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Characterization of grain boundaries in multicrystalline silicon with high lateral resolution using conductive atomic force microscopy

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In this work, the electrical characteristics of grain boundaries (GBs) in multicrystalline silicon with and without iron contamination are analyzed by fixed voltage current maps and local I/V curves using conductive AFM (cAFM). I/V characteristics reveal the formation of a Schottky contact between the AFM tip and the sample surface. The impact of both, the polarity of the applied voltage and the illumination by the AFM laser on the behavior of GBs was analyzed systematically. Depending on the polarity of the applied voltage and the iron content of the sample, grain boundaries alter significantly the recorded current flow compared to the surrounding material. The results also show a clear influence of the AFM laser light on the electrical behavior of the grain boundaries. Conductive AFM measurements are furthermore compared to data obtained by electron beam induced current (EBIC), indicating that cAFM provides complimentary information.

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INTRODUCTION

Multicrystalline silicon wafers fabricated from directional solidified silicon ingots have become a widely used material for photovoltaic applications.¹ However, such material contains electrically active defects like dislocations, grain boundaries, or impurity atoms which act as recombination centers and are, therefore, detrimental to the minority carrier lifetime and thus to the efficiency of the solar cell.² To be able to increase the efficiency of multicrystalline solar cells, the characteristics of such recombination centers (e.g., grain boundaries) have to be investigated thoroughly. Several research groups have already contributed to this topic. For example, the behavior of electrically active defects in silicon was investigated by, e.g., Martinuzzi,³ McHugo *et al.*,⁴ and Buonassisi *et al.*^{5,6} Fundamental papers and overviews concerning structure and electrical properties of grain boundaries in silicon have been published by, e.g., Seto,⁷ Grovener,⁸ and Seager.⁹

One common method to investigate the electrical activity of grain boundaries is the electron beam induced current (EBIC) technique which allows to detect recombination active defects.^{10,11} A promising approach to gain information on the properties of grain boundaries could be the characterization using scanning probe microscopy (SPM) techniques, e.g., conductive atomic force microscopy (cAFM).¹² AFM does not only provide the possibility of submicron resolution but the measurements can also be performed under ambient conditions. Several research groups (e.g., Rezek *et al.*,¹³ Azulay *et al.*,¹⁴ and Shen *et al.*¹⁵) have already used conductive atomic force microscopy (cAFM) to analyze the electrical properties of grain boundaries in hydrogenated microcrystalline silicon grown by chemical vapor deposition (CVD).

In contrast to those studies, this work examines multicrystalline substrates grown in a laboratory scale gradient

freeze furnace.¹⁶ Two different samples were analyzed by cAFM: One sample was grown from a melt intentionally contaminated with 400 ppm of iron; the other one was grown from a melt without intentional contamination. Furthermore, the influence of both, the polarity of the applied voltage and the illumination by the AFM laser was investigated systematically. To our knowledge, this has not been presented in the literature to this extend before.

EXPERIMENTAL

Sample preparation

Samples investigated in this study were obtained from multicrystalline silicon ingots with a diameter of 10 cm and a length of 6 cm which were grown by directional solidification. Electronic grade silicon was melted at 1440 °C in a silica crucible coated with Si₃N₄. The growth process was performed under argon atmosphere at a pressure of 500 mbar. To investigate the influence of iron contamination on the electrical behavior of the grain boundaries and the overall material, one ingot was grown from silicon melt intentionally contaminated with 400 ppm of iron. Since the melting point of iron is at 1535 °C,¹⁷ the contaminant was added in the form of FeSi₂ whose melting point is already reached at 1220 °C.¹⁸ Apart from the FeSi₂, no further contaminants or dopants were added to the silicon intentionally. However, a certain amount of boron is typically present in silicon ingots due to the high segregation of this element.¹⁹ In the following, the two samples investigated in this study are referred to as 0 ppm and 400 ppm, respectively, indicating the amount of iron added to the melt they were grown from.

Vertical slices with a thickness of around 1 mm were cut out of the grown ingots parallel to the growth direction and

polished afterwards in order to minimize their surface topography. This should prevent the occurrence of artifacts during the cAFM measurements. Since the maximum measurement area of the AFM used in this work is at $100 \times 100 \mu\text{m}^2$, the position of grain boundaries must be determined accurately to allow a locally resolved characterization of individual grain boundaries using cAFM in a reasonable amount of time. After polishing, however, the grain structure was not visible at the sample surface anymore. This makes it more or less impossible to position the AFM tip at a grain boundary using the optical microscope of the AFM. Therefore as a first preparation step, samples of approximately $1 \times 1 \text{ cm}^2$ were cut out of the silicon slices and characterized by EBIC. With use of the program "IMAGEJ,"²⁰ the geometrical dimensions of the silicon samples were merged with the EBIC images allowing to calculate the distance of different grain boundaries from a given reference point on the sample (e.g., one of the sample edges). This information was finally used for positioning the AFM tip on the samples. Furthermore, the EBIC results were correlated with the cAFM maps to compare the information provided by the two measurement techniques.

The average diameter of the columnar grains in the samples was determined to be around 5 mm. Since the thickness of the silicon specimens lies at about 1 mm, it is likely that the grain boundaries observed penetrate the samples from front to back. Nevertheless, it is possible that the grain boundaries do not proceed through the sample in vertical direction. This might lead to a lateral enlargement of the grain boundaries electrical signal recorded by the cAFM module.

In order to get a rough estimation of the iron content in the solidified 400 ppm sample, the concentration of iron in silicon was calculated by Scheil's equation.²¹ The calculations resulted in an average iron concentration of $2 \times 10^{14} \text{ cm}^{-3}$. For reference measurement purposes, standard monocrystalline silicon wafers with known resistivity and conductivity type (n-type: 2–6 Ωcm ; p-type: 5–30 Ωcm) were prepared.

AFM measurements

All AFM measurements were performed in ambient atmosphere using a Dimension ICON AFM by Bruker AXS. The microscope is equipped with a Nanoscope V controller and a "closed-loop" scanner which ensures high precision positioning of the probe. For electrical measurements, a DC voltage can be applied to the chuck while the probe is at ground potential. The resulting current is registered by a sensor that has to be attached to the AFM measurement head before the start of a cAFM measurement. The sensor can detect currents up to a limit of about 460 nA with an offset current of around 450 pA. All results presented in this study were corrected for the offset. During cAFM measurements, the microscope simultaneously records a map of the current flow through the sample and the topography of the measurement area. In addition, it is also possible to record local I/V characteristics. Thereto the probe remains in a fixed position and a defined voltage ramp is applied to the chuck. To simplify the recording of multiple I/V curves, the "NANOSCOPE"

software (Version 8.1) allows the definition of arrays that consist of an arbitrary number of measurement points with a defined distance between the points. During the actual measurement process the positioning of the AFM tip is done automatically for each point. Since the tip is lifted during the movement from one point of the array to the next, no additional wear out of the tip should occur.

To determine the impact of the AFM laser (GaAs, $\lambda = 670 \text{ nm}$) on the electrical characterization, so-called "dark lift" measurements were performed. Normally, the laser is used for the detection of the cantilever deflection which is the basis for the determination of the sample topography. Since the laser wavelength is suitable for generating additional carriers, the illumination might have an impact on the recorded current flow during cAFM measurements. During a "dark lift" measurement, two current maps are recorded simultaneously by scanning each line of the image twice in trace and retrace direction whereby the laser is deactivated during the second scan. Similarly local I/V measurements with the laser turned off are referred to as "dark ramp" measurements.

All cAFM measurements were performed in contact mode using conductive AFM probes (EFM-100) from NanoWorld AG, Switzerland, coated with Pt-Ir and a nominal tip radius of 25 nm. The work function of conductive silicon AFM probes with Pt-Ir coating is reported to be in the range of 5.3 eV to 5.5 eV.^{22–24} According to theory the lateral resolution of cAFM is governed by the contact area between AFM tip and sample surface.²⁵ Results published by Frammelsberger *et al.*²⁶ indicate that the contact area of EFM-100 tips can vary between 20 and 800 nm² depending on the contact force.

RESULTS AND DISCUSSION

The following presentation of the experimental results is divided into three parts. First, local I/V characteristics recorded on grains, in sufficient distance from grain boundaries are discussed and compared to results obtained on the monocrystalline reference samples. These measurements were used to determine the basic characteristics of the measurement setup and of the grown multicrystalline samples. Second, the current flow at grain boundaries is examined by current maps. The main emphasis is on investigating the electrical characteristics of the GBs with respect to the polarity of the applied voltage and the illumination by the AFM laser. Third, results of local I/V measurements at and near grain boundaries are presented and compared to results of the current maps. Every statement concerning an "increased" or "reduced" current flow refers to the absolute value of the current. If not stated otherwise, all AFM measurements were performed on areas of $50 \times 50 \mu\text{m}^2$.

Local I/V characteristics on grains of multicrystalline silicon samples and on monocrystalline silicon reference samples

As a reference local I/V curves with and without illumination by the AFM laser were recorded on standard 150 mm monocrystalline silicon wafers. The collected results were on

the one hand used to gain information about the basic characteristics of the measurement setup and served on the other hand as comparison for the laboratory-grown multicrystalline samples. Average values of several (25) I/V measurements performed with (“bright”) and without (“dark”) illumination are shown for a n-type (see Fig. 1(a)) and for a p-type (see Fig. 1(b)) monocrystalline reference sample. The averaging was performed to obtain a representative I/V curve and to present the results more clearly. As it is well known, local I/V curves recorded by cAFM at different locations of a sample exhibit a rather large spread due to the variation in contact resistance between the tip and the sample surface. However, the averaged values can be considered as representative for the given sample area.

The results of the “dark” measurements (with the laser turned off) on both samples reveal a clearly rectifying behavior. This fact leads to the assumption that a Schottky barrier is formed between the tip and the surface of the samples. Rezek *et al.*¹³ also report on the occurrence of a rectifying metal-semiconductor contact during their cAFM measurements on hydrogenated microcrystalline silicon. The results given here show that n- or p-type substrates can be clearly distinguished based on the recorded “dark” I/V curves by observing the polarity of the voltage applied in the forward bias regime. Since the measurement voltage is applied at the

chuck of the AFM, the Schottky contact on n-type samples is forward biased for negative measurement voltages (for p-type samples the behavior is vice versa).

Measurements performed under illumination of the laser exhibit a substantially increased reverse current, leading to a soft breakdown of the Schottky contact. Although several possible current transport mechanisms were taken into consideration (e.g., field induced barrier lowering, tunneling through the barrier, generation of photoelectric current), the reason for the observed current increase could not be unambiguously identified. Numerical simulations might lead to a better understanding of the observed effects.

Local I/V curves recorded on multicrystalline samples exhibit a very similar behavior. Figures 1(c) and 1(d) show the average values of 25 measurements performed with (“bright”) and without (“dark”) illumination on the 0 ppm (see Fig. 1(c)) and on the 400 ppm (see Fig. 1(d)) sample. As can be seen from the I/V characteristics, the 0 ppm sample showed n-type behavior, whereas the 400 ppm sample revealed p-type behavior, respectively. Further analysis of the grown ingots performed by Azizi *et al.*,¹⁶ suggest that the n-type conduction detected for the 0 ppm sample is caused by the generation of thermal donors during the cooling of the ingots. The presence of iron in the ingot seems to prevent the generation of the thermal donors, thus leading to

FIG. 1. Average values of 25 local I/V measurements recorded with (red squares) and without (dark triangles) illumination. (a) n-type reference sample. (b) p-type reference sample. (c) 0 ppm sample. (d) 400 ppm sample.

the expected p-type behavior of the 400 ppm sample (caused by boron present in the ingot).

For all samples, local oxidation could be observed during the measurements regardless of the polarity of the applied voltage. The oxidation process leads to a significant change in current flow, thus, resulting in rather poor repeatability of consecutive I/V measurements performed at the same position. The phenomenon of anodic oxidation occurring when the sample is positively biased with respect to the AFM tip is well described in many publications (e.g., Dagata *et al.*,²⁷ Gordon *et al.*,²⁸ and Stiévenard *et al.*²⁹). As mentioned before, oxidation was also discovered with the sample negatively biased with respect to the tip. Abadal *et al.*³⁰ who also observed oxidation for such conditions, explained this effect by the formation of H_2O_2 at the AFM tip.

Electrical behavior of grain boundaries

First, an intersection of multiple grain boundaries on the 400 ppm sample was investigated. Figure 2 shows topography (Fig. 2(a)), EBIC image (Fig. 2(b)), (different scale),

and current maps recorded with (Fig. 2(c)) and without (Fig. 2(d)) illumination by the laser. Since the cAFM measurement was performed with a negative voltage (-2V) applied to the chuck and the 400 ppm sample showed a “p-type” behavior, the Schottky contact between AFM tip and sample was reverse biased. The grain boundaries visible in the EBIC image (see Fig. 2(b)) can clearly be recognized in the current map recorded under illumination (see Fig. 2(c)). The current map shows that a significantly higher reverse current was recorded at the grain boundaries compared to the surrounding material. The topography image (see Fig. 2(a)) shows, however, no noticeable surface roughness variation, that could be responsible for the enhanced current flow, thus leading to the conclusion that the effect is related to a change in material properties. In contrast, the signal visible in the lower left of Figures 2(a), 2(c), and 2(d) (several line type features, marked by a white square) seems to be due to contamination on the sample surface since it is visible in the topography and the current maps. In the current maps, the detected width of the grain boundaries appears to be up to

FIG. 2. Intersection of multiple grain boundaries on the 400 ppm sample. (a) Topography map showing surface contamination (white square). (b) EBIC image, approximately $500 \times 500 \mu\text{m}^2$, red square indicates approximately cAFM measurement area. (c) Current map recorded with illumination by the laser, measurement voltage: -2V (reverse bias condition). (d) Current map recorded without illumination by the laser, measurement voltage: -2V (reverse bias condition).

FIG. 3. Current maps of an area slightly above the intersection of grain boundaries on the 400 ppm sample shown in Figs. 2(a)–2(d). (a) Current map recorded with illumination by the laser, measurement voltage: 0 V. (b) Current map recorded without illumination by the laser, measurement voltage: 0 V.

2 μm , exceeding the geometric width of silicon grain boundaries that is reported to be around 1 nm or several lattice constants.^{8,31} The cAFM results suggest, therefore, that the electrically relevant width of the GBs is larger than their geometric dimensions. Oualid *et al.*³² and Palm³³ present results of light beam induced current (LBIC) and EBIC measurements where grain boundaries influence the electrical signal for up to several tens of micrometers.

A comparison of the current maps shown in Figures 2(c) and 2(d) reveals the significant impact of the AFM laser on the appearance of the grain boundaries. Similar to the local I/V measurements presented before, the overall current flow in reverse bias conditions through the sample is strongly reduced without illumination by the laser (see Figs. 2(c) and 2(d); note the different current scaling). Furthermore, only the grain boundary on the left hand side of the intersection is still visible in the current map recorded without illumination, whereas the boundaries to the right show no enhanced current flow anymore. One possible explanation for this observation might be a locally increased photogeneration of carriers along the grain boundaries based on the photovoltaic impurity effect described by Wolf.³⁴ Shen *et al.*¹⁵ observed a similar effect in their study of microcrystalline solar cells by cAFM. The additionally generated carriers could be separated in the depletion region of the Schottky contact formed between the AFM tip and the sample surface, thus causing a locally enhanced reverse current flow.

For further investigation, cAFM measurements with zero bias were performed on the 400 ppm sample. Figure 3 shows corresponding current maps with (a) and without (b) illumination recorded at an area slightly above the intersection of multiple grain boundaries shown in Figure 2. As can be seen in Figure 3(a), the grain boundary is still visible under illumination and exhibits a negative current flow of around -100 pA which could result from a separation of generated carriers in the depletion region of the Schottky contact. This would support the assumption that increased generation of carriers takes place at the grain boundary.

To investigate the electrical behavior under forward bias conditions, measurements with a positive voltage of 1 V at the chuck were performed. Figure 4 illustrates the current flow at the same intersection as shown in Figure 2 with (a) and without (b) illumination by the laser. In contrast to the measurements with the reverse biased Schottky contact, the grain boundaries exhibit a reduced current flow compared to the surrounding material (e.g., with laser turned off, GBs: around 0–50 pA; Grains: around 200–300 pA). In this case, the AFM laser seems to have an opposite effect on the current flow at the grain boundaries compared to the results obtained under reverse bias conditions. With the laser turned on, only the grain boundary reaching from the intersection to the lower left can be detected in the current image (see Fig. 4(a)). When the laser is switched off, all grain boundaries are visible and the boundary to the left appears more pronounced in the current map (see Fig. 4(b)). The reduced current flow at the GBs could be related to an increased recombination.

Comparing the EBIC image with the current maps recorded on the 400 ppm sample reveals some major differences. First of all, one can observe a remarkable difference between EBIC and cAFM measurements concerning the intensity of the recorded current signal at the grain boundaries. As expected, the EBIC results show a reduced current flow at the grain boundaries that can be attributed to locally increased carrier recombination. However, as stated above, cAFM measurements reveal an enhanced photogeneration at the grain boundaries which seems to overcompensate the expected recombination. This interesting contrast between both measurement techniques could be due to the different interaction mechanisms between the beam responsible for carrier generation (cAFM: laser light, EBIC: electron beam) and the silicon samples. Nevertheless, further investigation is necessary before this issue can be addressed properly.

Looking at the cAFM current maps recorded at the grain boundary intersection, one can determine two more differences regarding the EBIC measurement. On the one hand, only the grain boundary on the left side of the intersection is

FIG. 4. Intersection of multiple grain boundaries on the 400 ppm sample (same area of the sample as in Fig. 5). (a) Current map recorded with illumination by the laser, measurement voltage: 1 V (forward bias condition). (b) Current map recorded without illumination by the laser, measurement voltage: 1 V (forward bias condition).

visible in each cAFM current map (see Figs. 2(c) and 2(d) and Figs. 4(a) and 4(b), respectively). In contrast, the EBIC image (see Fig. 2(b)) shows no significant difference concerning the electrical activity of the GBs. On the other hand, the neighboring grains exhibit a pronounced contrast in the EBIC image; whereas no difference can be detected looking at the cAFM current maps. The strong contrast seen in the EBIC might be due to different amount of iron incorporated in both grains, whereas a contact problem seems rather unlikely since the preparation process (i.e., evaporation of metal contacts) is well established.

The results on the 0 ppm sample (without intentional iron contamination) reveal a different electrical behavior of the grain boundaries. Figure 5 shows two current maps of a grain boundary segment on the 0 ppm sample recorded under illumination with a forward (a), (-2 V) and a reverse (b), (2 V; note the different scaling) biased Schottky contact. Most parts of the depicted grain boundary exhibit a reduced current flow compared to their surrounding grains regardless of the polarity of the applied voltage. Under forward bias conditions (negative voltages applied), the current flow at the grain boundary ranges from -3 nA to -10 nA, whereas the current at the surrounding grains ranges from -30 nA to -60 nA. For reverse bias conditions, the current values at the GB range from 2 nA to 4 nA, the current at the surrounding material is between 5 nA and 8 nA.

However, under forward bias conditions, the grain boundary appears wider, more uniform and “frayed” at its right hand side (see Fig. 5(a); except for the segment between “C” and “D.” The segment between “C” and “D” shown in Figure 5(a) deviates from the behavior observed elsewhere as the electrical characteristics of the grain boundary seem to fundamentally change for a distance of approximately $40\text{ }\mu\text{m}$. The grain boundary signal nearly disappears in the segment between “C” and “D,” leaving just a hint of a locally increased current flow (see Fig. 5(a)). In contrast to that, the grain boundary exhibits a reduced current flow

throughout the whole segment under reverse bias conditions and appears to be slightly more pronounced and broader where it has nearly vanished before (see Fig. 5(b), note the different current scaling). Moreover, a distinct difference concerning the current flow in the two neighboring grains becomes apparent in the reverse bias regime. The average current flow through the grain to the left of the grain boundary is about 1.5 times higher than the average current flow recorded for the grain to the right of the grain boundary.

The change in the electrical behavior of the grain boundary between “C” and “D” (see Fig. 5) is also visible in the corresponding EBIC image (see Fig. 6). The electrical activity of the grain boundary seems to be reduced in the area of interest. According to the EBIC image, one could also speculate that the grain boundary is intersected by twin boundaries coming from the upper right. This intersection might change the structure of the grain boundary and, therefore, its electrical properties as reported by Sarau *et al.*³⁵ for silicon or Russel *et al.*³⁶ for zinc selenide. Jiang³⁷ describes similar results in his work about the characterization of silicon grain boundaries by scanning capacitance microscopy (SCM). According to his investigations, the behavior of grain boundaries is strongly dependent on their structure and can vary even for boundaries that are located between the same two grains. Furthermore, except of the segment between “C” and “D,” the grain to the right of the grain boundary appears darker in the EBIC image (see Fig. 6) than its neighbor to the left. This might correlate with the slight difference concerning the average current flow through both grains recorded with the cAFM for reverse bias conditions (see Fig. 5(b)).

The impact of the illumination by the laser was investigated performing “dark lift” measurements at further sections of the same grain boundary on the 0 ppm sample. Figure 7 shows current maps recorded with (a) and (c) and without (b) and (d) illumination by the laser under forward (see Figs. 7(a) and 7(b)) and reverse (see Figs. 7(c) and 7(d)) bias conditions. With the Schottky contact forward biased

FIG. 5. Grain boundary segment on the 0 ppm sample. Both figures are combined out of two separate but adjacent measurements of $50 \times 50 \mu\text{m}^2$. All measurements were performed with illumination by the laser. (a) Current map recorded with measurement voltage: -2 V (forward bias condition). (b) Current map recorded with measurement voltage: 2 V (reverse bias condition).

(-2V), switching off the laser leads to a broadening of the grain boundaries signal (see Figs. 7(a) and 7(b), respectively). When a positive voltage of 2 V (reverse bias conditions) is applied to the chuck, the grain boundary is hardly visible with the laser turned off. The overall current flow through the sample is strongly reduced compared to the results recorded with illumination by the laser shown in Figure 7(c). As a consequence, the contrast between the grain boundary and the surrounding material becomes very low for a reverse voltage of 2 V . The current map shown in Figure 7(d) was, therefore, recorded with a reverse voltage of 4 V , thus enhancing the contrast between GB and the surrounding grains and making the grain boundary slightly visible again. Note, that the current maps in Figures 7(c) and 7(d) were recorded on different areas of the same grain boundary.

The comparison of the different results obtained on the 0 ppm and 400 ppm sample leads to the assumption that the

contamination of silicon with iron changes the electrical behavior of grain boundaries. Bounassisi *et al.*^{5,6} reported that iron rich clusters are not only present at structural defects along grain boundaries in silicon, but also substantially enhance the electrical activity of these GBs. Based on the results presented in this study, one might speculate that a sufficient amount of iron segregated at the grain boundaries could cause an enhanced generation of carriers that might be related to the photovoltaic impurity effect described by Wolf.³⁴

Local I/V characteristics at and near grain boundaries

In addition to the current maps shown above, local I/V curves were recorded at and near grain boundaries. For this purpose, arrays of 10×10 measuring points with a step

FIG. 6. EBIC image (approximately $500 \times 500 \mu\text{m}^2$) of grain boundary on the 0 ppm sample. The red square indicates the approximate cAFM measurement area shown in Fig. 5.

distance of 500 nm in X- and Y-direction were defined and I/V curves were recorded at each point. The applied voltage ramps ranged from -5 V to 5 V . Local oxidation at every point of the measurement arrays could be observed in current

maps that were recorded after completion of a measurement series (see, e.g., Fig. 8(a)).

For further analysis, measured currents of the local I/V curves were plotted as a function of the coordinates of their particular measurement point. Figure 8(a) shows the current map recorded by cAFM and Figure 8(b) a 3D plot of the maximum reverse currents. The measurements were performed on the 400 ppm sample under illumination by the laser. As Figure 8(b) clearly shows, the grain boundary can be easily identified based on the results of the local I/V characteristics. The reverse currents at the boundary exceed those in the surrounding material, thus, confirming the results of the current maps. In contrast, the grain boundary investigated on the 0 ppm sample could not be clearly reconstructed based on local I/V curves. This might be due to a smaller difference concerning the current flow at and near the grain boundary.

Concerning the consistency of the results from local I/V measurements and current maps, a comparison between both measurement modes was performed under illumination by the laser. Current maps for three different voltages (-2 V , -3 V , -4 V) were recorded at a grain boundary on the 400 ppm sample and the average current values at the boundary and both, from the grains to its left and its right were extracted. To avoid influences due to the local oxidation, the three maps were recorded at three neighboring segments of

FIG. 7. Grain boundary segments on the 0 ppm sample for different bias conditions. (a) Current map recorded with illumination by the laser, measurement voltage: -2 V (forward bias condition). (b) Current map recorded without illumination by the laser, measurement voltage: -2 V (forward bias condition). (c) Current map recorded with illumination by the laser, measurement voltage: 2 V (reverse bias condition). (d) Current map recorded without illumination by the laser, measurement voltage: 4 V (reverse bias condition) [Note: Different area of grain boundary depicted in figures (c) and (d)].

the GB. The average values of the currents were extracted using the “NANOSCOPE ANALYSIS” software (Version 1.2). These values were compared with local I/V curves (also shown in Fig. 8), which were recorded at the same grain boundary. Depending on the position of their measurement points, the I/V curves were defined to be recorded at the grain boundary, to its left or to its right. Average values for each of these three categories were calculated for every row of the measurement array. The results of the local I/V measurements were plotted together with the average values extracted from the cAFM maps (see Figure 9). The lines connecting the average values extracted from the cAFM maps serve as guide for the eye only. Looking at Figure 9, it becomes obvious that the results of local I/V measurements are in good agreement with the average values extracted from the current maps.

FIG. 8. Grain boundary on the 400ppm sample. (a) Current map of $5 \times 5 \mu\text{m}^2$ recorded with a measurement voltage of -1 V (reverse bias condition) after the measurement of 100 local I/V curves; the measuring points are visible as low current spots because of local oxidation during the I/V measurements. (b) Reverse current at -5 V from the local I/V curves, plotted as a function of the measurement position visible in figure (a). All measurements were performed with illumination by the laser.

FIG. 9. Comparison of average current values extracted from cAFM maps (lines serve as guide for the eye) and local I/V measurements (each line shows the average value of multiple measurements), all measurements performed on the 400 ppm sample.

SUMMARY

High resolution electrical characterization of grain boundaries in multicrystalline silicon samples grown by directional solidification with and without intentional iron contamination was performed using conductive AFM in ambient conditions. Recorded I/V characteristics show that a Schottky contact is formed between the AFM tip and the sample surface. Grain boundaries appear either as regions of reduced or enhanced current flow and can, therefore, be clearly detected in current maps. The electrical behavior of grain boundaries depends on the polarity of the applied voltage as well as on the illumination by the AFM laser. Measurements on a sample contaminated with iron suggest that enhanced carrier generation takes place at decorated grain boundaries, leading to a locally enhanced reverse current under illumination by the laser. In contrast, most parts of a grain boundary investigated on a sample without intentional iron contamination showed a reduced current flow regardless of the polarity of the applied voltage and the illumination by the AFM laser. The presented results furthermore proof that grain boundaries which do not exhibit any different behavior in EBIC measurements may indeed lead to different results when investigated by cAFM. This indicates that cAFM measurements at grain boundaries can be used to supplement EBIC results. However, further understanding is needed in this context, since the number of grain boundaries investigated in this study is too small to allow for any systematic relation between their appearance in EBIC and cAFM. The course of grain boundaries can be reconstructed based on local I/V curves recorded at and in the environment of the grain boundaries. Recorded local I/V characteristics are in good agreement with results from current maps.

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