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HIGH PURITY LOW DEFECT FZ SILICON

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The most common intrinsic defects in dislocation-free float zone (FZ) silicon crystals are the A- and B-type "swirl" defects. The mechanisms of their formation and annihilation have been extensively studied by Petroff and de Kock⁽¹⁾, Foell, Goesele and Kolbesen⁽²⁾, and others. Another type of defect in dislocation-free FZ crystals, found by Roksnoer and van den Boom⁽³⁾, is referred to as a D-type defect. Concentrations of these defects can be minimized by optimizing the growth conditions and the residual swirls can be reduced by the post-growth extrinsic gettering process.

Czochralski (Cz) silicon wafers are known to exhibit higher resistance to slip and warpage due to thermal stress than do FZ wafers. Sumino and Yonenaga⁽⁴⁾ examined various conditions under which dislocation-free Cz crystals have higher mechanical strength than dislocation-free FZ crystals. They observed no difference in the yield strength when external damage had been removed by chemical etching. The Cz crystals containing dislocations are more resistant to dislocation movement than dislocated FZ crystals because of the locking of dislocations by oxygen atoms present in the Cz crystals. Nitrogen doping of FZ wafers at concentrations as low as $1.5 \times 10^{15} \text{ cm}^{-3}$ was found by Sumino, et al.⁽⁵⁾ to make such wafers less susceptible to slip and warpage than undoped Cz and FZ wafers containing dislocations.

Recently we have applied a transverse magnetic field during the FZ growth of extrinsic silicon.* The objective was the study of the spatial distribution of the major dopant, under the assumption that the magnetic field would reduce fluctuations in the flow occurring in the melt and thus in the dopant concentration. Important changes in spatial distribution of the dopant were indeed observed, but the level of fluctuations was not substantially decreased. The observed flow patterns, as revealed by striation etching and spreading resistance in Ga-doped silicon crystals, indicate strong effects of the transverse magnetic field on the circulation within the melt. At fields of 5500 gauss, the fluid flow in the melt volume is so altered as to affect the morphology of the growing crystal. For crystals grown without rotation, the cross section of the crystal becomes elliptical, with the major axis of the ellipse aligned along the field direction. Melt flow in the directions parallel and perpendicular to the field are distinctly different. We believe that the magnetic field offers a unique experimental tool to help elucidate the mass and energy transport occurring in the float zone process.

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References:

- (1) Petroff, P.M. and de Kock, A.J.R., J. Crystal Growth 35, 4-10 (1976).
- (2) Foll, H., Gosele, U. and Kolbesen, B.O., J. Crystal Growth 40, 90-108 (1977).
- (3) Roksnoer, P.J. and van den Boom, M.M.B., J. Crystal Growth 53, 563-573 (1981).
- (4) Sumino, K. and Yonenaga, I., Japanese J. Appl. Physics, 20, L685-L688 (1981).
- (5) Sumino, K. and Yonenaga, I., Imai, M., and Abe, T., J. Appl. Physics 54, 5016 (1983).

Introduction

- FLOAT ZONE (FZ) Si CRYSTAL GROWTH
- CRYSTALLINE DEFECTS
- DEFECT GETTERING
- MECHANICAL STRENGTH OF FZ CRYSTALS

Growth of $\langle 100 \rangle$ Dislocation-Free FZ Si Crystals

PARAMETERS AFFECTING THE DISTRIBUTION AND CONCENTRATION OF INTRINSIC DEFECTS

- PULL RATE
- ROTATION RATE OF CRYSTAL
- AMBIENT ATMOSPHERE
- COOLING RATE OF CRYSTAL
- MAGNETIC FIELD

Observable Imperfections in Dislocation-Free FZ Si Crystals

- STRIATIONS
- "SWIRLS"
- STACKING FAULTS

Swirls

DENSELY PACKED DISCRETE DEFECTS

A-, B-, AND D-TYPE DEFECTS IN FZ DISLOCATION-FREE CRYSTALS

A-TYPE

- DISLOCATION LOOPS OR CLUSTERS OF DISLOCATION LOOPS WHICH ARE FORMED BY AGGLOMERATION OF Si SELF-INTERSTITIALS
- DISTRIBUTED IN STRIATED PATTERN NEAR BANDS OF IMPURITIES WITH DISTRIBUTION COEFFICIENT LESS THAN ONE
- SIZE: $\sim 1 - 40 \mu\text{m}$
- CONCENTRATION: $10^7 - 10^9 \text{ cm}^{-3}$ (H. FÖLL, et al.)
- PULL RATES OF CRYSTAL FOR FORMATION OF DEFECTS: 0.2 - 4.5 mm/min (23 mm diam)

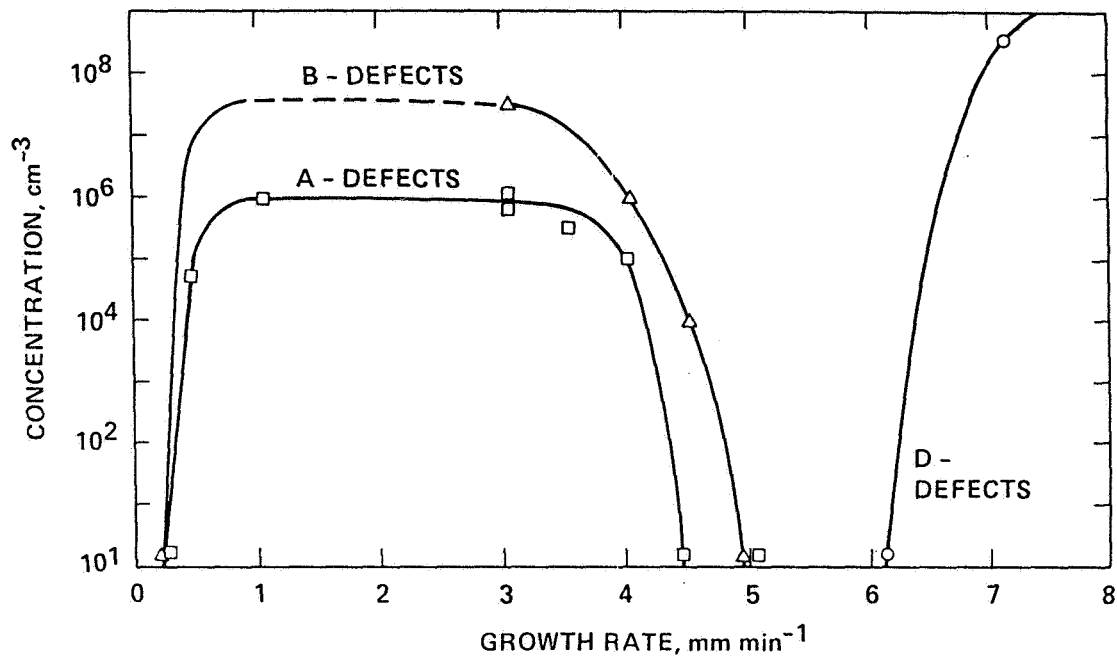
B-TYPE

- THREE DIMENSIONAL AGGLOMERATES OF Si SELF-INTERSTITIALS AND IMPURITY ATOMS, PARTICULARLY C
- DISTRIBUTED IN STRIATED PATTERN SIMILARLY TO A-TYPE SWIRLS
- SIZE: $\leq 30 \text{ nm}$
- CONCENTRATION: $< \sim 10^{11} \text{ cm}^{-3}$ (H. FÖLL, et al.)
- PULL RATES OF CRYSTAL FOR FORMATION OF DEFECTS: 0.2 - 5 mm/min (23 mm diam)

D-TYPE

- CONDENSATION OF POINT DEFECTS (VACANCIES), WHICH DO NOT PRECIPITATE ON CARBON NUCLEI
- HOMOGENEOUSLY DISTRIBUTED IN THE CRYSTAL
- SIZE: $30 \text{ nm} - 1 \mu\text{m}$
- CONCENTRATION: 10^9 cm^{-3} (P. J. ROKSNOER, et al.)
- PULL RATES OF CRYSTAL FOR FORMATION OF DEFECTS: $> 6 \text{ mm/min}$

Swirl Concentration vs Growth Rate



FROM: P. J. ROKSNOER AND M. M. B. van den BOOM
J. OF CRYSTAL GROWTH 53 (1981) 563 - 573

Extrinsic Gettering of Swirl Defects

ABRASION GETTERING (C. L. REED AND K. M. MAR)

- MATERIAL
 - <111>, P-DOPED, FZ Si CRYSTAL WITH SWIRL DEFECTS
 - WAFERS $305 \pm 13 \mu\text{m}$ THICK
 - SPECULAR DAMAGE-FREE FRONT SURFACE
 - CHEMICALLY ETCHED STRAIN-FREE BACK SURFACE
- ABRASION
 - BACK SIDE DAMAGE APPLIED BY MECHANICALLY LAPPING WITH 2 - 15 μm SIZE DIAMOND AND ALUMINA PARTICLES
- OXIDATION AND CHEMICAL ETCHING
 - OXIDIZED AT 1150°C TO ACTIVATE DEFECTS AND WRIGHT ETCHED TO REVEAL DEFECTS
- RESULTS
 - ABRASION GETTERING REDUCED THE ETCH PIT DENSITY FROM 5×10^5 TO $< 5 \times 10^2 \text{ cm}^{-2}$

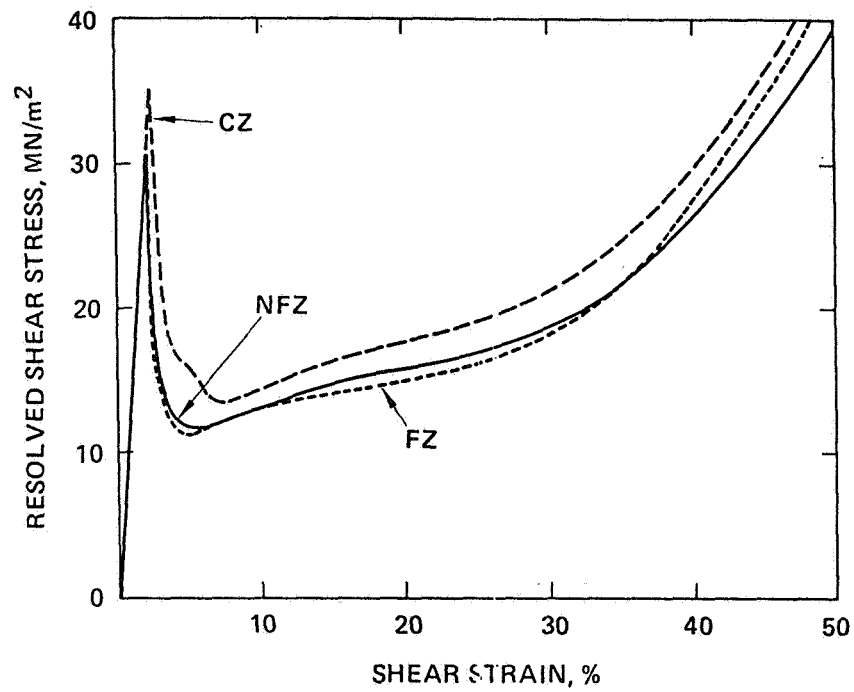
Mechanical Strength of FZ and Cz Si Crystals
(K. Sumino et al, 1981)

- YIELD STRENGTH SAME FOR DISLOCATION-FREE CHEMICALLY POLISHED FZ AND CZ WAFERS
- DIFFERENCE IN MECHANICAL STRENGTH DUE TO DISLOCATION PRIOR TO DEFORMATION
- DISLOCATIONS NOT GENERATED IN CZ FOR STRESSES $\tau < \tau_c$
- DISLOCATIONS GENERATED IN FZ FOR VERY LOW τ

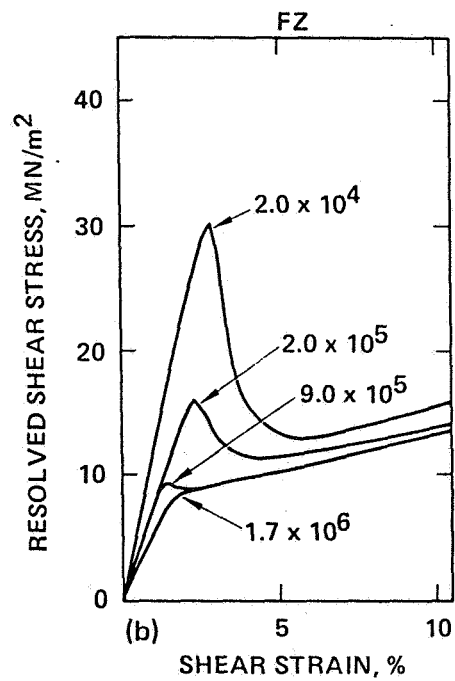
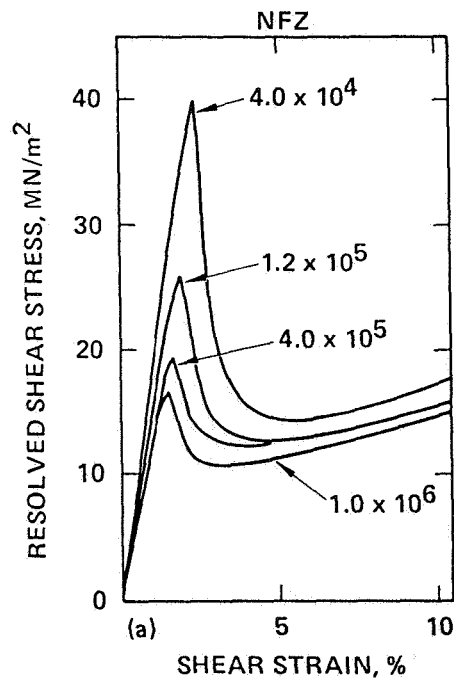
Effect of Nitrogen on Mechanical Strength
of FZ Crystals
(K. Sumino et al, 1983)

- $1.5 \times 10^{15} \text{ cm}^{-3}$ N-DOPED FZ LESS SUSCEPTIBLE TO SLIP
- N ELECTRICALLY INACTIVE IN Si. OCCUPIES INTERSTITIAL SITE
- DISLOCATION-FREE FZ, CZ HAVE SIMILAR STRESS-STRAIN CHARACTERISTICS
- DISLOCATED FZ N-DOPED HAVE HIGH UPPER YIELD STRESS
- DISLOCATIONS IN FZ N-DOPED IMMOBILIZED UNDER LOW STRESSES AT ELEVATED TEMPERATURES

Resolved Shear Stress vs Shear Strain



FROM: K. SUMINO, et al.
J. APPL. PHYS. 54 (9) SEPT 1983



FROM: K. SUMINO, et al.
J. APPL. PHYS. 54 (9) SEPT 1983

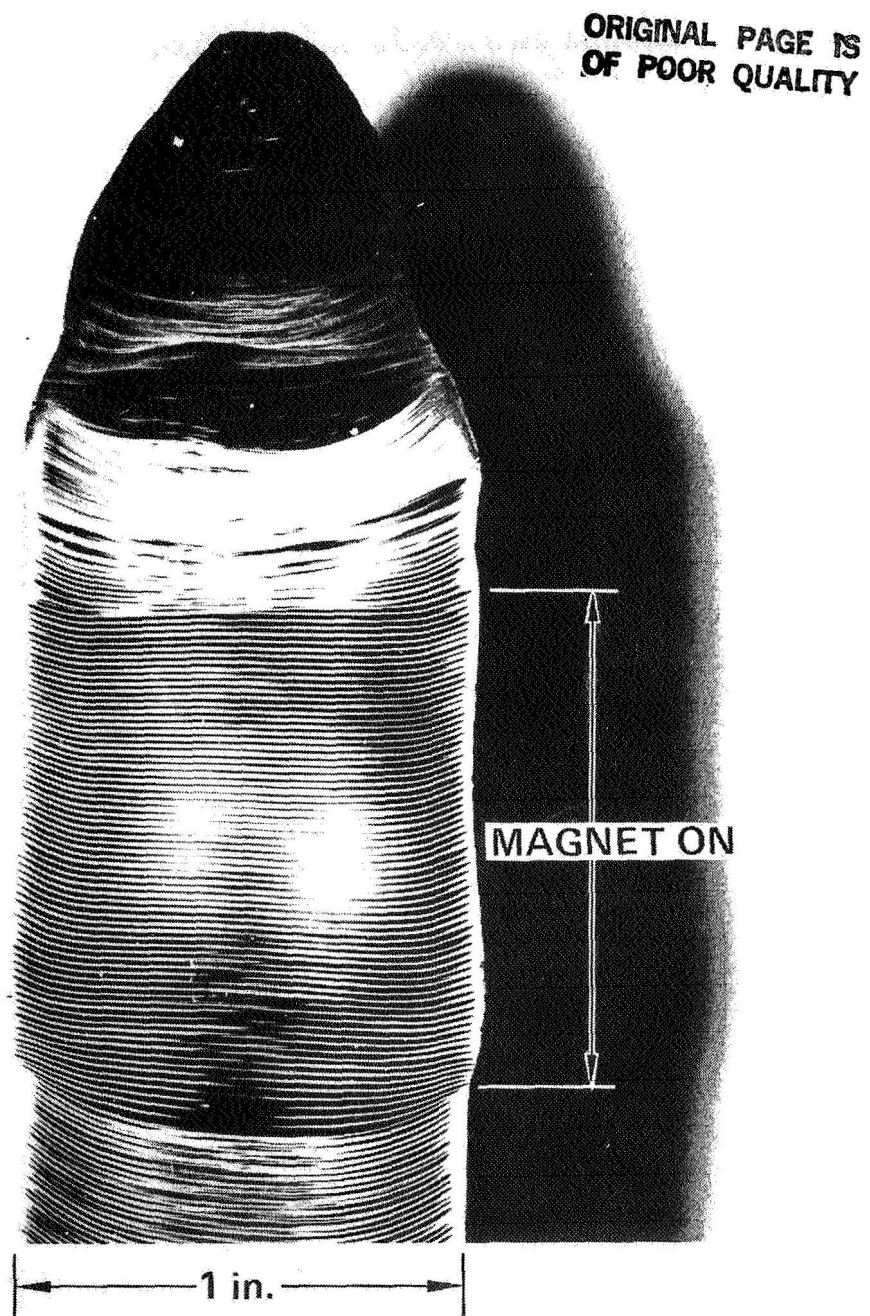
Introduction

- EXPERIMENTAL APPARATUS FOR APPLYING TRANSVERSE MAGNETIC FIELD TO FZ MOLTEN ZONE
- TYPICAL RESULTS FROM ROTATING CRYSTALS
- UNUSUAL RESULTS FOR NON-ROTATING CRYSTALS
- SIGNIFANCE OF RESULTS

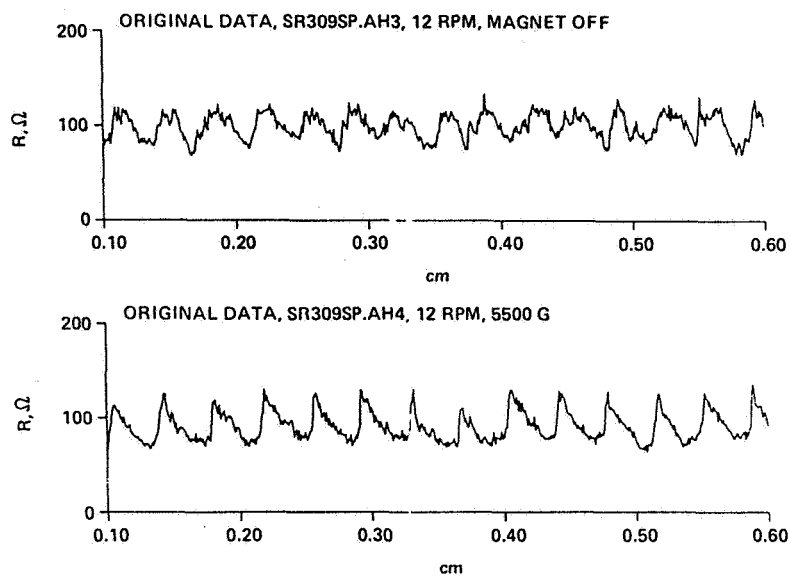
Experimental Parameters

- | | | |
|---------------------------|---|-----------------|
| ● CRYSTAL ORIENTATION | — | <100> AND <111> |
| ● CRYSTAL DIAMETER | — | UP TO 30 mm |
| ● PULL RATES | — | 2 TO 4 mm/min |
| ● ROTATIONAL RATES | — | 0 TO 12 rpm |
| ● MAGNETIC FIELD STRENGTH | — | 0 TO 5500 G |

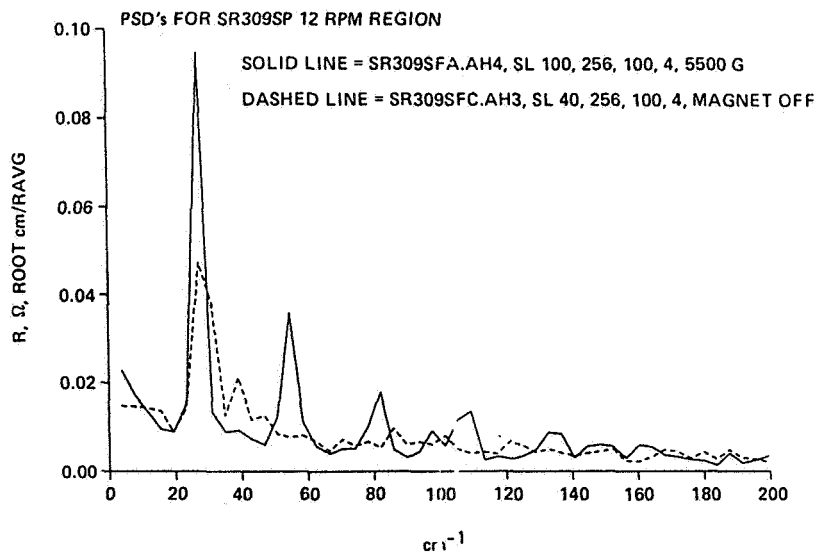
Striation Etch Pattern, Crystal Z309; 5000 Gauss
Transverse Magnetic Field (12 rpm Section)



Typical Spreading Resistance Data



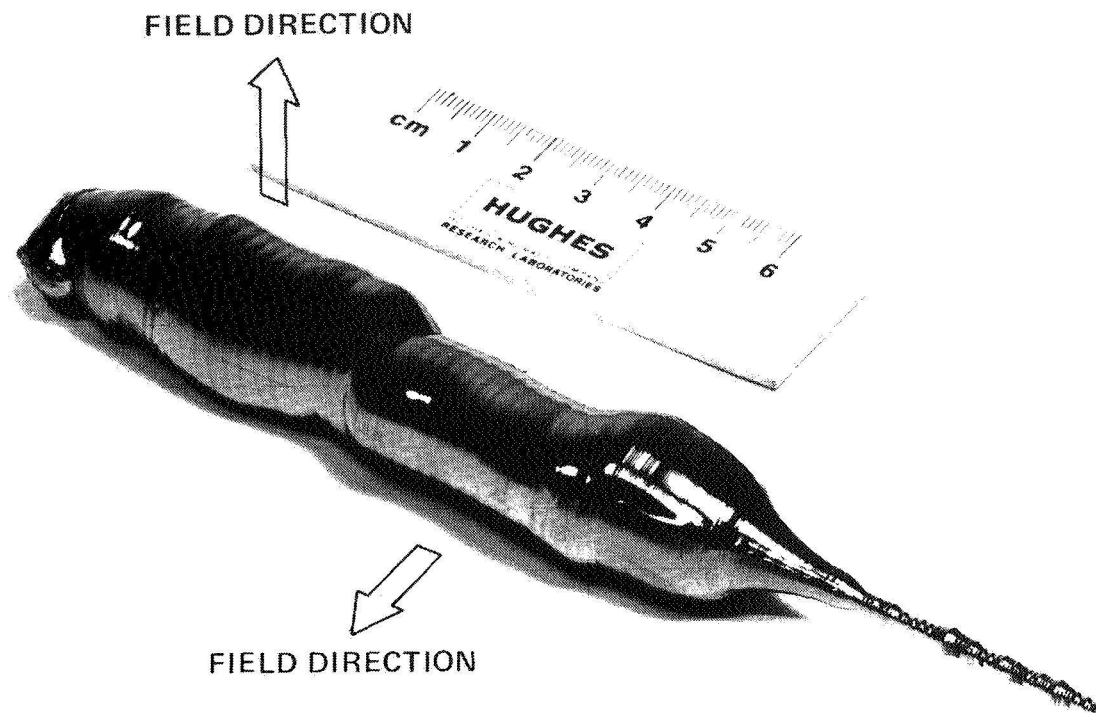
Power Spectral Densities



5500-Gauss Field Applied to Rotating Crystal

OBSERVATION	HYPOTHESIZED CAUSE
• GROWTH INTERFACE "FLATTENS"	ALTERED HEAT FLOW PATTERNS
• CRYSTAL DIAMETER INCREASES	FLATTENING OF INTERFACE
• DOPANT CONCENTRATION INCREASES	THICKENED DIFFUSION BOUNDARY LAYER
• ROTATIONAL STRIAE ENHANCED	REDUCED FLUCTUATIONS
• FINE STRIAE ALTERED	REDUCED "TURBULENCE"

Crystal Grown With No Rotation in 5000 Gauss Transverse Field (Z321)



5500-Gauss Field Applied to Non-Rotating Crystal

OBSERVATION	HYPOTHESIZED CAUSE
• DOPANT CONC INCREASES	THICKENED DIFFUSION BOUNDARY LAYER
• ROTATIONAL STRIAE ABSENT	AS EXPECTED FOR 0 RPM
• FINE STRIAE ALTERED	REASON NOT CLEAR YET
• SR FLUCTUATIONS REDUCED FOR $F < 300 \text{ cm}^{-1}$ ($\sim 2 \text{ Hz}$)	INCREASED MELT "VISCOSITY"
• CROSS SECTION \rightarrow ELLIPTICAL	$B \times V$ EFFECTS
• GROWTH INTERFACE \rightarrow CYLINDRICAL	ALTERED HEAT TRANSFER

Summary

- MAGNETIC FIELD IMPACTS CIRCULATION PATTERNS IN THE MOLTEN ZONE
- FORCES FROM FIELD ARE LARGE ENOUGH TO PRODUCE USEFUL EFFECTS
- FOURIER ANALYSIS OF SPREADING RESISTANCE MAY REVEAL FLUCTUATION SPECTRA IN MELT
- MAGNETIC FIELD PROVIDES NEW TOOL TO STUDY THE FZ GROWTH PROCESS

DISCUSSION

NEITZEL: I've heard the word turbulence mentioned a couple of times today and I think we have to be very careful when we mention the word turbulence because it implies something definite, in a fluid dynamic sense, that I don't think is occurring in your geometry for your region of parameter space.

ROBERTSON: Do you have some good reason for saying it's not occurring?

NEITZEL: I would guess it's not. We did a rough calculation for the maximum rotational Reynolds number that you should see in your configuration. It's probably on the order of 1000. For the transition to turbulence, the transitional Reynolds number is 3×10^5 .

ROBERTSON: All I can tell you is that the flows in that melt volume are very strong. They are being driven by electromagnetic forces. They are not being driven by convective forces or Marangoni surface-tension forces or the rotational forces. Those are all there, but superimposed on that is a circulation pattern from the electrodynamic forces. Professor Muhlbauer has just published information that indicates that that force is 50 to 100 times stronger than these other forces. If you look at particles in that melt zone, for example, they are in violent motion.

NEITZEL: I don't doubt that, but I just want to say that it's possible to have very oscillatory laminar flows that are not turbulent flows.