Carrier transport characterization of high-density plasma-induced p-to-n type converted MWIR HgCdTe material

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ABSTRACT

Exposure of p-type HgCdTe material to H_2-based plasma is known to result in p-to-n conductivity type conversion. While this phenomenon is generally undesirable when aiming to perform physical etching for device delineation and electrical isolation, it can be utilized in a novel process for formation of n-on-p junctions. The properties of this n-type converted material are dependent on the condition of the plasma to which it is exposed. This paper investigates the effect of varying the plasma process parameters in an inductively coupled plasma reactive ion etching (ICPRIE) tool on the carrier transport properties of the p-to-n type converted material. Quantitative mobility spectrum analysis of variable-field Hall and resistivity data has been used to extract the carrier transport properties. In the parameter space investigated, the n-type converted layer carrier transport properties and depth have been found to be most sensitive to the plasma process pressure and temperature. The levels of both RIE and ICP power have also been found to have a significant influence.

Keywords: HgCdTe, plasma processing, inductively coupled plasma reactive ion etching, type conversion, Hall effect, quantitative mobility spectrum analysis, carrier transport, material characterization.

1. INTRODUCTION

Traditional technologies for fabrication of high-performance n-on-p IR photodiodes on HgCdTe material are generally based on ion implantation or ion beam milling processes. [1] More recently, grown junctions have been demonstrated by molecular beam epitaxy (MBE). MBE is capable of very accurate control over the material and junction properties, although this technology is significantly more complicated and expensive. A novel fabrication process based on plasma-induced type conversion using a parallel-plate reactive ion etching (RIE) tool has previously been demonstrated to also be capable of producing high-performance HgCdTe photodiodes. [2] This plasma-induced type conversion process has major advantages over traditional ion beam milling and ion implantation processes since it does not require a post-exposure anneal to repair damage and activate dopants. Furthermore, because of the relatively low energy of impinging ions from the plasma, the passivation layer need not be reapplied after the type conversion process. This means that the fabrication process is truly planar and does not expose the junction surface to atmosphere at any stage.

The ion beam milling process utilizes a relatively high energy (typically 100-1000eV) beam of neutralized Ar ions which are accelerated directly at the p-type sample surface to result in physical sputtering of material, and p-to-n type conversion. Both the physical etch rate and type conversion depth have been found to be positively related to the ion current. [3] Ion beam milling is generally applied for physical etching steps in device fabrication processes, where the type conversion phenomenon is an unwanted side effect that degrades device performance. In the ion implantation process, dopant ions are accelerated at the sample in order to implant them in the crystal lattice. Depending on the dopant ion, the ion implantation process can be used to result in p-to-n or n-to-p type conversion. After the implantation step, a high temperature anneal is required to repair the damage caused by the high energy ion impact, and to activate the dopant species in the lattice.

In recent years, more advanced hybrid RIE tools incorporating secondary plasma generation power sources have been developed, such as electron cyclotron resonance (ECR) and inductively coupled plasma (ICP). While typical plasma density in the parallel plate RIE tool is of the order of 10^9 cm^{-3}, these hybrid tools are capable of generating plasma densities of approximately 10^{11} cm^{-3}. They also have the advantage of being able to separately control the power

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primarily applied to the plasma, and the power driving plasma ions at the sample surface. These advantages allow for much greater control over the plasma condition and, more specifically, enable the generation of higher density plasma without necessarily increasing the energy of ions driven at the sample surface. [4] The ICPRIE tool has advantages over the ECRRIE tool due to its lower setup and running costs, improved plasma uniformity, and easier scale up to production. A diagram of the ICPRIE configuration is shown in Figure 1.

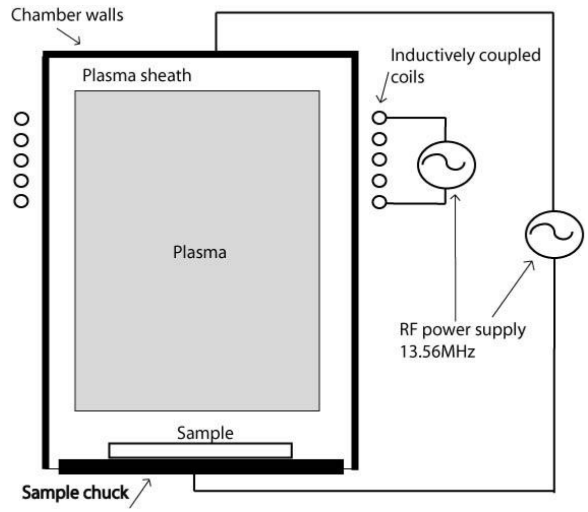

Fig.1: ICPRIE reactor configuration.

Many papers related to plasma etching of HgCdTe have focused on the application of the technology to dry physical etching of high-resolution and high aspect ratio structures typically required for current state-of-the-art HgCdTe IR focal plane arrays. [5,6] In these cases, it is desired to control the etch process for controlled anisotropic removal of material, resulting in smooth etched surfaces, high aspect ratios, and minimal modification of the electrical properties of the material. To this end, it has been reported in Ref [7] that the RIE plasma induced p-to-n type conversion process can be inhibited at low substrate temperatures in the order of 100K. It has also been shown in the same paper that for H_2/CH_4 gas chemistry, the type conversion depth tends to increase with increasing H_2 partial pressure in the gas mixture. In the case of plasma etching with Ar only, the junction depth increases with etch time, and passes through a peak with varying process pressure. As a result of this study, it was proposed that lowering the substrate temperature and decreasing the H_2 component of the gas mixture would reduce the extent of undesirable p-to-n type conversion. [7] In contrast to this investigation of RIE processing for dry etching applications, the aim of the present study is to identify plasma conditions which favour controlled p-to-n type conversion.

A number of theories have been published regarding the plasma-induced type conversion mechanism, which is believed to be related to similar type conversion known to occur under ion beam milling processes. It is most widely accepted that this type conversion mechanism is primarily due to the liberation of interstitial Hg at the exposed surface, which then diffuses into the HgCdTe material and recombines with vacancy sites, revealing background n-type doping. [7-11] It has also been proposed that three forms of H incorporation are also related to the plasma-induced type conversion phenomenon. [12]\ The type conversion phenomenon has also been observed for extrinsically doped HgCdTe material. An additional type conversion mechanism of “kick-out” has been proposed for extrinsically doped material, whereby interstitial Hg displaces extrinsic dopant atoms from the lattice. [13] This p-to-n type conversion can be reversed by a high-temperature anneal under Hg, which has been found to restore the original electrical properties of the exposed material. [14] It is intended that by investigating trends with changing ICPRIE process parameters with respect to the carrier transport properties of the p-to-n type converted material, this work will contribute towards further understanding of this type conversion mechanism.

The carrier transport properties of the type converted material have been investigated by variable-field Hall and resistivity measurements at various temperatures. Such measurements at a single field are able to provide information about the net contribution of all carriers in the sample to the conductivity. By taking these measurements at a number of fields, the process of quantitative mobility spectrum analysis (QMSA) can be used to extract the contribution of individual carriers to the conductivity. [15, 16] The temperature dependence of these carriers in terms of mobility and concentration can be interpreted to provide additional information, such as the scattering mechanisms and activation energy related to each carrier. [17]

In this paper, the carrier transport properties of the p-to-n type converted HgCdTe layer exposed to H2-based plasma in an ICPRIE tool will be investigated as a function of varying ICPRIE process conditions. These plasma process parameters include the RIE power, ICP power, process pressure, $H_2/CH_4/Ar$ gas flows, sample chuck temperature, He backside cooling pressure of the chuck, and the exposure time. The carrier transport properties are to be extracted via QMSA from variable-field Hall and resistivity data at fields of up to 12T and measured at various temperatures over the range 20-300K. Samples will be fabricated using the van der Pauw configuration, and wet etch-backs will be performed between measurements in order to determine the depth profile of the extracted carriers. The primary aim of this work is to begin to characterize the general plasma process for controlled type conversion, and more specifically to develop this process for the more advanced ICPRIE tool, which is expected to be capable of greater control of the plasma condition and thus the properties of the type converted material. This material characterization should develop this plasma processing technology for applications in n-on-p photodiode fabrication.

2. EXPERIMENT

Starting material for this work was vacancy-doped p-type $Hg_{0.7}Cd_{0.3}Te$ grown by LPE on CdZnTe and purchased from Fermionics Corporation. Most samples were taken from the same wafer, specified by Fermionics to have a p-type doping density of 1.1×10^{16} cm^{-3}, hole mobility of 400cm^2/V.s at 77K, cutoff wavelength of 5.2µm at 77K, and nominal epilayer thickness of 20µm.

Samples were initially cleaved into squares approximately 5mm×5mm. These wafers then underwent a standard hot organic clean in consecutive baths of trichloroethylene, acetone, and methanol, and were then dried under a high purity nitrogen flow. This was immediately followed by a 10s wet surface etch in 0.1% Br/methanol, which from known etch rates is expected to remove approximately 0.1µm of surface material, after which the sample was quenched with methanol, and dried with nitrogen.

The cleaned and thinned wafers were then bonded with Apeizon H vacuum grease to a silicon wafer for mounting in the ICPRIE tool. It has been found that the use of the Apeizon grease provides a better thermal bond between the sample and the temperature controlled chuck, resulting in better control of the sample surface temperature during plasma exposure. An Oxford Plasma Technologies System 100 with ICP180 was the ICPRIE tool used to perform type conversion of all samples. The sample chuck is equipped with a heater and may be cooled by He backside pressure. The sample and silicon carrier wafer were then loaded into the ICPRIE tool for processing under a particular set of plasma conditions. The sample was then removed from the plasma tool and a van der Pauw “Greek cross” structure was mesa etched in the HgCdTe down to the substrate, using a standard photolithography process followed by wet etch in 1% Br/HBr solution. This was followed by another photolithography step to define contact areas, and then Cr/Au contacts were deposited using a thermal evaporator. After metal liftoff, the samples were mounted in sample carriers and bonded out with Au wire, using pressed indium to the metal sample contacts and a silver epoxy to the sample carrier. The pressed indium to the sample contacts has been found to provide good ohmic contact, and minimum degradation in contact quality with removal and reapplication, which is required between wet etch back steps to determine the carrier transport depth profile.

Hall and resistivity measurements were performed in an Oxford Instruments superconducting magnet capable of generating magnetic fields from 0-12T of either polarity. The sample was mounted in a helium cooled cryostat capable of maintaining stable temperature over the measurement range of 20-300K. The sample was connected to a current source and digital voltmeter via a computer-controlled scanner equipped with a Keithley Model 7065 Hall Effect Card. The computer also controls the magnetic field and current bias, and records the voltage measurement data at each magnetic field point. The conductivity tensors were then calculated from the Hall and resistivity data using custom MatLab software, and QMSA software was used to extract the mobility spectra.

Once the initial Hall measurements and data analysis were completed, metal indium bonds were removed, taking care to cause minimal damage to the deposited Cr/Au contacts. The sample then underwent a standard hot organic clean, and was etched back for 10s in 0.1% Br/methanol solution. This first wet etch-back step was the same for each sample and is expected to remove approximately 0.1μm of material. The deposited metal contacts were not masked during these etch-back steps, since the methanol in the etch solution was found to dissolve the photoresist mask, resulting in a brown residue on the sample surface after etch back. A Br/HBr solution was an alternative which would not have attacked such a photoresist mask, however it is known to leave the HgCdTe surface nonstoichiometric. The Cr/Au contact quality was not observed to degrade with exposure to the Br/methanol solution, as determined by IV measurements using an HP4156 semiconductor parameter analyzer. After the wet surface etch-back, the sample was rebonded and mounted in the sample carrier. Measurement and analysis was then repeated for the remaining layer. This differential technique allows the depth profile of the type converted layer to be determined. The accuracy of the depth profile is limited by the thickness of the layer removed in each wet etch step, the spatial uniformity of the material removed, and the accuracy to which the etch depth is known.

3. RESULTS AND DISCUSSION

For comparison to previously reported results based on the traditional RIE tool, a sample (BP18) was prepared in the same parallel-plate RIE reactor from similar HgCdTe material and plasma conditions to those in Ref[17]. The RIE plasma conditions used to fabricate this sample were: H_2:CH_4 flow of 54:10 sccm at process pressure of 100mTorr, RIE power of 120W, and 2 min plasma exposure time. Irreversible temperature indicator stickers were exposed to the same plasma conditions and showed that the sample surface temperature remained below 77C during plasma exposure. It should be noted that these plasma conditions are slightly different from those under which the photodiodes reported in Ref [2] were exposed; total gas flow about 40 sccm of H2:CH4 ratio 5:1, at approximately 500mTorr process pressure, and RIE power 100W. The starting material for the present RIE sample was part of the same wafer used to prepare many of the other samples detailed in this paper. The proposed p-to-n type converted profile for HgCdTe material exposed to H_2/CH_4/Ar plasma is shown in Figure 3.1. The electron mobility spectra as a function of temperature obtained from this sample is shown in Figure 3.1, and is very similar to those reported in Ref[17] and Ref[18] for samples exposed to similar plasma process conditions. These mobility spectra can be interpreted to show temperature-independent low-mobility surface carriers which have been attributed to surface damage, combined with temperature dependent high-mobility bulk carriers. These high mobility bulk carriers are expected to be associated with regions close to the metallurgical junction and should result in good device performance. The depth profile of the sample prepared under higher pressure in Ref[18] was not measured, and as such can not be compared to the depth profile of the samples prepared at lower pressure in Ref[17] and in this paper. The latter results, and those in the present study, indicate that the type conversion process is repeatable and provides a baseline for comparison with mobility spectra obtained from samples fabricated using the ICPRIE plasma tool.

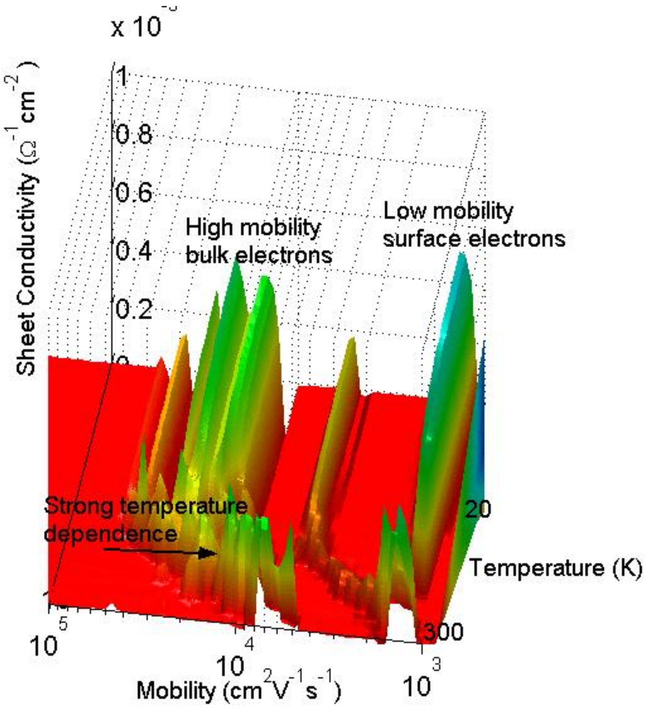

Figure 3.1 – Electron mobility spectra as a function of temperature for sample BP18 (exposed in parallel plate RIE tool) before wet etch-backs.

The plasma parameters for each sample fabricated using the ICPRIE tool and investigated in this paper are listed in Table 3.1.

Table 3.1 – ICPRIE plasma process parameters associated with each sample. A constant plasma exposure time of 2 min was used combined with He backside cooling pressure of 10 Torr.

Sample	RIE power (W)	ICP power (W)	H2 flow (sccm)	CH4 flow	Ar flow	Process pressure (mTorr)	Chuck temperature (C)
BP10	100	250	60	10	106	15	10
BP12	25	250	60	10	106	15	10
BP13	100	0	60	10	106	15	10
BP14	150	250	60	10	106	15	10
BP15	150	250	60	10	0	15	10
BP16	100	250	60	10	106	8	70
BP17	100	250	60	10	0	8	70
BP19	100	250	60	10	106	80	70

As a starting point, the temperature-dependent mobility spectra of the sample fabricated using the ICPRIE tool with the deepest p-to-n type conversion and highest electron mobility (BP19) is shown in Figure 3.2 for comparison to both the mobility spectra of the sample fabricated using the “old” plasma conditions in the parallel-plate RIE tool (Figure 3.1), as well as the other samples fabricated under different ICPRIE plasma process conditions. The junction depth of this sample, BP19, is significantly greater than that achieved for other samples, and therefore closest to the approximate target depth of 1-2μm. The mobility of bulk electrons in this sample is also the highest of all measured samples, which is not unexpected given that the deeper junction will incorporate higher quality material further from the lower quality HgCdTe surface.

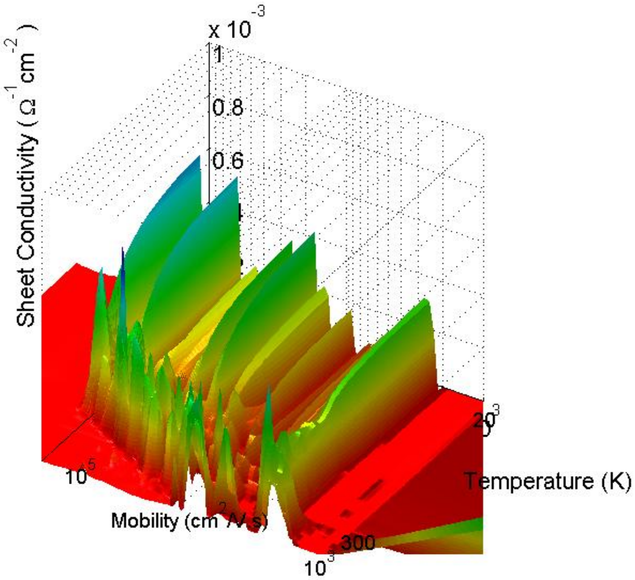

Fig. 3.2 – Electron mobility spectra as a function of temperature for sample BP19 before wet etch-backs.

The characteristics of the n-type mobility spectra of the p-to-n type converted region of the ICPRIE sample (BP19) shown in Figure 3.2 compared to the RIE sample (BP18) of Figure 3.1, include higher mobility n-type carriers and increased conductivity from these n-type carriers. Compared to the ICPRIE sample BP19, the most significant differences in plasma conditions for the RIE sample (BP18) are the lower substrate temperature without backside cooling or Apeizon grease, the higher process pressure of 100 mTorr, and the lack of Ar in the gas chemistry. It is difficult to draw a direct comparison between the nominal applied RIE power in the different reactor configurations, given differing rf power sources and plasma chamber dimensions. Irreversible temperature indicator stickers showed that the sample surface temperature reached approximately 77-81C during plasma exposure for sample BP19, while the surface temperature for the RIE sample was below that of the lowest indicator of 77C.

Considering the electrons with a range of high mobility values (approximately 1-8$\times10^4$ cm^2/V.s at 77K) which remain after the first etch-back step to be bulk electrons within the p-to-n type converted region, we can calculate a weighted average conductivity to obtain a representative single mobility carrier at each temperature point. Plots of weighted average electron mobilities and electron sheet doping densities as a function of temperature for both sample BP18 and BP19 are shown in Figure 3.3 and 3.4 respectively. These provide simplified representations of the information in the temperature dependent mobility spectra shown in Figure 3.1 and Figure 3.2. The proposed profile of the type converted layers is shown in Figure 3.5.

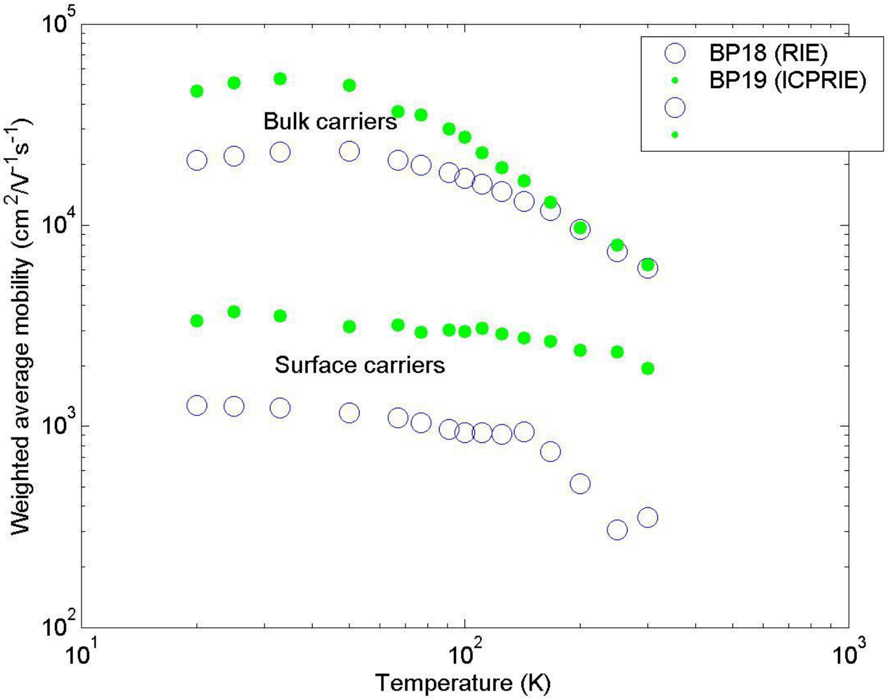

Fig. 3.3: Weighted average electron mobilities for sample BP18 and BP19 as a function of temperature. Each mobility point at a particular temperature is the weighted average of carriers with a range of mobility values. These curves can be related to the peaks of the respective mobility spectra shown in Figure 3.1 and Figure 3.2.

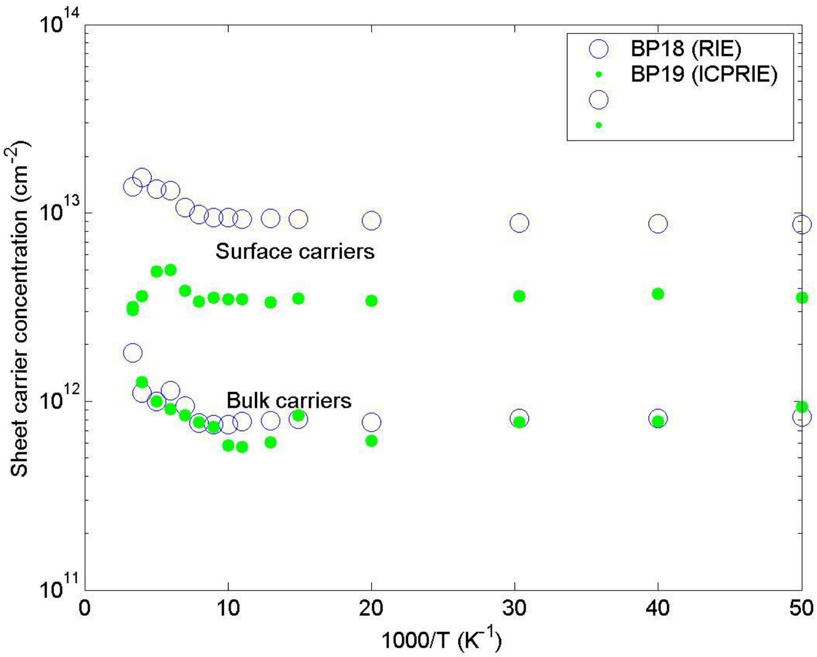

Fig. 3.4: Weighted sheet electron concentration for sample BP18 and BP19 as a function of temperature. These sheet concentrations correspond to the mobility values of Figure 3.3.

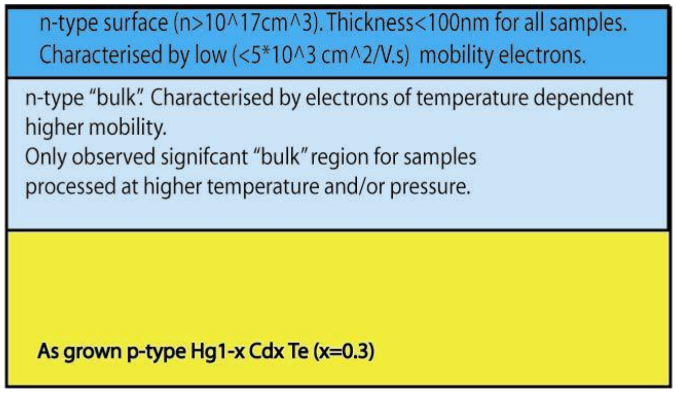

Figure 3.5 – Profile of p-to-n type converted layer in p-HgCdTe.

With reference to Figure 3.3, we observe a strong dependence of electron mobility on temperature for both sample BP18 and BP19, which is consistent with high quality bulk material. This relationship is a strong indication of a lattice scattering mechanism being the dominant mechanism, as a result of lattice vibrations increasing with increasing temperature. This leads to a greater scattering probability and hence lower mobility with increasing temperature. [17] Assuming that the thickness of the n-type layer is approximately 1μm for both samples BP18 and BP19, the electron concentration in the type converted layer is of the order of less than 10^{15}-10^{16} cm^{-3}, which is typical of high-quality n-type material. For ICPRIE sample BP19, after wet-etch backs to a cumulative etch depth of approximately 0.7μm, the p-to-n type converted layer is not completely removed, with electron mobility spectra obtained showing conductivity to be dominated by high mobility bulk electrons. The significantly increased asymmetry in the Hall and resistivity voltages, measured after this most recent wet etch-back, is similar to that observed in measurements of very thin and/or nonuniform n-on-p layers, so it is believed that the total p-to-n type conversion for sample BP19 is approximately 1μm. However, further wet etch-back(s) and Hall measurements are required to determine the total p-to-n type conversion depth for ICPRIE sample BP19.

From the results obtained so far for these samples, it appears that the p-to-n type converted layer of sample BP19 produced using the ICPRIE tool consists of increased conduction from higher mobility electrons than that of sample BP18, particularly at temperatures below approximately 100K. As such, it is reasonable to expect that photodiodes of similar or better performance to those fabricated in the RIE tool could be produced using these ICPRIE plasma process conditions.

Accurate placement of the junction depth is essential for producing high performance n-on-p photodiodes, with 2μm deep junctions in a 10μm thick epilayer being a typical target. For all samples prepared at a relatively low substrate temperature of 10C during plasma exposure, it was found that the first 10s wet etch-back removed all of the p-to-n type converted material. This result is consistent with reported type conversion depth figures measured by differential Hall for an RIE plasma-processed sample of less than 1μm, [17] and an ECRRIE plasma-processed sample for which the substrate temperature was not specified of less than 350nm. [19] It is known that the depth of the p-to-n type converted layer was less than approximately 0.1μm because the mobility spectra obtained after the first etch-back no longer contained significant contribution from n-type carriers, and the Hall coefficient obtained from the same measurements at 77K was positive for all applied magnetic fields. The temperature dependent mobility spectra obtained for sample BP14 before a wet etch-back is shown in Figure 3.6, which is similar to that obtained for other samples prepared at the chuck temperature of 10C.

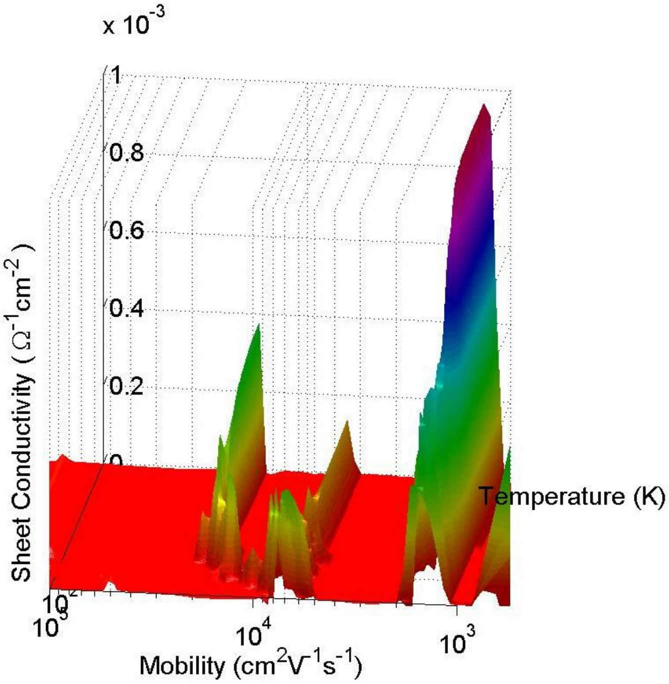

Fig. 3.6: Mobility spectra as a function of temperature for sample BP14 before wet etch-backs.

The mobility spectra for all samples prepared at the low chuck temperature of 10C, such as that shown in Figure 3.6, do not show bulk carriers of sufficiently high mobility for high-performance n-on-p devices. The existence of such high-mobility n-type carriers is not likely given the shallow type converted layer, in which the effects due to the surface and interface will act to reduce carrier mobility. The weakly-temperature-dependent mobility of bulk n-type carriers for these thin type converted layers was generally less than 10000cm^2/V.s at low temperatures, compared to extracted mobility up to over 50000 cm^2/V.s for type converted layers of thickness greater than 0.1μm, as in BP19, shown in Figure 3.2.

At an increased chuck temperature of 70C, significant changes were observed in the mobility spectra, and the depth of the n-type converted layer was confirmed to increase through differential measurements. Samples BP16, BP17, and BP19 were all processed at this higher chuck temperature, with samples BP16 and BP17 being prepared at a lower process pressure of 8mTorr compared to BP19 (80mTorr). The temperature dependent electron mobility spectra for sample BP17 is shown in Figure 3.7. For both samples BP16 and BP17, low mobility n-type surface carriers were removed by the first 0.1μm wet etch-back, and all of the high mobility bulk electrons were removed in a further 30s etch-back expected to remove an additional 0.3μm layer.

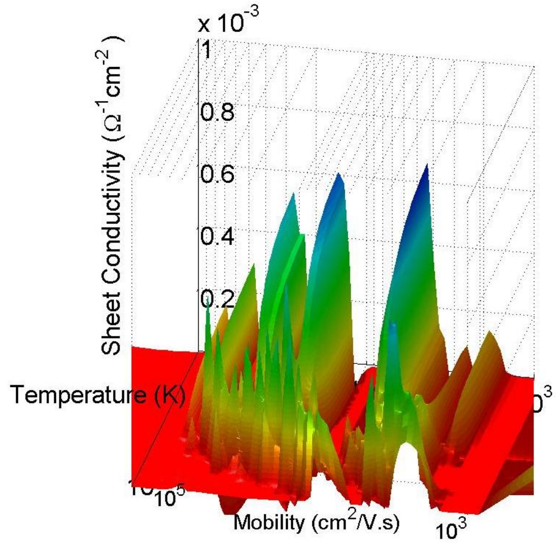

Fig. 3.7: Mobility spectra as a function of temperature for sample BP17 before wet etch-backs.

The electron mobility spectra for samples BP16 and BP17 were very similar at high mobilities, with the low mobility surface electrons of sample BP17 (exposed to H_2/CH_4 plasma – without Ar) being of slightly higher mobility (6000-9000 $cm^2/V.s$) and with a weak temperature dependence, compared to the lower mobility (2000-3000 $cm^2/V.s$) and temperature independent surface electrons of sample BP16 (exposed to $H_2/CH_4/Ar$ plasma). Lower mobility and a reduced degree of temperature dependence may be associated with lower x value and/or increased damage to the HgCdTe material. This may be an indication that the addition of Ar to the plasma chemistry results in a slightly increased degree of damage and/or Hg depletion in the thin surface layer, possibly due to increased lattice damage and preferential sputtering of Hg caused by heavier Ar ions. The bulk electron carrier transport properties were similar for both sample BP16 and BP17, and any difference in type conversion depth was smaller than the wet etch depth of 0.3µm. Further investigation is required before any conclusion can be drawn on the effect of Ar on the surface carrier transport properties.

CONCLUSIONS

The carrier transport properties of p-to-n type converted layers exposed to various ICPRIE plasma process parameters have been characterized. An ICPRIE plasma process parameter space has been identified which results in conductivity type conversion of vacancy-doped $Hg_{0.7}Cd_{0.3}Te$ material, to form n-type material with carrier transport properties similar or better than those reported for a similar process performed using a traditional parallel-plate RIE tool. This result indicates that a n-on-p photodiode fabrication process could be based on this ICPRIE plasma-induced type conversion, with junctions formed using the ICPRIE tool capable of improved performance compared to those formed by the RIE tool.

In the parameter space investigated, increasing the substrate temperature and process pressure during plasma exposure have been identified as changes in ICPRIE process parameters that have the most significant influence on increasing the depth of p-to-n type conversion. Further wet-etch back steps and Hall measurements are required to determine the extent to which increasing the process pressure increases the p-to-n type conversion depth.

Increasing the RIE power is also shown to affect the carrier transport properties of the n-type layer, although any effect on junction depth was not significant enough to be confirmed by differential measurements. The addition of ICP power was not observed to have a significant influence on type conversion depth, although it is expected to change the transport properties, in particular those of the surface layer. Addition of Ar to the plasma gas chemistry may increase the degree of surface damage and/or Hg depletion at the surface, with no significant effect observed on the bulk transport properties or junction depth.

For development of the ICPRIE plasma process for photodiode fabrication, the target junction depth can be primarily controlled with the substrate temperature and/or process pressure during plasma exposure. The surface damage resulting from this plasma exposure has been shown for all samples to be confined to a layer less than 0.1μm thick, and can be removed by a light wet chemical etch. It is expected that the carrier transport properties, including doping density, of the bulk type converted material can be more accurately controlled at the target junction depth by varying the RIE and/or ICP power.

For the application of ICPRIE processing to physical etching, it has been shown that the type conversion phenomenon is confined to a very thin layer of less than 0.1μm for low process pressure and temperature. In particular, the ICPRIE process is capable of maintaining the plasma at lower process pressures than the traditional RIE tool, where these lower process pressures have been shown to reduce the type conversion depth. While it is known that etch rates can be increased by increasing the RIE and/or ICP power, it has also been shown here that increasing power does not necessarily result in a significant increase in the p-to-n type conversion depth.

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