

# RECENT RESULTS FOR STUDY OF CERAMIC AND COPPER PLATING FOR POWER COUPLERS

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## Abstract

KEK has conducted a survey to select an optimum ceramic after withdrawal by a domestic manufacturing company two years ago. For this selection, there are four important items on the properties of ceramic; that is, relative permittivity, dielectric loss tangent, surface and volume resistance, and secondary electron emission coefficient. For measurements of these parameters, ten kinds of ceramic samples supplied from five companies were measured using three kinds of measurement systems. For measurement of secondary electron emission, scanning electron microscope (SEM) with beam blanking system was used. On the other hand, residual resistivity ratio (RRR) for copper plating, which is the most important item for quality control, has also been carried out by collaboration with KEK-CEA. In this report, the recent results for these studies will be presented in detailed.

## INTRODUCTION

In the E-XFEL, ESS, LCLS-II, etc. which have been constructed (or under construction) in recent years, several hundred superconducting cavities are used, and the quality control of the power coupler has become important. In particular, the manufacturing cost of copper plating covering the inner surface of the power coupler, ceramics used for RF windows, and titanium nitride coating (TiN coating) deposited on the surface of ceramic is also a large proportion of cost, which are the main items for this R&D. R&D for power coupler is also included in the framework of cost reduction R&D for the International Linear Collider (ILC) concluded between KEK and the US Fermi National Laboratory (FNAL) in 2017. Currently, various investigations and studies under the cooperation with affiliated companies, are being advanced at KEK [1].

## SURVEY AND RESEARCH ON COPPER PLATING

The quality control of copper plating is an item which became a problem frequently also in E-XFEL [2], which is still an important matter in the other projects under construction. The quality control is generally evaluated by RRR measurement. RRR measurement has been carried out in KEK/COI by using small refrigerator with conduction cooling method. Figure 1 shows the measurement system (upper) including the small cryostat in the COI building in KEK, and the stage (lower) for placing the sample. Four samples can be mounted at the same time and can be

measured with different electric current values (for example, three stages such as 1 mA, 10 mA, 100 mA). The electric current is bi-directionally loaded and the average resistance value is calculated. After the preliminary tests, the optimum electric current value was found to be 1 to 10 mA. And, the optimum sample size is fixed to 150 mm x 5 mm x 0.5 mm, because this system uses the conduction cooling method, and too large sample cannot be cooled.

As a way of evaluating the RRR of the copper plating, after measuring the resistance to a copper plating sample, the copper plating is peeled off and returned to the base material SUS316L, and resistance measurement is performed again. As the final step, by estimating with the ratio of the two measured values of room temperature and low temperature, the net RRR for copper plating is obtained. With respect to the SUS316L material, it is not necessary to peel off for all the copper plating samples in order to take almost the same resistance values.

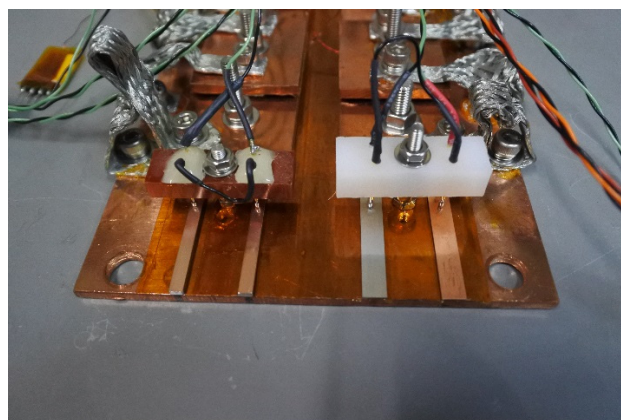


Figure 1: Residual Resistivity Ratio (RRR) measurement system (top) and four samples on the stage (bottom) in KEK/COI.

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After several commissioning tests, four copper plating samples were made and RRR measurements were made. As for two of these, it was found that the RRR changed remarkably from 15 to 75 as a result of measuring several times with the time being shifted. Since there was a possibility that a malfunctioning occurred in the measurement system, two samples were sent to IRFU/CEA in France and similar RRR measurements were carried out there, and the same result was obtained, therefore this phenomenon is not related to the measurement system. This result is still under investigation. Figure 2 shows the correlation plot between temperature and resistance of copper plating and SUS316L samples. Blue point shows the first result, and green one the second (three months later) for the same copper plating sample. Between two measurements, this sample was in desiccator with low humidity. SUS316L sample has the same result for every sample in every measurement.

Recently, new copper plating samples, fabricated by electroforming method, are ready for RRR measurement. It is possible to obtain the resistivity for copper plating directly. As a next step for this R&D, the temperature dependence and the impurity dependence on copper plating will be investigated in detailed.

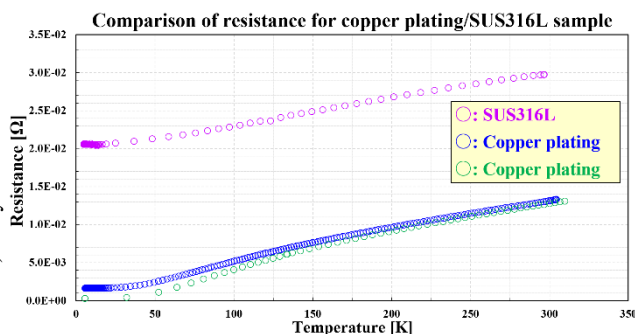


Figure 2: Correlation plot between resistance and temperature for copper plating and SUS316L samples. There are two data for one copper plating sample between different measurement periods.

## SURVEY AND RESEARCH ON CERAMIC

Surveys and research on ceramics are carried out on the following four items.

- Surface/volume resistance ( $\rho$ )
- Relative permittivity ( $\epsilon$ )
- Dielectric loss tangent ( $\tan\delta$ )
- Secondary electron emission coefficient ( $\delta_{SEE}$ )

Measurement of secondary electron emission coefficient is under progress and 10 types of samples were offered from 5 companies. For the measurement of  $\delta_{SEE}$ , it is common to use a scanning electron microscope (SEM). Moreover, it is essential to use a pulse beam, because ceramic is an insulator. For the generation of a pulse beam, beam blanking system (like a kicker magnet) was installed into SEM in KEK/COI. The following items show the specification for measurement system.

- Scanning electron microscope SU3500 (HITACHI)

- Cathode voltage: 0.6 to 30 kV (measured 4 times for each voltage)
- Use of beam blaker (for pulse beam generation)
- Use of pulse beam with pulse width of 1 msec
- Use of bias voltage (primary current: +50V, absorption current: -50V)
- Measuring with one pulse per point (avoidance of charge-up)
- Carbon target used for primary beam current measurement
- Area injection (avoidance of charge-up) for ceramics and point injection for a carbon target
- Current amplifier (KEITHLEY)

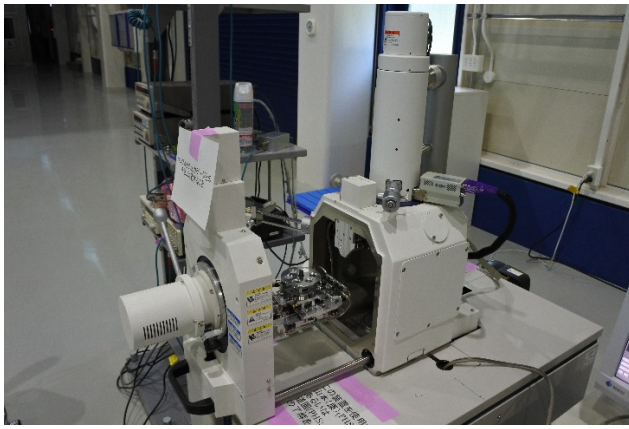
Figures 3 (a) and (b) show the SEM, and the sample holder where the sample is placed. On the stage, in addition to ceramic samples, carbon target for primary beam current measurement are placed together. In addition, the carbon target is provided with a non-penetrating hole with a diameter of 1 mm, and the beam is incident by the point injection. Figure 3 (c) shows the schematic view of measurement system including SEM, and observed signals for primary beam current on carbon and absorption current on ceramic. From the difference between the primary beam current and the absorption current, a reflected current including secondary electrons is obtained, and the  $\delta_{SEE}$  can be obtained by taking the ratio to the primary beam current. Figure 3 (d) shows the  $\delta_{SEE}$  of a ceramic sample. For ceramics, measurements are made four times at the same cathode voltage, and the average value is obtained, as shown by red line in Figure 3 (d). This result is in well-agreement with the previous result, therefore this means that the preliminary measurement for secondary electron emission coefficient was successfully done. At present, several ceramic samples were already measured. In near future, the comparison among 10 types of ceramic, the comparison between with and without TiN coating, the comparison among various rinsing methods including ultrapure water, ethanol, ultrasonic, and ozonized ( $O_3$ ) water will be done.

With regard to the other properties of ceramic, for one part of many samples, relative permittivity and dielectric loss tangent was measured by a common measurement device. Moreover, surface/volume resistivity was also measured by using two measurement devices. Finally, it turned out that one of two is more reliable. In this fiscal year, these tests will be completed.

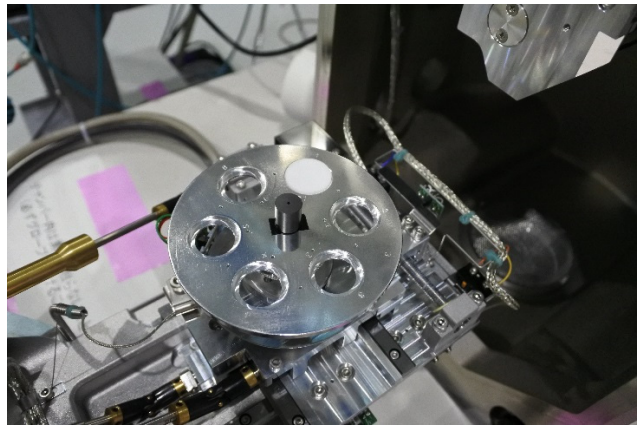
## CONCLUSION

For the cost reduction R&D for power coupler in ILC, the survey and the research for copper plating and ceramic property have been continued from 2017. As for copper plating, RRR measurement is under progress with collaboration among KEK, Nomura plating and IRFU/CEA. In KEK/COI, SEM with beam blanking system for the measurement of secondary electron emission coefficient was commissioned successfully, and several ceramic samples were already measured. New results will be presented in IPAC19 or SRF19.

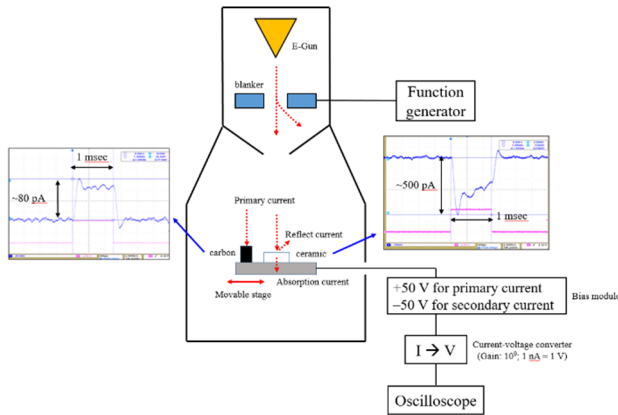
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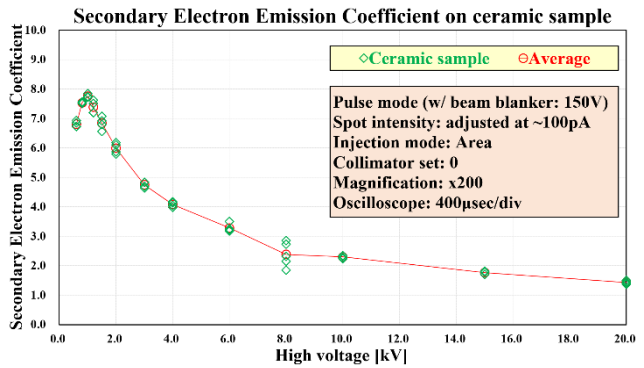
(a) Scanning electron microscope



(b) Sample holder



(c) Schematic view of measurement system



(d) Secondary electron emission coefficient for ceramic

Figure 3: (a) Scanning electron microscope (SEM) (b) Sample holder (c) Schematic view of measurement system (d) Secondary electron emission coefficient for ceramic.

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