Preparation of MgB2 superconducting microbridges by focused ion beam direct milling

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Abstract. MgB2 superconducting microbridges were prepared by focused ion beam (FIB) direct milling on MgB2 films. The surface topography of the microbridges were observed using SEM and AFM and the superconductivity was measured in this paper. Lots of cracks and holes were found near the milled area. And the superconducting transition temperature was decreased a lot and the bridges prepared were not superconducting due to ion damage after milled with large dose. Through these works, we explored the effect regular of FIB milling and experimental parameters on the performance of microbridges.

1. Introduction

The superconductivity of MgB2 material was found in 2001 and then much research has been done on it in a lot of countries due to its simple structure, high critical transition temperature (39 k), two weak coupling energy gap, the larger coherence length, strong and grain boundary connection and many other advantages.[1-5] MgB2 superconductor is expected easier to be prepared compared with high temperature superconductors and the cooling cost can be greatly reduced compared with low temperature superconductors such as Nb. Meanwhile, MgB2 has higher current density. Thus, the discovery of MgB2 superconductivity provides a new material of great potential to make superconducting electronics based on Josephson junctions.[6]

Traditional tunnel Josephson junction has the higher capacitance relatively due to geometric feature while microbridge Josephson junction has a lower one. So the electronic devices based on microbridge Josephson junction are very suitable for application in lower noise environment.[7] Therefore, to explore a simple and repeatable preparation method to fabricate submicron size devices is quite necessary.[8] In our work, a new method is explored on this problem. MgB2 microbridges were fabricated by focused ion beam direct milling on MgB2 film. We observed the morphology characteristics of MgB2 films and microbridges using SEM and AFM. The superconductivity of MgB2 films and microbridges was measured by PPMS. We explored the influence regular rule of the new method on MgB2 microbridge and film which has a great guiding significance for the fabrication of Josephson junctions and superconducting electronics.[9]
2. Experimental

2.1. The preparation of MgB$_2$ Film

The MgB$_2$ film used in this paper is made by HPCVD method on SiC substrate in Beijing university. The thickness of the film is about 100 nm. The superconducting transition temperature of MgB$_2$ film fabricated by HPCVD is up to 42 k and the zero resistance transition temperature is about 41 k. The superconducting transition width is less than 1 k. The quality of MgB$_2$ film prepared by HPCVD is greater than others. So it’s suitable to choose MgB$_2$ film made by HPCVD.

2.2. The Fabrication of MgB$_2$ microbridges

Focused ion beam milling progress is finished on the FIB/SEM dual beam 820 system. 30 KeV Ga$^+$ ion beam was used with different beam currents from 12 pA ~ 6300 pA in the process of the fabrication of MgB$_2$ microbridges. First of all the ion beam with 6300pA beam current was focused on the edge of the surface, and then the screen was freezed. Next we designed and prepare the rectangle frame with 300×800um$^2$ and the trench as shown in figure 1(a). Two exactly the same microbridges were made by this method. The image of one of the microbridges is shown in figure 1(b) with its partial enlarged image shown in the insert. The size of the bridge is about 7×10um$^2$. The other one was then milled to the size of 3×3um$^2$ with 12pA. The whole process will continue for several hours. In this method we prepared a series of microbridges which can be accurate to the nanometer level.

![Figure 1. SEM figures of MgB$_2$ microbridge by focused ion beam milling without scanning.](image)

What’s more, we fabricated only the outside frame without any trench in the middle area by direct milling with the same experimental parameters. One of them was scanned quickly for several times with the current of 2700pA. We observed the surface morphology of films and micro bridges and explore the effect of focused ion beam milling directly on the superconductivity of the films and microbridges by comprehensive physical property measurement system (PPMS).

3. Results and Discussion

The superconductivity of the film and microbridges was measured by (PPMS). The R-T curves are shown in figure 2. The inset shows the detail of the superconducting transition temperature of MgB$_2$ film on MgO substrate. It is obvious that the microbridges are not superconducting. This maybe due to the damage of MgB$_2$ film in the bridge in ion beam milling process. When MgB$_2$ film is milled with 6300pA in three hours or longer time, thermal effect is probably produced and the bridge area is affected seriously from it and lost the superconductivity.
**Figure 2.** R-T curves of MgB$_2$ microbridges and film on MgO substrate. Bridge 1 is 1.5 um long and wide. Bridge 2 is 3 um long and wide.

**Figure 3.** Spectrum image. analysis of micro bridge in scan mode

**Figure 4.** SEM figures of MgB$_2$ microbridge after measuring the superconductivity.
Figure 3 and table 1 shows the energy spectrum of the microbridge. It can be seen that content of O and Ga in the bridge is higher than other areas. This may be due to the decomposition of MgB$_2$ caused by thermal effect and ion beam bombarding when milling with large beam current for a long time. The SEM images of the edge of the milled frame and the area of middle bridge are shown in figure 4(a) and (b) respectively. We can see that the density of MgB$_2$ film becomes poorer. This illustrates that the film is damaged in milling process and thus confirms our aforementioned guess.

| Area | 1   | 2   | 3   | 4   | 5   |
|------|-----|-----|-----|-----|-----|
| O (Atomic %) | 1.41 | 1.36 | --- | --- | 0.79 |
| Mg (Atomic %) | 1.98 | 2.64 | 5.26 | --- | 2.48 |
| Ga (Atomic %) | 0.96 | 0.83 | --- | 1.33 | --- |

In order to make a compare, two rectangle frames were made only using the same experiment parameters. the width and depth of the frame were about 7um respectively and one frame was scanned with 2700pA ion beam for several times. The superconductivity was measured by PPMS as shown in figure 5. The MgB$_2$ film outside the frames has a starting transition temperature of 40.6 K with the zero resistance transition temperature of 40.2 K and the superconducting transition width is about 1 K. The starting transition temperature of MgB$_2$ film enclosed with frames is about 34 K and the zero resistance transition temperature is 25.8 K. The superconducting transition width is 8.2 K. The superconducting transition temperature is reduced by about 15 k. But the trend of the R-T curves of the two frames is nearly the same. It indicates that ion scanning for several times has little effect on the zero resistance transition temperature of MgB$_2$ film. But milling with large beam and long time indeed has a big effect on the superconductivity on the film.

**Figure 5.** R-T curves of MgB$_2$ films outside and enclosed the frames on SiC substrate. The area of frame 2 is scanned for several times by 2700pA ion beam.
The SEM images near and far from the milled area are shown in figure 6(a) and (b). It's obvious that there are a lot of holes occurred on the film and it is more serious as near the trench. From the images, we can see the affected area is in the range of about 6 um from the milled area edge as shown in figure 6(c), and the hole’s diameter is about 30 nm as shown in figure 6(d). This dedicates that the bridge area is completely damaged and is not superconducting.

In order to explain this phenomenon theoretically, the simulation was also carried out by using SRIM software. The penetration process and distribution of Ga+ ion into MgB2 film were simulated using 200, 1000, 5000, 25000 ions respectively as shown in figure 7. We can see that the penetration depth in MgB2 film is about 20-40nm and the ion concentration peak is appeared in the depth of 30nm with 30KeV ions. Only a few number of Ga+ can penetrate into the depth of the 50nm. The horizontal injection radius is about 15nm. In consideration of the actual size of ion beam spot, the diffusion radius should be much larger than 15nm. According to the simulation analysis, the film especially the middle bridge area is seriously damaged and form into cracks and holes due to heat effect under the condition of large ion beam current and as long as several hours. Therefore, the affected region can be as long as several microns from the milled area edge when MgB2 film is milled with depth and width of 7um using 6300pA beam current. It further illustrates the problem why the micro bridges prepared were not superconducting.
4. Conclusion

In all, MgB$_2$ microbridges were prepared by focused ion beam direct milling on MgB$_2$ film. It is found that the film near the milled area is seriously damaged and cracks and holes are produced after the film is milled with large beam current and long time. The superconducting transition temperature of MgB$_2$ film can be greatly reduced and the micro bridge is not superconducting due to the effect. Thus, although the new method to prepare micro bridges can avoid the disadvantage of traditional lithography, such as contacted with water vapor, there is still a big problem about ion damage. How to reduce the disadvantages of focused ion beam direct milling on MgB$_2$ film and microbridges will be our next focus. Some measures, such as changing the experiment parameters, to improve the quality of the film and microbridges, should be taken into. Our research provides an important guiding
significance and innovative idea for using focused ion beam milling technology to the prepare superconducting Josephson junction and device.

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