A study of the ferromagnetic transition of $\text{SrRuO}_3$ in nanometer thick bilayers with $\text{YBa}_2\text{Cu}_3\text{O}_{y}$, $\text{La}_{1.88}\text{Sr}_{0.12}\text{Cu}_4\text{O}_{4-y}$, $\text{Au}$ and $\text{Cr}$: Signature of injected carriers in the pseudogap regime

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(Dated: February 1, 2008)

The hypothesis regarding the existence of uncorrelated pre-formed pairs in the pseudogap regime of superconducting $\text{YBa}_2\text{Cu}_3\text{O}_y$ is tested experimentally using bilayers of $\text{YBa}_2\text{Cu}_3\text{O}_y$ and the itinerant ferromagnet $\text{SrRuO}_3$. In our study, we monitor the influence of $\text{YBa}_2\text{Cu}_3\text{O}_y$ on $\text{SrRuO}_3$, the ferromagnetic ordering temperature of $\text{SrRuO}_3$. Here, $T_p$ is the temperature of maximum $\text{dM}/\text{dT}$ or $\text{dR}/\text{dT}$ where $M$ and $R$ are the magnetization and resistance of $\text{SrRuO}_3$, respectively. We compare the results with similar measurements carried out on bilayers of $\text{La}_{1.88}\text{Sr}_{0.12}\text{Cu}_4\text{O}_{4-y}$, $\text{Au}$ and $\text{Cr}$ with $\text{SrRuO}_3$. We find that in bilayers made of underdoped 10 nm $\text{YBa}_2\text{Cu}_3\text{O}_y$/5 nm $\text{SrRuO}_3$, the $T_p$ values are shifted to lower temperatures by up to 6-8 K as compared to $T_p \approx 140$ K of the 5 nm thick reference $\text{SrRuO}_3$ film. In contrast, in the other type of bilayers, which are not in the pseudogap regime near $T_p$, only a smaller shift of up to $\pm 2$ K is observed. These differences are discussed in terms of a proximity effect, where carriers from the $\text{YBa}_2\text{Cu}_3\text{O}_y$ layer are injected into the $\text{SrRuO}_3$ layer and vice versa. We suggest that correlated electrons in the pseudogap regime of $\text{YBa}_2\text{Cu}_3\text{O}_y$ are responsible for the observed large $T_p$ shifts.

PACS numbers: 74.45.+c, 75.70.-i, 74.50.+r, 74.78.Bz

There exists a large body of experimental evidence to date for the existence of a pseudogap regime above $T_c$ in the high temperature superconducting cuprates \cite{1, 2}. Among the several possible models put forward to explain the origin of this phenomenon, the precursor pairs scenario carries a certain appeal. This model assumes the existence of pre-formed pairs in the pseudogap regime which do not show phase coherence. On lowering the temperature, these precursor pairs reach phase coherence at the superconducting transition temperature $T_c$ \cite{3}. The pre-formed pair scenario is consistent with many experimental results in the pseudogap regime, but experimental verification for the existence of the elusive pre-formed pairs is still lacking. In the present study, we investigate the proximity effect in the pseudogap regime using bilayers of $\text{YBa}_2\text{Cu}_3\text{O}_y$ (YBCO) and the itinerant ferromagnet $\text{SrRuO}_3$ (SRO). We observe that the proximity effect leads to a large decrease of the ferromagnetic transition temperature. Ferromagnetic order is incompatible with singlet Cooper pairs. Consequently, the presumed pre-formed pairs are likely candidates to explain this effect. Previous results on the proximity effect using superconducting-ferromagnetic (SF) bilayers and multilayers have been reported mostly for YBCO and a manganite such as $\text{La}_{2/3}\text{Ca}_{1/3}\text{Mn}_3\text{O}_4$ (LCMO) showing giant magnetoresistance. It was shown by magnetization measurements, that the temperatures where the magnetic moment saturates below the superconducting $T_c$, and of the onset of the ferromagnetic transition at $T_{\text{Curie}}$ above $T_c$, are both suppressed with increasing thickness of the superconducting layer \cite{4}. The results above $T_c$ were interpreted as due to a possible charge transfer from the LCMO to the YBCO layer. Similar results on the suppression of $T_{\text{Curie}}$ versus the YBCO thickness in multilayers of YBCO/LCMO above $T_c$ were obtained also in resistivity and susceptibility measurements \cite{5, 6}. The opposite effect, where the superconducting $T_c$ is depressed by the ferromagnetic layer is also possible due to pair breaking by spin polarized carriers penetrating the superconductor \cite{4, 5, 7}. No reference to the pseudogap or preformed pairs role in this context was mentioned or discussed in these studies.

In our experiment, epitaxial thin films and bilayers of SRO with either YBCO, $\text{La}_{1.88}\text{Sr}_{0.12}\text{Cu}_4\text{O}_{4-y}$ (LSCO), Cr or Au were deposited in-situ by laser ablation deposition on (100) $\text{SrTiO}_3$ (STO) wafers of $10 \times 10$ mm$^2$ area. For reference, nominally identical single layer films of these materials on STO were also prepared. All SRO, YBCO and LSCO layers in the different heterostructures were oriented with their $c$-axis normal to the wafer. The SRO and LSCO layers were prepared under the same deposition conditions as for obtaining high quality YBCO films (100 mT of oxygen flow and at 780°C). The Cr layer was deposited under vacuum at 30°C, while the Au layer was deposited in 400 mT oxygen at 150°C. Transmission electron microscope (TEM) images of similar SRO films deposited on (100) STO show an atomically smooth interface with the STO wafer and were found to grow in the layer-by-layer mode up to at least 10 nm thickness \cite{8}. Fig. 1 shows an image of the surface morphology of one of our SRO films measured by a scanning tunneling microscope (STM). One can see that this film consists of a stack of...
parallel and flat plates which are $\sim 60 - 90$ nm wide and atomically smooth. These plates are separated by steps of one unit cell height (the $c$-axis of SRO), and are formed due to the $\sim 0.5^\circ$ miscut angle of the STO substrate. Thus the area onto which the cover layers in the bilayers with SRO are deposited is atomically smooth and almost flat. In view of the small fraction of the total area of the film that the atomic steps occupy, we can safely assume that the dominant contribution to the properties of the bilayers originates in the flat areas. TEM images of the YBCO/SRO interface are also atomically smooth and similar to the YBCO/LCMO interfaces in super-lattices [10]. We can therefore conclude that the sharp and flat interfaces exclude any inter-diffusion and chemical reactions which could affect our results. Recently, epitaxial $SrTiO_3/LaAlO_3$ bilayers with different termination layers at the interface were investigated [11]. The results show that the effects of ionic and charge compensation for the different cases play a major role in enabling the creation of a sharp interface on a scale of a single atomic layer. In that study however [11], both components of the bilayer are ionic and insulating, while in our case, all the layers are metallic and conducting. Therefore, charge compensation is taken care of automatically, and the resulting interfaces are smooth and sharp. We also note that even if only one layer in the bilayer is conducting like in the SRO/STO case, the interface is clearly sharp [8].

In order to minimize effects of wafer to wafer variations between different deposition runs, some bilayers were prepared $m$-situ together with the reference films in the following way: first, the SRO layer was deposited on the whole area of the wafer. Then a shadow mask made of an MgO wafer was used to cover half of the sample area while the YBCO layer was deposited on the other half. This resulted in an SRO reference film on half the wafer and a bilayer on the other half. The various single layer films and bilayers deposited on the whole area of the wafer were not patterned. Wafers which were half coated with a bilayer and half with the SRO film were patterned by either wet etching or a scratch of a narrow stripe to separate the bilayer from the reference film. A very convenient feature of our experiment was that YBCO/SRO bilayers could be reannealed in $O_2$ to produce YBCO with different values of $T_c$ without affecting the properties of the SRO. We note that the oxygen annealing process was done at 450$^\circ$C which is about half the deposition temperature, and was fully reversible. Namely, we could switch back and forth between the different $T_c$ values of the bilayers without any change in their properties. This again is consistent with the absence of interdiffusion which would have affected the properties of the bilayer in a progressive and irreproducible manner. This convenient feature allowed us to compare the proximity effects at different doping levels on the same sample. Transport measurements were done by the standard 4-probe technique using gold coated contact tips. All in all, about 30 bilayers and reference films were prepared and measured, to establish the reliability and reproducibility of our results.

In the present study, resistance versus temperature measurements were used rather than direct magnetization measurements, because of the higher sensitivity that can be obtained in measuring a few nanometer thick SRO film (typically 5 nm). Theoretically, the issue of resistive anomalies and peaks associated with ferromagnetic transitions was discussed quite long ago [12, 13]. Here we wish to show first, experimentally, that near the ferromagnetic transition, the functional dependence of $\frac{dR}{dT}$ is very similar to that of $\frac{dM}{dT}$ where $R$ is the resistance, $T$ is the temperature and $M$ is the magnetization of the sample. For this, magnetization measurements as a function of temperature were performed using a SQUID magnetometer, and compared with measurements of $R$ and $\frac{dR}{dT}$ of the same sample. Fig. 2 shows the results of these measurements on a 200 nm thick SRO film. One can see that on cooling down, the magnetization curve starts rising at $T_{Curie} \approx 150$ K where the ferromagnetic order sets in, and that this temperature coincides with the corresponding sharp increase of both $\frac{dR}{dT}$ and $\frac{dM}{dT}$. The inflection point of $M$ versus $T$ yields a peak in $\frac{dM}{dT}$ which also coincides with the peak of $\frac{dR}{dT}$. We denote this temperature by $T_p$. Thus a basic correspondence between $\frac{dR}{dT}$ and $\frac{dM}{dT}$ near $T_{Curie}$ is well established. For the thin films of a few nm thickness used in this study, the magnetization signal was too small to measure. We therefore used measurements of $\frac{dR}{dT}$ to detect $T_p$, the midpoint of the ferromagnetic transition.
The choice of thickness of the films in this study was done by searching for the combination that would produce a large observable effect on the SRO transition. In general, to achieve good sensitivity, the resistances of the two components of the bilayer should be comparable. Second, the thickness should be in the range of the relevant penetration depths of the proximity effect. If one of the layers is much thicker than the penetration depth, the observable effect will be small. For example, with the YBCO film much thicker than the SRO, the SRO transition was suppressed to the point where an unambiguous identification of \( T_p \) became impossible. Finally, if the layers are too thin, the number of carriers available for injection into the other film is too small to produce an observable effect. For example, as shown below, in bilayers of 7.5 nm YBCO on 5 nm SRO, any suppression of the SRO transition temperature was smaller than the variation of \( T_p \) between different SRO films. In contrast, a clear suppression was observed in bilayers of 10 nm YBCO on 5 nm SRO. The thickness of the films used in this study indicates that the range of the proximity effect is on the order of a few nm. In view of the effects discussed above concerning the layers thickness in the bilayers, we also conclude that the measured results in the bilayers reflect mostly their bulk properties and not the interface.

Figs. 3 and 4 show the resistance versus temperature and the corresponding temperature derivatives \( dR/dT \) of a 5 nm thick SRO film, and of a 7.5 nm YBCO/5 nm SRO bilayer under three different oxygenation levels. The value of \( T_p \) in 5 nm thick SRO films is typically around 137-140 K, slightly lower than in the bulk (144 K, as seen in Fig. 2). It was established independently that the SRO films are insensitive to the oxygen annealing conditions used in the present study. In contrast, the YBCO layer in the bilayer is very sensitive to the oxygen annealing conditions, and in Figs. 3 and 4 we show the results for the 30, 60 and 90 K phases of YBCO obtained on the same bilayer through repeated annealing. We note that the oxygen annealing process is reversible, as we can switch between different \( T_c \) values with reproducible transport results. This multiple annealing without deterioration in the transport properties is apparently due to the fact that the oxygen annealing temperature (450°C) is much lower than the deposition temperature (780°C) where the layered structure is formed. Fig. 4 shows that at temperatures above 100 K, the 30 K bilayer data is almost coincident with that of the SRO film. This is due to the high normal resistivity of the 30 K YBCO phase and indicates very little interaction between the layers.
in the case. For the 60 and 90 K phases of YBCO this is no longer the case. A suppression of the magnitude of $dR/dT$ near $T_p$ is clearly observed. To account for the different resistances of the differently oxygenated bilayers as in Fig. 3, and for the sample to sample variability due to morphology differences and so on, we generally normalized the $dR/dT$ data to that of the SRO reference film in the temperature range of 160-180 K above the ferromagnetic transition at $T_{Curie}=150$ K. In cases where the $dR/dT$ curves did not fully overlap in this regime, normalization was done around 170 K, like in Fig. 4. The normalized curves show that $T_p$, the midpoint of the ferromagnetic transition (shown by the curves without symbols in Fig. 4) remains constant at 137 K, but the overall magnitude of the $dR/dT$ signal is still significantly suppressed in these bilayers.

In the next step, we prepared bilayers with a slightly thicker YBCO layer. Fig. 5 shows the $dR/dT$ data versus temperature of a bilayer of 10 nm thick YBCO film on top of a 5 nm thick SRO layer, together with the data of the corresponding reference films. Also shown in Fig. 5 is the expected resistance of the same bilayer structure, calculated when the two layers are assumed to be noninteracting and behave like two independent resistors connected in parallel (termed "calculated" in Fig. 5). The bilayer and films were annealed in a low oxygen pressure (of 10 mTorr $O_2$ flow) to produce the $T_c=60$ K phase of YBCO. At this doping level, the pseudogap regime of YBCO sets in below $T^*$ of about 170-200 K (this is seen as the broad shallow peak of the YBCO film in Fig. 5). As before, the SRO films are found to be insensitive to the oxygen annealing pressure, with $T_{Curie}$ of 150 K. This is easily seen by the change of slope at 150 K of the resistance versus temperature curve of the SRO film in Fig. 3, and by the corresponding sharp increase of $dR/dT$ in Fig. 5 at 150 K. These observations are in agreement with the results found in the literature [14, 15]. Just below 150 K, the measured $dR/dT$ of the bilayer in Fig. 5 is clearly lower than the calculated resistance of the corresponding noninteracting bilayer. Thus one can conclude that the different layers do affect one another in such a way that decreases their $dR/dT$ below the ferromagnetic transition temperature. Here, a shift of the ferromagnetic peak temperature $T_p$ to lower temperatures is observed, and this will be discussed in more detail next.
Figure 6 shows dR/dT results versus temperature of a 10 nm YBCO/5 nm SRO bilayer obtained on the same wafer by re-annealing in oxygen to produce the 30, 60 and 90 K YBCO phases. A clear shift to lower temperatures of 6-8 K is now observed in the peak position of the ferromagnetic ordering temperature $T_p$, in the bilayers of the 30 and 60 K YBCO phases, relative to the $T_p$ values of two reference SRO films with high and low resistances. We verified by atomic force microscopy, that the different resistances of the two 5 nm thick SRO films are related to morphology changes in the films. This can be due to a slight difference in the miscut angle of the (100) SrTiO$_3$ wafers or to small film thickness variations in different deposition runs. To minimize this large variability in the properties of the SRO films, we prepared additional samples with the reference SRO film on half the area of the wafer and the bilayer on the other half as described in the experimental part before. This geometry is depicted in the inset of Fig. 7. The results of the normalized dR/dT in such a sample are shown in Fig. 7. Basically, the results of Fig. 6 are now reproduced in Fig. 7 in a bilayer with the 60 K YBCO phase where again, a large 6 K shift of $T_p$ is found. This time however, the results were obtained with two SRO films (one in the bilayer and the other of the reference film) which were prepared in the same deposition run and on the same wafer. Therefore, the variability in the SRO properties in this case is minimal, and the reliability and reproducibility of the $T_p$ shift is well established. A smaller shift of $T_p$ of 2-5 K is found in the 90 K bilayer of Fig. 6, but the corresponding dR/dT peak is too small and broad for a clear determination of the shift. Suppression in the magnitude of dR/dT near the SRO peak is evident in all bilayers in Figs. 6 and 7, and more so in the 90 K bilayer, similar to the results of Fig. 4.

Since the largest shifts of the ferromagnetic ordering temperature $T_p$ are observed in the 30 and 60 K bilayers, it is tempting to attribute this behavior to the fact that around 140K, YBCO with $T_c$ of 30 K or 60 K is within its pseudogap regime, while the 90 K phase of YBCO is above its pseudogap regime, if at all, exists only very close to $T_c$. i. Bilayers of 5 nm SRO with 10 nm thick cuprate films for which the relevant temperature range is not in the pseudogap regime. These include $La_{1.88}Sr_{0.12}CuO$_4$_{-y}$/5 nm SRO, together with the dR/dT data of a 5 nm thick SRO reference film. Normalization to the reference film is done in the 160-180 K temperature range. The inset shows the raw data of R versus T.

SRO, while in case (iii) there is a competition between two different types of magnetic order parameters. The proximity effect with an underdoped YBCO should give similar results to case (iii), unless preformed pairs exist. In the latter case, each injected pair carries two charges with zero spin, which should decrease both the polarization and the ferromagnetic correlations in the SRO layer in a more noticeable way than in the case where the injected charges are uncorrelated. One can argue qualitatively that if the underdoped YBCO contains singlet Cooper pairs, the probability of injecting two charges with zero spin into the SRO is $P_{1↓} = 1$ because of the electron-electron correlations, while for uncorrelated electrons the injection probability $P_{1↓}$ would be only $P_{1↓} = 0.5 \times 0.5 = 0.25$. Thus clearly, the injection of correlated pairs should have a stronger effect on $T_p$, the midpoint of the ferromagnetic transition.

The dR/dT versus temperature results of the bilayers of the control experiments (of which the LSCO data was obtained in the configuration shown by the inset of Fig. 7), are shown in Figs. 8 and 9. This time the dR/dT peak of the 90 K YBCO bilayer in Fig. 8 is stronger and allows a clear determination of its peak value $T_p$. Fig. 8 shows that the $T_p$ values of both the LSCO and 90 K YBCO bilayers coincide to within the experimental error with that of the SRO reference film at $T_p \approx 139$ K. Moreover, the $T_p$ value of the Cr bilayer of Fig. 9 shifts by only 2 K to lower temperature relative to the reference SRO film, while in the Au bilayer a similar $T_p$ shift is observed but to higher temperatures. Thus, these $\pm 2$ K $T_p$ shifts are significantly smaller than the 6-8 K shifts observed in the 30 and 60 K bilayers of Figs. 6 and 7.
could still argue that the large preformed pairs scenario in the pseudogap regime. One in Figs. 6 and 7 for the 30 and 60 K bilayers are due to two 5 nm thick SRO films. Normalization is done Cr/5 nm SRO and 10 nm Au/5 nm SRO bilayers normalized to the large \( T_p \) observed. Thus the large \( T_p \) shifts observed when the YBCO layer was thicker (10 nm thick), have nothing to do with interface reactions, if any, and result from the added amount of YBCO. We also wish to stress that in both Cr/SRO and Au/SRO bilayers, deposition of the Cr and Au layers was done at low temperatures (30 and 150°C, respectively). Hence, no interdiffusion into the SRO could have occurred in these cases either. Concerning density of states considerations, we note that the similar resistivities of SRO, YBCO and LSCO indicates that the density of states in these materials is on the same order of magnitude. The density of states of the Cr and Au metals are much higher than that of SRO, and as a result a higher injection rate into the SRO is expected. This however, did not lead to larger \( T_p \) shifts in these cases. An alternative model for the pseudogap regime involves large superconducting fluctuations above \( T_c \), while phase coherence is reached at \( T_c \). Such a model is consistent with the Nernst results where the onset of this effect follows a higher temperature dome similar to the lower temperature dome at \( T_c \) [17]. Our results for the 12% Sr doped LSCO where no \( T_p \) shift was observed (Fig. 8), are consistent with the Nernst onset at 110-120 K, since this is out of the ferromagnetic transition range of SRO at 130-150 K. Unfortunately, there is no similar data on the Nernst onset for YBCO, so no further comparison with the YBCO results can be made. We note however that our LSCO results are in disagreement with the pseudogap \( T^* \) \( \approx \)160 K obtained from specific heat measurements [17], but agree well with the \( T^* \) \( \approx \)60 K obtained from STM results [16].

Finally, we discuss the above conclusion and consider alternative interpretations of our results. First, we wish to discuss possible interdiffusion effects at the interface on our results. From the atomically smooth surface morphology of the SRO layer as seen in Fig. 1, and the various TEM images [8, 9, 10], it is clear that the interface in the bilayers with SRO is sharp and flat. Therefore, interdiffusion in such an interface is quite unlikely. Moreover, even if we assume that reaction products are present at the interface, our results are not affected by them since in the 7.5 nm YBCO/5 nm SRO bilayers (see Fig. 4), no shift of \( T_p \) was observed. Thus we conclude that in this regime in YBCO, electrons with large \( T_p \) shifts are due to a loss of itinerant spin polarized electrons in the SRO layer caused by an inverse proximity effect, where these electrons are injected into the adjacent normal chromium layer. Injection of electrons in the opposite direction into the SRO layer should in principle enhance its ferromagnetism due to the presence of the exchange field. This seems to be the case in the Au/SRO bilayer in this Figure, where the gold has a higher density of conduction electrons than SRO, but without spin correlations. In the Cr/SRO bilayer, this is not the case since the carriers injected into the SRO, although uncorrelated, may have some degree of antiferromagnetic spin correlations which would tend to hinder the formation of the ferromagnetic order (\( T_p \)) in the SRO layer. This leads to the conclusion that the large \( T_p \) shifts observed in Figs. 6 and 7 for the 30 and 60 K bilayers are due to injection of correlated electrons, similarly to the preformed pairs scenario in the pseudogap regime. One could still argue that the large \( T_p \) shifts down are due to the inverse proximity effect, where a loss of itinerant spin polarized electrons occurs as in the chromium case. This however, would necessitate that the density of states near the Fermi surface of the 90 K YBCO phase be lower than that of the 30 and 60 K phases of YBCO, which is not the case. We note that the density of states in all the hole-doped cuprates, and thus also in YBCO, increases with increasing oxygen doping level. Thus one can rule out the inverse proximity effect as the responsible mechanism for the large \( T_p \) shifts. Our results therefore, are consistent with the preformed pairs scenario, but are not a definitive proof for their existence. However, our suggestion that a large effect on \( T_p \) is due to the injection of oppositely polarized correlated electrons, from the YBCO layer in the pseudogap regime into the SRO layer, is a significant step in support of the precursor superconductivity scenario in the cuprates.
opposite spins have to be correlated to produce the much larger effect, and this lends support for the preformed pairs scenario.

Acknowledgments: The authors are grateful to L. Klein and O. Millo for useful discussions, and I. Asulin for taking the STM image of Fig. 1. This research was supported in part by the Israel Science Foundation (grants # 1564/04 and 746/06), the Heinrich Hertz Minerva Center for HTSC, the Karl Stoll Chair in advanced materials, and by the Fund for the Promotion of Research at the Technion.

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