HIGH-PURITY SILICON CRYSTAL GROWTH INVESTIGATIONS
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Goals

A. OPTIMIZE DOPANTS AND MINORITY CARRIER LIFETIME IN FZ MATERIAL

B. IMPROVE THE CONTROL OF LIFETIME DEGRADATION MECHANISMS (IMPURITIES, THERMAL HISTORY, POINT DEFECTS, SURFACE EFFECTS)

C. CHARACTERIZE LIFETIME-RELATED CRYSTALLOGRAPHIC DEFECTS (VIA X-RAY TOPOGRAPHY AND OTHER METHODS)

Recent Emphasis

A. INSTALLATION OF RESIDUAL GAS ANALYSIS (RGA) ON FLOAT-ZONING FURNACE

B. IMPLEMENTATION OF MINORITY CARRIER LIFETIME MEASUREMENTS

C. DEVELOPMENT OF POINT DEFECT DECORATION PROCEDURES

D. X-RAY TOPOGRAPHY AND EBIC CHARACTERIZATION OF WEB AND FZ CRYSTALS

E. CALCULATIONS FOR PURIFICATION BY BOTH EVAPORATION AND SEGREGATION

F. INVESTIGATION OF THE DEPENDENCE OF LIFETIME ON CRYSTAL COOLING RATE (BOTH DISLOCATED AND DISLOCATION-FREE CRYSTALS)

G. GROWTH OF P-TYPE, HEAVILY Ga-DOPED CRYSTALS WITH HIGH LIFETIMES
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Current Potential Uses of RGA in High-Purity Silicon Growth

Qualitative:
1. Leak checking with growth chamber under argon pressure or under vacuum with helium gun.
2. Run-to-run comparisons of purity levels and location of impurity sources.

Quantitative:
1. Detection of evaporated growth zone impurities in vacuum.
2. Analysis of trace gas impurities in 1-3 Atm. growth ambient.
3. Verification of argon supply purity.
Minority Carrier Lifetime Measurements in Low Resistivity (<1 ohm-cm) Silicon

1. High current density at low applied voltages.
   a. Reduction of applied voltage.
   b. Control of sample temperature.
   c. Increased series resistor power ratings.
   d. Reduction of series R/sample R ratio.

2. Low PCD signal amplitude.
   a. Increased flash tube intensity by using an extra capacitor.
   b. Increased sample length.

3. Larger effect of noise on smaller PCD signal.
   a. Computer averaging of 20 to 100 oscilloscope traces.
Current Meter

Reversing Switch

Filament

Si Light Filter

Stroboscope
GenRad
GR 1538 A

DC Power Supply
HP 6206 B

1 μF

Oscilloscope
Nicolet 2090 III

Plotter
Printer
Disk Storage

Computer
HP-86B

Sample no: 50411u1TE
36 ohm-cm P-type (Ga)
600mA 26°C
30-250 μS LSF <20>
Sample number: 5041101010 Data: 5/13/85 Total of 20 traces averaged.
Peak voltage: 0.74564 V
Dissipation factor: 1.0056 - 3 at 10 μs
Sample dimensions: 7.5 x 7.5 x 27 OR Diameter: 0 Rs=1275.6
Measurement temperature: 26 Sample current: 300
Resistance: 2.5 Voltage: 0.72 Resistivity: 3.6 Type: P
Series R: 10.4 Maximum series current: 683
Predicted V at 2000 μs is: 4.97E-6
Plot for points from t=10 to 250 μs
LST Done 32.8 Minutes
LAPLOT
Procedure for Copper Decoration

1. ETCH 1 CM THICK SAMPLE IN NaOH
2. APPLY SATURATED, HOT CuSO$_4$/WATER TO WARM SAMPLE
3. HEAT TO 850°C FOR 1 HR.
4. COOL TO 750°C OVER A 4 MIN. PERIOD
5. COOL TO 400°C OVER A 2 MIN. PERIOD
6. COOL TO 200°C OVER A 4 MIN. PERIOD
7. COOL TO <50°C OVER A 4 MIN. PERIOD
8. REMOVE A THIN WAFER ABOUT 2 MM FROM SURFACE OF SAMPLE
\[ \frac{C_n(x)}{C_o} = \left( \frac{k}{k+g} \right)^n \left[ 1 - (1-k-g)Z_n e^{-(k+g)x} \right] \]

where

\[ Z_n = n - \sum_{s=1}^{n-1} (n-s)(k+g)^{s-1} e^{-(k+g) \left( (s+x)^{s-2} / s! \right)} \left( (s-1)x + (s+x)[1-(k+g)x] \right) \]

and

- \( n \) = number of zone passes
- \( k \) = effective segregation coefficient
- \( g \) = effective evaporation coefficient
- \( x \) = position in units of zone length
- \( C_o \) = original uniform impurity concentration
- \( C_n(x) \) = impurity concentration at \( x \) after \( n \) passes.

In the last zone (or for normal freezing):

\[ C_n(x) = C_n(N-1)(N-x)(k-1)^{n-1} e^{-g \left[ x-(N-1) \right]} \]

where \( N-1 < x < N \) and \( N \) = ingot length in zone length units.
### Advanced Silicon Sheet

**Graph Description:**
- The graph depicts the ratio of concentration ($C_n/C_0$) as a function of position (in zone lengths) for different values of $g$.
- The positions range from 0 to 20, and the concentration ratio ranges from $10^{-4}$ to $10^5$.

### Table: Impurity Effective Segregation Coefficient vs. Effective Evaporation Coefficient

<table>
<thead>
<tr>
<th>Impurity in Silicon</th>
<th>Effective Segregation Coefficient</th>
<th>Effective Evaporation Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>0.003 (a)</td>
<td>0.2 (e)</td>
</tr>
<tr>
<td>Antimony</td>
<td>0.07 (a)</td>
<td>100 (e)</td>
</tr>
<tr>
<td>Arsenic</td>
<td>0.5 (a)</td>
<td>7 (e)</td>
</tr>
<tr>
<td>Boron</td>
<td>0.9 (a)</td>
<td>0.007 (e)</td>
</tr>
<tr>
<td>Copper</td>
<td>0.04 (b)</td>
<td>0.035 (b)</td>
</tr>
<tr>
<td>Gallium</td>
<td>0.014 (a)</td>
<td>1.7 (e)</td>
</tr>
<tr>
<td>Gold</td>
<td>0.004 (b)</td>
<td>0.012 (b)</td>
</tr>
<tr>
<td>Indium</td>
<td>0.007 (a)</td>
<td>7 (e)</td>
</tr>
<tr>
<td>Iron</td>
<td>0.00001 (c)</td>
<td>0.035 (e)</td>
</tr>
<tr>
<td>Manganese</td>
<td>0.00005 (c)</td>
<td>0.35 (e)</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.45 (d)</td>
<td>0.71 (d)</td>
</tr>
</tbody>
</table>

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Growth configuration for studying minority carrier lifetime as a function of crystal cooling rate after solidification

Growth of a 34 mm diameter [100] dislocation-free FZ crystal
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Gallium Doping

\[ m = \frac{WCV}{kL_A} \]

Where

- \( m \) = Mass of pure gallium applied to seed end of ingot
- \( W \) = Atomic weight of gallium
- \( C \) = Desired uniform dopant concentration in crystal
- \( V \) = Zone melt volume
- \( k \) = Effective segregation coefficient of Ga in Si
- \( L_A \) = Avogadro's number

\( m \) is about 6 mg for a 0.3 Ohm-cm, 34 mm diameter crystal

(220) X-ray topograph (nuclear emulsion plate) of a dislocation-free, 0.34 Ohm-cm, Ga-doped, (100) wafer from a float-zoned, [100] crystal with minority carrier recombination lifetime >200 microseconds

(422) X-ray topograph (DEF-5 film) of a copper-decorated vacuum float-zoned, dislocation-free (111) silicon wafer
Fig. 3. The minority-carrier lifetime versus majority-carrier concentration relationship used for p-type material. For n-type material the Auger cutoff is shifted slightly towards lower concentrations. Otherwise, $\tau_p$ is taken as identical to $\tau_n$. The three circles indicate the points determined by the experts group (see Acknowledgment).
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Future Plans

A. INVESTIGATE DOPANT SPECIES EFFECTS ON MINORITY CARRIER LIFETIME
B. INVESTIGATE FEED ROD CLEANING EFFECTS ON MINORITY CARRIER LIFETIME
C. INVESTIGATE SURFACE PROXIMITY EFFECTS ON MINORITY CARRIER LIFETIME
D. GROW AND CHARACTERIZE HIGH-LIFETIME, HEAVILY-DOPED, FZ CRYSTALS