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Nb/Compound Semiconductor Heterostructures Epitaxially Grown by Interrupted Electron Beam Deposition

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Niobium heteroepitaxy onto (111) oriented InSb, (100) oriented GaAs, Al_{0.2}Ga_{0.8}As, and InP substrates was successfully achieved. Novel techniques² were conducted to realize the epitaxy: an interrupted electron beam deposition and a in-situ surface cleaning by hydrogen plasma. Both techniques brought about really effective processes in obtaining the epitaxial films. RHEED and x-ray diffraction measurements revealed that the epitaxial Nb films had (100) orientation regardless of the substrate choice, and clean interfaces were obtained by hydrogen plasma treatment at room temperature.

1. Introduction

Metal-semiconductor heterostructure has become of the prime technological importance in view of a number of future device applications. This structure enables us to fabricate ultra high speed devices such as metal-base hot-electron transistor^{1,2)}. Besides, a most attractive research target of the structure is a fabrication of a multiple - layered metal - semiconductor structure. It would lead new quantum effects and novel applications as the semiconductor heterostructure could. one of the expected quantum As effects, we have proposed a high Tc two dimensional excitonic superconductor which would have the Tc higher than 200K³⁾. To realize the quantum effect as well as the high speed devices, heteroepitaxial growth technique of metals on semiconductors must be established.

In recent papers, $^{4,5)}$ the metal epitaxy on GaAs has been reported. However, they required the molecular-beam epitaxy (MBE) of GaAs and the successive in-situ metal deposition in the UHV system. Furthermore, there is no report on the metal epitaxy on other compound semiconductors.

In the present work, we report the niobium heteroepitaxial growth onto several compound semiconductors. Novel techniques were conducted to achieve the direct epitaxy on the substrate wafers: an interrupted electron beam (EB) deposition and a room temperature hydrogen plasma cleaning of the semiconductor surfaces⁶⁾. Nb was chosen as an attractive metal candidate: refractory metal and superconductor. (111) oriented InSb, (100) oriented GaAs, AlGaAs, and InP wafers were used, which are promising materials⁷⁾ for the device applications and 2D excitonic superconductor.

2. Experimental Procedures

The Nb films were prepared in an electron beam (EB) deposition system. A residual gas pressure during the deposition was kept to be lower than 3.0×10^{-7} Torr. The deposition rate ranged from 0.3 to 1.0 Å/sec. At this growth step, the interrupted EB deposition method was introduced where the growth proceeds by the deposition of Nb for 30 sec and close of the shutter for 30-60 sec. and

this cycle repeated as many times as required. This method was really effective to avoid the radiation heating of the substrate surface and to give the sufficient migration time to Nb atoms arrived at the substrate. Mirror polished (111) oriented InSb ,(100) oriented GaAs, and InP wafers, and (100) oriented Al_{0.2}Ga_{0.8}As substrate prepared by MOCVD technique were used. All of them were etched in 20%HF solution for 2 minutes, and dried by N_2 . rinsed by water, The substrate wafers were set on the sample holder in the vacuum system. Prior to the deposition, H_2 plasma cleaning was done with the pressure of 0.06 Torr and the power of 80W at room temperature. The characterization of the prepared films was done by reflection high energy electron diffraction (RHEED) and X-ray diffraction measurements.

- 3. Results and Discussion
- 3.1 Nb heteroepitaxy

Nb heteroepitaxial growth was achieved on all of the substrates used in this study. Figure 1 (a) and (b) shows the RHEED patterns of the 40Å thick Nb film prepared on (111) oriented InSb. The film was deposited at the substrate temperature T_g of 200C after the 30 minutes H₂ plasma treatment. The pattern revealed that the epitaxial Nb film had (100) orientation: patterns (a) and (b), respectively, correspond to the ones from [120] and [110] incident directions of the (100)oriented Nb. The orientation was confirmed by x-ray diffraction measurement. Figure 2 shows the x-ray diffraction pattern of the about 60nm thick epitaxial Nb film. The peak observed at about 29=55° corresponds to the (200) texture of the (100) oriented Nb.

In order to determine the orientation relationship, thinner Nb film (-1.5nm) was deposited on the InSb substrate. Figure 3 gives the RHEED pattern from (100) Nb along [010] azimuth. The streaks correspond to the





Fig.1 RHEED patterns from (100) oriented Nb along (a) [120] and (b) [110].



Fig.2 X-ray diffraction pattern of Nb films prepared on (111) InSb.

pattern from (111) InSb along [112] azimuth. Furthermore, Nb[110]//InSb[413] relation was observed. As a result, the epitaxial relation was found to be Nb(100)//InSb(111) and Nb[100]//InSb[110] as drawn in Fig.4(a). This is an amazing result, because the epitaxial relation of Nb(111)//InSb(111) had been



Fig.3 RHEED pattern from (100) Nb and (111) InSb along [010] and [112], respectively.

expected to obtain in view of lattice mismatching and no misfit as given in Fig.4(b). In other word, the orientation relation of the metal on semiconductor was not simply determined by the surface strain energy⁸⁾. A core energy and/or pseudomorphic growth are the candidates for the explanation. One thing we should note here is the appearance of streak patterns of the substrate. Though the cleaned surface has stepped one, explained by the spot RHEED patterns(cf.Fig.6), only 1.5nm thick Nb growth brought about the InSb flat surface. It suggests that the atoms of the substrate at. near the surface were rearranged. Therefore, the pseudomorphic growth at the might interface happen. The minute observation during the film growth is now under way.

The (100) oriented Nb heteroepitaxy was achieved on (100) oriented GaAs, InP, and $Al_{0.2}Ga_{0.8}As$ wafers. We have not observed the epitaxial relations. But the growth with the relation of Nb(100) // GaAs(100) and Nb[100] // GaAs[110], so-called the Baker-Nutting relation, have been reported⁵⁾. Therefore, we could infer that the epitaxy on InP and AlGaAs has the same orientation relations.

3.2 H₂ plasma cleaning process

Surface cleaning is one of the most important factors to achieve the epitaxy. In



Fig.4 Lattice overlays of (a) Nb(100) on InSb
(111) and (b) Nb(111) on InSb(111).



Fig.5 Epitaxial grade as a function of the cleaning time. The plasma cleaning proceeded for the substrates kept at the room temperature.

this work, the cleaning process was done by H_2 plasma treatment at room temperature. Figure 5 shows the epitaxial film grade as a function of the cleaning time. Nb film were prepared on (100) oriented GaAs at T_S of 240-260°C. The film grade was determined by the (200) intensity fraction of both (200) and (110) intensities obtained by x-ray diffraction (non-epitaxial Nb has (110) natural orientation)³⁾. A glance at this figure







Fig.6 RHEED patterns from (100) GaAs surface along the [110] azimuth. (a) as-polished surface, (b) 20 min cleaned surface, and (c) 40 min cleaned surface.

revealed that the optimized process brought about the near perfect epitaxy. The epitaxial film grade is thought to serve the diagnosis of the surface cleanliness. Therefore, the 60 minutes plasma process would offer the quite clean surface of the GaAs wafer. The process for less and more than 60 minutes may lead to the surface cleaned insufficiently and damaged by the excess process, respectively.

The direct evidences were obtained by RHEED pattern. Figure 6 (a)- (c) shows the RHEED pattern dependency of the (100)surface on the $H_{
m o}$ plasma oriented GaAs cleaning time. The pattern indicated that the as-polished wafer had the step and oxidized surface, which can be explained by the net and halo patterns, respectively. The 20 minutes cleaning brought about the disappearance of the halo pattern and the clearer spot pattern. That is to say, the surface was cleaned by the room-temperature hydrogen plasma treatment. The 40 minutes cleaning, however, gave only a few spots and some ring patterns, which suggested that the unfavorable degradation began to develop. The tendency of the surface cleaning coincides with the results of the Nb epitaxial grade. Since this RHEED measurements were done in another chamber, the cleaning conditions were different from ones in Fig.5

4. Conclusion

Nb heteroepitaxial growth has been successfully achieved on (111) oriented InSb, (100) oriented GaAs, Al_{0.2}Ga_{0.8}As, and InP substrates. RHEED and x-ray diffraction measurements revealed that Nb epitaxial films have (100) orientation regardless of the substrate choice. Hydrogen plasma treatment of the substrates at room temperature brought about the clean surfaces of the substrates.

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