

# Effects of Phase Separation in Two Dimensional Thin Films

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Thin films with superconducting puddles and thin films with superconductor normal metal bilayer were investigated for evidence of a metallic phase as temperature approaches absolute zero.

## Introduction

Superconductors are materials that, at low temperature, are able to conduct electricity with no dissipation, or loss of energy. This, among other properties, has generated a large amount of interest in this phenomenon since its discovery in 1911. While the science of elemental superconductors such as Niobium and Tin are well understood, unconventional superconducting systems have proven to be more difficult to understand. In fact, the basic microscopic mechanics of disordered and phase separated superconducting systems remain relatively unknown. The study of such systems is more effectively conducted in two dimensions, as the effects are easier to observe and isolate.

We are interested in investigating the influence of phase separation on thin films. In particular, we expect that the variation of this parameter will lead us to an unusual metallic, or dissipative, state in these films. Although most theories only account for a superconducting or insulating state as temperature approaches zero, an anomalous metallic phase has previously been observed experimentally.<sup>1</sup>

In our experiments, we attempted to create this metallic state using thin films of normal metal with superconducting dots (see Fig. 2) and also in thin films with a metal-superconductor bilayer (see Fig. 1). We expect that by varying the dot sizes and spacing on the dot samples we could create the dissipation necessary to form the metallic phase. A similar metallic phase has been observed in other systems such as Josephson-junction arrays.

## Experimental Setup

Measurements in this experiment were taken on films of gold (Au) (normal metal) and niobium (Nb) (superconductor). We chose Nb because of its high critical temperature  $T_c$  of 9.3 K. The high  $T_c$  allowed us to test our bilayer samples in Helium-4 dewar with a standard 4K insert. We also tested the dot patterns at 4K, but used a 1K dunker to achieve colder temperatures.

Our films were fabricated on Silicon wafers with conventional photo and e-beam lithography techniques. The films were patterned into 4-probe structures, to allow for four wire measurements. Au was then evaporated onto our samples using a thermal evaporator. Our bilayer samples consisted of between 20 nm and 60 nm of Au, and a layer of Nb of about 60 nm – 90 nm in thickness.

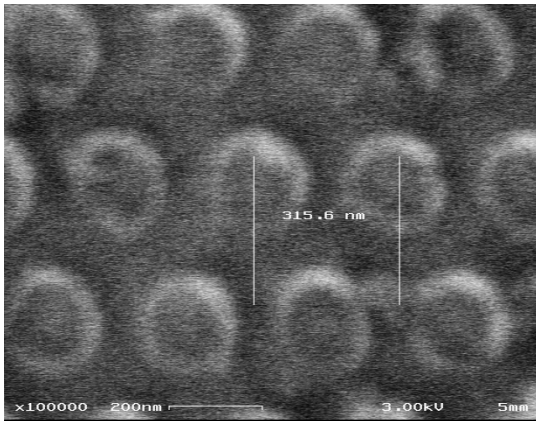
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<sup>1</sup> N. Mason, Ph.D thesis, Stanford University, 2001 (unpublished)



**Figure 1: Illustration of bilayer chips.**

Generally for our dot samples we would use 20 nm of Au and 60 nm – 90 nm of Nb. The pattern for the superconducting puddles was designed on the RAITH design program, and the dots were later patterned onto the samples with the use of the RAITH e-beam lithography system. The dots were typically spaced 200 nm - 600 nm apart with dot diameters about 140 nm. Up to six dot patterns could be patterned onto one chip, although typically we only patterned two.



**Figure 2: Scanning Electron Microscope image of 300 nm Spaced Dots. Note that the dot sizes are on the order of 140 nm.**

After the e-beam lithography, Nb was sputtered onto the samples by the Sputtering system. It was especially important to determine the sputtering parameters, as this fabrication step is very sensitive to small changes. Sputtering was used, as opposed to thermal

evaporation, because Nb is difficult to evaporate thermally, and it is generally not as homogenous. It was also important to calibrate our sputtering system to optimize  $T_c$  for thin films of Nb. This was meant to limit the amount of stress in our films, as stress depresses  $T_c$ . After sputtering, electrical contact to the samples was made via gold or aluminum wires.

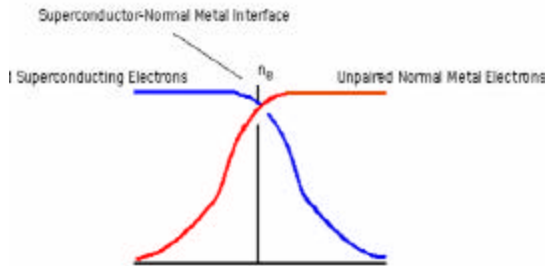
During the calibration phase of the experiment, which involved setting the ideal parameters for the fabrication process, a 4K insert was used. A current was sourced and a four wire measurement was taken by a Keithley 2400 source-measurement unit, and the signal was sent to a computer which was equipped with LabView. The temperature was measured on a Lakeshore Silicon diode thermometer. A current was sourced to the thermometer by a Keithley 220, and the resistance was read by a HP multimeter.

The dot samples were first measured in a 4K dunker and later in 1K dunker to measure to lower temperatures. The measurements were taken on a Keithley 2400 and a lock-in, and a similar LabView program was used to organize and plot the data. At low T (between 1.6 K and 10 K), a Lakeshore 331 heater/thermometer was used to control temperature variations.

## Results - Bilayers

In the first set of experiments, we wanted to verify that our fabrication methods yielded model samples, i.e., that the samples exhibited expected superconducting thin film behavior. These samples could then be used as controls for our phase-separated dot

samples. Alternatively, if these samples did not show ideal behavior, but instead showed an unusual metallic state we would know that a metal layer alone could induce dissipation in a superconductor. We anticipated that (a) adding to the thickness of superconducting layer raises  $T_c$ , and that (b) adding to the thickness of the normal metal layer lowers the  $T_c$  of the system.

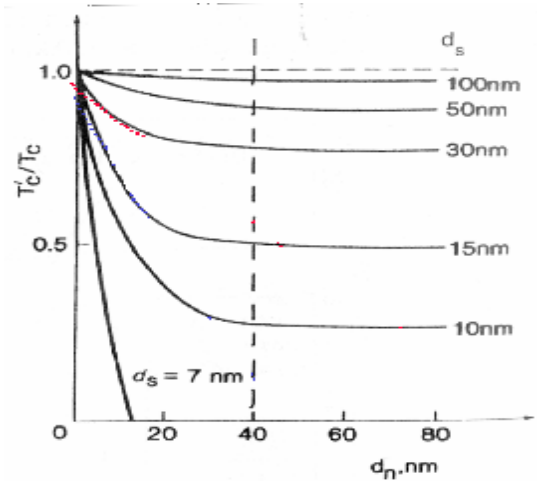


**Figure 3: Interface between superconducting and metallic domains in the intermediate state.**<sup>2</sup>

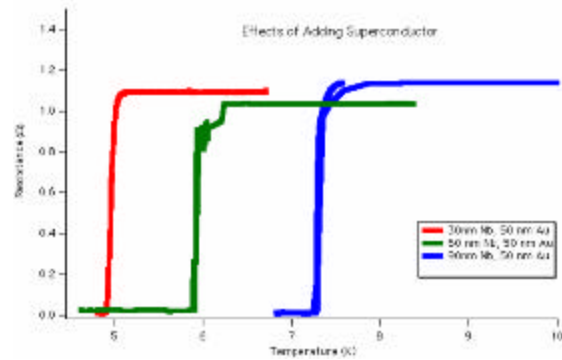
We expect our system to behave in this way because of the proximity effect. The superconducting electron pairs (Cooper pairs) penetrate into the normal metal, and the normal metal electrons penetrate into the superconducting metal. Thus, a superconducting region is created in the normal metal and a normal region is created within the superconducting metal. Fig. 3 illustrates the interface between superconducting and normal metal domains. The curves represent number of Cooper pairs (or electrons in normal metal) as a function of distance. We see that for some distance  $x$  from the interface boundary in either direction we will find both superconducting and normal metal electrons. As can be seen in Fig. 4 the  $T_c$  of such systems is then dependent upon the ratios of the

<sup>2</sup> Tinkham, *Introduction to Superconductivity*

thickness of superconductors to the thickness of normal metal.<sup>3</sup>



**Figure 4: This is a graph of expected values of  $T_c$  with respect to thickness of normal metal ( $d_n$ ). Each curve represents a different thickness of the superconducting layer ( $d_s$ ).**<sup>4</sup>



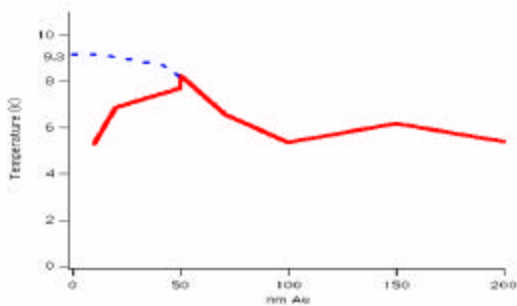
**Figure 5: These are the resistance vs. temperature curves for various samples. The red corresponds to 30 nm Nb, green 60 nm Nb, and blue 90 nm Nb. All three samples had 50 nm of Au deposited on them.**

In this part of experiment we find that the increase of superconductor thickness did lead to elevated  $T_c$ . Fig. 5 displays the results on three samples of differing thicknesses.

<sup>3</sup> But only to a certain point. If we examine Fig. 4, we notice that the addition of normal metal past a certain thickness does not affect the  $T_c$  any longer. From Fig. 2 we can see that normal electrons only penetrate somewhat into the superconductor and vice-versa.

<sup>4</sup> Adapted from N.R. Werthamer, *Phys. Rev.* **132**, 2440 (1963)

We also expect that with the addition of normal metal and a fixed amount of superconductor we would find a curve similar to Fig 4 where  $T_c$  flattens out as more normal metal is added to it, and goes toward bulk material  $T_c$  as normal metal is taken away. What we observed, however, did not hold to this pattern (see Fig. 6). We found that our  $T_c$ 's did not act as we expected when the Au layer was less than 40 nm thick.



**Figure 4:  $T_c$  vs nm Au. The red line is a plot of our actual results and the blue line suggests what we should have observed.**

It is possible that the  $T_c$  was depressed below 50 nm of Au thickness because of a lack of homogeneity in the Au layer below 40 nm. It is also possible that  $T_c$  suppression may have been caused by impurity of our Au evaporated in the first layers.

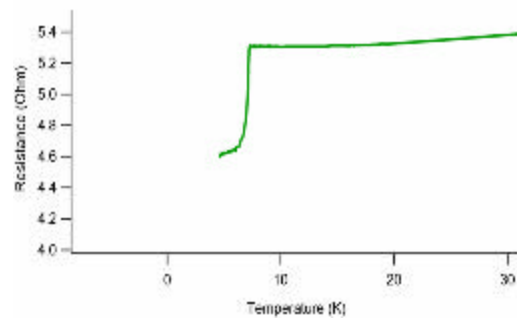
From this preliminary phase, we now know the limitations of our materials and equipment. And although the films did not behave ideally below a normal metal thickness of 40 nm, we were aware of the depressed  $T_c$ .

### Results – Dots

Thus far, we have fabricated samples with 20 nm of Au and 60 nm – 90 nm of Nb, with dot diameters  $\sim$  140 nm and spacing between 200 nm and 600 nm. We use 20 nm of Au because a thicker

layer of normal metal might suppress the superconductivity in these samples even more.

We wish to tune the parameters of this system, particularly the dot size and spacing to vary the phase separation to see a metallic phase at low T. We have just commenced our testing of these samples to 1 K, to see whether or not these samples exhibit the metallic phase that we aim to find. As can be seen in Fig 7, some of our dot samples seem to initially superconduct and then flatten out. Lower temperatures are needed to determine whether a metallic phase will appear.



**Figure 7: R vs T plot of one of our dot samples in the 4K dunker. We do not know whether this sample will superconduct or exhibit metallic characteristics at lower T.**

### Conclusion

In our experiment, we fabricated two different types of samples to attempt to find a metallic phase. We measured the  $T_c$  of our bilayer samples with different thicknesses of Au and Nb and were not able to obtain a metallic state. We measured our dot samples at 4K and did not find metallic behavior, we are now testing our samples at temperatures down to 1K.