Ultrathin MgB_2 Films for Hot Electron Bolometer Mixers Fabricated by HPCVD and Ion Milling

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Abstract— **Hot electron bolometer (HEB) mixers are frequently being used for high resolution spectroscopy at terahertz frequencies. Currently employed mixers are based on superconducting materials such as Nb and NbN. Magnesium diboride (MgB_2) with its higher critical temperature is a promising candidate that could significantly improve HEB performance in comparison to conventional superconductors. This study focuses on ultrathin MgB_2 films deposited by the hybrid physical chemical vapour deposition (HPCVD) technique for the employment in HEB mixers. Due to the Volmer-Weber growth mode encountered in the HPCVD process, films thinner than 10 nm are difficult to achieve as a result of incomplete film coverage. Therefore, ion milling of thicker MgB_2 films down to the desired thickness was explored as part of this study as well, resulting in uniform films of less than 10 nm. All MgB_2 films showed T_c higher than 36 K with residual resistivities below 26 μΩcm, where the milled films showed consistently higher T_c and lower resistivity in comparison to the regularly grown films. The thinnest films achieved in this fashion display critical currents of $5x10^6$ A/cm^2 at 15 K. The ion milling process also leads to an improved surface morphology with a roughness of less than 2 nm. The results show that ultrathin films fabricated by the HPCVD process and thinned down by ion milling exhibit improved qualities than films directly grown using the HPCVD process alone.**

INTRODUCTION

Hot electron bolometer mixers represent an important tool for terahertz astronomy and currently employed detectors contain NbN based devices [1]. Due to the critical temperature T_c of NbN being nearly 10 K [2], such mixers, however, need to be operated at temperatures close to 4.2 K. Furthermore, NbN based mixers suffer from a relatively low intermediate frequency (IF) bandwidth. The IF bandwidth is one of the key characteristics of HEB mixers and is in part determined by the electron relaxation time. The relaxation of hot electrons created in an HEB mixer during operation relies on the transfer of its energy to a phonon, which then escapes from the superconducting film into the substrate. As a result, the relaxation process highly depends on the electron-phonon interaction time [3]. NbN exhibits an electron-phonon relaxation time of about 12 ps at 10 K, leading to an IF gain bandwidth of up to 3 GHz [4, 5]. IF bandwidths of up to 6.5 GHz have been recently realized for NbN HEBs by further improving the mixer fabrication and design [6, 7]. As terahertz radiation in increasingly broader frequency ranges, such as Doppler broadened lines in interstellar molecular clouds, becomes the focus of current research, HEB mixers with IF bandwidths of more than 10 GHz are required. This necessitates the investigation of other materials than NbN and examining their feasibility for the employment in HEB mixers.

MgB_2, which exhibits T_c of up to 39 K [8], offers a viable substitute for NbN. As the estimated electron-phonon interaction time is just a few picoseconds [9], IF bandwidths beyond those of NbN HEB mixers could be achieved. While MgB_2 thin films have already been explored for HEB use in the past [10], the quality of the employed films led to devices with low T_c. Nevertheless, by achieving a mixer gain bandwidth of 3.4 GHz with a noise performance comparable to that of NbN HEB mixers, it was shown that with further improvements, MgB_2 could potentially rival NbN and other conventional superconductors.

The hybrid physical chemical vapour deposition process results in ultrathin MgB_2 films of significantly higher quality compared to any other method [11-14]. Not only does this process lead to films of high purity, but the intrinsic strain encountered in these films due to lattice mismatch between the film and substrate can lead to critical temperatures above bulk value. As the Volmer-Weber mode governs the growth of these films, however, thicknesses of less than 10 nm are difficult to achieve due to incomplete film coverage. In this work, we compare MgB_2 films grown by the regular HPCVD method with films that have been fabricated by thinning a subsequently thick HPCVD MgB_2 film down to the desired thickness using an Ar ion milling process. With this approach, uniform films of well below 10 nm have been achieved with relatively high critical temperatures and low residual resistivities.

EXPERIMENTAL

In the HPCVD process vapors of Mg and B, which are supplied by physical vapor deposition of Mg and chemical vapor deposition of B, react at high temperatures in a clean environment, resulting in the deposition of high quality

MgB_2 thin films [15, 16]. Thermodynamic stability of MgB_2 is crucial in this process which necessitates a high Mg vapor pressure. The high vapor pressure is achieved with evaporation of solid Mg pieces and formation of MgB_2 is initiated through the introduction of B_2H_6 (diluted to 5% in H_2) gas and its subsequent decomposition at high temperature. Oxygen contamination is omitted as the process takes place in a H_2 atmosphere. The employed HPCVD system has been described in detail by Zeng *et al.* [17] in the past.

An HPCVD MgB_2 thin film deposition requires four pieces of clean Mg which are placed on the edge of a stainless steel susceptor while 15x15 mm^2 substrates are placed at its center. The system is subsequently pumped down and flushed with hydrogen gas to remove any contaminants. After flushing, the chamber pressure is set to 40 Torr and the susceptor temperature is slowly raised. Once the Mg pieces begin to melt, the susceptor is kept at a stable temperature during deposition. The deposition of MgB_2 begins with the introduction of the B_2H_6 gas mixture into the chamber and ends once the B_2H_6 flow is shut down.

As the MgB_2 deposition rate depends on the B_2H_6 flow rate, ultrathin films have to be grown in a relatively low gas flow in order to provide accurate thickness control. The ultrathin films have therefore been grown at flow rates of 2 sccm and deposition times between 30 sec and 1 min. During regular HPCVD MgB_2 growth, the deposition temperature is fixed at around 15°C above the melting point of the Mg pieces. In the case of the ultrathin films, the susceptor temperature was kept just above the melting temperature, to keep particle diffusion and island formation to a minimum and achieve more uniform coverage. With this HPCVD process, ultrathin films of down to 10 nm were obtained. In order to acquire even thinner films, thicker MgB_2 films of 40 nm have been grown, which were subsequently milled down to the desired thickness using an IntlVAC Ar ion milling system. The films were milled at an angle of 45°, resulting in homogenous ultrathin films with a low surface roughness.

MgB_2 can successfully be grown on a variety of substrates [18]. In order to achieve high quality *c*-axis oriented epitaxial films, the choice of substrates is limited to single crystal (0001) Al_2O_3, (111) MgO, and (0001) SiC. Out of these options, (0001) SiC remains the most favorable substrate for HPCVD MgB_2 films. Since SiC offers the highest intrinsic stress amongst the three listed substrates, it results in the highest achievable T_c. It has been shown recently that SiC also exhibits THz transmission up to 2.5 THz [19], which is beneficial for HEB mixers. Semi-insulating SiC substrates have therefore been used for this study.

The transport properties of the fabricated MgB_2 films were characterized by four-point measurements according to van der Pauw [20]. Surface analysis was done using a FEI Quanta 450FEG scanning electron microscope (SEM). Thickness measurements were carried out with a Dektak profilometer and a Veeco atomic force microscope (AFM).

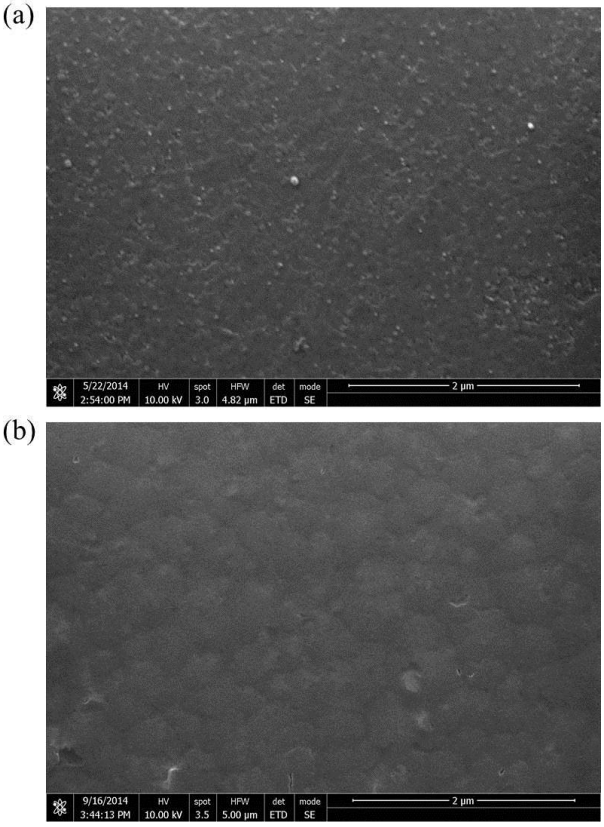

Fig. 1: A 10 nm thick MgB_2 film (a) grown under regular deposition conditions and (b) thinned down from a 100 nm film using ion milling.

RESULTS AND DISCUSSION

The SEM images of a regularly grown 10 nm MgB_2 film and an ion milled 10 nm film are shown in Fig.1. It is clearly visible that a directly grown film exhibits a higher granularity while a film obtained through ion milling of a much thicker MgB_2 film shows a more uniform coverage with larger, interconnected islands. AFM analysis of the grown 10 nm film showed a root-mean-squared roughness R_q of 3-4 nm, while the ion milled film results in a much smoother surface with an R_q of below 2 nm. The surface morphology of the ion milled films could be more beneficial for the use in HEB mixers as it might allow for a more even phonon cooling due to a uniform thickness and less grain boundaries being present. It has been recently shown that the regularly grown films still perform well when employed in a HEB mixer [19, 21] and it was possible to achieve IF bandwidths of more than 8 GHz at 25K. These films, however, display vortex pinning at increased magnetic

fields,which is most likely a result of the defects due to the highly granular structure of the film [22]. It is unclear at this point how the defects affect the HEB mixer performance, yet it is likely that the vortex pinning could be omitted in ion milled films due to their larger grain sizes.

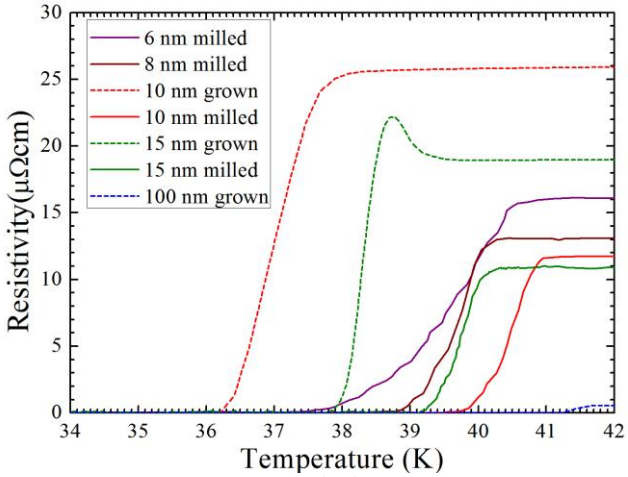

Figure 2: Resistivity vs. temperature for films directly grown with HPCVD and films fabricated by ion milling a 40 nm film down to the indicated thickness.

Fig. 2 shows a resistivity vs. temperature curve of various MgB_2 films either grown directly or milled from a thicker film. A drop in T_c can be observed for all films with decreasing thickness, independent from the fact if they were directly grown or ion milled. It is evident that the critical temperature of ion milled films is not as much affected by the film thickness as it is for directly grown films. The critical temperatures of all films are nonetheless well above 36 K and comparable to bulk value. The achieved critical temperatures surpass results obtained by other deposition methods in the past [23, 24] and theoretical studies showed recently that ultrathin MgB_2 films could still exhibit superconductivity down to thicknesses as low as approximately 1.5 nm [25].

The residual resistivity increases as well for both fabrication processes with decreasing film thickness. The increase in residual resistivity is more prominent for the directly grown films, which is expected to be a result of a disorder-induced suppression of the superconductivity due to the presence of pinholes and a predominantly granular structure. A similar outcome has been reported elsewhere [22, 13]. The change in residual resistivity is partly also a result of increased scattering of electrons off the film surface and substrate interface since the film thickness of the ultrathin films restricts the mean free path of the electrons. The ion milled films show a less pronounced increase in resistivity which further shows that film resistivity is more affected by the granularity of the films rather than its thickness.

While the surface structure and residual resistivity clearly show that the ion milled ultrathin films possess improved qualities than films directly deposited by HPCVD, it is possible that the ion milled films could be damaged to a certain point due to being exposed to highly energetic ions during the milling process. If this is the case, the current results do not present evidence that the film damage has a larger impact on the film quality than the granularity encountered in the directly grown films.

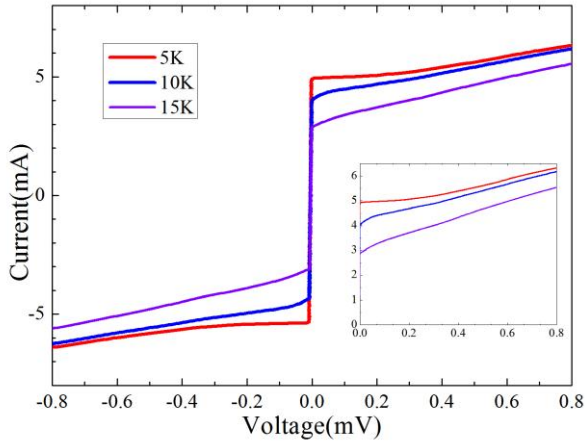

Fig. 3: I-V curves of an ion milled 6 nm film measured on a 300 μm x 10 μm micro strip.

Fig. 3 shows the I-V curves obtained for an ion milled 6 nm MgB_2 film. The I-V characteristics were measured on a 300 μm x 10 μm large micro strip. From the I-V measurements, a critical current density J_c of above $5x10^6$ A/cm^2 at 15 K was calculated. The result shows that ion milled HPCVD MgB_2 films not only exhibit high T_c, but also relatively high J_c. The improved characteristics can most likely be attributed to better connectivity and therefore an increase in the effective cross-sectional area. The obtained J_c values are encouraging as they reveal the capability to apply high local oscillator power to a MgB_2 based HEB mixer.

CONCLUSIONS AND FUTURE WORK

In summary, we have fabricated high quality superconducting ultrathin HPCVD MgB_2 films followed by thinning them down with an Ar ion milling process. In comparison to ultrathin films grown using the HPCVD process alone, the ion milled films exhibit higher critical temperatures and lower residual resistivities. The established process not only offers a way to achieve ultrathin MgB_2 films of extremely low thickness for HEB mixers, possibly leading to a higher IF bandwidth, but can also potentially be used for other applications based on thin superconductors, such as superconducting single photon detectors (SSPDs).

While film thicknesses of down to 6 nm have been achieved with this method so far, it should be possible to achieve even thinner films. Our future work involves the study of ion milled MgB_2 films below 6 nm and their superconducting properties.

Although these ion milled films have not been employed in HEB mixers yet, it is expected that they could surpass the performance of regularly grown films due to their improved characteristics in comparison to ultrathin MgB_2 films directly grown with HPCVD.

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