p⁺-doping analysis of laser fired contacts for silicon solar cells by Kelvin probe force microscopy

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Local rear contacts for silicon passivated emitter and rear contact solar cells can be established by point-wise treating an Al layer with laser radiation and thereby establishing an electrical contact between Al and Si bulk through the dielectric passivation layer. In this laser fired contacts (LFC) process, Al can establish a few μm thick p⁺-doped Si region below the metal/Si interface and forms in this way a local back surface field which reduces carrier recombination at the contacts. In this work, the applicability of Kelvin probe force microscopy (KPFM) to the investigation of LFCs considering the p⁺-doping distribution is demonstrated. The method is based on atomic force microscopy and enables the evaluation of the lateral 2D Fermi-level characteristics at sub-micrometer resolution. The distribution of the electrical potential and therefore the local hole concentration in and around the laser fired region can be measured. KPFM is performed on mechanically polished cross-sections of p⁺-doped Si regions formed by the LFC process. The sample preparation is of great importance because the KPFM signal is very surface sensitive. Furthermore, the measurement is responsive to sample illumination and the height of the applied voltage between tip and sample. With other measurement techniques like micro-Raman spectroscopy, electrochemical capacitance-voltage, and energy dispersive X-ray analysis, a high local hole concentration in the range of 10^{19}cm^{-3} is demonstrated in the laser fired region. This provides, in combination with the high spatial resolution of the doping distribution measured by KPFM, a promising approach for microscopic understanding and further optimization of the LFC process. © 2016 AIP Publishing LLC.
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I. INTRODUCTION

The laser fired contacts (LFC) process^{1–3} is an effective method to establish local rear contacts of silicon PERC (passivated emitter and rear contact) solar cells. A full area aluminum layer (deposited by physical vapor deposition, commercial Al foil or screen-printing⁴) is contacted point-wise by laser spots that are fired through the dielectric rear passivation layer to the silicon bulk. In this way, in contrast to other PERC concepts, dielectric layer opening and contact formation are performed in a single process step. Al can thereby establish a p⁺-doped Si region below the LFC. A high doping concentration is desirable for a low contact resistance,⁵ and the formed local back surface field (BSF) is beneficial for reducing carrier recombination at the Si/metal interface. As the solid solubility limit mainly defines the possible doping concentration of an alloyed contact, the addition of B to the Al layer is advantageous because it has a much higher solid solubility in crystalline Si than Al.^{6,7} Therefore, if the Al layer is formed by screen-printing, often pastes containing boron (B) additives are used.⁸ This leads to B-dominated doping and can strongly increase the resulting hole concentration compared with pure Al.⁸

There exist several methods to evaluate doping densities in Si. Electrochemical capacitance-voltage (ECV) allows the measurement of depth profiles of the electrically active

doping concentration. But the measurement spot is in the range of 1mm^2 leading to an area averaged value. Therefore, this method is only capable to determine the doping density of laterally uniform layers and quite large structures.

In contrast, spatially resolved secondary ion mass spectroscopy (SIMS) is applicable to the investigation of LFCs^{9,10} and allows for depth dependent measurements of the chemical concentration of doping atoms. Additionally, for spatially resolved measurements, Raman spectroscopy was recently applied for microscopic characterization of p⁺-doping densities.^{11–14} Its spatial resolution is in the range of $1\mu\text{m}$. Micro-photoluminescence spectroscopy (μPLS) offers about the same spatial resolution with the possibility to measure n-type doping, as well.^{15,16}

For measurements of doped regions with a spatial resolution in the sub-micrometer range, electron beam induced current (EBIC)^{3,17,18} can be used. Using n-type Si substrates, the LFC process results in a p⁺/n junction that can be detected qualitatively. But EBIC has the disadvantage that it requires complex technical equipment including a high-vacuum chamber. In contrast to EBIC, Kelvin probe force microscopy (KPFM) is based on atomic force microscopy (AFM), which requires a less complex setup and has also the advantage of a sub-micrometer resolution.^{19–28}

Aim of this work is to introduce KPFM as a valuable measurement technique for the investigation of the p⁺-doped region formed by the LFC process on Al-coated Si. This method is able to reveal the distribution of the electrical

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FIG. 1. Exemplary optical microscope images of polished cross-sections of LFCs with screen-printed Al paste. (a) After the first laser pulse; (b) after 5 laser pulses; and (c) after 9 laser pulses. The first laser pulses ablate part of the Al layer, then the contact between Al and Si is created leading to a crater in the Si.

potential and therefore the doping concentration in and around the area influenced by the laser.

II. EXPERIMENTAL

A. Sample preparation

Unless otherwise specified, for most of the samples in this investigation, n-type Czochralski (Cz) silicon wafers with a resistivity of $6 \Omega \text{ cm}$ are used. N-type wafers are not the material commonly used for LFC-PERC solar cells but are advantageous for KPFM measurements because in this case the Al doping results in a p^+/n junction. Compared with the usual p^+/p case (see Fig. 3 for results of a p^+/p structure), this leads to a higher potential difference (see, e.g., Fig. 4) and therefore increases the sensitivity of the measurement. However, the formation of the p^+ -doped region is expected to be comparable, which is why in this paper the term LFC is also used for the structures formed by laser treatment of an Al layer on n-type Si.

The wafers are saw-damage-etched in KOH solution and coated with a passivation layer of 75 nm plasma-enhanced chemical vapor deposited (PECVD) $\text{SiN}_x\text{:H}$. On top of the $\text{SiN}_x\text{:H}$, the Al layer with a thickness of about $30 \mu\text{m}$ is formed by screen-printing of a commercially available Al paste containing B additives. The samples are fired in a belt-furnace to form a laterally conducting Al layer. During firing, the $\text{SiN}_x\text{:H}$ acts as a barrier layer preventing full area contact formation between Al and Si. Afterwards, an array of LFCs is formed using a Nd:YAG laser (*Rofin Powerline 100D*) with a wavelength of 1064 nm and pulse duration of about 100 ns. From optical microscope images of the cross-sections of laser treated areas (see Fig. 1), it can be observed that applying this laser system with the parameters used several laser pulses per spot is needed to penetrate the Al layer and forms the Al-Si contact. The energy of the first laser pulse is completely absorbed by the Al layer and some Al is ablated (Fig. 1(a)). Additional laser pulses remove more Al (Fig. 1(b)) until the Si underneath is impacted by further heating up and the $\text{SiN}_x\text{:H}$ layer is opened. The contact and the p^+ -doped region are created at the Al/Si interface. After about 6–9 laser pulses (Fig. 1(c)), a few μm deep crater in the Si is observed. Only the crater edge represents the actual electrical contact between Al and Si.

For the KPFM samples, a two dimensional array with a small pitch of $250 \mu\text{m}$ between the LFCs is used to guarantee

that enough spots are hit during preparation of cross-sections later on.

The samples are subsequently annealed at 350°C . This is done to assure comparability to an LFC-PERC solar cell process in which this process step is used to improve contact resistance and curing of defects. Afterwards, the Al layer is removed in aqueous HCl solution and small parts of the wafers are glued on plastic holders in an angle of 30° to the surface (see Fig. 2) and embedded in epoxy resin. Followed by a grinding and double-phase polishing procedure, this leads to cross-sections of LFCs with a surface roughness of only some nanometers which is necessary for KPFM investigation. Contrary to an angle of 90° leading to a perpendicular cross-section, 30° leads to a stretched appearance of the layers by a factor of 2 increasing spatial resolution. A sketch to illustrate the geometry of the samples is shown in Fig. 2. The polished surfaces include LFCs at different depth positions. Depending on the grinding duration and sample tilt angle, different depths of the LFC crater can be represented. Suitable positions on the sample are selected using optical microscopy.

After performing the KPFM measurements, the cross-section samples can be further investigated by scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDX). Therefore, a 5–10 nm thick conducting layer of gold is sputtered on top of the samples to avoid charging by the electron beam.

B. Kelvin probe force microscopy

KPFM is based on AFM and evaluates the lateral 2D Fermi-level characteristics at sub-micrometer resolution on a given Si substrate. The method was first introduced by Nonnenmacher *et al.* in 1991 (Ref. 19) and later on applied

FIG. 2. Sketch of cross-section (not to scale) showing the geometry of the LFC sample preparation. Left: the Si-wafer with LFCs is embedded in epoxy resin at an angle of 30° , ground, and finally polished. Right: detailed view showing the desired position of an LFC crater at the polished surface.

to the measurement of p/n junctions.^{20–28} The sample preparation is of great importance because the KPFM signal is very surface sensitive.²³ But the KPFM signal is also responsive to sample illumination,^{20,24} tip geometry and tip-sample distance,²² applied tip-sample voltage,²⁵ surface charge, and therefore band bending effects. Hence, a quantitative measurement is not possible in ambient air. However, the spatial distribution of doping can be measured qualitatively with a resolution of around 100 nm. Thus, KPFM is capable to investigate LFCs and its cross-sections with regard to local p⁺-regions or p⁺/n-junctions; the latter is the case in this paper in order to get higher signal strength. KPFM is performed in amplitude modulated (AM) two-pass-mode (lift-mode)²⁷ applied on mechanically polished cross-sections of Si with the above described array of LFCs. The surface topology of each line is measured prior to each KPFM line scan. Afterwards, the Kelvin probe measurement is performed at constant sample-tip distance z . In general, neglecting the other effects mentioned, by applying an electric field, the electric force between the tip on the cantilever and the sample surface is given by²⁶

$$F = \frac{1}{2} \frac{\partial C}{\partial z} \left[\left\{ (V_{\text{ext}} - V_{\text{cpd}})^2 + \frac{1}{2} V_{\text{ac}}^2 \right\} + \left\{ 2(V_{\text{ext}} - V_{\text{cpd}}) V_{\text{ac}} \sin(\omega t) \right\} - \left\{ \frac{1}{2} V_{\text{ac}}^2 \cos(2\omega t) \right\} \right]. \quad (1)$$

V_{ext} represents a constant applied voltage between tip and sample, while V_{ac} is modulated by $\sin(\omega t)$ with ω being the frequency of the electrical excitation and t is the time. V_{cpd} is the potential (difference) between surface and tip being the quantity to be measured. C denotes the tip-sample capacitance.

For investigating the surface potential, the most important of the three terms in Eq. (1) is the second one. It depends on the potential difference between tip and sample multiplied by the applied AC voltage. For the measurement of the potential difference V_{cpd} , the second term is zero by adjusting V_{ext} so that $V_{\text{ext}} = V_{\text{cpd}}$. Therefore, V_{cpd} is indirectly measured by measuring V_{ext} .

The Kelvin probe measurements conducted in this work are performed with an *Asylum research MFP-3D* AFM in ambient air. The resonance frequency of the conducting cantilever is around 72 kHz, and the tip radius is approximately 28 nm. The resolution of the surface and potential mappings is set to 35 nm, and the distance between tip and sample during the potential measurement to the lowest value is possible (usually 40–50 nm below the average distance in AC-mode, which is conducted in 5% repulsive mode). The sample is as close as possible to thermal equilibrium conditions to avoid thermal drift. Scan rates differ from 0.25 to 1 Hz considering the relaxation time τ (around 3 ms) to changes in the amplitude, which is

$$\tau \approx \frac{Q}{f_0}, \quad (2)$$

with Q being the q-factor of the cantilever tip (between 140 and 190) and f_0 is the first mechanical oscillation frequency.

Amplitude images are flattened in 1st order. Unless otherwise specified, the potential data are extracted unmanipulated.²⁸

C. Raman measurements

Raman measurements can be used to detect the local doping density by evaluating the asymmetry of the Si Raman peak at wavenumber 520 cm^{-1} relative to the laser excitation.^{11,12} In this work, Raman measurements are performed with a commercial *WITec alpha300* confocal Raman microscope^{13,14} measuring the back-scattered spectrum. An excitation laser wavelength of 488 nm is used to get a signal from near the surface (excitation depth of $\sim 0.8 \mu\text{m}$). The automatic sample positioning stage allows mappings of specific areas with a laser spot size in the μm range, whereas the diameter of the LFC crater is about $50 \mu\text{m}$.

Instead of the Lorentzian distribution for undoped Si, the characteristic Raman peak shape for highly doped Si is described by the Fano distribution with the Fano parameter q .²⁹ $1/q$ characterizes the peak asymmetry and is proportional to the free hole concentration. To extract absolute values of doping concentration, the calibration is performed by ECV measurements of highly B-doped Si wafers or full area Al-alloyed Si samples.¹⁴

For data evaluation, the Raman spectrum of each measured point is corrected with the device specific offset and normalized. Then, the Fano distribution is fitted to the Si Raman peak. Afterwards, $1/q$ is correlated to the corresponding doping density resulting in a spatial distribution of the hole concentration. From the standard deviation of $1/q$ of 100 measuring points on 3 different homogeneously p-doped Si wafers, the sensitivity of the ECV-calibrated Raman measurement is estimated to be around $1 \times 10^{18} \text{ cm}^{-3}$.

III. RESULTS

A. Application of Kelvin probe force microscopy to LFCs

Polished cross-section samples are measured by AFM and KPFM. The samples feature a very smooth surface in the AFM topography with a variation in height of only a few 100 nm. This height difference occurs in a slightly rounded edge between the Si and the less grinding resistant resin region.

1. Measurement of a p⁺/p junction

As an example for a p⁺/p junction, a p-type Si wafer ($4\text{--}6 \Omega \text{ cm}$) is full-area printed with an Al and B containing paste and fired in a belt furnace. After etching off the Al layer in HCl, a 30° cross-section of the formed p⁺-layer is prepared and measured. The AFM and KPFM results are shown in Fig. 3. In Fig. 3(a), the AFM amplitude reveals the rough resin region on the left side, the slightly rounded edge of the Si wafer, resulting in higher amplitude, and the Si bulk with some scratches and particles on the surface. In Fig. 3(b), the KPFM potential distribution (corrected by a plane fit procedure to make minor differences visible) is depicted in color scale. Blue indicates the bulk p-type Si and green

FIG. 3. AFM and KPFM results of a 30° cross-section sample featuring a p^+ -layer that has been formed by full-area firing of a p-type Si wafer covered with an Al and B containing paste. (a) AFM amplitude image. (b) KPFM potential distribution (corrected by a plane fit procedure to make minor differences visible). Blue represents the bulk p-type Si. The p^+ -region near the wafer surface exhibits a slightly higher potential and appears in green. Red indicates the isolating and therefore charged epoxy resin region.

indicates the p^+ -region near the wafer surface. The green spots are artifacts resulting from particles on the polished surface. Red represents the isolating and therefore charged epoxy resin region. It can be seen that for this KPFM measurement of a p^+/p junction, the potential difference between p- and p^+ -Si is visible, but quite small (20–40 mV). Therefore, for the following investigation of LFCs, n-type Si wafers are chosen to enhance the potential difference of the p^+ -region.

2. Measurement of an LFC on n-Si

LFCs processed with 7 to 10 laser pulses exhibit a crater in the Si wafer which is easy to locate via microscopy. The AFM/KPFM scanning area is defined including the whole crater region of a specific LFC. For the exemplary measurement discussed in this paper, a position on the polished surface comparable to the one sketched by the dashed line in the right part of Fig. 2 was chosen. Fig. 4(a) shows an SEM image (InLens detector) of the investigated LFC structure with a high material contrast between Si and epoxy resin

which fills up the crater and covers the outside of the sample. The bubble-like structure shows most likely oxide residuals of the Al paste which are not completely removed during etching in HCl solution. In Fig. 4(b), the spatially resolved KPFM potential of the same measurement position is presented. Compared with the n-type bulk Si (which appears in blue), the few μm wide area around the crater exhibits a higher potential (green) which represents the expected p^+ -doped region. The isolating epoxy resin appears in yellow and red (high potential). Subsequent measurements of the same sample even raise the contact potential difference of these resin regions because they are additionally charged by each measurement, while the rest of the sample does not accumulate charges from the measurement.

In Fig. 4(b), the vertical green lines are KPFM measurement artifacts resulting in an offset and showing the scanning direction. Green dots on the left side are supposed to be particles on the surface leading to increased potential values. In Fig. 4(c), two exemplary line scans of the KPFM potential are shown in detail (positions marked with arrows in Fig. 4(b)). The values are normalized for better comparability.

FIG. 4. SEM and KPFM results of an LFC 30° cross-section. The measurements are performed on the plane which is sketched by the dashed line in the right part of Fig. 2. (a) SEM image showing the structure of the LFC. The bubbles are possibly oxide residuals of the Al paste. (b) KPFM potential distribution in color scale. Blue indicates the bulk n-type Si, green indicates the p^+ -region around the contact spot, and in yellow and red the isolating and therefore heavily charged epoxy resin region can be seen. (At the spot marked with X, the elemental composition is detected, results shown in Fig. 5(b).) (c) Normalized KPFM potential line scans corresponding to the arrows shown in (b). The potential step in line scan 1 caused by the p^+ -region at the crater edge is clearly visible compared with line scan 2 in an area not influenced by the laser pulse.

FIG. 5. (a) Combined SEM and EDX image of the same LFC cross-section as shown in Fig. 4. The red dots indicate detected Si. The Al concentration is below the EDX detection limit of 1%. (b) Exemplary EDX spectrum of the measurement point marked with X in Fig. 4(b).

The potential step in the middle of line scan 1 demonstrates the p^+ -doped region around the LFC crater. In contrast, line scan 2 next to the LFC shows the direct change from low potential of the n-Si base to high potential at the resin region.

B. Evaluation of additional influences on the KPFM potential

To verify that the region with increased potential around the LFC is p^+ -doped Si and not an Al-rich mixture of Al and Si (e.g., eutectic composition of 87.8% Al and 12.2% Si), EDX measurements are performed in the same scan area as the KPFM measurements before. Fig. 5(a) shows a Si EDX map superimposed with an SEM image of the corresponding region. The image shows that Si is detected in the Si bulk and in the region of interest around the LFC. In Fig. 5(b), an EDX spectrum is shown, taken at the position marked with X in Fig. 4(b). Using an integration period of 60 s, around 95.5 at. % Si and 4.5 at. % Au are detected. The Au results from the necessary conducting layer which avoids sample charging during SEM imaging. The EDX signal for Al is very low or below the detection limit of around 1% in relation to the entire spectrum. This result excludes the possibility of an Al-rich region instead of p^+ -doped Si.

Another possible explanation for the region around the LFC with increased KPFM potential could be a Si region which is molten by the laser and recrystallized with oxygen or nitrogen impurities incorporated from the ambient. To exclude this effect, a similar sample was prepared with the same n-type Si wafer material but no Al layer on top. The following LFC process creates a crater with possible crystal damage but without the possibility of incorporated Al or B. This sample does not show any increased KPFM signal near the crater edge region, and it can be concluded that laser damage does not influence the KPFM measurement.

C. Combination of KPFM with ECV calibrated Raman measurements

In former investigations concerning LFCs, quantitative Raman measurements were performed.¹⁴ For combination of KPFM and Raman, ideally the same sample is scanned over the whole LFC area. However, this is challenging for the embedded cross-section samples because the Raman excitation laser damages the epoxy resin which may influence the measurement even of the surrounding region. Therefore, single point Raman measurements are performed at selected spots

of the LFC which are easy to locate via microscope imaging and are in sufficiently large distance (about $1\ \mu\text{m}$ comparable to the size of the Raman laser spot) to the resin region. For example, the LFC edge bulge (see Fig. 6) is investigated. Fano evaluation, calibrated with ECV measurements of homogeneously B-doped Si-wafers, is used to calculate absolute values of the local hole concentration. For this example, local hole concentrations in the range of 10^{19} cm^{-3} (0.3 to $4.8 \times 10^{19}\text{ cm}^{-3}$) are determined (Fig. 6(b)). This is a comparable level of electrically active doping concentration as measured on a sample featuring a p^+ -layer that has been formed by full-area firing of an Al and B containing paste in a belt-furnace (as used for the measurements of Fig. 3).

With this combination of KPFM and Raman measurements, a valuable tool for investigation of the p^+ -region below LFCs is found. On the one hand, the sub-micrometer resolution of the KPFM potential image shows if the doped p^+ -region is continuously extending over the whole contact area and allows to determine its thickness. On the other hand, the absolute values of the hole concentration in the range of 10^{19} cm^{-3} , obtained from Raman measurement in the designated region, indicate that the p^+ -doping most likely originates from an alloying process of Al and B.

D. KPFM-measurement of an LFC with evaporated Al

In contrast to the $\approx 30\ \mu\text{m}$ thick screen-printed Al layer of the examples shown before, a $2\ \mu\text{m}$ thin layer of Al evaporated by electron beam can be laser fired with the same laser system applying single laser pulses. This results in a shallower crater and, apart from the use of n-type Si, in a

FIG. 6. (a) Optical microscope image of an LFC 30° cross section. (b) Detail of this image in the LFC edge bulge region indicating the selected Raman measurement spots for doping concentration measurements.

FIG. 7. (a) Sketch of 30° cross-section of a shallow LFC crater after removing the Al layer. (b) AFM amplitude image of an LFC (formed using evaporated Al) 30° cross-section corresponding to the scan area marked in (a). The border between resin and Si wafer can be seen from AFM amplitude signal in a gap of a few nm caused by polishing. (c) KPFM potential distribution is shown as color scale. Gray and blue indicates the bulk n-type Si with low potential, green indicates the p^+ -region following the LFC crater, and red indicates the epoxy resin region.

“standard LFC”.^{1–3} From a sample with this configuration, the Al layer is removed by etching in aqueous HCl solution and a 30° cross-section is prepared. AFM and KPFM measurements are performed over the scan area sketched in Fig. 7(a) including part of the LFC crater and the edge bulge. In Fig. 7(b), the AFM amplitude signal is shown. The border between resin and Si-wafer can be seen in a gap of a few nm caused by polishing. The Si edge is slightly rounded resulting in an increased amplitude. In Fig. 7(c), the KPFM potential of the same measurement position is presented. Within the Si, a region with increased potential is visible following the LFC crater (scaled to green). This region is supposed to be the p^+ -doped region. It features a reduced thickness compared with the screen-printed case (see Fig. 4(b)). Because of the 30° angle, the 2–3 μm thin layer corresponds to a 1–2 μm thin p^+ -doped layer. It can be observed that the layer is slightly thinner in the middle of the LFC crater where most likely the laser power is higher and therefore more Si is ablated or displaced towards the crater edge.

In Fig. 8, two exemplary line scans of the KPFM potential are shown in detail (positions marked with arrows

in Fig. 7(c)). The p^+ -doped region around the LFC crater is visible in the plateau in the middle of line scan 3 in contrast to the potential step between resin and Si in line scan 4 in the unaffected part besides the LFC. The potential in the resin region is increased compared with the Si region for both cases but does not reach the same level for both line scans. This could be due to charging effects during the measurement. As discussed before, subsequent measurements of the same sample area raise the contact potential difference of the resin regions, while the rest of the sample remains unaffected. This effect can also occur during a single measurement, e.g., when changing the measurement settings and scanning again. In Fig. 7(c), a change in the potential of the resin region can be observed at the crater edge near the end of the measurement (scanning direction is horizontal and proceeding from top to bottom). It can be seen that this area features a disturbed structure with some Si pellets besides the edge bulge possibly influencing the measurement. Nevertheless, this example shows that KPFM is well suitable for the detection of thin p^+ -doped regions. With the advantage of the high spatial resolution, KPFM measurements offer the possibility to check the uniformity of the formed p^+ -doped layer below LFCs.

IV. CONCLUSION

It could be demonstrated that KPFM is applicable to microscopic characterization of the p^+ -region formed by the LFC process. The spatially resolved measurement reveals the position and dimension of the Al doped p^+ -region below the metal/Si interface at sub-micrometer resolution. For this, a careful sample preparation is necessary with polished 30° cross-sections as a promising geometry. Additional EDX measurements have verified that the region with elevated KPFM potential is p^+ -doped Si and not an Al-rich mixture of Al and Si. In comparison with complementary Raman measurements, a high local hole concentration in the range of 10^{19} cm^{-3} was determined in this region. Therefore, a combination of quantitative Raman results and the highly

FIG. 8. KPFM potential line scans corresponding to the arrows drawn in Fig. 7(c). The potential step in line scan 3 caused by the p^+ -region at the crater edge is clearly visible compared with line scan 4 in an area not influenced by the laser pulse.

spatially resolved KPFM measurements allow for a more detailed investigation of the p^+ -region formed by the LFC process applied on Al-coated Si.

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