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UWFDM-1288

Proceedings of the Ninth International Ferrous Congress (INFACON 9), Quebec City,
June 3-6, 2001, pp. 338-351. Published by the Ferrous Association, Washington, DC.

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Steam Explosions of Single Drops of Molten Silicon-Rich Alloys

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ABSTRACT

Commercial quantities of ferrosilicon alloys and silicon metal in the form of granules are available. They are produced in water granulation, a process involving the risk of large-scale steam explosions. To learn about these steam explosions, we have investigated single drops of molten alloys when released into water.

When single drops of molten ferrosilicon alloy with 75 wt.% Si (FeSi75) or silicon metal at its liquidus or melting temperature were released into cold, room temperature or hot water, spontaneous explosions never occurred. We could, however, initiate steam explosions by exposing the drops to upward-directed pressure transients generated in the water with an encapsulated impactor.

For water at room temperature, we determined (a) the strength of the pressure transients needed to initiate the steam explosion of a single drop, and (b) the range of fall depths over which the drop can be triggered to explode with this transient. When varying the water temperature we learned that the triggered explosions became less vigorous as the temperature increased. The explosions became more vigorous also

when the Al and/or Ca content in the molten drop decreased.

Photographic images of the bubbles generated during the steam explosions indicate that the maximum energies released correspond to about 1% to 2% (dependent on drop size) of the total enthalpy of the molten ferrosilicon at its liquidus temperature. During the explosions, tiny bubbles of a permanent gas, presumably hydrogen, were generated.

Video images show that ferrosilicon drops with diameters between 3 mm and 11 mm fall through water at about 0.5 m/s. Shutter wheel photographs confirm these fall rates and also indicate the onset of solidification of the drops and show that steam explosions can occur in partially solidified drops.

1.0 INTRODUCTION

Silicon metal and ferrosilicon have important uses in the chemical and metallurgical industries. An increasing share of the products is supplied as granulated material. This is due to both ease in handling in various plant operations and better yield in the customer's process, e.g. alloying in steel production.¹ The size range

of the granulated material can vary a lot, mainly dependent on metal composition. Typically the granules are supplied to customers in the size from 3 to 15 mm. In the granulation process large quantities of granules are generated by breaking up a stream of molten metal into globules that are sprayed into water and quenched. The resulting solidified granules normally resemble confectioner's lozenges, have smooth surfaces and largely have the desired sizes. Unfortunately, the array of molten metal drops that forms in the water during granulation sometimes becomes the familiar "coarse premixture" that precedes a large and devastating steam explosion.^{1,2,3}

In order to understand the conditions that give rise to steam explosions of large numbers of drops of melt sprayed into water, we have begun a laboratory study of the release of single drops of molten silicon and of a molten ferrosilicon with 75 wt.% silicon (FeSi75) into water at several temperatures. In our early experiments, we discovered that the falling drops do not explode spontaneously after release into water as do drops of Sn, Bi and Pb.^{4,5,6} We also found that we could cause the drops to explode by introducing pressure transients into the water with a submersible mechanical impactor.⁷ Measurements with a tourmaline transducer in side experiments allowed us to determine the threshold peak pressures needed to initiate the explosions. Because of the ease of positioning the impactor, we also were able to determine the ranges of water depths over which the drops could be triggered with these threshold transients.

Our primary diagnostics for these experiments involved video imaging and time-exposed photography with two 35 mm cameras, one of which viewed the experiment through a rotating shutter wheel. Analyses of these images allowed us to determine the strength of the triggering pulse applied to the drops and also the time and location of the explosions. Also, these analyses provided us with estimates of the maximum volumes of the transient steam

bubbles formed during the explosions, and thus the pressure-volume (PV) energies they released. Also, the video and shutter wheel images allowed us to determine fall distances vs. time for the drops and in some cases we could identify the time and depth when an unexploded drop began to solidify. These images showed that a drop could explode even when partially solidified.

2.0 EXPERIMENTAL

2.1 TOWER

The apparatus was mounted in a 3 m-tall tower assembled from commercial steel U-beams. The tower was divided into three segments: the upper meter held the furnace and drop release apparatus; the water chamber was placed in the central meter; and there was a spill control container in the lower meter.

The furnace and drop release apparatus were mounted in a welded steel framework that was suspended on a trolley system attached to the top crossbeams of the tower. This trolley system allowed us to bring the furnace to the desired temperature with the furnace at the left side of the tower before an experiment while we prepared the water chamber at the right side of the tower. Shortly before the experiment, the heated furnace was moved to the right and centered above the water chamber. After the experiment, the furnace was moved to the left again to allow us to retrieve the quenched materials from the water before the furnace had cooled completely.

2.2 WATER CHAMBER

The water chamber is made of optically clear 12.7 mm-thick polycarbonate sheets with cemented seams. It is 1m-tall, open at the top, with 0.3 m ID square cross section. It is normally filled to the brim with 90 L of deionized water. The chamber is strengthened by four sets of horizontal reinforcing crossrods, one each at the top

and bottom and two placed 0.33 m apart vertically.

2.3 SILICON CARBIDE FURNACE

We used the tube furnace with a helical silicon carbide resistive heating element and firebrick insulation described previously.⁷ According to its manufacturer (I Squared R Element Company, Akron, New York), this element can operate in air at a maximum temperature of about 1675 °C. This is more than adequate to produce drops of molten silicon and FeSi75 alloy that have melting and liquidus temperatures of 1410 °C and about 1320°C, respectively. The furnace tube is made of mullite, 25 mm ID, 32 mm OD and 382 mm long and is centered within the silicon carbide helix, both with axes vertical.

2.4 PREPARATION OF DROPS OF THE MOLTEN METALS

As reported previously,⁷ the drops of molten alloy were prepared by lowering the bottom end of a nominally 100 mm-long rod of silicon or FeSi75 into the hot zone of the furnace preheated to ~1550 °C or ~1450 °C, respectively. While the rods were being heated, the furnace was flushed downward with a mixture of argon +1% hydrogen prepared from commercial grade gases. This atmosphere was extended almost to the water surface with an extension tube mounted beneath the lower end of the furnace tube. A pendant drop of molten metal formed on the lower tip of the rod and detached spontaneously after several minutes. This drop then fell about 400 mm through the argon-hydrogen mixture before entering the 1 m-deep water below. After the first drop had fallen, the rod could be lowered appropriately to allow another drop to form and detach several minutes later while the furnace was still hot. This procedure could be repeated several times depending on the initial length of the rod.

It should be emphasized that the technique described above produces pendant drops that

are inherently at the temperature of the solid-liquid interface of the metal; that is, all experiments were performed with melt subcooled below the melting or liquidus temperature.

A diagram of the apparatus is shown in Figure 1.

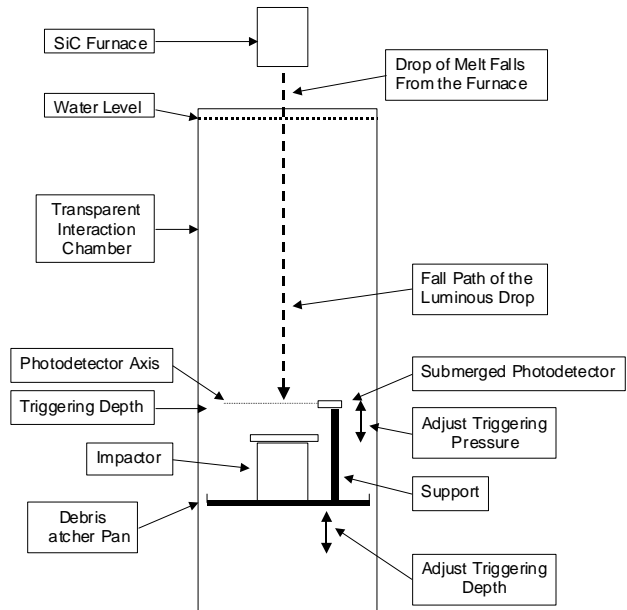


Figure 1. Arrangement for triggering the steam explosion of a luminous molten drop with a submerged photodetector and a pneumatic impactor.

2.5 MATERIALS

The drops of molten silicon and FeSi75 were prepared from rods provided by SINTEF Materials Technology, Trondheim, Norway. These rods had diameters of 10 mm and were nominally 100 mm long. The drops produced from these rods were nominally 9 mm in diameter. Although the rods of both metals had Si and Fe fractions typical of commercial granules, the starting materials were highly pure. Analyses showed other metals always to be <0.01 wt. %. Some of the rods were intentionally alloyed with small amounts of Al and/or Ca (both <0.5 wt. %).

released into water at three temperatures: cold, room temperature and hot (about 7 °C, 20 °C and 80 °C, respectively).

2.6 IMAGING

We used video imaging to determine fall and triggering distances and times, the occurrence and approximate magnitude of the explosions and the duration of luminous and non-luminous phenomena associated with the drops both before and after they were triggered. The video images also provided information about bubble behavior and water motion that accompany the explosions. The video images were recorded with a Sylvania Model HQ-VHS camcorder, either by the self-luminosity of the drops in a darkened room or with reflected light. The records were analyzed in continuous or frame-by-frame modes with a Mitsubishi HS-UG681 video cassette recorder (VCR) with a time resolution of 30 f/s (0.033 s per frame).

We also produced photographic streak images of the fall and explosion of the incandescent drops of molten silicon and FeSi75 in a darkened room with open shutter, time-exposed 35 mm photography (Minolta X-370N camera, 50 mm, 1:1.7 lens, ISO 200 film, f/8 to f/16). The camera was placed 0.38 m from the water chamber.

We also placed another 35 mm camera (Ricoh XR-10 with a 50 mm, 1:2 lens using ISO 200 film and a lens aperture of f/8) about 2 m from the water chamber to simultaneously record a second time-exposed image of the luminous drops. This camera viewed the falling drops through a rotating five-bladed shutter wheel to produce “dotted” tracks for the luminous globule of melt; time resolution was 12 chops per second (0.083 s per chop). Distances on the images were calibrated by placing a pair of vertical fiducial rods with known separations in the fields of view of the video and 35 mm cameras.

2.7 INITIATION OF INTERACTIONS WITH PRESSURE TRANSIENTS

We did not experience any spontaneous explosions with drops of either molten silicon or FeSi75. Therefore we initiated their explosions with pressure transients applied to the water with a submerged mechanical impactor. We developed three devices during this work: Impactor 1, driven with an electrical solenoid;⁷ Impactor 2, driven by a pneumatic piston; and Impactor 3, a device that employs an air-driven slug. Each device generates a pressure transient in the water by the impact of a metallic element on the underside of the steel cover of a water-tight canister in which the impactor is mounted.

To measure the pressure transients generated by the impactors, we used the tourmaline transducer-oscilloscope combination described previously.⁷ For these measurements, we placed the surface of the impactor at a depth of 400 mm in the water, and the tourmaline transducer 100 mm above at a depth of 300 mm. The peak pressures of the transients generated by Impactors 1, 2 and 3 with this positioning are nominally 0.13 MPa, 0.30 MPa and 1.96 MPa, respectively.

2.8 TRIGGERING WITH A SUBMERGED PHOTODETECTOR

The impactor is fired when a luminous drop of silicon or FeSi75 passes a submerged photodetector; its placement in the water at various distances above the impactor is indicated by the upper double-headed arrow in Figure 1. The placement of the impactor at various depths in the water is indicated by the lower double-headed arrow in Figure 1.

2.9 HYDROGEN COLLECTOR

The hydrogen collection concept is based on experiments in which single drops of a molten Al-Li alloy were released into water.⁸ In those experiments, the gases were analyzed by mass spectrometry. In the

current experiments, the hydrogen bubbles generated by the steam explosion of a single drop of melt were gathered in a conical collector at the top of the water chamber and their combined volume was measured by displacement of water contained in a graduated cylinder. After the drop of melt falls into the water at the start of an experiment, the sliding funnel above the conical collector closes quickly, as shown in Figure 2.

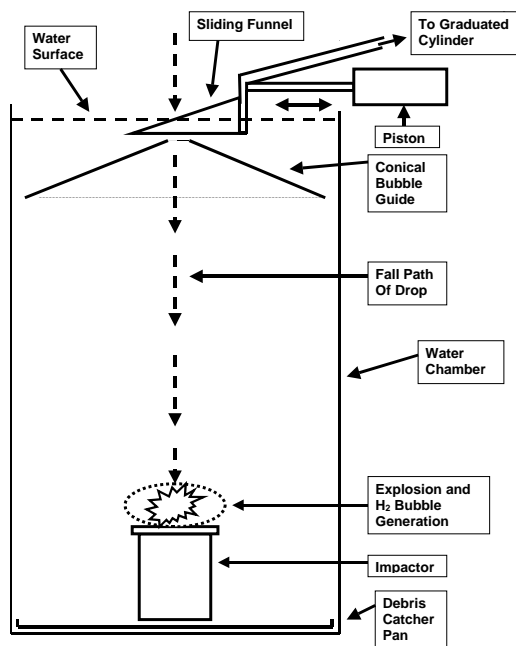


Figure 2. Schematic drawing of the hydrogen collector and its relationship to the explosion of a typical drop.

2.10 EXAMINATION OF THE SOLIDS

The weights of the molten drops released into the water were determined by the differences between the weights of the rods before and after the experiments. The granular debris recovered after an experiment, which included solidified drops that had not exploded and the coarse and fine material from drops that had completely or partially exploded, was also weighed. Both the rods and the granular materials were imaged with 35 mm photography.

We discovered that a large fraction of the original weight of each drop that exploded (10% to 20% for FeSi75 drops; sometimes as much as 60% for silicon drops) remained suspended in the water as a colloid that did not settle for many hours. We attempted to investigate this material (a) with inductively coupled plasma mass spectrometry (ICPMS)⁹ by directly injecting the colloid-containing water into the spectrometer, (b) by weighing the material that remained after known amounts of the colloid-containing water had been boiled dry, and (c) by X-ray diffraction of these dry residues.

2.11 SIEVE ANALYSES

We did sieve analyses on some of the debris resulting from the various melt-water interactions. The analyses were done by hand sieving and weighing of the fractions; unfortunately this procedure is not very exact for small samples, because losses up to 15% of the mass were detected. This makes it difficult to be too conclusive, but still general trends are provided, and there is a substantial difference between coarse-fragmented debris and debris resulting from a steam explosion.

3.0 RESULTS

3.1 CHARACTERISTICS AND BEHAVIOR OF THE DROPS

In this work, luminous drops of the molten FeSi75 alloy at its liquidus temperature of about 1320°C and of molten silicon at its melting temperature of 1410°C were released into 90 L of deionized water contained in a transparent 1 m-tall chamber. The water was at three specified temperatures for the ferrosilicon drops and at room temperature for the silicon drops. When these drops fell undisturbed through the water, spontaneous explosions never occurred; instead, the drops froze benignly, producing lozenge-shaped particles. The intact drops generated a few hydrogen bubbles as they fell through the water and

after they landed on a stainless steel plate placed at the bottom of the chamber.

The weights of the drops that detached spontaneously from the FeSi75 rods were quite reproducible-- 1.34 ± 0.10 g, averaged over 38 unexploded globules. These weights yielded drops with equivalent spherical diameters of 9.2 mm, calculated using a density for the molten alloy of 3.2 g/ml.¹⁰ The drops that detached from the 10 mm-diameter rods of silicon were less reproducible, weighing 0.8 g to 1.0 g. These weights corresponded to equivalent spherical diameters of 8.4 to 9.1 mm, as estimated from the density of molten silicon, 2.54 g/ml. For simplicity, we refer to the diameters of both sets of drops as 9 mm.

From the behavior of our photodetector (filtered to operate in the near infrared) drops of molten silicon seem to have much higher luminosity than drops of molten ferrosilicon--perhaps several times greater than purely thermal emission, $(T_{Si}/T_{FeSi})^4$ (the ratio of absolute release temperatures, 1683 K (1410 °C), for the silicon drops vs. 1593 K (1320 °C) for the ferrosilicon drops). We believe that a thin shell of solid silicon with high emissivity ($e_{solid} = 0.55$) coats each drop immediately after it enters the water, causing it to appear much brighter than the completely molten drop with low emissivity ($e_{liquid} = 0.26$). But an analogous shell of solid probably cannot form quickly on the ferrosilicon drop because the binary melt must first pass through a mushy state during which there is little change in emissivity. Knowledge of this behavior is important for estimating radiative heat transfer during the water granulation of molten silicon and ferrosilicon.

3.2 INITIATION OF EXPLOSIONS WITH PRESSURE TRANSIENTS

We used the tourmaline transducer with oscilloscope⁷ to record the pressurizations generated in the water (a) when the impactor was activated alone, before the drop was released, or (b) during the triggered

explosive interaction, after the drop was released. Often, the initial portions of the traces recorded in (b) show the triggering transients recorded in (a) clearly separated from the pressurizations generated later by the explosive interactions. A trace of this sort for a 9 mm-diameter drop of molten silicon is shown in Figure 3, where the pulses PT 1 and PT 2 are essentially those generated by the impactor alone and pulse PT 3 was produced by the explosion of the drop. In this experiment, the transducer was 120 mm from the centerpoint of the top of the impactor and 90 mm from the explosion. For comparison with our earlier report,⁷ the peak pressures of PT 1, 2 and 3, normalized to 100 mm using the $1/r$ relationship, were 1.57 MPa, 0.44 MPa and 2.19 MPa, respectively. By analogy with the behavior of drops of molten iron oxide,¹¹ we attribute pressure transient PT3 in Figure 3 to the collapse of a bubble generated by the steam explosion of the drop of molten silicon.

3.3 PHOTOGRAPHY OF THE TRIGGERED INTERACTIONS

In Figures 4a and 4b, we compare typical time-exposed photos of explosions of single 9 mm-diameter drops of molten ferrosilicon and silicon, both triggered with Impactor 2 in water at room temperature. Both photographs were recorded in a darkened room with an open-shutter 35 mm camera by the self-luminosity of the drops and the hot incandescent melt particles produced during the interactions. Although the images of the explosions are similar, a much larger triggering pressure (about 2.1 MPa) was required to initiate the explosion of the drop of molten silicon than was required for the ferrosilicon drop (about 0.6 MPa).

In Figure 5, we show the time-exposed image of the triggered coarse fragmentation of a drop of molten FeSi75 in hot water (about 80 °C). Apparently, the drop first

broke into a clump of large, luminous fragments. These particles then seemed to become enclosed in hydrogen (plus steam) bubbles that slowly rise together and ultimately break through the surface of the water. Inside each bubble there seems to be a particle burning luminously in a self-sustained fashion. With time-exposure photography, this slow rise of the clump of bubbles produces “seaweed-like” images of the luminous burning particles shown in Figure 5.

3.4 VIDEO IMAGING OF THE INTERACTIONS

Video images of the triggered interactions were recorded with either reflected light or in a darkened room during open-shutter photography. The video images, recorded at 30 frames/second, are used for observing and timing various aspects of the interactions that can last from fractions of a second to many minutes, e. g., fall distances vs. time, times to explode or quench, and the generation of both the transient steam bubbles and the permanent hydrogen bubbles.

3.5 THRESHOLDS FOR TRIGGERING STEAM EXPLOSIONS

We determined the thresholds for initiating steam explosions of 9 mm diameter drops of FeSi75 and silicon released into water at room temperature. We estimated both the magnitudes of the triggering transients needed and the range of depths over which the explosions could be initiated with these transients. The transients for both sets of experiments were generated by the pneumatic piston-driven Impactor 2. The results are summarized in Table 1.

Several conclusions can be drawn from Table 1: (a) the pressure transients required to initiate the explosions of drops of molten silicon are about 50% stronger than those required for molten ferrosilicon, (b) drops of molten ferrosilicon remain explosive over much longer fall depths (150 mm to at least 785 mm) than the silicon (only between 150 mm and 400 mm), and (c) ferrosilicon drops cannot be triggered to explode in water shallower than about 150 mm. (Our video records indicate that the air bag that forms around the drop as it enters the water does

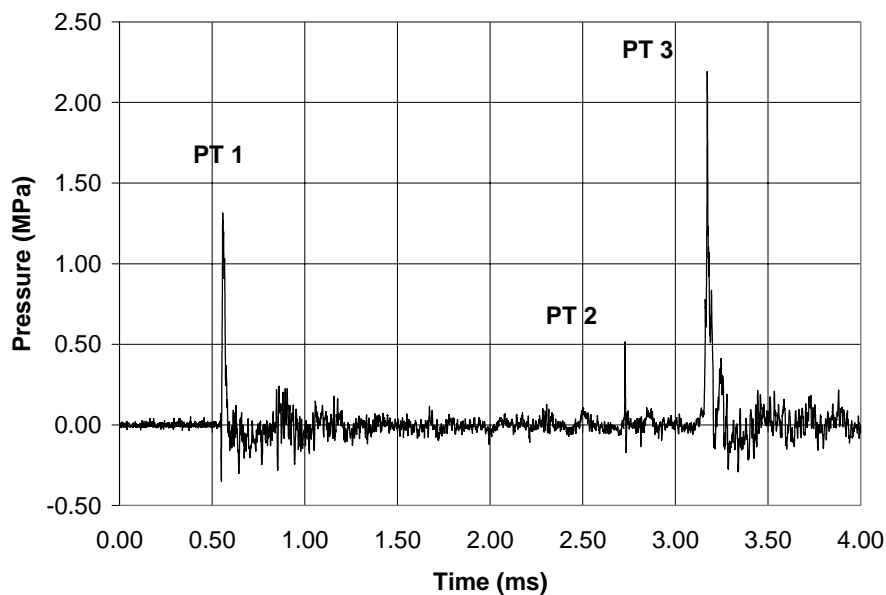


Figure 3. Pressure-time trace recorded during the steam explosion of a 9-mm diameter drop of molten silicon. The triggering pressure transient was generated by Impactor 3.

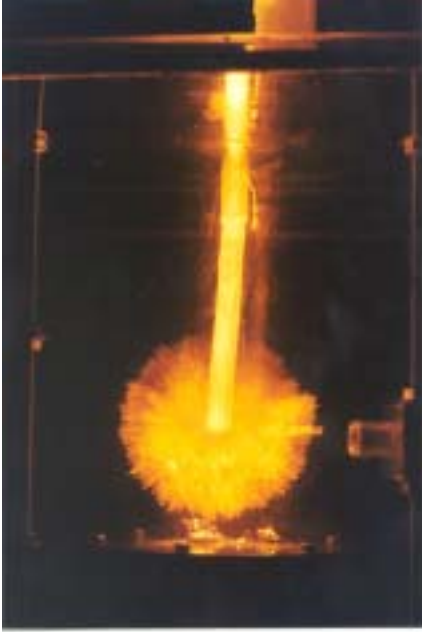


Figure 4a. Time-exposed photograph of the steam explosion of a 9 mm-diameter drop of molten ferrosilicon alloy. (D-11-4-4)



Figure 4b. Time-exposed photograph of the steam explosion of a 9 mm-diameter drop of molten silicon. (D-59-1-2)



Figure 5. Time-exposed photograph of the coarse fragmentation of a 9 mm-diameter drop of molten ferrosilicon alloy in water at 83 °C. (C-203-1-6)

not close and thus the boiling film cannot stabilize around the drop until it reaches a depth of about 125 mm.)

3.6 WATER TEMPERATURE EFFECT

We tested the effects of water temperature on impactor-triggered interactions of molten ferrosilicon drops. We used water at three temperatures—nominally 8 °C, 20 °C (room temperature) and 80 °C. In these experiments:

- Steam explosions never occurred spontaneously at any water temperature.
- Explosive interactions could be triggered with drops of the molten ferrosilicon at the three water temperatures when pressure transients were applied to the water from the impactor. These interactions were most vigorous in the cold water, vigorous in the water at room temperature and mild in the hot water.

- In cold and room temperature water, vigorous explosions occurred, with fine fragmentation and the formation of much colloid.
- In hot water, however, the FeSi75 drops fragmented coarsely, producing particles that burned luminously within bubbles that presumably contain both hydrogen and steam as they rose toward the surface of the water (see Figure 5). Smaller amounts of colloid were deposited when the experiments were performed in hot water.

3.7 ENERGETICS OF THE EXPLOSIONS

We can estimate the energies transferred to the water by the steam explosions of the drops of molten ferrosilicon and silicon from the pressure-volume (PV) products of the bubbles generated by the explosions. The bubble volumes are determined from the photographic images of the explosions and converted into energies with Equation (1):

$$E(J) = 1013[P_{amb}(MPa) \times V_{max}(liters)] \quad (1)$$

where P_{amb} is the ambient pressure against which the bubble grows (assumed to be $P_{atm} = 0.1$ MPa) and V_{max} is the maximum volume of the bubble. Thus, if we determine the true dimensions of the steam explosion bubbles from our photographs, we can estimate the amount of energy transferred to the water.

In side experiments, we compared the images of steam explosions recorded by high-speed photography in reflected light with time-exposed images recorded in a darkened room by the self-luminosity of the melt particles. We learned that the time-exposed images could be used with good accuracy to estimate V_{max} . Because these estimates can be performed quickly and inexpensively without the need for high-speed photography, we relied on this procedure exclusively during this work.

The PV_{max} bubble energies produced by the triggered explosions of 9 mm-diameter drops of molten ferrosilicon and silicon were estimated from the time-exposed images and plotted per gram of melt vs. depth of triggering; they are shown in Figure 6 as the solid diamonds and open circles,

Triggering						
Depth (mm)	Ferrosilicon Pmax = 0.3 Mpa	Ferrosilicon Pmax = 0.6 Mpa	Ferrosilicon Pmax = 1.3 Mpa	Silicon Pmax <2 Mpa	Silicon Pmax >2 Mpa	
0						
50						
100				OOOO		
150	OOOOOOOOOO	OOOOOOOOOX	OOOOXX			
200			XXX	OOOOOOOOOOOOOX	XXX	
250						
300	OOOOO		XXXX			X
350						
400			XXXX	O		O? X
450						
500			XX			O
550						
600						
650						
700			XX			
7850			XX			O
(Bottom)						

Table 1. Explosiveness of 9-mm diameter drops of molten ferrosilicon and silicon vs. fall distance in room temperature water. Drops were triggered with Impactor 2. O = no explosion; X = explosion.

respectively. For both metals, we note that (a) there is a maximum energy release of about 50 J/g at explosion depths of 200-300 mm, and (b) the outer envelope of the points seems to trend toward smaller PV energies per gram of melt as fall distance increases.

3.8 FALL BEHAVIOR OF DROPS OF THE MOLTEN METALS

Our video records indicate that drops of molten silicon fall through the water more slowly than comparable drops of molten

FeSi75. This is shown at the upper and lower left sides of Figure 7 as the times required for 9 mm-diameter drops of these metals to fall to a depth of 311 mm. Although their depth-time relationships scatter somewhat, the drops of molten silicon always fell about $\frac{3}{4}$ as fast (200 to 300 mm/s) as their denser FeSi75 counterparts (about 350 mm/s).

Alloying elements like Al and Ca are present in the parent metal prior to water granulation in varying concentrations.

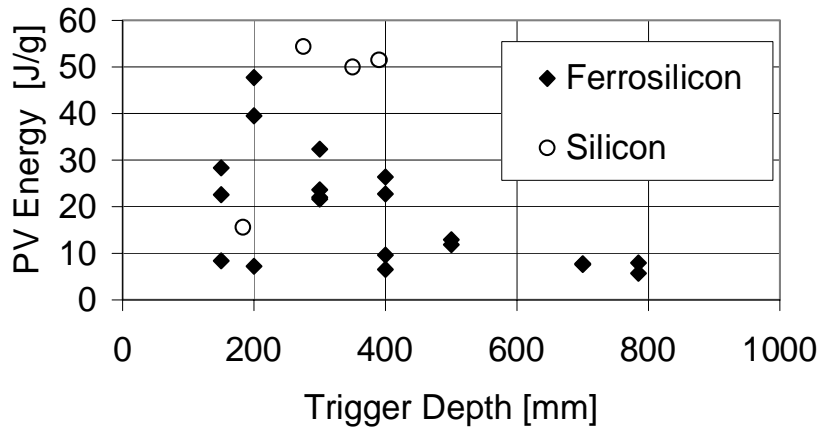


Figure 6. Bubble energies of molten silicon and ferrosilicon drops per gram of melt released plotted as a function of triggering depth.

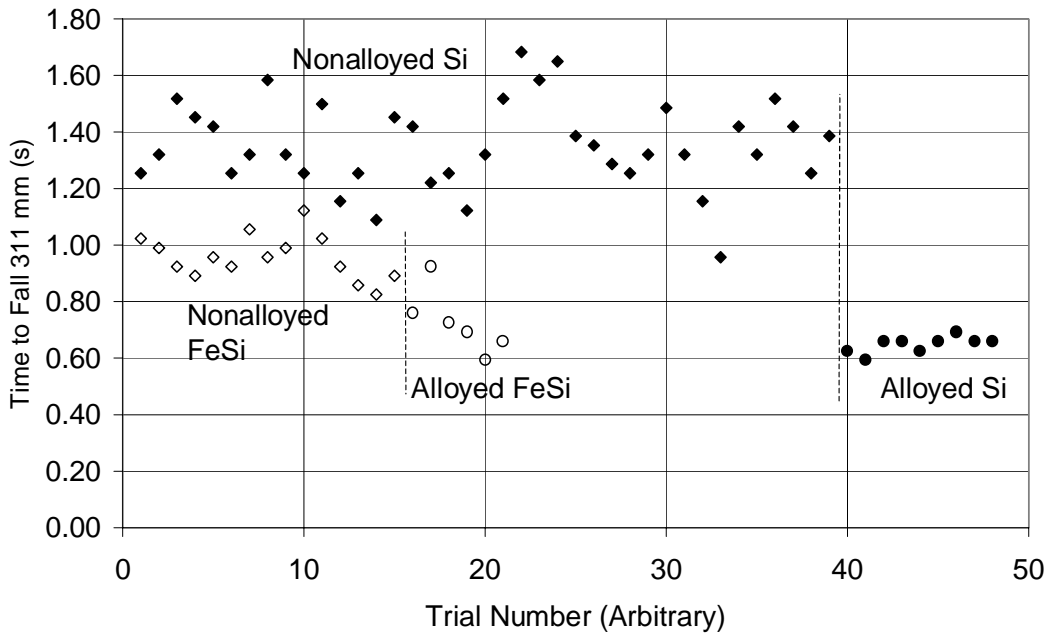
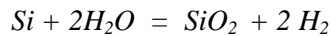


Figure 7. Fall histories of drops of molten alloyed and nonalloyed silicon and ferrosilicon in water at room temperature.

In this work, we discovered that the fall velocities of drops of molten silicon are strongly affected by the presence of small amounts of Al (0.4 wt. %) and/or Ca (0.04 wt. %) in the parent metals. This is shown at the right side of Figure 7. Note that the fall times were drastically reduced by the presence of the additives! In fact, the drops of alloyed silicon fell even faster than 9 mm-diameter drops of the denser nonalloyed FeSi75. A similar but less dramatic effect seems to occur with drops of alloyed FeSi75, as shown at the lower right of Figure 7. Although there were only a few measurements, it appears that drops of both alloyed silicon and alloyed FeSi75 fell at about the same rates, in strong contrast to the behavior of drops of the nonalloyed metals.

3.9 GENERATION OF HYDROGEN BY DROPS OF THE MOLTEN METALS

Our video records consistently showed the generation of bubbles during the interactions of drops of both silicon and FeSi75 with water. We believe the bubbles contain hydrogen due to the reaction



The sizes and numbers of the bubbles depended primarily on the triggering pulses applied to the drops of melt as they fell through the water:

(a) When no pulse or only a low trigger

pulse was applied, the drops did not fragment; only a train of bubbles, each several millimeters in diameter, was released as each non-fragmented drop quenched.

(b) When a drop was exposed to a mild trigger pulse, it fragmented coarsely, and larger hydrogen bubbles were formed. Sometimes these coarse fragments burned luminously and rose within bubbles that presumably contain both hydrogen and steam, producing “seaweed-like” photographic images of the sort shown in Figure 5.

(c) When the triggering transient was strong enough to initiate a vigorous explosion, hundreds of tiny bubbles about 1 mm in diameter were generated. These bubbles rose individually and broke through the surface of the water without coalescing.

With the apparatus shown in Figure 2, we measured typical amounts of hydrogen generated during interactions of types (a), (b) and (c). These are summarized in Table 2. Note that alloying the silicon with small amounts of Al and Ca seemed to increase the amounts of hydrogen generated.

3.10 DEBRIS

3.10.1 GRANULAR DEBRIS

The granular debris recovered within an hour or so after the interactions of drops of FeSi75 and silicon is generally black, and

Type of Interaction	Silicon	Alloyed Silicon	Ferrosilicon
(a) Benign Solidification	1 ml	B: about 2 ml C: about 2 ml D: about 2 ml	About 0.2 ml
(b) Coarse Fragmentation	2-3 ml	B: about 6 ml C: about 4 ml D: about 7 ml	NM
(c) Vigorous Explosions	About 5ml	B: about 6 ml C, D: NM	2-4 ml

Table 2. Amounts of hydrogen generated during the interactions of single 9 mm-diameter drops of molten silicon and ferrosilicon (75 wt. % Si). Alloy compositions: B = Si + 0.4 wt. % Al; C = Si + 0.04 wt. % Ca; D = Si + 0.4 wt. % Al + 0.04 wt. % Ca.

may include lozenge-shaped frozen drops, angular and smooth chunks, material that resembles black sand and sometimes a fine black powdery material. None of the granular debris from either metal appears white to the unaided eye. We have not attempted to analyze the granular debris chemically, but have determined the weights and taken dimensionally calibrated 35 mm photographs as appropriate. The weight lost by each rod (assumed to be the weight of the drop of melt released into the water) is then compared with the weight of the recovered debris. These are usually identical unless an explosive interaction occurred; then, significant fractions of the original drops are not recovered and we assume this material remained suspended in the water as a colloid.

3.10.2 COLLOIDAL DEBRIS

Colloidal products are generated when drops of molten FeSi75 and silicon interact with water. The formation of this material is observed visually and with video imaging both as the drops fall and after the interactions. The colloidal suspensions are very stable, with little change noticed many hours or even days after an experiment. The suspensions produced from the two metals are very similar, except that those formed from silicon seem more copious and longer lasting.

Our only quantitative diagnostic for the colloidal material is the difference between the weight of melt released into the water and the weight of the granular debris recovered after the experiment. Without an explosion, this difference was very small, but when a vigorous explosion occurred, as much as 50% to 60% of a silicon drop or 10% to 20% of a FeSi75 was lost, presumably remaining suspended as colloid.

We analyzed the colloidal material produced from drops of FeSi75 (a) with inductively coupled plasma mass spectrometry (ICPMS),⁹ (b) by weighing residues that remained after known amounts of the

colloid-containing water had been boiled dry, and (c) by X-ray diffraction of the material that remained after the colloid-containing water had been boiled dry. These analyses were not completely definitive, however, except to indicate that the colloid seems to have exactly the same composition as the starting material.

When a silicon drop quenches without a trigger, the video images recorded with reflected light show that a thin, wispy column of colloid starts to rise in the water very late in the quenching, just before solidification seems almost complete. Similar columnar formation of colloid was not noticed with the FeSi75 drops.

When the silicon was alloyed with small amounts of Al and/or Ca, there was an unexpected decrease in the deposition of the colloidal material. In each experiment, either with or without an explosion, the difference between the weight of the drop released into the water and the weight of the granular debris recovered was negligible or at most only a few percent. This indicates that very little colloidal material was deposited in the water during triggered interactions of the alloyed silicon. This is in strong contrast to the experiments performed with drops of non-alloyed silicon, in which as much as half the molten drop might remain in colloidal suspension after an explosion.

3.11 PARTICLE SIZE DISTRIBUTIONS

Although our sieve analyses are still preliminary, significant trends are observed. For example, Figure 8a shows the distribution curve for debris from a steam explosion while Figure 8b shows a typical distribution curve for coarse-fragmented debris. An interesting aspect is the apparent cut-off for the smallest particle size for the finest debris. This could indicate that particles smaller than about 45 μm stay in the water and do not settle. Unfortunately, as mentioned earlier, we cannot exclude the

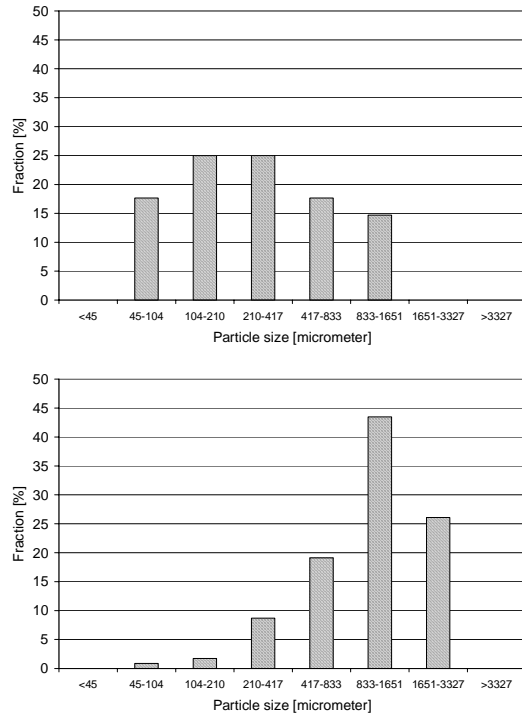


Figure 8. Typical particle size distributions determined by the sieve analyses of the debris recovered from the steam explosion (a, above) and the coarse fragmentation (b, below) of a 9 mm-diameter drop of molten silicon.

possibility that this material was lost during the sieve analyses.

3.12 SOLIDIFICATION OF THE DROPS

In some experiments, we could detect the solidification of the falling drops in the shutter wheel photographs. As a typical example, we observed a 9 mm-diameter drop of FeSi75 that fell through water at room temperature to a depth of 785 mm before being triggered to explode. The “dot” images produced by the rotating shutter wheel indicated that this drop first fell as a completely molten globule for about 1.67 s to a depth of about 500 mm, then started to tumble as a partially solidified “lozenge” before it was triggered and exploded at 2.33 s and a depth of 785 mm. Similar tumbling of silicon drops was

detected on photographs, along with recovered debris that contained both chunks of solid a millimeter or so across plus powder.

Observations of this type are significant because they show that globules of both FeSi75 and silicon can still be triggered to explode even after they have partially solidified! (The steam explosion of a partially solidified drop of laser-melted iron oxide has been reported earlier.¹¹)

4.0 CONCLUSIONS

In experiments at several water temperatures, we learned that the triggered explosions became less vigorous as the water temperature increased from cold to warm. In hot water, however, the drops no longer exploded, but instead broke into coarse fragments that often burned luminously and generated hydrogen.

We also learned that the addition of small amounts of Al and Ca to both molten silicon and ferrosilicon causes the fall velocity of the drops through the water to increase. Also, the explosions became less vigorous when the Al and/or Ca content in the molten drop increased.

We examined the solid materials that remained in the water chamber after the interactions. If the drops froze benignly without exploding, we recovered solidified globules similar to those produced in the granulation process. But if the drops exploded, we recovered coarse fragments or powders. Sieve analyses of the powders showed an unexpected absence of the smallest particle diameters.

We also found that when both silicon and ferrosilicon drops explode, they leave behind in the water a large fraction of their original masses suspended as slow settling colloidal material. The fineness of the colloidal particles suggests a gas-phase combustion-type origin. Although some hydrogen was generated during the

explosions, we have not been able to identify oxygen-containing solid combustion products in the residues. Most of the colloid seems to have a composition identical to the starting material.

We did sieve analyses on some of the debris resulting from the various melt-water interactions. The analyses were done by hand sieving and weighing of the fractions; unfortunately this procedure is not very exact for small samples, because losses up to 15% of the mass were detected.

We have triggered steam explosions of drops of both ferrosilicon and silicon after they have partially solidified.

ACKNOWLEDGEMENTS

This work was performed at the University of Wisconsin-Madison; it was supported by SINTEF Materials Technology, Trondheim, Norway.

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