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JPL IN-HOUSE FLUIDIZED-BED REACTOR RESEARCH

JET PROPULSION LABORATORY

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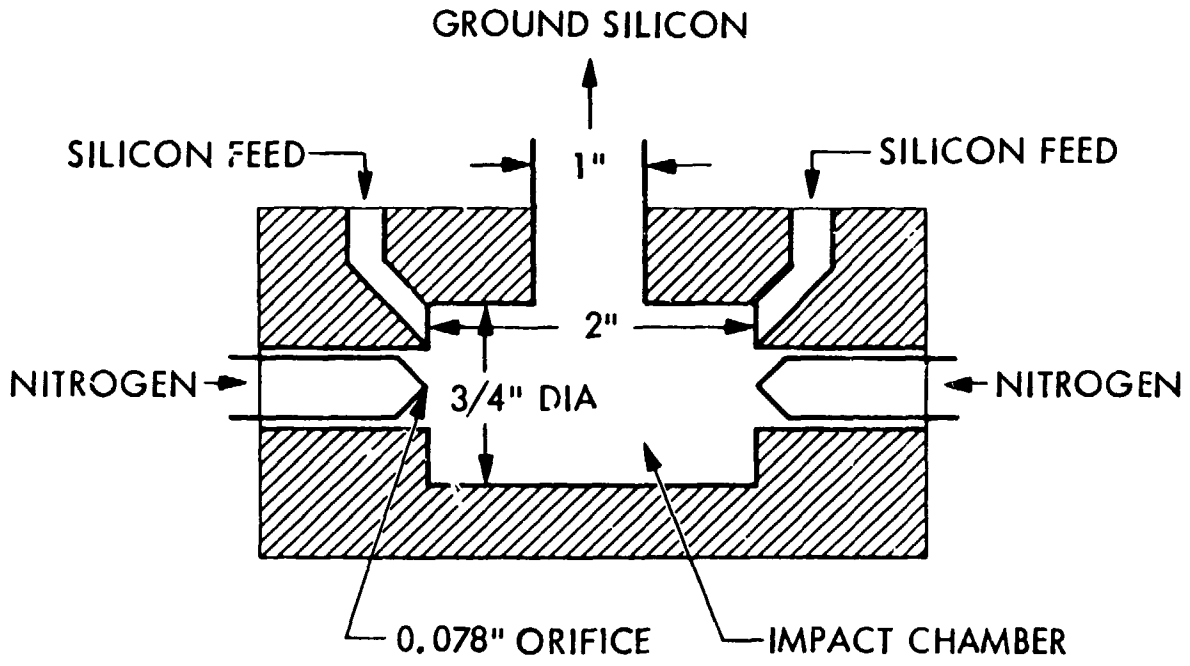
Objectives

- ESTABLISH ANALYTICAL TECHNIQUES TO VERIFY METALLIC IMPURITIES IN SILICON
- OBTAIN CLEAN SILICON SEED PARTICLES
- CONDUCT PURITY EXPERIMENTS
- DRAW SINGLE CRYSTAL Cz INGOT USING SILICON PARTICLES GROWN IN THE FLUIDIZED BED REACTOR.

Silicon Seed Particles

- SILICON PARTICLES OF LESS THAN 2 MM SIZE WERE PURCHASED FROM DYNAMIT NOBEL. THESE PARTICLES WERE PREPARED BY MECHANICAL BREAKING OF SILICON ROD PRODUCED BY THE SIEMENS PROCESS.
- THE SIZE OF THESE PARTICLES WAS FURTHER REDUCED TO 200 TO 300 μM DIAMETER BY THE FLUID JET MILL.
- PARTICLES WERE ACID CLEANED AND DRIED UNDER NITROGEN BLANKET BEFORE FED INTO THE FLUIDIZED BED REACTOR.

Jet Milling Device for Seed Particle Generation



Silicon Seed Particles from Jet Mill

FEED, 20x

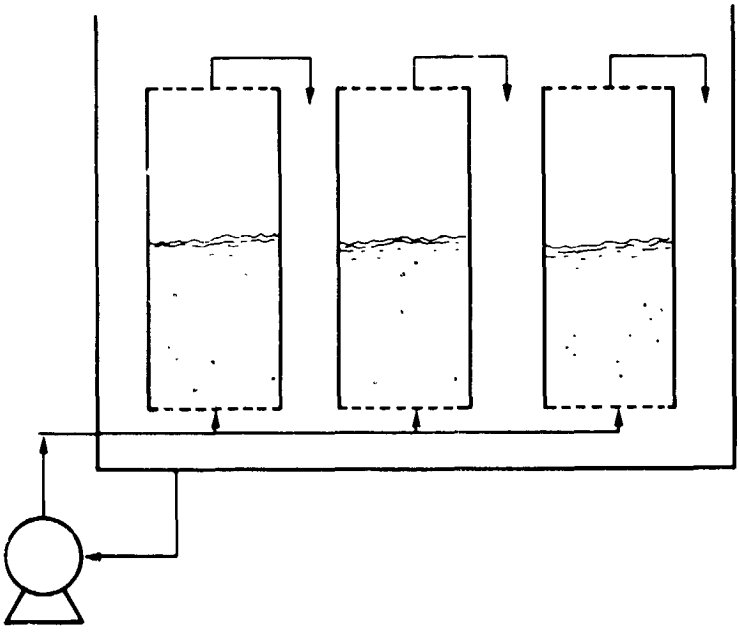
PRODUCT, 20x

NITROGEN FLOW RATE : 20 SCFM
SILICON FEED RATE : 40 gram/min

Silicon Cleaning Procedure

- DEIONIZED WATER WASH TO REMOVE FINE SILICON PARTICLES
- CLEANING IN THE MIXTURE OF TWO PARTS OF I2N HCl AND ONE PART OF 16N HNO_3 FOR 20 MINUTES.
- WASHING WITH DEIONIZED WATER UNTIL EFFLUENT WATER IS NEUTRAL.
- ETCHING WITH 48% HF FOR 20 MINUTES
- WASHING WITH DEIONIZED WATER UNTIL EFFLUENT WATER IS NEUTRAL AND HAS A RESISTIVITY OF 16 MEGAOHMS.
- DRYING IN DIFFUSION FURNACE AT ~120°C UNDER NITROGEN BLANKET

Fluidized Bed Cleaning of Silicon Seed Particles



Purity Experiment

- SEED PARTICLES WERE PREPARED VIA JET MILL GRINDING OF LESS THAN 2 MM SIZE SILICON PARTICLES PURCHASED FROM THE DYNAMITE NOBEL.
- PARTICLES WERE WASHED AND CLEANED VIA FLUIDIZED BED CLEANING SYSTEM.
- EXPERIMENTAL CONDITIONS
    - AVG. INITIAL SEED PARTICLE SIZE: 250 μM
    - INITIAL BED WEIGHT: 11 KG (≈ 24" BED HEIGHT)
    - $U/U_{MF} = 4$
    - SILANE CONCENTRATION: 30% (IN H_2)
    - BED TEMPERATURE: 650°C
    - DURATION OF RUN: 4.30 HRS.

Results

- MASS BALANCE
    - TOTAL SILICON FED: 7.3 KG
    - SILICON DEPOSITED ON PARTICLES: 6.8 KG (93.1%)
    - SILICON RECOVERED AS FINES: 0.4 KG (5.5%)
- PRODUCTION RATE: 1.5 KG/HR.
- PARTICLE GROWTH: ≈ 10 μM (RADIUS).

Purity of Silicon (PPma)*

ELEMENTS	RAW PARTICLES "AS PURCHASED"	JET MILLED AND ACID CLEANED "SEED FOR FBR"	FBR PRODUCT
P	0.2	0.2	0.1
Fe	20	≤0.6	≤0.6
Cr	0.05	0.03	<0.02
Ni	10	<0.5	<0.5
Cu	0.06	≤0.02	<0.02
Zn	<0.02	≤0.04	<0.04
Co	≤0.1	≤0.1	≤0.1
Mn	0.5	≤0.02	<0.02
Na	≤0.1	≤0.1	≤0.1
Mg	≤1	<1	<1
Al	2	0.05	0.05
S	<1	<1	<1
K	≤0.07	<0.1	<0.1
Ca	0.6	0.1	0.1

* SPARK SOURCE MASS SPECTROSCOPY

Work in Progress

- DRAW SINGLE CRYSTAL Cz INGOT USING SILICON PARTICLES GROWN IN THE FLUIDIZED BED REACTOR.
- DETERMINE METALLIC IMPURITIES IN INGOT AND SILICON LEFT IN THE CRUCIBLE.
- FABRICATION OF SOLAR CELLS
- CHARACTERIZATION OF SOLAR CELLS