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Exploring the Proximity Effect in Mo/Au Bilayers

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***Abstract*—We report on the sensitivity of superconducting transition temperature (T_c) to the individual layers' thickness in Mo/Au proximity bilayers to be used in Transition-Edge Sensors. The achieved good reproducibility and quality of the bilayers allow a clear determination of the superconducting critical temperature T_c as a function of the Mo and Au thicknesses. One objective of this work is to analyse the quality of the Mo-Au interface and to assess the possible effects of the double Au layer we use to fabricate these bilayers and TESs based on them. Experimental data are analysed on the base of Usadel equations using the model developed by Martinis and co-workers, in which the proximity effect in the bilayer is mainly governed by the interface transparency between the superconductor and the normal metal. We find that this model describes quite well the behaviour of T_c, even for quite thick Au layers, and that the double Au layer does not play any relevant role on the proximity effect.**

***Index Terms*—Proximity Effects, Superconductors, Transition-Edge Sensors, X-ray Detectors**

I. Introduction

MOLYBDENUM/GOLD bilayers are among the most suitable materials for Transition-Edge Sensors (TESs), in cryogenic microcalorimeters and bolometers for the detection of radiation in a wide frequency range, from millimetre waves to gamma-rays [1,2]. These high performance devices are the base for ultra-sensitive detectors required for instruments in future space missions such as X-IFU-Athena [3] or SAFARI-SPICA [4] or for ground-based astronomy, and for other applications in science and industry.

TESs are constituted by superconducting thin films operated in the superconducting transition region, which takes place usually at temperatures ~100-400 mK. Most often, the superconducting film is a superconducting/normal metal (S/M) bilayer, whose superconducting critical temperature T_c can be tuned using the proximity effect by changing the thickness of the individual layers, respectively d_S and d_M. The quality of the interface between them also plays an important role on the T_c value and on the sharpness of the transition, which is ultimately related to the detectors performances.

For a bulk superconductor, the contact with a normal metal layer slightly reduces T_c but, for superconducting films thinner than the superconducting coherence length ($d_S<\xi_S$), T_c is reduced as the normal metal layer thickness (d_M) increases, till d_M reaches a critical value ξ_M , which is defined as the distance inside the normal metal where the superconductivity propagates: if $d_M \geq \xi_M$ no further suppression of T_c is found when changing d_M [5]. It is important to remark that different combinations of d_S and d_M values can provide the same T_c. As a consequence, the most often used approach to obtain a proximity bilayer with the desired T_c for TES devices consists on choosing the normal metal thickness (most usually Au or Cu) to adjust the resistance of the system to the required value, and then vary the superconducting layer thickness to tune T_c to the desired operating value.

In this work, we explore the evolution of the T_c of Mo/Au bilayers as a function of the Mo and Au thicknesses, d_{Mo} and d_{Au}, with the goal of analysing the quality of Mo/Au interface and the resulting proximity effect. Indeed, Mo/Au bilayers display two challenges to achieving the outstanding fabrication control required by TESs: the intrinsic high sensitivity of the critical temperature of molybdenum to the presence of impurities and stress, and the easy oxidation of Mo. We have already explored the dependence of the critical temperature of Mo films on their stress and thickness [6,7] and have demonstrated our ability to produce high quality reproducible Mo/Au bilayers using a trilayer approximation, Mo/Au/Au [8]; here we use the fabrication control achieved to characterize the proximity effect and determine the $T_c(d_{Mo},d_{Au})$, paying also attention to the possible impact of the double Au layer used to preserve the Mo/Au interface and reduce bilayer resistance.

Different models allow analytically determining the critical temperature of a S/M bilayer as a function of the interface quality and the individual layers' thickness [9,10,11,12]. We discuss our results in the framework of one of them, the Martinis approximation [12]. We find that this quite simple model describes well the $T_c(d_{Mo},d_{Au})$ dependences, even for the thicker Au layers used. Moreover, these results and the transparency values obtained reveal that the double Au layer has apparently no significant consequences regarding the proximity effect in these bilayers.

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II. Experimental

Mo/Au bilayers were deposited at room temperature by magnetron sputtering and electron beam evaporation on top of Si (100) single crystal substrates covered by a low stress Si_3N_4 layer, 0.5 or 1 μm-thick [8,13]. The Mo layers were deposited by RF magnetron sputtering in a UHV chamber, whereas the Au layer deposition procedure is twofold: first a protective Au layer with a thickness of 15 nm is deposited in-situ by DC magnetron sputtering immediately after Mo deposition. Then, an additional Au layer is evaporated ex-situ by e-beam. The two objectives of this deposition procedure are: to avoid Mo surface contamination by covering it in situ, and to reduce the bilayer electrical resistivity by increasing the gold grain size (e-beam provides larger grain sizes than sputtering, and thus lower Au resistivity). Optimum deposition conditions ensuring high quality, strain free Mo layers have been described elsewhere [6,13].

For this study several batches of bilayers were fabricated, with either Mo or Au layer thicknesses kept constant. d_{Mo} and d_{Au} were selected to range from 40 to 120 nm and from 15 to 575 nm, respectively. The lower limit for Mo thickness is chosen in order to avoid finite size effects that could affect Mo functional properties, mainly T_c [7].

For the purpose of the analysis reported here, a good control of thicknesses is essential, since thickness variations will result in apparent T_c irreproducibility or the broadening of the transition. The thickness of sputtered Mo and Au films is measured by X-ray reflectometry; the thickness control in these films is better than 3%, for the thickness range explored in this work. For Au films deposited by e-beam, the thickness is measured by a profilometer; in this case, the control is of the order of 2-3%.

Transverse Transmission Electron Microscopy (TEM) has been performed in selected bilayers in order to analyse the morphology of the Au and Mo layers and their interface.

The temperature dependence of the resistance and the critical temperature of the different bilayers down to 350 mK were measured by using the four points resistance method in a commercial Physical Property Measurement System (PPMS) from Quantum Design. At lower temperatures, in the range between 30 mK and 350 mK, a commercial dilution refrigerator (Kelvinox MX40) from Oxford Instruments with an AVS-47 resistance bridge was used. In both cases the bias current was 10 μA. The transition temperature T_c was defined as the temperature at which the measured resistance departs from zero.

Resistivity values of the layers are [6]: 125nΩm for Mo films above 30nm, 40nΩm for 14-nm-thick sputtered Au, and 16, 6.3 and 4nΩm for Au films deposited by e-beam with thicknesses 15, 45 and >130nm, respectively.

III. Results and Discussion

Fig.1 displays a transverse TEM image of a bilayer constituted by 55 nm Mo, 15 nm sputtered Au and 115 nm Au deposited by e-beam. Since all the bilayers include 15nm sputtered Au, from now on we will refer to only two thicknesses, the Mo and the total Au; in this case, a film of 55/15/115 will be described as 55/130. As reported previously, the interface between Mo and Au is sharp, and Au deposited by sputtering and by e-beam are not distinguishable. Both metals (Mo, Au) display columnar growth. The average Mo column width is 20 nm. Au initially grows following Mo columns, with average grain size ~20 nm at the interface. As the thickness of the gold layer increases the grain size and its dispersion increase, up to 70-100 nm on average [6,14]. Thus, the thickest Au layers studied in this work display column widths of the order of 100 nm and increasing disorder.

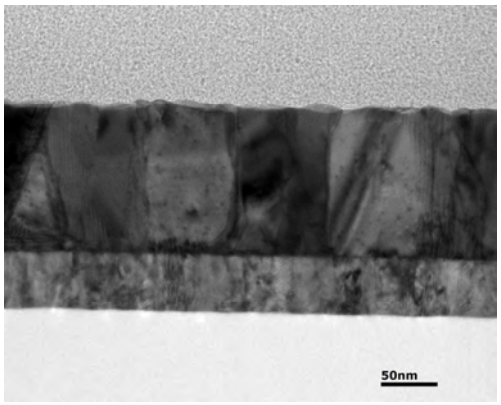

Fig. 1. Transverse Transmission Electron Microscope image of a Mo/Au bilayer constituted by 55 nm Mo, 15 nm sputtered Au and 115 nm Au deposited by e-beam. Columnar morphology of both molybdenum and gold is observed, as well as a sharp, flat interface between them.

The average critical temperature of Mo films with thickness d_{Mo}>35-40 nm grown in parallel to the bilayers analysed is T_{co}=999±11 mK; for thinner films finite thickness effects are observed [7]. This value is in good agreement with previous results of T_{co} for bare sputtered Mo films, full within the range of critical temperatures reported for Mo thin films [6,7,16,17,18] but somehow above the critical temperature of bulk Mo, 915 mK.

The superconducting transitions of several selected Mo/Au bilayers with different Mo and Au thicknesses are shown in Figs. 2. Transitions shift in a parallel way (without broadening) by changing the superconductor or metal thicknesses. All of them are extremely sharp, with typical transition widths <5 mK. This sharpness, in large area, unpatterned bilayers [15], reveals their uniformity and the high quality of the Mo/Au interface, and therefore the high foreseeable TES sensitivity [13].

The dependence of T_c on d_{Mo} and d_{Au} is shown in Figs. 3. As expected, T_c diminishes with decreasing Mo thickness. In addition, T_c decreases monotonously with increasing d_{Au}, from the critical temperature of Mo, T_{co}, to nearly zero (our lowest accessible temperature is ~30 mK).

The critical temperature of S/M bilayers can be estimated by using the model developed by Martinis et al. [12], based on Usadel equations [19]. Within the so called thin films limit, in which both layers are thin enough to assume that the superconducting order parameter is approximately constant across the film thickness, T_c is given in terms of the critical temperature of the superconducting layer T_{co}, the superconducting and normal metal layers' thicknesses d_S and d_M and the interface transparency t as follows:

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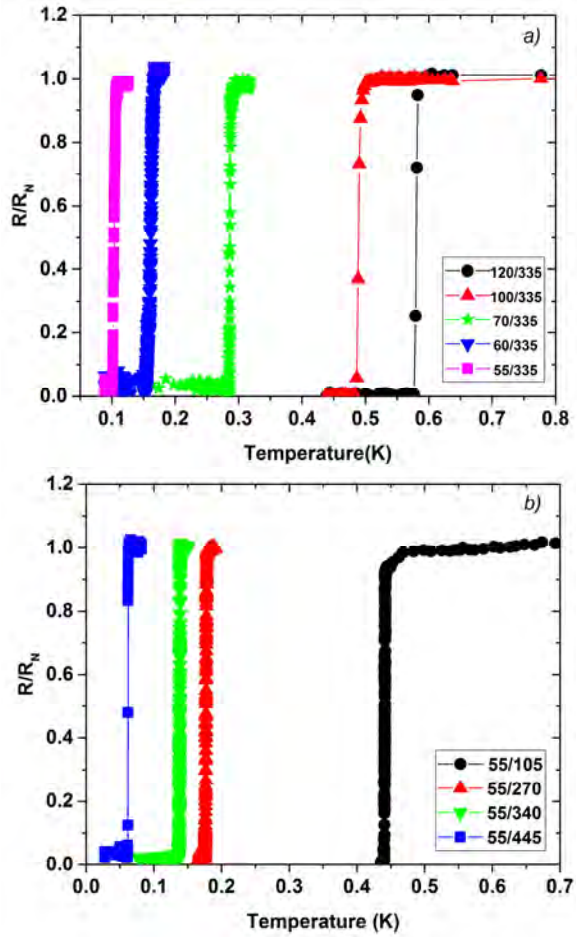
$$T_c = T_{co}\left[\frac{d_S}{d_0}\frac{1}{1.13(1+1/b)}\frac{1}{t}\right]^b$$
$$\frac{1}{d_0} = \frac{\pi}{2}k_B T_{co}\lambda_f^2 n_S \qquad (1)$$
$$b = d_M n_M / d_S n_S$$

where λ_f is the normal metal Fermi wavelength, and n_M and n_S are the density of states in the normal and superconducting layers respectively. Note that the dependence of T_c on d_M is only through the thickness ratio d_M/d_S (inside parameter b), whereas d_S appears also alone in (1).

In the case where the layers cannot be assumed to be in the thin limit, Martinis et al. [12] showed that (1) may still hold but substituting t by and effective transparency t', dependent on the resistivity of the layer or layers, according to:

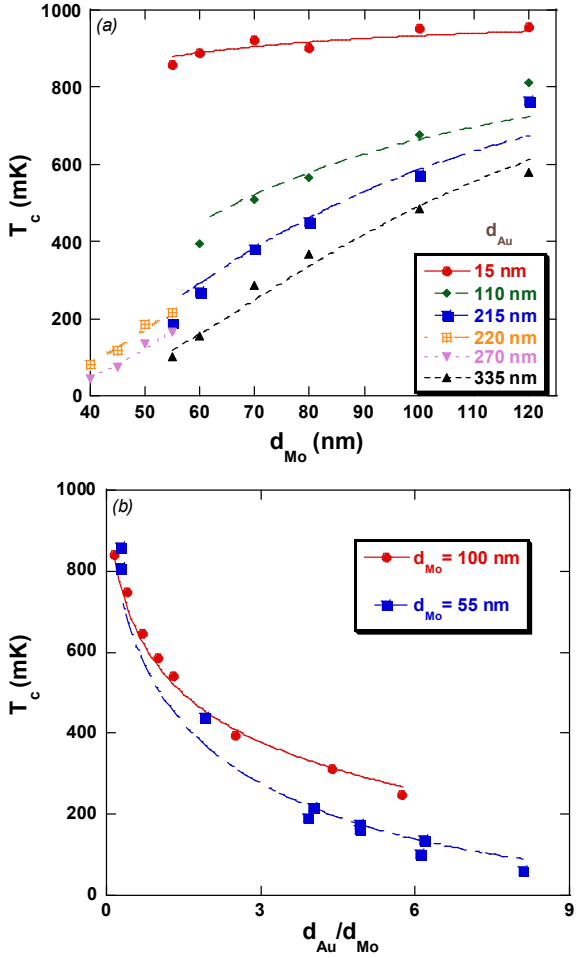
$$\frac{1}{t'} = \frac{1}{t} + \frac{1}{3}\left(\frac{d_S}{0.013\,\mu m}\frac{\rho_S}{\rho_{S0}} + \frac{d_M}{0{,}153\,\mu m}\frac{\rho_M}{\rho_{M0}}\right) \qquad (2)$$



Figs. 2. Resistive transition of several Mo/Au bilayers with changing Mo thickness (a) and Au thickness (b). The superconducting critical temperature is shown to decrease for increasing Au thickness and decreasing Mo thickness.

ρ_{S0} and ρ_S (ρ_{M0} and ρ_M) in (2) are respectively the room temperature nominal resistivity and the thin film resistivity of the S (M) material. This expression reveals that the thick-film correction results effectively in a reduction of the interface transparency, and thus a smoother decrease of T_c with increasing normal metal thickness should be expected. Equivalently, slightly higher T_c's would result for bilayers of given thicknesses d_{Mo}, d_{Au} if this correction applies. The effective reduction of the interface transparency for thick films is enhanced as the resistivities of the layers increase. We find that this thick film correction is not significant compared to our experimental error bar.

Indeed, data in Figs.3 are fitted to (1) with t as a single fitting parameter, without use of the thick film corrections (2), and using T_{co}=1000 mK, the densities of states of Mo and Au $n_{Mo}=0.29\cdot10^{23}$ states/eVcm^3 [12], $n_{Au}=0.107\cdot10^{23}$ states/eVcm^3 [20], and the Fermi length of Au, λ_f=0.524 nm [20]. The interface transparencies obtained from these fits are summarized in Table I. Several conclusions can be extracted from these fits. First, (1) describes well the behavior of the bilayers critical temperature as a function of the Mo and Au thicknesses.



Figs. 3. Evolution of the critical temperature of bilayers with Mo thickness for several dAu (a), and with Au thickness, for two different Mo thicknesses (b). Lines are fits to (1).

This is even more important because the high sensitivity of the critical temperature of Mo to impurities and stress results often in a scatter of T_{co} values which difficults analyzing the proximity effect. The transparency values shown in Table I reveal that

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the reproducibility achieved with the analysed bilayers is quite good. We have further checked this by performing the following test: a set of bilayers with d_{Mo}=55 nm and belonging to different batches with fabrication dates spanning during several months (set labeled as "mixed" batch in Table I) was fitted, and the results compared to those of a new batch with the same d_{Mo}; finally, both batches were fitted together. As table I reveals, the agreement of the three fits is very good.

It is also remarkable that all the interface transparency values obtained for the different bilayer sets are very close, around t~0.1 (except for the batch with d_{Mo}=100 nm, which displays a higher value, t~0.13). This gives further coherence to the analyses and fits. The t values obtained are similar to those reported for Mo-based bilayers that constitute TESs with excellent performances [16,17,18,20,21].

The ability of (1) to describe our $T_c(d_{Mo},d_{Au})$ is significant in two other respects: on one hand, this, together with the similarity of the t values obtained to other reported, means that the trilayer structure of our bilayers, whereby the gold layer is in fact constituted by two layers deposited by sputtering and e-beam, has no relevance regarding the Mo/Au proximity effect; that is, only the Mo/Au interface plays a role. This fact is in agreement with the observation by TEM that there is not a clear interface between the Au layers deposited by either technique, being them undistinguishable. On the other hand, (1) holds even for quite thick gold layers, up to 575 nm. We have checked that by performing two fits with the data of the bilayers with d_{Mo}=100 nm: (i) taking all the data points, and (ii) using only the data for d_{Au}<130 nm. If the thin film limit did not apply for the thicker d_{Au}, the fits to (1) would still work but, according to (2), the effective transparency would be lower. Therefore, fits using data up to 130 nm should provide an interface transparency higher than when using all the data up to the highest d_{Au}. The fact that identical transparency values are obtained in both cases is indicative that the thick film correction is irrelevant within the ~10% error of the fitted t values. This is somehow surprising. Indeed, the thin film limit is expected to hold when the layers' thicknesses are smaller than the coherence lengths of either layer, that is, when $d_{Mo}<\xi_{Mo}$ or $d_{Au}<\xi_{Au}$. According to [22], the coherence length of the normal metal ξ_M can be estimated as follows:

$$\xi_M = \sqrt{\frac{\hbar v_M^f \ell_M}{6\pi k_B T_{co}}} \qquad (3)$$

where T_{co} is the critical temperature of the superconducting layer (molybdenum), and v_M^f and ℓ_M are the Fermi velocity and the mean free path of the normal metal layer (gold), respectively. In the case of highly disordered metallic thin films, like the bilayers under study, the mean free path is limited by the grain size. From the column widths extracted from TEM analyses (see above), ℓ_M is found to range from 20 to 100 nm. Then, according to (3) the Au coherence length is calculated to be ξ_{Au} ~ 100 - 230 nm by using the experimental T_{co}=1000 mK and v_m^f=1.39·10^6 m/s [23]. Hence, some of the bilayers in this study (in particular those with thicker Au in batch Z41) should require the thick film correction for Au. However, the quality of the fits does not deteriorate for higher d_{Au}, nor is there any significant change in the transparency extracted from them. Therefore, the simplified expression (1) is able to describe the $T_c(d_{Au},d_{Mo})$ behaviour of the bilayers up to the maximum thicknesses analysed, well above the estimated value for ξ_{Au}.

TABLE I
INTERFACE TRANSPARENCIES

Batch	d_{Mo}(nm)	d_{Au}(nm)	t
Z32	55	15-445	0.093±0.007
Mixed*	55	15-335	0.093±0.007
Z32+mixed*	55	15-445	0.097±0.006
Z41	100	15-575	0.132±0.003
Z41#	100	15-130#	0.13±0.01
Z31	60-120	110	0.08±0.007
Z29	55-120	215	0.09±0.004
Z35	40-55	220	0.095±0.002
Z35	40-55	270	0.100±0.002
Z31	55-120	335	0.100±0.002

Interface transparency t obtained from the fits of data in Figs. 2 to (1). T_{co} was settled to 1000 mK. Batches of bilayers varying d_{Mo} and/or d_{Au} have been tested.
*The "mixed" batch stands for a set of bilayers belonging to different batches and with d_{Mo}=55 nm. In order to check reproducibility, fits were performed on data from this batch, from batch Z32 and from both batches together (row 3). Very good agreement between the three fits is obtained.
Fits corresponding to data from batch Z41 limited to d_{Au}<130 nm (see text).

In the case of Mo, the intrinsic coherence length of sputtered Mo films similar to those in the bilayers studied here was estimated to be ξ_S~85 nm [7]. Since the maximum Mo thickness in this study is 120 nm, it is reasonable to assume that the superconducting layer is in the thin film limit or near it, so that the thick film correction for Mo will be small and can be reasonably ignored. The fact that T_c in Fig.3a is still far from T_{co} for d_{Mo}=120 nm supports this assumption.

IV. CONCLUSIONS

The superconducting critical temperature of Mo/Au bilayers has been studied as a function of the thicknesses of both the superconductor and the normal metal layers, in order to study the proximity effect. The goals of this study were to evaluate the impact of the double Au layer used on the proximity effect, as well as the quality of the interface. It is found that $T_c(d_{Mo},d_{Au})$ is well described by the expression obtained by Martinis et al., even for the bilayers with quite thick Au (above 500 nm). The results evidence the good quality of our bilayers and the reproducibility of the fabrication process. They also reveal that the double Au layer used has no consequences regarding the superconducting/metal proximity effect.

This study allows selection of bilayers with different thickness and targeted T_c suitable for TESs, thus allowing also the choice of the devices resistance. We have fabricated bare square TESs (without banks or other structures) with T_c=100 mK have been fabricated using 55/335 nm and 45/265 nm bilayers, with R_n respectively 15 and 19 mΩ.

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