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ADVANCED PROCESS TO EMBED OPTICAL FIBER SENSORS INTO CASTING
MOLD FOR SMART MANUFACTURING

by

RAGHAVENDER REDDY JAKKA

A THESIS

Presented to the Graduate Faculty of the

MISSOURI UNIVERSITY OF SCIENCE AND TECHNOLOGY

In Partial Fulfillment of the Requirements for the Degree

MASTER OF SCIENCE

in

MANUFACTURING ENGINEERING

2018

Approved by

Lianyi Chen, Advisor

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Jie Huang

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PUBLICATION THESIS OPTION

This thesis consists of the following article that will be submitted for publication as follows:

Paper I: Achieving enhanced sensing range and sensitivity in embedded optical fiber sensors for smart manufacturing, pages 3-18 are intended for submission in Sensors and Actuators A: Physical

ABSTRACT

Optical fiber sensors embedded in metals with distributed sensing can sense temperature at multiple points with single fiber. This is useful for smart manufacturing like structural health monitoring in aerospace industry and smart molds in manufacturing plants. There is a huge difference in thermal coefficient of expansion for fiber and metal. This is the reason for the increase in sensitivity for embedded fiber sensors. However, at high temperatures, the stress on the fiber increases, eventually damaging the sensor. The fiber-metal interface determines the sensor performance. A tight interface results in high sensitivity and a gap in the interface enhances sensing range. There is a dilemma to choose either high sensitivity or high sensing range. The objective of this study is to enhance the interface to have both high sensitivity and high sensing range which can be used for casting application. Extrinsic Fabry-Perot interferometer (EFPI) sensors with a single sensing point and cavity length around 50 μm are embedded into copper substrate using electrodeposition. The embedded sensors are 300 μm deep from the surface. Three different interface: chemical plated, copper painted, and dual-layer interface, were tested. The results show that dual-layer interface can provide both high sensitivity of 45 $\text{pm}/^\circ\text{C}$ and high sensing range of 700 $^\circ\text{C}$ at the same time, which overcomes sensitivity-sensing range dilemma. The analysis shows that one layer in the dual-layer interface increases the longitudinal strain for sensitivity and the other layer reduces the radial strain which enhances the sensing range. This new dual-layer interface developed in this research can have high sensitivity and high sensing range at the same time. Aluminum casting was done to test the effectiveness of the dual-layer interface. The cooling curve data from the EFPI sensor is consistent with the thermocouple data.

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SECTION

1. INTRODUCTION

Optical fiber sensors are superior to conventional sensors with compact size, electromagnetic immunity, multiplexing, and corrosion resistance. A single optical fiber sensor can have multiple sensing points along its length called as distributed sensing. These fiber sensors have the potential to measure temperature, strain, pressure, etc. As the shift in the wavelength of light is used, even a small change in the input can be detected. There are different types of fiber sensors depending on the fabrication method used. Of them, fiber grating sensors and interferometers are the majorly used optical fiber sensors.

Smart manufacturing is the real-time understanding of all the manufacturing process facilitated by advanced sensor-based data analytics. This is applied for structural health monitoring in aerospace industry and smart molds. The feedback from the sensors is used to control the process. Temperature monitoring is critical in some process like continuous casting where fluctuation of temperature has to be monitored to reduce the quality issues. Conventional sensors are integrated in the casting molds by drilling holes. This destroys the integrity of the mold and moreover, sensing points are limited by the number of holes that can be drilled in a mold.

Embedding fiber sensors into the metal molds can enhance the temperature monitoring process by having many sensing points in a single fiber. Embedding fiber sensor into metal enhances the sensitivity at the cost of limited sensing range. This is because of the mismatch between the thermal properties of fiber and the metal. For example, thermal expansion coefficient of copper is thirty times higher than that of silica fiber. When the temperature is increased, copper induces stress on the fiber and eventually damaging the

sensor at high temperatures. The interface transfers the stress from metal into the fiber. Interface that is too tight has high sensitivity but not high sensing range. If there is a gap in the interface, it has high sensing range but low sensitivity.

This research focus on developing a new interface for embedding fiber sensors in metal to have both high sensitivity and high sensing range at the same time. Three fiber-metal interfaces, namely: chemical plated, copper painted, and dual-layer interface, are tested. Extrinsic Fabry-Perot interferometer (EFPI) sensors are embedded into copper substrate using electrodeposition. After annealing the samples, they are tested in a tube furnace. Dual-layer interface had a linear sensitivity of $45 \text{ pm}/^\circ\text{C}$ and high sensing range of 700°C . The analysis of the dual-layer interface was carried out using scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS).

PAPER

I. ACHIEVING ENHANCED SENSING RANGE AND SENSITIVITY IN EMBEDDED OPTICAL FIBER SENSORS FOR SMART MANUFACTURING

ABSTRACT

Fiber sensors embedded in the metals can be used to measure temperature at multiple points with a single fiber. The difference in the thermal expansion coefficient of metal and fiber makes it difficult to achieve both high sensitivity and high sensing range at the same time. There is a dilemma of choosing either high sensitivity or high sensing range and not both. Fiber-metal interface plays an important role in transferring stress from the metal to fiber. Here, we developed a dual-layer interface to achieve both high sensitivity and high sensing range. Longitudinal strain governs the sensitivity while the radial strain affects sensing range. Dual-layer interface with a carbon layer to increase longitudinal stress and a porous copper paint layer to reduce the radial stress had high sensitivity and sensing range at same time. A dual-layer interface can be used for high temperature applications with high sensitivity.

1. INTRODUCTION

Optical fiber sensors are compact and immune to electro-magnetic interference when compared to conventional sensors [1]. They can sense external stimuli like temperature, pressure, strain, etc., when embedded in metals [2, 3]. With distributed sensing capability, a single fiber embedded in metal can detect temperature at various points. Embedding fiber sensor into metals has also enhanced the sensitivity of the sensor which is useful for detecting small changes in the temperature [4]. Using the real-time feedback from these

embedded sensors and controlling the process is one of the important aspects in smart manufacturing [5–7]. Smart manufacturing is employed in various areas, e.g. aerospace for structural health monitoring, manufacturing plants for smart molds [8–13].

There are various techniques of embedding fiber sensor into metals which determines the service temperature and sensitivity of the sensor. Fiber sensors in general are fragile and require metal coatings to increase strength and sensitivity [4, 14]. Embedding sensor with the polymer coating inside a metal limits the temperature range to 200 °C [15]. Chemical plated fibers have been used to increase the strength of the fiber for further processing [14]. Metal coated fiber sensors embedded into metal sheets by ultrasonic consolidation were damaged due to the pressure and vibration [16–18]. Layered manufacturing method can be used to embed fibers into metals [19–21]. When selective laser melting technique is applied for embedding fibers, defects such as gap between fiber and metal, delamination of metal occurs [19, 22]. Previous works proved electrodeposition to be effective and stress-free method to embed fibers. Fibers are made conductive using chemical plating process and then embedded into metals by electrodeposition [4, 23].

The problem with the embedded optical fiber sensors is the mismatch of thermal expansion coefficient. Metals have a high thermal expansion coefficient when compared to optical fiber. When the embedded fiber sensors are subjected to high temperatures, the expansion of metal is higher than that of fiber. This induces strain in the fiber which deforms fiber along with the metal. Fiber-metal interface plays an important role in transferring this strain. If the interface is tight, the strain in the fiber becomes high at elevated temperatures which eventually damages the sensor. Drilling and placing the sensor in the holes create a gap in the interface which will reduce the sensitivity and increase the response time. Moreover, drilling holes will affect the mechanical integrity of the metal. This makes it difficult to achieve both high sensitivity and high sensing range at the same time.

In this paper, a dual-layer interface with high sensitivity and high sensing range was demonstrated. By altering the interface, the sensitivity was increased by 50 times with improved sensing range, room temperature to 700°C. Extrinsic Fabry-Perot Interferometer (EFPI) sensors are embedded into copper substrate using electrodeposition. Three different fiber-metal interfaces are developed, and tested. The analysis of the interface was carried out using scanning electron microscopy (SEM) micrographs and energy-dispersive X-ray spectroscopy (EDS). There is strain in two directions in the fiber, longitudinal and radial which result in sensitivity and sensing range respectively.

2. MATERIALS AND METHODS

The sensor used in this research is Extrinsic Fabry-Perot Interferometer (EFPI) sensor. They are one of the two types of optical fiber Fabry Perot interferometric sensors [24]. The schematic and principle of EFPI sensor is shown in Figure 1. There are two reflectors one at each end of the capillary tube and Single Mode Fiber (SMF) junction. When light is injected into the EFPI sensor, a fraction of light is reflected by the first reflector while the rest travels through the capillary tube and again a part of it is reflected by the second reflector. The reflected beams create an interference pattern. When temperature changes, the cavity (capillary tube) length changes and the interference pattern shifts. EFPI sensors are more commonly used than Intrinsic Fabry-Perot Interferometer (IFPI) sensors as it is easier to fabricate. The fabrication process involves splicing silica capillary tube between single mode fibers. The ends of capillary tube act as reflectors and partially reflect light. These two reflected beams interfere with each other forming an interference spectrum. The basic principle of EFPI sensor is the shift in interference spectrum with the change in cavity length. This shift in the interference spectrum can be used to detect temperature and strain [25]. The geometric parameters of the EFPI sensor govern the application of the sensor [26].

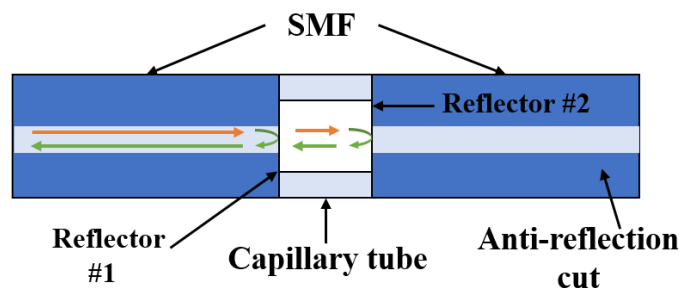


Figure 1. Schematic of EFPI sensor

The fabricated EFPI sensor was embedded into the metal by electrodeposition. Electrodeposition is an electro-chemical process wherein the metal from the anode deposits on cathode (Figure 2). The setup contains two electrodes that are immersed in a solution called electrolyte [27]. When DC current with required potential difference is applied between the electrodes, metal from anode reduces into ions and dissolves in electrolyte. These metal ions in the solution are reduced on the surface of the cathode forming metal coating. Cathode surface which is free from surface irregularities and hydrophobic substance ensures good deposition strength.

The surface of the fiber should be made conductive to embed it using electrodeposition. This can be done by chemical/electro-less plating or copper paint. Chemical plating involves a redox reaction on the surface of the fiber to form a metal layer. Chemical plating does not require external electrical energy as electroplating. It involves three solutions, namely: sensitizing, activating, and chemical plating solutions (Table 1). Fiber needs to be hydroxylated using sulfuric acid (ACS reagent 95-98% from SigmaAldrich) for 2-5 minutes before chemical plating. Copper paint on the other hand involves a simple dip coating of copper paint from Caswell Inc. This serves as initial conductive layer and can be used for electrodeposition.

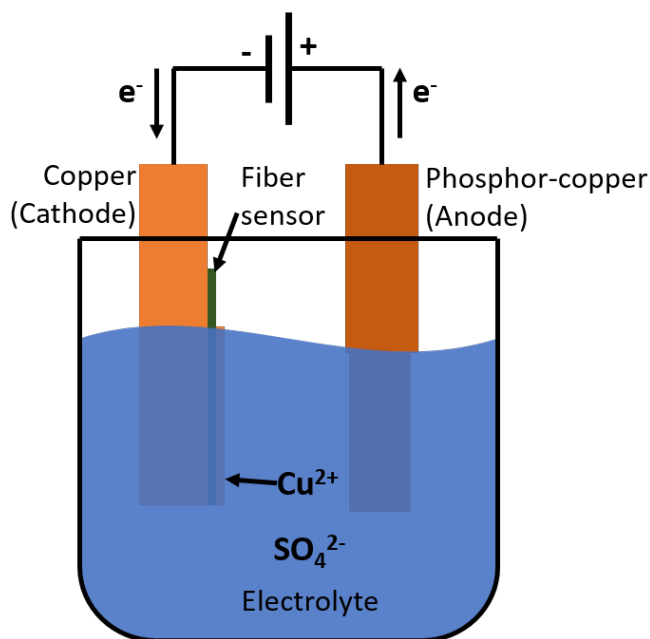


Figure 2. Schematic of electrodeposition setup.

Table 1. Chemical composition and dipping time of the solutions required for chemical plating of copper

Solution	Chemical composition (/lit)	Dipping time
Sensitizing Solution	SnCl ₂ .2H ₂ O 10 gm	10 min
	HCl (37%) 40 mL	
Activating Solution	PdCl ₂ .2H ₂ O 0.5 gm	10 min
	HCl (37%) 40 mL	
Chemical plating Solution (pH=12)	CuSO ₄ .5H ₂ O 10 gm	90 min
	NaKC ₄ H ₄ O ₆ .4H ₂ O 35 mL	
	NaOH 8gm	
	Na ₂ CO ₃ 1 gm	
	NiCl ₂ .6H ₂ O 0.5 gm	
HCHO 20mL		

The cathode and anode used for the electrodeposition setup are 99.99% pure copper from Mc-master and phosphor-copper from Caswell Inc respectively. Surface preparation of the electrodes involves grinding till 1200 grit sandpaper and ultrasonically cleaning them. Sulfide bath electrolyte was made using 42 grams of anhydrous copper sulfate (anhydrous, 99-100.5% from Sigma Aldrich) and 7.5 mL of sulfuric acid (ACS reagent 95-98% from Sigma Aldrich) in 345 mL of deionized water. Prior to the electrodeposition, electrodes are etched using an acid dip whose composition is 15% hydrochloric acid (ACS reagent, 37% from Sigma Aldrich) and 7% sulfuric acid (ACS reagent 95-98% from Sigma Aldrich). Two small bits of Kapton tape used to hold the fiber in place were removed after 3 hours of electrodeposition while the whole process was continued for 12 hours. Agilent U8031A DC power supply operated in constant current mode at 0.5 A was used to apply the potential difference for deposition.

Tight interface had better sensitivity with a low sensing range and a gap in the interface resulted in low sensitivity and high sensing range. To overcome the dilemma of sensing range and sensitivity, the interface was altered. A dual-layer interface was developed as a solution to this problem. This research demonstrates three different fiber-copper interfaces. They are chemically plated, copper painted and dual-layer interface. Chemically plated interface has chemical plated copper layer in the interface. Copper paint and additives make the interface porous in case of copper painted sample. Dual-layer interface is prepared by dip coating the fiber with wax and then copper paint. Annealing in testing process leaves carbon residue in the interface along with copper paint layer making it a dual-layer interface.

Testing was done to determine the effectiveness of the sensor. The testing setup includes a tube furnace (Lindberg/blue M TF55035A) for heating the samples while broadband source (BBS; Thorlabs ASE-FL7002C4) as a light source and optical spectrum analyzer (OSA; Ando AQ6317B) to record the reflected spectrum (Figure 3). The 50-50 coupler routes the light from BBS into the sensor and sends the reflected light to OSA. The samples

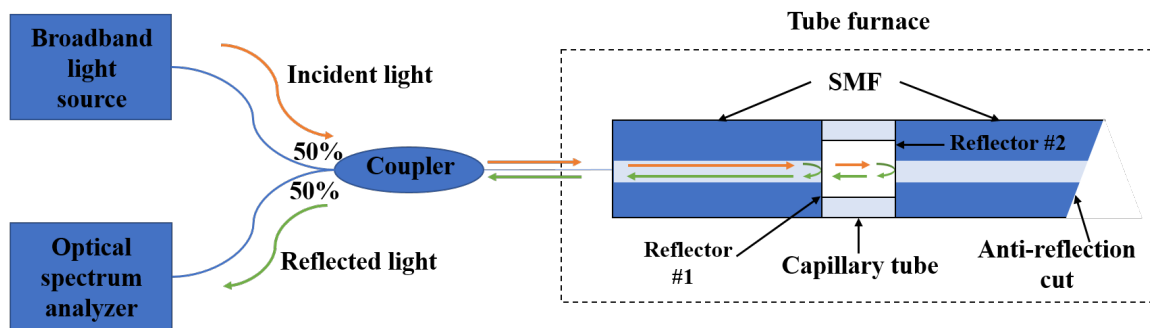


Figure 3. Schematic layout of the electrodeposition setup.

were annealed at 350°C for 15 minutes to remove residual stress and the additives to evaporate. After annealing, the variation of wavelength spectrum with temperature was recorded by heating the samples to 700°C.

3. RESULTS

EFPI sensors with around 50 microns of cavity length were fabricated and embedded into copper substrate. The whole fiber sensor was successfully embedded into the substrate by electrodeposition for 14 hours (Figure 4). Three different fiber-copper interface samples were prepared and tested along with bare fiber. The bare fiber spectrum was recorded and used as a reference.

The spectrum recorded by the Optical Spectrum Analyzer (OSA) is a sinusoidal wave at different wavelengths and corresponding power level (Figure 5). A peak was chosen and tracked its shift with the change in temperature.

The trends in the variation of the wavelength with temperature was analyzed for different fiber-copper interface (Figure 6). The bare fiber had a linear variation with a low sensitivity. The sensitivity in the chemically plated fiber was not uniform with an increase

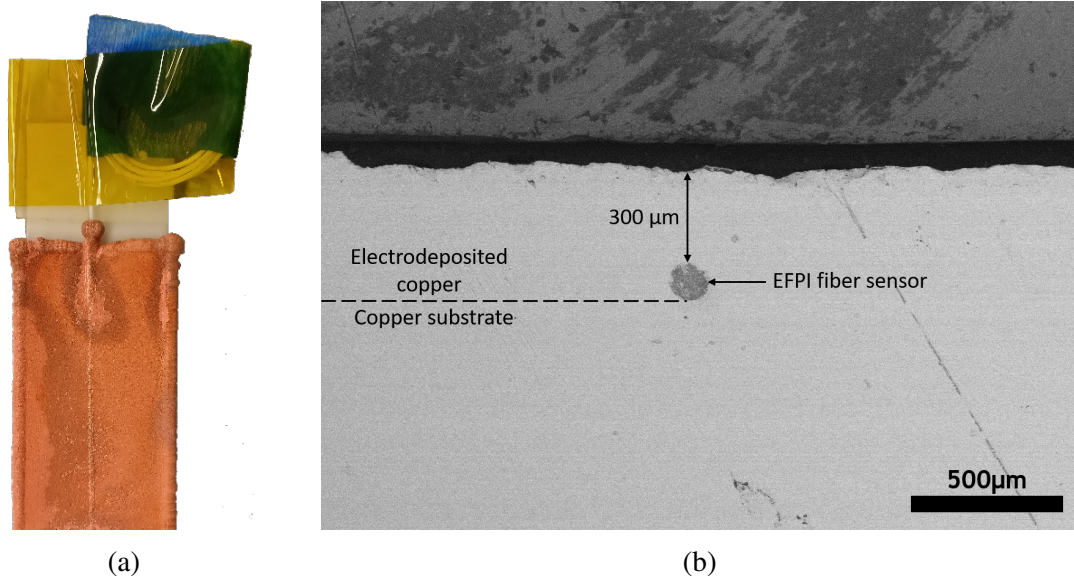


Figure 4. EFPI sensor embedded into copper substrate by electrodeposition. a. The sensor is fully embedded into the substrate by electrodeposition. b. SEM image showing the distance of embedded sensor from the surface.

and decrease in the slope and finally damaged at 600°C. Copper painted fiber initially had sensitivity similar to bare fiber but after 350°C it increased. Dual-layer interface had the sensitivity of the chemical plated fiber and maintained its linear sensitivity up to 700°C.

4. DISCUSSION

Sensitivity and sensing range are the parameters used for comparing different fiber-metal interface. Sensitivity is the change in output of sensor with respect to the change in input. On the other hand, sensing range is the maximum and minimum value of the parameter that can be measured. The sensitivity of EFPI sensor is the variation of wavelength with respect to the temperature.

$$Sensitivity = \frac{\Delta\lambda}{\Delta T} \quad (1)$$

Where $\Delta\lambda$ is the shift in wavelength and ΔT is the change in temperature.

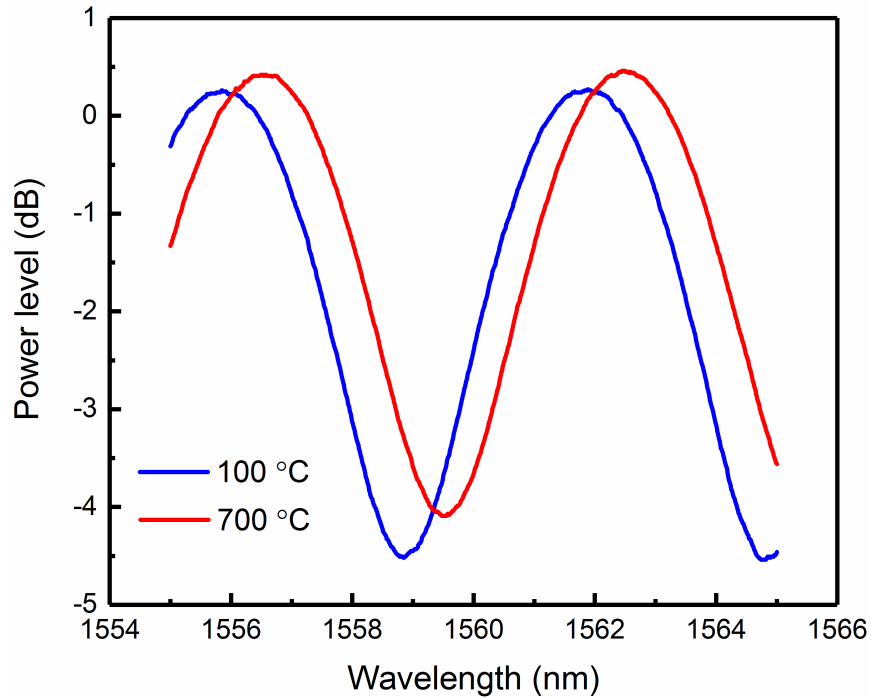


Figure 5. Spectrum recorded by Optical Spectrum Analyzer (OSA) for bare fiber at 100°C and 700°C.

Different interface has different mechanism in inducing stress on fiber from the metal. When there is a rise in temperature, the metal around the fiber expands and applies longitudinal stress as well as radial stress. There is no stress from metal in case of the bare fiber. Sensitivity for the bare fiber is solely because of the thermal expansion of the fiber (Figure 8a).

Chemically plated sample had high sensitivity of around 50 pm/°C. This can be attributed to the immediate chemical plated copper layer on the fiber. Hydroxylation of the fiber before chemical plating ensures copper-silica bonding. This bonding is evident from the high sensitivity of 50 pm/°C till 250°C. Thereafter, copper-silica debonding occurs due to the high strain on fiber due to the surrounding copper. The negative slope in the graph suggests that there is a decrease in cavity length because of debonding. The cavity length

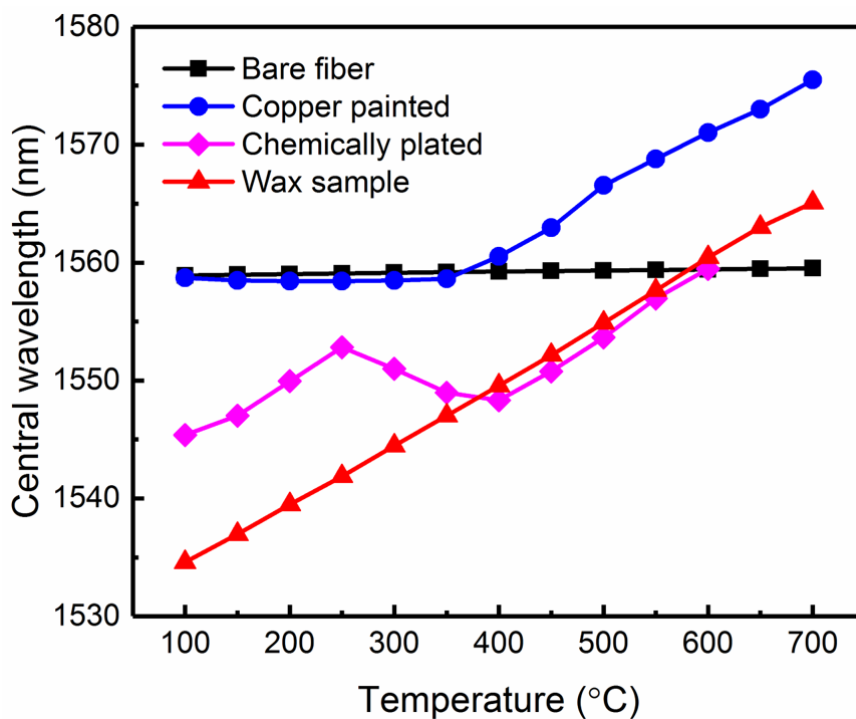


Figure 6. Variation of wavelength with temperature for different fiber-copper interface.

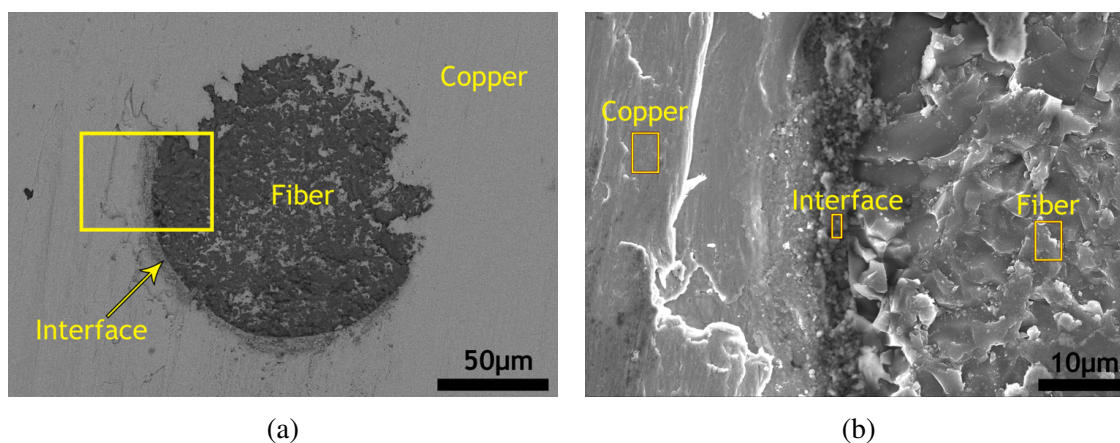


Figure 7. Scanning Electron Microscopy (SEM) images of the dual-layer interface.
 a. Wax interface with porous carbon and copper paint in the interface b. Close-up of the area highlighted in 7a. with yellow rectangle

starts to increase again from 400°C suggests that there was strain due to static friction between copper and fiber. At around 600°C, the noise in the signal suggested that sensor was damaged (Figure 8b).

Table 2. EDS analysis of the dual-layer interface

Element	Composition		
	Copper	Interface	Fiber
Carbon	2.53	32.02	4.46
Oxygen	0.80	6.54	35.14
Silicon	1.55	1.32	57.07
Copper	95.13	60.11	3.33

There was a porous copper layer in the interface for copper painted sample. As the temperature increased, the strain in the copper paint cannot be transferred to fiber due to the air gaps in copper paint. This continued till 350°C and then the strain from copper was induced directly into the fiber as the air gaps are closed (Figure 8c). The sensitivity increased from 2 pm/°C to 48 pm/°C because of the strain from the copper.

Dual-layer interface had high sensitivity of 45pm/°C and maintained its linearity even at 700°C. After annealing, the wax evaporates leaving carbon residue in the interface. The EDS analysis shows carbon residue and copper paint in the interface (Table 2). Dual-layer interface had two layers in the interface: carbon residue and copper paint. The sensitivity is almost same as that of chemical plated. Although this does not fail at 700°C, cavity length increase is higher than that of chemical plated suggesting the strain in the direction of fiber, longitudinal strain, is high. The sensor damage in the chemical plated sample was the result of high radial stress. In case of dual layer interface, the porous copper paint reduces the radial stress from the electrodeposited copper. This protects the sensor by decreasing the radial stress and leading to high sensing range. The carbon in the interface, bonds with silicon atom in the fiber and copper, applies longitudinal strain on the fiber with increase in temperature. This can be seen from the slope of dual-layer interface in the graph. This high longitudinal strain transfer results in high sensitivity in dual-layer interface.

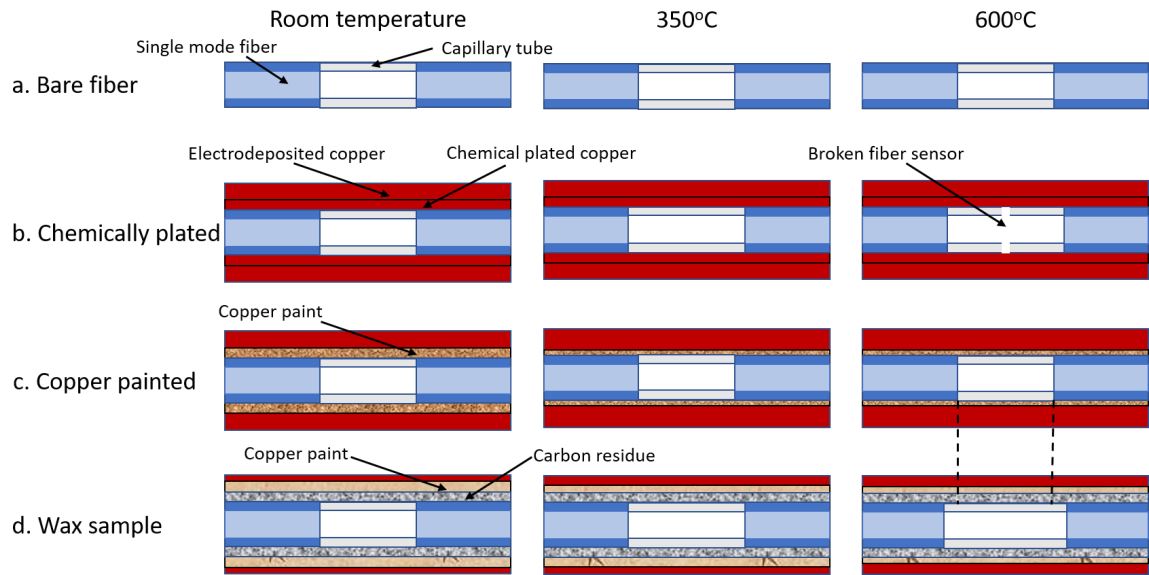


Figure 8. Schematic of the mechanism of the sensor at 350°C and 600°C for different fiber-copper interface.

5. CONCLUSIONS

EFPI sensors were fabricated and embedded into the copper substrate using electrodeposition. Sensing range and sensitivity of the embedded fiber sensors is determined by the fiber-metal interface. By developing a new interface, sensitivity and sensing range are enhanced. Dual-layer interface with one layer to apply longitudinal stress on the sensor and the other two reduce the radial stress has high sensitivity and high sensing range. Dual-layer interface had a linear and high sensitivity with high sensing range of 700°C with the carbon and copper paint layers in the interface.

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SECTION

2. APPLICATION: ALUMINUM CASTING WITH DUAL-LAYER INTERFACE

The high sensitivity and high sensing range of dual-layer interface was applied for aluminum casting. The casting setup involved a steel mold with embedded sensor as one of the faces (Figure 2.1). C-clamps were used to hold the mold in place. A thermocouple was placed in the mold as a reference. Aluminum was heated to 800°C in a furnace and transferred to the mold. The data from the sensor was recorded at 1Hz. A similar testing setup (Paper I, Figure 3) was used to record the data from the sensor.

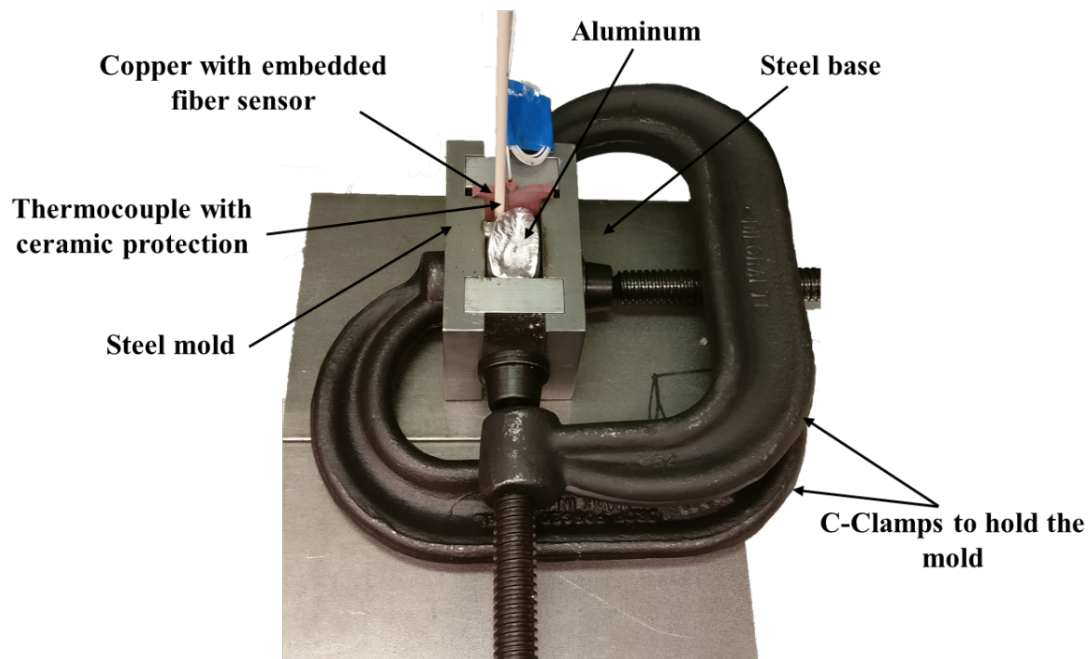


Figure 2.1. Casting setup used for aluminum casting.

The data from thermocouple and EFPI sensor was analyzed (Figure 2.2). The maximum temperature recorded by the thermocouple is around 600°C whereas 320°C for the EFPI sensor. This is because the thermocouple is close to the center of the mold. The

cooling curve of EFPI sensor is consistent with that of the thermocouple. The exponential decrease in the wavelength with time shows that the dual-layer interface retains the linearity even when subjected to a rapid change in temperature.

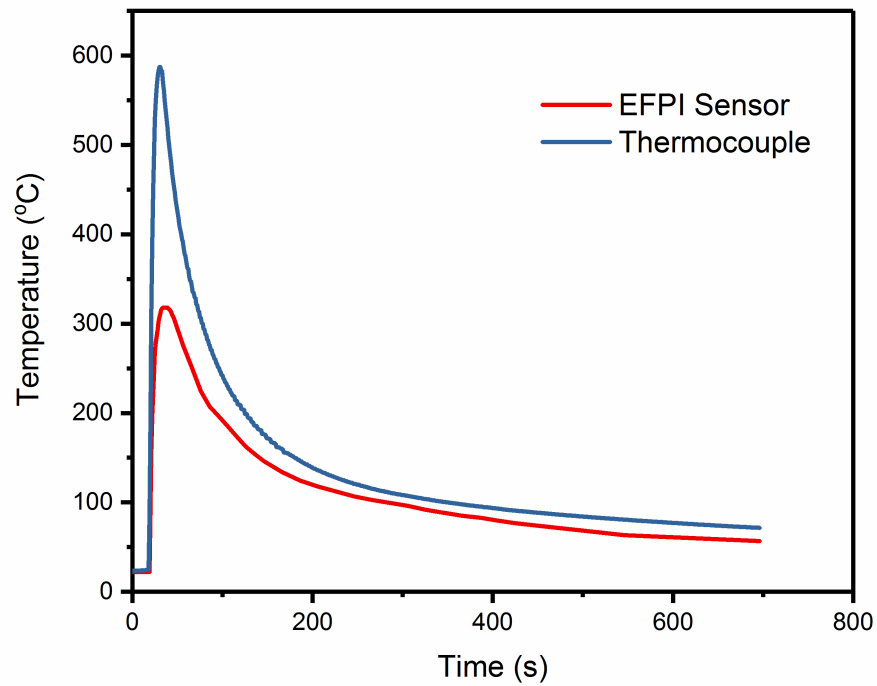


Figure 2.2. Variation of temperature data collected from EFPI sensor and thermocouple.

3. CONCLUSIONS

Embedding fiber sensors into metal can enhance the sensitivity of the sensor. Sensing range of the sensor is affected by the radial strain and sensitivity is due to the longitudinal strain. The dilemma of choosing either high sensitivity or high sensing range was solved by developing a dual-layer interface. This advanced embedding process with dual-layer interface ensured longitudinal strain in the fiber and at the same time minimizing the radial strain. The sensitivity of the dual-layer interface was 40 times higher than that of the bare fiber and survived a temperature of 700°C while maintaining the linearity. This can be attributed to the carbon bonding with the fiber and copper.

This dual-layer interface can be used to embed optical fiber sensors into metals for high temperature applications. Aluminum casting has been demonstrated in this work as an example. The data from the embedded sensor followed a similar trend to that of thermocouple. The embedded EFPI sensor effectively detected the rapid change in the temperature.

VITA

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