ISBN: 978-3-95450-191-5 doi:10.18429/JACoW-SRF2017-MOPB061

QUALITY CONTROL OF COPPER PLATING IN STF-2 INPUT POWER COUPLERS

E. Kako[1, 2 †], R. Ueki[1], H. Kutsuna[3], J. Taguchi[3], K. Okihira[4], K. Sennyu[4], H. Umezawa[5], F. Saito[5], K. Tetsuka[6] and O. Yushiro[6]
[1] KEK, High Energy Accelerator Research Organization, Tsukuba, Ibaraki, Japan
[2] SOKENDAI, The Graduate University for Advanced Studies, Hayama, Kanagawa, Japan
[3] Nomura Plating Co. Ltd., Osaka, Japan
[4] Mitsubishi Heavy Industries Mechatronics Systems Ltd., Kobe, Japan
[5] Tokyo Denkai Co. Ltd., Tokyo, Japan
[6] Toshiba Electron Tubes and devises Co. Ltd., Ohtawara, Japan

Abstract

Purity of thin copper plating used for input power couplers in a superconducting cavity system is one of important characteristics for considering thermal losses at low temperature. Various samples of thin copper plating on stainless sheets were fabricated by four venders with their own plating techniques. The RRR values of the samples with different thickness of copper plating were compared in the condition before and after heat treatment at 800ºC in a brazing furnace. Deterioration of the RRR was observed in all of samples after heat treatment. The results of the RRR measurements and sample analysis of impurities are reported in this paper.

INTRODUCTION

The primary role of a high power input coupler is to transfer RF power from a generator to a cavity and beams. High power input couplers are one of the most critical components of a superconducting RF cavity system and includes varieties of key technologies in design, fabrication, conditioning and operation. Many types of high power couplers for CW and pulsed operation were fabricated, and the RF conditioning was carried out at KEK, [1, 2]. Reduction of both thermal loads, which consists of a dynamic loss due to RF losses and a static loss due to heat leak from outside, is an important consideration in input couplers. In this view point, quality control of Cu-plating of input couplers has a significant role for supressing thermal losses. Purity and thickness of Cu-plating are essential parameters for the fabrication process of input couplers. Heat treatment at 800ºC for brazing gives a remarkable influence in property of Cu-plating. Investigation for quality control of Cu-plating is discussed in this paper.

DYNAMIC LOAD OF INPUT COUPLER

International cryomodule test named S-1 Global [3] in 2010 was aimed to compare each performance of different components included in a cavity package. One of the purposes in S-1 Global was to compare the performance of input couplers between TTF-III couplers [4] developed by DESY and STF-2 couplers [5] developed by KEK.

† eiji.kako@kek.jp

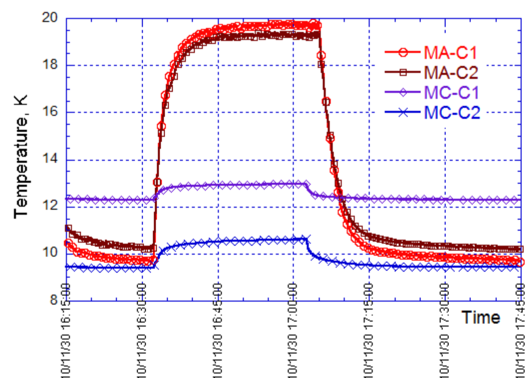

Figure 1: Temperature rises at connection flanges of input couplers at the condition of detuned 32 MV/m: MA-C1 and MA-C2 show the temperature at STF-2 couplers (red lines). MC-C1 and MC-C2 show the temperature at TTF-III couplers (blue lines) [6].

Table 1: Measured dynamic heat loads in a single cavity operation; a total dynamic heat load of a cavity and a coupler ($P_{loss-(Total)}$), a dynamic heat load of a coupler under a detuned condition ($P_{loss-(Detune)}$) and a net dynamic heat load of a cavity ($P_{loss-(Cavity)} = P_{loss-(Total)} - P_{loss-(Detune)}$) [6].

Cavity	Z-109	AES-004	MHI-07	MHI-06
Coupler	TTF-III	TTF-III	STF-2	STF-2
Eacc [MV/m]	28	25	32	32
$P_{loss-(Total)}$ [W]	0.8	1.4	2.8	2.6
$P_{loss-(Detune)}$ [W]	0.1	0.2	0.7	1.2
$P_{loss-(Cavity)}$ [W]	0.7	1.2	2.1	1.4
Q_0	8.8 x 10^9	4.3 x 10^9	4.3x 10^9	6.5 x 10^9

Figure 1 shows a comparison of temperature rises due to dynamic RF losses of TTF-III and STF-2 couplers [6]. The temperature rise was less than 1 degree in the TTF-III couplers and around 10 degrees in the STF-2 couplers. Measured results of the dynamic heat loads in the cavity equipped with each coupler are summarized in Table 1. The dynamic heat load of a coupler is shown by

ISBN: 978-3-95450-191-5 doi:10.18429/JACoW-SRF2017-MOPB061

$P_{loss\text{-}(Detune)}$, and it was found that the STF-2 couplers had much higher heat load than that of the TTF-III couplers. Two experimental results of temperature rises and heat loads confirmed that the STF-2 coupler has a high heat production and a low thermal extraction through thermal anchor. These were a clear motivation to check a quality of Cu-plating in the STF-2 coupler.

ADHESION PROBLEM OF CU PLATING

Adhesion problems of Cu-plating occurred during the fabrication process of the STF-2 input couplers [7]. Many severe swellings and micro-projections were observed

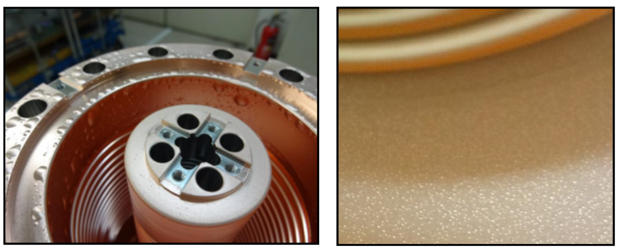

Figure 2: Swellings (left) and micro-projections (right) of Cu-plating after heat treatment at a brazing temperature.

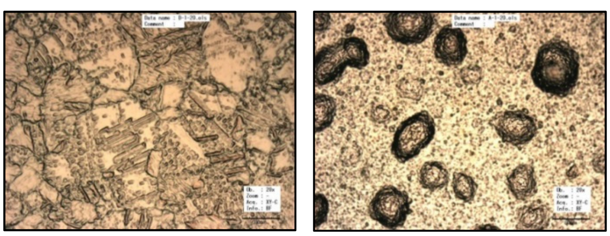

Figure 3: Surface condition of Cu-plating with a normal smooth surface (left) and many micro-projections (right), after heat treatment at a brazing temperature.

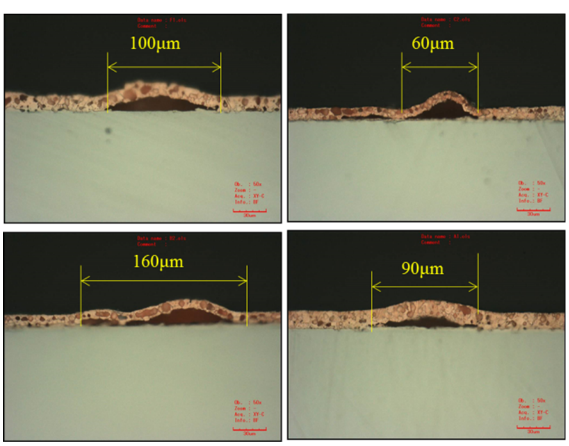

Figure 4: Cross-section of four micro-projection sites in Cu-plating (20 μm) and Ni strike-plating (0.5 μm) on a stainless steel plate (SUS316L).

after the brazing at 800°C, as shown in Figure 2. The surface condition and cross-section of micro-projection were shown in Figures 3 and 4, respectively. The cause of these adhesion problems were investigated by sample analysis. As the consequence, contamination by an aged solution of a plating acid or incorrect surface finishing before plating was found as the responsible reasons. Therefore, use of a fresh plating acid and a careful surface cleaning procedure of a plating area were applied for solving this problem.

SAMPLES FOR RRR MEASUREMENT

Measurements of a residual resistivity ratio (RRR) are useful for evaluating a purity of Cu-plating, because a purity of Cu-plating has a strong relation with a thermal conductivity and an electrical resistivity. Several Cu-plating samples for the RRR measurements were fabricated by four companies, which have different plating technologies, as shown in Table 1. Here, the samples were prepared by different parameters like a thickness of Cu-plating, a material for strike plating and an acid solution for Cu- plating. Figure 5 shows the Cu-plating samples for the RRR measurements. The size of a sample plate made of stainless steel (SUS316L) is a length of 100 mm, a width of 5 mm and a thickness of 1 mm. The sample plates of SUS316L were fabricated by wire-cutting to avoid a thermal influence.

Precise measurement of a thickness of Cu-plating is important for evaluation of the RRR values. Figures 6 and 7 shows the cross-section of Cu-plating samples measured by a digital microscope and a laser microscope.

Table 2: Cu-plating Samples for RRR Measurements from Four Venders [7–10]

Sample	Plating vender	Target thickness	Measured thickness	Strike plating	Solution for copper-plating
A	X	20 μm	15.9 μm	Au	Copper-Pyrophosphate
B	X	5 μm	5.9 μm	Au	Copper-Pyrophosphate
C	X	20 μm	20.7 μm	Ni	Copper-Pyrophosphate
D	X	5 μm	3.9 μm	Ni	Copper-Pyrophosphate
E	Y	5 μm	4.1 μm	Ni	Copper-Cyanide
F	Y	20 μm	6.8 μm	Ni	Copper-Cyanide
G	Z	10 μm	10.7 μm	Ni	Copper-Cyanide
H	Z	35 μm	29.1 μm	Ni	Copper-Cyanide
I	V	10 μm	13.7 μm	Ni	Copper-Sulfate
J	V	20 μm	19.3 μm	Ni	Copper-Sulfate
K	V	30 μm	31.1 μm	Ni	Copper-Sulfate

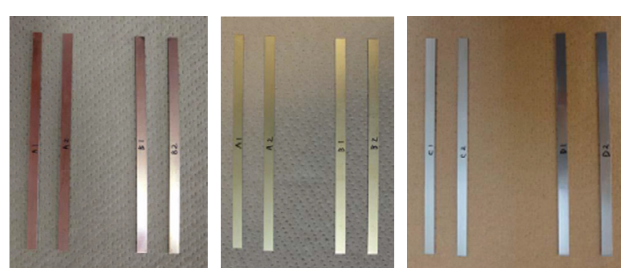

Figure 5: Cu-plating samples with a different Cu-plating thickness (left), samples with Au strike-plating after removing Cu-plating (centre), and samples with Ni strike-plating after removing Cu-plating (right).

ISBN: 978-3-95450-191-5 doi:10.18429/JACoW-SRF2017-MOPB061

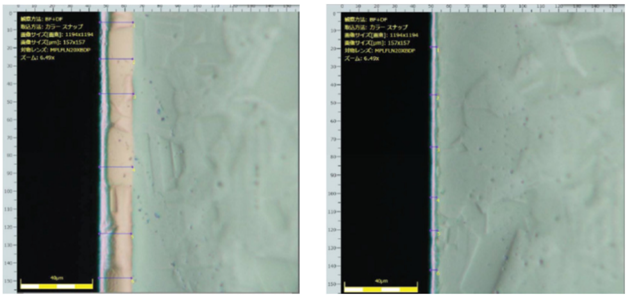

Figure 6: Cross-section of Cu-plating on SUS316L measured by a digital microscope. The thickness of Cu-plating is 18 μm (left) and 3 μm (right) [11].

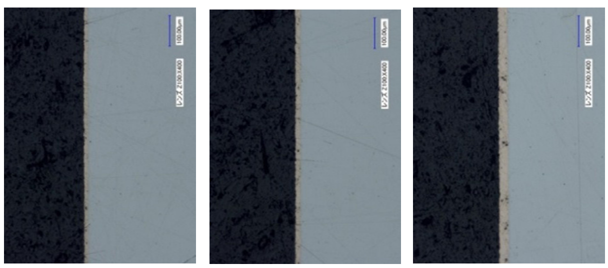

Figure 7: Cross-section of Cu-plating on SUS316L measured by a laser microscope. The thickness of Cu-plating is 14 μm (left), 19 μm (centre) and 31 μm (right) [10].

RRR MEASUREMENT SYSTEM

A measurement system for RRR of Cu-plating is shown in Figure 8 (left). Four cables connected with four terminals were soldered on a sample plate, as seen in Figure 8 (right). A constant DC current is supplied from both sides of the sample, and a voltage induced by the constant current is measured by a digital voltmeter to estimate the resistivity of the samples. The samples were inserted into a small cryostat and were cooled down from a room temperature to 4.2 K by liquid helium. An example of the measured resistivity as a function of temperature is shown in Figure 9. The results of Cu-plating samples with a different thickness are shown in Figure 9 (top). It is known that a thin Cu-plating layer on a substrate is easily removed by a nitric acid. After the Cu-plating on the sample plate were removed, a resistivity of the remaining stainless steel plate (SUS316L) was similarly measured as shown in Figure 9 (bottom). Values of RRR of Cu-plating were calculated by a simple parallel model as shown in Figure 10. Here, the resistivity of only Cu-plating was determined by a difference of the resistivity of Cu-plating on SUS316L in Figure 9 (top) and the resistivity of only SUS316L in Figure 9 (bottom). Finally, the RRR of Cu-plating was obtained by a ratio of the resistivity at room temperature and that at 4.2K.

EFFECT OF HEAT TREATMENT

A ceramic disk window of the STF-2 input coupler was joined with coaxial parts by brazing at 800^{o}C. Therefore, Cu-plating of the cold outer conductor of input couplers was also heated up to 800^{o}C during fabrication process.

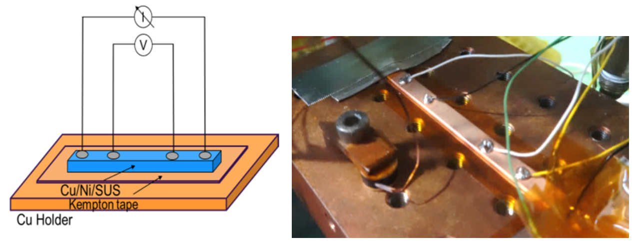

Figure 8: RRR measurement system with four terminals for Cu-plating samples.

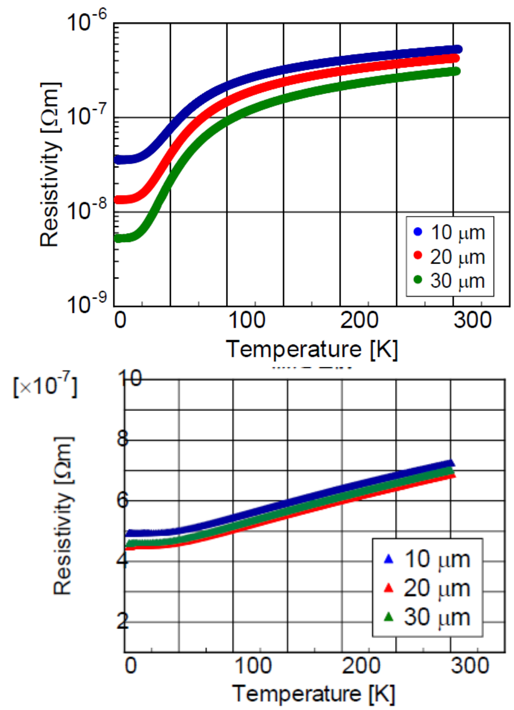

Figure 9: Temperature dependence of the resistivity of Cu-plating with 10, 20 and 30 μm thickness on SUS316L (top), and temperature dependence of the resistivity of SUS samples after removing Cu-plating (bottom) [10].

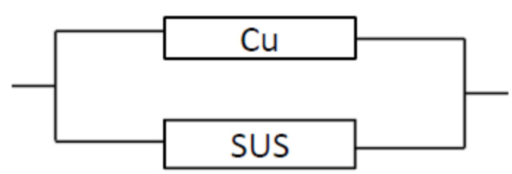

Figure 10: Simple parallel model to calculate RRR values of Cu-plating on SUS316L samples.

On the other hand, a cylindrical ceramic window of the TTF-III input couplers were joined by laser welding without any heating process. This must be the main reason to explain the different dynamic heat loads between the TTF-III and the STF-2 input couplers, as shown in Figure 1. Heat treatment at 800^{o}C by a hydrogen furnace was carried out in eight Cu-plating samples (A~H) shown in Table 1. Other three samples (I, J and K) were heat-treated at 800^{o}C by a vacuum furnace. Figure 11 shows a comparison of the measured resistivity

ISBN: 978-3-95450-191-5 doi:10.18429/JACoW-SRF2017-MOPB061

of three Cu-plating samples before and after heat treatment at 800°C of a brazing temperature. Figure 12 shows the RRR values of twelve Cu-plating samples in Table 1, before and after heat treatment at 800°C of a brazing temperature. Clear deterioration of the RRR was observed in all of samples after heat treatment at 800°C by both of hydrogen and vacuum furnace. It was confirmed that degradation of the purity of Cu-plating in the STF-2 input couplers had been caused by the brazing process.

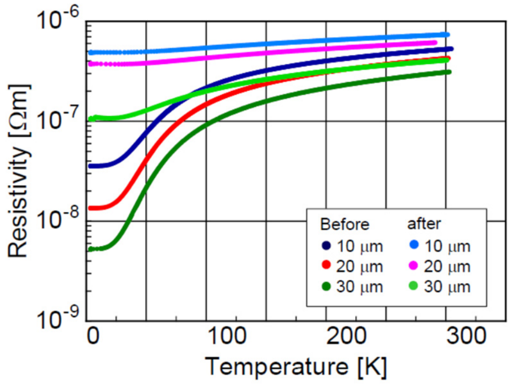

Figure 11: Comparison of measured resistivity between before and after heat treatment at 800°C of a brazing temperature: Sample-I (10 μm), Sample-J (20 μm) and Sample-I (30 μm).

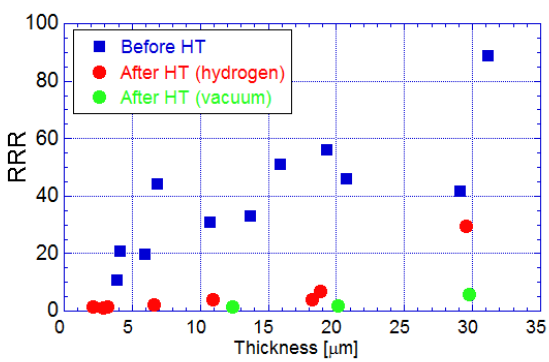

Figure 12: Degradation of RRR values of Cu-plating as a function of thickness of Cu-plating after heat treatment at 800°C of a brazing temperature in a hydrogen furnace (red circles) and a vacuum furnace (green circles).

COMPOSITION ANALYSIS AFTER HT

Analysis of impurity components into Cu-plating was performed in order to understand a reason of the degradation of the RRR after heat treatment at 800°C. Figure 13 shows the cross-section of Cu-plating with Ni-strike on SUS plate. Impurity composition was measured by EDXS (energy dispersive x-ray spectroscopy) at seven points in Figure 13. The results are summarized in Figure 14. After heat treatment in a vacuum furnace at 800°C of brazing temperature, impurity compositions like Ni, Fe and Cr were diffused into Cu-plating around the boundary area. On the other hand, it was found that there was no impurity composition at the area of ~26 μm close to RF

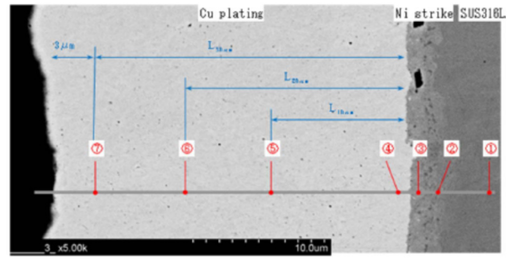

Figure 13: Cross-section of Cu-plating (30 μm) with Ni-strike (0.5 μm) on SUS316L by SEM [12].

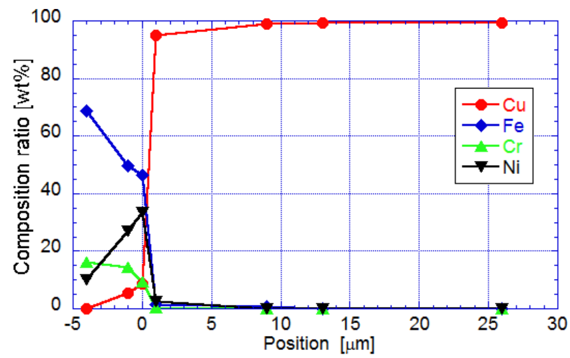

Figure 14: Impurity composition diffused into Cu-plating of 30 μm, after heat treatment in a vacuum furnace at 800°C of brazing temperature.

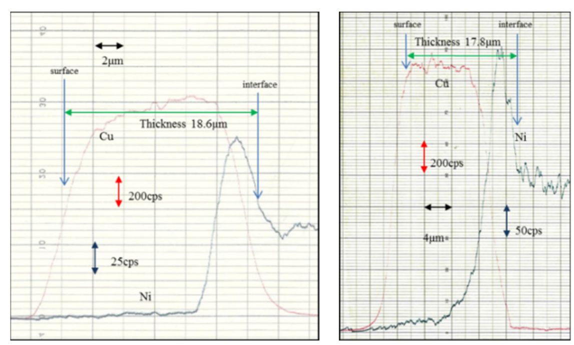

Figure 15: Observation of Ni diffusion into Cu-plating before (left) and after (right) heat treatment at 800°C of a brazing temperature.

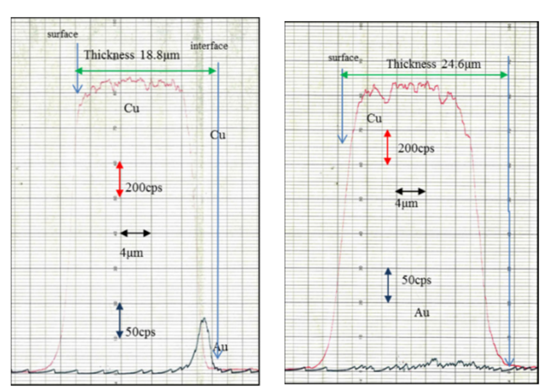

Figure 16: Observation of Au diffusion into Cu-plating before (left) and after (right) heat treatment at 800°C of a brazing temperature.

ISBN: 978-3-95450-191-5 doi:10.18429/JACoW-SRF2017-MOPB061

surface. This means that high purity of Cu-plating around RF surface has been kept even after heat treatment. Therefore, thicker Cu-plating of more than 20 µm will be necessary to keep a high purity at an RF surface layer of several µm.

The use of strike-plating by Ni or Au is a standard procedure to obtain a good adhesion between Cu-plating and stainless steel. Diffusion of Ni and Au into Cu-plating were observed before and after heat treatment at 800ºC, as shown in Figures 15 and 16, respectively. The diffusion layer of Ni was increased from a few µm to ~10 µm after the heat treatment at 800ºC. On the other hand, the layer of Au strike-plating has completely disappeared after heat treatment and dissolved into Cu-plating. This was also confirmed by the Cu-plating samples, as shown in Figure 17.

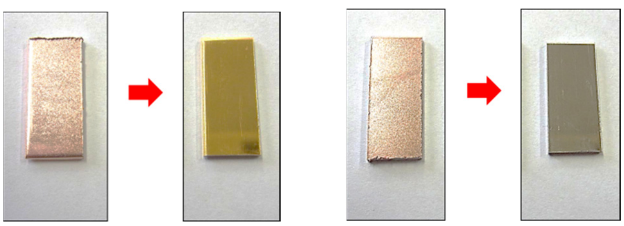

Figure 17: Diffusion of Au strike-plating into Cu-plating before (left) and after (right) heat treatment at 800ºC of a brazing temperature. The Cu-plating in each right side was removed.

SUMMARY

- Values of RRR of Cu-plating remarkably degraded after heat treatment at 800ºC for brazing in both of hydrogen and vacuum furnaces.
- Thin layer of Ni and Au for strike plating diffused into Cu-plating after heat treatment.
- Thicker Cu-plating of more than 20 µm will be necessary to keep a high purity at an RF surface layer of several µm.
- Preferable design is to separate a cold outer-conductor from brazed coupler components.

REFERENCES

[1] E. Kako *et al.*, "Advances and performance of input coupler at KEK", Proc. of SRF2009, Berlin, Germany (2009) 485-490.

[2] E. Kako *et al.*, "High Power Tests of CW Input Couplers for cERL Injector Cryomodule", Proc. of IPAC2012, New Orleans, Louisiana, USA (2012) 2230-2232.

[3] E. Kako *et al.*, "S1-Global Module Tests at STF/KEK", Proc. of IPAC'11, San Sebastian, Spain (2011) p38-40.

[4] W.-D. Moeller, "High power coupler for the TESLA test facility", SRF'99, Santa Fe, NM, USA (1999) p577.

[5] E. Kako *et al*., "Cryomodule test of four Tesla-like cavities in the superconducting test facility at KEK", Phys. Rev. ST-AB, 13, 041002, (2010).

[6] N. Ohuchi *et al.*, "Thermal Performance of the S1-Global Cryomodule for ILC", Proc. of IPAC2011, San Sebastian, Spain (2011) 2472-2474.

[7] H. Kutsuna *et al.*, "Quality Control of Copper Plating for Couplers", Proc. of LCWS2013, Tokyo, Japan (2013), Proc. of LCWS2013, Tokyo, Japan (2013). http://www.icepp.s.u-tokyo.ac.jp/lcws13/proceedings.php

[8] H. Umezawa *et al.*, "RRR and Thickness Measurements of Cu-plating", Proc. of LCWS2013, Tokyo, Japan (2013) *ibid.*, [7].

[9] O. Yushiro *et al.*, "Comparison Fabrication Techniques in TTF-III and STF-2 Couplers", Proc. of LCWS2013, Tokyo, Japan (2013) *ibid.*, [7].

[10] K. Kanaoka *et al.*, "Development for Mass-production of Superconducting Cavity by MHI", Proc. of ERL2015 at Stony Brook University, NY, USA (2015).

[11] H. Umezawa *et al.*, "Measurement of RRR and Thickness on Thin Cu-plating used for Input Couplers in Superconducting Cavities", Proc. of 10th Annual Meeting of Particle Accelerator Society Japan, Nagoya, Japan (2013) 569-572.

[12] K. Okihira and K. Sennyu, in private communication.