to be a useful research tool for turbulence research because they provide a more accurate means to measure energy levels of small-scale turbulent motions than is possible with other measurement devices.

TABLE OF CONTENTS

1. INTRODUCTION

The following report is intended as a guide to persons wishing to construct subminiature hot-wire sensors. The techniques described have been used successfully to construct subminiature hot-wires; however, as individuals master the techniques involved, each will develop his or her own unique procedure. The two most important equipment items required are a three-dimensional micromanipulator and a variable-power, binocular microscope. Other items are mentioned in the text. For information on the response of normal-sized and subminiature hot-wire sensors, the reader is referred to archival journal papers by P. M. Ligrani and P. Bradshaw.

2. THE WIRE

The subminiature hot-wire sensors are constructed using 0.625 μm diameter, 90% platinum/10% rhodium wire. This wire is manufactured by the Wollaston process and is available from Sigmund Cohn Corporation (121 South Columbus Avenue, Mt. Vernon, New York; export distributor -Leico Industries, Inc., 250 West 57th Street, New York, New York 10019). In the Sigmund Cohn Corporation manufacturing process, the platinum/rhodium wire is first inserted into a tube of silver. The resulting composite is then drawn to a preselected final overall diameter of about 30-40 μm. This wire may then be easily handled and manipulated during the construction procedure.

3. BENDING THE WIRE

In order to work with the plated platinum-rhodium wire, one end of a 2 cm long segment is glued to a 1 mm diameter brass wire. Brass wire was chosen as a means to hold the plated Pt-Rh wire for soldering because it was available, easy to work with, and easily bendable. The brass wire is then held by a PRIOR 59638 three-dimensional micromanipulator so that position and orientation are easily controlled.

With the silver-plated platinum-rhodium wire attached to the brass wire, and the brass wire held by the three-dimensional micromanipulator, the plated Pt-Rh wire may be bent. Each bend should be made with only one motion so that minimal cold-working of the platinum-rhodium wire takes place. Using a surgeon's pointed tweezers, two bends are required so that the wire appears to make three sides of a rectangle. As shown in Figure 1, the bends at the corners of the rectangle should have a small radius and not be too sharp or abrupt. The middle side of the rectangle should be about 500 μm in length; the other sides should be 3 mm-5 mm. This configuration ensures that, during measurements, there is sufficient distance between sensor and the main part of the probe support so that flow near the sensor is unaffected by any disturbances caused by the larger parts of the probe support.

4. SOLDERING THE WIRE

As shown in Figure 1, the Pt-Rh wire is soldered to two stainless steel prongs which are placed about 0.5 mm apart. It is very important to solder the wires in place with no added deformation or strain. Otherwise, during the etching process, the wires will probably break when the silver plating is removed.

Before soldering, the bent Pt-Rh wire is placed on the two prongs. This is done while viewing the wire and prongs using a variable-power Watson Barnet model 142183 binocular microscope to ensure that one wire and prong do not come in contact before the other wire and prong. If this is not the case, the Pt-Rh wire and/or the prongs are repositioned to give better alignment. The plane of the bent Pt-Rh wire "loop" is soldered so that is is inclined relative to the plane of the prongs. With this arrangement, the sensor will then be lower and closer to the wall than the prongs during a measuring traverse, as shown in Figure 1.

After the wire is in position, a drop of orthophosporic acid is added using a pipette to the location where the Pt-Rh wire touches the prong. This is done without use of the microscope; however, afterwards, the microscope is used for a look at the location and size of the acid drop. The acid is in a satisfactory position if in contact with both the wire and prong. Next, a pointed soldering iron having about a 2 mm diameter is used to place a drop of solder at the location of the acid flux. Again, this is done without the use of the microscope; however, a look afterwards gives an indication of whether the soldering was satisfactory. If not, the step is repeated until successful, and then the wire is soldered to the second prong. At soldering, a hiss indicates that the hot solder has made contact with the orthophosphoric acid. After soldering, a small Exacto knife is used to cut off loose Pt-Rh wire ends. This is sometimes done, as appropriate, after the wire is soldered to one prong.

Figure 1 shows how the wire should look after soldering and Exacto knife trimming.

5. ETCHING

In order to remove the silver plating from the Pt-Rh wire, a bath of 5 percent nitric acid is required. The Watson Barnet variable-power, binocular microscope is placed at a 60° angle relative to horizontal, and focussed on or just above the surface of the acid which is contained in a 250 ml beaker. This microscope is variable-power from 10x to 100x and is connected to a pivot allowing rotation of the optical column along two different axes. The probe holder, with the bent Pt-Rh wire soldered to it, is held using the three-dimensional micromanipulator so that the plane of the wire loop is nearly vertical and easily seen through the microscope.

Next, the electro-etching device is attached to the connecting prongs on the probe holder, and to an electrode immersed in the nitric acid. This electro-etching device, shown schematically in Figure 2, is a DC voltage generator. The output of the device is adjustable, and capable of producing current levels between 0 and 25 milliamperes. During the etching process, the positive output terminal (anode) is connected to the probe, and the negative output terminal (cathode) is connected to the acid electrode.

With the electro-etching device turned off (no current), the end of the Pt-Rh wire loop is immersed in the acid bath. A complete circuit then exists for the current to and from the etching device. From the output terminal of the etching device, the current will pass from the probe holder, to the plated Pt-Rh wire, through the acid, to the platinum electrode immersed in the acid, and finally back to the etching device.

At the completion of the etching procedure, the aim is to obtain a 200-400 μm length of Pt-Rh wire connected to tapered silver plating. With a voltage level across the etching device terminals of about 1.5 volts, the current is set to about 150 μ amperes. Progressively smaller currents and voltages are required as the silver plating diameter decreases in size.

While viewing the Pt-Rh wire through the binocular microscope, the Pt-Rh sensor wire is left in the acid until sufficient silver is removed so that the diameter decreases to 5-10 μm. Smaller and smaller parts of the loop are left in the bath as time increases so that the silver plating will be tapered to the location where the Pt-Rh wire will be completely exposed. This process requires 10 to 15 minutes, during which, it may be helpful to move the Pt-Rh wire in and out of the acid bath. This helps to mechanically remove silver plating as the wire loop passes through the surface of the acid. In the beginning, silver bits come off in small slivers, and later, in even smaller granules. Before the partially plated Pt-Rh wire is moved across the surface of the acid, the electro-etching device is generally turned off and current levels allowed to become zero.

Removing the last 5-10 μm thickness of silver from the Pt-Rh wire requires extra care. When the Pt-Rh wire is exposed after etching, the sensor is in its most delicate mechanical state. The Pt-Rh wire, exposed just as all silver is removed, could also be melted away if current levels for etching are too high.

In order to remove the last parts of the silver plating, three different techniques have been used successfully. Each may require 1-5 minutes to complete. The techniques are recommended in decreasing order, as follows:

(1) The acid bath technique is continued using current levels which are one-sixth to one-tenth of initially set values. These lower current levels are obtained by connecting a resistor in the electric circuit. With such levels, the Pt-Rh wire may be moved freely in and out of the bath with little or no danger of melting. This approach also has the advantage that removal of portions of silver plating, spread nonuniformly along the Pt-Rh wire, is easily accomplished. In some cases, a few residue bits of silver may remain; however, with care, these may also be removed.

(2) When 5-10 μm of silver plating still remains attached to the Pt-Rh wire, it should be placed near the liquid surface so that surface tension is holding the liquid around the wire. The etching device is then quickly turned on and then off, to send an impulse of current to the sensor. If successful, the last silver plating falls away leaving exposed Pt-Rh wire out of the nitric acid (with the electrical circuit broken) since the surface tension (which is related to wire diameter) is not large enough to maintain liquid around the wire.

(3) The removal of all of the silver plating along a segment of the Wollaston wire may be done using a jet of acid. The wire is placed near the edge of the acid jet so that contact with the wire is due to surface tension.

When the Pt-Rh wire becomes exposed, the circuit is broken (because surface tension decreases), minimizing the risk of a melted wire.

6. WASHING

A bath of distilled water is now used for sensor washing. The sensor is again held in the three-dimensional micromanipulator and viewed using the binocular microscope. The loop of silver plated wire, now with an exposed portion of Pt-Rh, is slowly immersed in the distilled water so that the entire sensor, except for the soldered connections, is in contact for 3-5 minutes. During operation of different sensors, it was found that those which were washed had the smallest drift. Thus, the drift is believed to be due to chemical residue on the Pt-Rh wire and supporting silver.

7. PLATING

As shown in Figure 3, portions of the sensor, near its corners, are plated with copper. This is done in order to obtain a Pt-Rh wire having the desired sensing length for a particular measurement application. The copper plating also ensures that good mechanical and electrical connections exist to the sensing portion of the sensor. After etching, the silver plating may be mechanically weak because small portions are unconnected with the main body of the probe.

The plating solution requires four components:

(1) 750 ml distilled water,

(2) 10-12 cc sulfuric acid,

(3) 6-7 grams potash alum,

(4) 100 grams copper sulfate.

To make the plating solution, copper sulfate and potash alum are first dissolved in a portion of the distilled water at room temperature. All of the distilled water is then added, stirring the solution for a time so that all components are well mixed. The sulfuric acid is then added very slowly with stirring. Finally, the solution is placed in a sealed flask and well shaken. If, after plating, the copper is too brittle, a less acidic and more dilute solution is required.

The probe holder is held on the three-dimensional micromanipulator so that the two prongs (to which the silver plated leads are soldered) are at a 45 degree angle relative to horizontal, oriented so that both prongs are approximately in the same vertical plane. A slight inclination may be necessary so that the probe may be easily seen with the binocular microscope.

Next, the probe is held just above the surface of the copper plating solution so that it is easily viewed using the binocular microscope. An electro-plating device, shown schematically in Figure 2, is then connected to the probe connectors and to an electrode placed in the copper plating solution. In the completed electric circuit, the probe is then the cathode, and the immersed electrode is the anode. The corner of the probe is then immersed in the plating solution. The current and voltage on the electro-plating device are then set at 20 to 150 μ amps and 90 to 150 m v, respectively, where these levels depend on how much of the sensor is immersed in the solution. These voltage and current levels will change as more copper plating is added. Both a portion of the silver plating and a small portion of the Pt-Rh wire should be plated in the process, which requires anywhere from 2 to 10 minutes depending on the immersed surface area. The plating should be done very slowly with the smallest levels of current possible, in order to give a surface which is hard and solid, and not soft and crystalline.

The diameter of plating added to the Pt-Rh wire should be 10-25 μm, as required. After completion of the plating on one corner of the wire, the process is then repeated on the opposite corner after rotating the wire

180 degrees.

8. FINAL WASHING

After plating, the sensor is again washed, using the same procedure as before.

9. PROBE CHECK

After the final washing, the sensor resistance is measured. The resistance per unit length of the 0.625 μm diameter platinum/rhodium wire is 0.577 ohms/μm. The length is then computed from the resistance and compared with the length measured using a 200x Vickers microscope. If the two lengths do not agree within about ±5 percent, one of several problems may exist:

(1) a chemical residue may be present on the Pt-Rh wire and additional washing is required;

(2) the Pt-Rh wire is not entirely clean - bits of silver may be present;

(3) the silver plating is partially broken off; or

(4) the copper and/or silver plated portions may not be entirely solid.

The alignment of the wire with the horizontal plane may also be checked using a high power microscope. This is done by rotating the wire about the probe axis until the entire length of wire is in focus. If part of the wire is out of focus, it is not contained in the microscope field of view. After alignment with the field of view, the angle of the plane of the connecting prongs (which normally should be horizontal) is measured.

Alignment of the probe with respect to vertical probe stem may be checked when the sensor is operated in a flow of known direction.

After operation of the wire, 4-6 hours of aging is required for minimal drift. During this time, the magnitude of the drift (change of voltage with respect to time) seems to decrease monotonically.

A photograph of a completed subminiature hot-wire sensor is shown in Figure 4. In this case, the probe sensing length is 225 μm, and the distance between the two silver supports is about 500 μm. The right-hand portion of the Pt-Rh sensing wire appears very large since it is reflecting light and because an exposure time of 30 seconds was used to obtain the photograph. Because very little light is reflected from the left-hand portion, it appears to be very small or nonexistent.

10. SUMMARY

A description of subminiature hot-wire construction procedures has been presented. By carefully following the procedure and after some practice, a reasonably careful person may use the step-by-step procedure to make a sensor in 2 to 3 hours. If handled and operated properly, the sensors then have minimal draft and may be used without breakage for an indefinite period. Subminiature sensors are a valuable fluid mechanics research tool since they provide more accurate measurement of small-scale turbulence than has been possible in the past with longer sensors having less spatial resolution.

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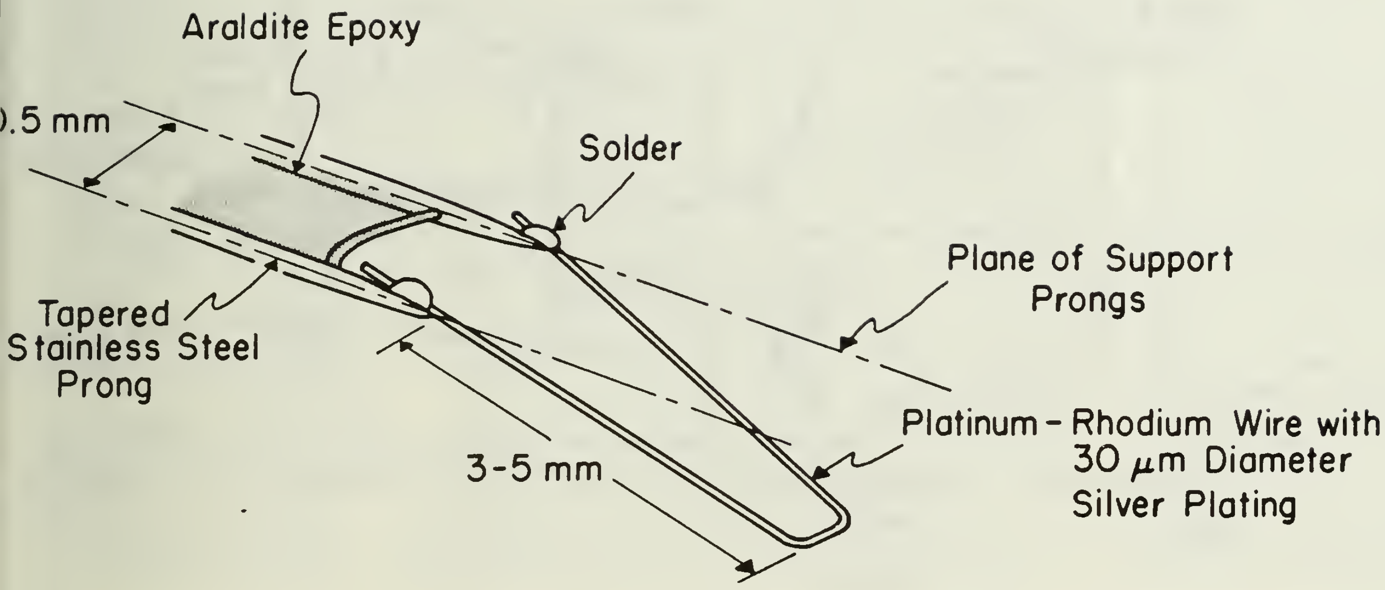

Figure 1. Stainless Steel Prongs with Plated Platinum-Rhodium Wire Loop Soldered into Place.

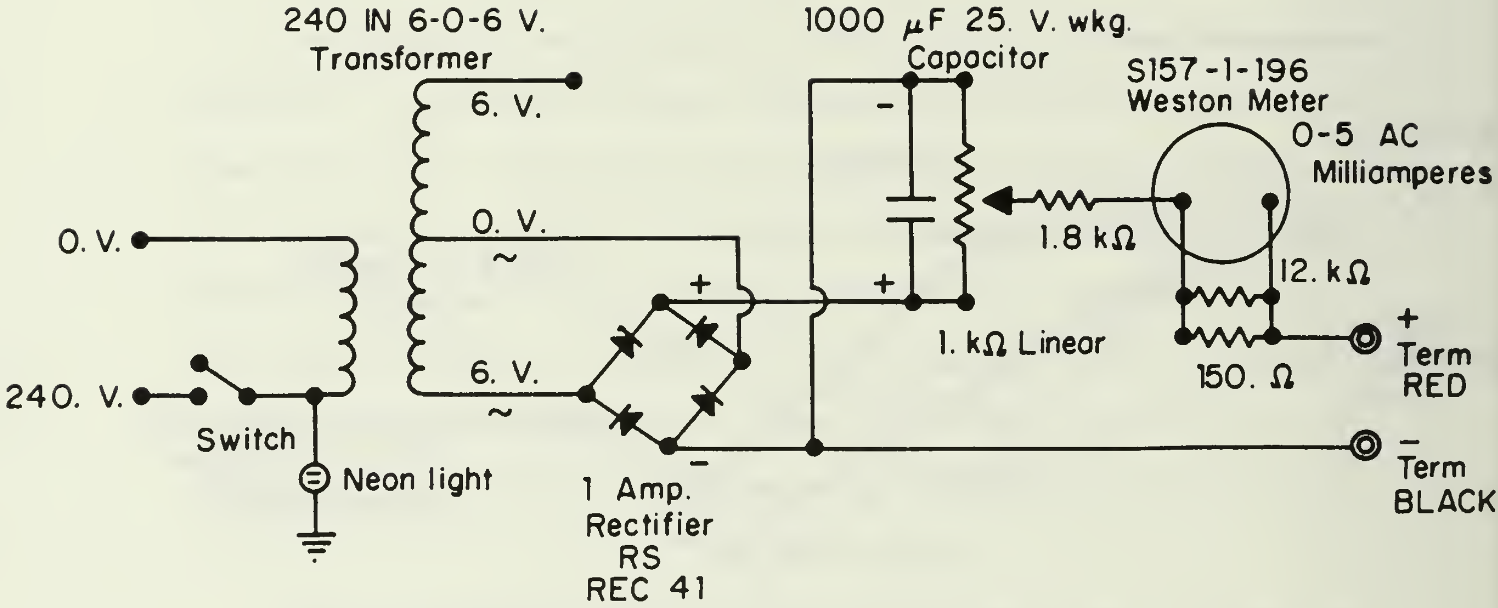

Figure 2. Schematic of Circuitry of Electro-Plating/Electro-Etching Device. Components shown are for plating.

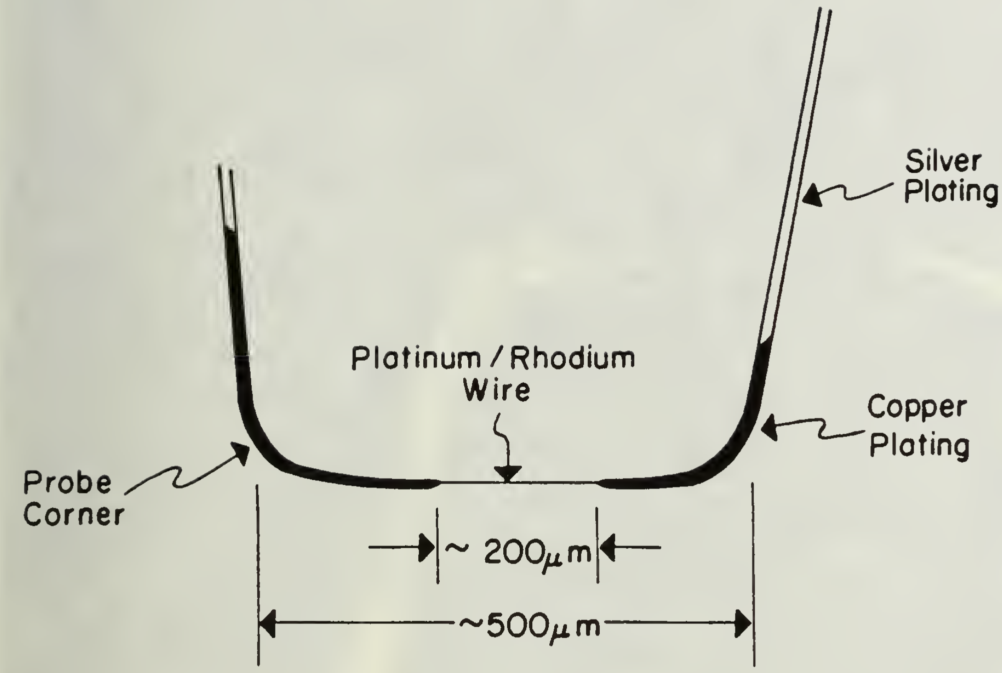

Figure 3. Completed Sub-Miniature Hot-Wire Sensor.