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ABSTRACT

Two tube furnaces were used to prepare single 10-20 mm-diameter pendant drops of a molten alloy, Fe-75 w/o Si; one was inductively heated and the other, resistively heated. The liquidus temperature of this alloy is about 1350°C. The drops were formed in argon at the lower end of a 10 mm-diameter rod of the alloy positioned within the respective furnaces. After melting, the pendant drops were released into free fall spontaneously or by moving the rod upward rapidly.

The resistively heated furnace consisted of an insulated helical silicon carbide element that surrounded a mullite tube. The furnace can operate in air at temperatures of at least 1650°C. This furnace is compact, easily moved and repositioned and simple, safe and inexpensive to build and operate.

INTRODUCTION

In this informal report, we describe two procedures used at the University of Wisconsin-Madison to successfully produce drops of molten ferrosilicon with diameters in the range 10-20 mm. The starting material consisted of the 10 mm x nominally 100 mm rods of Fe-75 w/o Si supplied by Dr. Karl Forwald of Elkem Metals Company, Trondheim, Norway. We believe both procedures could be used for physical and chemical studies of the behavior of freely falling molten metallic globules. Straightforward quantitative studies such as the following should be possible:

- Globule cooling rates in both gaseous and aqueous media.
- Effects of supercooling and nucleation on granule quality.
- Generation of hydrogen and the hazards of its combustion and explosion.
- Initiation and suppression of vapor explosions.

EXPERIMENTAL

Four drop formation experiments were performed, two with induction heating and two with resistive heating. The heating and melting were done in an atmosphere of argon in all four experiments. Schematic diagrams of the heating arrangements are shown in Figures 1 and 2.

The induction melting was done with a 10 kW, 450 kHz unit with a coil about 3 cm in inside diameter placed with axis vertical in an argon-filled chamber. The coil was about 12 cm long and had a quartz tube inside. The ferrosilicon rod was suspended inside the tube with a spring steel clamp. No temperature measurements were attempted.

The resistive heating was done with a 4 ohm helical silicon carbide element powered with 110 volt AC controlled by a 60 ampere variable transformer. A mullite tube with 25 mm ID passed vertically upward through the helical element and contained an atmosphere of argon. The argon flowed upward from a one-inch steel pipe tee held beneath the heating element in which the mullite tube rested. The side arm held the argon inlet; the gas was forced to flow upward by a thin sheet of polyvinyl chloride (PVC) stretched and fastened across the lower end of the pipe tee. This thin PVC sheet provides an essentially nonintrusive rapid-opening closure as the falling hot drop melts through it.

The heating element and the mullite tube were insulated with firebrick. A horizontal viewing hole was drilled through the firebrick at the level of the hot zone to permit temperature measurements with an optical pyrometer. Although the maximum operating temperature recommended by the supplier of the heating element is 1650°C, we did not exceed 1550°C in either resistively heated experiment.

In three of the four experiments we used a graphite catcher for the molten drops after release; in the fourth we used a steel container with 57 cm-deep water.

RESULTS

The four ferrosilicon drop formation experiments are summarized in Table 1.

Table 1. Summary of Ferrosilicon Drop Releases. All melting was done in an argon atmosphere.

	Heating	Release by	Weights (g)		Diameter of	
Expt. No	Method	Shaking	Main Drop	Minor Drops	Main Drop (mm)*	Catcher
B-62-1-1	Induction	No	NR	NR	--	Graphite
B-62-1-2	Induction	No	3.98	0.25	13.4	Graphite
B-62-1-3	Resistive	Yes	6.15**	NR**	15.4	Graphite
B-62-1-4	Resistive	Yes	4.29	0.14	13.7	Water

* Assumes spherical drop with melt density = 3.2 g/cc.

** All drops were collected together

NR = Not recorded

Induction Heating Experiments

Experiment B-62-1-1

The first experiment was performed with one of the 10 mm x 100 mm ferrosilicon rods grooved around its circumference about 2 mm deep and about 10 mm from one end. The groove was intended to limit the drop diameter to about 10 mm. The rod was supported with the grooved end down just below the upper turn of the induction coil. When the power was increased, a small shower of about eight irregular luminous drops formed. Afterward, the tip of the rod had a smooth hemispherical shape below the groove. Apparently some melt had formed at the tip of the rod, but was not removed as a single drop. We did not attempt to weigh these drops.

Experiment B-62-1-2

We then inverted the same ferrosilicon rod and placed its (nongrooved) lower end at about the middle of the induction coil. Again, in the argon atmosphere, when the induction power was increased, a sizable luminous globule and several tiny drops detached spontaneously. The solidified large drop was flattened and had a few protuberances extending upward from the surface in several places. These protuberances appeared to have formed by ejection of melt through cracks or holes in a solidified outer shell. One such protuberance was also found near the lower tip of the rod from which the molten globule had detached.

The large globule weighed 3.98 g, while the tiny globules weighed a total of 0.25 g. If we use a density of 3.2 g/cm^3 for the molten alloy, the diameter of a 3.98 g spherical drop would be 13.4 mm. These values are entered in Table 1.

Resistive Heating Experiments

Experiment B-62-1-3

This was the first experiment performed in the resistively heated tube furnace shown in Figure 2. Before heating began, a new 10 mm x 100 mm ferrosilicon rod with a 2 mm-deep groove at the upper end was suspended from the midpoint of a steel rod by nichrome wire. The wire was wound around and fastened in the groove and then attached to the steel rod in a way such that the lower end of the ferrosilicon rod would be positioned midway within the hot zone of the tube furnace. The steel rod and its attached ferrosilicon rod were positioned away from the furnace during a 40 minute heatup period.

Once the furnace reached 1425°C, as read by the optical pyrometer, the ferrosilicon rod was hung vertically in the argon flow of the tube furnace by placing the steel rod transversely across the top of the furnace. When the furnace reached 1455°C two minutes later, the steel rod was abruptly shaken to release a drop; none fell. But ten minutes later, when the furnace had reached 1525°C the steel rod was shaken again; a luminous globule fell through the thin PVC sheet and then through air into the graphite catcher.

We could not differentiate between the solidified major and minor drops in this experiment because the frozen melt cracked into several pieces when we tried to remove it from the graphite catcher. We assumed that several minor drops may have coalesced with the large drop during freezing. The total weight of solidified material was 6.15 g, corresponding to a sphere of 15.4 mm diameter (again using the melt density of 3.2 g/cc).

The lower tip of the rod from which the drop was released showed a tapered eight-pointed star-like shape formed by the flow of melt during detachment. Its almost perfect symmetry was striking. There was a tiny protuberance of solidified melt near the base of one of the points of the frozen star.

Experiment B-62-1-4

This experiment was performed essentially identically to experiment B-62-1-3 with the exception that the catcher was changed from graphite to a steel container with 57 cm-deep water. The molten drops had a 30 cm fall path in air between the PVC foil at the bottom of the furnace and the surface of the water.

In this final experiment, a new ferrosilicon rod was placed in the argon flow of the tube furnace when its temperature was 1445°C. Eight minutes later, when the furnace temperature had increased to 1545°C, we shook the transverse steel rod. A luminous globule was seen to fall immediately afterward. Its entry into and fall through the water was uneventful.

After draining the water, we recovered a large black deformed leaf-like piece of solidified melt along with about seven one- or two-millimeter diameter black spheres. There were also a few black spherical particles. The major globule was flat, roughly elliptical, about 20 mm x 30 mm, and weighed 4.29 g, corresponding to a spherical drop of 13.7 mm in diameter (ρ = 3.2 g/cc). The remainder of the small debris weighed about 0.14 g.

The lower tip of the rod from which the melt detached in this experiment showed a somewhat tapered star-like pattern, much like that of experiment B-62-1-3, with the exception that a relatively large growth of solidified melt protruded below and to one side of the tip.

DISCUSSION

The brief scoping study described in this report indicates that 10-20 mm diameter drops of molten ferrosilicon can be generated by two techniques currently available at the University of Wisconsin-Madison: induction and resistive heating. By detaching a pendant drop of the molten alloy from a rod of the solid, it seems from Table 1 that reasonably reproducible drop diameters can be achieved for a variety of physical and chemical studies of value to the ferroalloy industry. Moreover, it is likely that drop diameters can be varied in this desired range by changing the diameter of the rods from which the pendant drops are formed and detached.

It should be noted, however, that in this containerless procedure the molten drops will always be formed with little or no superheat. This is, of course, a necessary condition of their production from an essentially melt-solid equilibrium temperature at the drop-rod interface. This situation may provide the important advantages for the desired studies of (a) a known temperature of release of the drop fixed by the melting temperature of the rod and (b) a minimum of internal heat to be removed from the drop before solidification can be initiated. Characteristic (a) should be valuable for both cooling rate and supercooling studies by providing a known starting point for drop release. Characteristic (b) should be useful when carrying out falling drop studies with limited fall distances.

If superheat of the melt is important, it would still be possible to use the two available heating techniques described here: via containerless levitation melting with the induction heating system and either a tilting or bottom-drain crucible with the resistive heating system.

The resistive heating unit described here provides several advantages over the induction heating system:

- Small size and weight. The unit is essentially portable and may easily be positioned where needed, e.g., above tall drop tubes.
- Inexpensive. The entire furnace costs less than $300. No elaborate electrical and cooling water systems are needed.
- Readily replaced and modified. The silicon carbide elements are commercially obtainable at relatively low cost in a variety of standard configurations and sizes. Also, special designs usually can be accommodated.
- Safe. The furnace does not generate excessive electrical and magnetic fields, does not require elaborate shielding, and does not require official certification and testing prior to operation.
- High maximum temperatures. The silicon carbide heating elements are rated for operation up to 1650°C in air. This should be adequate for most ferroalloy and silicon melting. And, although some extra temperature margin is probably desirable, the silicon carbide elements might even be used to produce drops of pure iron.

CONCLUSIONS

With a few preliminary experiments performed with internal funding we have shown that single drops of molten ferrosilicon alloy with 10-20 mm diameters can be produced reproducibly with two heating techniques available at the University of Wisconsin-Madison. Moreover, both techniques should be applicable to prepare drops of other ferroalloys, silicon and perhaps iron.

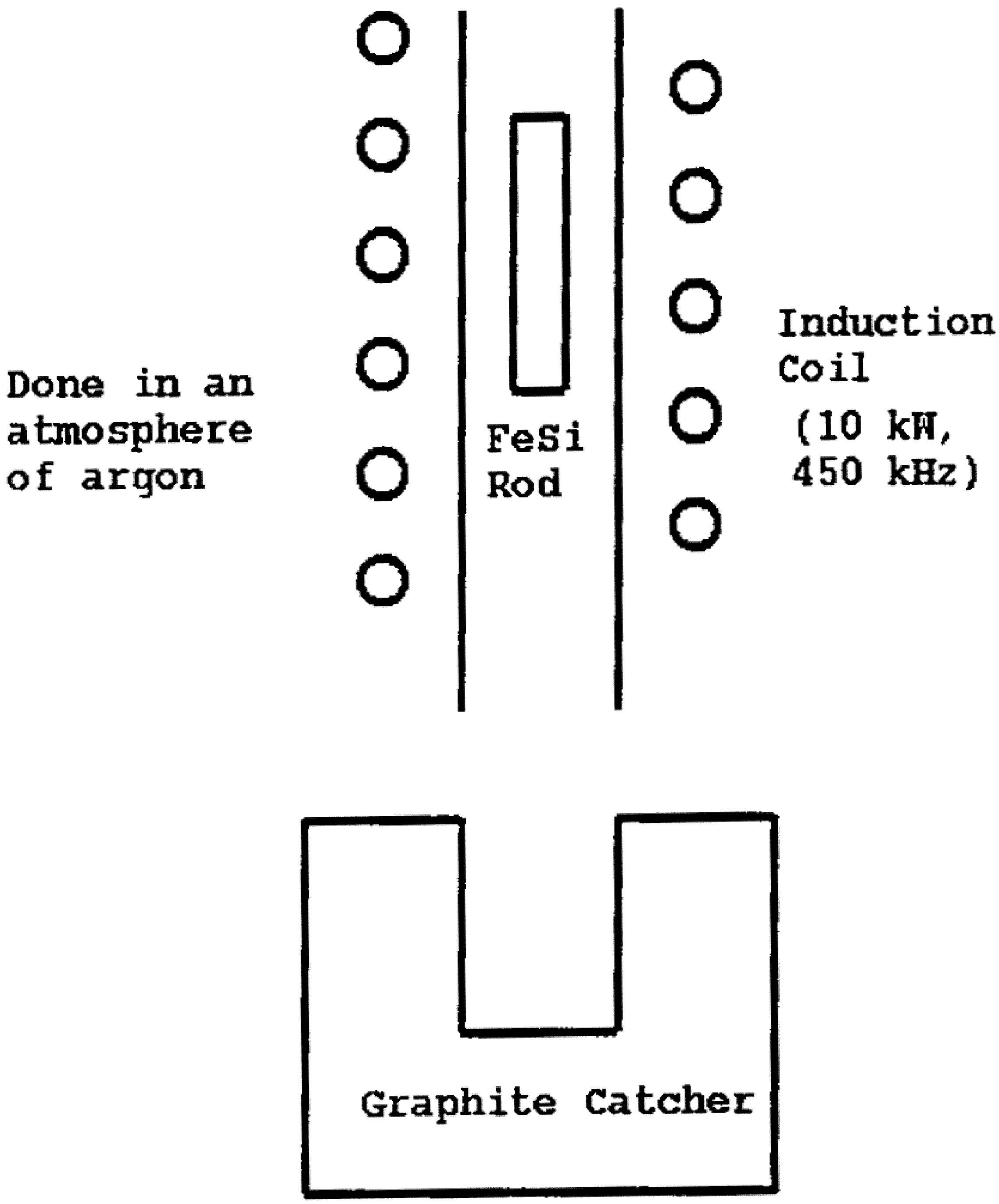


Figure 1. Schematic diagram of the induction heating apparatus used to prepare molten ferrosilicon drops.

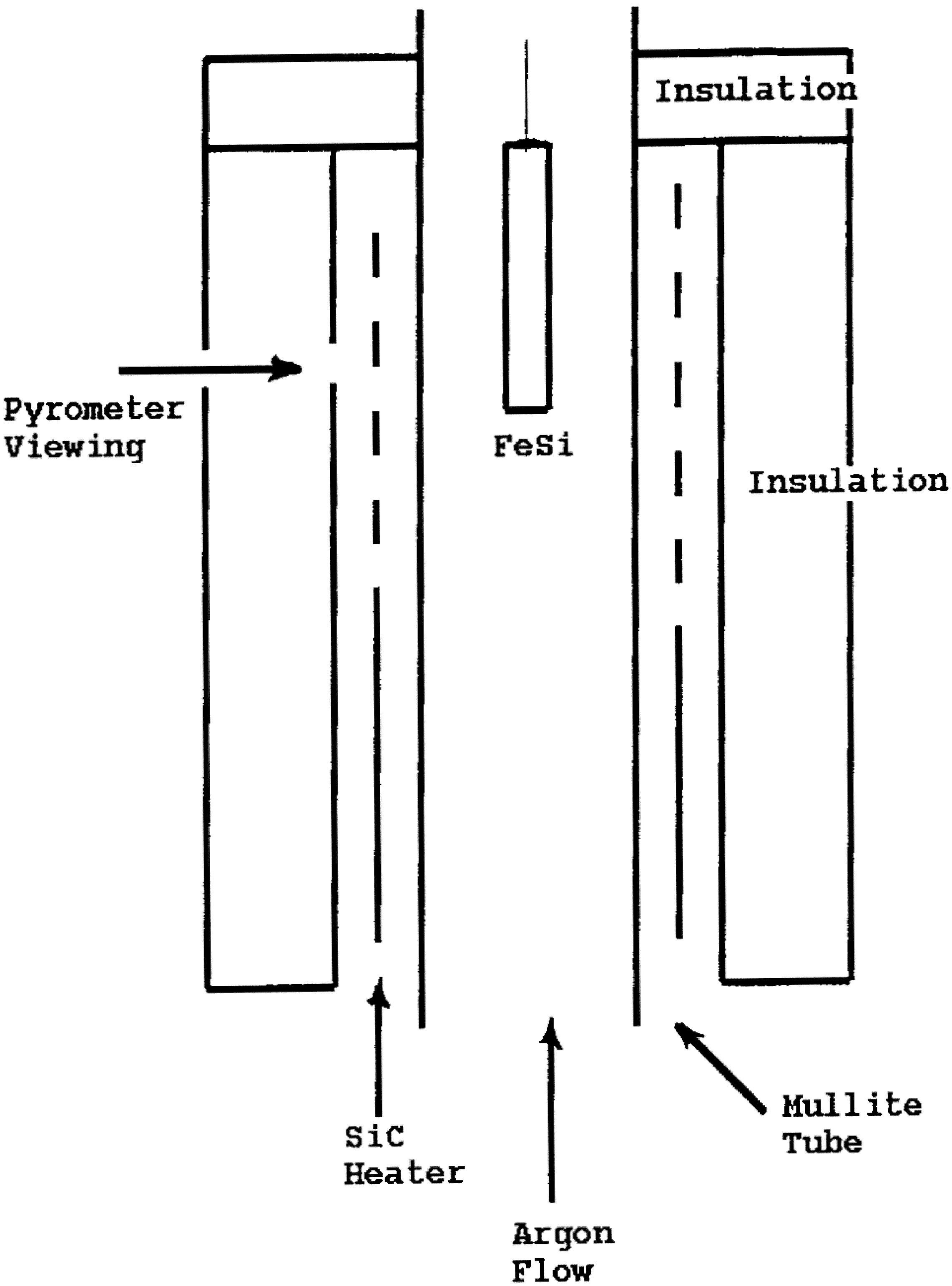


Figure 2. Schematic diagram of the resistively heated tube furnace used to prepare molten ferrosilicon drops.

Appendix A

Photographs of the Ferrosilicon Rod Melting Experiments

In this appendix, we present a few photographs of the apparatus used for generating single 10-20 mm drops of molten ferrosilicon. We also include photographs of the rods and frozen materials recovered after the experiments.

The rods and frozen materials have been archived at the University of Wisconsin-Madison, but could be made available for further examination if desired.

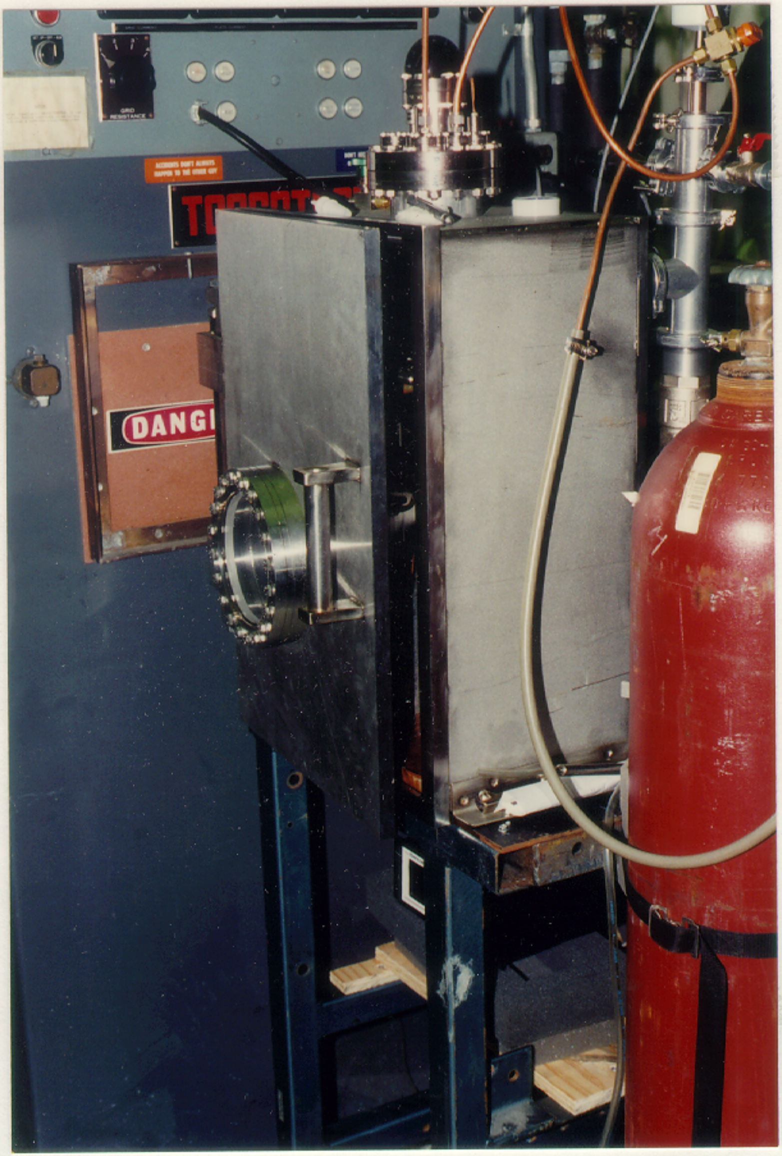


Figure A-1. External view of vacuum chamber (center), power unit (left) and argon supply (right) used for induction melting experiments. (B-68-3)

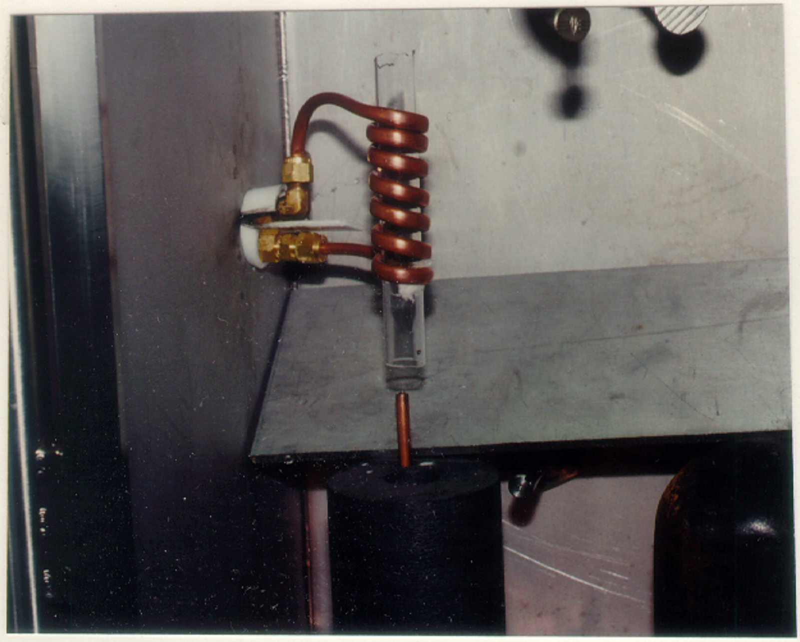


Figure A-2. Interior of vacuum chamber showing induction coil, quartz tube and graphite catcher (below). (B-68-2)

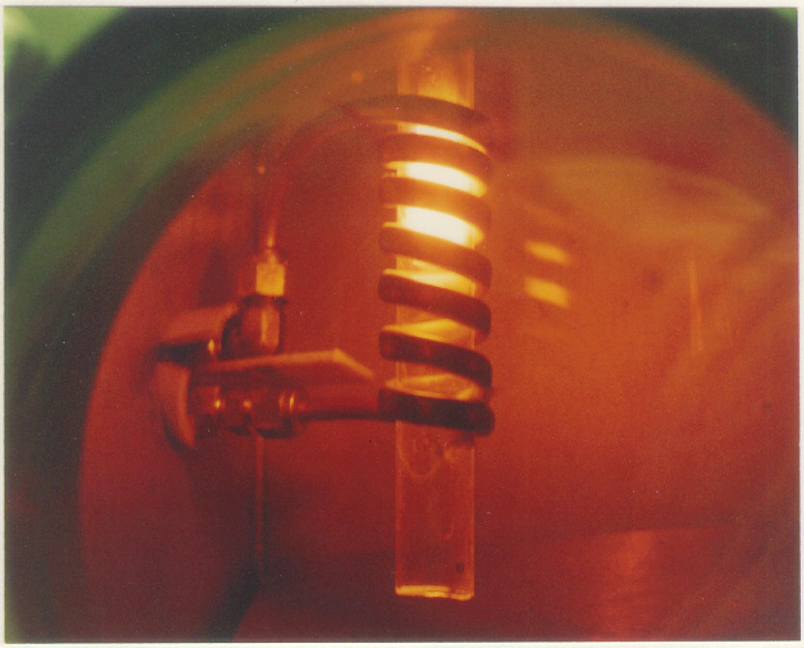
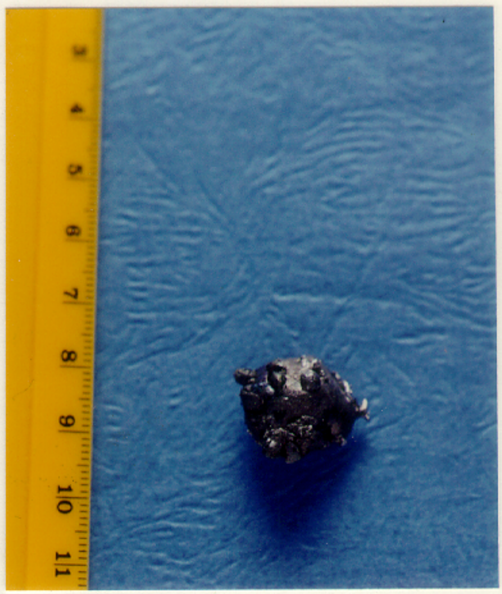


Figure A.3. Photograph of ferrosilicon rod just before drop detached in experiment B-62-1-2. (B-68-4)

a. Grooved ferrosilicon rod before the experiments. (B-62-1b)

b. Irregular droplets formed in experiment B-62-1-1. Groove is at the bottom of the rod. (B-62-1-1)

c. Large and smaller drops formed in experiments B-62-1-2. Groove is now at the top of the rod. (B-62-1-2c)



d. Enlarged view of the large drop formed in experiment B-62-1-2. (B-62-1-2i)

Figure A-4. Materials related to induction melting of ferrosilicon rods.

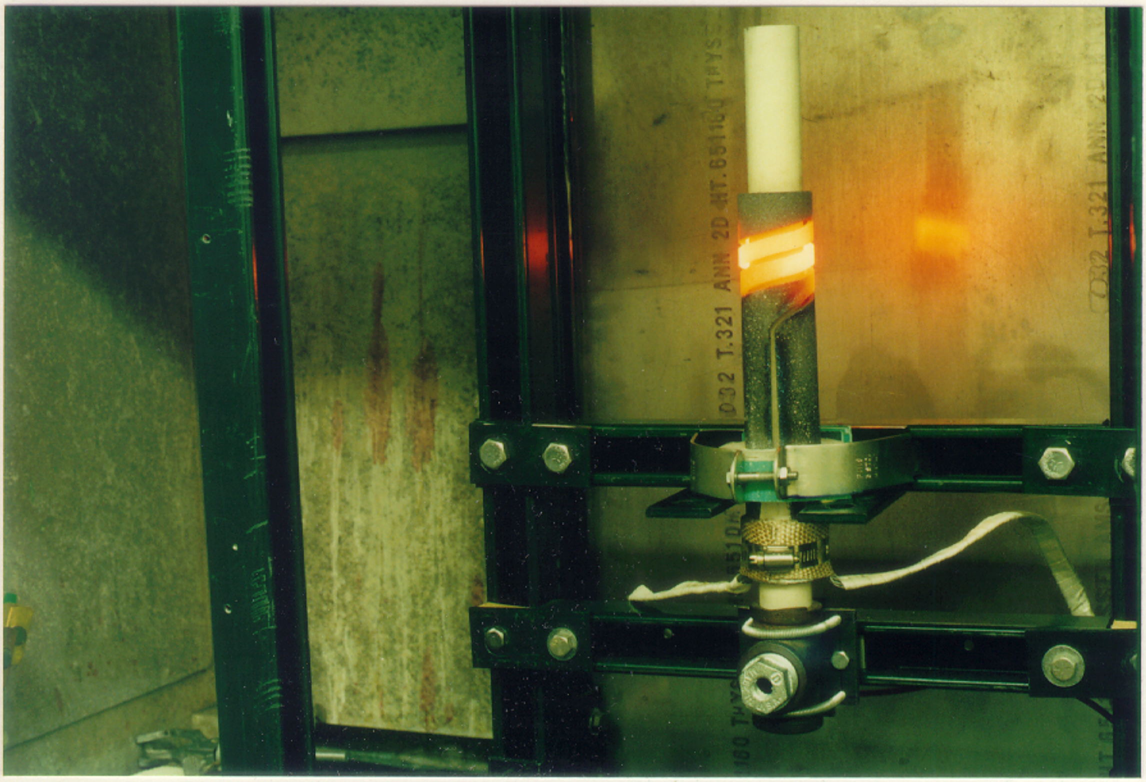


Figure A-5. Test of cylindrical silicon carbide resistive heating element without insulation. Note mullite tube that passes vertically through the element. The tube rests on the steel pipe tee. Argon entry port faces forward. Drop falls through the bottom of the tee. (B-72-1-2)

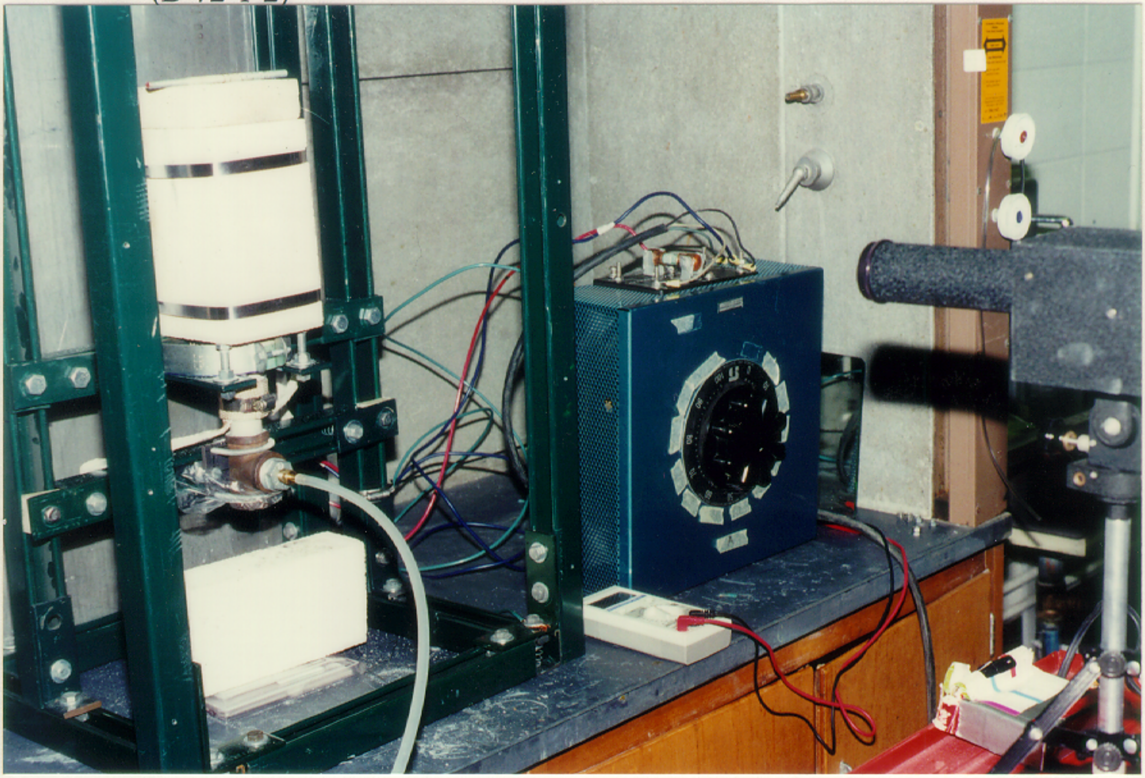


Figure A-6. Resistively heated tube furnace set up for operation with firebrick insulation and argon line in place. Thin polyvinyl chloride sheet may be seen stretched across bottom of pipe tee. Variable transformer is at the center and optical pyrometer is at the right. (B-72-1-7)

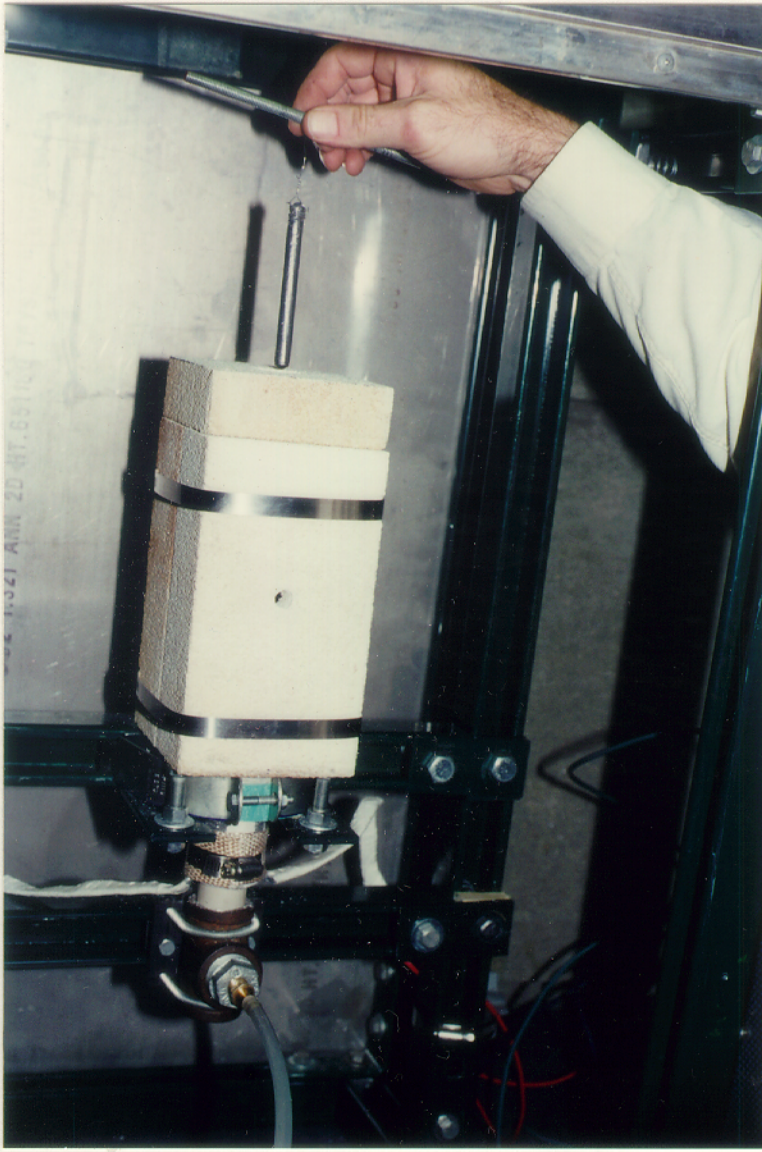


Figure A-7. Suspension of ferrosilicon rod being checked before an experiment with the resistively heated tube furnace. (B-72-1-5)

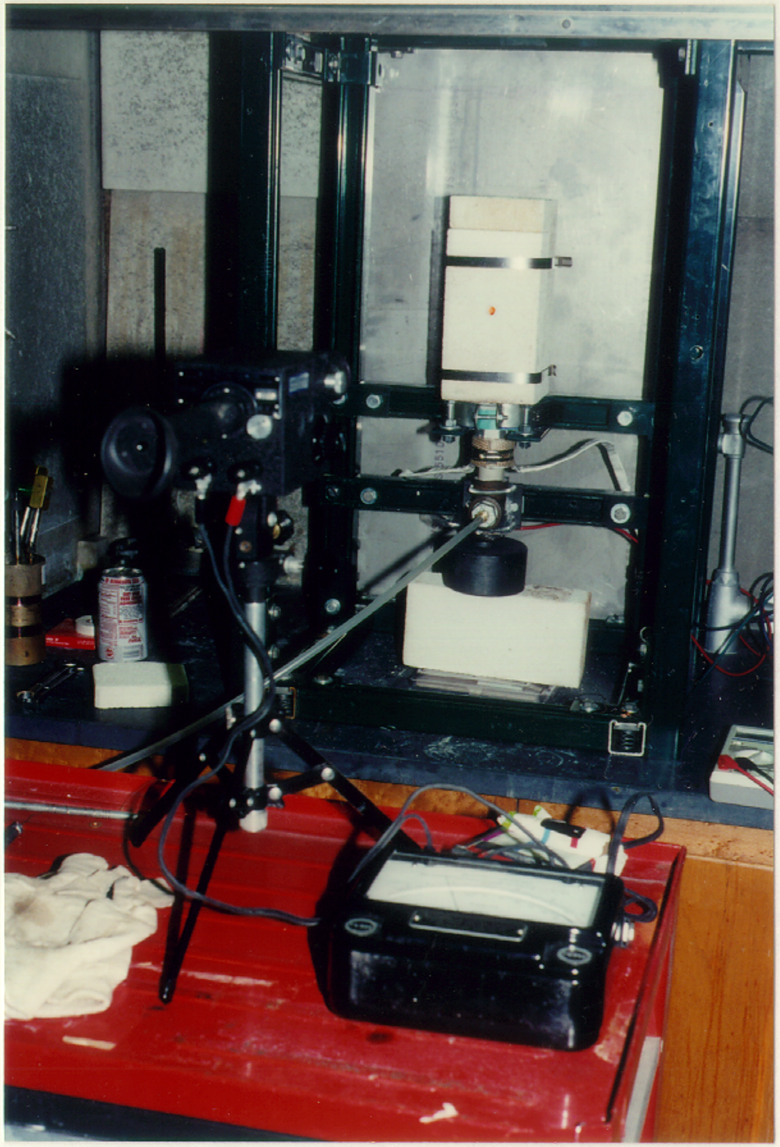


Figure A-8. Resistively heated tube furnace being brought up to temperature for experiment B-62-1-3. Graphite catcher is in place beneath the furnace. Optical pyrometer (foreground) is trained on the viewing port (center of firebrick insulation). (B-72-1-6)

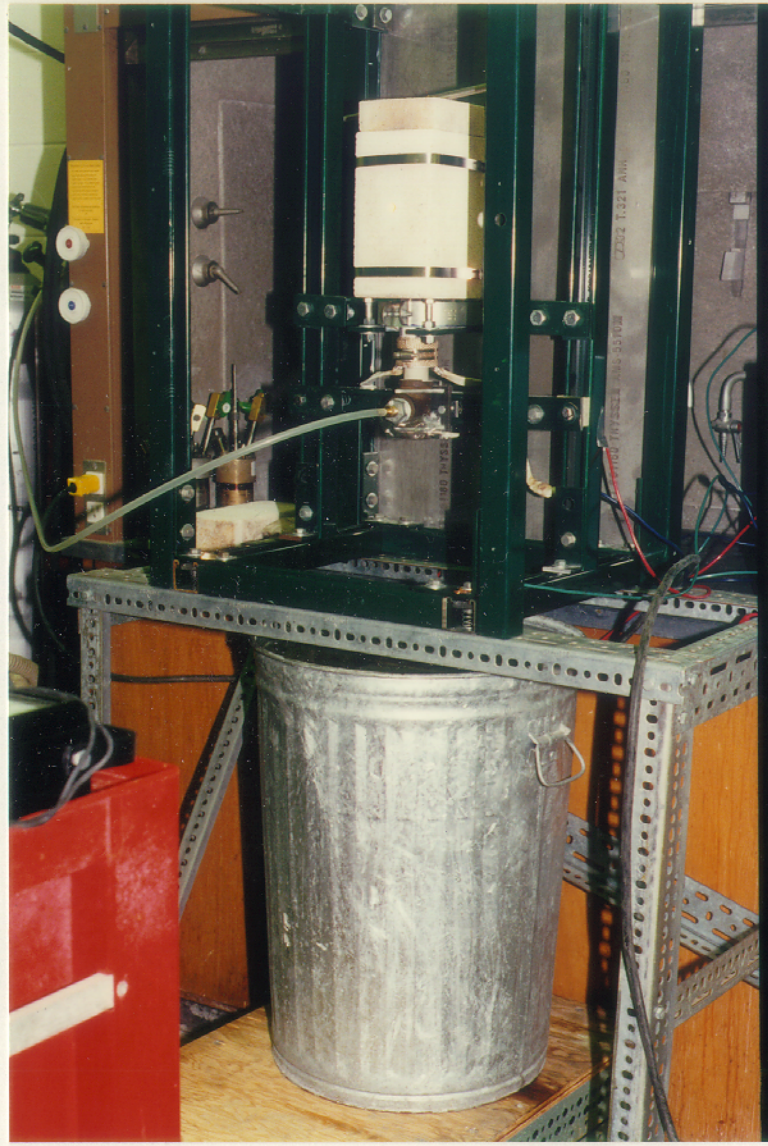


Figure A-9. Setup used for ferrosilicon rod melting experiment B-62-1-4. Furnace has been positioned above water-filled steel chamber. (B-62-1-4b)

a. Material from experiment B-62-1-3. It was broken during removal from the graphite catcher. Nichrome wire used to suspend the rod is visible at the top of the photograph. (B-62-1-3a)

b. Tip of the rod from which the molten ferrosilicon detached in experiment B-62-1-3. Note tapered star-like tip small proturberance of solidified melt at the left (B-62-1-3e)

c. Frozen material recovered from the water filled steel chamber in experiment B-62-1-4. (B-62-1-4g)

d. Tip of the rod from which the molten ferrosilicon detached in experiment B-62-1-4. Note the partial tapered star-like tip with large proturberance of frozen melt at the left. (B-62-1-4j)

Figure A-10. Products from resistively heated ferrosilicon rod melting experiments. Diameter of the rods is 10 mm in all photographs.