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REDUCTION OF RF LOSSES AT 35 GHz IN HIGH PURITY COPPER RESONANT CAVITIES BY COOLING TO CRYOGENIC TEMPERATURE

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Résumé. — La résistance de surface R_s de cavités résonnantes réalisées en cuivre très pur a été mesurée à 35 GHz pour trois températures (293, 77 et 4,2 K). Après une série de traitements mécaniques, chimiques et thermiques, la valeur de R_s pour un monocristal de cuivre, décroît d'un facteur 4,5 entre 293 et 4,2 K, en bon accord avec les prévisions théoriques.

Abstract. — Surface resistance R_s of microwave cavities made of high purity copper is measured at a frequency of 35 GHz and at three temperatures (293, 77 and 4.2 K). After convenient mechanical, chemical and annealing treatments, the experimental R_s value for a copper single crystal decreases by a factor of 4.5 when cooled down to 4.2 K, in excellent agreement with theory.

1. Introduction. — In a very low noise millimeter wave receiver, the effective noise temperature increases rapidly with the RF losses in the walls of the waveguide connecting antenna and mixer diode. Decreasing these losses will therefore improve the ultimate sensitivity of the receiver. The main object of our work was to realize low loss millimeter waveguides and cavities operating at the working temperature (16 to 20 K) of low-noise cryogenic receivers now in use in radioastronomy.

In this range of temperatures, classical superconducting materials such as niobium or lead cannot be used, their critical temperature T_c being well below 16 K. Among high T_c superconducting compounds or alloys, like Nb_3Sn ($T_c = 18.3$ K), or NbGe ($T_c > 20$ K), Nb_3Sn only is known to exhibit sufficiently low losses at microwave frequencies below 12 GHz. By extrapolation of experimental results, the theoretical value of R_s at $T = 16$ K and $f = 35$ GHz would be $R_s \simeq 5 \times 10^{-4} \Omega$, a factor of about 20 lower than of high purity copper at the same temperature. But it would be very difficult with real surfaces to obtain in waveguides or cavities and particularly in the millimeter wave domain these R_s theoretical values. In addition, at temperatures very close to T_c , R_s would strongly depend on T , leading to variations of the receiver sensitivity, following the fluctuations or drifts of the cryogenic refrigerator temperature.

For these reasons, in a first attempt, we have only considered the possible utilization of high purity copper, for which R_s — like for all normal metals — presents the advantage to be nearly independent of T in the domain $T < 20$ K.

In the past, the surface resistance of copper single crystals was carefully measured at liquid helium temperature by Pippard [1] at 22.7 GHz, using circular disc with plane face very easy to polish. Our purpose was to try to obtain, by an appropriate technology, values of R_s as good as those of Pippard in microwave structures of complicate shape.

We report here experimental results on the lowering of R_s by cooling our resonant copper cavities working at 35 GHz from $T = 300$ K to $T = 4.2$ K, these results being compared to the theoretical R_s values.

2. RF surface resistance of copper at low temperature. — Cooling down a metal leads to an increase of its conductivity σ due to an increase in the electron mean free path l . For RF currents, the surface resistance decreases first as $\sigma^{-1/2}$ by lowering the temperature until a certain limit and at relatively low frequencies. But at very low temperature and very high frequencies, l becomes larger than the RF field penetration depth δ and R_s tends towards a limit although σ increases continuously. The RF field amplitude changes rapidly in space along a distance l , and in time during the time τ between two successive collisions; for both reasons Ohm's law can no longer be applied.

This anomalous skin effect was first studied by Reuter and Sondheimer [2] (R. S.), neglecting the effect of electron relaxation time at microwave frequencies. Then Dingle [3], simplified the expressions given by R. S., leading to equations which can be used at all temperatures and microwave frequencies, considering both specular and diffuse electron reflection at the metal surface, but neglecting relaxation. He also gave tables showing frequency and temperature dependences of the surface impedance in the microwave and infrared region for a number of values of frequency and conductivity, taking relaxation into account. Finally, Chambers [4], pointed out that at frequencies higher than 20 GHz relaxation can no longer be neglected for metals having low σ/l ratio.

The temperature dependence of copper surface resistance can be seen on figure 1 (OFHC, diffuse

FIG. 1. — Temperature dependence of R_s for OFHC copper :
1) computed - - - diffuse reflection ;
2) experimental $\left\{ \begin{array}{l} \bullet \text{ before chemical treatment} \\ \star \text{ after chemical treatment} \\ \blacksquare \text{ after annealing in hydrogen.} \end{array} \right.$

electron-reflection) and figure 2 (single crystal, diffuse and specular electron reflection). The curves were obtained by computation of Dingle exact formula considering relaxation effect, at 35 GHz. The values of the relaxation time (which is only a correcting factor) are not exactly known, and are taken in all cases as those given by Chambers [5] (2.7×10^{-14} s at room temperature, 2.4×10^{-11} s at 4.2 K). The surface resistance of a copper single crystal is slightly lower — particularly at 4.2 K — than for OFHC polycrystalline copper, due to a lower d.c. resistivity. In our experiments the single crystal of copper was supplied by Metals Research, without any data on d.c. electrical resistivity ρ . Therefore, we assumed that ρ (293 K) = $1.59 \mu\Omega\cdot\text{cm}$, a value given by

FIG. 2. — Temperature dependence of R_s for single crystal of copper : 1) computed $\left\{ \begin{array}{l} - - - \text{ diffuse reflection} \\ - \text{ specular reflection} \end{array} \right.$
2) experimental $\left\{ \begin{array}{l} \bullet \text{ before chemical treatment} \\ \star \text{ after chemical treatment} \\ \blacksquare \text{ after annealing in hydrogen.} \end{array} \right.$

H. H. Mende [6] for copper single crystals instead of $1.68 \mu\Omega\cdot\text{cm}$, the commonly adopted value for high purity OFHC copper. This assumption leads to a theoretical value of R_s in agreement with our best experimental value obtained at $T = 293$ K after polishing and annealing of the surface in hydrogen atmosphere.

Despite of a decrease by a factor 10^3 of the d.c. resistivity, theoretical $R_s(T)$ curves show that the limit imposed by the anomalous skin effect to the RF surface resistance is only a few times lower than its room temperature value. The ratio $R_s(293 \text{ K})/R_s(T)$ is 2.6 and 4.1 respectively at liquid nitrogen and liquid helium temperatures for OFHC copper assuming diffuse electron reflection, 2.5 and 4.0 for single crystal and also diffuse electron reflection ($R_s(293 \text{ K})$ being smaller for single crystal than for OFHC copper), 2.8 and 5.0 for single crystal and specular electron reflection.

The program we developed needs the value of the following parameters : frequency, d.c. electrical resistivity at each temperature, density of free electrons, in order to compute R_s over the entire temperature range. Computation using the same formula, but neglecting relaxation gives similar results within 1 %, leading to the conclusion that at a frequency of 35 GHz, the influence of relaxation on R_s may be neglected.

3. R_s measurement method and experimental set up. — The measurement of surface resistance can be achieved by measuring the quality factor Q_0 of a resonator obtained by short-circuiting a section of a rectangular waveguide.

The losses in such a cavity, coupled to the RF power supply and the measuring system, can be described by a loaded quality factor Q_L . Knowing the coupling coefficients β_1 and β_2 , the unloaded quality factor Q_0 can be written as :

$$Q_0 = Q_L(1 + \beta_1 + \beta_2). \quad (1)$$

As the cavity is pumped down to a few 10^{-1} Pa, the dielectric absorption losses in residual gas are negligible.

The surface resistance is then derived from Q_0 using the relation

$$Q_0 = AR_s \quad (2)$$

where A is a geometric factor depending only on the cavity dimensions and the RF chosen mode. In our case (rectangular cavity, TE_{013} mode), $A = 299 \Omega$. Two rectangular cavities were fabricated from bulk copper rods by spark machining : by this process, rectangular cavities were obtained with a good surface finish and the very thin mechanically disturbed surface layer could be removed easily by chemical polishing. A measure of the residual microreliefs existing on the wall surface before polishing was performed on a stylus type profilometer, showing a surface finish of $0.5 \mu\text{m}$ r.m.s. A chemical polishing in a $1/3 \text{H}_3\text{PO}_4$, $1/3 \text{HNO}_3$, $1/3 \text{CH}_3\text{COOH}$ during a few minutes at a temperature of about 60°C is sufficient for obtaining mirror-like surfaces. One cavity was made out of oxygen-free high-conductivity copper with a purity of 99.9 %; the other, out of a single crystal copper cylinder (purity 99.999 %) with an axis oriented in the $[100]$ direction.

The transverse inner dimensions of each cavity have been taken the same as those of standard 26.5-40 GHz waveguide (WR 28) : $7.10 \times 3.55 \text{ mm}^2$. The length was chosen $L = 3\lambda_g/2 = 16.1 \text{ mm}$ at 35 GHz, determining the lower mode of resonance to be TE_{013} . With such a mode, having an odd longitudinal number, the cavity could be made of two identical parts, without additional losses, as no current flows across the connection between the two half-cavities (Fig. 3).

FIG. 3. — Cavity diagram.

In the single crystal rectangular cavity, while the bottom walls are in the planes (100) the side walls are not exactly in the planes (010) and (001) as they should be, but make an angle θ of about 10° with them. Results obtained by Pippard [1] show that in these conditions the surface resistance is still isotropic with a value slightly higher than those corresponding to $\theta = 0^\circ$.

The cavity was cooled down in a cryostat filled up with liquid nitrogen or liquid helium, the working temperatures being respectively 77 K and 4.2 K. The cavity was kept under vacuum in a low pressure can (a few 10^{-1} Pa). Two stainless steel circular waveguides — 8 mm diameter and about 1 m long — were used to carry the RF input energy from the supply (an 0-type Thomson-CSF carcinotron) to the cavity, and the transmitted energy out of the cavity. Two tapered transitions at the hot end of each waveguide allow the connection with standard rectangular waveguides (Fig. 4).

FIG. 4. — Experimental set up.

The two connecting guides going into the cryostat were made of thin walled stainless steel tubes instead of copper, to reduce the thermal losses. Despite the relatively bad conductivity of stainless steel, the insertion losses are less than 2 dB in each waveguide, in which the TE_{11} mode is excited.

The cavity with its supporting waveguides was pumped down at a low pressure ($\approx 10^{-1}$ Pa) to

avoid gas condensation on the walls during the cooling process. Two pressure-tight windows were placed between the transitions and the rectangular waveguides (see Fig. 4).

The cavity resonance frequency f_0 is first measured with a conventional transmission cavity wavemeter. In a second experiment, a part of the energy supplied by the carcinotron is coupled to a transmission semi-confocal Pérot-Fabry resonator 130 mm in diameter. This resonator made of two copper mirrors has a quality factor in the range 100 000-200 000, depending on the resonance mode, a figure at least 10 times greater than the loaded cavity quality factor to be measured. This open resonator is thus used as a wavemeter to measure the frequency difference Δf between the two -3 dB points of the cavity resonance transmission curve observed on a power-meter. One of the resonator mirrors can be moved by using a micrometric screw, with a precision better than $\pm 1 \mu\text{m}$ resulting in a relative error on Δf of the order of 3 %.

The loaded cavity quality factor is then obtained as : $Q_L = f_0/\Delta f$. Assuming the coupling coefficients β_1 and β_2 to be identical, expression (1) becomes :

$$Q_0 = Q_L(1 + 2\beta) \quad (3)$$

which allows the unloaded quality factor Q_0 to be derived from the measurement of the cavity insertion loss factor of the cavity T :

$$T = \frac{P_{\text{output}}}{P_{\text{input}}}$$

Knowing that $\sqrt{T} = \frac{2\beta}{1+2\beta}$, (3) can be written :

$$Q_0 = \frac{Q_L}{1 - \sqrt{T}} \quad (4)$$

This ratio T has been experimentally evaluated *in situ* by means of two successive insertion loss measurements. In a first run, the cavity resonance frequency is observed and the power ratio T_1 is measured at each temperature for the ensemble composed of the two pressure windows and the rectangular-circular transitions, the two sections of circular waveguides, the bend and the cavity. In a second experiment, the cavity is replaced by a section of circular OFHC copper 8 mm in diameter waveguide of the same length, and the power ratio (T_2) is measured at the same frequency and the same temperature. The insertion loss of the cavity (T) is then taken as T_1/T_2 , neglecting the insertion loss of the short section of circular waveguide, which in any case is less than 1 dB.

4. Experimental results. — Theoretical and experimental values of R_s can also be compared *versus* temperature both for OFHC (Fig. 1) and single crystal (Fig. 2).

We first tested the OFHC copper cavity before any chemical or heat treatment right after spark machining (SM). Then this cavity was chemically polished (SM + CP), and finally annealed in hydrogen atmosphere at 400 °C for 4 hours (SM + CP + A) to remove the superficial oxides, R_s values decrease after each treatment and appears to be no more

TABLE I
Theoretical and experimental data

	Treatment	(K)	Q_0 th	R_s th (Ω)	Q_0 exp	R_s exp (Ω)	$\frac{R_s \text{ th}}{R_s \text{ exp}}$	$\Gamma_1 = \frac{R_s \text{ exp } 293 \text{ K}}{R_s \text{ exp } T \text{ K}}$	α_{exp} (dB/m)	$\frac{\alpha_{\text{stand}}}{\alpha_{\text{exp}}} (***)$
OFHC copper	SM	293	—	—	4 200	7.12×10^{-2}	69 %	—	0.79	1.4
	SM + CP	293	6 100 (**)	4.9×10^{-2}	5 500	5.44×10^{-2}	90 %	—	0.60	1.8
	SM + CP + A	293	—	—	5 700	5.25×10^{-2}	93 %	—	0.58	1.9
	SM	77	—	—	6 100	4.90×10^{-2}	39 %	1.5	0.54	2.0
	SM + CP	77	15 800 (**)	1.89×10^{-2}	10 300	2.90×10^{-2}	65 %	1.9	0.32	3.4
	SM + CP + A	77	—	—	13 500	2.21×10^{-2}	86 %	2.4	0.24	4.5
	SM + CP	4.2	25 100 (**)	1.19×10^{-2}	15 700	1.90×10^{-2}	63 %	2.9	0.21	5.2
	SM + CP + A	4.2	—	—	21 500	1.39×10^{-2}	86 %	3.8	0.15	7.3
copper single crystal 99.999 % purity	SM	293	6 400	—	5 400	5.54×10^{-2}	84 %	—	0.61	1.8
	SM + CP	293	(*)	4.67×10^{-2}	5 700	5.25×10^{-2}	89 %	—	0.58	1.9
	SM + CP + A	293	(**)	—	6 300	4.75×10^{-2}	98 %	—	0.53	2.1
	SM	77	16 000 (**)	1.87×10^{-2}	12 900	2.32×10^{-2}	71 % (*)	2.4	0.26	4.2
	SM + CP	77	18 050 (*)	1.66×10^{-2}	14 300	2.09×10^{-2}	79 % (*)	2.5	0.23	4.7
	SM + CP + A	77	—	—	16 100	1.86×10^{-2}	89 % (*)	2.6	0.21	5.2
	SM	4.2	25 300 (**)	1.18×10^{-2}	14 000	2.14×10^{-2}	43 % (*)	2.6	0.24	4.5
	SM + CP + A	4.2	32 100 (*)	0.93×10^{-2}	28 600	1.05×10^{-2}	88 % (*)	4.5	0.12	9.1

(*) Considering specular electron reflection R_s theoretical value.

(**) Considering diffuse electron reflection R_s theoretical value.

(***) With $\alpha_{\text{stand}} = 1.09 \text{ dB/m}$.

than 1.2 times the theoretical values which have been computed assuming diffuse electron reflection as it is suitable for polycrystalline copper. Our limit value of R_s for the OFHC copper cavity is in good agreement with the value obtained by Fawcett [7] at $f \simeq 36$ GHz on a plane sample.

The same treatments have been repeated on the single crystal copper cavity. The results are in all cases better than for OFHC copper, and on figure 2 one can notice that the best experimental value obtained at $T = 4.2$ K, is situated between diffuse and specular values. If we consider now the R_s value quoted by Pippard [1] for $\theta \simeq 10^\circ$ ($R_s \simeq 0.83 \times 10^{-2} \Omega$ at $f = 22.7$ GHz and $T = 4.2$ K), we obtain by extrapolation to $f = 35$ GHz $R_s \simeq 1.1 \times 10^{-2} \Omega$, corresponding to our measured value.

The quantities reported in table I resume the respective data obtained respectively by numerical computation and measurement, and give a few basis of comparison between them. Also Γ represents R_s amelioration factor when cooling down, α_{exp} is the corresponding attenuation (in dB) in one meter of waveguide made of the same metal as the cavity, in the same conditions, and $\frac{\alpha_{\text{stand}}}{\alpha_{\text{exp}}}$ is the improvement factor in the attenuation of a waveguide using cooled down OFHC or single crystal copper machined and prepared at it is described in this paper, compared to a good commercial waveguide ($\alpha_{\text{stand}} = 1.09$ dB/m) working at room temperature.

The relative error on our experimental Q_0 values comes mainly from the measurement of Δf ($< \pm 3\%$) and from the cavity insertion loss ($< \pm 4\%$) resulting in a total relative error on Q_0 of about $\pm 5\%$.

5. Conclusion. — Similar measurements on resonant cavities has already been done but only at room temperature by F. J. Tischer [8] ($R_s \text{ th}/R_s \text{ exp} = 88\%$) and

J. S. Thorp [9] ($R_s \text{ th}/R_s \text{ exp} = 99\%$) in the same conditions (35 GHz, high purity copper annealed in hydrogen). At lower frequency, F. Biquard and A. Septier [10] also observed on OFHC electro-polished copper a decrease of R_s with temperature by a factor 5.6 ($R_s \text{ th}/R_s \text{ exp} = 99.6\%$ and 91% respectively at room and liquid helium temperature).

Our measurements show that the anomalous skin effect does not limit the room temperature RF electrical conduction in copper at 35 GHz; using appropriate surface preparation techniques on a cavity machined in a copper single crystal, the theoretical value of R_s can be experimentally obtained at $T = 300$ K and an improvement factor of about 2 is observed compared to a standard commercial waveguide.

Working at very low temperature leads to a very interesting result for the improvement of the sensitivity of RF high sensitivity receivers used in radio-astronomy. A comparison (see last column of table I) between standard waveguide and components machined in copper single crystal shows that the wave attenuation coefficient α may be reduced by a factor between 7 and 9 at 4.2 K. Our preparation techniques lead to values of R_s as low as those obtained by Pippard [1] for plane circular samples, even for structures of complicate shape machined as a whole. A slight decrease of R_s would be still possible by a precise orientation of the cavities plane faces in the crystal planes [100], [010] and [001].

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References

- [1] PIPPAARD, A. B., *Phil. Trans. R. Soc. A* **250** (1957) 325-357.
- [2] REUTER, G. E., SONDHEIMER, E. H., *Proc. R. Soc. (London)* **A 195** (1948) 336-364.
- [3] DINGLE, R. B., *Physica* **19** (1953) 311-364.
- [4] CHAMBERS, R. G., *Physica* **19** (1953) 365-370.
- [5] CHAMBERS, R. G., *Proc. R. Soc. (London)* **A 215** (1952) 481-497.
- [6] MENDE, H. H., THUMES, G., *Appl. Phys.* **6** (1975) 93-97.
- [7] FAWCETT, E., *Proc. R. Soc. A* **232** (1955) 519-536.
- [8] TISCHER, J. F., *IEEE Trans. Microw. Th. and Techn.* **MTT 24**, n° 11 (1976) 853-858.
- [9] THORP, J. S., *IEEE Proc.* **101** III n° 74 (1954) 357-359.
- [10] BIQUARD, F., SEPTIER, A., *Nucl. Instrum. Methods* **44** (1966) 18-28.