# Development of Zinc Coating Methods on Fiber Bragg Grating Temperature Sensors

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Abstract-To make practical use of the high-temperature superconducting (HTS) magnets, it is effective to detect a sign of failures by the temperature monitoring, because HTS magnets have temperature distribution when it is cooled by conduction cooling. Therefore, reliable multipoint temperature monitoring method is necessary. The optical temperature sensor can measure multipoint temperatures with a single fiber. The optical fiber is not affected by the fluctuation of magnetic field and has low heat invasions. A Fiber Bragg Grating (FBG) is a type of the optical fiber temperature sensor. When the FBG sensor thermally contracts, the refractive index of it changes and the wavelength of the light reflected from it changes. The thermal expansion rate of an optical fiber decreases at cryogenic temperature. Therefore, the accuracy of the measurement also decreases. It has been proposed to coat the FBG sensor with a metal or a resin to increase thermal contraction at cryogenic temperature. Zinc is suitable for the coating material, because it has high thermal expansion rate and its coating process is simple. Three types of zinc coating methods were evaluated in this research: sputtering, electroplating after sputtering titanium and copper. electroplating after electroless nickel plating. The production cost of these methods was evaluated. These zinc-coated FBG sensors were compared in the sensitivity at cryogenic temperature, the durability against vibration and thermal shock, the repeatability during the cooling cycle. We report the evaluation results of the zinc coating method on the optical fibers as a cryogenic temperature sensor.

*Index Terms*— Coatings, Cryogenics, Optical sensors, Superconducting magnet, Temperature sensors

## I. INTRODUCTION

Maglev [1]-[4], magnetic bearings [5]-[7], motors [8]-[10] etc. using superconducting magnets are being developed for practical application by many research groups throughout the world. The superconducting magnet is a very important component in these devices. If the magnetic function fails, the whole system will be affected. Thermometry of the whole surface of the superconducting magnet is effective to detect failures at an early stage.

This requires multiple-point measurement. When using conventional resistance temperature sensors, such as thermocouples, CERNOX or platinum sensors, multiple sensors and measurement lines are needed for multiple-point measurement. There is a problem that this in turn can cause heat intrusion in the superconducting magnet.

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When using an optical fiber temperature sensor, however, it is possible to measure temperatures of multiple points with one thread, as shown in Fig. 1. An optical fiber is nonconductive, and heat intrusion by an optical fiber is small. Therefore the optical fiber sensor has a smaller influence on cooling of the superconducting magnet than conventional sensors.

1



Although optical fiber temperature sensors for use at room temperature are already available on the market, the purpose of this study is to develop a sensor for use at cryogenic temperatures below 50 K (- $223^{\circ}$ C).

The optical fiber temperature sensor is classified by measuring methods, into a scattering type and a FBG (fiber Bragg grating) type.

The scattering type measures a thermal contraction by a wavelength change of the scattering light in the optical fiber. There are three kinds of scattering light that can measure thermal contraction: Brillouin scattering light, Rayleigh scattering light, and Raman scattering light.

In a past study, cryogenic measurement experiments were conducted to test the performance of a Brillouin scattering type optical fiber sensor and a Rayleigh scattering type sensor. A Raman scattering type was not tested because it cannot make measurements at cryogenic temperatures.

However, the scattering type optical fiber sensor has a demerit that its measuring equipment is very expensive. Therefore the FBG type sensor was adopted for experimentation. Temperature measuring experiments were performed by cooling a room to about 10 K, producing a satisfactory level of reproducibility. Therefore trials revealed that the sensitivity to wavelength shift fell at temperatures below 50 K, although it was possible to restore sensitivity by applying coating. [11-12]

Further trials were conducted to discover ways to enhance sensitivity by improving the optical fiber coating material and

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method. The thermometry of the superconducting magnet was also conducted. The results of these trials are reported in this paper.

# II. ENHANCING SENSITIVITY BY IMPROVING THE OPTICAL FIBER COATING MATERIAL AND METHOD

# A. Selection of the coating material

The principle of measurement of the FBG type is described below. At first, an optical fiber is processed by fiber Bragg grating. The refractive index changes at the FBG part, and the FBG part reflects the light of a specific wavelength. When the FBG part is cooled, the refractive index increases due to thermal contraction. And the wavelength of reflected light becomes small. From the change of the wavelength, the quantity of thermal contraction of the FBG part can be measured.

However, the optical fiber has decreased thermal expansion rate at cryogenic temperatures, and the sensitivity of the measurement also decreased. To improve sensitivity of the measurement, methods were proposed that coat the surface of the optical fiber FBG part and improve the sensitivity.

Past proposals to bond the stainless steel tube to the FBG sensor [13], included coating the FBG component with a metal such as aluminum, copper, lead or indium [14-15], and so on. The highest sensitivity was obtained with a sensor coated with both aluminum and indium. These studies however did not attempt to predict the change in FBG wavelength shift due to modification of the coating material properties.

This study therefore was undertaken to predict wavelength shift based on the thermal expansion coefficient and Young's modulus of the materials, and to examine a method to improve sensor sensitivity at cryogenic temperatures.

Results confirmed that the thermal expansion coefficient of the coating material influences sensitivity and that the rate of heat conduction influences the stability of the output.

Various materials to date have been tested as a coating material, enabling the confirmation of their characteristics. The sensitivity of acrylic resin-coated optical fiber was about 6.4 times higher than optical fiber without coating. Nevertheless, temperature differences between the object of measurement and the FBG component, and measurements were unstable, due to low thermal conductivity of the acrylic resin.

The acrylic resin was coated with metal to improve thermal conductivity. Nickel and copper were tried, since they have high thermal conductivity. Tests showed stabilized measurements but the metals had a low thermal contraction rate which interfered with the thermal contraction of the acrylic resin, which in turn meant that sensitivity did not improve with either nickel or copper.

When employing an optical fiber as a thermal sensor, measurement stability is a prerequisite. Consequently another metal with high thermal conductivity was selected as a coating material and tested. In each case where the optical fiber was coated with nickel or silver, sensitivity did not improve. With copper however, sensitivity improved by about 1.8 times compared to optical fiber without coating. [14-15]

In this paper, zinc was selected as the coating material, because of its superior thermal expansion properties compared to copper, and because the coating process is simple.

## B. Selection of the coating method

There are two methods for applying a zinc coating: sputtering and electroplating. Two samples were prepared using each method. These samples were then tested and compared to determine their influence on sensitivity.

The sputtering method applies zinc which then adheres directly to the FBG part of an optical fiber.

Three kinds of metal were used in the electroplating method. After sputtering titanium first to enhance zinc adhesion to the FBG part of an optical fiber, copper was then used as a pole in the plating process. Finally, the optical fiber was electroplated with zinc.

## C. Experimental methods

Two optical fiber sensors each treated with one of the above measures were placed on a cooling plate in the cryogenic equipment. Resistance temperature sensors were also placed on the same plate for comparison.

A vacuum was formed inside the cryogenic equipment, then it was cooled with a cryocooler from room temperature down to about 10 K. The cryocooler was then stopped and the equipment was left to return to room temperature naturally.

While the equipment returned to room temperature after cooling, the temperature and wavelength of the coated FBG component were measured.

# D. Results of the experiments

Fig. 2 shows the experimental results.

The sensitivity to temperature of the optical fiber coated by sputtering fell in comparison to non-coated optical fiber.

The sensitivity to temperature of the electroplated optical fiber increased by about 3 times compared to non-coated optical fiber, and the FBG wavelength shift remained stable with the changes in temperature. This showed the best sensitivity in coating materials of the optical fiber which was examined so far.



Fig. 2. Temperature and wavelength shift at the FBG part

## E. Analyses and discussions about the sputtering method

Touching the sputtered surface after coating left traces of powdered zinc on the fingers. It is thought that this is due to the surface of the zinc being rough and porous, and thus easily detached. Consequently interfacial peeling occurred during the cooling and causing the coating to lose its effectiveness.

The worsening of sensitivity of the coated fiber compared to non-coated fiber is thought to be due to the interfacial peeling, which meant that the coating inhibited the thermal contraction of the FBG component.

# F. Analyses and discussions about the electroplate method

An optical fiber sensor made by the electroplate method showed the best sensitivity in coating materials which was examined.

It is thought that zinc adherence to the optical fiber, thereby fixing it in place, can be achieved by sputtering titanium as a foundation. Furthermore, it is deemed that sensitivity improved because the coating layer thickness could be made thicker through electroplating than with the sputtering method. Zinc has high thermal expansion, and copper has high thermal conductivity. It was thus assumed that coating the optical fiber with layers of these metals could stabilize output and sensitivity.

# III. THERMOMETRY IN THE THERMAL SIMULATOR

The optical fiber sensor studied in this report is intended to be used in the thermometry of the superconducting magnet. This report confirmed the ability of the optical fiber sensor to measure the temperature of the conduction cooling superconducting magnet.

A thermal simulator was used which has a conduction cooling simulation coil made with stainless steel and which can reproduce the distribution of temperature in the cryostat. Experiments were performed to test the performance of the electroplated optical fiber sensor.

In addition, the performance of a Brillouin scattering type sensors and a Rayleigh scattering type were tested at the same time, and evaluated for the ability to measure the temperature of the conduction cooling superconducting magnet.

Both of scattering type sensors used normal optical fiber without zinc coating, and measured the frequency shift of each scattering light that was caused by cooling.

#### A. Experimental methods

Fig. 3 shows the temperature measuring points of a optical fiber temperature sensor in the thermal simulator.

The figure illustrates the installed electroplated optical fiber sensors, Brillouin scattering type sensors, Rayleigh scattering type sensors and a CERNOX thermometer to measure the temperature of the imitation coil surface (about 1210 mm in width and about 610 mm in height) and the refrigeration machine cold head.

This set of sensors was then cooled in a refrigerator and the

#### temperature was measured.



Fig. 3. Thermometry experiment using the thermal simulator

## B. Experimental result

Fig. 4, 5 and 6 show the experimental results.

These figures show the relationship between the temperature and the wavelength shift of the FBG part or the frequency shift of the scattering type at the coil surface measuring point, in the cooling process from about 290 K to about 60 K.

It was confirmed that each type of optical fiber sensor, namely the electroplated type, Brillouin scattering type and Rayleigh scattering type, was able to measure the temperature of the conduction cooling coil surface without any problems, in the range from about 290 K to about 60 K.



Fig. 4. Temperature and wavelength shift at the coil surface measuring point (the electroplating method type optical fiber sensor)





#### IV. DURABILITY EVALUATION EXPERIMENT

For the electroplating type, the ability of thermometry in the cryogenic and the ability of thermometry of the conduction cooling coil surface was confirmed.

If an optical fiber sensor is really used for a measurement as a sensor, durability against thermal fluctuation and vibration is necessary. Therefore durability evaluation experiments were conducted.

# A. Experimental methods

Three kinds of experiments were conducted in the durability evaluation experiments. The experiment used three optical fibers, one for each experiment.

At first, an electroplating type optical fiber sensor was placed on a cooling plate in the cryogenic equipment. Cooling from room temperature to 16 K was repeated fifteen times.

Then, the optical fiber was soaked into liquid nitrogen and cooled rapidly from room temperature to 77 K three times.

Finally, the optical fiber was placed on a vibrator and vibrated. The acceleration of vibration was selected as 150  $m/s^2$  based on an actual acceleration of a running train vehicle. The experiment time was selected as 800 hours. It was based on operative time and maintenance cycle of a railway vehicle.

After the experiment was conducted, abnormalities such as

disconnection of the optical fiber and detachment of the electroplating coating were investigated.

B. Experimental results

Fig. 7 and 8 show the experimental results.



Fig. 7. Temperature characteristics of wavelength for repeated cooling



Fig. 8. Time history of the wavelength for rapid cooling

After the vibration experiment, the optical conduction of the sensor was investigated. And a liquid nitrogen thermometry experiment was conducted. As a result, it was confirmed that the ability of the sensor was maintained even when an optical fiber sensor was vibrated.

# C. Analyses and discussions

Repeated cooling, rapid cooling, and vibration cause the detachment of coating and disconnection of the optical fiber. Experiments that increase these conditions were conducted and the durability of the electroplating coating type optical fiber sensor was evaluated.

The optical fiber sensor output the normal wavelength in all processes of the experiments. And there were no abnormalities on surface, such as peeling.

From these results, it was confirmed that the electroplating coating type optical fiber sensor maintained the ability of the temperature sensor even under repeated cooling, rapid cooling, or vibration.

# V. IMPROVEMENT OF THE ELECTROPLATING METHOD

As a zinc coating method, the electroplating method was proposed. This method requires an exclusive device and many man-hours for sputtering of titanium and copper. The production cost of this method is equal to CERNOX or platinum sensors.

For production cost reduction, we studied an electroless plating method which was a low cost method of coating metal instead of sputtering. [16]

A combined plating method was proposed, in which zinc was coated by electroplating after electroless plating of nickel. A prototype optical fiber sensor was made by the combined plating method, and the experiment that evaluates its sensitivity was conducted.

## A. Trial of the combined plating method

The optical fiber cannot be electroplated, because it is an insulator. The electroless plating method is a plating method that can plate metal to an insulator. In the method, a plating object is soaked in a reducing agent, and a nickel film precipitates it by electrons which are released by oxidation reactions.

Using this method, nickel was plated to a normal optical fiber. Because the surface of the optical fiber was smooth, nickel did not adhere to the surface, and peeling occurred as in the sputtering method.

Then, an optical fiber was etched before plating to increase the coefficient of friction. After performing etching with an alkaline solution, nickel was able to adhere to the surface by electroless plating.

However, silicon which is a chief ingredient of the optical fiber dissolves in alkaline solution. It was revealed that the optical fiber dissolved in solution and became thinner by this method. Furthermore, the optical fiber dissolved when the alkalinity of the solution was high.

When an FBG part is eroded by alkaline, the function of the sensor may disappear. An optical fiber was etched with an acid solution which does not dissolve the silicon. The coefficient of friction was improved without dissolving the silicon by adjusting the ingredients of the solution.

A prototype optical fiber sensor was made by this method; the FBG part was etched with acid solution, coated with nickel by electroless plating, and coated with zinc by electroplating. The prototype was soaked in liquid nitrogen and cooled rapidly. The coating did not peel and there were no abnormalities on the surface and no abnormal output.

However, the electroless plating method has a problem in which the thickness of nickel is easy to become non-uniform. Some prototypes of electroless plating were made, and the most uniform one was coated with zinc.

Fig. 9 shows a prototype optical fiber sensor made by the combined plating method.

# B. Experimental method

The prototype optical fiber was cooled from about 280 K to 16 K, and the shift of the wavelength of its FBG part was measured.

5



Fig. 9. A temperature detection part of an optical fiber sensor (made by the combined plating method)

## C. Experimental result

Fig. 10 shows the experimental result.

It was confirmed that the combined plating type optical fiber sensor could measure the temperature without any problems in the range from about 280 K to about 16 K.



## D. Analyses and discussions

The combined plating method was proposed, and it was confirmed that the optical fiber sensor made by this method can measure cryogenic temperatures.

The combined plating method can make an optical fiber sensor at low cost. However, as shown in Fig. 11, the thickness of nickel coating easily becomes non-uniform in the electroless plating process.

This is the problem of the combined plating method, because the non-uniformity causes dispersion between sensors.

We will improve the accuracy by adjusting pH and soaking time. A new prototype will be made by the improved method, and a durability experiment will be conducted.



Fig. 11. The rejected prototype of the electroless plating

## VI. CONCLUSIONS

The optical fiber temperature sensor that can measure cryogenic temperatures was produced by the electroplating method that coats zinc by electroplating after sputtering titanium and copper to the FBG part of an optical fiber.

The sensitivity to temperature of the optical fiber sensor coated by sputtering fell, because interfacial peeling occurred during the cooling.

The electroplating method that coats zinc by electroplating after sputtering titanium and copper showed the best sensitivity which was experimented so far. It is thought that zinc adherence to the optical fiber can be achieved by sputtering titanium as an under coating.

Experiments were performed to measure temperatures of the conduction-cooled superconducting magnet. An electroplating type optical fiber sensor, a Brillouin scattering type sensor, and a Rayleigh scattering type sensor were tested. It was confirmed that each type of optical fiber sensor could measure the temperature of the surface of the conductioncooled coil without any problems, from about 290 K to about 60 K.

Durability evaluation experiments of the electroplating type optical fiber sensor were conducted. It was confirmed that the electroplating type optical fiber sensor maintained the ability of the temperature sensor even under repeated cooling, rapid cooling, and vibration.

For production cost reduction, the combined plating method that coats zinc by electroplating after electroless plating of nickel was proposed. The experiment that evaluates the ability of thermometry was conducted.

It was confirmed that the optical fiber sensor made by this method has the ability of thermometry at cryogenic temperatures. Accuracy of the thickness is the problem of this method, because the non-uniformity causes dispersion. We will improve the accuracy of the thickness.

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