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ESTABLISH METHODS FOR CRYSTAL GROWTH OF Si-Ge

Parke Mathematical Laboratories, Inc.

Joseph A. Adamski and John S. Bailey

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13. ABSTRACT *(Maximum 200 words)*

The SiGe alloy system will result in a whole new set of high performance electronic and optoelectronic devices, such as, thermoelectric generators, infrared detectors, and high speed optical transmitter-receivers. The production of high speed SiGe devices has been limited by thin-film alloy growth techniques which are compatible with silicon substrates. The alloy composition of thin films is limited because of strain between the substrate and the thin film. This contractual effort is aimed at producing bulk alloy SiGe crystals of uniform composition for use as substrates. These new substrates will expand the range of lattice-matched thin film alloys available for development of high speed SiGe devices. SiGe alloy electronic devices offer some advantages over III-V and II-VI compound semiconductors. Their chemical and thermo-mechanical properties will allow them to be closely compatible with established Si processing techniques and Si integrated circuits.

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BULK CRYSTAL GROWTH OF SILICON-GERMANIUM

Introduction

This is the final report for Contract F19628-95-C-0172 Non-Conservative Bulk Growth of Silicon-Germanium for high performance electronic and optoelectronic devices. The work period for this contract was from August 1, 1995 to February 28, 1997. This summary documents that progress was made in developing methods of solid solution bulk growth of uniform composition alloys from non-congruent melts. Bulk growth of Silicon-Germanium crystals was attained utilizing a liquid encapsulated zone-melting (LEZM) method. In addition, float zone studies of single crystals using a high gradient melt zone were undertaken.

Experimental Procedure

Crystal growth and alloying experiments were conducted using a Bridgman/Float Zone Crystal Growth System with a water cooled quartz chamber (see fig.1). A Lepel induction generator coupled to a split-ring concentrator provided the needed thermal gradient. A vertical Bridgman method combined with a liquid-encapsulated zone melting method was used. To contain the silicon-germanium alloy, silicon seed, and the calcium chloride encapsulant, a quartz ampoule was employed. A number of quartz ampoule designs and sizes, including pointed and flat bottomed, and vacuum sealed were used. The ampoule was attached to the puller shaft which was then raised or lowered into the RF concentrator zone (see fig.2). A quartz tube between the concentrator and the ampoule served as a susceptor retainer and barrier in the event of a melt leakage. A quartz tube with the bottom shaft was also utilized to raise and lower the susceptor into the RF concentrator.

To measure the temperature profile of the melted zone and establish the growth interface of the silicon-germanium alloy, a fiber optic thermometer was used. A pre-drilled silicon-germanium charge was used and the sapphire fiber extracted from the hole at a rate of a few millimeters per hour. A refractory metal coated tip sapphire fiber system made by Luxtron Inc. recorded the temperature as the fiber passed from the silicon seed up though the silicon-germanium alloy.

Charges of silicon-germanium alloy were made using three techniques: a silicon rod with germanium melted on top, pre-drilled silicon with a germanium rod in the center, and zone melting.

Silicon-germanium crystal and alloy experiments were conducted either under an Argon atmosphere with a flow rate of 3 sccm per/min or under vacuum. Coupling to the charge was provided by placing a graphite susceptor ring inside the concentrator coil and thus an open source of radiant heat was provided. Experiments using direct coupling to the charge were also made.

Float zone experiments were undertaken in which the top alloy rotated in one direction (see fig. 3) and the bottom in another direction. Other single rotational direction experiments were performed where the alloy and seed were pinned (see fig 4) to the quartz tube.

Initial wetting experiments were carried out by placing the silicon or the silicon germanium alloy on a substrate in argon atmosphere. Then the wetting angle was measured to determined compatibility of various container materials.

Results

The bulk growth of single crystals of alloys of silicon-germanium was attained by the Liquid Encapsulated Zone Melting method. Composition uniformity of single crystals alloys of 4.5 at% of germanium were attained by this technique. The use of a non-wetting liquid calcium chloride encapsulant aided in the release of the SiGe alloy from the quartz ampoule and suppressed the nucleation of grains. The encapsulant also prevented reaction between the quartz ampoule and SiGe alloy. The liquid alloy and the melted silicon seed are not soluble in calcium chloride. The pre alloying and zone leveling of SiGe in the liquid encapsulant resulted in a compositional homogeneous ingot from seed to tail end as reported in a paper to be published in the Journal of Crystal Growth.

The axial temperature profile of the growing bulk silicon-germanium crystal was obtained. The Accufiber temperature profile measurements revealed that the molten zone length was approximately 8.2 mm in length, and that there is a high thermal gradient. The thermal gradient for solidification between the seed and the melt was measured to be 170^0C/cm, while the melting gradient above the molten zone is 210^0C/cm.

Bulk growth of SiGe alloy using the float zone method resulted in ingots where the periphery was melted but the central core did not reach the molten temperature. Such variables as melting temperatures, electrical conductivity, and convective motion associated with feed-rod rotation between SiGe alloy and the Si seed limited the successful growth of crystals.

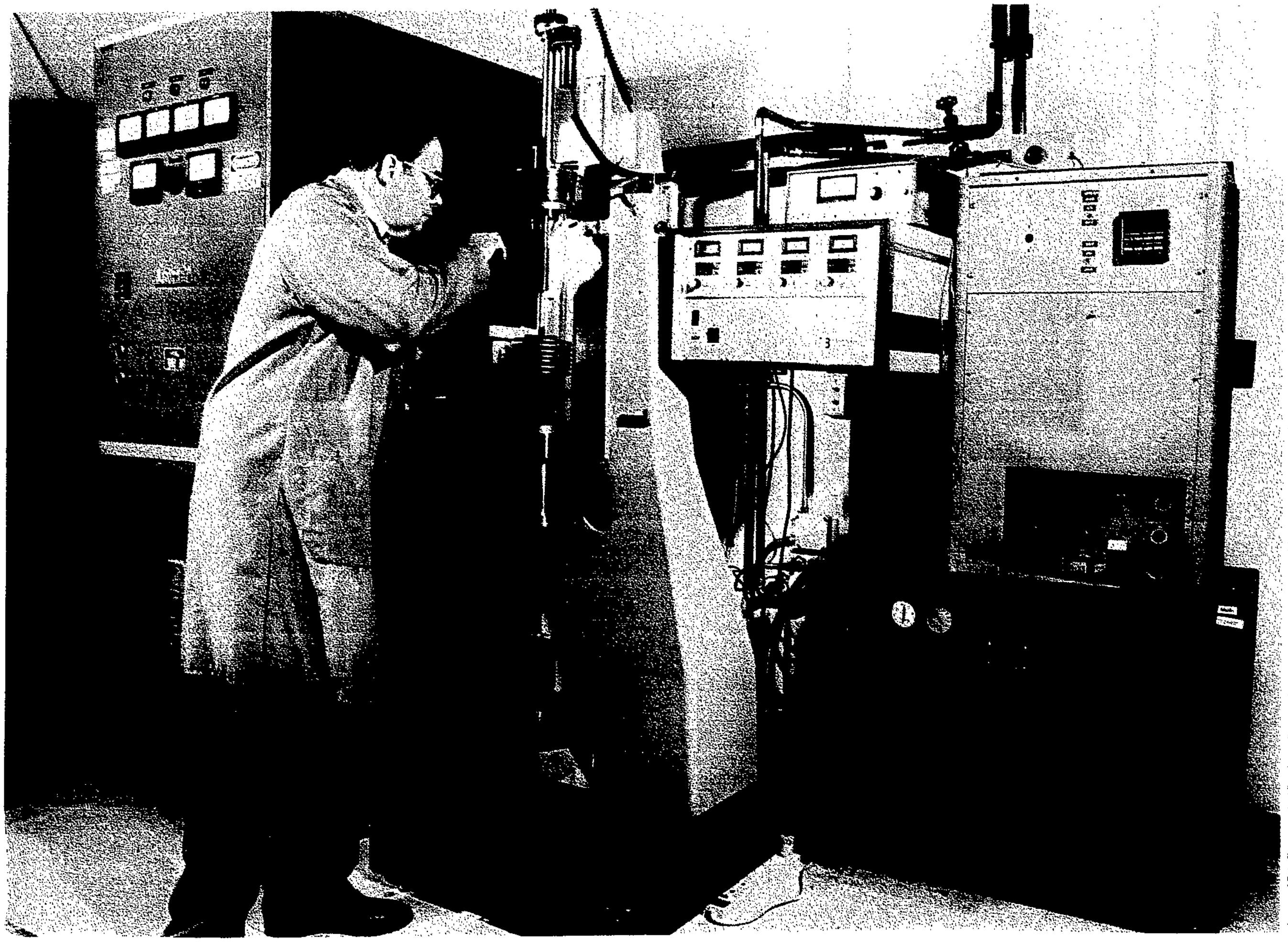
Summary

The LEZM method has been demonstrated for bulk growth of SiGe mixed crystals. Between the alloying and the crystal growth runs more than 94 experiments were carried out. More than 14 wetting experiments were also performed. Four float zone experiments were investigated utilizing the concentrator. A paper will appear in the Journal of Crystal Growth 175(1997) entitled, “Silicon-germanium bulk alloy growth by liquid encapsulated zone melting.” A poster was presented at the Tenth American Conference on Crystal Growth, Vail, Colorado August 4-9, 1996. A patent application

was executed for the invention for Liquid Encapsulation Zone Melting (LEZM) Method for Silicon Bulk Alloys.

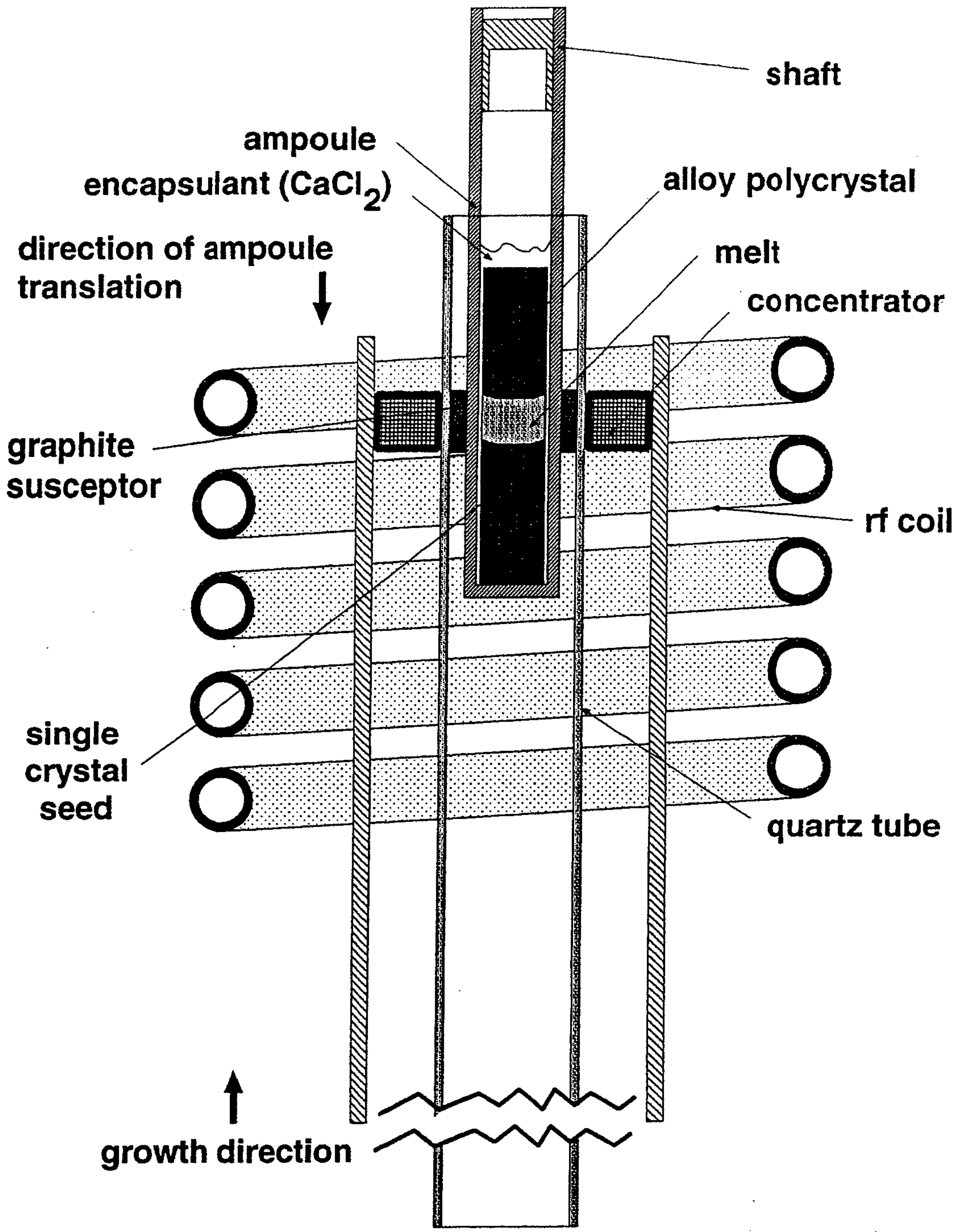
References

"Silicon-germanium bulk alloy growth by liquid encapsulated zone melting," D. Bliss, B. Demczyk, A. Anselmo, J. Bailey, Journal of Crystal Growth, in-press



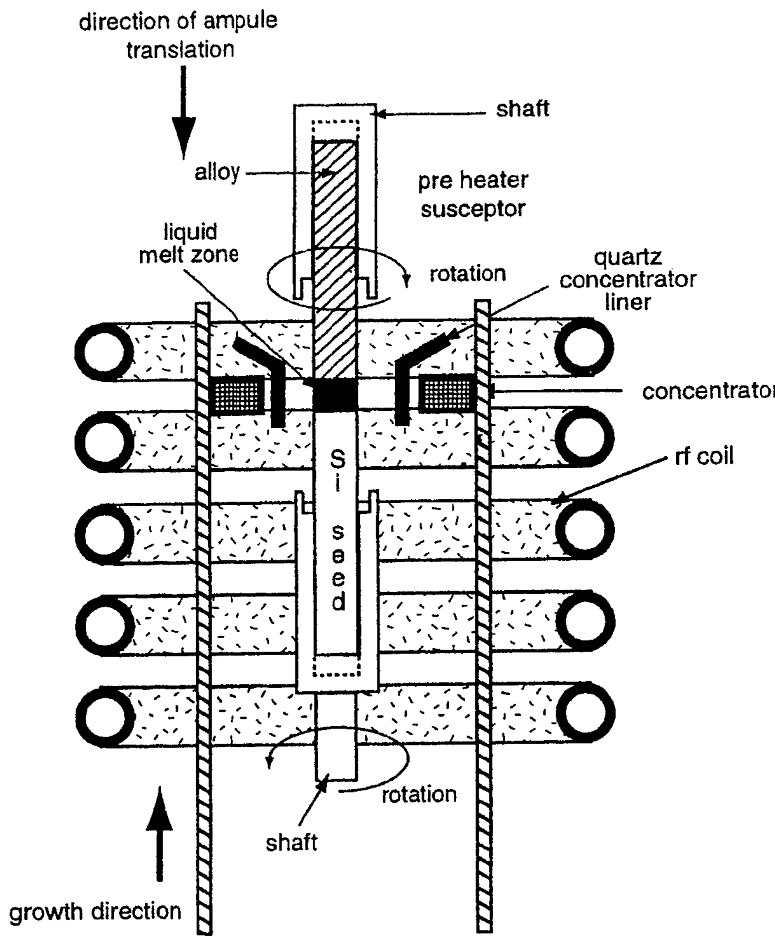
Bridgman/Float Zone Crystal System

Figure 1



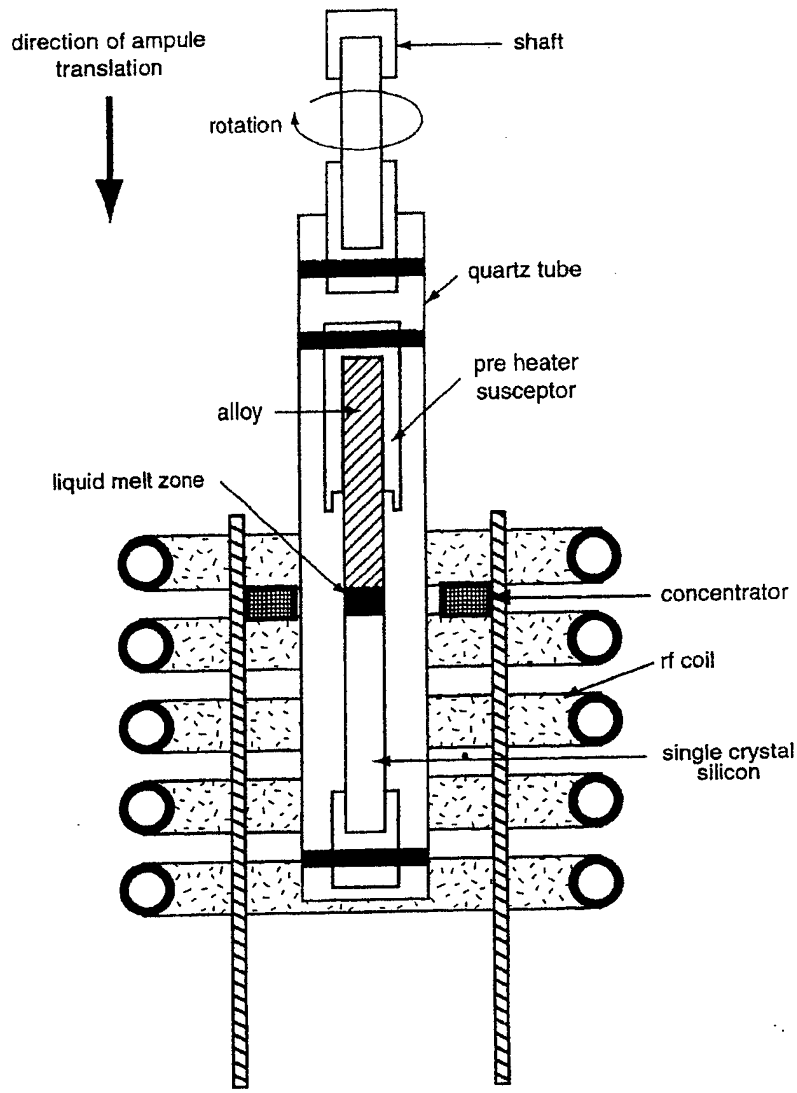
RF Concentrator

Figure 2



<u>Rotation experiment where top and bottom alloys were rotated in opposite directions</u>

Figure 3



Rotational experiment where alloy & seed were pinned to quartz tube

Figure 4

TABLE 1

SiGe Run Sheet

I.D.	Date	Objective	Composition	Hot Zone	Results
RL-SG-5001	8/03/95	Ge on Carbon wetting	Ge	#1 concentrator.	No wetting
RL-SG-5002	8/07/95	Ge on SiC wetting	Ge	#1concentrator.	
RL-SG-5003	8/07/95	Si on SiC wetting	Si	#1concentrator	No melting*
RL-SG-5004	8/08/95	Si on SiC wetting	Si	#1concentrator	No melting
RL-SG-5005	8/08/95	Si on SiC wetting new Susceptor	Si	#1concentrator	Melt&wetting
RL-SG-5006	8/08/95	SiGe on SiC wetting " "	SiGe 3to1wt.%	#1concentrator	Melt&wetting
RL-SG-5007	8/09/95	SiGe on quartz wetting	SiGe 3to1wt.%	#1concentrator	Melt slight wet.
RL-SG-5008	8/09/95	SiGe on Sapphire wetting	SiGe 3to 1wt.%	#1concentrator	Melt slight wet.
RL-SG-5009	8/10/96	SiGe on BN wetting	SiGe 3to1wt.%	#1concentrator	Melt slight wet.
RL-SG-5010	8/10/95	SiGe on single crystal SiC wetting	SiGe 3to1wt.%	#1concentrator	Melt&wetting
RL-SG-5011	8/10/95	SiGe on Poco carbon wetting	SiGe 3to1wt.%	#1concentrator	Melt&wetting
RL-SG-5012	8/10/95	SiGe on vitrious carbon wetting	SiGe 3to1wt.%	#1concentrator	Melt&wetting
RL-SG-5100	8/15/95	water leak			
RL-SG-5101	8/22/95	water leak			
RL-SG-5102	8/24/95	SiGe in quartz crucible	SiGe 3to1wt.%	#2	No melting
RL-SG-5103	8/29/95	SiGe in alumina crucible	SiGe 3to1wt.%	#2+lid	melt&wetting
RL-SG-5104	8/31/95	SiGe in quartz	SiGe 3to1wt.%	#2+lid2	melt&wetting
RL-GE-5106	9/07/95	SiGe in quartz ampoule	Si ge 3yo1wt.%	#2+lid2	melt&wetting
RL-SG-5107	9/19/95	SiGe in quartz ampoule	SiGe 3to1wt.%	#3	melt&wetting
RL-SG-5108	9/21/95	SiGe in quartz ampoule	SiGe 3to1wt.%	#3+heater	melt+wetting
RL-SG-5109	9/26/95	SiGe in quartz ampoule#2	SiGe 3to1wt.%	#3+heater	melt+wetting
RL-SG-5110	10/05/95	SiGe in quartz ampoule#1+CaCl2	SiGe 3ti1wt.%	#3+heater	melt+no wet
RL-SG-5111	10/11/95	SiGe in quartz ampuole#1CaCl2	SiGe 3to1wt%	#3+heater	melt+no wet
RL-SG-5112	10/12/95	SiGe inquartz ampoule#1CaCl2	SiGe 3to1wt%	#3+heater	melt+no wet
RL-SG-5113	10/16/96	SiGe in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#3+heater	melt+no wet
RL-SG-5114	10/17/95	SiGe in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#3+heater	melt no wet
RL-SG-5115	10/18/95	SiGe in quartz ampoule#1CaCl2	SiGe 3to1wt.%	#3+heater	quench+no wet
RL-SG-5116	10/18/95	SiGe in quartz ampoule#1CaCl2	SiGe 3to1wt.%	#3+heater	quench+no wet
RL-SG-5117	10/24/95	SiGe in quartz ampoule#1 CaCl2	SiGe 3to1wt.%	#3+heater	quench+no wet
RL-SG-5118	10/24/95	SiGe in quartz ampoule#1CaCl2	SiGe 3to1wt.%	#3+heater	quench+no wet
RL- SG-5119	10/24/95	SiGe in quartz ampoule#1CaCl2	SiGe 3to1wt.%	#3+heater	quench+no wet
RL-SG-5120	10/25/95	SiGein quartz ampoule#1CaCl2	SiGe 3to1wt.%	#3+heater	quench+no wet
RL-SG-5121	10/25/95	SiGe in quartz ampoule#1CaCl2	SiGe 3to1wt.%	#3+heater	quench+no wet
RL-SG-5122	10/26/95	SiGe in quartz ampoule#1CaCl2	SiGe 3to1wt>%	#3+heater	quench+no wet
RL-SG-5123	11/08/95	SiGe in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#4 Concentrator	melt+no wet
RL-SG-5124	11/15/95	SiGe in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#5 concentrator	melt+no wet
RL-SG-5125	11/20/95	SiGe in quartz ampoule#2 CaCl2	SiGe 3to1wt.%	#5 concentrator	melt no wet
RL-SG-5126	11/28/95	SiGe in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#5 concentrator	melt+no wet
RL-SG-5126b	12/05/95	SiGe in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#5 concentrator	melt+no wet
RL-SG-5127	12/29/95	Ge in quartz ampoule	Ge	#6 concentrator	melt+wet
RL-SG-5128	1/02/96	Si in quartz ampoule	Si	#6 concentrator	melt+wet
RL-SG-5129	1/08/96	SiGe in quartz ampoule	SiGe 3to1wt.%	#6 concentrator	melt+wet
RL-SG-5130	1/16/96	SiGe in quartz ampoule2CaCl2	SiGe 3to1wt.%	#6 concentrator	melt+no wet
RL-SG_5131	2/05/96	Si in quartz ampoule-no susceptor	Si	#6 concentrator	non-melt temp.
RL-SG-5132	2/06/96	SiGe in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#6 concentrator	melt+no wet
RL-SG-5133	2/12/96	SiGe In quartz ampoule#2CaCl2	SiGe 3to1wt.%	#6 concentrator	melt+no wet
RL-SG-5134	2/13/96	SiGe in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#6 concentrator	melt+no wet

TABLE 1

SiGe Run Sheet

I.D.	Date	Objective	Composition	Hot Zone	Results
RL-SG-5135	2/20/96	SiGe in quartz anpoule #2CaCl2	SiGe 3to1wt.%	#6 concentrator	melt+no wet
RL-SG-5136	2/26/96	SiGe in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#6 concentrator	melt +no wet
RL-SG-5137	2/27/96	SiGe in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#6 concentrator	melt+no wet
RL-SG-5138	3/05/96	SiGe in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#6 concentrator	melt+no wet
RL-SG-5139	3/12/96	SiGE in quartz ampoule#2CaCl2	SiGe 3to1wt.%	#6 concentrator	melt+no wet
RL-SG-5140	3/21/96	SiGe in quartz ampoule#3CaCl2	SiGe 3to1wt.%	#6 concentrator	melt+no wet
RL-SG-5141	3/26/96	SiGe in quartz ampoule#3CaCl2	SiGe 50%wt.%	#6 concentrator	melt +no wet
RL-SG-5142	3/27/96	SiGe in quartz ampoule#3CaCl2	SiGe 50%wt.%	#6 concentrator	melt+no wet
RL-SG-5143	4/02/96	SiGe in quartz ampoule#3CaCl2	SiGe 50%wt.%	#6 concentrator	melt+no wet
RL-SG-5144	4/02/96	SiGe in quartz ampoule#3CaCl2	SiGe 50%wt.%	#6 concentrator	melt+no wet
RL-SG-5145	4/03/96	SiGe in quartz ampoule#3CaCl2	SiGe 50%wt.%	#6 concentrator	melt+no wet
RL-SG-5146	4/09/96	SiGe in quartz ampoule#3CaCl2	SiGe 50%wt.%	#6 concentrator	melt+no wet
RL-SG-5147	4/10/96	SiGe in quartz ampoule#3CaCl2	SiGe 50%wt.%	#6 concentrator	melt+no wet
RL-SG-5148	4/16/96	SiGe in quartz ampoule#3CaCl2	SiGe 50%wt.%	#6 concentrator	melt+no wet
RL-SG-5149	4/17/96	SiGe in quartz ampoule#3CaCl2*	SiGe 40%wt.%	#6 concentrator	melt+no wet
RL-SG-5151	4/23/96	SiGe in quartz ampoule#3CaCl2*	SiGe 40%wt.%	#6 concentrator	melt+no wet
RL-SG-5152	4/24/96	SiGe in quartz ampoule#3CaCl2*	SiGe 40%wt.%	#6 concentrator	melt+no wet
RL-SG-5153	4/30/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to1Wt.%	#6 concentrator	melt+no wet
RL-SG-5154	5/06/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to1wt.%	#6 concentrator	melt+no wet
RL-SG-5155	5/15/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5156	5/22/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5156	5/29/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5156	5/29/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5157	6/10/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5158	6/21/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5159	6/24/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5160	6/25/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5161	6/26/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5162	7/01/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5163	7/02/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5164	7/08/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5165	7/16/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5166	7/23/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5167	8/19/96	SiGe in quartz ampoule#3CaCl2*	SiGe 2to1wt.%	#6 concentrator	melt no wet
RL-SG-5168	8/26/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to1wt%	#6 concentrator	melt no wet
RL-SG-5169	8/27/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5170	8/28/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5171	9/03/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5172	9/09/96	SiGe in quartz ampoule#3sCaCl2*	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5173	9/10/96	SiGe in quartz ampoule#3CaCl2*	SiGe 2to1wt.%	#6 concentrator	melt no wet
RL-SG-5174	9/17/96	SiGe in quartz ampoule#3sCaCl2*	SiGe 2to8wt.%	#6 concentrator	melt no wet
RL-SG-5175	9/16/96	SiGe in quartz ampoule#3sCaCl2*	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5176	10/03/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5177	10/15/96	SiGe in quartz ampoule#3CaCl2*	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5178	10/22/96	SiGe in quartz ampoule#3CaCl2	SiGe 3to1wt%	#6 concentrator	melt no wet
RL-SG-5179	11/05/96	SiGe In quartz ampoule#3CaCl2	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5180	11/06/96	SiGe in quartz ampoule#3CaCl2	SiGe 3to1wt.%	#6 concentrator	melt no wet

TABLE 1

SiGe Run Sheet

I.D.	Date	Objective	Composition	Hot Zone	Results
RL-SG-5181	11/19/96	SiGe in quartz ampoule#3CaCl2	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5182	11/25/96	SiGe in quartz ampoule#3CaCl2	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5183	11/26/96	SiGe in quartz ampoule#3CaCl2	SiGe 3to1wt%	#6 concentrator	melt no wet
RL-SG-5184	11/27/96	SiGe in quartz ampoule#3CaCl2	SiGe 3to1wt.%	#6 concentrator	melt no wet
RL-SG-5185	12/02/96	SiGe in quartz ampoule#3CaCl2**	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5186	12/03/96	SiGe in quartz ampoule#3CaCl2**	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5187	12/10/96	SiGe in quartz ampoule#3CaCl2	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5188	12/11/96	SiGe in quartz ampoule#2CaCl2	SiGe 3to 1wt.%	#6 concentrator	melt no wet
RL-SG-5189	12/17/96	SiGe in quaetz ampoule#3CaCl2	SiGe 3 to1wt.%	#6 concentrator	melt no wet
RL-SG-5190	01/03/97	SiGe in quartz ampoule #3CaCl2	SiGe 3 to1wt.%	#6 concentrator	melt no wet
RL-SG-5191	01/07/97	SiGe in quartz ampoule #3CaCl2	SiGe 3 to 1wt.%	# 6 concentrator	melt no wet
RL-SG-5192	01/14/97	SiGe in quartz ampoule #3CaCl2	SiGe 3to1wt.%	Box furnace	melt no wet
RL-SG-5193	01/22/97	SiGe in quartz ampoule #3CaCl2	SiGe 3to1wt.%	Box furnace	melt no wet
RL-SG-5194	01/23/97	SiGe in quartz ampoule #3CaCl2	SiGe 3to1wt.%	Box furnace	melt no wet
RL-SG-5195	02/06/97	Float Zone	SiGe 3to1wt.%	Box furnace	
RL-SG-5196	02/14/97	Float Zone	SiGe 3to1wt.%	Box furnace	
RL-SG-5197	02/20/97	Float Zone	SiGe 3 to 1wt.%	Box furnace	
RL-SG-5198	02/28/97	Float Zone	SiGe 3to1wt.%	Box furnace	

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