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A SILICON INGOT LIFETIME TESTER FOR LARGE CRYSTALS

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ABSTRACT

A lifetime-measurement instrument has been developed to characterize large silicon ingots prior to wafering and polishing. It uses the direct-current photoconductance decay method and localized probing and illumination to achieve the necessary sensitivity on low-resistivity, large samples. A 940-nm, 60-Wp, pulsed-laser diode beam (250-μs width, <100-ns cut-off) lights the as-cropped silicon surface between two ohmic-contact probes. A user-friendly graphical interface supports data acquisition, lifetime calculation, and data storage. Pneumatic systems position the ingot and probes. Three-dimensional, finite-element analysis indicates that the detection depth of this technique is much better than the microwave or radio-frequency techniques. It also shows that the as-cropped surface finish is adequate for measuring bulk lifetimes on the order of 50 μs or less–a typical range for Czochralski ingots used in photovoltaic module production. Measurement repeatability and clear distinction among different grades of feedstock materials have been demonstrated.

INTRODUCTION

For applications in which minority charge-carrier lifetime is an important parameter, it is useful to measure bulk lifetime τ on a crystal ingot before carrying out expensive wafering and polishing steps. While this was routinely done when ingot sizes were small, today's large-diameter ingots present a challenge to such a measurement. The volume of a sample affected by the light flash is a small fraction γ of the total volume, even for long wavelength light (the light energy must be greater than the band-gap value). In addition, the carrier injection ratio is proportional to $\Delta\sigma/\sigma$ where σ is the conductivity. And $\Delta\sigma$ is proportional to τ and the excitation light intensity. By all practical means, the signal-to-noise ratio that is proportional to $\gamma\Delta\sigma/\sigma$ would be too small for any meaningful measurement on a massive, low-lifetime, low-resistivity Si ingot. Of course, a smaller sample segment could be removed from the ingot and measured by the ASTM F28-91 standard photoconductive decay (PCD) method (1), but this involves destructive, laborious, and therefore expensive sample preparation and contacting procedures–especially if the lifetime is to be tested in several ingot locations. The ASTM method is not directly applicable on large ingots.

The commonly used contactless techniques, e.g., microwave reflection or radio-frequency photoconductance decay (μ-wave-PCD or RF-PCD), have a carrier-density-dependent skin effect that emphasizes surface recombination and nonlinear circuit response to photoconductance. They generally require a tuning operation for each measurement. Our objective was to develop a nondestructive, inexpensive, preparation-free, and easy-to-use instrument to measure bulk minority-carrier lifetimes on an as-cropped surface of a low-resistivity, large ingot. One of the applications for such an instrument is in the commercial production of crystalline-silicon solar cells, in which a variety of feedstock materials may be used due to cost and availability considerations. Czochralski-silicon (CZ-Si) crystals grown from such feed materials could have varying impurity levels and defects that reduce solar cell efficiency. Lifetime characterization of the crystal ingots before slicing could save the unnecessary processing costs of these materials.

SILICON INGOT LIFETIME TESTER

We have developed an instrument to directly measure the bulk minority charge-carrier lifetime τ on large, cropped silicon ingots. The instrument uses the direct-current photoconductance decay (DC-PCD) method for detection of a transient photoconductance signal and localized probing and illumination to achieve the necessary sensitivity on low-resistivity and large samples. We chose the DC-PCD method for circuit linearity and simplicity. To eliminate the need for contact preparation and to generate a sufficient photoconductance decay signal for low-resistivity, large ingots, a pair of probes having ohmic-contact behavior with silicon is used as schematically shown in Fig. 1. A long-pulsed laser beam (~250-μs pulse width and <100-ns cut-off) is delivered onto the as-cropped silicon surface between the probes by a high-powered (60-Wp) 940-nm laser diode array.

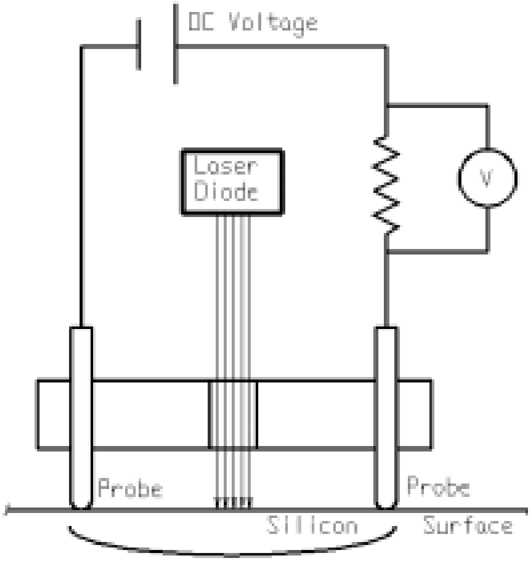

Fig. 1. A schematic diagram of the probe system used for measuring τ on large, cropped Si ingots

The system configuration is shown in the more detailed schematic of Fig. 2. A digital-storage oscilloscope records the average of many photo-induced conductance transients, stores the measurements, and transmits the data to a personal computer (PC) running a LabVIEW® (National Instruments Corporation) graphical data acquisition program. A least-squares fit to the data and an effective lifetime are calculated within an operator-chosen time subinterval of the conductance transient. The program can store the raw data for later retrieval and refitting during different time intervals, and can print a hard copy. The choice of the time interval for the lifetime calculation as well as the relevance of the effective lifetime to the bulk lifetime will be discussed in the following section.

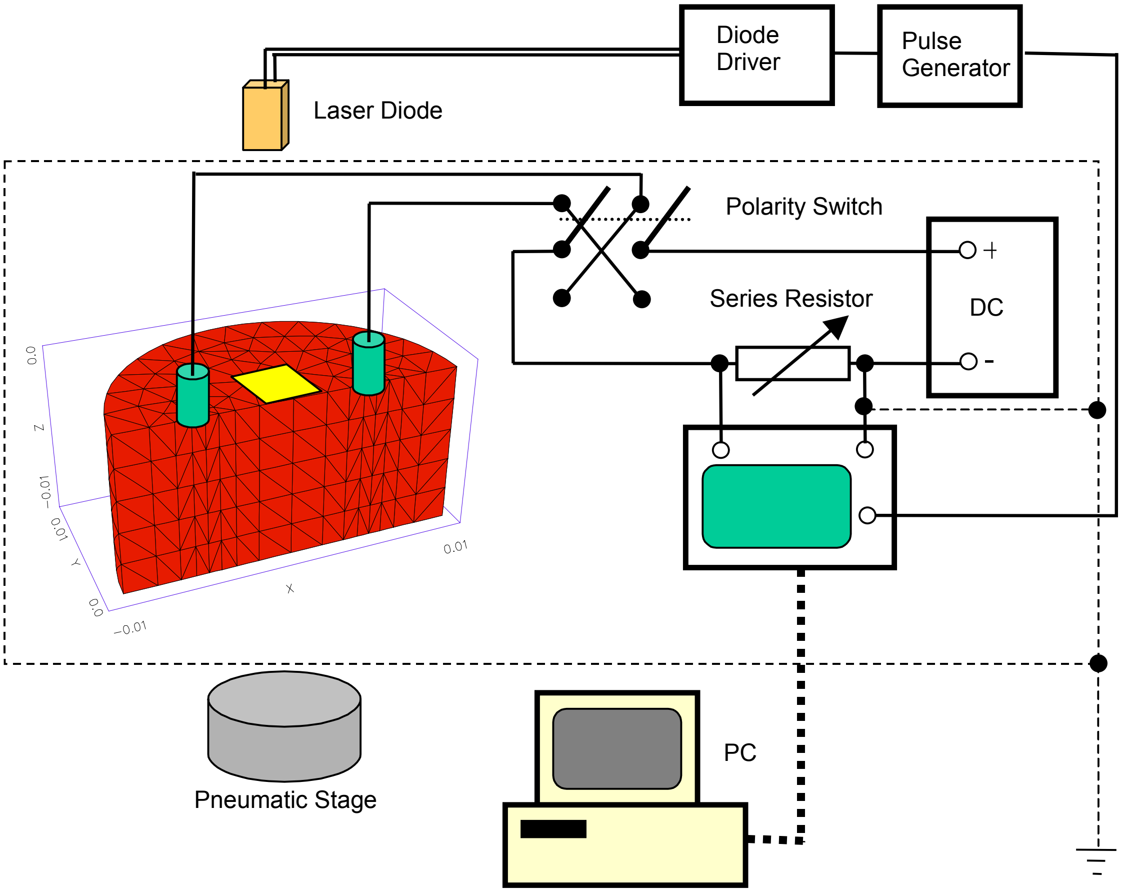

Fig. 2. Schematic block diagram of the lifetime instrument. Half of the silicon ingot sample is shown along with the finite-element grid used for PCD simulation.

The system has a pneumatic stage for positioning and bringing the ingot into contact with the probes. The load spring in the probe assembly, a proximity switch, and a limit switch determine the probe pressure, which does not affect a given measurement if the pressure is maintained constant during the signal transient. A photograph of the silicon ingot lifetime tester is shown in Fig. 3. In principle, any size of ingot can be measured.

In use, the cropped ingot is placed on the ingot-holder platform, a switch is pressed to activate the pneumatic mechanism that automatically raises the ingot to measurement position in contact with the probes, and the door is closed to electrically shield the measurement region. Information about the ingot is entered into the PC, and a transient trace is established on the oscilloscope. The system then averages a selected number of transient conductance traces and transmits the average transient to the PC. There, a best fit is calculated to the transient data, and this is displayed along with the data. From the best fit over a selected sub-interval of the transient time, the lifetime is calculated and displayed. The lifetime, the transient data, and the best-fit curve can all be printed and/or stored. The door is then opened and the ingot is repositioned for additional measurements at other radial positions or on the other end. An auxiliary cup-shaped holder can be used for measurements on cropped crystal transitions or termination cones.

MODELING

A three-dimensional finite-element analysis was performed to determine how the electric potential and current are distributed in the sample and how much of an effect the surface has on the PCD signal for the probe configuration described above. For analogous 2-D modeling, see Wang and Ciszek (2). To minimize computation, a small sample geometry (radius = 1.0 cm, height = 1.0 cm) was chosen, but a much larger ingot does not change the distribution substantially for the same probe spacing. Figs. 4a and 4b show the calculated potential contours on the top surface and the center vertical plane, respectively. Even when a total voltage of 10 volts was applied between the two probes, only about 1 volt is actually dropped in the center

Fig. 3. Photograph of the silicon ingot lifetime tester

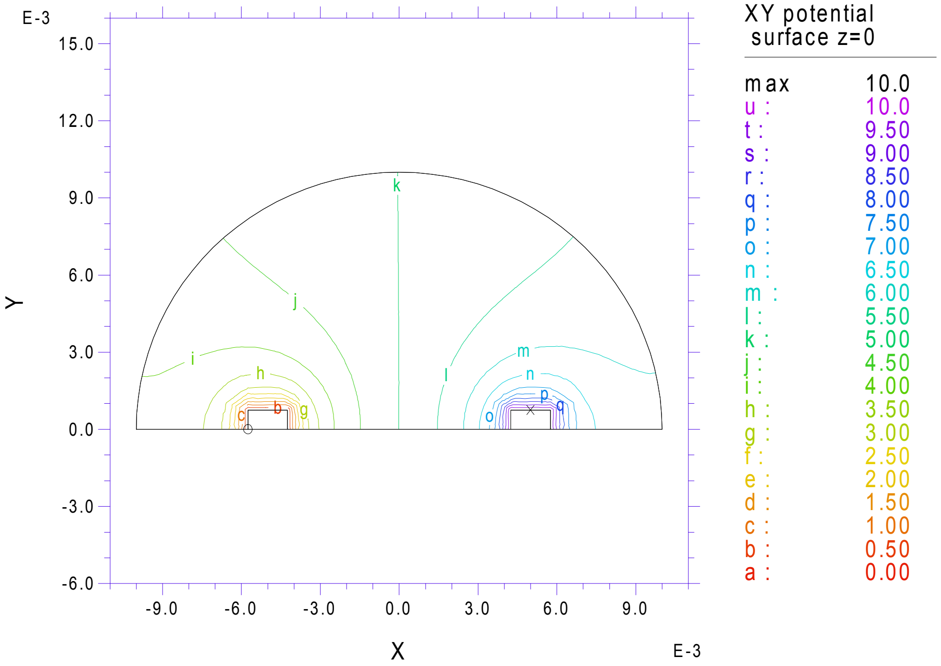

Fig. 4a. Electric-potential contours on the top sample surface.

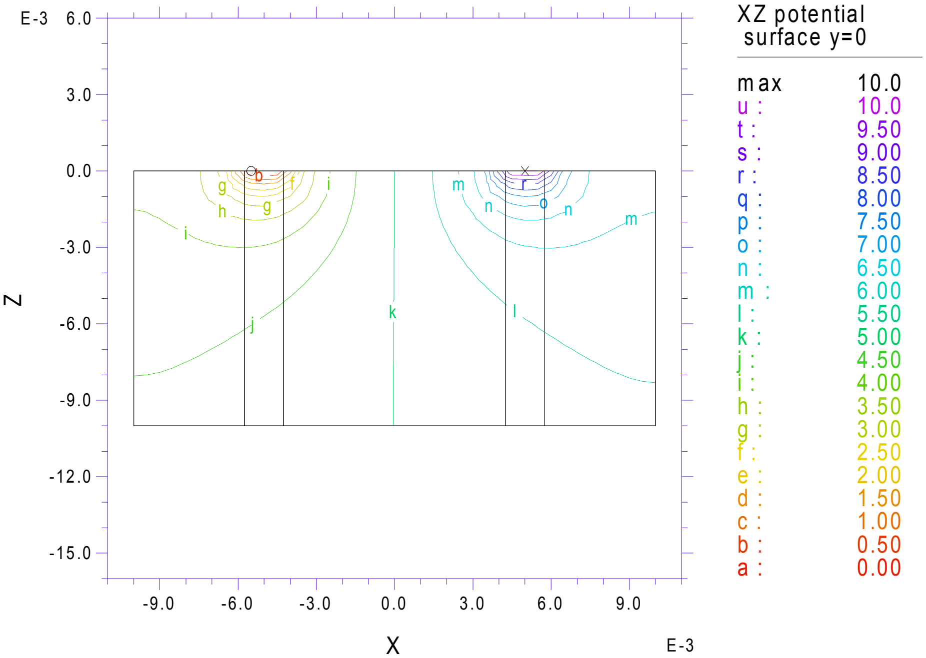

Fig. 4b. Electric-potential contours on the center vertical plane containing the probes

excitation region of about 3-mm width. A large voltage drop occurs around each probe tip. This makes the PCD signal sensitive to mechanical stability of the contacts, and adequate precautions must be made in the probe head and pneumatic mechanism design.

The current density in the center cross section between the two probes is shown in Fig. 5. The current is somewhat concentrated near the center line (represented by the upper left corner) between the probes. Yet, it also spreads out as deep as 5 mm before the current density drops by a factor of ~2. This implies that the surface effect is minimal, especially for the relatively low bulk lifetimes characteristic of commercial PV-grade CZ-Si. In addition, the influence is confined to the top surface, and hence is much less than that from the two surfaces of a wafer.

We have simulated the PCD signal for the case in which the surface recombination velocity is 10^5 cm/s compared with the signal when there is no surface recombination velocity. The actual bulk lifetime was taken to be 20 µs. The surface effect only lowers the effective lifetime to 18.8 µs. Therefore, as long as the ingot lifetime is relatively low (~50 µs or less), the effective lifetime is a reasonably close approximation to the actual bulk lifetime, within measurement error. For higher-lifetime ingots, it would be possible to develop a surface recombination velocity correction algorithm based on appropriate finite element analysis and simulations.

In making actual measurements, the initial segment of the PCD conductance transient signal should be ignored because of the high-order surface effect. The tail segment should also be ingnored because of complications arising from carrier drifting by the electric field to the contact area and possible trapping effects. For higher-lifetime ingots, a larger probe spacing could be used to reduce these effects.

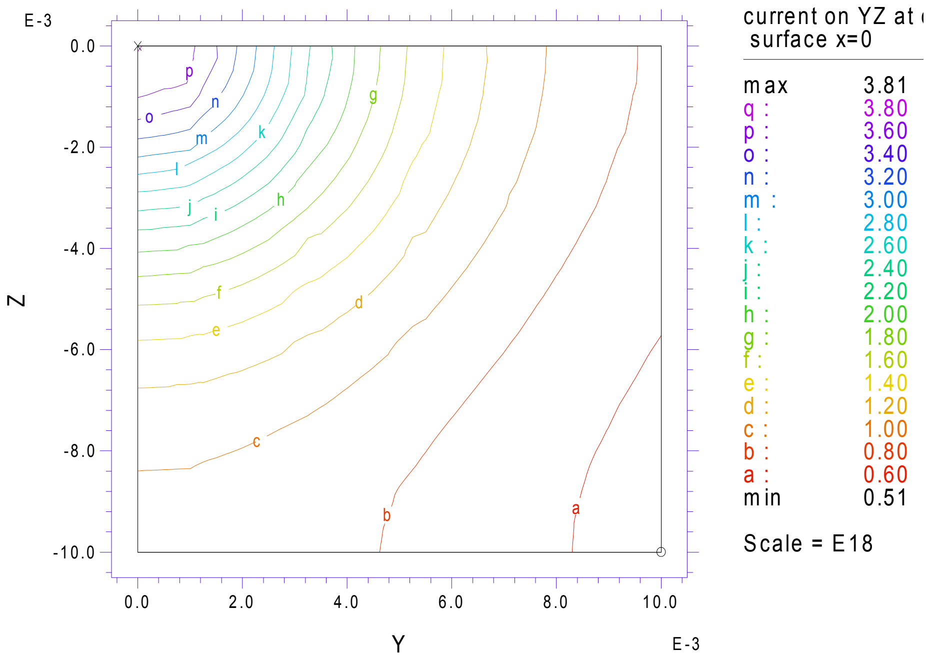

Fig. 5. Current density distribution in the center vertical plane between the probes

LIFETIME CHARACTERIZATION EXAMPLES

Measurement Repeatability

A repeatability study following the balanced Analysis of Variances (ANOVA) procedure [3] has shown that the instrument is capable of detecting the difference between a) the seed end and tail end of the ingots, and b) various locations on the same end of the ingot. In both cases, repeated measurement at the same location is not a significant source of variation, as Table I shows. The P-value is the probability that the source factor is not significant.

Table 1. Lifetime Measurement Repeatability Results by ANOVA

a) Analysis of Variance for Lifetime: Ingot seed end and tail end.

Source	DF	SS	MS	F	P
Ingot	8	10303.80	1287.97	22.24	0.000
Seed/Tail	1	1579.34	1579.34	27.27	0.000
Rept Up/Down	2	25.80	12.90	0.22	0.801
Rept Only	1	15.56	15.56	0.27	0.605
Error	95	5501.38	57.91		
Total	107	17425.88			

b) Analysis of Variance for Lifetime. Location.

Source	DF	SS	MS	F	P
Location	3	470.792	156.931	23.67	0.000
Rept Up/Down	2	6.750	3.375	0.51	0.610
Rept Only	1	3.375	3.375	0.51	0.485
Error	17	112.708	6.630		
total	23	593.625			

Lifetime Differences Due to Type of Feedstock Material

More than 400 CZ ingots were grown from remelt, virgin, and pot-scrap feedstock material blends, and measured using the lifetime tester. Remelt is scrap material from previously grown CZ ingots including tops and tails, whereas pot-scrap is residual materal left in the crucible from previous growth runs. Fig. 6 presents the lifetime values measured for different categories of source material. The data clearly show that pot-scrap feed material yields lower lifetimes than remelt or virgin material.

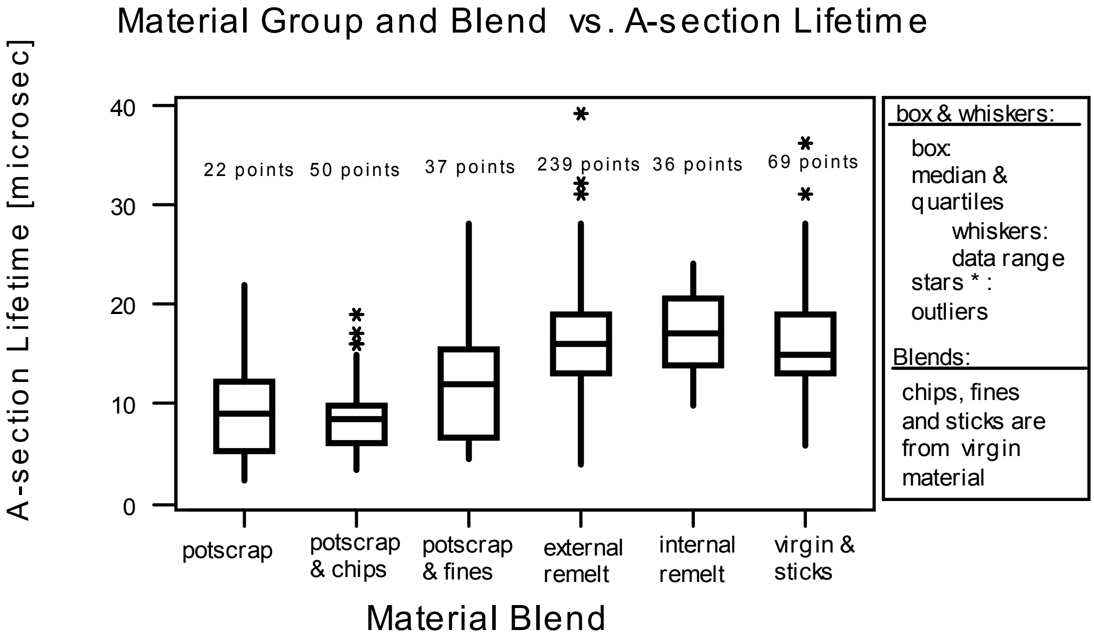

Fig. 6. CZ-Si ingot lifetimes vs. feedstock source material. Median and quartile data are shown, as well as highest and lowest values.

Lifetime Differences for Sequential Crystals Grown from the Same Melt

Ingots grown from low-quality feedstock occasionally lose their dislocation-free single-crystal structure. At that point, the ingot (A) is tailed and pulled from the melt and another ingot (B) is grown from the remaining silicon charge. Lifetimes of the cropped tail ends of both A and B crystals were measured and are shown in Fig. 7. The lifetimes of B crystals were usually lower than tose of A crystals, probably due to a higher impurity content because of impurity segregation.

SUMMARY AND DISCUSSION

We have developed a DC-PCD lifetime instrument to characterize large cropped silicon ingots before they are subjected to expensive slicing and wafering. The key feature that allows an adequate signal to noise ratio for this measurement is a localized

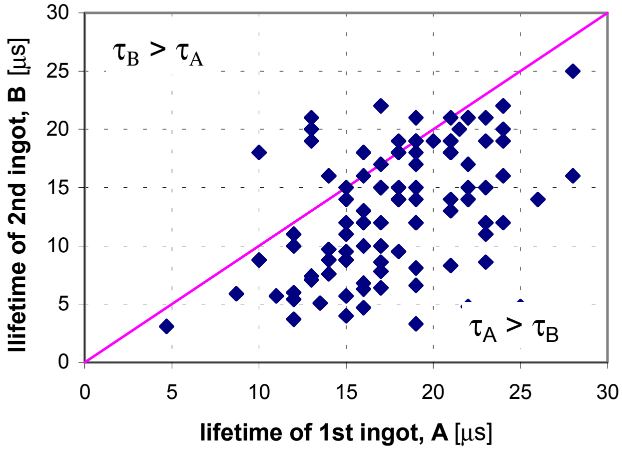

Fig. 7. Lifetimes of (A) and (B) crystals

probe and light source configuration. A three-dimensional, finite-element analysis indicates that an as-cut ingot surface finish is adequate for measuring bulk lifetime on the order of 50 µs or less. This level is typical of CZ ingots grown from low-quality silicon feedstock for PV applications. For higher lifetime material, a greater probe separation can be used. Additionally, a surface recombination velocity correction algorithm could be determined from finite-element analysis modeling. Measurement repeatability and clear distinction among lifetimes resulting from different grades of feedstock materials have been demonstrated.

Oxygen thermal-donor effects could be present in all the measurement results. To establish a relationship between ingot lifetime and final device performance (assuming no significant contamination from subsequent processing), thermal-donor annealing of the cropped ingots may be necessary before reliable lifetime measurements can be made.

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