

# PROPERTIES OF COPPER CAVITIES COATED WITH NIOBIUM USING DIFFERENT DISCHARGE GASES

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## Abstract

Experimental evidence is presented showing that in conformity with theoretical expectations, discharge gas atoms are trapped in sputtered films whenever a gas of atomic mass smaller than that of the cathode is used. In such a case, discharge gas atoms may be reflected by the cathode as high energy neutrals and get incorporated in the growing film. Niobium films have been produced using Ne, Ar, Kr, Xe and then analysed for rare gas content by thermal extraction. The gas concentrations are found to vary from the several percent range for Ne down to the ppm level for Kr and Xe. The noble gas concentration in the film influences the RRR and, in the case of high concentration, also the critical temperature. To study the effect of the implanted noble gas on the superconducting RF parameters, several 1.5 GHz copper cavities have been niobium-coated using the different discharge gases. The noble gases trapped in the film affect the penetration depth, the temperature dependent losses ( $R_{BCS}$ ), the losses induced by the presence of trapped fluxons, but have no significant influence on the residual resistance.

## 1 Introduction

Theoretical arguments suggest that discharge gas atoms may be trapped in the film whenever a gas of atomic mass smaller than that of the cathode material is used. In this case, discharge gas atoms may be reflected by the cathode as high energy neutrals and get incorporated in the growing film.

The energy at which atoms of the discharge gas are reflected from the niobium cathode is a function of the gas mass and of the voltage used in the discharge as illustrated in table I.

Noble gas	Atomic mass	E'/E
helium	4	0.92
neon	20	0.65
argon	40	0.40
krypton	84	0.05
xenon	131	reflection impossible

Table I. Ratio of reflected (E') to initial (E) energy for different noble gases incident on a niobium atom (atomic mass ~ 93) at rest calculated for a 90° scattering.

## 2 Samples studies

The samples are produced using a cylindrical magnetron sputtering configuration similar to that used for cavity coating [1]. Three sample-holders are mounted at the level of the equator of a 500 MHz stainless-steel cavity. The coating pressure, as read from an ionisation gauge, is  $9 \times 10^{-4}$  mbar and the discharge current is 7.5 A. A solenoid allows changing the ionisation efficiency in the magnetron in order to keep the current constant while varying the voltage. Voltages lower than 300 V cannot be used without reducing the discharge current. The substrate temperature is kept at the same temperature during bake-out and coating. The coating thickness is 1.5  $\mu\text{m}$  for all samples.

The influence of the discharge voltage on the quantity of gas trapped in the film has been studied using neon, argon, krypton and xenon. Niobium films were also produced using different argon-neon mixtures at constant gauge reading. The samples are analysed for RRR and for gas content using a thermal extraction method described elsewhere [2].

## 3 Results on samples

The results obtained for samples produced at different voltages and with different gases are summarised in table II and table III. These results complete those already presented [2].

Noble gas	Discharge voltage [V]	Gas content [at. %]	RRR Nb on copper
Neon	360	50	3.0±0.3
Argon	300	0.12	39.0±6
Argon	350	0.35	25.0±5
Argon	400	0.64	19.0±2
Argon	500	2.1	11.4±1
Argon	600	5.7	10.4±1
Argon	700	6.2	8.9±1
Krypton	400		23.0±2
Krypton	500		34.0±5
Krypton	600		27.0±4
Krypton	700		14.0±1

Mixture	Neon content [at.%]	RRR (on quartz)	RRR (on copper)	$T_c$ [K]
100%Ne	5.8	3.0	too fragile	8.8
90%Ne-10%Ar	3.5	6.0	too fragile	9.1
80%Ne-20%Ar	2.2	8.8	4.3±.6	9.4
67%Ne-33%Ar	1.3	10.0	5.4±.7	9.5
50%Ne-50%Ar	0.7	13.4	not measured	9.5
20%Ne-80%Ar	0.17	14.8	9.2±1	not measured
100%Ar	0	17.8	12±1	9.5

Table II and Table III. Samples are coated at 200 °C, except for samples produced with gas mixtures which are coated at 150°C. The error on the measurements of neon content is less than 20%, the error on  $T_c$  is ~0.1K. The RRR of the samples with the highest Ne content on copper could not be measured because the film was destroyed when dissolving the copper substrate.

From these data the following conclusions may be derived.

The increase of discharge voltage results in a higher energy of backscattered neutrals and in a higher implantation probability in the growing film with a consequent increase of the residual resistivity (measured at 10 K), as shown in fig. 1.

The rare gas concentration in the film is strongly dependent on its mass. For krypton and xenon, the values are in the range of 10 ppm, which is very close to the experimental detection limit. The content is about 0.5 % for argon, and goes up to 5 % in the case of neon. This results in a significant increase of the residual resistivity when using neon, as it is shown in Fig.2, while a further decrease of the gas content does not reduce appreciably the residual resistivity of the film.

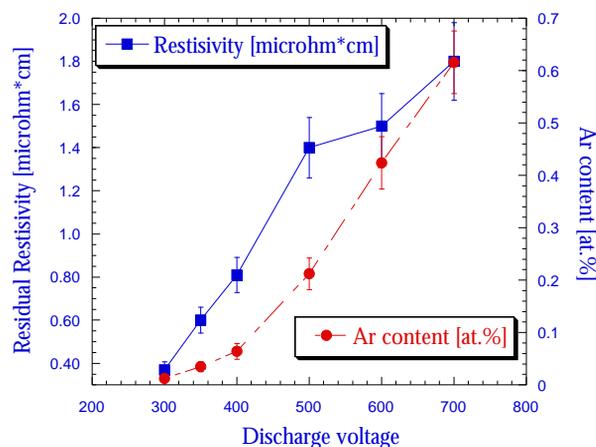


Fig. 1. Residual resistivity and argon content in the film as a function of the discharge voltage. The  $T_c$  remains unchanged.

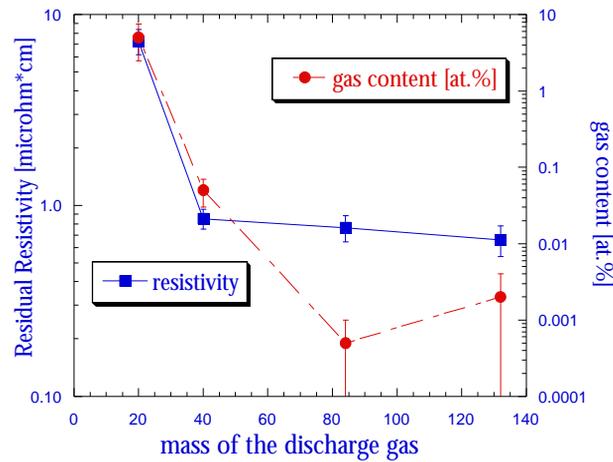


Fig. 2. Gas content and residual resistivity for niobium film coated at 400 V using different gases. For Xe a voltage of 600 V had to be used to maintain the same sputtering current.

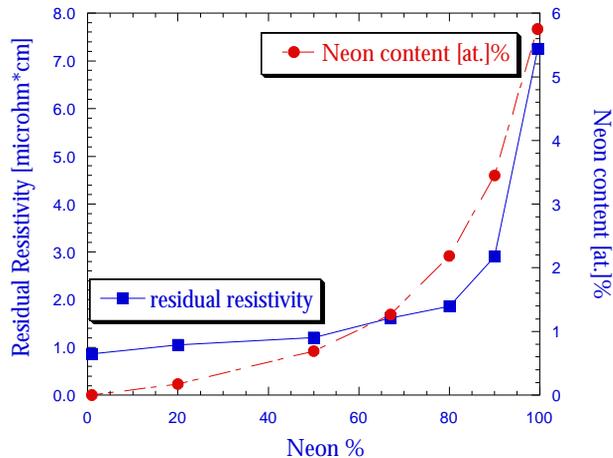


Fig. 3. Residual resistivity and neon content as a function of the fraction of neon present in the discharge.

Fig. 3 shows the neon content calculated by measuring the film pumping throughput, as a function of the neon percentage in the discharge gas, and the related variation of the residual resistivity. Since the flux of backscattered atoms is higher at the iris than at the equator of the cavity, the gas content thus calculated may be overestimated.

#### 4 Results on coated cavities

Several 1.5 GHz copper cavities have been coated with a niobium film using different discharge gases according to the standard LEP procedure [1]. In addition to the standard surface resistance versus RF field measurement at different temperatures, the penetration

depth  $\lambda$  and the effect of trapped magnetic flux on the surface resistance ( $R_s$ ) have been measured. The results are parametrised in the form  $R_s=R_{res}+R_{BCS}+R_{fl}$  with  $R_{fl}=(R_{fl}^0+R_{fl}^1H_{rf})H_{ext}$ . Temperature dependence of  $R_{fl}$  is expressed in terms of  $K_{fl} = R_{fl}(4.2K)/R_{fl}(1.7K)$ . The details of the measurement of the fluxons induced losses are given elsewhere [3].

The variation of the cavity resonance frequency  $f$  when  $T$  approaches  $T_c$  provides a direct measurement of the temperature dependence of the penetration depth [4],  $\lambda(t)=\lambda(0)F(t)$  where  $\lambda(0)=\lambda_0(1+0.5\pi\xi_0/\ell)^{1/2}$ ,  $\lambda$  is the mean free path,  $\xi_0$  is the coherence length and  $t=T/T_c$  is the reduced temperature. The  $\lambda$  values are normalised to the value measured for annealed bulk niobium which is taken as the clean limit.

In table IV we report the results obtained with different gases and gas mixtures. All the superconducting properties of the niobium films, except for  $R_{res}$ , are influenced by the presence of rare gas trapped in the film.

Gas	$R_{BCS}[n\Omega]$	$R_{fl}^0[n\Omega G^{-1}]$	$R_{fl}^1[n\Omega G^{-1}mT^{-1}]$	$K_{fl}$	$\lambda/\lambda_{clean}$
Xenon	440	6.4	1.02	2.58	1.51
Krypton	401	2.9	0.56	2.64	1.68
Argon	401	4.8	1.13	2.72	1.59
80Ar/20Ne	384	4.4	1.07	2.53	1.63
50Ar/20Ne	406	6.2	2.2	2.60	2.19
33Ar/67Ne	444	6.3	3.4	2.28	2.17
20Ar/80Ne	633	14.	9.5	1.60	2.69
10Ar/90Ne	655	21	21.	1.83	3.18
Neon	1006	139	75.	1.45	4.25

Table IV. Measured properties of cavities coated with various discharge gases or gas mixtures. The coating conditions are the same.

The results are often an average over more than one cavity (28 cavities in case of argon). All the measured parameters are very well reproducible except for the residual resistance: the fluctuations of the residual resistance are partly due to the different surface roughness, but other causes cannot be excluded. We also measure  $T_c$  (which is in agreement with what is reported for samples) and the gap  $\Delta$ . Both quantities decrease slightly when Ar-Ne mixtures are used due to the high quantity of neon trapped in the film.

From the values of  $\lambda$  measured on cavities we can calculate  $\lambda/\xi_0$  and compare it with the value derived from the RRR of samples using the relation  $\rho_n^*\ell=4.1\times 10^{-6}\mu\Omega cm^2$  [5] and  $\xi_0=390\text{\AA}$  (Fig. 4). The agreement is quite good and the observed discrepancies are at least partly due to the different substrates [6]. The noble gas concentration affects the RRR,  $\ell$  and in turn the  $R_{BCS}$ , as is shown in Fig. 5.  $R_{BCS}$  varies with noble gas concentration as expected from the change of the mean free path.

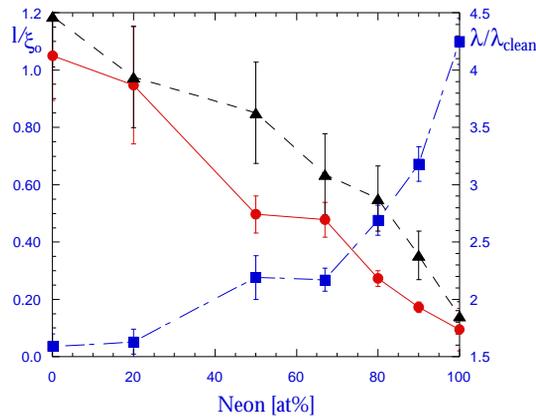


Fig. 4.  $l/\xi_0$  for the data from cavities (dots) and samples (triangles) together with the  $\lambda$  (squares) as a function of the atomic percent of neon in the mixture.

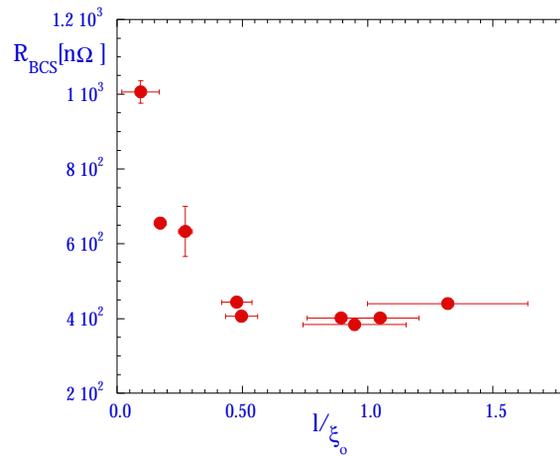


Fig. 5.  $R_{BCS}$  as function of  $\lambda/\xi_0$ . The shown behaviour is in agreement with the BCS theory.

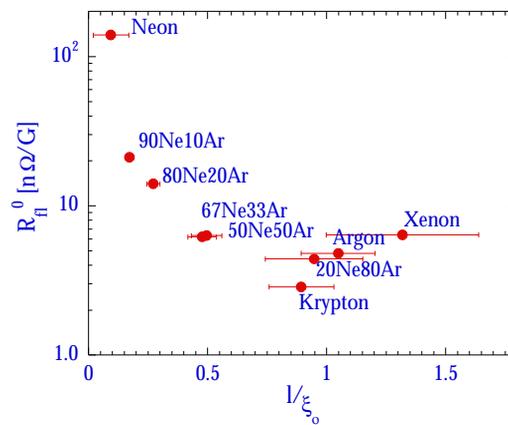


Fig. 6. The variation of fluxon induced losses at low accelerating field as a function of the mean free path.

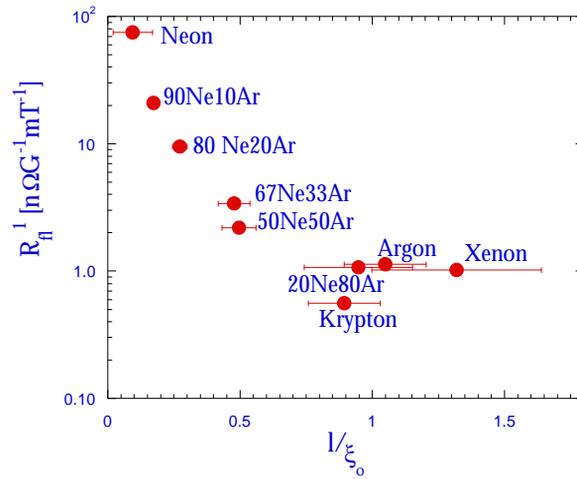


Fig. 7. The dependence of fluxon induced losses on the RF field as a function of the mean free path.

The dependence of  $R_{fl}^0$  and  $R_{fl}^1$  on the mean free path is reported in Fig. 6 and Fig. 7. This behaviour is found to be similar to that of the BCS resistance with a minimum value for  $l \sim \xi_0$ .

For  $l < \xi_0$  the density of noble gas atoms is such that their pinning potentials overlap and the pinning action becomes weaker. The resulting increased mobility of the fluxons is interpreted as being the cause of the increase of the associated RF losses.

The temperature dependence of the fluxon induced losses is affected only by high noble gas concentrations which result in  $l \leq 0.5\xi_0$  as is illustrated in Fig. 8.

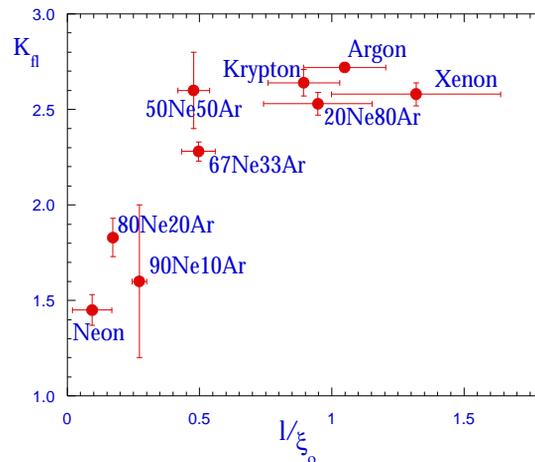


Fig. 8.  $K_{fl}$  as a function of the mean free path. The presence of a high quantity of neon causes a reduction of the temperature dependence of the fluxon induced losses.

## 5 Conclusions

The amount of the noble gas in the Nb films may be controlled by varying the discharge gas and the coating parameters.

The presence of noble gas influences the RF superconducting properties of the niobium films such as  $R_{BCS}$ ,  $\lambda$ ,  $R_{fl}^0$  and  $R_{fl}^1$ . The behaviour of all these quantities may be qualitatively described in terms of the reduction of the mean free path due to the increase of the noble gas concentration.

Only the residual resistance of the films show no correlation with the discharge gas presence up to concentrations in the percent range.

## References

- [1] C. Benvenuti, D. Bloess, E. Chiaveri, N. Hilleret, M. Minestrini, W. Weingarten in: *Proc. of the III Workshop on RF Superconductivity*, ANL-PHY-88 (ANL, Argonne, 1988) p. 445
- [2] C. Benvenuti, S. Calatroni, P. Chiggiato, M. Marino, and R. Russo, VII Workshop on RF Superconductivity, 17-20 October 1995, Gif-sur-Yvette, France
- [3] C. Benvenuti, S. Calatroni, I. E. Campisi, P. Darriulat, M. Marino, M. Peck, R. Russo, paper presented to this workshop
- [4] C. Benvenuti, S. Calatroni, I. E. Campisi, P. Darriulat, M. Marino, M. Peck, R. Russo and A.-M. Valente, to be published
- [5] A. Andreone, A. DiChiara, G. Peluso, M. Santoro, C. Attanasio; L. Maritato; and R. Vaglio, *Journal-of-Applied-Physics* 73,.9,(1993) p.4500
- [6] R. Russo and S. Sgobba to be published