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INFLUENCE OF VARIOUS VACUUM SURFACE TREATMENTS ON THE SECONDARY

ELECTRON YIELD OF NIOBIUM

by

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ABSTRACT

The secondary electron emission of technical niobium surfaces has been studied in the context of the development of superconducting radio-frequency cavities for particle acceleration. In these devices, spurious secondary electrons can be accelerated and multiplied, enhancing the losses in the cavities and finally causing a transition to the normal state. In order to limit these effects, a program has been undertaken to study niobium surface treatments which decrease the secondary electron yield (SEY). Consecutive in-situ bakeouts at temperatures increasing from 80°C to 350°C are found to reduce the maximum SEY from about 2.5 to about 1.3, depending on the initial chemical cleaning of the surface. The energy interval over which the SEY exceeds 1 is also reduced from 20-2000 eV in the as-received state, to 100-1100 eV after a 350°C bakeout. Glow-discharge cleaning treatments using different gases have also been tried. Argon produces a surface with a SEY close to that measured on high purity niobium surfaces. Chemically reactive gases introduced during the discharge can also reduce the SEY and create a relatively inert surface, less sensitive to subsequent air exposure.

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1. INTRODUCTION

Superconducting accelerating systems are at present under development at CERN (European laboratory for particle physics) for installation in the large electron-positron storage ring LEP. In this machine, the particle beams will radiate a large amount of energy by synchrotron radiation (1.6 MW with 6 mA at 55 GeV electron energy), the corresponding energy loss being fed back into the beams by powerful accelerating systems. Due to their lower surface resistance, superconducting Nb accelerating cavities offer a substantial reduction over conventional copper cavities in terms of the total power consumption. However, these devices are extremely sensitive to all sources of energy loss, including those induced by spurious electron emission from the cavity surface under the possibly combined action of electron bombardment and/or high electrical field.

In order to reduce these energy losses, various vacuum surface treatments have been investigated which aim at a reduction of the electron induced secondary electron emission. This emission is characterized by the secondary electron yield δ , the number of electrons re-emitted per incident electron and which depends essentially on the nature of the surface and on the energy (E) of the incident primary electrons. The general shape of the curve $\delta(E)$ for bulk materials is shown inset on figure 2b. One can define certain values of δ and E (hereafter called characteristics values) having a particular interest in the context of this work. These are:

- δ_{100} (δ_{1000}): value of δ at 100 eV (1000 eV) primary electron energy
- δ_m : maximum value of δ
- E_1 (E_2): energy below (above) which δ is less than 1
- E_m : energy at which δ_m is attained

δ_m , E_1 , E_2 and E_m are illustrated in figure 2b.

2. EXPERIMENTAL SYSTEM AND MEASUREMENT PROCEDURE

Secondary electron emission is a strongly surface dependent phenomenon; it is therefore especially important to make any measurements in a clean and well defined but nevertheless realistic environment. Furthermore, the perturbations

introduced to the surface by the electron bombardment, which is necessary for the measurements, can affect the secondary electron yield (1) if the electron dose exceeds 10^{-6} C.m⁻². For these reasons, all measurements quoted here were made in a bakeable U.H.V. system using very small electron dose to record the variation of δ as a function of the electron energy. The use of a micro-processor to drive the electron gun and to collect the data allowed the electron dose required for the measurement of δ at one electron energy to be reduced as low as 10^{-4} C.m⁻². δ is obtained by measuring simultaneously the net electron current on the sample (i_s) and on a secondary electron collector (i_c). For the calculation of δ , 10 individual measurements of these 2 currents are taken during a 30 ms electron pulse. The mean values are used for the calculation of δ from the expression:

$$\delta = i_c / (i_c + i_s)$$

In the present set-up, it was possible to measure δ at very low primary energy (down to 10 eV) by biasing the sample.

The gas composition in the vacuum vessel was monitored using a residual gas analyser. Before bakeout and after two days of pumping, the pressure in the system levelled off at 1×10^{-6} Pa, water vapour being the main constituent. After a 300°C, 24 hour bakeout the pressure attained was typically 1×10^{-8} Pa (88% H₂, 8% CH₄, 4% CO). For the glow discharge treatments, the gas was introduced in the system through a bakeable leak valve at a pressure close to 1 Pa and the sample was negatively biased to approximately 1 kV in order to activate the discharge and achieve the desired ion bombardment.

3. VARIATION OF δ WITH THE BAKEOUT TEMPERATURE

The niobium sample was submitted to consecutive in-situ bakeouts at increasing temperatures in order to study this influence on δ . Prior to this treatment the sample had been chemically polished in an aqueous solution of HF, HNO₃, H₃PO₄ and rinsed in demineralized water.

After each bakeout the variation of δ with the incident electron energy was measured; the characteristic values of $\delta(E)$ and E are given in the table 1. All characteristic values of δ decrease with increasing bakeout temperature; δ_m falling as low as 1.22 after a 350°C bakeout. The variations are presented in figures 1 and 2. The energy E_1 is only marginally affected below 200°C; above that temperature it rises and exceeds 100 eV after a 350°C bakeout. The energy

E_2 exceeds 2000 eV for bakeout temperatures lower than 200°C and falls below 1000 eV after a 350°C bakeout. In this scheme we are not able to distinguish between the effects from bakeout temperature and those from the accumulation of successive additional bakeouts.

4. VARIATION OF δ WITH AIR EXPOSURE

After the final bakeout at 350°C, laboratory air was introduced in a controlled way into the system to investigate any influence on the secondary electron emission. The variation of the characteristic values of δ with the air exposure (Pa.s) are shown in figure 3 and the corresponding variations for E_1 and E_2 are shown in the figure 4. It is seen that δ is only marginally affected for an air exposure smaller than 1.3×10^6 Pa.s (1×10^4 Torr.s). The most important effect is visible on δ_m for air exposures greater than 1.3×10^9 Pa.s (1×10^7 Torr.s). The low energy part of the curve $\delta(E)$ is more affected by the air exposure than the high energy part: δ_{100} increases from 1.0 to 1.5 while δ_{1000} increases for the same exposure of 2×10^{11} Pa.s, from 1.0 to 1.3. The effect is important for the energy E_1 which decreases from 105 eV to 51 eV for an air exposure of 4×10^6 Pa.s (3.3×10^4 Torr.s). The energy E_2 is only affected by larger air exposures of $\sim 2 \times 10^{10}$ Pa.s (1.6×10^8 Torr.s). In general these characteristic values do not recover their former 'as received' level (e.g. $\delta_m = 1.74$ compared to the initial 2.29) even after a long air exposure of 2.7×10^{11} Pa.s (2×10^9 Torr.s or the equivalent of 30 days at atmospheric pressure).

5. INFLUENCE OF GLOW DISCHARGE TREATMENTS

Various gases, including pure argon, nitrogen, methane and a mixture of 90% argon with 10% oxygen, have been used to study the influence of ion bombardment in a glow-discharge on δ . The curves of $\delta(E)$ measured in-situ after a glow discharge with these gases on separate samples are shown in figure 5 and the characteristic values are given in the table 2. For all these glow discharges, the ion dose used was 3×10^{18} ions.cm⁻². As it would be difficult in practice to make these treatments in situ, the sequence of events and the choice of the gas used to let the system and newly prepared sample up to atmospheric pressure may be important. After most of the glow discharge treatments the system was first exposed to 1 atmosphere of dry nitrogen, rebaked under vacuum (150°C) before exposing to air, then again rebaking (150°C) under vacuum before measurement. The characteristic values after the different stages of this treatment are also given in table 2.

A methane glow discharge results in the formation of a carbon layer on the sample as shown by Auger analysis. This treatment, although it increases δ at low primary electron energy, it does give the lowest δ for electron energies above 1000 eV.

Argon and argon-oxygen glow discharge treatments produce surfaces with δ close to those measured on pure niobium (2)(3).

A nitrogen glow discharge gives the smallest δ_m (1.09 at 400 eV), and the smallest energy interval E_1, E_2 in which δ exceeds unity.

It is seen from table 2 that the effect of air exposure after these treatments depends largely on the gas used for the glow discharge. Nitrogen and argon-oxygen glow discharge produce the surfaces least sensitive to contamination by subsequent air exposure.

6. DISCUSSION

6.1 Influence of bakeout temperature and of air exposure

Secondary electron emission is, in general, strongly influenced by surface adsorbates (4). These adsorbates may stem from cleaning treatments and/or contact with the air. The modifications introduced to δ by bakeout and air exposure are mainly related to changes in these adsorbates. A bakeout at 80°C, by removing loosely bound adsorbates, influences in particular the low energy values of δ (δ_m and δ_{100}) which are the more sensitive to the superficial surface. Between 80 and 150°C, the change in the electron emissivity of niobium is marginal and bakeout temperatures exceeding 200°C are needed in order to produce notable effects on the other characteristic values of δ . This behaviour is linked to the desorption of more tightly bound adsorbates and/or to the modification of the surface oxide layer. In accelerating cavities, multiplication effects are generally due to low energy electrons (5) and hence the energy E_1 is of great importance: it can be considered as the threshold above which electron multiplication and enhanced losses can occur. An improvement in the performance of such devices can be obtained by raising E_1 , i.e. by raising the in-situ bakeout temperature above 200°C.

The measurements have also shown that E_1 is strongly affected by an air exposure: 30 s at atmospheric pressure is enough to lose most of the benefit of a bakeout. The effect of air exposure on E_2 and δ_m is less marked; after a day

at atmospheric pressure δ_m increased from 1.22 to 1.5, compared with 2.29 in the 'as received' state. After one week at atmospheric pressure δ_m was still lower (1.73) than in the initial state. The bakeout appears to have permanently removed or modified some surface properties present after the initial chemical treatment of the sample. An air exposure re-creates an adsorbed layer, still with a relatively low δ but, nevertheless, having a low E_1 and hence providing a potential source of electron multiplication and associated energy losses.

6.2 Influence of glow discharge treatments

The effect of glow discharge on surfaces is much more pronounced, as might be expected, since the energetic ion bombardment involved in this process is able to remove completely the surface oxide layer.

A non-reactive gas like argon creates a surface with characteristics which are very similar to those obtained after a 350°C bakeout (comparable E_1 , E_2 and δ_m). When exposed to air this glow-discharged surface is also very reactive and, for example, E_1 decreases to 38 eV, as in the case following a 350°C bakeout.

A methane discharge can be interesting in special circumstances where a very low δ is required at high incident electron energy.

Reactive gases, such as the argon-10% oxygen mixture or nitrogen, can provide a surface more immune to subsequent air exposure by creating a stable protective layer (60 to 100 Å) with the stoichiometry of NbN in the case of a nitrogen glow-discharge as shown by Auger measurements). For the nitrogen discharge E_1 still exceeds 70 eV after a 48 hours air exposure and is increased to 90 eV after a subsequent in-situ bakeout to 150°C.

7. CONCLUSION

These measurements have shown the strong influence of surface changes on the secondary electron emission from technical niobium. These changes are due to modification of the adsorbed layer and/or of the surface oxide. Passivation layers such as those created by glow discharge treatments with reactive gases can give a certain protection to the surface and stabilize the situation with respect to air exposure. They result in a permanent increase of the threshold energy E_1 and a reduction of δ_m , both factors favourable to a safer use of superconducting R.F. cavities.

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TABLE 1
INFLUENCE OF THE BAKEOUT TEMPERATURE
ON THE CHARACTERISTIC VALUES

Bakeout Temperature (°C)	100eV	1000eV	E ₁ (eV)	E ₂ (eV)	E _{max} (eV)	max
As received	1.97	1.64	27.6	-	250	2.29
80	1.63	1.48	29	-	300	2.
100	1.59	1.47	31.1	-	300	1.97
120	1.5	1.47	26.6	-	300	1.98
150	1.46	1.43	33.3	-	300	1.95
200	1.31	1.27	37.8	1900.	300	1.67
250	1.17	1.18	51.3	1600.	300	1.53
300	1.04	1.05	86.8	1220.	300	1.32
350	0.96	0.99	117	950.	350	1.22

TABLE 2

INFLUENCE OF GLOW DISCHARGE TREATMENTS
ON THE CHARACTERISTIC VALUES

Treatment	δ_{100} (eV)	δ_{1000} (eV)	E ₁ (eV)	E ₂ (eV)	E _m	δ_m
NITROGEN GLOW DISCHARGE						
after G.D.	0.83	0.94	150	750	400	1.09
94h. N ₂	1.01	1.03	100	1100	400	1.23
rebaked	0.9	0.99	150	950	300	1.23
48 h. air	1.07	1.13	70	1450	350	1.42
rebaked	1.07	1.09	90	1300	300	1.37
ARGON GLOW DISCHARGE						
after G.D.	0.87	0.98	115	980	300	1.25
8h. air	1.5	1.4	38	-	300	1.86
rebaked	1.03	1.05	90	1150	300	1.31
METHANE GLOW DISCHARGE						
after G.D	1.75	0.9	140	850	200	2
ARGON + 10% OXYGEN GLOW DISCHARGE						
after G.D.	1.03	0.94	120	1100	350	1.25
66h. N ₂	0.99	1.08	103	1250	350	1.32
rebaked	0.96	1.07	105	1250	350	1.31
66 h. air	1.09	1.16	80	1570	350	1.44
rebaked	1	1.1	100	1350	350	1.35

Figure 1 : Influence of bakeout temperature on δ_m , δ_{100} , δ_{1000}

Figure 2 : Influence of bakeout temperature on E_1 and E_2

Figure 2b: Definition of the characteristic values δ_m , E_1 , E_2 , E_m

Figure 3 : Variation of δ_m , δ_{100} , δ_{1000} with air exposure

Figure 4 : Variation of E_1 and E_2 with air exposure

Figure 5 : $\delta(E)$ curves after various glow discharge treatments