Insulator to metallic transition due to intermediate band formation in Ti-implanted silicon

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Abstract

We have analyzed by means of electrical transport measurements, the insulator to metallic transition due to intermediate band (IB) formation (insulator to metallic-IB) in silicon layers. The samples were implanted with titanium concentrations well above the solid solubility limit and subsequently pulsed laser melted (PLM). Whereas the doping of silicon with Ti impurity concentrations below the Mott limit is known to produce deep levels which act as non-radiative recombination centers, the introduction of a high concentration of deep impurities above this limit could form an IB. Time-of-flight secondary ion mass spectrometry (ToF-SIMS) measurements show the remaining titanium concentration profile after PLM, indicating whether this concentration is above or below the theoretical limit for IB formation in the different implanted samples. Sheet resistance and Hall effect measurements performed in the temperature range (100–300 K) show that insulator to metallic-IB transition takes place for concentrations above \( \sim 10^{20} \text{ cm}^{-3} \). This transition becomes apparent in a rectifying behavior observed in van der Pauw and transversal I-V electrical measurements at low temperatures. Contacts exhibit Schottky or ohmic behavior for samples with Ti concentrations below or above the transition, respectively. All these results point out to the metallic behavior of the IB and provide a powerful tool to determine the IB formation in a semiconductor.

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1. Introduction

To increase the efficiency of solar cells a great technological effort has been devoted over the last years. The intermediate band solar cell (IBSC) is a promising device based on the intermediate band (IB) concept that has been proposed within the third generation of photovoltaics to exceed the efficiency of conventional solar cells [1]. An IB material is characterized by the existence of an electronic energy band of allowed states within the conventional band gap that enables the absorption of sub-band gap photons. In this process, one photon pumps an electron from the valence band (VB) to the IB and a second photon pumps an electron from the IB to the conduction band (CB). To ensure the occurrence of both processes, the IB must be a semi-filled or “metallic” band. Since the introduction of ultrahigh concentrations of deep centers can give rise to the formation of an IB, with suppression of non-radiative recombination. This is due to the delocalization of the impurity electron wavefunctions and the consequent suppression of the multiple phonon emission process [5]. In this way, chalcogenides supersaturated silicon materials have been investigated [6,7]. The deep level impurity concentration required to form an IB is denominated Mott limit and has been theoretically calculated (\( \sim 6 \times 10^{19} \text{ cm}^{-3} \)) [5]. The experimental determination of the delocalization transition is an essential point to investigate the IB formation.

Recently, we have proposed deep-level atoms impurified semiconductors as promising candidates to form an IB material [8,9] by means of Ti ion implantation in silicon at the high concentrations required to exceed the IB formation limit [5]. The behavior of transition metal atoms in silicon has been deeply studied in the past, mainly because the deep levels associated to them are known to increase the amount of non-radiative recombination and thus to reduce the carrier lifetime. However, we have achieved evidences of carrier lifetime recovery in these

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ultrahighly Ti impurified silicon layers, conversely to the classic understanding of the deep level impurity effect in semiconductors and supporting the predictions of a non-radiative recombination suppression when an IB is formed [10].

We have reported that silicon implanted with very high Ti concentrations and subsequently pulsed laser melted (PLM) forms an IB material [8,9]. In these studies, we remarked an important aspect: the electrical decoupling behavior observed at low temperatures between the IB material and the n-Si substrate that is under the implanted layer. This electrical decoupling behavior of the sheet resistance at low temperatures results in a resistivity higher than the Si substrate. This is not possible if a simple conduction model with two parallel layers is considered, since the total sheet resistance could never be higher than the sheet resistance of one of the layers. This indicates that an electrical decoupling effect of the layers is produced at low temperatures. We have proposed a two-layer analytical model based on the van der Pauw setup that explains satisfactorily both sheet resistance and Hall effect measurements of Ti-implanted Si assuming the IB formation. The model takes into account the current limitation in the junction between the IB layer and the substrate that has as a consequence the rectifying behavior. The analytical model fully agrees with the electrical measurements obtained from the Ti impurified layers. The details dealing with this junction were previously reported in Ref. [11].

Additionally to these electrical measurements we have reported recently a strong sub-band gap absorption in these samples that cannot be explained in terms of conventional absorption processes, such as defects or free carrier absorption and that has been suggested that could be produced by the IB formation [12].

In this work, we show an insulator to metallic-IB transition in ultrahighly Ti impurified crystalline silicon. We have measured the electrical behavior of silicon layers implanted with a very high Ti concentration by means of sheet resistance and Hall effect in the (100–300 K) range, in order to evaluate the critical concentration to obtain an IB material. We have investigated the insulator to metallic-IB transition by measuring the electrical rectifying behavior associated to IB formation and the type of electrical contact formed. The analyzed samples show the transition for Ti concentrations between $10^{20}$ and $8.5 \times 10^{20}$ cm$^{-3}$.

2. Experimental

Single crystal n-type Si (111) wafers with a thickness of 300 μm ($\rho = 200$ Ωcm; $\mu = 1500$ cm$^2$/Vs; $n = 2.2 \times 10^{13}$ cm$^{-3}$ at room temperature) were implanted in an IBS refurbished VARIAN CF3000 Ion Implanter with $^{48}$Ti$^{+}$ at different doses ($10^{13}$, $10^{14}$, $10^{15}$ and $10^{16}$ cm$^{-2}$) at 33 keV using a 7° tilt angle. After implantation, all the samples were PLM annealed at 0.8 J/cm$^2$ with a single 20 ns pulse using a KrF excimer laser (248 nm) at J.P. Sercel Associates Inc. (New Hampshire, USA). We have analyzed a non implanted sample but PLM annealed sample for comparative purposes.

Depth profiles of Ti concentration in the Si lattice were obtained by time-of-flight secondary ion mass spectrometry (ToF-SIMS) characterizations. These were carried out with a TOF-SIMS IV system manufactured by ION-TOF, using a 25 keV positive primary ion pulsed Bi$^{3+}$ beam at 45° incidence that scanned an area of 250 $\times$ 250 μm$^2$. The secondary ions generated were extracted with a 10 keV voltage and their time of flight from the sample to the detector was measured in a reflection mass spectrometer. The Ti concentration profiles were calibrated using the non saturated signal of Si$^{28}$.

The samples were electrically characterized by means of sheet resistance and Hall effect measurements with the van der Pauw configuration at variable temperature (100–300 K) using a Keithley SCS 4200 model with four Source and Measure Units. Samples were $1 \times 1$ cm$^2$ pieces of silicon wafers with four aluminum electrodes in the corners. The magnetic field used in Hall effect measurements was 0.88 T. The samples were placed inside a homemade liquid nitrogen cryostat attached to a vacuum pump to avoid moisture condensation. Measurements were performed in the four van der Pauw configurations. For each configuration the polarity of the current source and the direction of the magnetic field were changed, (for a total of 16 measurements), in order to minimize spurious thermo-galvanomagnetic effects.

The electrical characterization was complemented with transversal $I$–$V$ measurements, to further investigate the influence of the Ti implanted dose on the electrical rectifying effect. Prior to Ti implantation, the back side of these samples was superficially implanted with phosphorus at 80 keV with a $10^{15}$ cm$^{-2}$ dose followed by RTA process at 900 °C during 20 s in an Ar atmosphere to obtain a n$^+$ layer and optimum ohmic back contacts. To carry out these measurements, frontal guard ring contacts with a 1.5 mm of dot diameter and a dot-ring separation of 50 μm, were deposited on the top of the Ti implanted layer to avoid surface current leakages. These contacts were made with a pattern defined by photolithography and subsequently evaporating 200 nm of Al metallic electrodes by e-beam. No additional treatment was done in the Ti implanted layer to improve the contact.

3. Results

In previous papers we have analyzed Ti implanted Si samples with concentrations well above the Mott limit [8,11]. In this work, we extend the study with samples with Ti concentrations below the Mott limit to explore the insulator to metallic transition. Fig. 1 shows the Ti concentration depth profiles obtained from ToF-SIMS measurements of samples implanted with different Ti doses ($10^{13}$, $10^{14}$, $10^{15}$ and $10^{16}$ cm$^{-2}$) and PLM annealed at 0.8 J/cm$^2$ [13]. A push effect of the Ti impurities towards the surface with respect to the characteristic Gaussian profile of an as-implanted sample is observed as a consequence of the PLM annealing process, which tends to expel the impurities. However, the PLM permits to obtain Ti impurity concentrations above the solid solubility limit, even for the sample implanted with the lowest Ti dose. The theoretical Mott insulator-metallic limit is only clearly surpassed in the samples implanted with doses above
10^{14} \text{ cm}^{-2}. In fact, after the PLM process the Ti concentration above this Mott insulator to metallic limit extends over a reduced depth with respect to the as-implanted profile [12].

For the samples implanted with 10^{13}, 10^{14} and 10^{15} \text{ cm}^{-2} doses a double peak structure can be clearly observed, and the valley between the two peaks indicates the laser penetration depth [13]. As it can be observed, the depth of this valley increases with increasing the implanted dose. This is in agreement with the fact that the laser depth penetration increases as the crystallinity of the implanted layer degrades [14]. Moreover, the Si crystal recovery after PLM, decreases as the implanted dose is increased [13]. The peak near the surface is a result of the push effect during the melting process. The deeper peak corresponds to a zone that probably has been warmed but not melted. However the high temperatures reached together with the lower damage of the lattice in this region, produce high quality crystal layers. In a detailed structural analysis of this sample we do not detect Ti-Si precipitates or any phase formation different from silicon [13]. Silicide phase formation has been weakly detected by means of Raman spectroscopy, only in samples with very higher Ti implanted doses (5 \times 10^{16} \text{ cm}^{-2}). In these samples the lattice crystal recovery was not achieved [15].

Fig. 2 shows the sheet resistance measured in the 100–300 K range for the Ti implanted samples with doses from 10^{14} to 10^{16} \text{ cm}^{-2} and PLM annealed at 0.8 J/cm^{2}. The behavior of the sample implanted with a dose of 10^{13} \text{ cm}^{-2} is very similar to the one of the sample implanted with a dose of 10^{14} \text{ cm}^{-2}. The sheet resistance of a sample not implanted but PLM annealed has been previously analyzed as a control test to show that there is not effect due just to PLM. For the sake of clarity the results for these samples are not shown in the figure. For comparative purposes we have also included the sheet resistance of an unimplanted Si substrate. The sheet resistance of this unimplanted reference sample decreases as the temperature is reduced due to the decrease of optical phonon scattering [16]. The sheet resistance of the samples implanted with 10^{15} and 10^{16} \text{ cm}^{-2} doses is higher than the substrate sheet resistance at low temperatures. For these samples the Ti concentration is well above the theoretical Mott insulator to metallic-IB transition. This electrical decoupling behavior at low temperatures has been observed and satisfactorily explained in terms of the IB formation in the Ti implanted silicon region with a Ti concentration above the Mott limit [11]. This electrical decoupling effect cannot be associated to implantation/PLM induced defects, since TEM and High resolution TEM images of the Ti implanted sample with a Ti dose of 10^{15} \text{ cm}^{-2} show a very high crystal quality after the PLM. Similar results in relation to lattice reconstruction have been reported recently in the formation of single crystal of sulfur implanted in Si at similar very high doses (from 10^{15} to 10^{16} \text{ cm}^{-2}) [17]. TEM and HRTEM images of these samples do not exhibit extended defects and implanted and the underlying Si crystal are indistinguishable. On the other hand, four-point probe measurements at RT of "non reactive" Ar implanted Si layers subsequently annealed by PLM [18], exhibit a very high resistance, higher than non-implanted Si substrate. Since evaluated Ti implanted Si samples show a sheet resistance at RT lower or equal to the Si substrate, we can discard that the observed electrical behavior is associated to implantation/PLM induced defects.

Conversely, the sample implanted with the 10^{14} \text{ cm}^{-2} dose shows a sheet resistance behavior almost equal to the Si substrate. This result suggests that the Ti concentration for this sample is not enough to form an IB, and therefore similar electrical properties to the substrate are measured. The ToF-SIMS Ti concentration obtained for the sample implanted with a dose of 10^{14} \text{ cm}^{-2} is slightly higher than the theoretical Mott limit concentration, but just in an extremely thin layer (∼10 nm) close to the surface. Moreover, although 6 \times 10^{16} \text{ cm}^{-2} has been assumed as a reference concentration for the IB formation [5], this value could have been slightly underestimated and the actual Mott limit may be just over the maximum concentration obtained for this sample. Insulator to metal transition in sulfur-doped silicon with sulfur concentrations between 1.8 and 4.3 \times 10^{20} \text{ cm}^{-3} has been recently reported [19]. These results are in agreement with the range of 10^{20} and 8.5 \times 10^{20} \text{ cm}^{-3} observed in the maximum of the ToF-SIMS concentration profiles of the samples implanted with 10^{14} and 10^{15} \text{ cm}^{-2}, respectively. This suggests that metallic transition could be relatively independent of the impurity introduced in the Si host material [5].

Fig. 3 displays the Hall mobility measurements in the 100–300 K temperature range for the Ti implanted samples. Hall mobility of an unimplanted Si substrate is also shown. All samples exhibit n-type behavior over the whole analyzed temperature range. As in the case of sheet resistance measurements, the sample implanted with a Ti dose of 10^{13} and 10^{14} \text{ cm}^{-2} exhibits the same behavior as the substrate. In these analyzed samples, the Hall mobility increases as temperature decreases due to the decrease of the optical phonon scattering. On the other hand, samples implanted with doses of 10^{15} \text{ cm}^{-2} or higher deviate from this behavior. The electrical behavior of these samples has
been described by a two-layer analytical model taking into account the IB formation in the implanted layer [11]. This model reproduces with high accuracy both the Hall mobility and the sheet resistance measurements assuming the same initial parameters. Therefore, the sheet resistance and the Hall mobility behaviors of samples implanted with Ti doses of $10^{15}$ cm$^{-2}$ or higher is attributed to the formation of the IB. The analytical model also predicts the measured behavior for the substrate and the sample implanted with a Ti dose of $10^{14}$ when the IB is not formed. These results clearly indicate that the insulator to metallic transition produced by the IB formation has taken place for the samples implanted with Ti doses above $10^{14}$ cm$^{-2}$. The samples implanted with lower doses do not present the IB behavior, and thus we can assume that the IB has not been formed in these samples. In conclusion, the insulator-metal transition limit that would mark the IB formation in Ti implanted Si should be in the $10^{20}$–8 $\times$ $10^{20}$ cm$^{-3}$ range for the ion implantation and PLM conditions used in this work. As the implantation dose of $10^{15}$ cm$^{-2}$ produces a Ti concentration very close to the calculated theoretical Mott limit, we can conclude that this limit is valid as an initial reference for IB formation in Ti implanted Si [5].

Transversal electrical measurements have been performed in order to elucidate the nature of the electrical frontal contacts deposited on the Ti implanted layer, as a function of Ti concentration and the electrical decoupling mechanism observed in the samples with the highest Ti implanted doses. Recently reported transversal electrical measurements for the sample implanted with the $10^{15}$ cm$^{-2}$ dose show a progressive rectifying electrical effect when temperature decreases, in complete agreement with the previous sheet resistance and Hall effect measurements [8,9].

Fig. 4a and b show the current–voltage (I–V) electrical characteristic for the samples implanted with $10^{13}$, $10^{14}$, $10^{15}$ and $10^{16}$ cm$^{-2}$ doses measured at room temperature and 100 K, respectively. The I–V characteristic of the Si substrate at room temperature has been added also for comparative purposes. As it has been mentioned in the experimental, the back contact has been $n^+$ doped to obtain an ohmic contact with very low resistivity. Hence, the transversal I–V measurements at RT would take into account the series resistance of the Si substrate, the Ti implanted layer and the front contact. At room temperature, samples implanted with concentrations above the Mott insulator to metallic-IB limit exhibit an ohmic behavior, while samples with Ti concentrations below this limit and the Si substrate present a clear rectifying effect. The ohmic behavior observed in reverse bias for the bilayer with Ti concentrations above the Mott limit is in agreement with the electrical coupling of the Ti implanted-Si substrate bilayer at high temperature [8]. From an estimation of the silicon substrate resistance from the slope of the Fig. 4a in forward bias, a resistance of 330 $\Omega$ is obtained. Calculating the silicon substrate resistance with a 1.5 mm contact diameter and the silicon carrier concentration and mobility at room temperature, a value of 365 $\Omega$ is obtained. This indicates a good agreement between the van der Pauw measurements and transversal electrical measurements. As can be seen also in the Fig. 4a, the sample of $10^{16}$ cm$^{-2}$ present a slope in forward bias practically equal to the silicon substrate. It points out that the Ti implanted layer in this sample has a very low resistance as it would correspond to a semi-metallic layer and that an ohmic contact with negligible resistance has formed where the only current restriction is due to the substrate resistance. On the contrary, for the Ti implanted sample with a dose of $10^{15}$ cm$^{-2}$, discrepancies between the resistance obtained from the slope of the figure 4a (1 k$\Omega$) and the van der Pauw measurements at room temperature (365 $\Omega$) are obtained. This reveals an increase of the resistance in the transversal measurements that could be originated in the imperfect junction between the Ti implanted layer and the silicon substrate since the depth of the PLM annealing at 0.8 J/cm$^2$ for this sample it was slightly lower than the Ti implanted region [12]. As it is revealed after, a poor ohmic contact, can be discarded for this sample.

Fig. 4b shows the I–V characteristics measured at 100 K for all the implanted samples. At this temperature, for the samples implanted at $10^{15}$ and $10^{16}$ cm$^{-2}$, the electrical decoupling effect is observed in the sheet resistance and the Hall mobility measurements (Figs. 2 and 3) and it is expected in I–V measurements rectifying behavior. Indeed, the rectifying behavior is observed for all the samples at this temperature, but there is a strong difference in the magnitude of the reverse current intensity between the samples with concentrations below and above the Mott limit. For the samples with Ti concentrations above the Mott limit, the electrical rectifying function is attributed to the electrical decoupling effect between the implanted layer and the substrate due to IB formation, corroborating the results obtained from the van der Pauw measurements. As it has been seen, for the samples with Ti concentration below the Mott limit, the sheet resistance and the Hall mobility measurements do not exhibit a decoupling effect suggesting that the IB is not formed in these samples. Since the I–V measurements for these samples show a rectifying effect, we could assume that a Schottky contact had formed. The saturation current
associated to Schottky contacts is given by [17]

\[ I_0 = \frac{S}{A} T^2 \exp(-\phi_B/kT) \]  

(1)

where \( S \) is the area of contact, \( A \) is the Richardson constant, and \( \phi_B \) is the metal-semiconductor barrier height. A simple calculation of the ratio of reverse currents at the measured temperatures considering \( \phi_B = 0.7 \text{ eV} \) for metallic aluminum Schottky contacts [20], gives \( I_0/(300 \text{ K})/I_0/(100 \text{ K}) = 10^{24} \). Since \( I_0/(300 \text{ K}) \) is in the order of milliamperes to microamperes, the \( I_0/(100 \text{ K}) \) is clearly below our experimental setup resolution. This can be observed in the inset of the Fig. 4b where a detail of the \( I-V \) measurements of the samples implanted with \( 10^{13} \) and \( 10^{14} \text{ cm}^{-2} \) is compared with the signal of the measurement system in open circuit. In this inset, the signal of these low concentration Ti implanted samples in reverse bias is comparable to the measurement obtained in the order of nA from a measure in open circuit. Conversely, measurable \( I_0 \) reverse currents are observed for the samples with Ti concentration above the Mott limit, pointing out to a different type of electrical contact than Schottky junction. Hence the ohmic contact observed at RT for the samples with Ti concentration above the Mott limit, together with the measurable \( I_0 \) in reverse bias due to the electrical decoupling at 100 K, are a strong electrical evidence of a metallic-IB formation in the ultraheavily Ti implanted Si layers.

Finally, the analytical two layer model for the sheet resistance also considers the \( V/\Delta V \) relation in the van der Pauw setup [11], where \( V \) is the voltage developed at the current source when \( \Delta V \) is the voltage difference generated in the opposite corners of the van der Pauw setup, as is represented in the scheme of the inset in Fig. 5. For a single layer it can be obtained that the \( V/\Delta V \) relation should be equal to \( \pi \eta \ln 2 \) in the case of ohmic contacts with low resistance [11]. The \( \eta \) factor takes into account the geometrical relation between the electrode size and the sample size. For the samples analyzed in this work \( \eta \approx 2 \) [11]. In this analytical two layer model, if the IB is formed in the layer with the evaporated metallic contacts, \( V/\Delta V \) goes to \( \pi \eta \ln 2 \) when the bilayer is electrically coupled at high temperature or completely decoupled at very low temperatures. During the electrical decoupling effect, attributed to the IB presence, the \( V/\Delta V \) function has a maximum, deviating from the single layer expected behavior.

Fig. 5 shows the \( V/\Delta V \) relation as function of temperature (100–300 K) for the silicon substrate and the samples implanted with Ti doses of \( 10^{13}, 10^{14}, 10^{15} \) and \( 10^{16} \text{ cm}^{-2} \) after PLM. The reference \( 2\pi \ln 2 \) value is also shown. The samples implanted with Ti doses of \( 10^{15} \) and \( 10^{16} \text{ cm}^{-2} \) present the electrical behavior for the \( V/\Delta V \) function predicted by the analytical model for a bilayer considering that the IB is formed [11], in agreement with the decoupling electrical behavior observed in the sheet resistance and the Hall mobility measurements. On the other hand, for the Si substrate, and the samples implanted with doses of \( 10^{13} \) and \( 10^{14} \text{ cm}^{-2} \), the \( V/\Delta V \) function increases as temperature decreases, without reaching a maximum in the function. This is different to the behavior predicted by the model assuming that ohmic contacts are formed. These samples did not show the electrical decoupling behavior associated to IB formation. Therefore a single layer behavior with a constant value of \( V/\Delta V = 2\pi \ln 2 \) is expected. This disagreement between theoretical and experimental results points out that non ohmic contacts are obtained for the Si substrate and the samples impurified with Ti concentrations below the Mott metallic-IB limit formation at low temperatures. We have analyzed also the behavior of the \( V/\Delta V \) function for a non implanted sample but PLM annealed. For the sake of clarity the results for this sample is not shown in the figure. The \( V/\Delta V \) function of this sample has a similar trend than Si substrate. This indicates that a non ohmic behavior is formed and rules out the possibility of PLM creates donor type vacancies which would produce the ohmic behavior in the samples impurified with Ti concentrations above the Mott limit. The Al ohmic contact behavior observed for the samples implanted with Ti doses of \( 10^{15} \) and \( 10^{16} \text{ cm}^{-2} \) seems to indicate that a different kind of electrical contacts are obtained when the IB is formed, suggesting Schottky or ohmic electrical contacts depending on the Ti concentration. These results are in agreement with the previous \( I-V \) transversal electrical measurements, indicating that Ti concentrations below the Mott limit produce metal-semiconductor Schottky contacts and Ti concentrations above the Mott limit produce excellent ohmic contacts. Differences observed in the \( V/\Delta V \) function between the Si substrate and the samples implanted with \( 10^{13} \) and \( 10^{14} \text{ cm}^{-2} \) can be attributed to differences of the resistance associated to the Schottky contact, as Ti implanted dose is increased.

The measurements of the \( V/\Delta V \) relation confirms the transversal \( I-V \) electrical measurements indicating that the Al contact forms an ohmic or a Schottky rectifying contact with the Ti implanted layer when the Ti concentration is above or below the insulator to metallic-IB limit, respectively. This also indicates the metallic behavior of the IB in the Ti implanted Si layers that have Ti concentrations above the Mott transition.

4. Conclusions

In this study, we observe the insulator to metallic-IB transition in ultraheavily Ti implanted Si layers by means of the van der Pauw and transversal \( I-V \) electrical measurements. We show that the electrical decoupling behavior associated to IB formation can be only achieved with implanted doses of \( 10^{15} \text{ cm}^{-2} \) or higher, while for samples implanted with lower doses the measured electrical properties are the same as in the unimplanted reference. We find that insulator to metallic-IB transition occurs for Ti concentrations about \( 10^{19} \text{ cm}^{-3} \) in good agreement with the theoretical Mott limit previously calculated. The measurements of the \( V/\Delta V \) relation reveals that deposited aluminum in the corners forms ohmic contacts for the samples with Ti concentrations above the insulator metallic-IB Mott limit and Schottky rectifying contacts for the samples with Ti concentrations below this limit. These results are in completely agreement with the transversal \( I-V \) electrical measurements pointing out the metallic character of the IB. All these results open a new route to identify intermediate band materials.
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