INFLUENCE of HEAT TREATMENT on THIN ELECTRODEPOSITED CU LAYERS

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INTRODUCTION

The TESLA input coupler has to transmit pulses of up to 1MW rf peak power at an average of up to 3 kW at 1.3 GHz. It makes the connection between room temperature WR650 waveguide and the coaxial port of the 1.8 K superconducting TESLA cavity. Hence its transmission line parts should combine low heat conductivity and high electrical surface conductivity. This is solved by constructing the transmission line walls of about 0.5 mm stainless steel and coating them with thin (10µ..20µ) copper layers. Rf losses occur only in the copper layer because current penetration depth is only a few microns. High purity of the copper coatings guarantees very low rf surface losses especially at low temperature. Electrical conductivity, heat conductivity and thickness of the copper layer dominate the heat budget at the 1.8K end of the coupler because the stainless steel heat conductivity is very low at this temperature. Consequently it is very important to control the physical properties of the copper layer.

Problems arise if the copper coated parts have to be heat treated. This is necessary for the DESY type TESLA coupler because the final steps of its assembly are done by brazing in a UHV furnace. Two braze alloys are used at two different steps of manufacturing. The first braze alloy is eutectic CuAg with a working temperature of 820°C, the second one is $AuGe_{10}Cu_2$ braze with a working temperature of 500°C. Only a few minutes at maximum temperature are necessary for melting and brazing.

In this situation it is important to know the influence of heat treatment on RRR and heat conductivity of copper layers. The purpose is to achieve a coating quality with RRR≈30 at a thickness of 10µm. RRR means Residual Resistivity Ratio and is determined as the ratio of electrical resistivities at room temperature and at 4.2 K. RRR>30 cannot further reduce the low temperature surface resistance of copper at microwave frequencies because of the anomalous skin effect.

Similar problems were the subject of references /1/,/2/. They also present RRR data of similar electrolytic copper layers which are heat treated. The 100 µm thickness of the investigated and analyzed layers is essentially bigger than here requested. It was very surprising when after first tests it turned out that these results were not transferable to our application.

RRR MEASUREMENTS and DISCUSSION

According to /1/, it was expected that the RRR of electrolytically deposited copper layers on stainless steel might still exceed 30 after a heat treatment of only a few minutes at maximum 820°C. However first test measurements of samples with Cu layers of 10µm to 20µm showed after this procedure very low RRR values between <1 and 10. This surprise initiated an investigation of different copper layers which is reported here. All of the necessary copper coatings were manufactured by a Swiss company

/3/. The aim was to understand the reasons of RRR degradation and to find copper coatings with as low as possible sensitivity towards heat treatment. Clearly the basic mechanism of RRR degradation is diffusion of foreign atoms from the substratum (consisting of stainless steel and an intermediate layer) to the copper layer. Even small amounts of impurities in the copper result in large reductions of electrical and heat conductivity.

a) INFLUENCE of INTERMEDIATE LAYERS

Table 1 (ser.1) shows measurement results of a first series of Cu coatings on stainless steel. They differ by the intermediate layers. The Cu coating ser.1, sample 1 (: s. 1) e.g. is based on a thin layer of Ni plus an additional thin layer of Au. 'Flash' means chemical deposition at a thickness of roughly 0.1µm. Sample 4 has a Ni-Flash layer and in addition 10,4µm Ni which are electrodeposited. Finally it is coated by 19,5µm Cu. Intermediate layers between copper and stainless steel are primarily needed in order to allow electrolytic deposition of copper. In addition they could work as diffusion barriers.
Results from Table 1 (ser. 1) are that Ni and Au as intermediate layers are not ideal diffusion barriers. The best RRR result after 820°C treatment is obtained with the thinnest intermediate Ni layer. The intermediate layers reduce diffusion between stainless steel and Cu layer but they diffuse themselves into the copper layer at 820°C treatment. Au even seems to be a bigger perturbation in the Cu lattice than Ni . Besides diffusion there are recrystallization, crystal growth with temperature, reduction of initial porosity (Fig. 1) and degassing observable which dominate RRR reduction due to diffusion and lead to considerable increase of RRR at a temperature of about 400°C. The following dramatic reduction of RRR at increase of temperature up to 820°C is due to the fact that diffusion depth even after 5 min at that temperature is already comparable to the thickness of the copper layer (10µm .. 20µm). It has to be mentioned that the heat treatments followed a temperature vs time profile of about 2-3 hours to heat up to 820°C and about 5-6 hours to cool down to room temperature again.

b) COATING THICKNESS

Table 2 (ser. 2: s.3, s.5, s.6) illustrates the RRR results as a function of coating thickness and heat treatment. Clearly after all heat treatments higher RRR values are obtained with increasing thickness of copper plating because diffusion depth is independent of thickness. The worst sample is (s.7). It has an additional 1µm intermediate Ni layer. The pulse current deposition technique of (s.4) gives no improvement of RRR. At 550°C treatment the RRR first improves, but with increasing time it degrades due to increasing diffusion depth. After about 8 hours the diffusion depth is so large that all samples have RRR values between 1 and 2. This is nearly the same result as for 5 min 820°C treatment. The change of microstructure of (s.3) due to heat treatment is demonstrated in Fig.1 .

c)DIFFERENT DEPOSITION TECHNIQUES

In a third test series Table 3 (ser.3) the quality of different deposition techniques is investigated. Additionally the manufacturer succeeded to improve the RRR starting values. Samples (s.1) , (s.2) , (s.3) , (s. 3.1) exhibit those improved RRR values 'as received' and also confirm the results of Table 2.

Samples (s. 3.2) , (s. 3.3) are the worst ones. They have additives which make the copper surface shiny and work as impurities. Samples (s. 4) , (s. 6) use an additional Ag layer. (s. 6) performs worse because the Cu layer gets impurities from 2 sides.

Sample (s. 11) shows the highest RRR values after heat treatment. An unusual feature of this sample is 0.1µm Cu-Flash as intermediate layer.

APPLICATION of a MATHEMATICAL DIFFUSION MODEL

At two samples an X ray spectrum analysis was performed at a few points near the transition planes of the intermediate layer and thus concentration values of Fe out of the stainless steel and of Ni in the Cu layer were gained. With this knowledge of foreign atom concentration at one certain position and the time temperature profile of heat treatment it was possible to determine the actual diffusion constants of Fe and Ni in the Cu layer by application of Fick's law /4/. This allowed us to compute the concentration profile of Fe or Ni in the copper layers depending on heat treatment. A typical concentration profile is given in Fig. 2. Given the concentration, it was also possible to calculate the expected RRR values for a given coating thickness and heat treatment. Some of these values are shown in Table 3. Their accuracy is limited due to high sensitivity against small amounts of impurities and presence of more than only one impurity.

CONCLUSIONS

None of the investigated copper coatings reveals a value of RRR >=30 after 820°C heat treatment. Maximum values of 16 were achieved. Values of RRR≈30 at coating thickness>=10µm are achievable up to furnacing temperatures of roughly 550°C. Heat treatment up to 400°C can improve low RRR values 'as received' to maximum values of even more than 200. However values of RRR >30 are not necessary anyway at microwave frequencies because of the anomalous skin effect. The mechanism reducing RRR of thin copper layers is diffusion from the substratum at high temperatures. The reduction is dramatic if the diffusion depth reaches the coating thickness. Following impression was gained from the investigations: Minimization of RRR reduction is difficult because substratum materials of low diffusion rate in copper (e.g. Fe) show high influence on RRR. On the other hand materials with less influence on RRR (e.g. Ni) have higher diffusion rates.

REFERENCES

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2. J.M. Dalin, J. Hague, Note Technique MT-SM/93-06, April 1993.
3. Werner Flühmann AG, Dübendorf, Switzerland.
4. J. Crank, The Mathematics of Diffusion, Oxford, 1970

RRR of electrodeposited Cu layers

Table 1

Deposition characteristics, ser. 1	Heat treatment procedure		
	As receiv.	820°C 5 min	450°C 1h
Ni-Flash Au-Flash19,4µmCu (s. 1)	4	2,7	137,5
Ni-Flash 21,4µm Cu (s. 2)	4	14,8	126,5
Ni-Flash 1µmNi 19,8µm Cu (s. 3)	7	2,9	33,9
Ni-Flash 10,4µmNi 19,5µm Cu (s. 4)	9,6	2,1	42,5
Ni-Flash 1µmNi 12,4µm Cu (s.5)	7	0,9	12,1

Table 2

Deposition characteristics, ser. 2	Heat treatment procedure						
	As receiv.	820°C 5 min	350°C 1 h	550°C 10 min	550°C 1 h	550°C 4 h	550°C 8 h
Ni-Flash 5µmCuPyro (s. 6)	10,6	1,3	56,3	11,6	17,2	4,9	1,1
Ni-Flash 1µmNi 5µm CuPyro (s. 7)	13,3	1,1	35,7	4,8	5,1	2	1
Ni-Flash 10µm CuPyro (s. 5)	10,3	1,9	91,4	84,2	19,3	12	1,6
Ni-Flash 20µm CuPyro (s. 3)	10,8	10,3	100,8	131	61,1	35,6	1,5
Ni-Flash 20µm CuPyro pulse cur.(s.4)	13,3	7,2	86,2	48,9	36,6	21,9	1,5

Table 3

Deposition characteristics, ser. 3	Heat treatment procedure								
	As receiv.	820°C, 5 min.		400°C, 1 h		550°C, 10 min.		550°C, 4 h	
		exp.	calc.	exp.	calc.	exp.	calc.	exp.	calc.
Ni-Flash 5,9µmCuPyro (s. 1)	24,6	0,3	0,9	33,9	35	22	34	16,3	19
Ni-Flash 11,9µm CuPyro (s. 2)	30,4	1,9	1,8	84,3	54	62	53	26,2	40
Ni-Flash 23µm CuPyro (s. 3)	35,9	12,1	3,7	147,5	114	124	113	111,9	96
Ni-Flash 100µm CuPyro (s. 3.1)	43,7	22	23	195,6	208	193	208	205,7	205
Ni-Flash 11,3µm CuPyroGlanz (s. 3.2)	6,9	2,9		18		14		5,5	
Ni-Flash 23µm CuPyroGlanz (s. 3.3)	7,2	2,6		29,8		23		21,2	
Ni-Flash 3µmAgRein 23µm CuPyro (s. 4)	48,3	2		174,7		145		83,7	
Ni-Flash 21µm CuPyroOptalloy (s. 5)	45,7	1,3		61,8		15		3,1	
Ni-Flash 20µm CuPyro3µmAgRein (s. 6)	35,8	4,5		101,1		63		27,8	
Ni-Flash 20µm CuSauer1 (s. 7)	50,8	3,8		82,5		60		27,9	
Ni-Flash 24µm CuSauer2 (s. 8)	60,1	15,4		226,4		213		123,3	
Cu-Flash11,5µm CuPyro (s. 10)	16,3	1,7	1,4	132,1	18	103	18	33,5	16
Cu-Flash 23µm CuPyro (s. 11)	41,9	16,3	3,8	227,2	165	213	165	233,7	130

ser. = series; (s. 1) = sample 1

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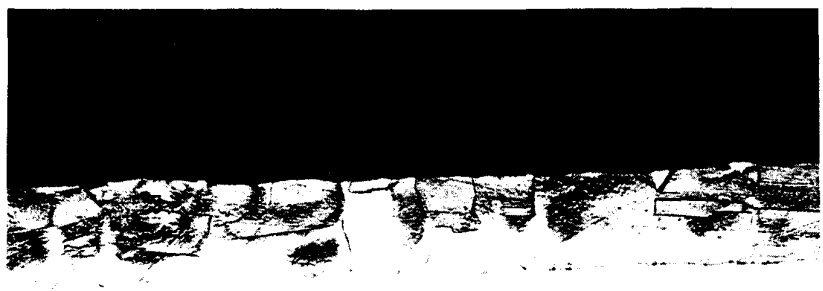
as received

550°C/10min



820°C/5min

Figure 1:Mikrostructure of NiFl20µmCuPyro samples; magnification 500:1

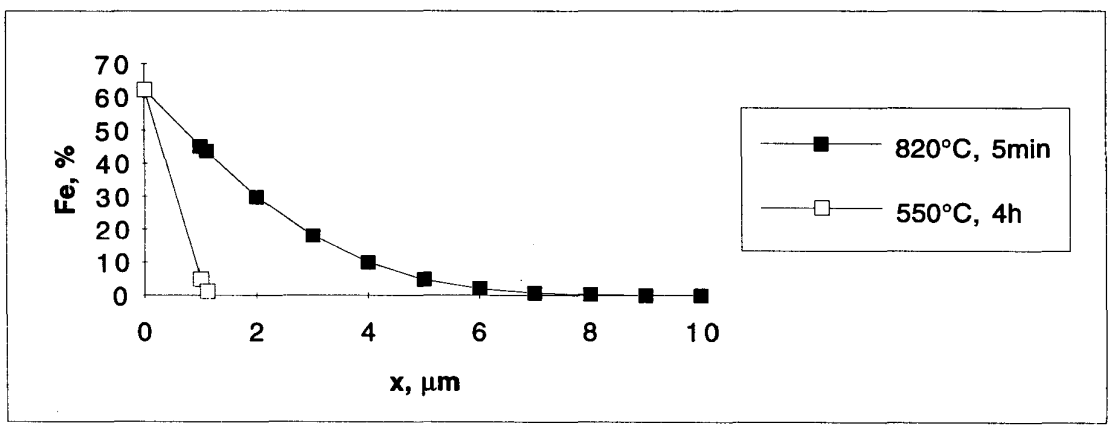


Figure 2: Distribution of Fe in copper layer of CuFl20µmCuPyro sample