

Growth and characterization of highly oriented diamond films on silicon

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ABSTRACT

Highly oriented, (100) textured diamond (HOD) films have been grown on single crystal Si substrates via microwave plasma enhanced chemical vapor deposition. A multi-step deposition process including bias-enhanced nucleation (BEN) and textured growth was used to obtain smooth films consisting of epitaxial grains with only low angle grain boundaries. Boron-doped layers were selectively deposited onto the surface of these oriented films and temperature dependent Hall effect measurements indicated a 3 to 5 times improvement in hole mobility over polycrystalline films grown under similar conditions. Room temperature hole mobilities between 135 and 278 cm²/V-s were measured for the highly oriented samples as compared to 2 to 50 cm²/V-s for typical polycrystalline films. Grain size effects and a comparison between the transport properties of polycrystalline, highly oriented and homoepitaxial films will be discussed.

1. INTRODUCTION

Significant progress has been made in the past several years towards the growth of single crystal diamond films on non-diamond substrates via low pressure thermal and plasma assisted chemical vapor deposition (CVD). Heteroepitaxy has been observed on Ni^{1, 2, 3}, c-BN^{4, 5}, SiC^{6, 7, 8} and most recently Si^{9, 10} single crystals. The epitaxial nucleation of diamond on silicon has been shown to occur via the formation of an interfacial β -SiC conversion layer thus making diamond/SiC/Si the actual epitaxial system. The advantage of this system is that one can utilize the less expensive and more readily available Si substrates thus making the development process more economical.

In addition to the growth of discrete epitaxial nuclei recent progress has led to the deposition of complete and highly oriented films on c-BN⁴, SiC¹¹ and Si^{10, 12, 13, 14} substrates. The epitaxial films grown on c-BN single crystals were highly oriented although the size and quality of the substrate material has limited the exploitation and further development of this epitaxial system. The initial heteroepitaxial nucleation and subsequent growth of highly oriented films on SiC was encouraging in that it provided an alternative heteroepitaxial substrate to c-BN, however

availability of large area, high quality substrates has hindered development. Most recently, researchers in this laboratory and others have obtained highly oriented, (100) textured diamond (HOD) films on (100) silicon single crystals over areas in excess of 1.5 in diameter. Initial Hall effect measurements performed on B-doped epitaxial layers selectively deposited on the surface of these highly oriented films resulted in room temperature mobilities in excess of 165 cm²/V-s; a factor of 5 higher than typical polycrystalline boron doped films.^{13, 15}

These results on Si represent a dramatic improvement over preexisting electronic transport properties of polycrystalline films and support the feasibility of fabricating active electronic devices from diamond films grown on non-diamond substrates. This paper will focus on the growth and characterization of the HOD films on Si. The films were characterized via scanning electron microscopy (SEM), Hall effect measurements, polar x-ray diffraction (XRD) and polarized Raman spectroscopy. Polar XRD is an effective technique for determining the texture, orientation and crystallographic quality of crystalline films^{11, 16, 17}. Polarized Raman spectroscopy is not as popular, however it can provide useful crystallographic information and is often more accessible to those researching diamond thin films.^{4, 18}

19, 20 A comparison between the two analytical techniques will be presented as well as correlations between the electronic and crystallographic properties of highly oriented, (100) textured diamond films on Si. A better understanding and control of the electronic properties of these films will ultimately lead to the fabrication of superior active electronic devices.

2. EXPERIMENTAL

2.1. Oriented Diamond Growth

Previous work from one of the authors has shown that diamond may be nucleated on pristine (unscratched) silicon wafers via a bias-enhanced nucleation (BEN) process.^{21, 22} This BEN process allowed nucleation densities in excess of $10^9/\text{cm}^2$ to be obtained *in situ* just prior to diamond deposition and without the need for conventional nucleation pretreatments. An in depth analytical study of the BEN process on Si indicated that diamond nucleation occurred on the surface of a carbon-rich, silicon-carbide interfacial layer that formed during the BEN pretreatment process.²² Subsequently it was shown that heteroepitaxy was achieved if BEN was performed on single crystal β -SiC substrates.^{6, 7, 8} As mentioned above, this work led to the epitaxial nucleation of diamond on (100) Si via the formation of a SiC interlayer.⁹ By converting the surface of the silicon to a thin epitaxial SiC layer using microwave plasma CVD, crystallographic registry was maintained across the interface allowing diamond epitaxy to occur.

The BEN and growth processes used in this study were performed in a stainless steel microwave plasma CVD reactor that has been modified to allow for *in situ* substrate biasing. A schematic of the system and a more detailed description of the BEN process has been reported elsewhere.^{12, 22} The plasma conditions for the BEN step are similar to those typically used for diamond growth; 1-5% methane in hydrogen at 15-25 torr, 1kW of power and a substrate temperature of 600-800 °C.

In the work presented by Wolter et al.⁹ it was shown that 30-50% of the grains were oriented relative to the (100) silicon substrate following the BEN step. To become useful as a substrate on which to fabricate diamond electronic devices, close to 100% of the grains should be epitaxially oriented. It was therefore necessary to either achieve 100% epitaxy or start with a partially oriented film and grow under conditions which promote (100) texturing. The latter approach was demonstrated by both Clausing et al.²³ and Wild et

al.^{17, 24} as a means to obtain highly textured diamond films from randomly oriented seed crystals. This approach utilizes the van der Drift theory of crystal growth evolution where the resulting texture is a function of the growth competition between different crystal orientations.²⁵ For an initial distribution of randomly oriented diamond nuclei, the resulting film-texture will be a function of the relative growth rate parameter (α) as described by Wild et al.;^{17, 24}

$$\alpha = \sqrt{3} \cdot V_{100} / V_{111};$$

where V_{100} and V_{111} represent the relative growth rates of the <100> and <111> directions respectively. For $3.0 < \alpha < 2.6$ the resulting textured will be strongly (100).

The HOD samples grown in this study utilized the following multi-step deposition process on (100) Si substrates. This process has been discussed in detail elsewhere and consists of; (i) a BEN step used to epitaxially nucleate diamond on the silicon substrate and (ii) a textured growth process utilized to grow out only the grains which are initially epitaxially oriented following the BEN step. Four samples were prepared for this study and grown out to thicknesses of 1, 4, 20 and 30 μm . SEM, Polar-XRD and polarized Raman were used to observe the texture and orientational development as a function of thickness and are described in more detail below.

2.2. Polar X-ray Diffraction

X-ray diffraction polar measurements were performed on the five samples of increasing thickness mentioned above so as to observe the development of texture and orientation during the (100) van der Drift grow out step. This study is similar to that performed by Kohl et al. where X-ray pole figures were obtained for highly oriented diamond films grown under both (100) and (110) texturing conditions¹¹. They demonstrated that both orientation and texture would either improve or degrade for films grown under (100) or (110) texturing conditions respectively. The XRD analysis was conducted on a Philips MRD 1880/HR with a 3 kW Cu radiation source. Texture mapping was facilitated via Philips "TEX" software. Incident radiation passed through a pinhole aperture while the diffracted radiation passed through a set of collimating slits and was reflected off a graphite plate prior to entering the detector.

2.3. Polarized Raman Spectroscopy

Polarized Raman spectroscopy was then used to determine the epitaxial quality of the highly oriented, (100) textured diamond films as a function of thickness. This technique has been shown previously to be a useful technique for determining the crystallographic orientation of single crystal films¹⁹. More recently it was used by Yoshikawa et al. to determine the epitaxial relationship of diamond films grown on cubic boron-nitride^{4, 20}. For a (100) oriented substrate the Raman intensity;

$$I \propto \sin^2(2\theta).$$

A polycrystalline film though should be invariant to polarization angle (θ);

$$I = \text{constant.}$$

Polar responses for both diamond film and silicon substrate were measured for each of the 4 films mentioned above.

2.4. Hall Effect Characterization

Hall effect measurements were performed to correlate the electronic transport with the crystallographic properties. The 30 μm HOD film was utilized for the electrical characterization study. A 4 μm thick B-doped layer was grown on the surface of the undoped film using B_2H_6 and CH_4 in H_2 at a B/C ratio of 44 ppm in the gas phase. The doped layer was selectively deposited using sputtered SiO_2 to mask the edges of the substrate so that the p-type layer would be isolated in the center of the samples to avoid current leakage to the Si. The SiO_2 was removed in HF following growth, and the films were subsequently annealed at 600 °C for 30 min. under vacuum at 10^{-7} torr in order to dissociate hydrogen from the film surface. After annealing, the sample was boiled in a solution of $\text{CrO}_3 + \text{H}_2\text{SO}_4$ at 200 °C to remove graphite which may have formed during growth or annealing. Finally, the film was cleaned in Aqua Regia and RCA solutions to prepare the surface prior to deposition of the electrical contacts. A van der Pauw configuration was arranged using Ti/Au contacts of ~500 μm in diameter. Hall effect measurements were performed between 80 and 600 K.

3. RESULTS AND DISCUSSION

3.1. Oriented Diamond Growth

The SEMs of the 1, 4, and 20 μm thick HOD samples are shown in Figures 1(a)-(c). Also displayed in this figure are the corresponding XRD and polar Raman data which will be discussed in the following sections. This thickness series displays clearly the van der Drift texture evolution. At 1 μm approximately 60% of the grains on the surface are epitaxial with respect to the substrate. After 4 μm the percentage of epitaxial grains has increased to 85% and at 20 μm the epitaxial grains have coalesced into larger (100) faces, forming only low-angle grain boundaries of several degrees. Low-angle grain boundaries develop due to a high density of misfit dislocations at the diamond/SiC interface, creating azimuthal and transversal misorientations in the individual diamond grains.^{7, 8}

Although far superior to polycrystalline films, the low angle grain boundaries limit the potential electronic transport properties of boron-dope films grown on the surface of these highly oriented intrinsic layers. Previous work has reported that highly oriented (100) films have room temperature mobilities an order of magnitude higher than polycrystalline films and 1/3 lower than similarly doped films grown on single crystal diamond^{13, 15}. This represents a significant improvement over polycrystalline films although for diamond to be utilized as a substrate material for the fabrication of active electronic devices these low angle grain boundaries must be further reduced or eliminated. The role of boundaries on the reduction of mobility and carrier transport in polycrystalline and highly oriented diamond films will be discussed in greater detail in the section on Hall effect characterization.

3.2. Polar X-ray Diffraction

XRD polar measurements were performed at diffracting conditions for the {004}, {022} and {111} projections on the 3 thinner films shown in the previous section. The orientation and texture evolution may best be seen by looking at the {022} polar progression as a function of film thickness as shown in Figure 1. Data were taken at a diffraction angle of $2\theta = 75.3^\circ$ and plotted on a stereographic projection representing the angular distribution in the range; $0^\circ \leq \chi < 85^\circ$ and $0^\circ \leq \phi \leq 360^\circ$. At 1 μm there is clear evidence of both the (100) four-fold symmetry and preferred orientation, however one can also observe the {022} twin spots indicated by the arrows on Figure 1(a). Further evidence of epitaxy is in the relationship between the D{022} and the Si{331} peaks located on

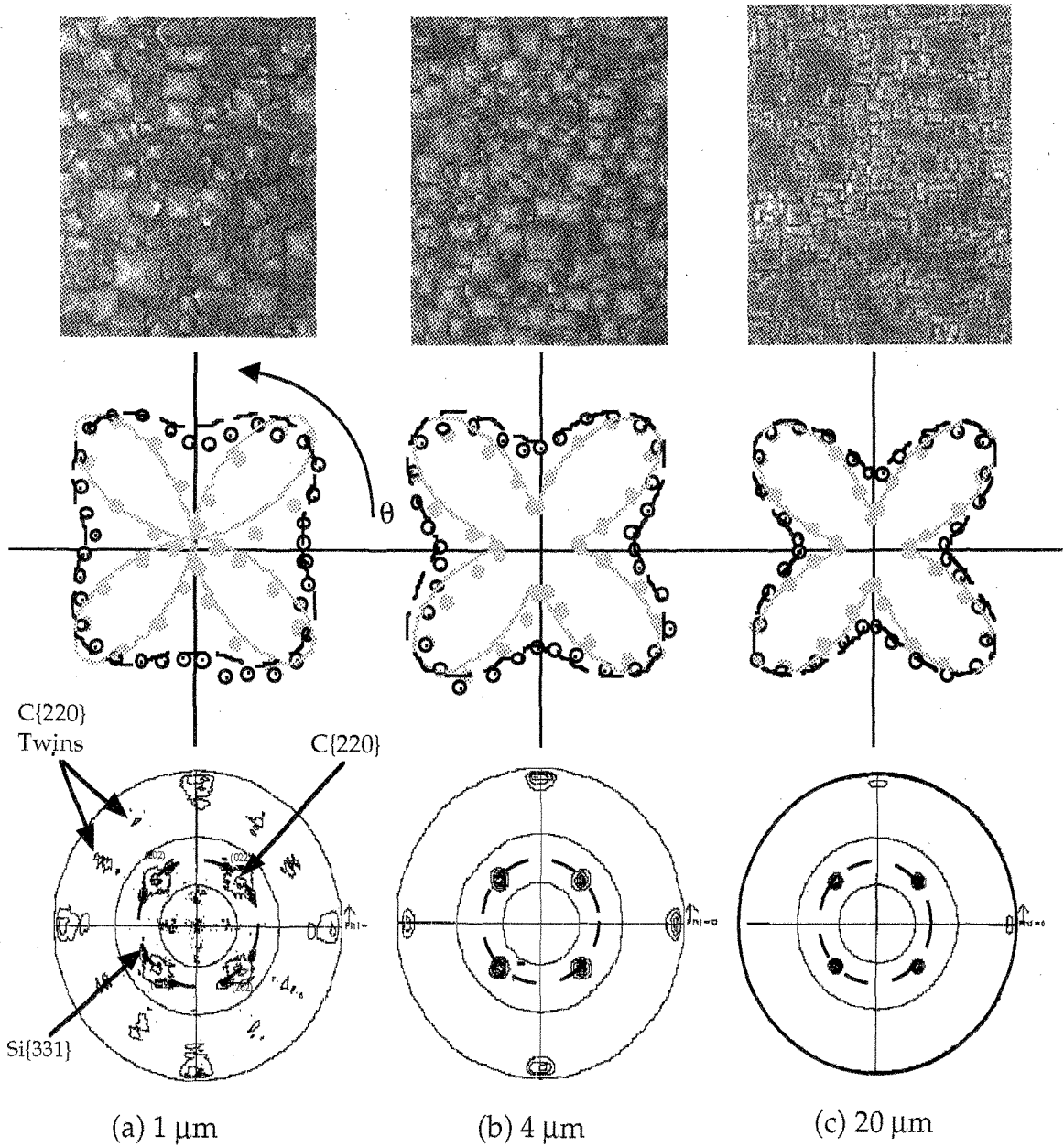


Figure 1: SEM, polar Raman and polar XRD spectra taken from (a) 1 μm, (b) 4 μm and (c) 20 μm thick HOD films. The open circles on the polar Raman plot are from the diamond film, the filled circles are from the Si substrate.

the dashed circle at $\chi = 45^\circ$. Both sets of spots may be seen since silicon and diamond have Bragg reflections with $2\theta_{hkl}$ near 75.3° (Si at $2\theta = 76.4^\circ$ and Diamond at $2\theta = 75.3^\circ$). At $4 \mu\text{m}$ the orientation and texture have greatly improved as can be seen by Figure 1(b). The full width at half maximum (fwhm) of the {022} peaks have decreased from 18 to 14 degrees and the twin spots are no longer visible. By $20 \mu\text{m}$ the fwhm has further improved to 9 degrees.

3.3. Polarized Raman Spectroscopy

As mentioned in the experimental section the polar Raman intensity (I) will vary as;

$$I = A \sin^2(2\theta)$$

for {100} single crystals and will be invariant for polycrystalline films (i.e. $I = \text{const.}$). In this study, the polarization data from both the film and substrate are fit to the relationship;

$$I = A \sin^2(2\theta) + B,$$

where B represents the component of the Raman intensity that is invariant to polarization angle (θ). This invariant intensity can be attributed to; (i) a polycrystalline component, (ii) surface roughness or (iii) the 0-6 degree misorientation that has been commonly observed for epitaxial diamond on SiC. A random distribution of {100} oriented grains, misoriented by less than 6 degrees would have an angular response with an invariant component (i.e. the intensity (I) would never go to zero). The SEMs indicated that there is a polycrystalline component visible up to $15 \mu\text{m}$ and it has also been shown via XRD above that misorientations exist between the epitaxial grains. Since it is not possible at this time to differentiate between these effects all are combined into the single invariant term (B). The ratio of $A:(A+B)$ can thus represent the fraction of the total intensity that is polarization sensitive and is a useful measure of the relative single crystalline quality of the highly oriented films. As the film quality approaches that of an ideal single crystal the ratio;

$$R_{(100)} = A/(A+B),$$

approaches unity and for polycrystalline films it will go to zero.

The polar response for both the diamond film and silicon substrate are normalized and plotted for increasing thicknesses in Figure 1. The solid circles refer to the silicon data and open circles to the diamond film. At $1 \mu\text{m}$ in thickness as also indicated by the SEM and XRD data there is clear evidence that a component of the diamond film is oriented with respect

to the silicon substrate. $R_{(100)}$ for this film was calculated to be 0.27 indicating that polarized Raman can be used to accurately detect partial epitaxy for diamond films on the order of $1 \mu\text{m}$ thick. From the SEM analysis approximately 60% of the grains appear oriented. As the thickness increases, the polarized response improves to $R_{(100)} = 0.39$ at $4 \mu\text{m}$ and then to 0.55 by $20 \mu\text{m}$. One also notices that the silicon intensity response does not go entirely to zero. Since the silicon is known to be single crystal with negligible misorientation, this constant component for the silicon signal is speculated to be due to roughness and scattering at the diamond/SiC/Si interfaces. Although difficult to calculate given the data available, this scattering component due to the interface should also affect the diamond polar response. As thickness increases, the component of the diamond signal that is due to interfacial scattering should decrease and eventually go to zero as the film thickness surpasses the penetration depth for Raman spectroscopy. By 30 microns, the silicon signal is no longer detectable and $R_{(100)}$ was measured to be 0.7. The XRD data indicated a dramatic improvement in orientation and texture as thickness increased from 1 to $30 \mu\text{m}$. The full width at half maximum at $30 \mu\text{m}$ was measured to be approximately 6 degrees as compared to less than 1 for the silicon substrate. The polarized Raman intensity ratio, $R_{(100)}$, increased from 0.25 at $1 \mu\text{m}$ to 0.7 by $30 \mu\text{m}$ suggesting that polarized Raman can be a sensitive measure of the orientational quality of these HOD films.

3.4. Hall Effect Characterization

The temperature dependent Hall effect data for the boron-doped HOD film are shown in Figure 2. The room temperature mobility and carrier concentration for this film were measured to be $278 \text{ cm}^2/\text{V-s}$ and $6 \times 10^{13} / \text{cm}^3$ respectively. For comparison, typical room temperature Hall mobility data for boron doped films deposited on; (i) polycrystalline, (ii) highly oriented, and (iii) single crystal diamond substrates; was obtained from the literature^{13,26,27,29-32}. These data represent typical films doped *in-situ* via the gas phase (to dopant concentrations of $N_A = 10^{17} - 10^{19} / \text{cm}^3$) and clearly indicate three separate ranges of values with little overlap. There are polycrystalline films with room temperature mobilities ranging from $2 - 50 \text{ cm}^2/\text{V-s}$ at room temperature; highly oriented films falling between 135 and $278 \text{ cm}^2/\text{V-s}$; and single crystal films with values ranging from 250 to $1340 \text{ cm}^2/\text{V-s}$. Much of the variations within each film class can be attributed variations in deposition quality, differences in boron concentration, or substrate morphology and condition

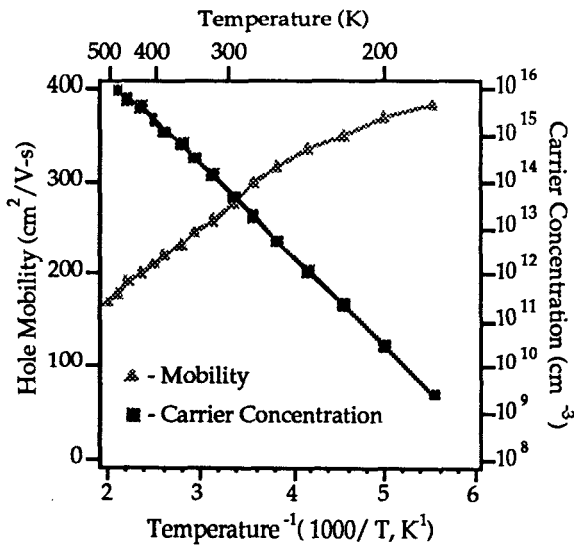


Figure 2: Temperature dependent Hall effect data showing mobility and carrier concentration of the B-doped HOD film.

prior to deposition of the p-type layer. However, it is clear in general that single crystal films have superior transport properties to highly oriented films which again have better properties than polycrystalline films deposited under similar growth conditions. If one looks at just the highest mobilities in each of the three categories it is evident that the films represent three very separate classes of film quality;

polycrystalline: $\mu = 50 \text{ cm}^2/\text{V-s}$,
 highly oriented: $\mu = 278 \text{ cm}^2/\text{V-s}$, and
 single crystal: $\mu = 1340 \text{ cm}^2/\text{V-s}$.

These values are for films with similar room temperature carrier concentrations; $5 \times 10^{13} < p < 5 \times 10^{14} \text{ cm}^{-3}$.

These data indicate that although the highly oriented films have mobilities 5 times higher than polycrystalline films, they are still only 1/5 that of single crystal homoepitaxial layers. These differences emphasize the importance of further reducing the film misorientation and associated defects at grain boundaries. Malta et. al, 26 showed that degradation in transport properties of polycrystalline films was attributed to charge carrier trapping and scattering from defects at the grain boundaries. By going to the HOD film the intergranular defects are reduced, however the presence of low angle grain boundaries still creates scattering and trapping centers at densities much higher than for single crystals. Fox and coworkers 27 presented a comparison

of the transport properties of boron doped films grown simultaneously on; polycrystalline, highly oriented, and single crystal substrates. From a model developed by Seto²⁸ to explain conduction in polycrystalline silicon, Fox et. al, calculated the trap density in both polycrystalline and highly oriented films. When traps at the grain boundaries are filled a potential barrier, of height E_B , develops. The expression for the degradation in mobility due to the formation of a potential barrier is;

$$\mu = \mu_0 \exp(-E_B/kT)$$

where μ_0 represents the intragranular mobility and k is the Boltzman constant. The barrier height (E_B) when all the traps are filled will be proportional to the square of the trap density assuming a constant carrier concentration. The HOD films in the study by Fox et. al, showed a 50% reduction in interfacial trap density resulting in an improved mobility of almost 3 times that of the polycrystalline film at room temperature. These data emphasize the importance of further reducing the intergranular defect density and associated trapping centers. Currently the misorientation in some of the higher quality films ranges between 2-6 degrees with an XRD fwhm measured at 6 degrees for the (004) peak. For highly oriented films to achieve transport properties similar to those currently obtained from single crystal films, the interfacial trap density must be approximately an order of magnitude lower (or reduced by an additional 90% from the current levels). To reduce the trap density one needs to either decrease the misorientation or reduce the grain boundaries density.

4. SUMMARY

Highly oriented, (100) textured diamond films deposited on (100) Si substrates have been characterized by a wide range of analytical techniques. Both polar XRD and polarized Raman spectroscopy show that epitaxy and a preferred orientation can be identified for films as thin as 1 μm and that orientation improves dramatically under deposition conditions that promote (100) texture. Polarized Raman was shown to be sensitive to changes in the quality of orientation thus making it a useful analytical tool when polar XRD is not available. Hall effect measurements on boron-doped, HOD films indicated room temperature hole mobilities of 278 $\text{cm}^2/\text{V-s}$ at a carrier concentration of $6 \times 10^{13} \text{ cm}^{-3}$. This mobility represents a great improvement in the transport properties over typical polycrystalline films however further improvements will be linked to the ability to reduce the misorientation and corresponding density of interfacial defects.

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