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Experiments on Interactions Between Zirconium-Containing Melt and Water

Prepared by
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U.S. Nuclear Regulatory Commission

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ABSTRACT

The results of two series of experiments on explosive interactions between zirconium-containing melt and water are described. The first series of experiments involved dropping 1 kg batches of zirconium/zirconium dioxide (Zr/ZrO_2) mixture melt into a column of water while the second series employed 1.2 kg batches of zirconium/stainless steel (Zr/SS) mixture melt. A total of 14 successful tests (nine triggered and five untriggered) were conducted with Zr/ZrO_2 mixtures and a total of eight tests (five triggered and three untriggered) with Zr/SS mixtures. Explosions took place only in those tests which were externally triggered. The test results indicate that the explosion energetics were augmented by the zirconium-water reaction, the extent of augmentation being dependent on the zirconium content in the melt. The explosion energies for tests involving high zirconium contents were mostly in the range of 2-3% of the combined thermal and chemical energy available, while the energy conversion ratios for Zr/SS mixtures of low zirconium content were considerably lower.

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EXECUTIVE SUMMARY

The potential for an explosive interaction between core melt and water (often referred to as a fuel-coolant interaction or FCI) has long been recognized as a concern in assessment of nuclear reactor severe accidents. Mechanical energy release from such an interaction is of interest in evaluating the structural integrity of the reactor vessel as well as of the containment. For example, technical issues relevant from an accident management standpoint include reactor vessel lower head loading due to in-vessel interactions and cavity structure loading due to ex-vessel interactions.

Usually, the source for the mechanical energy release in a melt-water interaction is considered to be the rapid transfer of heat from the high-temperature melt to the water, producing steam fast enough to cause an explosive event ("steam explosion"). When the melt contains a metallic component that is chemically reactive with water, however, there could be an additional source for the energy release, which is the heat release and gas (i.e., hydrogen) production due to the metal-water chemical reaction. For a reactive metal component such as zirconium, the potential chemical reaction energy is about seven times greater than the stored thermal energy of the melt. Thus, if a significant fraction of the chemical energy could be released during an explosive interaction, the explosion energy might be strongly augmented by the chemical reaction. At the last SERG-2 Workshop, questions were raised regarding the possible extent of such chemical augmentation of the fuel-coolant interaction energetics due to the zirconium-water reaction. It was noted at the Workshop that while the importance of the chemical augmentation would depend on the zirconium content in the melt, quantitative data needed for evaluation of FCI issues involving zirconium-containing melts was missing. Accordingly, in an effort to provide the needed data, an experimental program called ZREX (for ZiRconium EXplosion) was carried out at Argonne National Laboratory. This report describes the results of the experiments conducted under this program.

The experimental apparatus consisted of a melt furnace/drop assembly and an interaction vessel. A 1.0 kg mass of zirconium/zirconium dioxide (Zr/ZrO_2) or a 1.2 kg mass of zirconium/stainless steel (Zr/SS) mixture was melted in a graphite crucible by induction heating. The melt was then dropped into a column of water in the interaction vessel, which was a one-dimensional tube geometry, similar to that of KROTOS at JRC (Joint Research Center, Ispra, Italy) or WFCI at the University of Wisconsin. Because of the rigid radial constraint of the test section, the explosion propagation and expansion was essentially one-dimensional, maximizing the axial output of the mechanical energy release. All components of the apparatus were placed in an inerted containment chamber that allowed collection and measurement of the hydrogen generated in the interactions. The containment chamber also provided blast protection from the explosive interaction.

Two series of experiments were performed, the first with Zr/ZrO_2 mixtures (1.0 kg) and the second with Zr/SS mixtures (1.2 kg). A total of 14 successful tests (nine triggered and five untriggered) were conducted with Zr/ZrO_2 mixtures and a total of eight tests (five triggered and three untriggered) with Zr/SS mixtures. The primary objective of these tests was to determine the effect of zirconium content in the melt on the explosion energetics. To accomplish this objective, the melt composition was varied over a wide range. For the Zr/ZrO_2 mixture, the zirconium content ranged from 60 to 100% while the range was from 0 to 100% for the Zr/SS mixture. The major findings of these tests are as follows:

- Explosions took place only in those tests which were externally triggered. In the untriggered tests, the interaction was benign (i.e., no explosion), and the melt quenched rapidly.

- In both series of experiments involving Zr/ZrO_2 and Zr/SS mixtures, the explosion energy increased significantly with increasing content of zirconium in the melt. These test results indicate that the energetics of explosive interactions between zirconium-containing melt and water were augmented by

the zirconium-water chemical reaction, the extent of augmentation being dependent on the zirconium content in the melt. The explosion energies for tests involving high zirconium contents were mostly in the range of 2-3% of the combined thermal and chemical energy (i.e., melt stored thermal energy plus potential chemical energy release from the oxidation reaction of zirconium and water.), while the energy conversion ratios for Zr/SS mixtures of low zirconium content were considerably lower (the conversion ratio for 36 w/o Zr was about 0.8%).

- Hydrogen production in the triggered tests involving explosive interactions was very extensive. For many of the triggered tests, the extent of zirconium oxidation was in the range of 70-100% of the available zirconium. For the untriggered tests, it was significantly lower, ranging from 5 to 25% of the available zirconium.

The experimental results suggest that the extent of chemical augmentation is approximately proportional to the zirconium content in the melt. Thus the impact of chemical augmentation of FCI energetics on reactor safety assessments should be examined on a case-by-case basis since the zirconium content of the melt may vary. In the present experiments it was not possible to distinguish between the relative contributions of thermal and chemical energy to the overall explosion work. There is no evidence that the chemical energy release was converted into mechanical work in any more or less efficient manner than the stored thermal energy. However, the energy conversion efficiency is likely to depend on the boundary conditions for the interaction, including the constraint. Thus the conversion ratios reported herein are considered to be specific to the apparatus employed and should not be generalized to other geometries without modeling efforts that would elucidate the relevant energy conversion processes. This report provides an experimental data base for such modeling efforts.

ACKNOWLEDGEMENTS

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1 INTRODUCTION

The potential for an explosive interaction between core melt and water (often referred to as a fuel-coolant interaction or FCI) has long been recognized as a concern in assessment of nuclear reactor severe accidents. Mechanical energy release from such an interaction is of interest in evaluating the structural integrity of the reactor vessel as well as of the containment (e.g., reactor vessel lower head and cavity structure). Usually, the source for the energy release is considered to be the rapid transfer of heat from the high-temperature melt to the water, producing steam fast enough to cause an explosive event ("steam explosion"). When the melt contains a component that is chemically reactive with water, however, there could be an additional source for the energy release, which is the heat release and gas (i.e., hydrogen) production due to the metal-water chemical reaction.[1] This chemical contribution to the explosion energetics could be important depending on the time scale of the chemical energy release. If this release occurs over a millisecond time scale the energy will be included in the explosive event while if the release occurs over seconds or minutes the energy will only increase the heat loading of the system.

In 1990-1991, Sandia National Laboratories conducted a series of tests investigating explosive interactions between aluminum melt and water as part of New Production Reactor safety studies.[2] These tests, typically involving 10-kg masses of aluminum melt, produced very energetic explosions. In fact, the best estimate mean work release was five to six times the maximum thermodynamic work potential for a pure steam explosion. The investigators suggested that 30-40% of the aluminum melt was oxidized on an explosion time scale during the tests. These findings generated considerable interest in the possibility of the steam explosion energetics being chemically augmented for melts containing a reactive metal such as zirconium.[3] Zirconium is of particular interest, since the heat of reaction with water is about seven times greater than the thermal energy stored in its melt.

In past assessments of the hazard from a steam explosion (e.g. alpha-mode failure of containment), a conservative approach was used with regard to the energetics [4]. For example, given an estimate for melt-water mixing, the energy yield from the explosion was assumed to be a maximum based on thermodynamic considerations. (Subsequent research has revealed that there is a limit on melt-water mixing and more importantly that the energetic yield is substantially lower than the maximum thermodynamic limit.) Note that these otherwise conservative assessments, however, did not take into account the possible contribution of the zirconium-water chemical reaction to the explosion energetics. It was felt that the assessments yielded sufficient safety margins to offset any lack of conservatism arising from phenomena that were not accounted for (e.g. chemical augmentation) or still had uncertainties. Nevertheless, at the SERG-2 Workshop [4] questions were raised regarding the possible extent of chemical augmentation of the FCI energetics due to zirconium-water reaction. It was noted at the Workshop that while the importance of the chemical augmentation would depend on the zirconium content in the melt, quantitative data needed for evaluation of FCI issues involving zirconium-containing melts was missing. Accordingly, in an effort to provide the needed data, an experimental program called ZREX[1] was carried out at ANL.

Two series of experiments were conducted under the ZREX program, with the primary objective of determining the effect of zirconium content in the melt on the explosion energetics. The first series of experiments involved dropping 1-kg batches of zirconium-zirconium dioxide (Zr/ZrO_2) mixture melt into a column of water while the second series employed 1.2-kg batches of zirconium-stainless steel (Zr/SS) mixture melt. The results of these experiments are described in this report.

Section 2 provides details of the experimental apparatus while Section 3 describes the experimental

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[1] ZREX for ZiRconium EXplosion

procedure. Section 4 gives a brief summary of all experiments including the scoping as well as data experiments. The main results of the data experiments are described and discussed in Section 5. Concluding remarks are presented in Section 6. Appendix A shows a check list for the test procedures. Some details of the scoping experiments are given in Appendix B. Appendix C chronologically summarizes all data experiments, successful as well as unsuccessful ones. Detailed descriptions of the successful data experiments including compilations of all data are given in Appendix D.

2 APPARATUS

A schematic of the apparatus is shown in Fig. 2.1. Briefly, the apparatus consists of a melt furnace/release assembly and a test section. The test section is a one-dimensional tube geometry similar to that of KROTOS or WFCI, as discussed in Ref.[5]. Because of the rigid radial constraint of the test section, the explosion propagation and expansion is essentially one-dimensional, maximizing the axial output of the mechanical energy release. All components of the apparatus are placed in an inerted containment chamber that allows collection and measurement of the hydrogen generated in the melt-water interactions. The containment chamber also provides blast protection from the explosive interaction.

Containment Chamber. The body of the containment chamber is a 1.8 m-long section of a 91-cm O.D., 2.5-cm thick carbon steel DN900 pipe. A small electric fan is placed in the chamber to circulate the atmosphere and help provide well mixed gas samples. The electrical and mechanical penetrations are through the top and bottom lids of the chamber. A photograph of the containment chamber is shown in Fig. 2.2.

Test Section. As shown in Fig. 2.3 (schematic) and Fig. 2.4 (photograph), the test section is a one-dimensional tube geometry and is located beneath the melt furnace/release assembly. It is a 98-cm long section of a 10-cm I.D., 3.8-cm thick carbon steel tube. The base of the test section is a welded carbon steel plug with a 3.2-cm diameter threaded hole for mounting of a force transducer.

Melt Furnace/Release Assembly. As shown in Fig. 2.5 (schematic) and Fig 2.6 (photograph), the melt furnace/release assembly is an inductively heated graphite crucible, equipped with an integral plug valve. To release the melt, the plug is pulled from its seat in the crucible base by a pneumatic cylinder mounted on the top lid of the containment chamber. The diameter of the hole in the crucible base is 2.5 cm.

Trigger Device. The trigger device is made up of five parts:

- A length of detonating cord passing from outside the test section, through a hole at the bottom, and into the test section cavity. The trigger energy is adjusted by varying the amount of detonating cord inside the test section. For all tests but one, one g of PETN (PentaErythritolTetraNitrate) was the trigger.

- A commercial #8 blasting cap attached to the external length of detonating cord. On receipt of an electrical signal, the cap explodes, initiating the explosion of the detonating cord. As the cap is outside the test section, it does not add to the trigger energy. The cap and external detonating cord are covered with a steel tube to protect transducer signal cables from blast effects and shrapnel.

- An electrical firing circuit attached to the blasting cap. This circuit uses a Silicon Controlled Rectifier (SCR) to discharge a capacitor through the cap. Charging of the capacitor is controlled by a key lock switch in the experiment control room. The SCR is fired by a signal from a burn wire break detector.

- A burn wire break detector that senses the opening of either of two circuits and then sends a signal to the firing circuit. The two circuits are normally wire grids formed by wrapping 30 ga. magnet wire around phenolic plastic rings with a 9 cm diameter opening such that the area within the ring is crisscrossed with wire, leaving gaps no larger than ½ cm. Melt falling on the grid melts the wire, opening the circuit.

- A break wire, wrapped around the blasting cap to detect when the trigger occurred. The signal

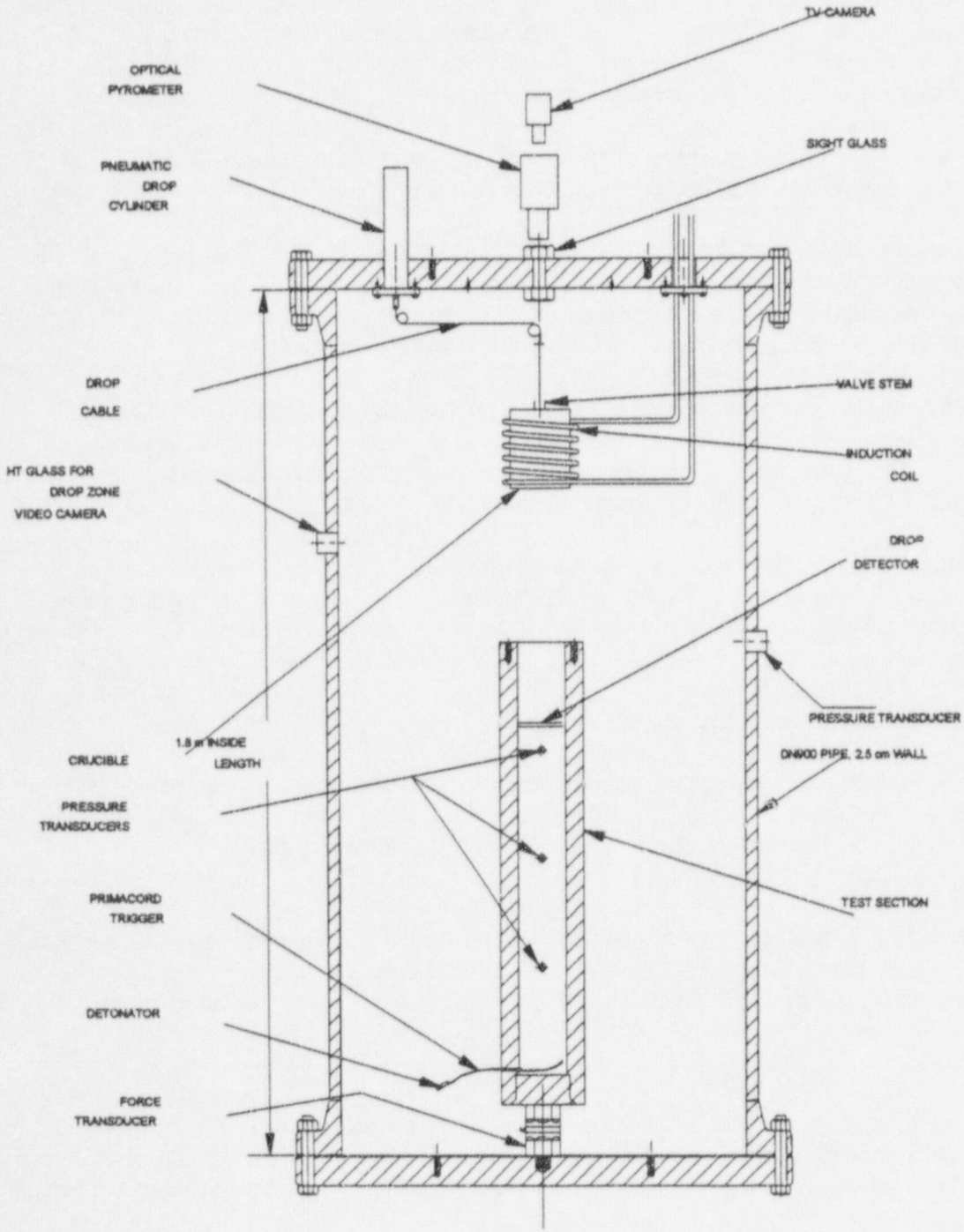

Figure 2.1 Schematic of ZREX Apparatus

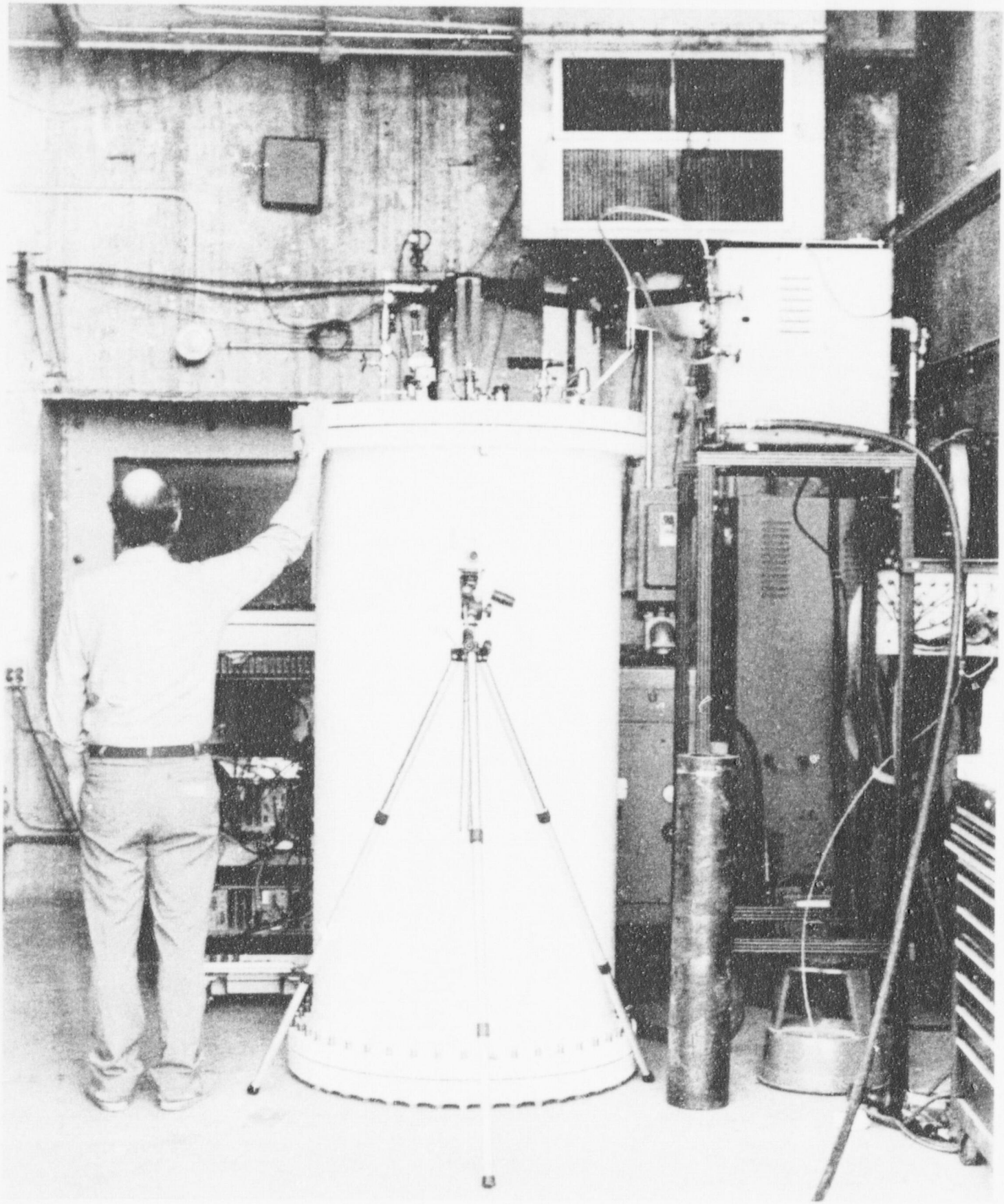

Figure 2.2 Photograph of Containment Chamber

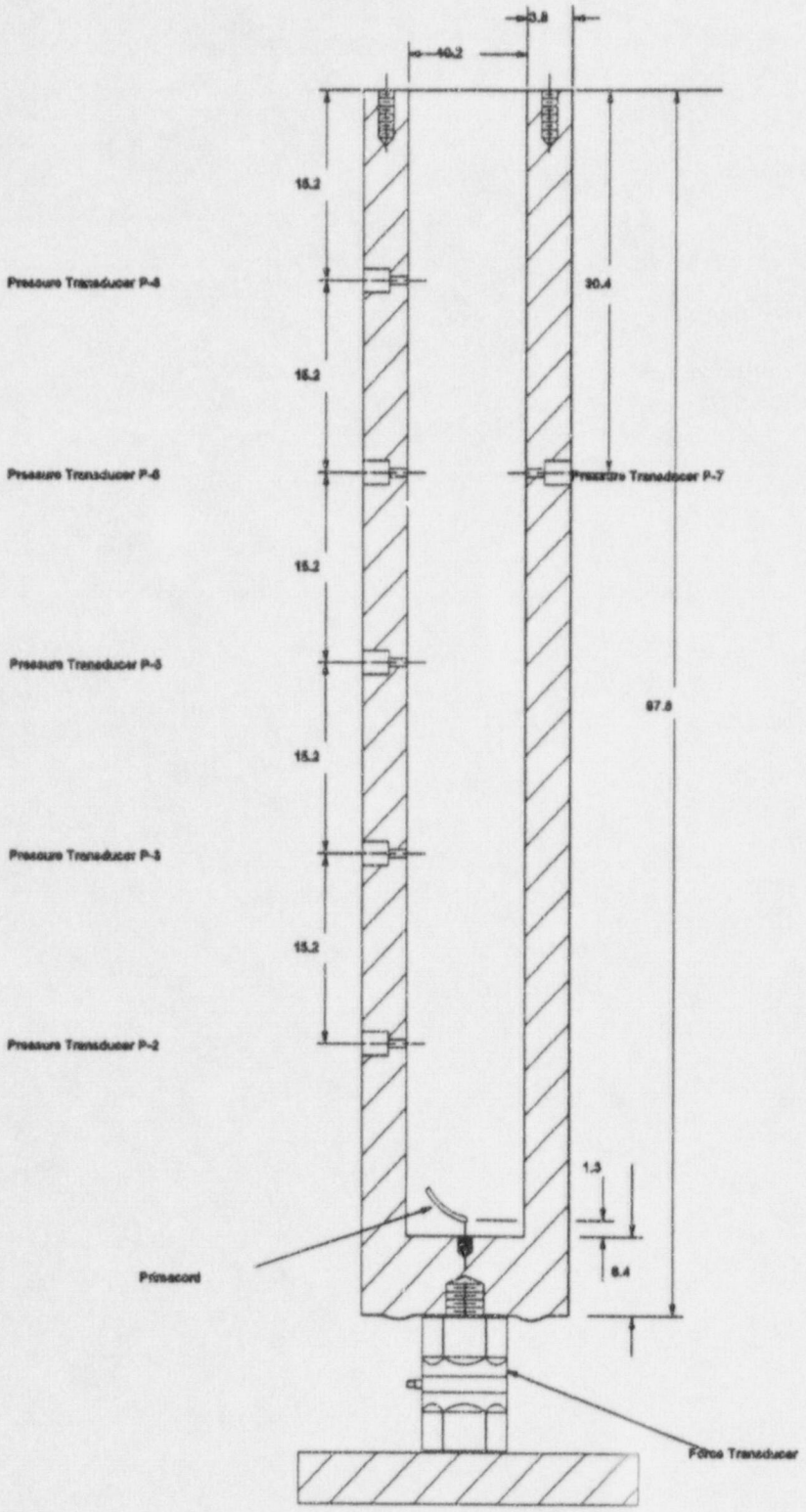

Figure 2.3 Schematic of Test Section

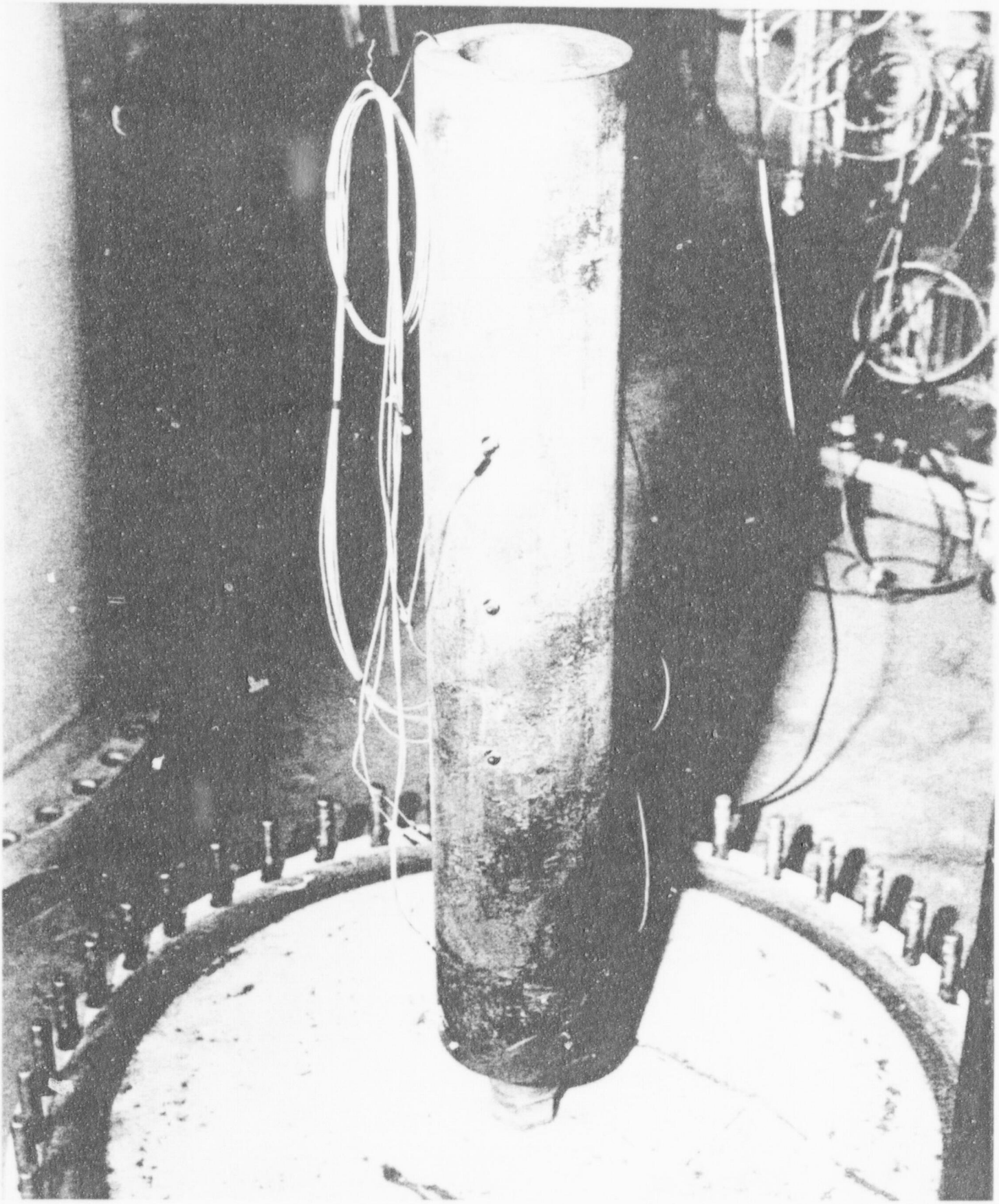

Figure 2.4 Photograph of Test Section

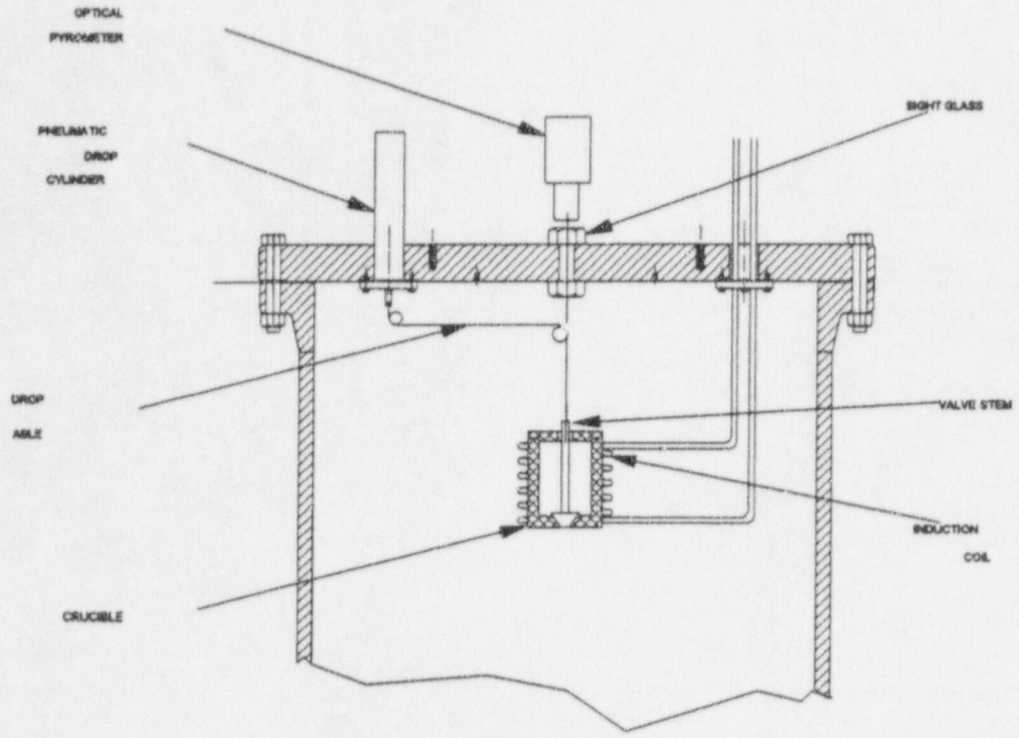

Figure 2.5 Schematic of Melt Furnace/Release Assembly

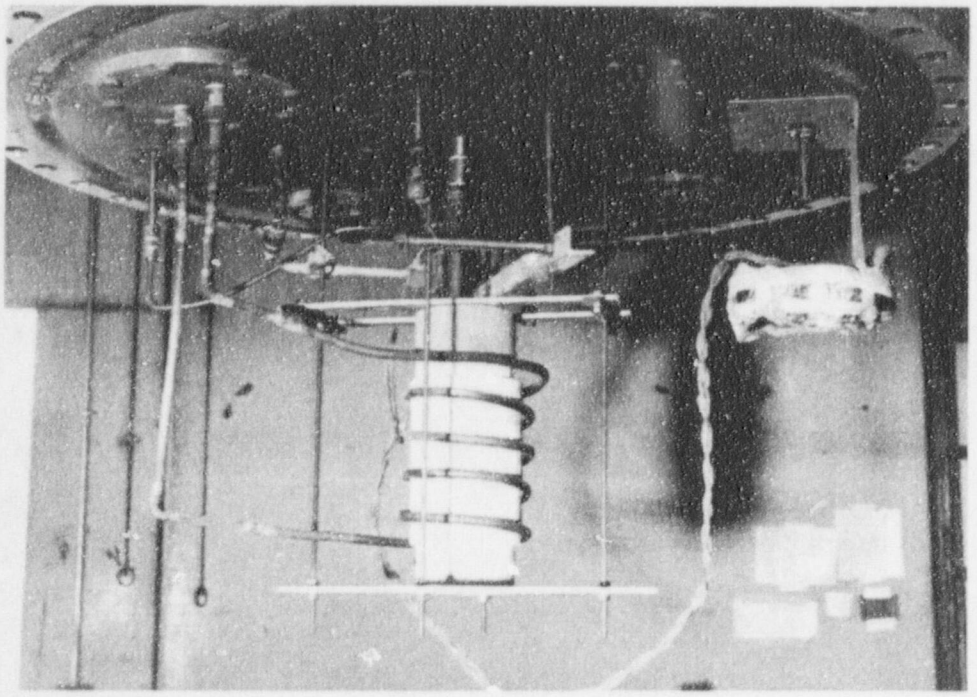

Figure 2.6 Photograph of Melt Furnace/Release Assembly

from this is used to start the PC records and provide a coincidence signal to the backup tape recorder and the slow PC recorder.

A calibration test was conducted to characterize the external trigger used in the triggered experiments. The trigger explosive (i.e., 1 g of PETN) was placed in the test section filled with water and detonated. The resulting pressure and force pulses were measured. The pressure pulses were sharp and narrow, having a peak of 30-40 MPa and a duration of about 100 μs (see Fig. 2.7). From these measurements the trigger energy was estimated by using the shock wave energy formula given by Cole [6], viz

$$E = \frac{A}{\rho c} \int (\Delta p)^2 \, dt$$

where Δp is the overpressure, A is the test section cross-sectional area, ρ and c are the density and the speed of sound of water, respectively, and t is time. Based on the above formula, the trigger energy was estimated to be about 400 J. This trigger energy is a small fraction of the theoretical explosion energy of one gram of PETN, which is 6 kJ.

Water Fill System. Water addition is accomplished by using an external water tank connected to the test section through a pump. An operator watching the water level with a TV camera controls the fill system manually from a remote location.

Gas Sampling System. Gas samples are taken for later chromatographic analysis using a gas sampling system consisting of two sample cylinders, with manual valves at the inlet and outlet of each, in the exhaust line from the containment chamber. The cylinders are placed between remotely operated solenoid valves that control the capture of a gas sample. When a sample is desired, the solenoid valves are closed, stopping exhaust flow and capturing gas in the cylinders. The manual valves are then closed and the cylinders removed and replaced with new ones. The gas sample analysis measured the concentrations of hydrogen, nitrogen, oxygen, carbon monoxide, carbon dioxide, and argon.

Instrumentation System. An optical pyrometer is used to measure the melt temperature in the furnace. The pyrometer views the melt surface through a quartz window in the top chamber lid, through a tube flushed with flowing argon, and through a hole in the top lid of the crucible. A TV camera is placed to view through the pyrometer alignment sight. This gives the operator a view of the melt during the experiment. A second TV camera, looking through a second quartz window in the side wall of the containment chamber, views the drop space between the bottom of the crucible and the top of the test section. A third TV camera views the top of the containment chamber, containing both the water fill tank and dump pneumatic cylinder in its field of view.

A number of type-K thermocouples measure temperatures in the test section, in the containment chamber gas space, and in the exit cooling water from the induction coil. An additional thermocouple controls test section heaters in those runs using preheated water.

Five piezoelectric pressure transducers mounted on the wall of the test section measure dynamic pressures during the melt/water interactions. (The locations of these pressure transducers are shown in Fig. 2.3). These transducers have full-scale ranges of 700 MPa and rise times of 3 μs. A force transducer mounted on the bottom of the test section measures the reaction forces during the interactions. The force transducer has a full-scale range of 0.5 MN and a resonant frequency of 20 kHz (unloaded). Both a static pressure transducer in the top lid and a dynamic pressure transducer mounted on the wall of the containment chamber measure pressure in the containment chamber. The dynamic pressure transducer has a full-scale range of 700 kPa and a rise time of 3 μs while the static transducer has a full-scale range of 3.5 MPa and a rise time of 250 ms.

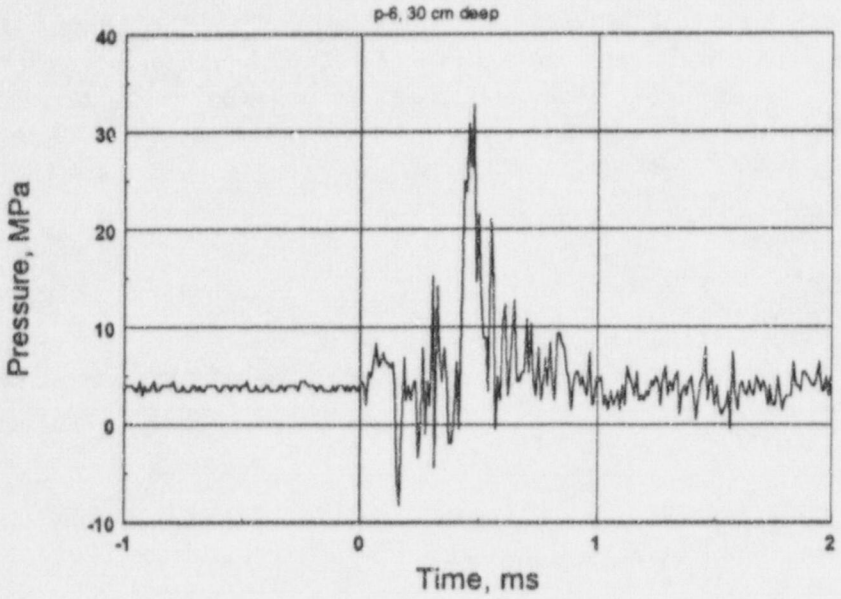

Figure 2.7 Trigger Calibration Pressure Trace

Strain gauges also are mounted on the wall and top lid of the containment chamber.

Current and voltage sensors are mounted on the induction coil leads and monitor the electrical power to the crucible. A burn wire array consisting of four wire grids suspended in the test section water measures the velocity of the falling melt by indicating the time at which each grid is broken by contact with the melt. Each frame is made of a phenolic insulator ring wrapped with a length of 30-ga enameled copper wire so that each wrap passes over the center hole in the ring. The center opening of the ring is about 9 cm in diameter and the wire wrap leaves no opening greater than 1 cm. For those experiments where an external trigger is used, one or two of the grids are selected to serve as the trigger signal for the detonator firing circuit. The breaking of a wire wrapped around the detonator is used as a signal to start the fast data recording. (In the absence of a detonator, a switch on the dump pneumatic cylinder or melting of the top wire in the melt drop sensor is used to start the recording.)

The fast transducer data were recorded on Keithley-MetraByte DAS-50 data acquisition boards in two PC computers. Each of these boards is capable of recording a total of one Mega (1,048,576) samples from four different input channels at rates as fast as one sample/μs. An acquisition rate of one sample each 10 μs for each channel was used in the ZREX experiments. The boards sample data continuously, storing the most recent one Mega samples. A delay timer is started (with a pre-set delay time ranging from zero to the time required to record one Mega samples) when a trigger signal is received. At the end of the delay time, the data memory is frozen, giving a block of one Mega data points within a fixed time interval with a start time that can vary from the interval ending at the trigger time to the interval starting at the trigger time.

A break wire wrapped around the trigger detonator provided the trigger signal when the wire was broken by the explosion of the detonator. For each channel, data was recorded from 2 ms before the trigger to 2.5 s after the trigger. A check was made of the time jitter between triggering the two PCS from the same signal. The jitter was found to be less than 10 μs.

The drop sensor data is recorded by a third PC while all other data is recorded through a multiplexer and DVM onto a fourth PC. A digital tape recorder was used for backup on critical data channels for some experiments. An audio tape recorder placed near the control console records operator comments during the experiment.

An outline of the instrumentation system is shown in Fig. 2.8 where the numbers in parenthesis refer to the number of transducers of that type.

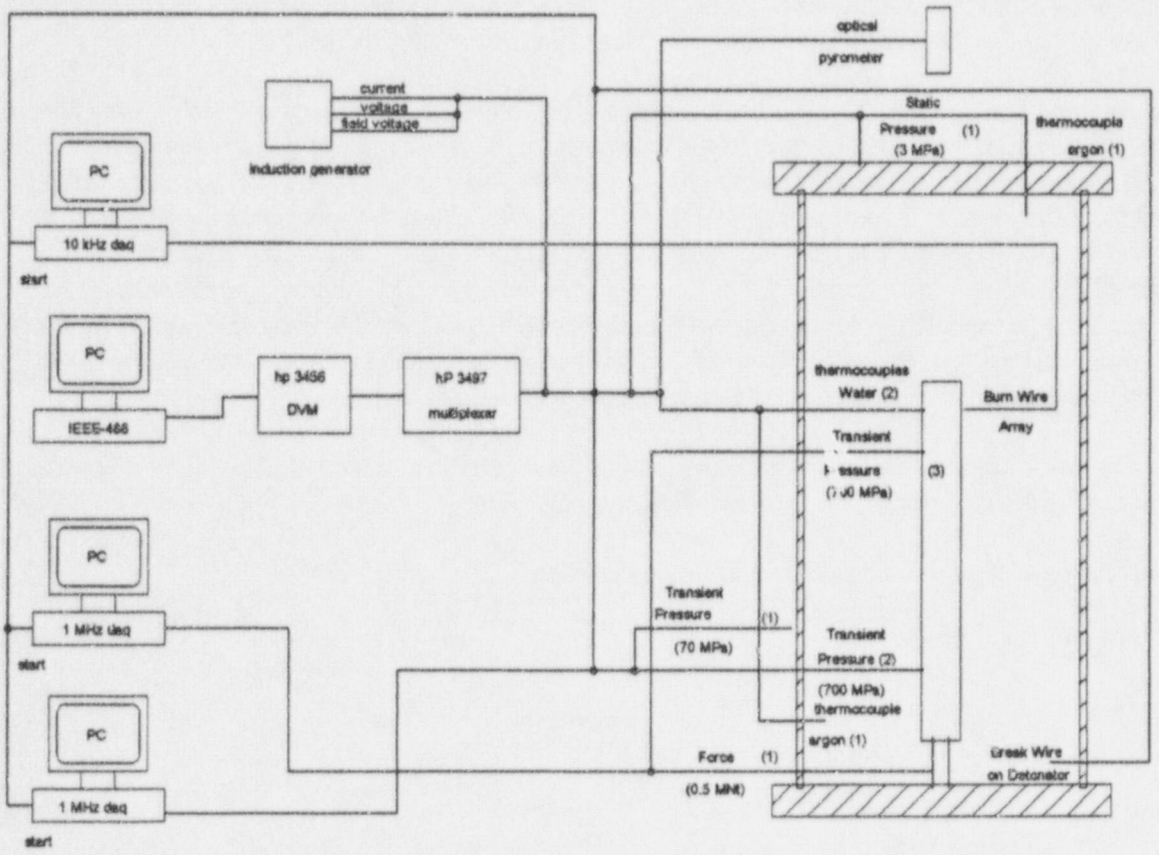

Figure 2.8. Outline of Instrumentation System

3 EXPERIMENTAL PROCEDURE

A 1-kg batch of 1.3-cm diameter zirconium rod or a mixture of zirconium and zirconium dioxide powders (or in the case of ZRSS runs, a 1.2 kg mixture of zirconium powder and 304 stainless steel shot) is loaded into the dropping crucible. The top and bottom of the crucible are insulated with zirconia and alumina board, and zirconia felt is wrapped around the side of the crucible. The crucible and insulation are placed in the induction heating coil and supported on a platform suspended from the containment chamber lid. A steel wire cable is attached to the crucible plug and connected to a pneumatic cylinder. The pyrometer and its associated TV camera are aligned with the crucible and window at this time. A schematic drawing of the system used to control the experiment is shown in Fig. 3.1.

If the experiment is to be triggered, the detonating cord and detonator are added to the test section. The containment chamber is assembled, and final connections and alignment checks are made. The chamber is pumped down . Argon is backfilled into the chamber to a pressure a few kPa above atmospheric when the exhaust line is opened. An argon purge of six liter/minute is continued throughout the experiment. The induction generator is turned on and crucible heating is begun.

When the luminosity of the melt is high enough, a final check of the pyrometer alignment is made, a neutral density filter is placed over the pyrometer camera and all personnel leave the experiment bay. The water fill system is turned on, filling the test section with water. The temperature of the melt is monitored, and when the predetermined (melting temperature plus some superheat) temperature is reached, the argon purge is stopped and the exhaust is closed, trapping a gas sample representative of the atmosphere at the time of melt dumping. Then the dump mechanism is actuated. This procedure is modified for those runs where a waiting or soaking period is desired at some temperature. This is usually done to allow mixing and dissolution of zirconia into zirconium. Then the water addition is delayed until after the soak interval.

After the melt dump, the gas sample cylinders are removed and replaced with others. Fifteen minutes later (the internal fan is mixing the containment chamber atmosphere during this delay), the exhaust valves are opened and the exhaust passes through the gas sample cylinders into an exhaust line that exits the building and enters the atmosphere in front of a fan 7 m above ground level. When the pressure in the containment chamber has dropped at least 3 kPa from the pressure recorded when the exhaust valves were opened, the manual sample valves are closed and the gas sample cylinders are removed. Dummy sample cylinders are placed in the line and exhausting the containment chamber continues. When the pressure reaches 110 kPa, the argon purge valve is opened and a cylinder of argon is purged through the containment chamber to remove any hydrogen and carbon monoxide generated during the experiment.

The following day, the apparatus is disassembled, gas samples are sent to an analytical lab, data is read out from the recording devices, and debris is collected and photographed.

A check list for the test procedures is shown in Appendix A.

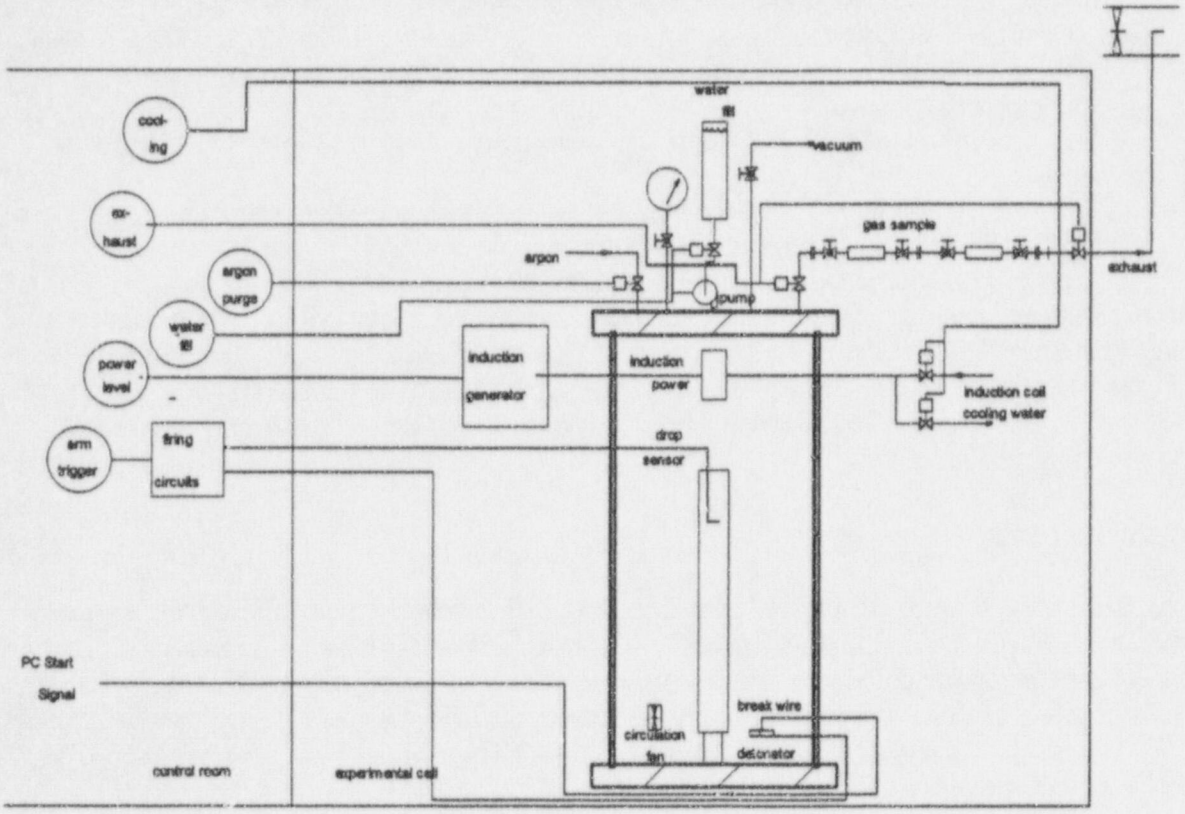

Figure 3.1. Outline of Control System

4 SUMMARY OF EXPERIMENTS

4.1 Scoping Experiments

Prior to the experiments for data, a series of scoping experiments had been conducted to develop the experimental techniques. The scoping experiments started using 200 g batches of Zircaloy-4 melt. The numbering of the experiments starts with the first shakedown test conducted in the containment chamber, which is ZREX-1. These experiments provided information that led to several improvements to the original design of the apparatus. These included

- An insulation scheme was developed that allowed reliable melting of the crucible load.
- A pump was added to the water fill system to force water into the apparatus against a pressurized environment.
- A triggering sensor based on the falling melt breaking an electrical circuit incorporating a fine wire grid was developed.
- A method of looking through the optical pyrometer viewer onto the surface of the melt was devised.
- The melt composition was changed from Zircaloy to zirconium when it was found that tin from the Zircaloy was depositing on the pyrometer viewport.

The scoping experiments included two successful untriggered tests in which the melt simply quenched without an explosive interaction. The first successful test provided information on the quenched debris as well as on hydrogen production during the quenching. Details of this test are given in Appendix B. Although several scoping tests were used to develop techniques for triggering an explosive interaction, no successful triggered tests were conducted. The 18 scoping experiments conducted are summarized in Table B.1 of Appendix B.

4.2 Data Experiments

All experiments for data (successful as well as unsuccessful ones) conducted as part of the program are summarized in Tables C.1 and C.2 of Appendix C. All data experiments involving 1-kg batches of zirconium or zirconium-zirconium dioxide mixture melt were designated as ZREX experiments while the experiments using 1.2 kg batches of zirconium-stainless steel mixture melt were designated as ZRSS (the additional mass for ZRSS corrected for the greater density of the zirconium-stainless-steel melts and gave approximately equal volumes for the two sets of experiments.)

A total of 14 successful tests (nine triggered and five untriggered) were conducted with Zr/ZrO_2 mixtures and a total of eight tests (five triggered and three untriggered) with Zr/SS mixtures. The successful data experiments are summarized in Table 4.1a (ZREX) and Table 4.1b (ZRSS). Detailed descriptions of the successful data experiments including compilations of all data are given in Appendix D. The major findings of these tests are as follows:

- Explosions took place only in those tests which were externally triggered. These explosions caused considerable damage to the internals of the containment chamber. In the untriggered tests, the interaction was benign (i.e., no explosion), and the melt quenched rapidly.

Table 4.1a. Summary of Zr/ZrO_2 experiments

Test No.	Melt Composition		Melt Temp. K (superheat K)[a]	Water Temp. K (subcooling K)	Trigger	Result	
	w/o Zr	w/o ZrO_2				Interaction Behavior	% Zr Oxidation
ZREX-19	100	0	2278 (150)	295 (78)	No	quenching	4.8
ZREX-20	100	0	2373 (245)	296 (77)	Yes	explosion	55.8
ZREX-21	90	10	2573 (252)	293 (80)	No	quenching	9.8
ZREX-22	90	10	2583 (262)	295 (78)	Yes	explosion	48.3
ZREX-23	100	0	2273 (145)	343 (30)	No	quenching	17.0
ZREX-24	100	0	2373 (245)	367 (6)	Yes	explosion	92.3
ZREX-25a	60	40	2583 (243)	298 (75)	No	quenching	13.7
ZREX-27	90	10	2473 (152)	363 (10)	No	quenching	25.6
ZREX-28	90	10	2473 (152)	363 (10)	Yes	explosion	66.9
ZREX-34	60	40	2573 (233)	291 (82)	Yes	explosion	80.0
ZREX-36	80	20	2673 (268)	290 (83)	Yes	explosion	73.0
ZREX-41	90	10	2473 (152)	293 (80)	Yes	explosion	99.2
ZREX-47	100	0	2373 (245)	298 (75)	Yes	explosion	82.6
ZREX-50	100	0	2373 (245)	299 (74)	Yes	explosion	87.1

[a] temperature of melt above the liquidus temperature of Zr-O at the indicated composition

Table 4.1b. Summary of Zr/SS experiments

Test No.	Melt Composition		Melt Temp. K (superheat K)[a]	Water Temp. K (subcooling K)	Trigger	Result		
	w/o Zr	w/o SS				Interaction Behavior	% Oxidation	
							Zr + SS[b]	Zr[c]
ZRSS-4	36	64	2153 (233)	293 (80)	No	quenching	5.7	15.9
ZRSS-5	36	64	2163 (243)	293 (80)	Yes	explosion	26.9	74.8
ZRSS-6	0	100	2153 (342)	296 (77)	No	quenching	3.1	-
ZRSS-7	0	100	2043 (232)	296 (77)	No	quenching	-	-
ZRSS-8	0	100	2153 (342)	297 (76)	Yes	explosion	7.5	-
ZRSS-9	0	100	2123 (312)	300 (73)	Yes	explosion	7.1	-
ZRSS-10	60	40	2173 (293)	301 (72)	Yes	explosion	60.6	100
ZRSS-11	80	20	2123 (717)	304 (69)	Yes	explosion	80.7	100

[a] temperature of melt above the liquidus temperature of Zr-Fe at the indicated composition

[b] $2Cr + 2Fe + Ni + 7H_2O = Cr_2O_3 + Fe_2O_3 + NiO + 7H_2$

[c] $Zr + 2H_2O = ZrO_2 + 2H_2$

- In both series of experiments involving Zr/ZrO_2 and Zr/SS mixtures, the explosion energy increased significantly with increasing content of zirconium in the melt. These test results indicate that the energetics of explosive interactions between zirconium-containing melt and water were augmented by the zirconium-water chemical reaction, the extent of augmentation being dependent on the zirconium content in the melt. The explosion energies were mostly in the range of 2-3% of the combined thermal and chemical energy (i.e., melt stored thermal energy plus chemical energy release from the oxidation reaction of zirconium and water), while the energy conversion ratios for Zr/SS mixtures of low zirconium content were considerably lower. These energy conversion ratios are considered to be specific to the apparatus employed.

- Hydrogen production in the triggered tests involving explosive interactions was very extensive. For many of the triggered tests, the extent of zirconium oxidation was in the range of 70-100% of the available zirconium. For the untriggered tests, it was significantly lower, ranging from 5 to 25% of the available zirconium.

5 RESULTS AND DISCUSSION

A total of 14 successful tests (9 triggered and 5 untriggered) were conducted with Zr/ZrO_2 mixtures and a total of 8 tests (5 triggered and 3 untriggered) with Zr/SS mixtures. Explosions took place in all triggered tests and caused considerable damage to the internals of the containment chamber. In the untriggered tests, the interaction was benign (i.e., no explosions) and the melt quenched rapidly. Salient features of the results are discussed below.

5.1 Debris Characteristics in Untriggered Tests

In the untriggered tests, the melt simply quenched and no explosion took place. The characteristics of the quenched debris seemed to depend on the melt composition as well as on the melt superheat. For pure zirconium melt with a modest superheat (ZREX-19 and -23), the quenched debris was mostly in the form of a long ropy mass with the appearance of a folded ribbon. Most of the debris was found to be resting on one of the burnwires. It appeared that the melt had quenched and frozen within a water depth of 39 cm in ZREX-19 and 78 cm in ZREX-23. Photographs of ZREX-19 and ZREX-23 debris are shown in Figs. 5.1 and 5.2, respectively.

For a $Zr-ZrO_2$ mixture melt with a modest superheat (ZREX-25a and -27), the quenched debris consisted of a loose collection of irregularly shaped particles. Photographs of ZREX-25a and ZREX-27 debris are shown in Figs. 5.3 and 5.4, respectively. It appears that the melt breakup was enhanced by the addition of ZrO_2 to the zirconium melt. Compare, for example, Fig. 5.1 (100w/o Zr) with Fig 5.3 (60w/o Zr/40w/o ZrO_2) or Fig 5.2 (100w/o Zr) with Fig 5.4 (90w/o Zr/10w/o ZrO_2). The enhanced breakup is thought to be related to changes in the melt thermophysical properties brought about by adding ZrO_2 to the zirconium melt.

For a Zr-SS mixture melt with a modest superheat (ZRSS-4), most of the melt fragmented into lumps, sheets, and sharp rods (see the photograph of Fig. 5.5). For pure stainless steel melt with a high superheat (ZRSS-6), the debris was primarily in the form of a cake resting on the bottom of the test section. Apparently, the melt had remained molten throughout its fall and pooled in the catch cup at the bottom of the test section.

5.2 Explosive Events in Triggered Tests

In the triggered tests, an external trigger was used and it was the detonation of a length of Primacord containing 1 g of PETN placed near the bottom of the test section. (One exception was ZREX-50 where 2 g of PETN was used for trigger). The start signal for the trigger was generated by the melt burning through a burnwire grid submerged in the water. The choice of the burnwire location for the trigger signal was made based on consideration of the melt quenching in the test section as observed in the untriggered tests. For the tests with highly subcooled water, the trigger burnwire was located at 30 cm below the top of the test section. (Exceptions were ZREX-20 and -22 where the trigger burnwire location was 20 cm). For the tests with water near saturation (ZREX-24 and -28), the corresponding location was 60 cm.

Damage to the apparatus from the explosive interactions in the triggered tests was qualitatively similar. Typically, all of the mechanism hanging from the vessel lid was destroyed (Fig. 5.6). The crucible was shattered (Fig 5.7). The induction coil, crucible support, valve opening gear, and sight tube were bent or torn. The lid and wall of the vessel were coated with a thick (~1 mm) layer of grey dust (Fig 5.8). The bottom of the vessel contained large pieces of debris (probably crucible pieces) and was coated with a mud made up of water and more of the grey dust along with small pieces of debris (Fig 5.9). When inspected under magnification (30x), the dust had the appearance of a collection of transparent fibers, small white spheres and small black spheres. The three types of insulation used in the experiment, namely, alumina board, zirconia board, and zirconia felt, consisted entirely of fibrous particles with no spherical parts. Thus, it may be

Figure 5.1 ZREX-19 Debris (100w/o Zr and 295 K Water)

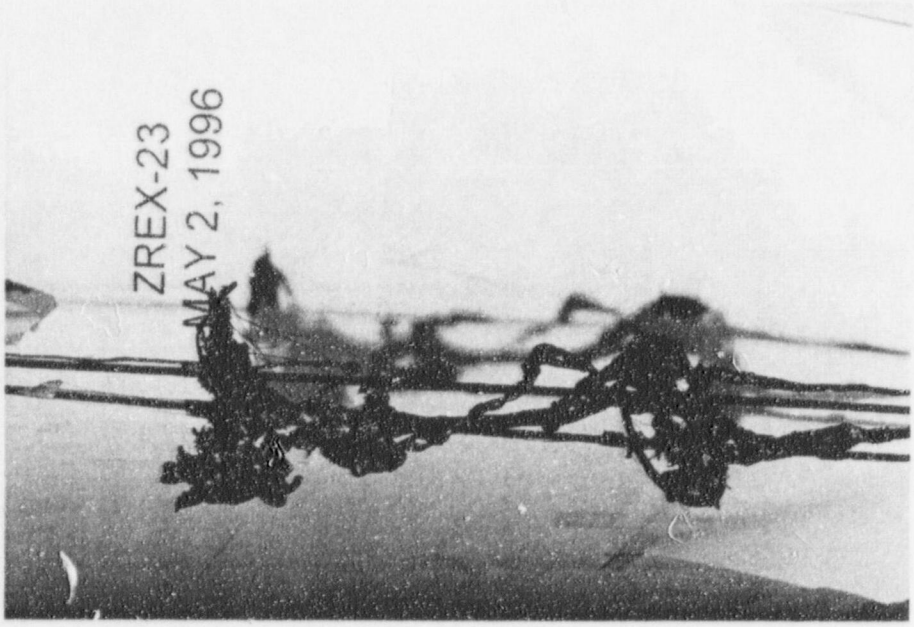

Figure 5.2 ZREX-23 Debris (100w/0 Zr and 343 K Water)

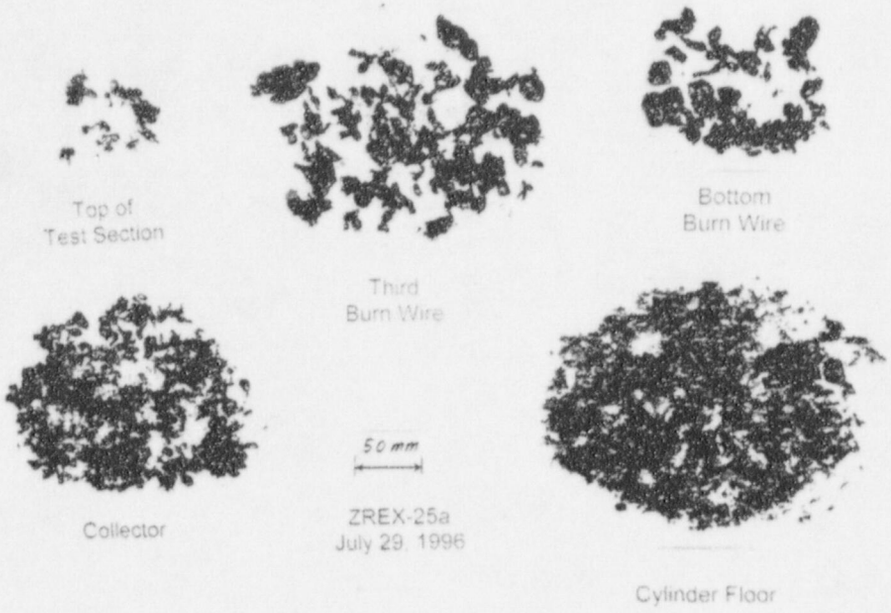

Figure 5.3 ZREX-25a Debris (60w/o Zr/40w/o ZrO_2 and 298 K Water)

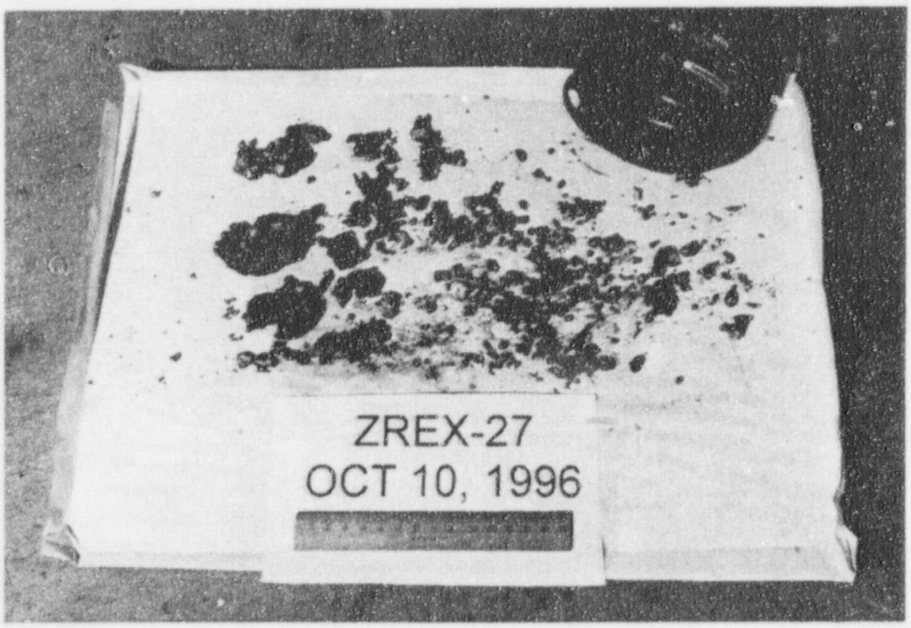

Figure 5.4 ZREX-27 Debris (90w/o Zr/10w/o ZrO_2 and 363 K Water)

Figure 5.5 ZRSS-4 Debris (36w/o Zr/64w/o SS and 293 K Water)

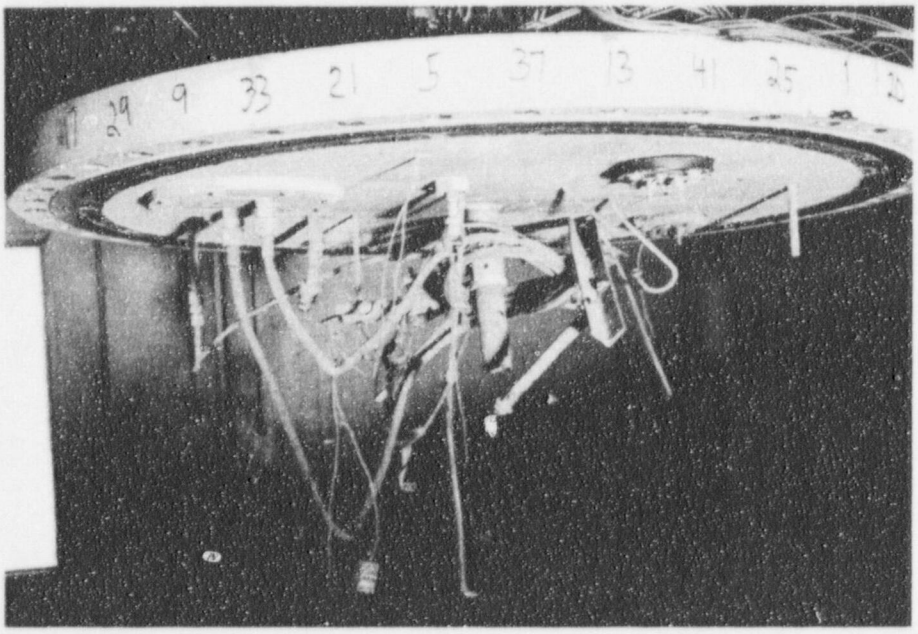

Figure 5.6 Inside View of Top Lid of Containment Chamber after Explosion

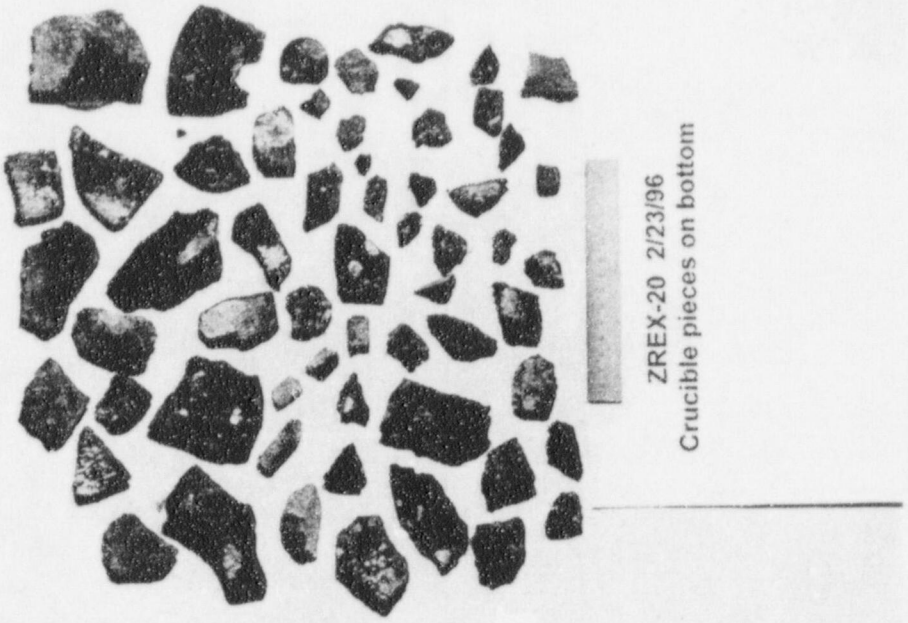

Figure 5.7 Pieces of Shattered Crucible

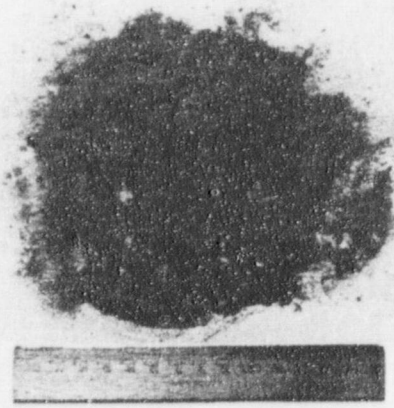

Figure 5.8 Gray Dust Deposited on Top Lid (Inside) of Containment Chamber after Explosion

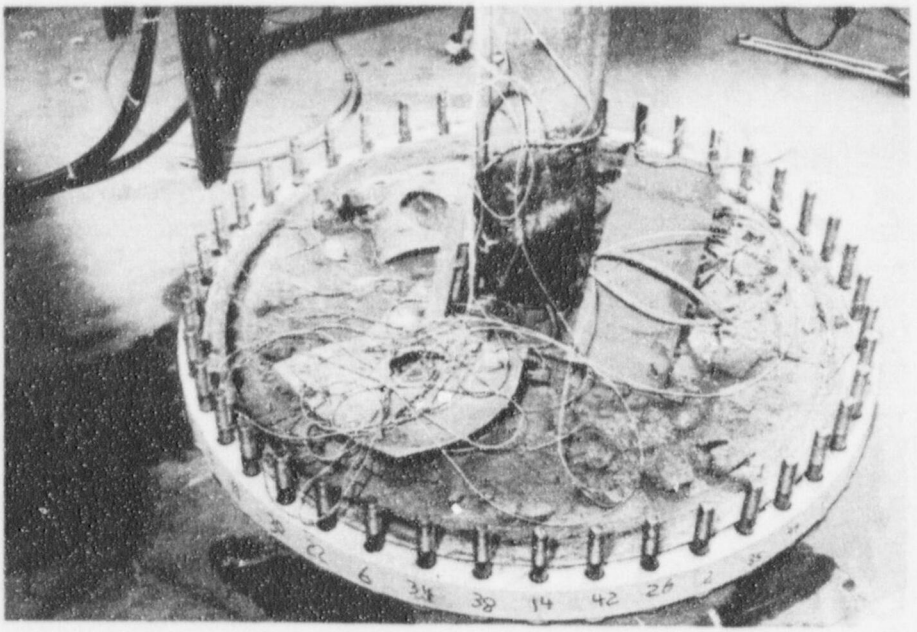

Figure 5.9 Inside View of Bottom of Containment Chamber after Explosion

concluded that all of the spheres are zirconium and zirconia from the explosion. The diameters of these spheres varied over a wide range from under 10 μm to over 100 μm (see Fig. 5.10).

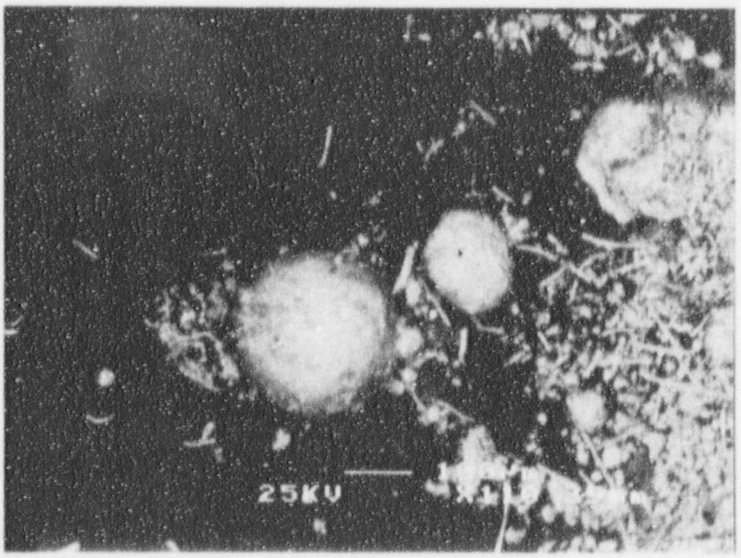

Figure 5.10 SEM Photograph of Explosion Debris

5.3 Hydrogen Production

The gas samples of the containment chamber were analyzed by the Institute of Gas Technology Analytical Laboratory using a gas chromatographic method. Using these gas sample analysis results along with the measured temperatures and pressures of the containment chamber atmosphere, the amounts of hydrogen produced in the experiments were estimated. (The estimate method is separately described below.) The results of the gas sample analyses as well as the estimates of hydrogen production are summarized in Table 5.1.

As can be seen from the last column of Table 5.1, hydrogen production in the triggered tests involving explosive interactions was very extensive. For many of the triggered tests, the extent of zirconium oxidation was in the range of 70-100% of the available zirconium. The amounts of hydrogen production in the untriggered tests were relatively small compared to those in the triggered tests, the extent of zirconium oxidation ranging from 5 to 25%. Also, for the untriggered tests, the extent of zirconium oxidation appeared to increase with increasing temperature of the water (compare ZREX-19 with ZREX-23 and ZREX-21 with ZREX-27).

Estimate Method of Hydrogen Production. There are a number of ways of proceeding with the estimate, each relying on different assumptions. The gas sample before melt-drop was taken from an environment that had a large undetermined thermal gradient due to the hot crucible. Thus the total number of moles of the pre-drop containment chamber gas was not well known. The gas sample after melt-drop was taken after a 15 minute delay while a fan circulated the gas in the containment chamber. The thermal gradient, as indicated by the thermocouples near the top and bottom of the containment chamber, was relatively small, only a few degrees from the bottom to the top. After an explosion the containment chamber atmosphere was very uniform as the hot crucible had been fragmented and mixed with water from the test section. Thus the total number of moles of the containment chamber gas was better known for the post-drop samples. Accordingly, unless there was an indication of a leak from the containment chamber, the estimates were based on the post-drop gas sample analysis results as follows.

Assume oxygen reacts with the hot crucible as follows:

$$O_2 + C = CO_2 \qquad \text{or} \qquad O_2 + 2C = 2CO$$

The total number of moles of oxygen in the containment gas whether elemental or in compound form will be

$$\{O_2\}_{\text{in gas}} = \{CO_2\} + \{O_2\} + \tfrac{1}{2}\{CO\}$$

where the brackets indicate the total number of moles.

Any nitrogen in the gas sample is assumed to be derived from air leakage into the system and the leak would have provided an equivalent amount of oxygen:

$$\{O_2\}_{\text{from air}} = (\{O_2\}/\{N_2\})_{\text{air}} * \{N_2\}_{\text{in gas}}$$

where $(\{O_2\}/\{N_2\})_{\text{air}} = 0.268$.

Subtracting the leakage oxygen from the total oxygen in the gas gives the oxygen that was removed from the water by the producer gas reaction between water and the hot graphite crucible:

$$\{O_2\}_{\text{from water}} = \{O_2\}_{\text{in gas}} - \{O_2\}_{\text{from air}}$$

Table 5.1 Gas sample analysis results and hydrogen production estimates

Test No.	Gas Sample Analysis Results (volume %)							Hydrogen Production Estimates	
	Constituent	H_2	CO	N_2	O_2	Ar	CO_2	No. of H_2 Moles	% Reaction
ZREX-19	Before drop	0.08	0.22	0.65	<1.0	99.0	<0.03	1.06	4.8
	After drop	3.22	0.72	0.61	<1.0	95.3	0.17		
ZREX-20	Before drop	0.11	0.32	1.04	<1.0	98.5	<0.03	12.3	55.8
	After drop	23.0	2.28	0.71	<1.0	73.5	0.39		
ZREX-21	Before drop	0.22	0.61	0.75	<1.0	98.4	<0.03	1.94	9.8
	After drop	5.02	1.11	0.84	<1.0	92.9	0.09		
ZREX-22	Before drop	--	--	--	--	--	--	9.54	48.3
	After drop	18.8	2.15	1.26	<1.0	77.7	<0.03		
ZREX-23	Before drop	0.15	0.25	2.35	<1.0	92.2	<0.03	3.75	17.0
	After drop	8.32	2.60	2.17	<1.0	86.8	0.08		
ZREX-24	Before drop	0.38	0.22	1.41	<1.0	98.0	<0.03	20.3	92.3
	After drop	34.0	3.03	0.89	<1.0	61.9	0.18		
ZREX-25a	Before drop	0.36	0.92	2.17	<1.0	96.5	0.06	1.68	13.7
	After drop	6.61	1.78	1.70	<1.0	89.8	0.09		
ZREX-27	Before drop	1.67	2.50	3.60	<1.0	92.1	0.11	5.07	25.6
	After drop	13.1	3.48	3.15	<1.0	80.1	0.11		
ZREX-28	Before drop	1.20	2.19	7.75	<1.0	88.4	0.48	13.2	66.9
	After drop	22.8	3.56	5.97	<1.0	67.0	0.70		

Table 5.1 Gas sample analysis results and hydrogen production estimates (cont'd)

Test No.	Gas Sample Analysis Results (volume %)							Hydrogen Production Estimates	
	Constituent	H_2	CO	N_2	O_2	Ar	CO_2	No. of H_2 Moles	% Reaction
ZREX-34	Before drop	0.24	1.47	10.1	3.47	84.2	0.52	8.75	79.7
	After drop	15.3	3.17	8.71	<1.0	72.0	0.83		
ZREX-36	Before drop	0.14	1.51	12.9	1.7	82.5	0.52	12.5	72.6
	After drop	21.0	3.18	10.1	<1.0	64.3	1.46		
ZREX-41	Before drop	0.56	1.34	3.88	<1.0	94.1	0.11	19.6	99.2
	After drop	30.5	3.06	2.60	<1.0	63.4	0.40		
ZREX-41a[a]	Before drop	0.55	1.33	4.12	<1.0	93.8	0.12	19.8	100.5
	After drop	31.3	3.06	2.54	<1.0	62.5	0.53		
ZREX-47[b]	Before drop	0.17	0.53	4.42	2.76	92.0	0.13	15.4	70.0
	After drop	24.9	1.78	12.1	3.50	57.2	0.47		
ZREX-47a[a]	Before drop	0.20	0.55	4.45	<1.0	94.6	0.22	18.1	82.6
	After drop	26.5	1.74	11.6	<1.0	59.7	0.46		
ZREX-50[c]	Before drop	0.04	0.55	11.3	<1.0	87.7	0.45	19.1	87.1
	After drop	21.2	1.43	34.1	<1.0	42.6	0.57		

[a] analysis using backup samples

[b] gasket blowout, vessel pressure at zero gage, sample by argon pressurization

[c] gasket blowout, vessel pressure at zero gage, sample by evacuation through sample bottles (see Appendix D for more detail)

Table 5.1 Gas sample analysis results and hydrogen production estimates (cont'd)

Test No.	Gas Sample Analysis Results (volume %)							Hydrogen Production Estimates	
	Constituent	H_2	CO	N_2	O_2	Ar	CO_2	No. of H_2 Moles	% Reaction {based on Zr}
ZRSS-4	Before drop	0.65	1.15	8.81	<1.0	89.1	0.34	1.51	15.9
	After drop	3.02	2.19	8.82	<1.0	85.5	0.48		
ZRSS-5	Before drop	0.67	0.99	8.85	<1.0	89.3	0.16	7.1	74.9
	After drop	12.8	2.26	7.69	<1.0	76.7	0.52		
ZRSS-6	Before drop	0.79	0.82	6.89	<1.0	91.4	0.07	0.8	3.1
	After drop	1.63	1.67	6.82	<1.0	89.5	0.39		
ZRSS-8	Before drop	0.69	0.53	5.29	<1.0	93.3	0.13	2.92	7.5
	After drop	5.19	3.41	4.86	<1.0	86.0	0.45		
ZRSS-9	Before drop	0.72	0.50	6.39	<1.0	91.8	0.12	1.86	7.1
	After drop	3.61	2.13	6.48	<1.0	87.4	0.36		
ZRSS-10	Before drop	0.36	0.82	9.18	<1.0	89.3	0.33	16.45	60.6 {100.2}
	After drop	27.1	3.23	6.52	<1.0	62.7	0.47		
ZRSS-11	Before drop	0.37	1.05	15.6	<1.0	82.2	0.76	21.42	80.7 {92.1}
	After drop	34.8	3.88	9.8	<1.0	50.6	0.87		

$$C + H_2O = CO + H_2 \qquad \text{or} \qquad \tfrac{1}{2} C + H_2O = \tfrac{1}{2} CO_2 + H_2$$

Thus the hydrogen in the sample gas that was produced by the reaction between the crucible and water is:

$$\{H_2\}_{\text{from C}} = 2 * \{O_2\}_{\text{from water}}$$

But then the remainder of the hydrogen came from the reaction between zirconium and water:

$$\tfrac{1}{2} Zr + H_2O = \tfrac{1}{2} ZrO_2 + H_2$$

$$\{H_2\}_{\text{from Zr}} = \{H_2\}_{\text{in gas}} - \{H_2\}_{\text{from C}}$$

And, the number of moles of zirconium oxidized is

$$\{Zr\}_{\text{from ZrO2}} = \tfrac{1}{2}\{H_2\}_{\text{from Zr}}$$

5.4 Containment Pressure Spikes

In the triggered as well as the untriggered tests, upon melt drop into the test section, the containment chamber gas pressure (static) exhibited a sharp spike. The rise time of the spike was on the order of one second. This pressure spike was probably caused by a combination of the addition of gas to the chamber by the generation of a hydrogen/steam mixture and the rapid heating of the containment gaseous atmosphere. It is believed, however, that the primary cause was the containment gas heating brought about by the metal/water reaction. To illustrate the point, the containment pressure spike data were plotted against the amounts of hydrogen produced in the tests. This plot is shown in Fig. 5.11, which clearly indicates a strong correlation (almost linear) between the containment pressure spike and the amount of hydrogen production.

The containment pressure spikes discussed above were recorded by a static pressure transducer in the top lid. In addition, there was a dynamic pressure transducer mounted on the inside wall of the containment chamber at the same elevation as the top of the test section. This dynamic transducer measured gas shock pressures in the containment, which were observed only during the explosive events. Fig 5.12 shows a representative gas shock pressure in the containment during an explosion (ZREX-24). The gas shock pressure records for all explosive events are presented in Appendix D.

5.5 Test Section Explosion Pressures

The test section pressure traces of all triggered tests are presented in Appendix D. Table 5.2 summarizes the characteristics of these pressure traces, including the peak explosion pressures and specific impulses.

A representative explosion pressure trace in the test section is shown in Fig. 5.13. This trace was obtained from the ZREX-47 test where a 1-kg mass of 100w/o zirconium melt was dropped into room-temperature water. The pressure transducer was located at 30 cm below the top of the test section, which was the same location as that of the trigger burnwire. Thus the pressure transducer was located at the lower boundary of the melt-water mixing zone, so the pressure trace shown in Fig 5.13 may be considered to be representative of the interaction zone pressure. It had an abrupt rise to a peak at 0.4 ms, which appeared to coincide with the arrival of the trigger pressure pulse at the burnwire location. (The zero time corresponded to the firing of the trigger detonator at the bottom of the test section which produced an electrical noise spike. The acoustic travel time from the bottom of the test section to the trigger burnwire was approximately 0.4 ms) Thus the initial narrow spikes seen in Fig 5.13 are considered to be primarily due to the trigger pressure pulse.

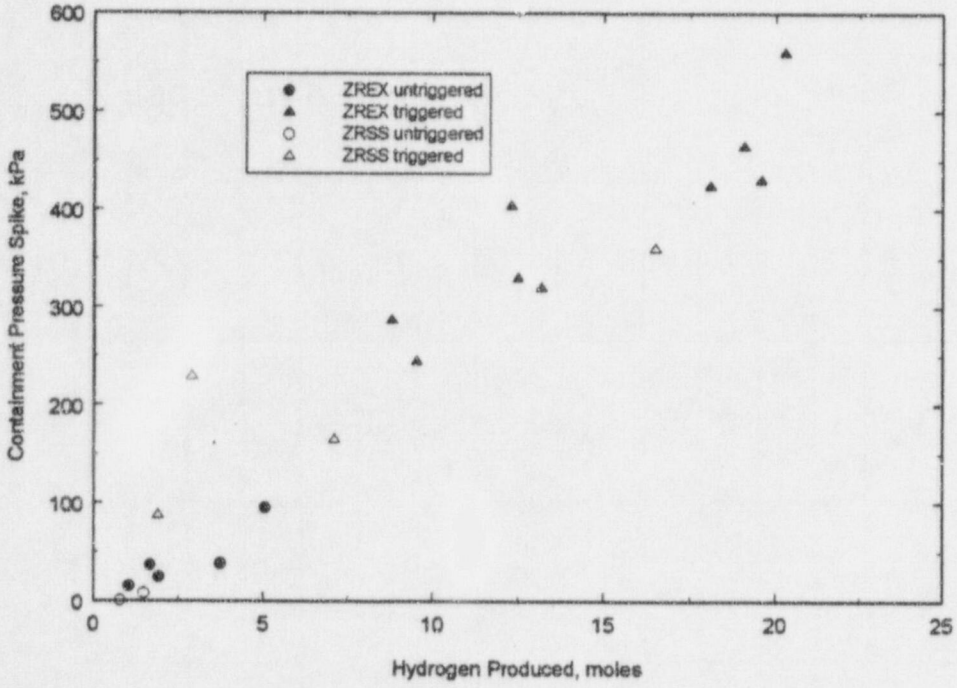

Figure 5.11 Containment Pressure Spikes

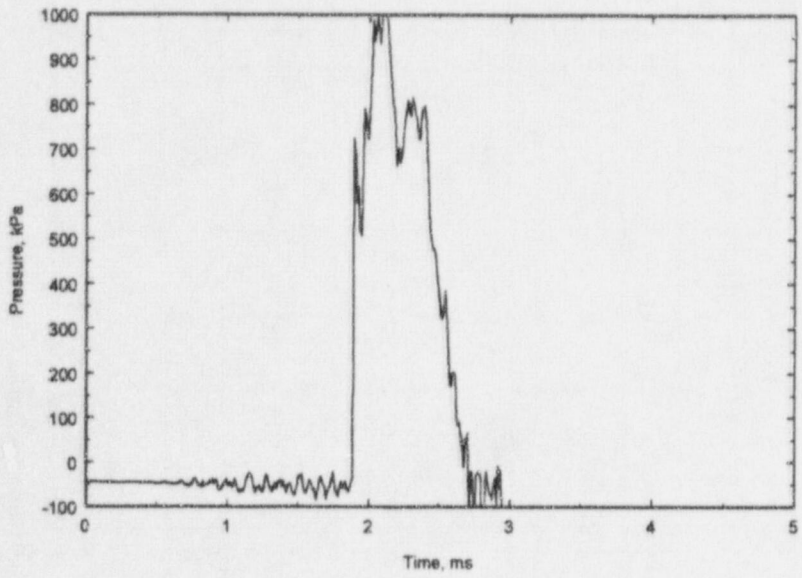

Figure 5.12 Gas Shock Pressure in Containment Due to ZREX-24 Explosion

Table 5.2 Explosion pressures and specific impulses

Test No.	Transducers									
	P-2		-3		P-5		P-6		P-7	
	Peak Pressure MPa	Specific Impulse kPa-s	Peak Pressure MPa	Specific Impulse kPa-s	Peak Pressure MPa	Specific Impulse kPa-s	Peak Pressure MPa	Specific Impulse kPa-s	Peak Pressure MPa	Specific Impulse kPa-s
ZREX-20	--	--	--	--	--	--	40	56	--	--
ZREX-22	--	--	--	--	--	--	22	35	--	--
ZREX-24	--	--	45	70	37	40[a]	42	44	48	62[a]
ZREX-28	--	--	20	75	20	60	20	47	--	--
ZREX-34	90	81	75	48	--	--	55	55	40	66
ZREX-36	55	69	--	--	--	--	40	66	42	72
ZREX-41	110	84	110	90	--	--	60	77	--	--
ZREX-47	55	30[b]	180	138	105	130	47	62	75	95
ZREX-50	85	95	75	120[b]	65	110	23	35[b]	42	87
ZRSS-5	27	21	--	--	25	25[c]	25	18	30	39
ZRSS-8	23	9	32	31	25	24	8	12	12	28
ZRSS-9	23	14	25	25	--	--	6	10	11	18
ZRSS-10	--	--	95	100	55	55	20	37	28	70
ZRSS-11	--	--	--	--	75	75	8	3[b]	70	94

[a] early termination due to instrument failure
[b] pressure record appears faulty
[c] erratic signal, piecewise integration

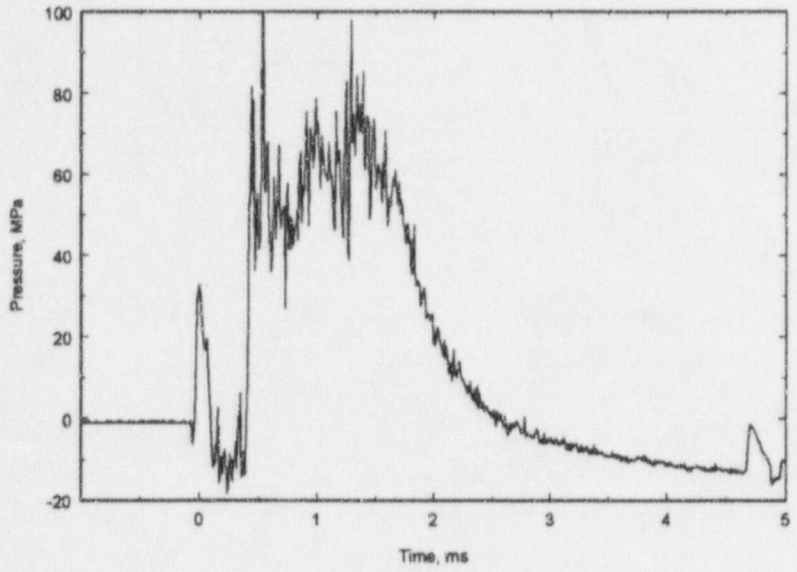

Figure 5.13 Representative Test Section Explosion Pressure

As the trigger pressure decayed, the explosion pressure began to rise, peaking at about 80 MPa, which occurred much later, at 1.2 ms.

Information on propagation of the pressure pulse in the interaction zone was obtained from the ZREX-24 test. In this test, which involved dropping a 1-kg mass of zirconium melt into water near saturation, the trigger burnwire was located at 60 cm below the top of the test section, so the melt-water mixing zone was about 60 cm long. Fig 5.14 shows a composite of three pressure traces at 30, 46, and 61 cm from the top of the test section. These traces were somewhat incomplete, since the decay portions of the pressures were missing, probably due to signal cable failures. Nevertheless, they reveal some details of the explosion propagation characteristics. A close examination of the three pressure traces shown in Fig 5.14 indicates that the propagation velocity of the explosion pressure pulse was about 450 m/s.

5.6 Explosion Energetics

The force measurements at the bottom of the test section were used to estimate the mechanical energy release from the explosion (the force traces of all triggered tests are presented in Appendix D.) Considering one-dimensional acceleration of an inertial mass (slug) caused by the explosion forces in the test section, the kinetic energy of the mass was given by $I^2/2M$, where M is the slug mass being ejected upward by the explosion and I is the impulse load on the bottom of the test section (which was obtained by integrating the force pulse over time). For example, consider the reaction force trace of ZREX-20 as shown in Fig. 5.15. Integration of this force pulse over time gave an impulse of 650 N-s. Assuming an inertial mass of 2.5 kg (all of the zirconium melt plus the volume of water above the trigger burnwire minus the volume of the melt), the slug kinetic energy then was calculated to be 85 kJ. Similar estimates of the slug kinetic energy were made for other triggered tests. These estimates are summarized in Table 5.3.

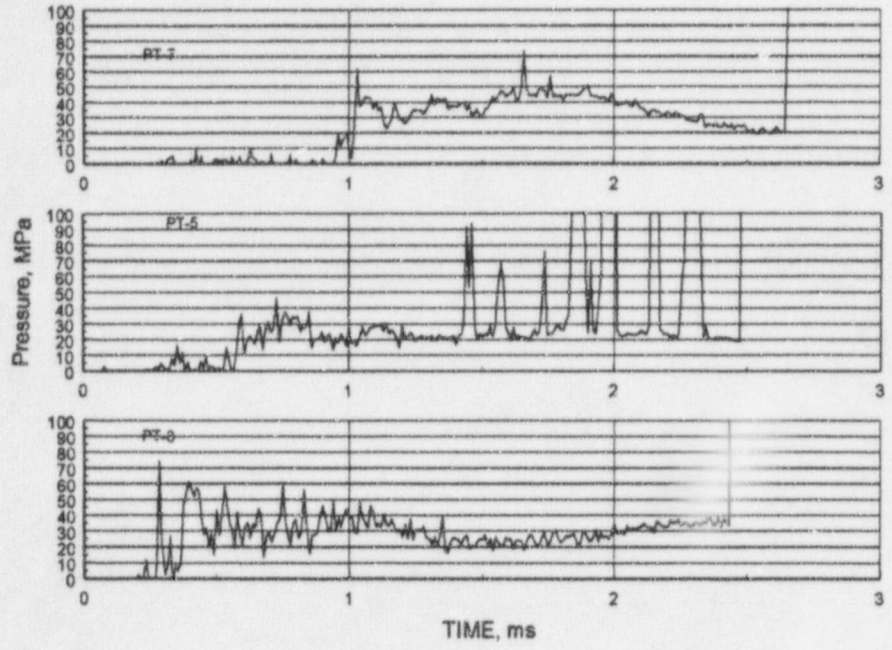

Figure 5.14 Explosion Pressure Propagation in Interaction Zone

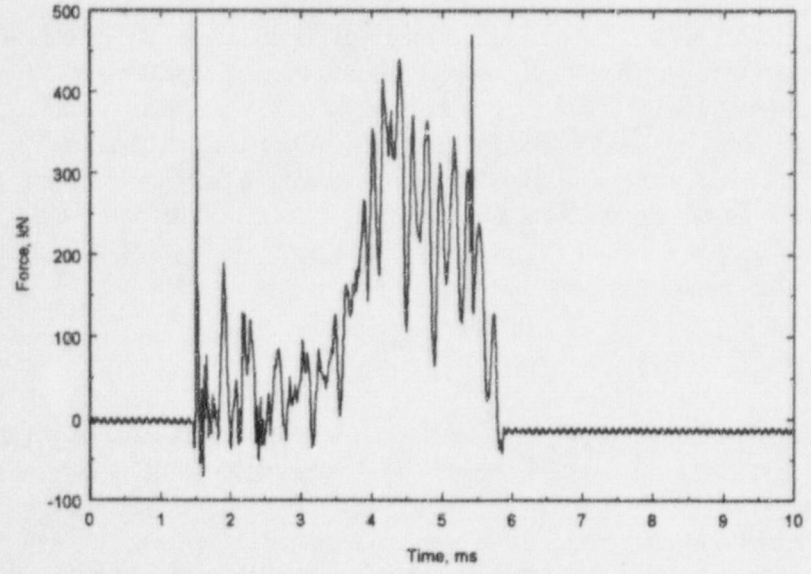

Figure 5.15 Reaction Force Trace of ZREX-20

Table 5.3 Explosion energy estimates

Test No.	Peak Force kN	Impulse N-s	Estimated Expulsion Mass kg	Slug Energy kJ
ZREX-20	420	650	2.5	85
ZREX-22	350	370	2.5	27
ZREX-24	450	800	5.7	106
ZREX-28	300	500	5.7	22
ZREX-34	>500	680	3.1	74
ZREX-36	>500	1000	3.2	153
ZREX-41	>500	1000	3.2	153
ZREX-47	>500	1200	5.7	222
ZREX-50	>500	1125	5.7	196
ZRSS-5	450	480	3.4	33
ZRSS-8	--	267	3.4	10
ZRSS-9	--	304	3.4	13
ZRSS-10	>500	743	3.4	81
ZRSS-11	>500	980	3.4	140

With the exception of two cases where the force transducer failed completely (ZRSS-8 & 9) the integrated force measurement was used to determine the impulse even in those cases where the force transducer was over-ranged. The impulses for ZRSS-8 & 9 were calculated from integrated pressure transducer measurements. All of the calculations were made using assumptions that produced low estimates of the explosion energy. For instance, it was assumed that all of the melt was in the reaction zone at the time of an explosion although the actual amount could not be quantified. Also all of the melt and water with no vapor volume was assumed for the slug mass. There was probably a vapor vent between the explosion zone and the ambient atmosphere during explosions that reduced the conversion of explosion energy to work.

The estimates of the mechanical energy release for all the triggered tests are summarized and compared to the available energies of the melt in Tables 5.4a and 5.4b. The available thermal and chemical energies of melt were calculated using the HSC Chemistry thermodynamics software [7] which is based on the well-known SOLGAS-MIX program. Also, for illustrative purposes, energy conversion ratios were calculated based on two different assumptions regarding the available energy, i.e., that the chemical energy from all of the reactants was available or that only the chemical energy from the reaction as measured in the experiments was available . The results are summarized in Table 5.5. It is seen that the explosion energies (i.e., mechanical energy outputs) were small compared to the available energy source (i.e., melt stored thermal energy plus chemical energy release from the oxidation reaction of zirconium and water). For tests with high zirconium

Table 5.4a. Explosion energies compared to available energies (Zr/ZrO_2 Tests)

Test No.	Estimated Explosion (mechanical) Energy kJ	Available Energy, kJ		
		Stored Thermal Energy of Melt	Maximum Potential Chemical Energy (100% reaction)	Estimated Chemical Energy Release Based on Hydrogen Production Measurement
ZREX-20	85	992	5790	3230
ZREX-22[a]	27	1120	5190	2510
ZREX-24	106	991	5780	5340
ZREX-28[a]	22	1070	5190	3470
ZREX-34	74	1010	2880	2310
ZREX-36	153	1140	4480	3250
ZREX-41	153	1070	5190	5150
ZREX-47	222	991	5780	4780
ZREX-50	196	988	5760	5020

[a] Melt delivery less than satisfactory (see text.)

Table 5.4b. Explosion energies compared to available energies (Zr/SS Tests)

Test No.	Estimated Explosion (mechanical) Energy kJ	Available Energy, kJ		
		Stored Thermal Energy of Melt	Maximum Potential Chemical Energy (100% reaction)	Estimated Chemical Energy Release Based on Hydrogen Production Measurement*
ZRSS-5	33	1530	2770	747(1970)
ZRSS-8	10	1770	444	33
ZRSS-9	13	1740	444	32
ZRSS-10	81	1380	4330	2620(4150)
ZRSS-11	140	1210	5620	4540(5530)

* Numbers in parentheses indicate chemical energy release estimated assuming that Zr alone reacted with water.

Table 5.5. Energy conversion ratios
(explosion energy/available energy)

Test No.	Available Energy Assumed	
	Stored thermal energy plus maximum potential chemical energy	Stored thermal energy plus chemical energy release estimated based on hydrogen production measurement
ZREX-20	0.013	0.020
ZREX-22[a]	0.0043	0.0074
ZREX-24	0.016	0.017
ZREX-28[a]	0.0035	0.0048
ZREX-34	0.019	0.022
ZREX-36	0.027	0.035
ZREX-41	0.024	0.025
ZREX-47	0.033	0.038
ZREX-50	0.029	0.033
ZRSS-5	0.0077	0.014 (0.0094)[b]
ZRSS-8	0.0045	0.0055
ZRSS-9	0.0060	0.0073
ZRSS-10	0.014	0.020 (0.015)[b]
ZRSS-11	0.020	0.024 (0.021)[b]

[a] Melt delivery less than satisfactory (see text.)

[b] Numbers in parentheses indicate chemical energy release estimated assuming that Zr alone reacted with water.

contents, the explosion energies were mostly in the range of 2-3% of the combined thermal and chemical energy potentially available[2]. However, the energy conversion ratios for Zr/SS mixtures of low zirconium content were significantly smaller (the conversion ratio for 36 w/o Zr was about 0.8%).

5.7 Parametric Effects

The primary focus of the experiments was on investigating the effect of melt composition (i.e. the content of ZrO_2 or SS in the zirconium melt) on the FCI energetics. To illustrate the effect of melt composition, the explosion work energies summarized in Table 5.3 were plotted as a function of ZrO_2 content for the ZREX experiments (Fig. 5.16)[3] and as a function of SS content for the ZRSS experiments (Fig. 5.17). These plots include only those runs that were made using the normal values of melt penetration depth (i.e., 30 cm) and water subcooling (i.e., room-temperature water.) It is seen that the explosion work energy decreases significantly with increasing proportion of ZrO_2 or SS in the melt. Clearly, in both ZREX and ZRSS series of experiments, the explosion energy increases with increasing content of Zr. This effect of Zr content on the explosion energy cannot be explained without considering the role of chemical energy release from the Zr-water reaction.

It appears instructive to compare the measured explosion energies of ZREX-47 and -50 (100% Zr) with those of ZRSS-8 and -9 (100% SS) (See Table 5.3). The measured explosion energies of Zr are seen to be about 20 times larger than those of SS. The SS explosions may be considered to be pure steam explosions involving no chemical contribution to the explosion energy. Now, the stored thermal energies of the Zr melt were only 56-57% of those of the SS melt, so one might expect that if the Zr explosions had been pure steam explosions, the Zr explosion energies would have been less than those of the SS explosions, rather than being 20 times larger. Thus, one is led to conclude that the Zr explosions were combined steam and chemical explosions with the chemical contribution being dominant.

As indicated earlier, the energy conversion ratios of the explosions involving high zirconium contents were mostly in the range of 2-4% of the combined thermal and chemical energy available. These conversion ratios are comparable to those of the energetic steam explosions that occurred with alumina melt in the KROTOS experiments [8]. It should be noted, however, that in the KROTOS experiments, the available energy source was thermal only. A comparison of representative ZREX and KROTOS tests (ZREX-47 and KROTOS-44) is given in Table 5.6.

If it is assumed that the ZREX-47 explosion had been a pure steam explosion (i.e., no chemical contribution), the thermal-to-mechanical energy conversion ratio would have been 22.4%, approaching the thermodynamic maximum. However, it is highly unlikely that the ZREX-47 explosion was a pure steam explosion, for it would imply that the Zr explosion was an order-of-magnitude more efficient than that of KROTOS-44. There is no reason to believe that this was the case (both ZREX-47 and KROTOS-44 were conducted in a very similar one-dimensional tube geometry). Again, one is led to believe that the ZREX-47 explosion was a combined steam and chemical explosion in which both the melt thermal energy and the Zr-H_2O chemical reaction energy contributed to the explosion energetics.

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[2] The melt delivery mode involved in ZREX-22 and -28 deviated somewhat from the intended, normal one for these tests. It is believed that the melt masses submerged in the water at the time of the explosion were considerably smaller than 1 kg. The low values of explosion energy for ZREX-22 and -28 are attributed to this less-than-satisfactory melt delivery.

[3] The explosion energies of ZREX-34 and ZREX-36 were normalized to the melt mass of 1 kg.

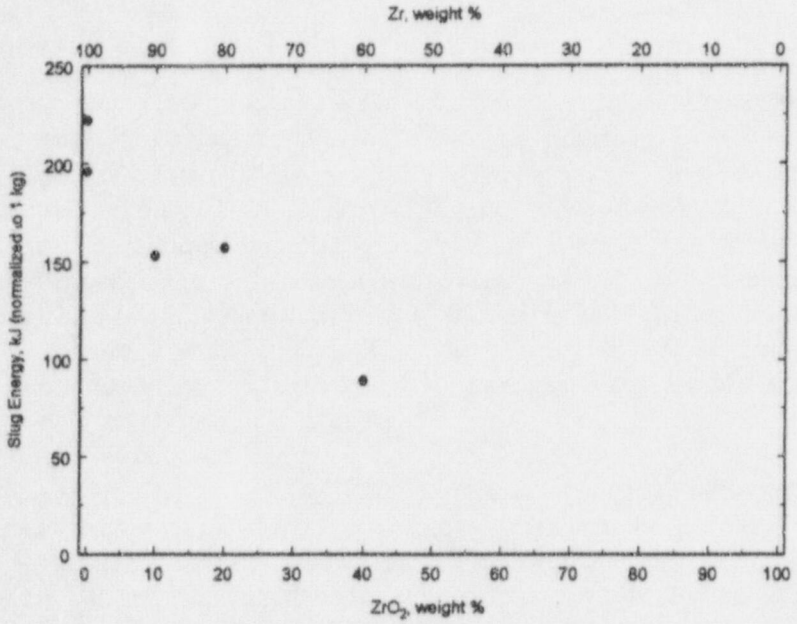

Figure 5.16 ZREX Explosion Energies

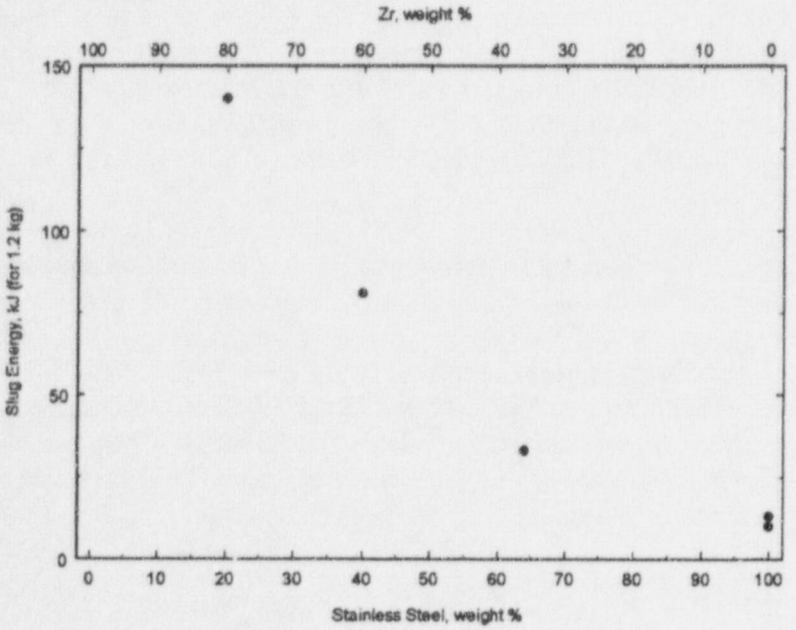

Figure 5.17 ZRSS Explosion Energies

Table 5.6 Comparison of Representative ZREX and KROTOS Tests

	ZREX-47	KROTOS-44
Melt material	zirconium	alumina
Melt energy (thermal), kJ/kg	991	4111
Melt energy (chemical, released), kJ/kg	4780	-----
Total melt energy, kJ/kg	5771	4111
Explosion energy, kJ/kg	222	102
Conversion ratio, % (based on combined thermal & chemical energy)	3.85	2.48
Conversion ratio, % (based on thermal energy only)	22.4	2.48

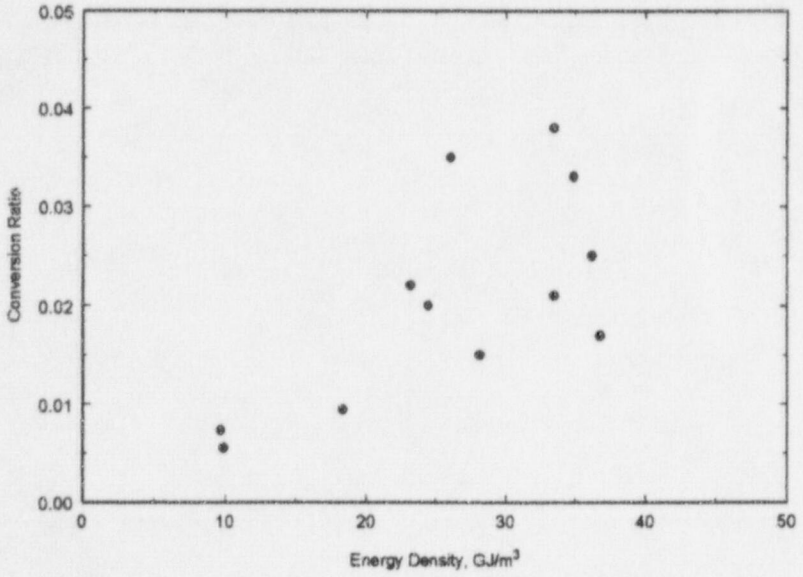

Figure 5.18 Energy Conversion Ratio versus Melt Energy Density

An examination of Table 5.5 seems to suggest that the energy conversion ratio decreases with increasing content of ZrO_2 or SS in the melt. This trend may be related to the total melt energy density (thermal plus released chemical energy available per unit volume). Fig. 5.18 shows the energy conversion ratios (column 3 of Table 5.5) which were plotted against the melt energy density (for ZRSS-5, -10, and -11, it was assumed that Zr alone reacted with water.) Despite a large amount of scatter, it is seen that the energy conversion ratio

increases with increasing melt energy densities. The difference between the highest (Zr explosions) and lowest (SS explosions) melt energy density is particularly striking. Corradini et al made a similar observation based on the data of KROTOS and WFCI experiments [5].

In most of the experiments conducted, the water was at room temperature, the depth of melt penetration into the water at the time of the explosion (melt penetration depth) was 30 cm, and the trigger energy was 1 g of PETN. There were a few exceptions to these normal conditions. Based on the results of experiments involving the exceptions, the following observations are made.

- In ZREX-50, the trigger energy was twice the normal value (2 g vs. 1 g of PETN). A comparison of the results of ZREX-50 with those of ZREX-47 indicates that doubling the trigger energy had little effect on the explosion energy. It appears that the detonation of 1 g of PETN was sufficient (perhaps more than sufficient) to trigger the maximum possible explosions in the present apparatus.

- In ZREX-20, the melt penetration depth was 20 cm. Comparison of the results of ZREX-20 with those of ZREX-47 (which used the normal value of melt penetration depth of 30 cm) shows that the explosion energy of ZREX-20 was significantly lower than that of ZREX-47. This difference may be due to the combination of smaller inertial constraint and earlier venting associated with the shallow melt penetration depth of ZREX-20 although it is possible that less melt was in the reaction zone at the time of the explosion because the reaction zone was shorter.

- In ZREX-24, the depth of melt penetration into the water at the time of explosion was 60 cm and the water was near saturation at 367 K. The results of ZREX-24 may be compared with those of ZREX-47 where the water was highly subcooled at 298 K. It is seen that the explosion energy of ZREX-24 was significantly lower than that of ZREX-47. This difference may reflect the effect of the high void fraction of the melt-water mixing zone associated with the nearly saturated water.

6 CONCLUDING REMARKS

The experimental results suggest that the extent of chemical augmentation is approximately proportional to the zirconium content in the melt. Thus the impact of chemical augmentation of FCI energetics on reactor safety assessments should be examined on a case-by-case basis since the zirconium content of the melt may vary. In the present experiments it was not possible to distinguish between the relative contributions of thermal and chemical energy to the overall explosion work. There is no evidence that the chemical energy release was converted into mechanical work in any more or less efficient manner than the stored thermal energy. However, the energy conversion efficiency is likely to depend on the boundary conditions for the interaction, including the constraint. Thus the conversion ratios reported herein are considered to be specific to the apparatus employed and should not be generalized to other geometries without modeling efforts that would elucidate the relevant energy conversion processes. This report provides an experimental data base for such modeling efforts.

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APPENDIX A

CHECK LIST FOR TEST PROCEDURES

date_____

Experiment Description :

Crucible reference drawing #

{ VESSEL 1.800 m high x 0.866 m I.D. = 1.06 m^3 }
{ test section 91.5 cm high x 10.1 cm I.D. }
{ crucible to test section gap - 29 cm }

1. **Loading**
   a. METAL
      i. Material _____
      ii. Amount _____
      iii. Crucible _____
   b. WATER
      i. Amount _____
2. **Pre-Test Procedures**
   a. VESSEL INTERNAL ACTIVITIES
      i. Clean pyrometer window.
      ii. Inspect and replace transducer leads.
      iii. Position and tape transducer leads.
      iv. Test all force and pressure transducers.
      v. Assemble burnwire array.
      vi. Insert Primacord and glue in place (length inside Test Section _____).
      vii. Plug Primacord hole if not triggering.
      viii. Assure that all former Primacord holes in Test Section are plugged.
      ix. Check that Primacord doesn't cross over itself.
      x. Install and connect burnwire array to trigger circuit.
      xi. Check burnwire integrity and trigger operation.
      xii. Install shorting plug on vessel shell connector.
      xiii. Check that shorting plug not cross threaded.
      xiv. Attach detonator to Primacord.
      xv. Attach break wire to Primacord.
      xvi. Cover Primacord and detonator with shrapnel tube.
      xvii. Place vessel cylinder on base plate.
      xviii. Drape instrument leads outside vessel.
      xix. Connect detonator to vessel shell connector(red - C-D - wht/org).
      xx. Connect break wire to vessel shell connector (black - A-B - wht/blk).
      xxi. Insulate detonator connectors.
      xxii. Check detonator wires for continuity
         (1) detonator (2.7 Ω) _____
         (2) break wire (0.3 Ω) _____
      xxiii. Replace shorting plug.
      xxiv. Connect water fill lines.
      xxv. Bolt up cylinder.
      xxvi. Check circulation fan on lid.
      xxvii. Move containment lid into place, support on blocks
         (1) Connect water fill line.
         (2) Connect Test Section theromocouple.

(3) Connect thermocouple in bottom insulation.
(4) Connect Test Section control thermocouple.
(5) Connect Test Section heater power.

xxviii. Run "DUMPS" to check instruments.

xxix. Lower lid into place.

xxx. Position pyrometer.

xxxi. Turn off pyrometer light and bolt up lid.

b. EX-VESSEL ACTIVITIES

i. Connect leads to TOCCO.

ii. Connect gas line to cylinder.

iii. Connect gas line to water fill valve.

iv. Connect gas line to argon purge.

v. Connect water fill line after filling line with water.

vi. Fill water reservoir.

vii. Connect coil cooling water lines.

viii. Plug in water pump.

ix. Connect vacuum line.

x. Connect exhaust line (check if open).

xi. Connect control cables :

(1) Argon purge solenoid.
(2) Exhaust solenoids.
(3) Water fill solenoid.
(4) Dump solenoid.
(5) Cooling coil solenoids.
(6) Heater power.

xii. Connect instrument cables:

(1) TV cameras:
(a) Pyrometer.
(b) Induction generator.
(c) Lid overview.
(d) Drop view.

(2) Thermocouples:
(a) Top Vessel atmosphere.
(b) Bottom Vessel atmosphere.
(c) Test section water.
(d) Crucible insulation.
(e) Test section heater controller.

(3) Pyrometer.

(4) Pressure transducers:
(a) Static on lid.
(b) Dynamic on vessel side.

xiii. Evacuate vessel.

xiv. Back fill with Ar.

xv. Purge vessel with argon (set purge flow to 2 lpm).

xvi. Set test section heater control to ____ and turn on.

xvii. Load water fill system with ___ l.

xviii. Lid strain gauge _____. $Vr=\Delta Vout/(Vin*G)$; G=gain=100

xix. Cylinder strain gauge _____. Vin=9.130V; GF=2.03; $\epsilon=4*Vr/[GF*(1+2*Vr)]$

xx. Adjust polaroid filter to mark and lens f/stop.

(1) Pyrometer camera - f/22.
(2) Drop view camera - f/22 + 2.0 ND.

xxi. Unplug solenoid valve to lid vacuum gage.

xxii. Power to internal circulating fan.

xxiii. Power to :

(1) Charge amps for test section pressure (3).
(2) Burn wire trigger unit.
(3) Static pressure transducer supply.
(4) PCB charge amps(1).
(5) Drop view camera.
(6) Pyrometer.
(7) Induction generator sensors.

xxiv. Close and lock gate.

xxv. Inspect fenced enclosure.

xxvi. Turn on warning lights.

xxvii. Power up trigger system.

(1) For triggered run connect cable from PC Trigger to on vessel break wire connector (bk/wt wires).
(2) For non-triggered run connect cable from PC Trigger to top burn wire tee on Burn Wire Trigger box.
(3) Plug in vessel detonator connector.
(4) Check PC triggering.
(5) Check trigger circuit to fire circuit connection.
(6) Connect detonator to fire circuit (or/wt wires).

xxviii. Increase argon purge to 6 lpm.

xxix. Turn on argon supply to dump.

xxx. Set crucible valve cylinder operating pressure to _____.

xxxi. Turn water reservoir light.

xxxii. Set up audio recorder.

xxxiii. Turn on all PCs.

(1) Gateway (#4) run "DUMPS" 20 s/min
(2) DTK (#2) run "ZREX201" auto backup
(3) DTK (#3) run "ZREX201" auto backup
(4) MIS (#1) run "UWBW1.

xxxiv. Turn on coil cooling valves (manual & solenoid).

xxxv. Turn on main induction generator cooling water.

xxxvi. Balance induction coil if necessary.

xxxvii. Calibrate pyrometer.

xxxviii. Turn on induction generator.

xxxix. Set induction generator to remote.

xl. Generator readings

(1) initial I____% V____% P____% Lead____°
(2) later I____% V____% P____% Lead____°

xli. Adjust TV camera alignment and Polaroid.

(1) Pyrometer camera (f/11).
(2) Drop view camera (f/8 + 2.0 ND).

xlii. Turn off water reservoir heater.

xliii. Turn off water reservoir stirrer.

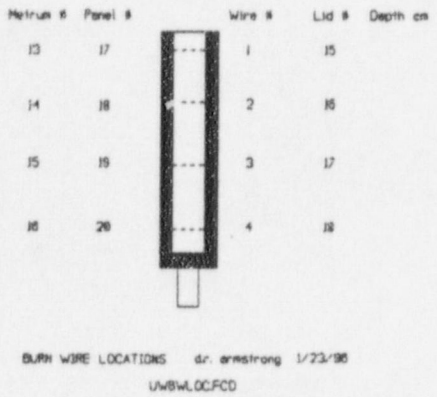
xliv. Add cork to water reservoir.

3. **Run Procedures**
   a. Start audio recorder.
   b. Top up water level in reservoir.
   c. Turn off water reservoir heater.
   d. Start VCR recorders.
   e. Load water into test section.
   f. Monitor crucible temperature.
   g. Check PCs.
   h. Turn on detonator power.
   i. Turn off test section heater.
   j. Close dump switch when temperature reaches _____°C.
   k. If explosion and if vessel pressure is dropping - close coil cooling valves.
4. **Post-Run**
   a. Note time _____.
   b. Back up data.
   c. Turn off VCRs.
   d. Break off record enable tabs.
   e. Close exhaust valves.
   f. Close argon purge.
   g. Turn off induction generator Variac.
   h. Turn off "detonator arm".
   i. Turn off "dump".
   j. Check vessel pressure _____.
   k. Stop audio recorder.
   l. Break off record enable tab.
   m. Enter cell.
   n. Close sample bottle valves and remove sample bottle containing pre-test gas sample.
   o. Install new gas sample bottle and open valves.
   p. Turn off generator.
   q. Turn off Test Section heater controller.
   r. Turn on exhaust fan and note time _____.
   s. Run "SAMPLES" on Gateway.
   t. Wait 15 min.
   u. Open vessel exhaust for 10 sec to 1 min or $\Delta P = 3$ kPa.
   v. Note temperature _____ and Pressure _____.
   w. Close sample bottle top valve.
   x. Wait 3 minutes and close remaining valves (top down).
   y. Close exhaust.
   z. Remove sample bottles.
   aa. Install new sample bottle and open valves.
   bb. Open exhaust.
   cc. After pressure reaches 10 kPa over ambient, open argon purge.
   dd. Purge one full argon cylinder.
   ee. Turn off exhaust fan.
   ff. Turn off circulation fan.

REMARKS :

Cable Chan	Rcrd Chan	Type	M/N	S/N	Amp S/N	Sens	Location
1	DTK3-1	flink	937	46350	PCB-1	100 kn/V 0.991 (50 kN /V out)	see drawing
2	DTK3-2	ptran-w	109	3832	D-4	77.5 MPa/V	" 76.2 cm
3	DTK2-3	"	109	3688	D-1	72.6 MPa/V	" 61.0 cm
4	---	"	---	---	--	-------	" 61.0 cm
5	DTK2-0	"	109	3833	D-2	72.6 MPa/V	" 45.7 cm
6	DTK2-1	"	119	2055	6	-100 MPa/V	" 30.5 cm
7	DTK2-2	"	109	3836	PC-1	71.8 MPa/V	" 30.5 cm
8	---	"	---	---	---	------	" 15.2 cm
9	DTK3-0	ptran-g	Dytran 2200 VI	114	D-3	109 kPa/V	side of vessel
10							
11							
14	DTK3-3	brk wire				5 V on/off	on detonator
	3497-0	T cold junc		AD592		1 mV/K	terminal box
	3497-1	P vessel				50 psi/V	vessel lid
	3497-2	I gen				%0 A/V	input matching box
	3497-3	V gen				39 V/V	input matching box
	3497-6	pyrometer				20000 C/V	axial view thru lid
	3497-7	T test sec				Type K	30 cm below top
	3497-8	T insulation				Type K	in insulation below cruc.
	3497-9	T vessel				Type K	10 cm below vessel lid
	3497-10	T vessel				Type K	30 cm above vessel base
	3497-11	T reservoir				Type K	submerged in reservoir
	3497-12	T coil				Type K	outlet of induction coil

Tran Chan	Rcrd Chan	Type	Metrum	Vessel Cable #	Location
17 M13	G1-0	burn wire 1	13	15	see figure
18 M14	G1-1	burn wire 2	14	16	see figure
19 M15	G1-2	burn wire 3	15	17	see figure
20 M16	G1-3	burn wire 4	16	18	see figure
			-		



APPENDIX B

SCOPING EXPERIMENTS

1 SUMMARY OF SCOPING EXPERIMENTS

After the assembly of the apparatus had been completed, a series of scoping experiments were performed in the containment chamber. These experiments were preceded by several shakedown tests which involved dropping of Zircaloy melt in the absence of the interaction vessel. All scoping experiments including the shakedown tests are summarized in Table B.1. All these tests involved 200-g batches of melt. The numbering of the experiments starts with the first shakedown test, which is ZREX 1. The first successful scoping experiment (ZREX-10) is described in detail below. The remainder of the scoping tests were used to develop techniques for triggering an explosion, improve the temperature measurements, and to try dropping a mixture of zirconium and zirconium dioxide.

2 DESCRIPTION OF THE FIRST SUCCESSFUL SCOPING EXPERIMENT (ZREX-10)

ZREX-10 was the first successful scoping experiment in which a 0.2-kg mass of Zircaloy-4 melt was dropped into a column of water. The pull-plug melt release method worked very well. The primary purpose of this test was to demonstrate the workings of the experimental apparatus and procedure in the absence of an external trigger. ZREX-10 was run on August 4, 1995.

2.1 Operation

Two hundred g of Zircaloy-4 were loaded into the graphite crucible. Insulation was placed on all of the crucible surfaces. It was put into an induction coil and supported in a jig suspended from the lid of the containment vessel. The containment vessel was evacuated and back-filled with argon. Heating was started at 90% current level giving 88% power (the percent measurements are from meters on the control panel of the induction generator) and held there for the entire run. The current sensor showed 123 amps. The water pump was started and the test section filled with water as soon as the crucible had heated to a temperature where the self luminosity was enough to allow a final check of the pyrometer alignment. It took about sixty seconds to fill the test section with 305 K water. A solid piece of the Zircaloy load was visible in the TV view through the pyrometer during the initial stages of the heatup. When the temperature reached about 2100 K at 450 s after the start of heating, the piece began to shift indicating melting was occurring. After a few seconds, the piece fell out of view and the pyrometer looked directly at the melt surface. The temperature abruptly jumped to 2150 K at this time. As the indicated temperature approached 2300 K, the argon purge and vessel exhaust valves were closed in preparation for the melt dump. Closing the exhaust valves automatically isolated a sample bottle containing some of the vessel atmosphere. The temperature indicated by the pyrometer immediately dropped as smoke from the crucible partially obscured the pyrometer view of the crucible. (The record of the pyrometer temperature and the induction generator current is shown in Fig. B.1.) Argon was turned back on and the dump initiated. The melt temperature at the time of the dump is estimated to be somewhere between 2,300 and 2,400 K, although its exact value is not known due to the obscuring effects of the aforementioned smoke. The estimate was based on an extrapolation of the pyrometer readings taken prior to the appearance of smoke obscuring the pyrometer view. Sometime after the induction power was turned off, a fan inside the containment chamber was turned on, circulating the containment atmosphere for about fifteen minutes. A gas sample was then taken from the containment chamber.

2.2 Observation

Post-test examination of the apparatus showed that the operation had been a total success. The Zircaloy load had completely melted and run out of the crucible. The crucible showed very little damage, although the

mating valve surfaces were slightly roughened. All of the melt had fallen into the test section. Some water had splashed out of the test section (the water level was down about 3 cm from the top). The Zircaloy debris was removed from the test section. About 180 g of ZREX-10 debris was recovered.

The interaction between the Zircaloy melt and water was benign. There was no explosive interaction. No dynamic pressurization was recorded. The minimum resolution of the data acquisition system was 400 kPa, so dynamic pressures less than 400 kPa would not be recorded.

As indicated above, the debris had dark grey or black hue and a globular shape. Most of the debris consisted of about fifty particles ranging from 5 to 20 mm in size. A few of the particles were spherical, but most were like thick flakes as can be seen in Fig. B.2. Details of the particle size data are presented in Tables B.2(a) and B.2(b).

Two samples of the containment chamber atmosphere were taken, one immediately before the melt drop and another about fifteen minutes after the melt drop. These gas samples were analyzed by the ANL Analytical Chemistry Laboratory using the mass spectrometric method. The results of the analysis are summarized in Table B.3, which shows the constituent compositions in terms of mole percent. An analysis of the hydrogen data given in Table B.3 indicates that about 9 g of zirconium or 5% of the melt reacted with the water in the ZREX-10 experiment.

Table B.1 Summary of scoping experiments

Test No.	Setup	Purpose	Result
ZREX-1	Side view pyrometer, direct pull on valve, zirconia felt insulation top, bottom and side, flat bottom crucible, dry test section. 200 g Zircaloy-4 load.	First test of apparatus	Partial melting. Valve opened. No melt drop.
ZREX-2	Fire brick insulation added top and bottom	Insulation test	Valve did not open. No melt drop. Fire brick melted
ZREX-3	Zirconia and alumina board added to top and bottom insulation. Bottom insulation had 1 mm felt covering drop hole.	Insulation test	Valve opened. Melt collected and frozen on bottom 1mm felt insulation.
ZREX-4	Hole cut in bottom insulation beneath drop hole	Insulation test.	Valve opened. Melt dropped and collected in a sand bucket
ZREX-5	Changed location of side pyrometer hole	Temperature measurement test.	Did not attempt to open valve. Fully melted load.
ZREX-6	Moved pyrometer to axial position looking down on melt surface. Indirect pull on valve. Conical bottom crucible. Water to be added from external reservoir.	Test new pyrometer position and crucible design. Try water addition.	Run aborted due to failure of water to fill test section.
ZREX-6a	Same as ZREX-6. Re-try without disassembling apparatus	Above	Water added but melt did not drop. Fully melted load.
ZREX-7	Same as ZREX-6	Try water addition and dump.	Water addition very slow. Valve did not open
ZREX-8	Current and voltage sensors added to induction work coil	Try water addition and dump	Valve did not open. No melt drop. Low temperatures indicated. Found dirty pyrometer window causing errors of about 300 K. water addition very poor.
ZREX-9	Pump added in water fill line	Try water addition	Run aborted due to pyrometer view shift. Again found dirty pyrometer window giving 300 K error. Fully melted load although temperature indication below melting point.
ZREX-10	Same as ZREX-9	Try water addition and dump.	Successful test. No explosion. Melt fragmented. Dirty pyrometer window

Table B.1 Summary of scoping experiments (cont'd)

Test No.	Setup	Purpose	Result
ZREX-11	Same as ZREX-10	Try water addition and dump.	Successful test. No explosion. Dirty pyrometer window. Dirt sample sent for analysis
ZREX-12	Added an explosive trigger of blasting cap and detonating cord. Firing signal obtained from melt through of a burn wire grid suspended beneath the crucible. Adjustable delay in signal circuit.	Try a triggered drop	Crucible valve leaked. Trigger fired long after melt drop.
ZREX-13	Replace burn wire firing signal with valve cylinder motion signal.	Try a triggered drop	Valve opened. Trigger did not fire.
ZREX-14	Same as ZREX-13	Try a triggered drop	Valve did not open. Trigger fired after programmed delay sending jet of water into crucible. Explosion in crucible.
ZREX-15	Same as ZREX-14	Try a triggered drop	Valve did not open. Trigger fired after programmed delay. No explosion.
ZREX-16	60w/o Zr, 40 w/o ZrO_2 load. Changed from Zircaloy to Zr as Sn from Zircaloy was coating pyrometer window causing erroneous temperature readings. Added three burn wire grids submerged in test section water	Try a non-triggered drop with Zr/ZrO_2 melt. Measure fall rate in water.	Valve did not open. No drop
ZREX-17	Same as ZREX-16 with an array of four burn wire grids.	As above	Temperature low but attempt made any way. Valve opened but melt only sagged without falling. Apparently not completely melted.
ZREX-18	Same as ZREX-17 with the addition of a Type-C thermocouple in the melt.	As above	The pyrometer temperature indicated about 2300 C and the thermocouple 2050 C at the time of the drop. The valve opened and a few drops fell but most remained in the crucible and hanging from it as a stalactite.

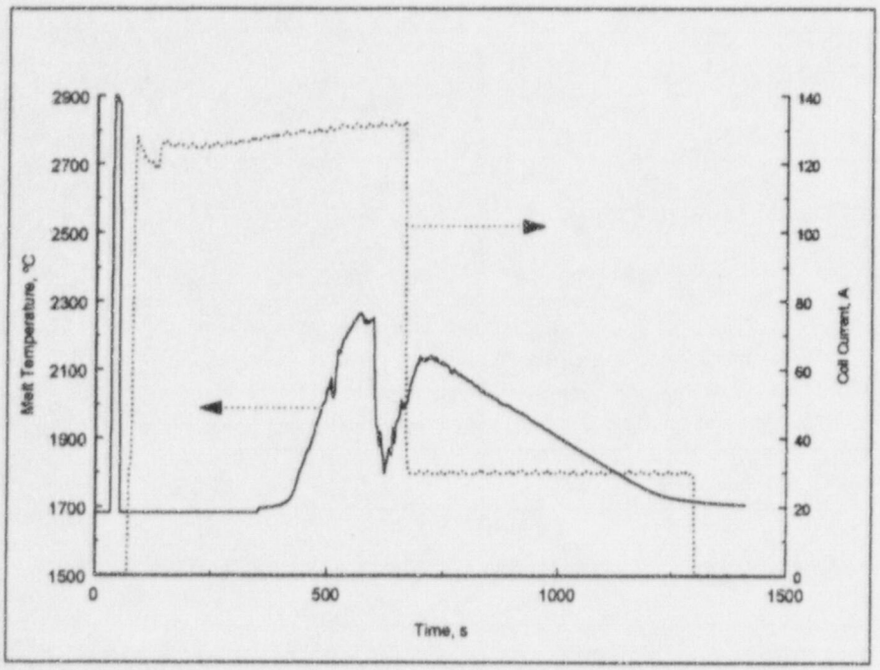


Figure B.1 ZREX-10 Melt Heating Record

Figure B.2 ZREX-10 Debris

Table B.2(a). Particle Size Data for ZREX-10
(Individually measured flakes)

Particle #	Length (maximum) mm	Width (maximum) mm	Thickness mm	Weight g
1	18.0	11.4	1-7	4.42
2	26.4	9.9	3-5	4.40
3	27.2	16.0	1-5	4.35
4	27.4	14.0	1-5	4.04
5	22.4	14,5	1-7	4.02
6	18.0	13.2	2-7	3.38
7	25.7	16.5	1-3	3.22
8	19.3	11.9	2-4	3.16
9	20.8	14.0	1-4	3.09
10	15.0	13.5	1-5	3.07
11	17.5	14.5	1-4	3.03
12	19.8	17.0	1-5	2.83
13	24.4	15.5	1-3	2.81
14	22.4	11.9	1-3	2.77
15	30.5	15.5	1-3	2.50
16	18.5	10.4	1-5	2.39
17	21.6	11.9	1-6	2.30
18	14.2	10.2	2-5	1.67
19	13.0	9.9	1-5	1.56
20	13.2	7.9	2-4	1.48
21	22.4	8.1	1-2	1.34
22	14.2	8.6	1-3	0.83
23	14.5	6.4	1-3	0.68
24	11.9	6.1	1-2	0.60

Table B.2(a) (contd.)

Particle #	Length (maximum) mm	Width (maximum) mm	Thickness mm	Weight mm
25	12.7	6.4	1-2	0.60
26	14.0	4.8	1-2	0.46
27	13.7	4.3	1-2	0.38
28	10.2	8.4	1-2	0.30
29	15.0	6.1	1-2	0.27
30	10.4	7.6	1-2	0.26

Table B.2(b). Particle Size Data for ZREX-10
(sieved globular particles)

Particle #	Mesh mm	Weight g
31-71	+6.3	95.10
72-94	-6.3, +3.4	7.67
95-125	-3.4, +2.4	2.10
~25 particles	-2.4, +2.0	0.65
	-2.0	0.20

Total weight = 171.9 g

Table B.3 Gas Sample Analysis Results for ZREX-10

	Ar	H_2	CO	N_2	O_2	CO_2
Before	96.0	0.277	0.436	1.45	0.154	0.030
After	93.8	1.77	1.41	2.22	0.105	0.107

APPENDIX C

SUMMARY OF DATA EXPERIMENTS (ZREX-19 TO ZREX-52, ZRSS-1 TO ZRSS-11)

Table C.1 Summary of data experiments with Zr-ZrO_2 mixtures

Test No.	Zr %	Melt Source	Melt Temp. K	Water Temp. K	Crucible[a]	Bottom Insulation[b]	Trigger	Success	Result
ZREX-19	100	rod	2278	295	type-1	type-A	no	yes	No explosion. Residue was ribbon-like or ropy. Minor fragmentation. Minor to moderate hydrogen production.
ZREX-20	100	rod	2373	296	type-1	type-A	yes	yes	Explosion. Fine debris. Extensive hydrogen production.
ZREX-21	90	powder	2573	293	type-1	type-A	no	yes	No explosion. Residue was a long, thin, ropy mass. Minor fragmentation. Moderate hydrogen production.
ZREX-22	90	powder	2583	295	type-1	type-A	yes	maybe	Premature trigger caused by leak of melt. Explosion. Fine debris. Extensive hydrogen production.
ZREX-23	100	rod	2273	343	type-1	type-A	no	yes	No explosion. Residue was a long, thin, ropy mass. Minor fragmentation. Moderate hydrogen production.
ZREX-24	100	rod	2373	367	type-1	type-A	yes	yes	Explosion. Fine debris. Very extensive hydrogen production
ZREX-25	60	ingot	2573	298	type-1	type-A	no	no	Crucible valve did not open. No melt drop.
ZREX-25a	60	ZREX-25	2583	298	ZREX-25	type-A	no	yes	No explosion. Moderate fragmentation. ~25% of debris expelled from the test section. Moderate hydrogen production.
ZREX-26	90	ingot	2583	371	type-1	type-A	yes	no	Crucible valve did not open. No melt drop.
ZREX-26a	90	ZREX-26	2581	371	ZREX-26	type-A	yes	no	Crucible valve did not open. No melt drop
ZREX-26b	90	ZREX-26a	2623	368	ZREX-26a	type-A	yes	no	Crucible valve did not open. No melt drop

[a] See Figs. C.1 - C.3

[b] See Figs. C.4 - C.8

Table C.1 Summary of data experiments with $Zr-ZrO_2$ mixtures (contd.)

Test No.	Zr %	Melt Source	Melt Temp. K	Water Temp. K	Crucible[a]	Bottom Insulation[b]	Trigger	Success	Result
ZREX-27	90	powder	2473	~363	type-1	type-A	no	yes	No explosion. Moderate fragmentation. Over 50% of debris expelled from the test section. Moderate to extensive hydrogen production.
ZREX-28	90	powder	2473	363	type-1	type-A	yes	maybe	Crucible valve partially opened, producing a thin melt stream. Explosion. Fine debris. Extensive hydrogen production.
ZREX-29	80	powder	2573	300	type-1	type-A	yes	no	Crucible valve did not open. No melt drop
ZREX-29a	80	ZREX-29	2593	296	ZREX-29	type-A	yes	no	Crucible valve did not open. No melt drop
ZREX-30	80	powder	2523	293	type-1	type-A	yes	no	Crucible valve did not open. No melt drop
ZREX-31	80	ZREX-30	2473	294	type-2	type-A	yes	no	Crucible valve did not open. No melt drop
ZREX-32	60	powder	2573	293	type-2	type-A	yes	no	Crucible valve did not open. No melt drop
ZREX-33	60	ZREX-32	2573	294	type-3[c]	type-A	yes	no	Crucible valve did not open. No melt drop
ZREX-34	60	ZREX-33	2573	293	type-3	type-A	yes	yes	Explosion. Fine debris. Extensive hydrogen production
ZREX-35	80	ZREX-29& 31	2573	293	type-3[d]	type-A	yes	no	Crucible valve did not open. No melt drop.
ZREX-36	80	ZREX-35	2623	291	ZREX-35	type-B	yes	yes	Explosion. Fine debris. Extensive hydrogen production.
ZREX-37	50	ingot	2673	291	type-2	type-B	yes	no	Melt leaked and froze in bottom insulation hole. No melt drop
ZREX-38	50	ingot	2723	290	type-2	type-B	yes	no	Melt leaked and froze in bottom insulation hole. Partial melt drop. No triggering. No explosion.
ZREX-39	50	ingot	2723	292	type-3	type-B	yes	no	Melt leaked and froze in bottom insulation hole. No melt drop

[a] See Figs. C.1 - C.3
[b] See Figs. C.4 - C.8
[c] See Fig. C.10
[d] See Fig. C.11

Table C.1 Summary of data experiments with Zr-ZrO_2 mixtures (cont'd.)

Test No.	Zr %	Melt Source	Melt Temp. K	Water Temp. K	Crucible[a]	Bottom Insulation[b]	Trigger	Success	Result
ZREX-40	70	ingot	2573	291	type-3	type-B	yes	no	Melt leaked and froze in bottom insulation hole. No melt drop
ZREX-41	90	powder	2473	293	type-2	type-C	yes	yes	Explosion. Fine debris.
ZREX-42	70	powder	2573	297	type-2	type-C	yes	no	Crucible valve did not open. No melt drop
ZREX-43	70	ZREX-42	2623	293	ZREX-42	type-D	yes	no	Crucible valve did not open. No melt drop
ZREX-44	70	ZREX-43	2648	293	ZREX-43	type-B	yes	no	Crucible valve did not open. No melt drop
ZREX-45	90	powder	2423	304	type-5	type-C	yes	no	Water leaked from test section. Partial melt drop. No explosion.
ZREX-46	100	rod	2373	301	type-5	type-C	yes	no	Melt leaked and froze in bottom insulation hole. No melt drop
ZREX-47	100	rod	2373	298	type-4	type-A	yes	yes	Explosion. Extensive damage to apparatus
ZREX-48	90	powder	2300-2400 (?)	302	type-4	type-E	yes	no	Pyrometer field-of-view shifted during heating. Incomplete melting.
ZREX-49	90	powder	2523	297	type-4	type-E	yes	no	Crucible valve did not open. No melt drop
ZREX-50	100	rod	2373	299	type-4	type-E	yes	yes	Explosion. Extensive damage to apparatus.
ZREX-51	100	rod	2373	366	type-4	type-E	yes	no	Melt leaked and froze in bottom insulation hole. No melt drop
ZREX-52	100	rod	2373	372	type-4	type-E	yes	no	Crucible valve did not open. No melt drop

[a] See Figs. C.1 - C.3

[b] See Figs. C.4 - C.8

Table C.2 Summary of data experiments with Zr-SS Mixtures

Test No.	Zr %	Melt Source	Melt Temp. K	Water Temp. K	Crucible[a]	Bottom Insulation[b]	Trigger	Success	Result
ZRSS-1	36	powder shot	2083	no water	type-6	type-A	no	yes	Attack of crucible by melt was minimal
ZRSS-2	36	powder shot	2213	no water	type-6	type-A	no	yes	No water in the test section. Melt dropped into the empty test section.
ZRSS-3	36	powder shot	2213	no water	type-6	type-A	no	no	Melt dropped prematurely before the test section was filled with water.
ZRSS-4	36	powder shot	2153	293 (est.)	type-5	type-A	no	yes	Melt quenched without explosive violence. Some fragmentation.
ZRSS-5	36	powder shot	2163	293	type-5	type-A	yes	yes	Explosion. Damage less than Zr/ZrO explosions.
ZRSS-6	0	shot	2153	295	type-5	type-A	no	yes	Melt quenched without explosive violence. Some fragmentation.
ZRSS-7	0	shot	2043	296	type-5	type-A	no	yes	Melt dropped into test section. Trigger failed to detonate due to damage to burn wires. No explosion.
ZRSS-8	0	shot	2123	297	type-5	type-A	yes	yes	Explosion. Damage similar to ZRSS-5
ZRSS-9	0	shot	2123	300	type-5	type-A	yes	yes	Explosion. Damage similar to ZRSS-8
ZRSS-10	60	powder shot	2173	301	type-5	type-A	yes	yes	Explosion. Damage similar to Zr/ZrO explosions.
ZRSS-11	80	powder shot	2123	304	type-5	type-A	yes	yes	Explosion. Damage similar to Zr/ZrO explosions.

[a] See Figs. C.1 - C.3
[b] See Figs C.4 - C.8

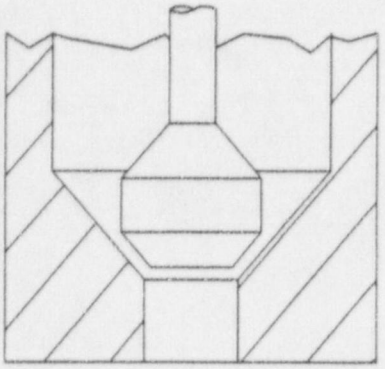


Figure C.1 Type-1 Crucible

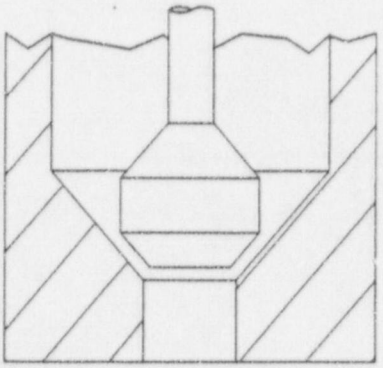


Figure C.2 Type-2 Crucible

Type-3 is a Type-2 valve coated with two layers of TiC slurry, air-dried, followed by two coats of an aerosol spray of Y_2O_3 powder. The crucible was then baked at 530 K for four hours.

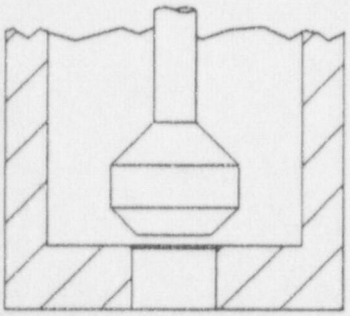


Figure C.3 Type-4 Crucible

Type-5 is a Type-4 coated with two layers of Y_2O_3 each baked at 875 K.
Type-6 is a Type-2 coated with two layers of Y_2O_3 each baked at 875 K.

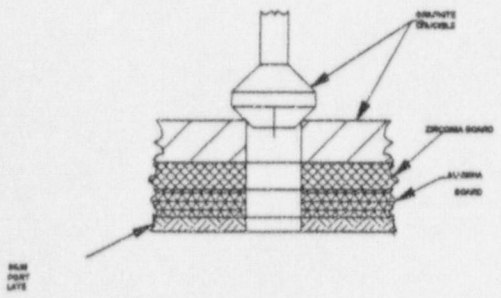


Figure C.4 Type-A Insulation

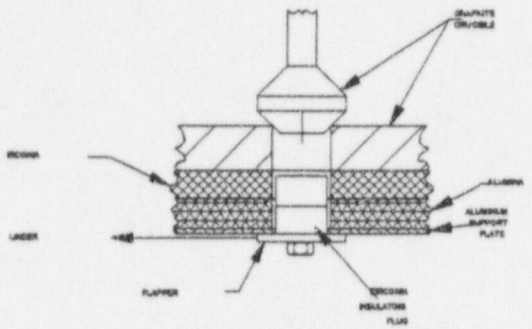


Figure C.5 Type-B Insulation

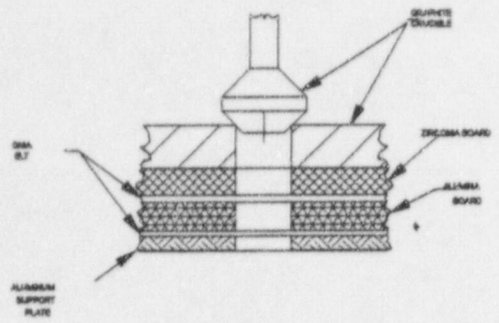


Figure C.6 Type-C Insulation

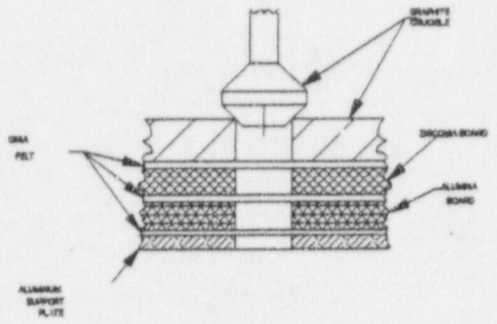


Figure C.7 Type-D Insulation

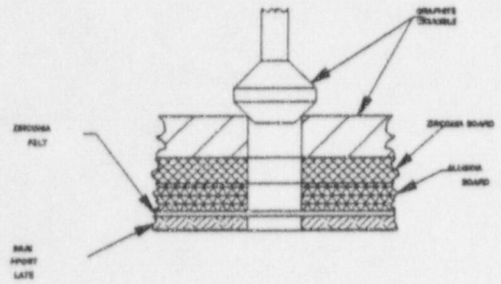


Figure C.8 Type-E Insulation

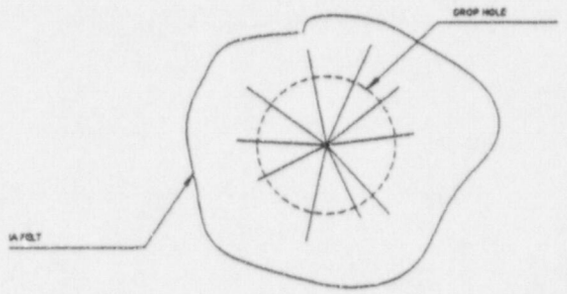


Figure C.9 Slitting Pattern in Zirconia Felt Used in Type-C, D, and E Insulation

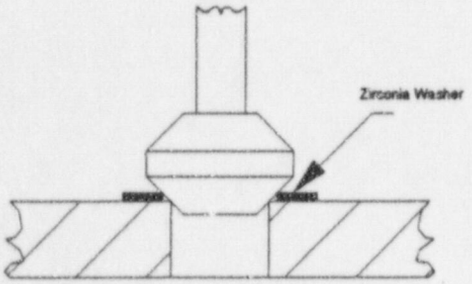


Figure C.10 ZREX-33 Valve Detail

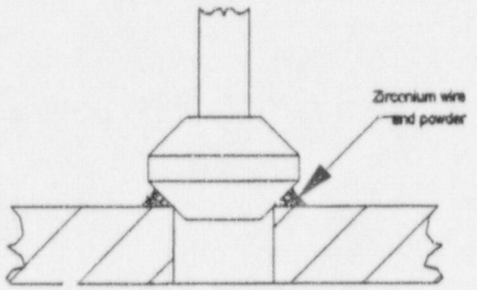


Figure C.11 ZREX-35 Valve Detail

APPENDIX D

DETAILED DESCRIPTION OF SUCCESSFUL EXPERIMENTS

1 ZREX-19

ZREX-19 was run on February 7, 1996. This experiment was an un-triggered drop of one kg of molten zirconium metal into room-temperature water. A set of four burnwire frames were suspended beneath the crucible. The top frame (#1) was placed 7 cm below the crucible in the argon atmosphere. The other three frames were beneath the water in the test section at depths of 1/2 (#2), 38-1/2 (#3), and 77-1/2 (#4) cm below the surface. Each wire frame was made a part of a resistive divider attached to a voltage source.

1.1 Operation

1003 g of 1.3-cm diameter zirconium rod was loaded in the crucible. Heating was started at 90% current level giving 82% power and held there for most of the run. Current as measured by the current sensor showed ~130 A. The water pump was started and the test section filled with room-temperature (295 K) water. Heating proceeded normally until the temperature reached about 2103 K when the slope of the heating rate curve decreased. No obvious change was seen in the view through the crucible lid but a few seconds later at an indicated temperature of 2131 K the zirconium rods started to move, indicating the incipient melting of the rods. This indication of melting is very consistent with the known melting point of pure zirconium, which is 2125 K. The rods continued to melt and move with erratic temperature readings until the surface of the molten pool could be seen and the pyrometer showed the temperature to be 2190 K. Heating continued normally to 2273 K when the decision to drop was made (see the pyrometer record shown in Fig. D.1). The 2900 K spike early in the temperature record is a calibration mark used to calibrate the optical pyrometer. A similar spike is seen in most of the pyrometer records. The temperature increased another 5 K while the drop procedures were initiated. Molten material dropped out of the crucible in about 0.5 s, dropping somewhat aslant of vertical (as seen from the VCR recording of the drop zone camera).

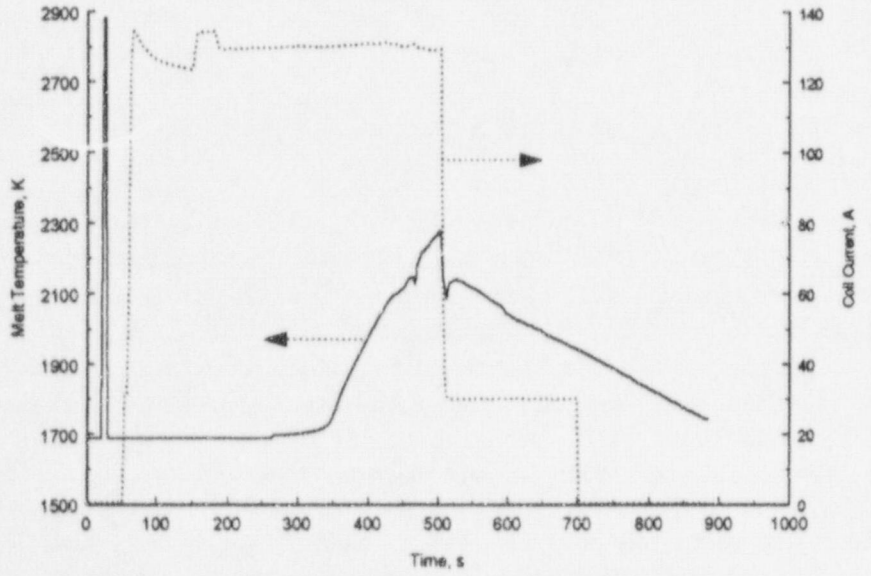


Figure D.1. ZREX-19 Melt Heating Record.

1.2 Observations

The interaction between the zirconium melt and water was benign. There was no explosion. No dynamic pressures were registered by the test section pressure transducers. The test section thermocouples failed, apparently because the melt had fallen on the thermocouple wires where they left the test section. Thus, no data is available on the temperature change in the test section. However, the thermocouple in the gas space 10 cm below the top of the containment chamber indicated an increase in temperature from 347 K to 377 K. Also, as shown in Fig. D.2, the containment chamber pressure jumped from 106 kPa to 121 kPa. This pressure jump was probably caused by a combination of the generation of hydrogen and steam by the melt/water reaction and the rapid heating of the containment gas by the reaction and the falling melt. The water level in the test section was down about 15 cm from the top after the test.

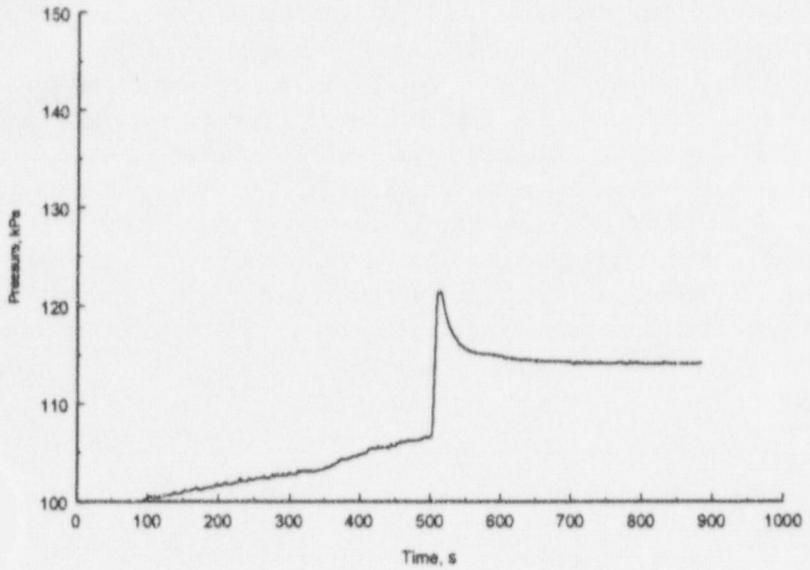


Figure D.2. ZREX-19 Containment Pressure

The burnwires beneath the crucible (#1) and at the top of the test section (#2) were both burned through with a delay between them of about 0.05 s. There was an indication that the bottom wire (#4) burned out 0.33 s later. However, post-test examination showed that the lead wire at the top of the test section rather than the burnwire array had failed. The middle (#3) and bottom (#4) burnwire arrays never opened. Post-test examination of the apparatus showed all (except for a few grams in the valve hole) of the melt in the crucible had fallen out. A small amount (25 g) remained in the hole through the insulation immediately under the crucible. A circular pattern on the bottom of the crucible formed by the hole in the underlying insulation showed a slight misalignment of the crucible and bottom insulation that probably explains the slightly off-vertical drop of the melt. A large amount (254 g) of the frozen melt was suspended from the top edge of the test section and #2 burnwire frame with the appearance of a cylindrical stalactite. A piece of melt with the appearance of a folded ribbon and weighing 399 g was resting on the middle submerged burnwire (#3). This ribbon shape varied from 2 to 6 cm wide and a few mm thick. An additional 160 g of lump material also was on the (#3) burnwire. The wire was not significantly damaged. A small amount (25 g) of particles (several millimeters in size) were found in the catch can below the bottom burnwire. An additional 82 g of melt fell outside the test section and was collected from the containment vessel floor. A photograph of the debris is shown in Fig. D.3.

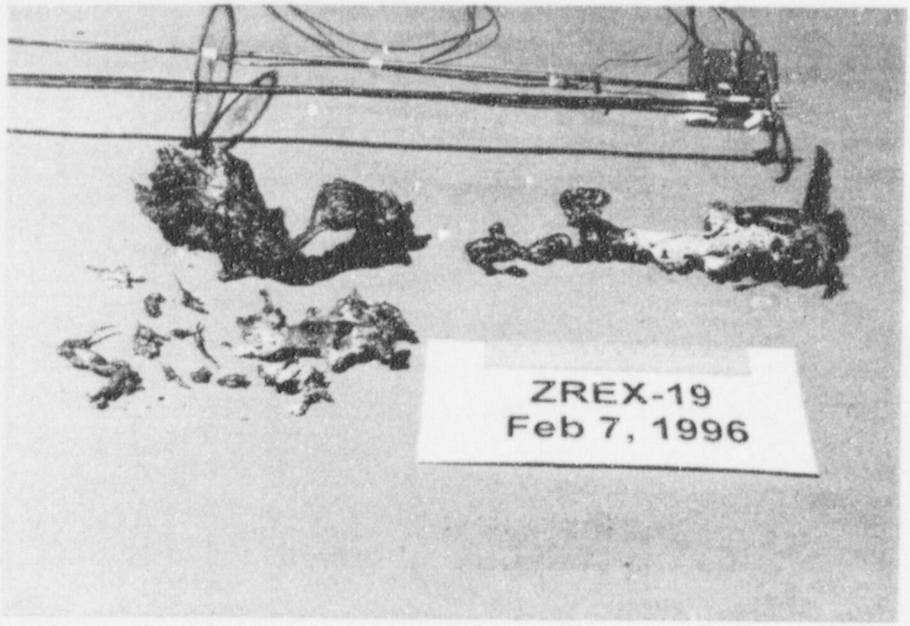


Figure D.3. ZREX-19 Debris.

2 ZREX-20

ZREX-20 was run on February 23, 1996. This experiment was a triggered drop of one kg of molten zirconium metal into room-temperature water. The trigger was the detonation of a length of Primacord containing 1 g of PETN (Penta Erythritol Tetra Nitrate). The start signal for the trigger was generated by the melting through of a wire grid 20 cm below the top of the test section. (There was another burnwire grid 1 cm lower for redundancy.) This choice of the location for the trigger start signal was based on consideration of possible rapid quenching of the melt in the water. The results of ZREX-19 seemed to indicate that the melt could quench and freeze within a water depth of 39 cm. It was thought that the 20-cm water depth would be sufficient to submerge the 1-kg melt while minimizing quenching so that the melt is fully molten at the time of triggering.

2.1 Operation

1004 g of 1.3-cm diameter zirconium rod was loaded into the crucible. The containment vessel was evacuated and backfilled with argon. Heating was started at 90% current level giving 82% power and held there for most of the run. Current as measured by the current sensor showed ~130 A.

The water pump was started and the test section filled with 296 K water. Heating proceeded normally with no obvious change seen in the view through the crucible lid until at an indicated temperature of 2187 K the zirconium rods started to move, indicating the incipient melting of the rods. They continued to melt and move with erratic temperature readings until the surface of the molten pool could be seen and the pyrometer showed the temperature to be 2263 K (see the pyrometer record shown in Fig. D.4). The heating rate was faster than

earlier runs probably due to the reduced crucible wall thickness and consequent improved coupling efficiency into the zirconium melt. Heating continued normally to 2323 K when the trigger circuit was armed. At this time a leak occurred with a number of individual small drops falling out of the crucible. (These drops were small enough that they probably solidified while falling through 20 cm of water before contacting the trigger burnwire). This prompted the decision to drop the melt which was done at 2373 K. Molten material dropped out of the crucible in about 0.5 s, falling directly into the crucible and initiating the trigger.

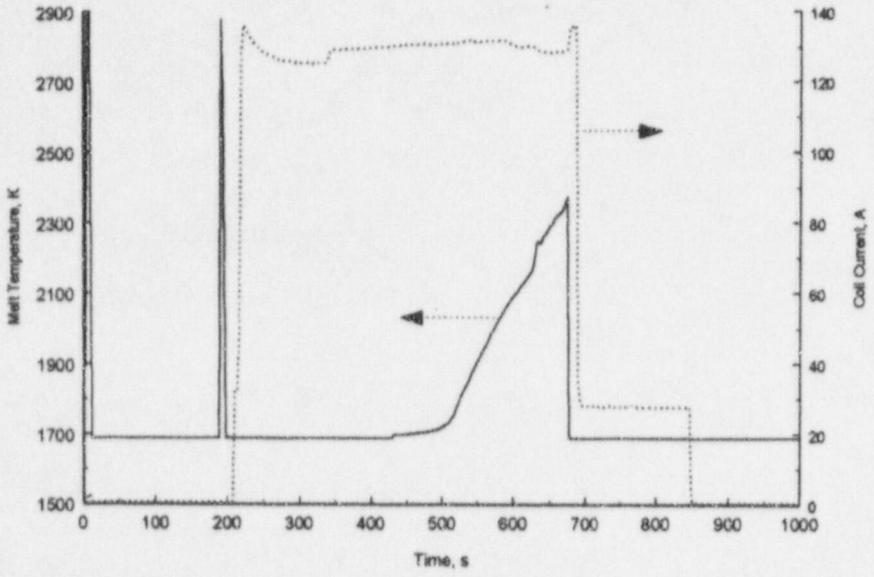


Figure D.4. ZREX-20 Melt Heating Record.

After the melt drop, the induction power was turned off and gas samples of the containment chamber were collected and stored for later analysis.

2.2 Observations

An explosion took place. Post-test examination of the apparatus showed that much damage had been done. All of the mechanism hanging from the vessel lid was destroyed (see Fig. D.5). The crucible was shattered. The induction coil, crucible support, valve opening gear, and sight tube were bent or torn. The lid and wall of the vessel were coated with a thick (~1 mm) layer of grey dust (36 g on the lid and 243 g on the wall) . The bottom of the vessel contained large pieces of debris (probably crucible pieces, 1147 g) and was coated with a mud made up of water and more of the grey dust along with small pieces of debris (1354 g) (see Fig. D.6). A few fragments identifiable as zirconium were found in the bottom debris (47 g). The collector at the bottom of the test section had zirconium particles and thin flattened globules weighing a total of 98 g. When inspected under magnification (30x), the dust had the appearance of a collection of transparent fibers, small white spheres and small black spheres. A scanning electron microscope (SEM) photograph of the debris is shown in Fig D.7. The three types of insulation used in the experiment, namely, alumina board, zirconia board, and zirconia felt, consisted entirely of fibrous particles with no spherical parts. Thus we can assume that the spheres are zirconium and zirconia from the explosion.

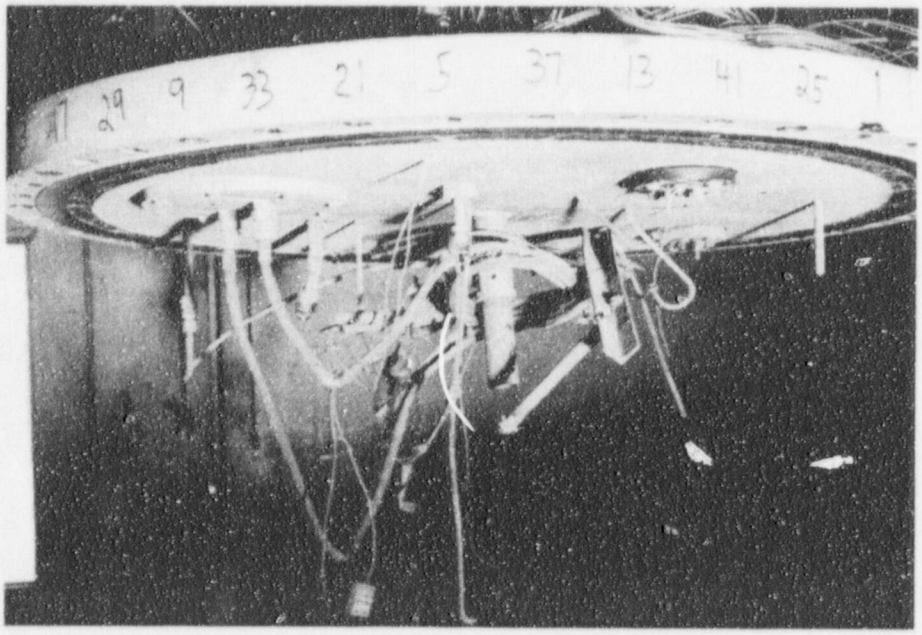


Figure D.5. ZREX-20 Containment Vessel Lid.

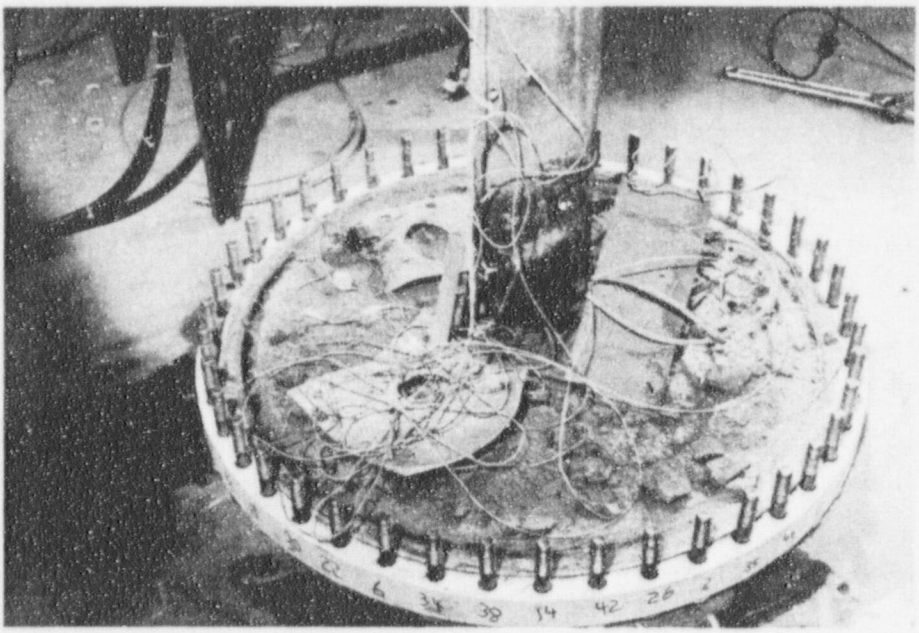


Figure D.6. ZREX-20 Debris.

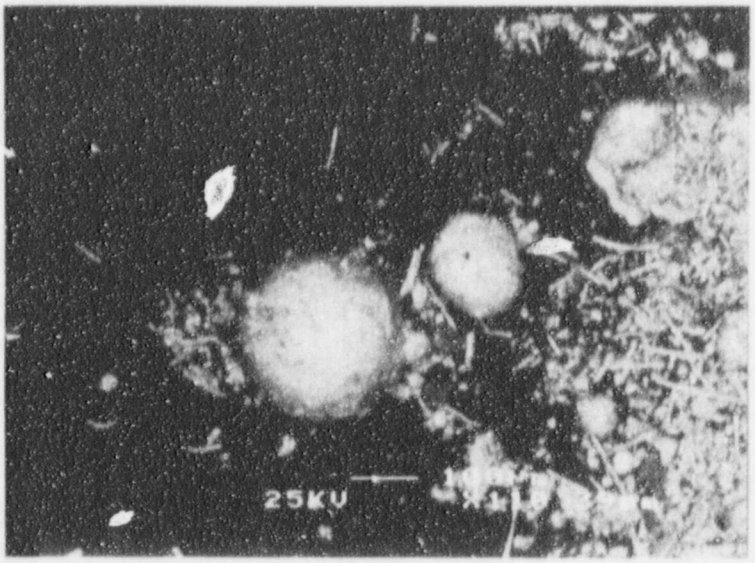


Figure D.7. ZREX-20 SEM Photo of Debris.

The burnwire at the top of the test section showed the making and breaking of the circuit a number of times (60) before it permanently opened. The onset of these signals happened 12.5 s before the signal from the 20 cm deep underwater burnwire that triggered the explosion. This agrees well with a 12 s delay between the beginning of leak and the trigger estimated from the video tape of the drop zone camera which also showed about 75 individual drops from the leak. Two of the three burnwire grids were gone. The third one at 21 cm below the top of the test section remained. This suggests that the lowest part of the explosion zone was between 20 and 21 cm.

The slow instruments all responded to the explosion. The lower test section thermocouple failed while the upper jumped from 296 K to over 770 K and back to 359 K. A thermocouple trace indicated a temperature increase from 341 K to 692 K in the gas space under the top cover of the containment chamber. At the same time, the containment vessel pressure jumped from 108 kPa to 512 kPa, probably caused by the rapid heating of the containment gas coupled with a sudden release of gas (hydrogen mixed with steam) (see Fig. D.8).

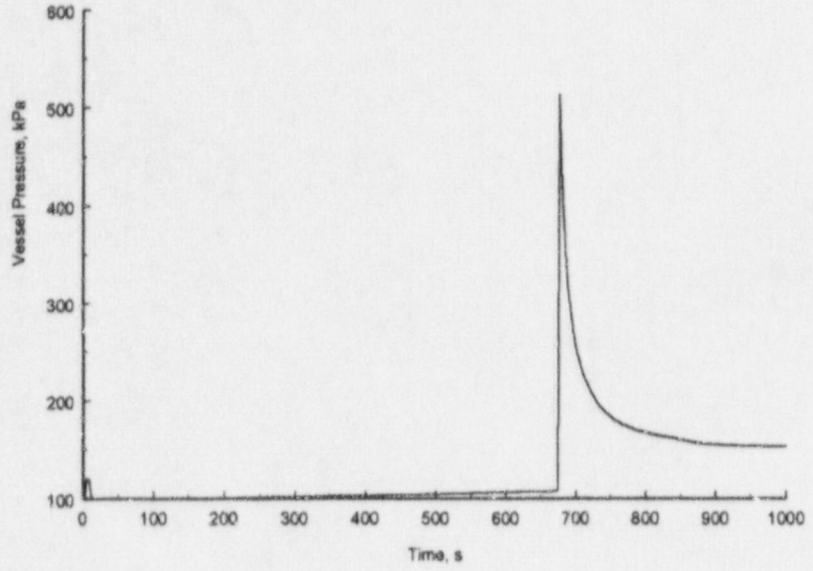


Figure D.8. ZREX-20 Containment Pressure.

The fast instruments, i.e, the force link and dynamic pressure transducers all produced signals, but it was later found that only two of the five pressure transducers in the test section were functioning. One of the two working pressure transducers was located at 15 cm below the top of the test section and probably surrounded by a multi phase mixture in the interaction zone. The pressure trace of this transducer is so noisy that it would be extremely difficult to extract useful information from this trace (see Fig. D.9). The other working pressure transducer was located at 30 cm below the top of the test section. The pressure trace of this transducer is shown in Fig. D.10. It shows the electrical noise pulse from the trigger at about 1.5 ms followed by a noisy pressure pulse of about 3 ms duration with an averaged peak pressure about 40 MPa above the baseline. Probably the best data was obtained from the force transducer supporting the test section. It is shown in Fig. D.11. It shows a force pulse, starting about 1.5 ms after the trigger, with a width of about 3 ms and a peak force of ~ 360 kN. Fig. D.12 shows the output of the fast response containment pressure transducer mounted in the sidewall of the containment vessel. It has a high noise level of 10 kHz from the induction generator, an electrical spike at the trigger time, and a very high spike at about 2.3 ms that does not correlate with anything.

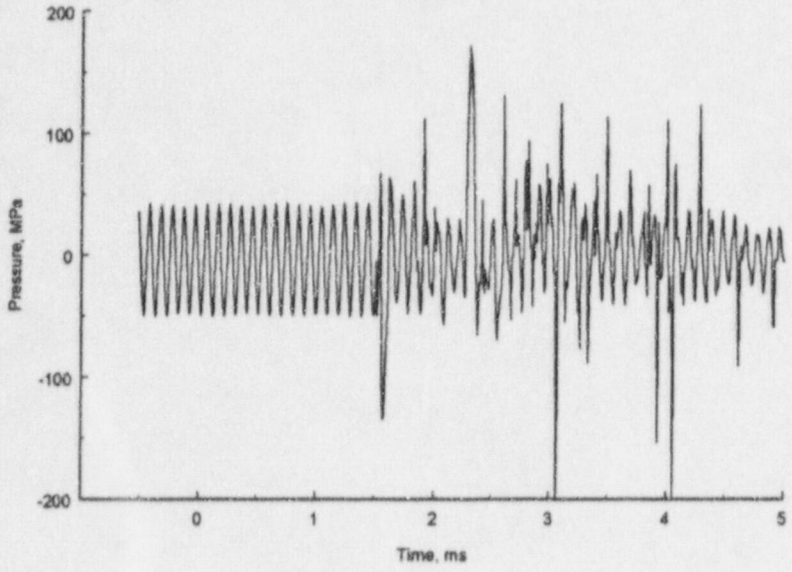


Figure D.9. ZREX-20 P-8 Pressure Record.

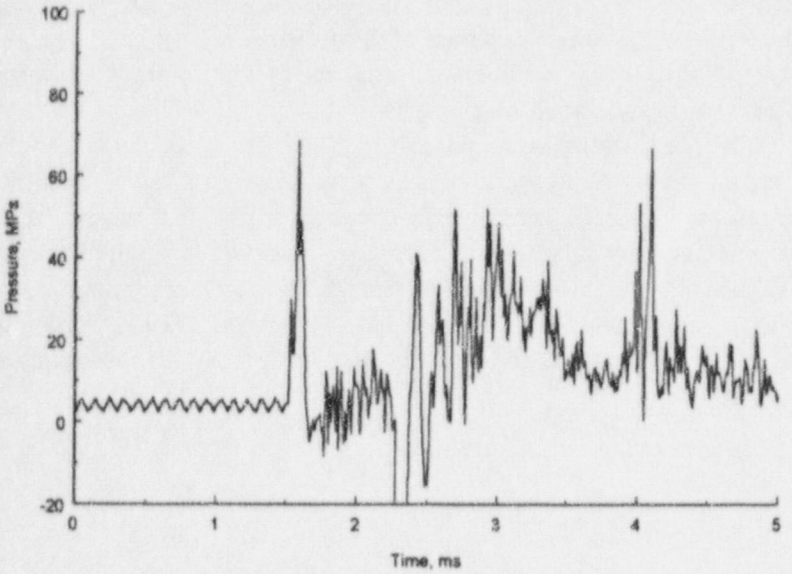


Figure D.10. ZREX-20 P-6 Pressure Record.

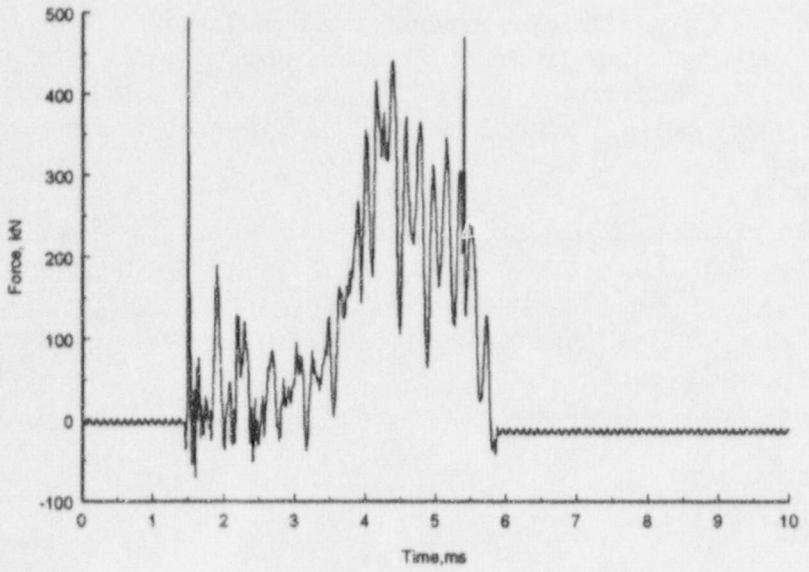


Figure D.11. ZREX-20 Force Record.

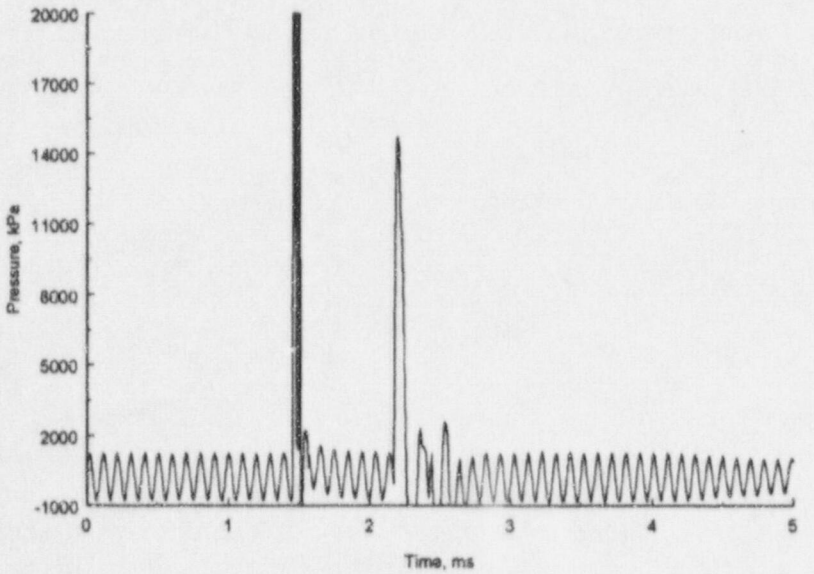


Figure D.12. ZREX-20 Fast Response Containment Pressure.

3 ZREX-21

ZREX-21 was run on March 22, 1996. This experiment was an un-triggered drop of one kg of molten mixture of 90 w/o zirconium metal and 10 w/o zirconium dioxide into room-temperature water. Four burnwire frames were placed at 5, 20, 30, and 78 cm below the top of the test section. Also, an additional type K thermocouple was installed in the containment vessel, 30 cm above the bottom and 10 cm in from the wall.

900 g of zirconium powder and 100 g of ZrO_2 powder were mixed, placed in polyethylene bags and loaded into the crucible. Heating was started at 92% current level giving 77% power and held there for most of the run. Current as measured by the current sensor showed ~ 130 A. The water pump was started and the test section filled with 293 K water. Heating proceeded normally with no obvious change seen in the view through the crucible except a few moving dark patches. Heating continued normally to 2573 K when the crucible valve was actuated to drop the melt (see the pyrometer record shown in Fig. D.13).

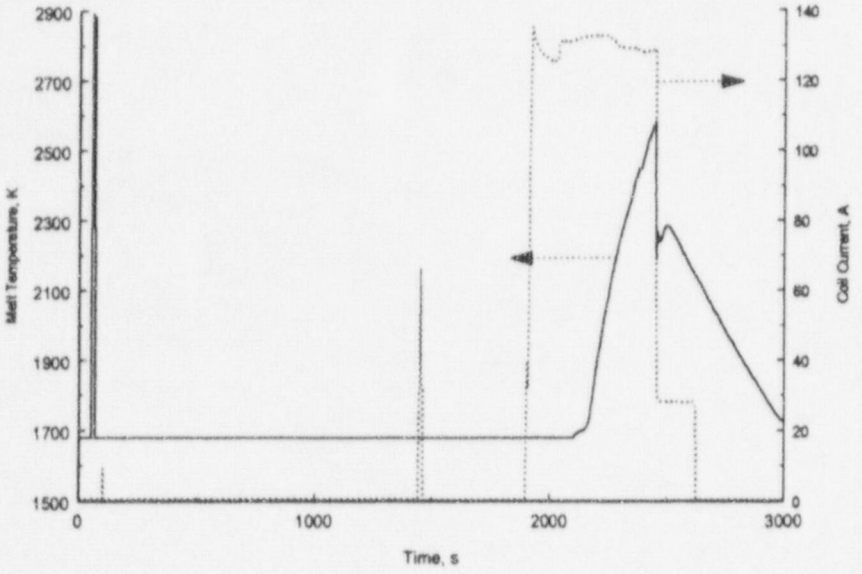


Figure D.13. ZREX-21 Melt Heating Record.

3.1 Observations

The top burnwire was found to be inoperative. The next to the top burn wire (i.e., 20 cm from the top of the test section) showed an open circuit, apparently due to contact with the melt. (The circuits of the bottom two burn wires also opened, but it was not possible to correlate this opening with the travel of the melt in the test section.) The test section thermocouples failed following the melt drop. The thermocouple in the gas space below the top of the containment vessel showed a temperature jump from 358 K to 388 K, while the vessel pressure exhibited a spike from 112 kPa to 136 kPa (see Fig. D.14). None of the fast instruments, i.e, the test section force and pressure transducers, produced perceptible signals. There was no explosion.

Post-test examination of the apparatus showed that no damage had been done. Most of the melt fell into the

test section. A small amount (20 g) was on the top edge of the test section and 180 g remained in the crucible. The material in the test section formed a ropy mass (752 g) extending from the top burn wire to below the third burn wire, a length of 30 cm. This mass was suspended by the burn wire structure. A small amount (34 g) fell through to the catch can. Most of the water remained in the test section after the experiment with the final level 17 cm below the top. A photograph of the debris is shown in Fig. D.15.

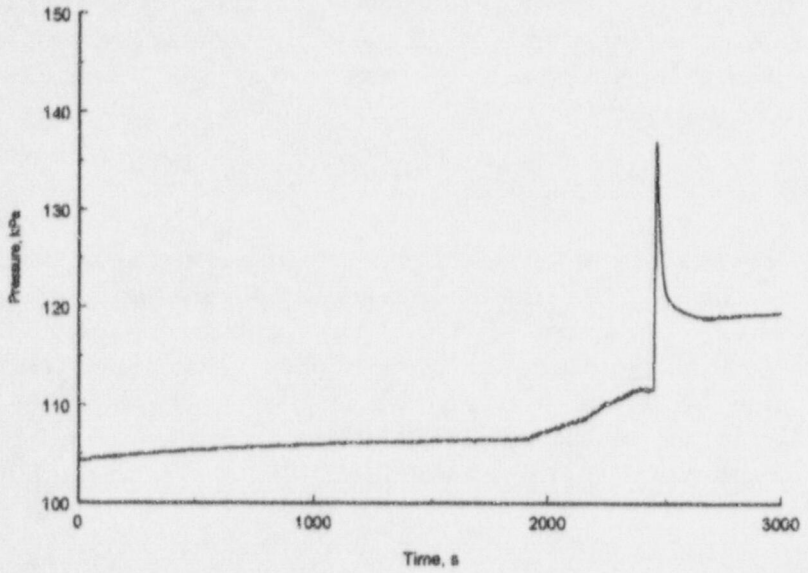


Figure D.14. ZREX-21 Containment Pressure.

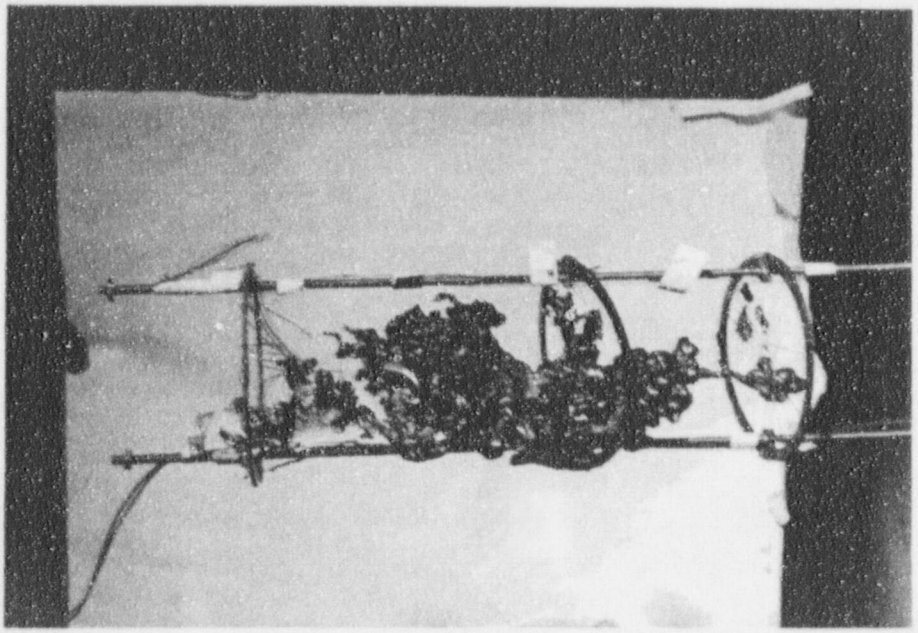


Figure D.15. ZREX-21 Debris.

4 ZREX-22

ZREX-22 was run on April 4, 1996. This experiment was a triggered drop of one kg of molten mixture of 90 w/o zirconium metal and 10 w/o zirconia into room-temperature water. Some minor modifications were made to the apparatus. The internal circulation fan was moved from the base of the containment vessel to the lid and the direction changed from horizontal to vertical. Instrumentation for ZREX-22 was identical to that for ZREX-20, including the burnwire arrangement for starting the trigger pulse. Two burnwire grids were placed 20 and 21 cm below the water level in the test section, respectively. These burnwire grids were attached to a circuit that would fire the trigger if either grid opened.

4.1 Operation

900 g of zirconium powder and 100 g of ZrO_2 powder were mixed, placed in four polyethylene bags and loaded into the crucible. Heating was started at 95% current level giving 85% power and held there for most of the run. Current as measured by the current sensor showed ~ 135 A. The water pump was started and the test section filled with water at 295 K. Heating proceeded normally with no obvious change seen in the view through the crucible except a few moving dark patches. Heating continued normally to 2570 K (see the pyrometer record shown in Fig. D.16) when the detonator circuit for the trigger was armed. Soon (~ 3 s) after, melt began to leak out of the un-opened crucible valve in a large stream. Within ~1 s, the trigger fired, ending the experiment.

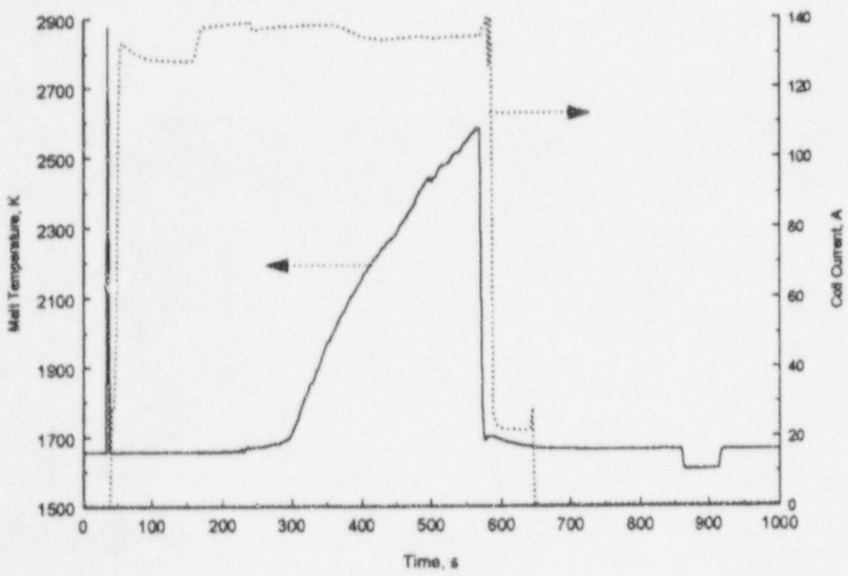


Figure D.16. ZREX-22 Melt Heating Record.

4.2 Observations

An explosive interaction similar to that of ZREX-20 took place. Post test examination of the apparatus showed that much damage had been done although the damage was somewhat less that ZREX-20. All of the

mechanism hanging from the vessel lid was destroyed. The crucible was broken into a few large pieces. The induction coil, crucible support, valve opening gear, and sight tube were bent or torn. The lid and wall of the containment vessel were coated with a patchy layer of gray dust. The total weight of debris attached to the vessel lid including large pieces of crucible resting on the support platform was 819 g, while the weight of dust on the wall was 81 g. The bottom of the vessel was coated with a mud made up of water from the experiment and more of the gray dust and filled with pieces of debris. The total weight of debris on the vessel bottom was 2197 g. A small amount of material (139 g) was in the collector cup at the bottom of the test section. The total debris weight was 3236 g, which is 58 g less than the combined weights of the loaded crucible plus all of the insulation. The debris from the vessel floor was sifted through a #6 U.S. standard sieve and the retained material washed with water and manually sorted for fragments with a metallic appearance, a total of 207 g.

It appears that the explosion was triggered by the burnwire located 20 cm below the water surface. The two thermocouples of the test section jumped to over 373 K and returned to less than 313 K within 200 s. The temperature in the containment vessel jumped from 344 to 387 K for the upper thermocouple and 301 to 559 K for the lower one, while the vessel pressure increased from 114 kPa to 359 kPa (see Fig. D.17).

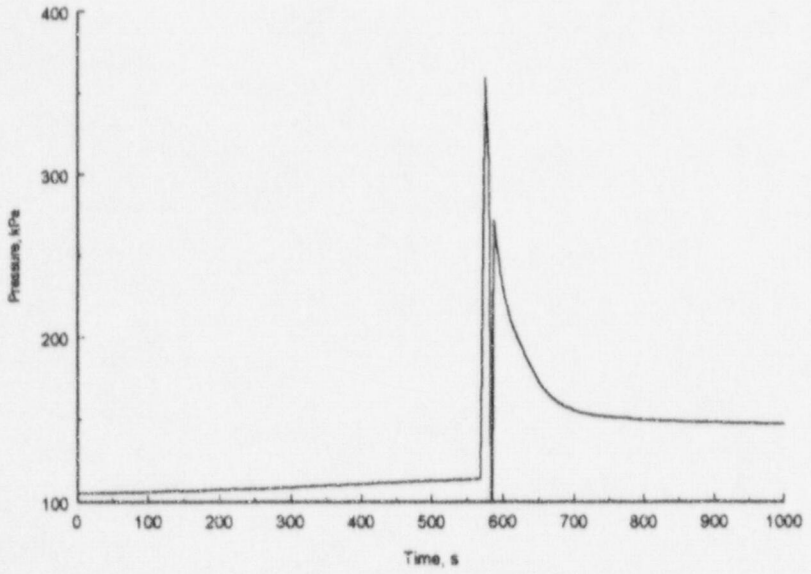


Figure D.17. ZREX-22 Containment Pressure.

An examination of the test section pressure and force traces indicates that the explosion started shortly after the trigger pressure pulse (delayed by passage through the test section water column) reached the falling melt. The peak pressure in the test section was estimated to be about 25 MPa. The pressure transducer signals were all quite noisy, due mainly to the induction generator being at full power during the explosion. The one pressure transducer that used an external amplifier (P-6, located 30 cm deep) had a fairly small noise and is shown in Fig. D.18. The reaction force trace had a peak value of about 300 kN with negligible noise and is shown in Fig. D.19. Another indication of the explosion event was the gas shock pressure recorded by the dynamic pressure transducer mounted on the wall of the containment chamber at the same elevation as the top of the test section. The gas shock had a peak pressure of about 150 kPa, as shown in Fig. D.20.

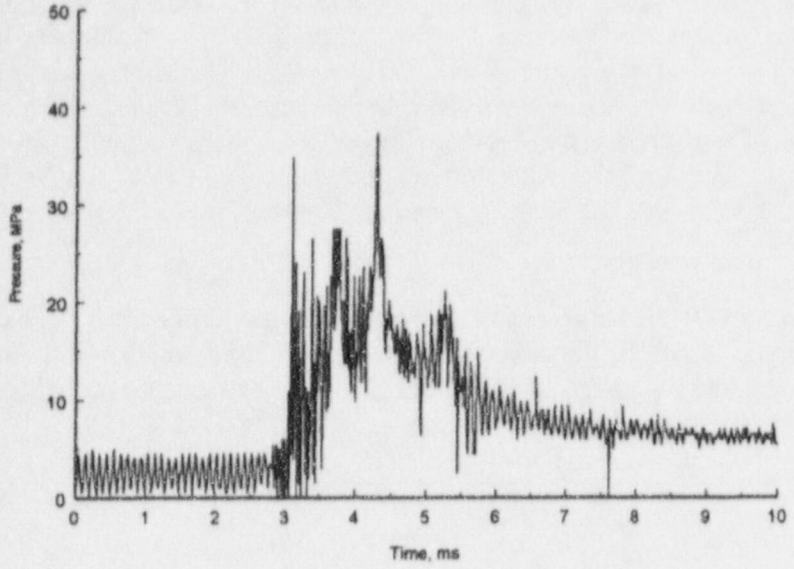


Figure D.18. ZREX-22 P-6 Pressure Record.

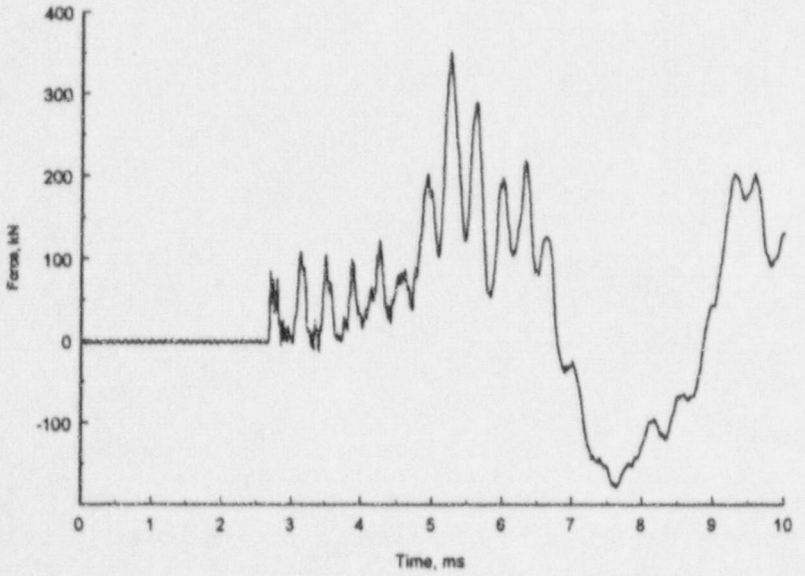


Figure D.19. ZREX-22 Force Record.

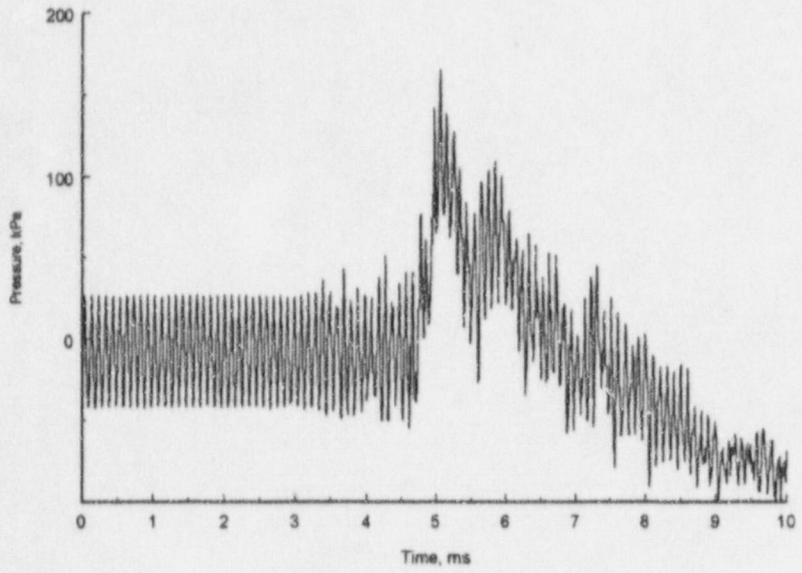


Figure D.20. ZREX-22 Fast Response Containment Pressure.

5 ZREX-23

ZREX-23 was run on May 2, 1996. This experiment was an un-triggered drop of one-kg of zirconium melt into preheated water. Four 500 W band heaters were put on the test section and a control thermocouple was clamped on the external wall of the test section. The procedure used for heating the water was to pre-heat the test section to a level greater than the desired temperature and dump room temperature water into it. A set of four burnwire frames were suspended beneath the crucible. They were placed 5, 29, 54 and 78 cm below the top of the test section.

5.1 Operation

1004 g of zirconium rod was loaded into the crucible. Power was applied to the test section heaters about two hours before the run, heating the test section to 388 K. Heating was started at 98% current level giving 80% power and held there for most of the run. Current as measured by the current sensor showed ~140A. The water pump was started and the test section filled with water at room temperature. Heating proceeded normally (based on the pyrometer readings) with no obvious change seen in the view through the crucible until the temperature reached 2123 K when the zirconium rods began to move. When the temperature reached 2273 K, the dump was initiated (see the pyrometer record shown in Fig. D.21)

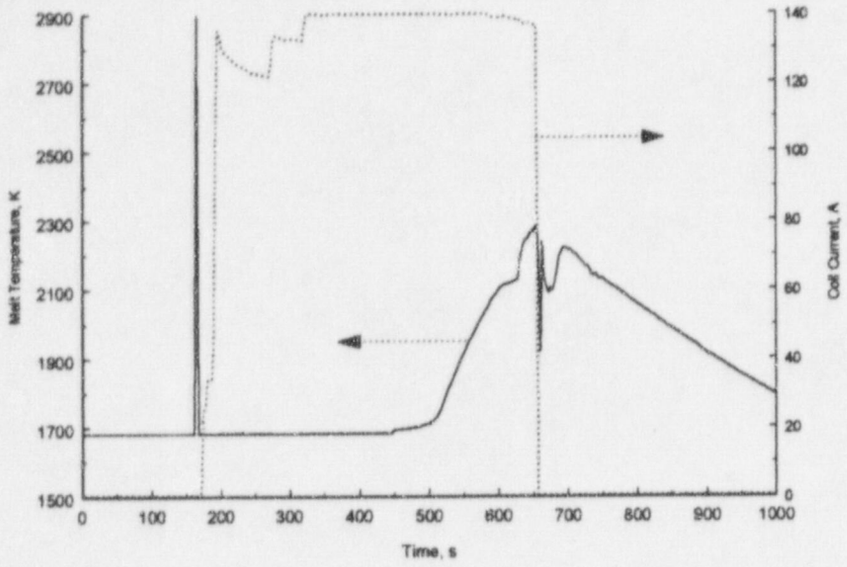


Figure D.21. ZREX-23 Melt Heating Record.

5.2 Observations

Water entered the test section, lowering the test section temperature to about 333 K. It then began warming as heat was transferred from the hot test section wall. The test section thermocouple indicated 343 K when the melt dump occurred, burning out the thermocouple. The top burn wire malfunctioned, giving numerous false signals before the dump. The lower three melted through at intervals of 0.5 and 1.4 s. The temperature recorded by the upper thermocouple in the containment vessel jumped from 328 K to 347 K while the vessel pressure exhibited a spike from 124 kPa to 162 kPa (see Fig. D.22). However, as expected in a non-explosive test, no discernable signals appeared on the fast pressure transducer channels.

Post-test examination of the apparatus showed that no damage had been done. The debris was in the form of a long, thin, ropy, connected mass extending from the second to the fourth burn wire, a distance of 48 cm. This debris shape is quite similar to that observed previously in ZREX-19 and ZREX-21 There was no zirconium left in the crucible. There was a thin white shell adhering to the inside wall of the crucible and in spots on the valve stem. This may be a layer of zirconium that was oxidized (zirconium dioxide) by the high concentration of water vapor in the vessel immediately after the zirconium was dropped. A photograph of the debris is shown in Fig. D.23.

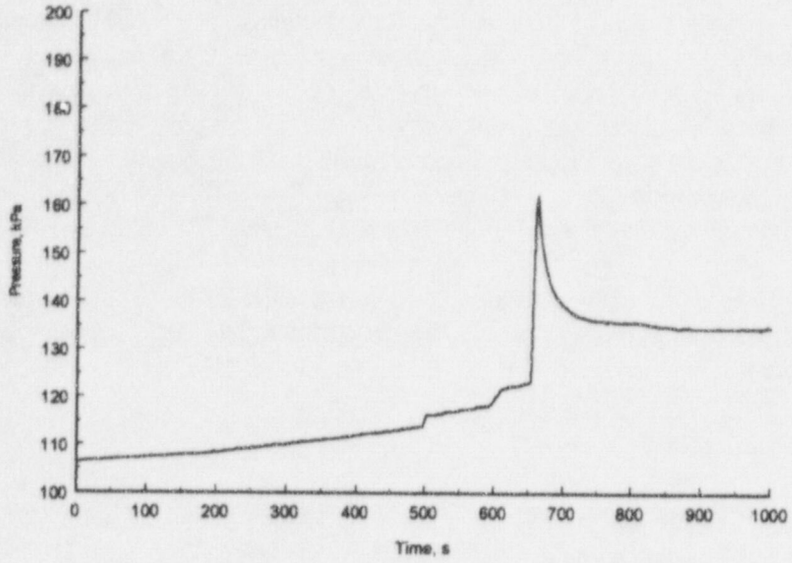


Figure D.22. ZREX-23 Containment Pressure.

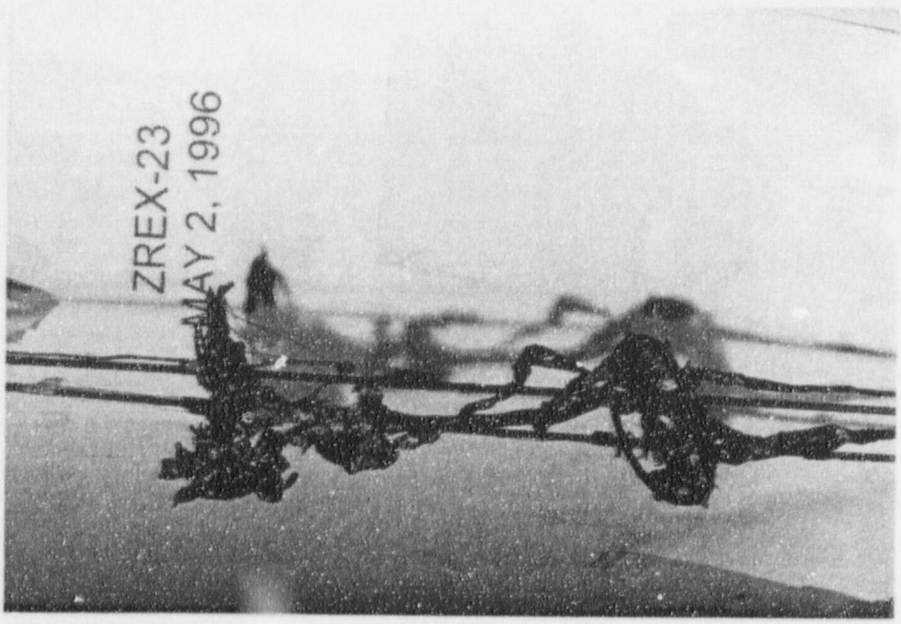


Figure D.23. ZREX-23 Debris.

6 ZREX-24

ZREX-24 was run on May 15, 1996. This experiment was a triggered drop of one kg of zirconium melt into preheated water near saturation. The trigger was a length of Primacord containing 1 g of PETN detonated by the melt melting through a wire grid 61 cm below the top of the test section. A set of two burnwire frames were suspended in the test section at 60.5 and 61.5 cm below the water level, i.e, the top of the test section. The two submerged burnwire grids were attached to a circuit that would fire the trigger if either grid opened. This choice of the burnwire location for the trigger signal was made based on considerations of the melt quenching in the test section as observed in the un-triggered test ZREX-23. It was thought that the quenching distance of the melt for nearly saturated water would at least be 60 cm, as it reached 78 cm in ZREX-23.

A 500-W immersion heater was placed in the water reservoir to preheat the water for this test. The procedure used for heating the water was to pre-heat the test section to a level greater than the desired temperature and then dump the preheated water into it.

6.1 Operation

1003 g of zirconium rod was loaded into the crucible. Power was applied to the test section and water reservoir heaters about two hours before the run, heating the test section to 388 K and the water to 351 K. Heating was started at 95% current level giving 75% power and held there for most of the run. Current as measured by the current sensor showed ~135 A. The water pump was started and the test section filled with preheated water. Heating proceeded with no obvious change seen in the view through the crucible until the temperature reached 2163 K when the zirconium rods began to move. When the temperature reached 2373 K, the dump was initiated (see the pyrometer record shown in Fig. D.24). The water temperature at the time of the melt dump was 367 K, as indicated by the test section thermocouple.

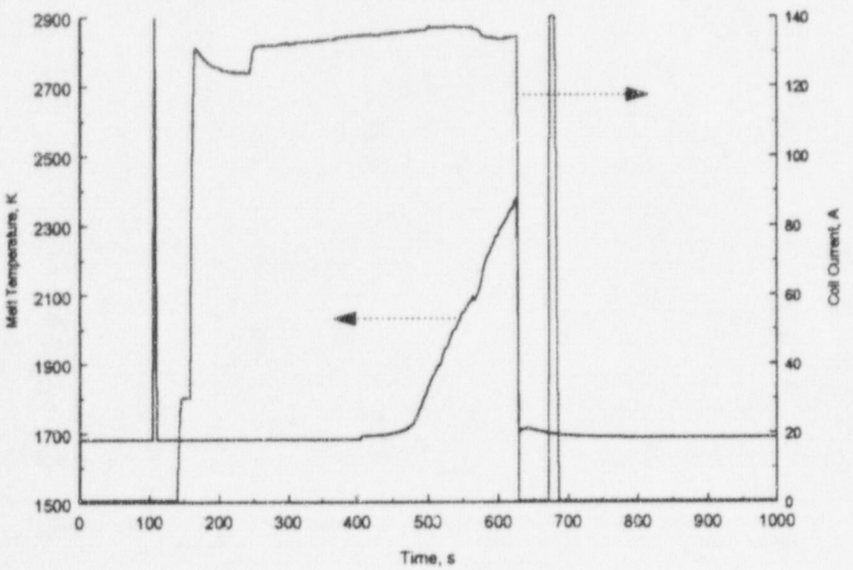


Figure D.24. ZREX-24 Melt Heating Record.

6.2 Observations

After the melt drop, the trigger was initiated and an explosive interaction took place, causing extensive damage to the apparatus. Post-test examination showed that the damage was very similar to that for ZREX-20 previously described, although it appeared to be somewhat more extensive. The debris characteristics also were very similar to those of ZREX-20.

Water entered the test section, lowering the test section temperature to about 363 K. It then began warming as heat was transferred from the hot test section wall. The test section thermocouple indicated 367 K when the melt was dumped. Immediately following the explosion, the temperature in the containment vessel jumped from 313 K to 512 K for the upper thermocouple and from 327 K to 435 K for the lower thermocouple, while the vessel pressure showed a spike from 112 kPa to 672 kPa (see Fig. D.25).

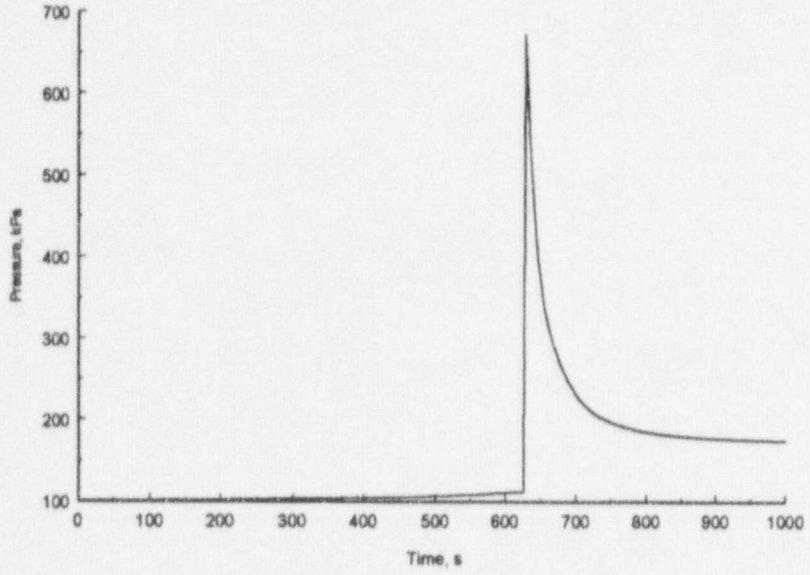


Figure D.25. ZREX-24 Containment Pressure.

Figures D.26, 27, and 28 show, respectively, the pressure traces of three test section transducers located at 30, 46, and 61 cm from the top of the test section (i.e, the water surface level). These traces are somewhat incomplete, since the decay portions of the dynamic pressures are missing, probably due to signal cable failures caused by the explosion. Nevertheless, they reveal important details of the explosion characteristics. The pressure trace at 30 cm from the top of the test section (shown in Fig. D.26) is similar to that for ZREX-20 (shown in Fig. D.10), except that the peak pressure is somewhat higher (about 50 MPa vs. 40 MPa) and that the duration of the pressure is presumably somewhat longer. An examination of the three pressure traces shown in Figs. D.26-28 shows different arrival times of the pressure pulse which indicate that the propagation velocity of the pressure pulse was about 450 m/s. The trace of the reaction force is shown in Fig. D.29. The force peaked at 450 KN, but the transducer failed before it gave the full pulse. (It had a duration of about 2 ms before failure. The duration of the full pulse would have been longer.)

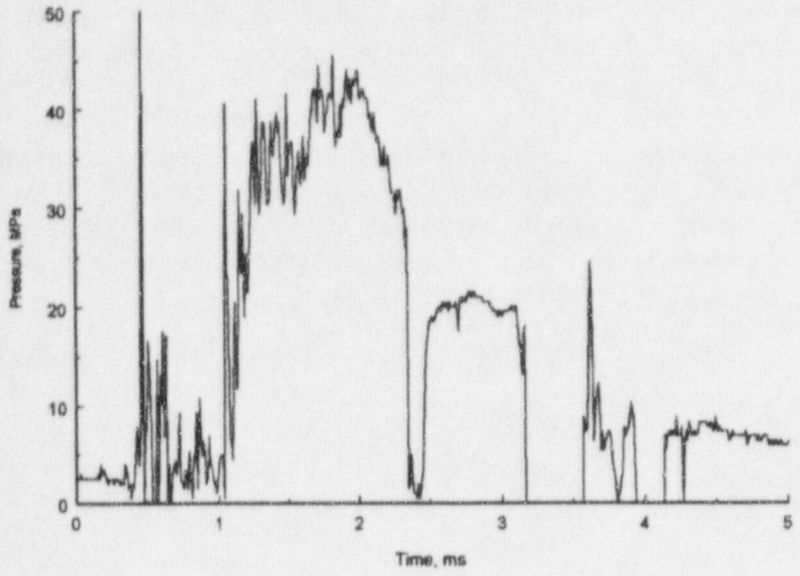


Figure D.26. ZREX-24 P-6 Pressure Record.

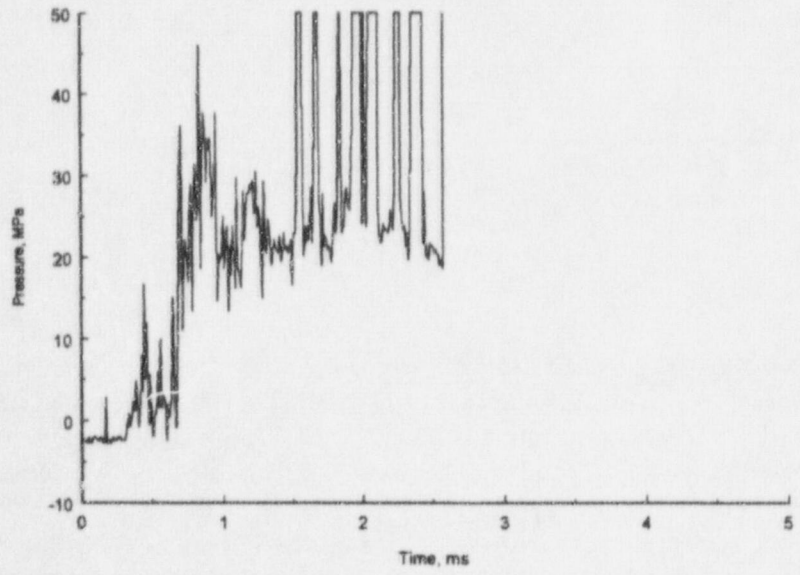


Figure D.27. ZREX-24 P-5 Pressure Record.

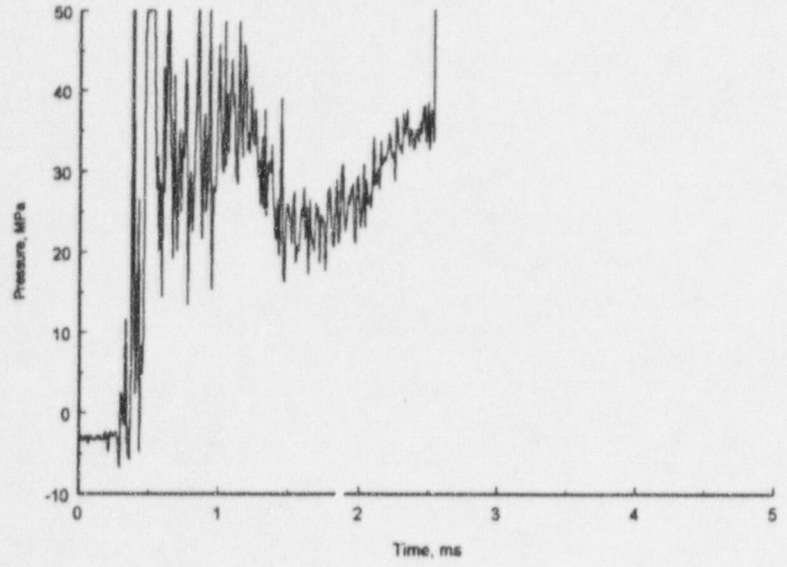


Figure D.28. ZREX-24 P-3 Pressure Record.

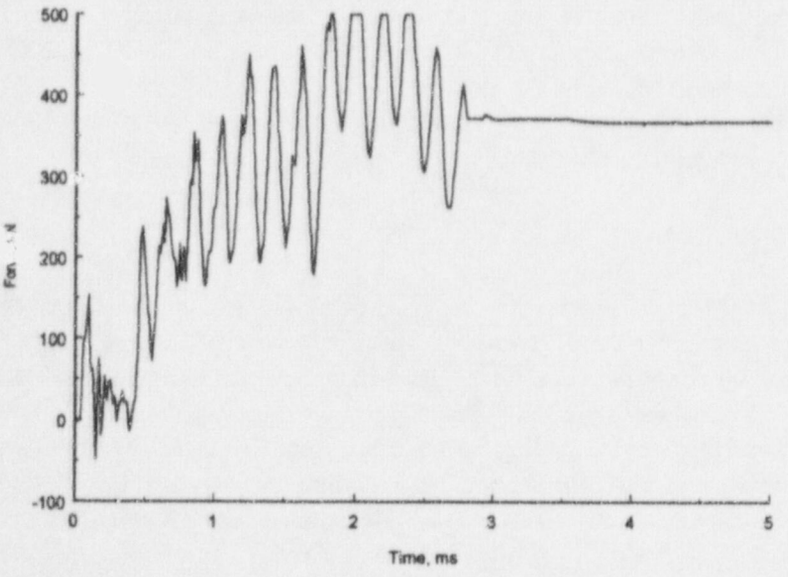


Figure D.29. ZREX-24 Force Record.

Another indication of the explosion event in ZREX-24 was the gas shock pressure recorded by the dynamic pressure transducer mounted on the wall of the containment chamber at the same elevation as the top of the test section. The gas shock had a peak pressure of about 1 MPa, as shown in Fig. D.30.

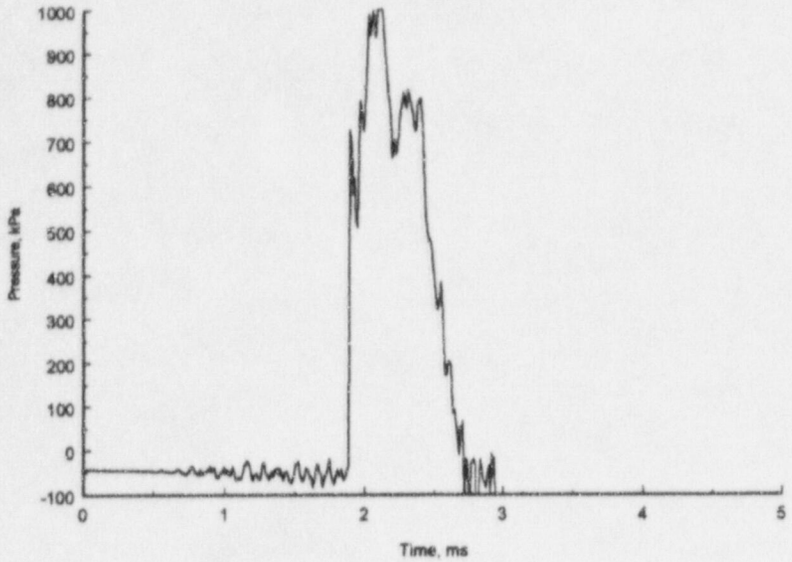


Figure D.30. ZREX-24 Fast ResponseContainment Pressure.

7 ZREX-25a

ZREX-25a was run on July 29, 1996. This experiment was an un-triggered drop of nominally 1-kg melt of 60 w/o Zr-40 w/o ZrO_2 mixture into room-temperature water. It re-used the crucible and load from ZREX-25 which was unsuccessful. The actual mass of the Zr-ZrO_2 mixture was 0.933 kg (some material losses were encountered while pieces were being sawed out of the ingot). A set of four burnwire frames were suspended in the test section beneath the water. The frames were placed at 5.0, 28.5, 52.5, and 76.0 cm deep.

7.1 Operation

Heating was started at 92% current level giving 77% power. The water pump was started and the test section filled with water. Heating proceeded normally with no obvious change seen in the view through the crucible until the temperature reached about 2300 K. At this time the temperature of the water exiting the induction coil was noticed to be above 363 K. If the water in the coil boiled, the coil would fail, ending the experiment. Hence the power to the coil was reduced to lower the cooling water temperature. For about the next 400 seconds, the power was adjusted to continue heating the crucible while keeping the cooling water temperature below boiling. When the crucible temperature reached about 2580 K, the dump mechanism was actuated (see the pyrometer record shown in Fig. D.31. The dump proceeded normally.

7.2 Observations

All of the burn wires opened. The top wire (#1) opened first, followed by the third wire after 0.16 s, then the second 0.06 s later, and finally the bottom wire (#4) 0.27 s after the second. (The opening of the third wire before the second was not expected and is not understood.) The slow instrumentation all responded to the melt drop. The test section thermocouple jumped from 299 to over 373 K (the melt may have hit the thermocouple). Vessel temperature jumped from 358 to 378 K for the upper containment thermocouple and

323 to 329 K for the lower containment, while the containment pressure increased from 112 kPa to 148 kPa (see Fig. D.32). However, no dynamic pressures were measured in the test section or in the containment vessel. The drop view camera showed a brighter drop than usual, the view was completely overexposed during the drop providing no details on dispersion of the falling melt stream.

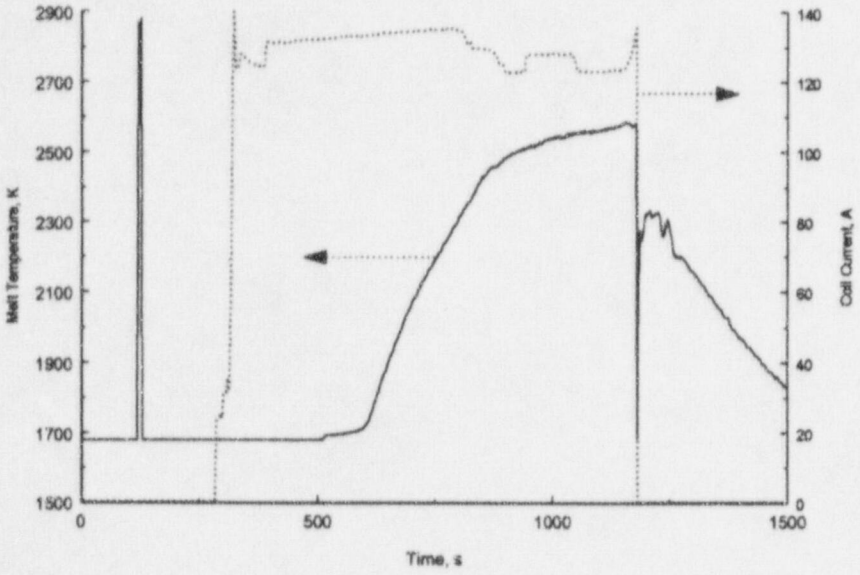


Figure D.31. ZREX-25a Melt Heating Record.

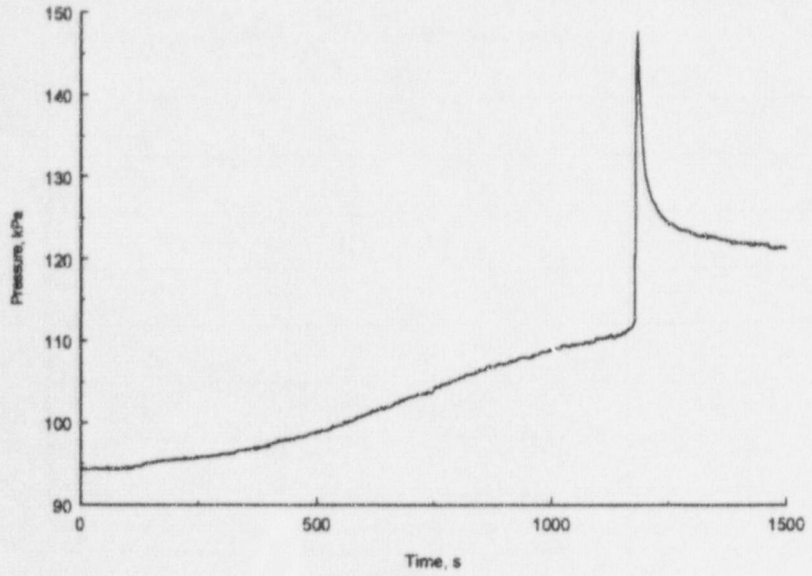


Figure D.32. ZREX-25a Containment Pressure.

Post-test examination of the apparatus showed that complete dumping of the melt had occurred. There was no explosion in this experiment. The water level in the test section was found to be 40 cm below the top after the test. Large globular melt fragments were found on the burn wire arrays (275 g on #3 and 135 g on #4) and in the collector (176 g). An additional 280 g was found on the vessel floor. A small amount , 15 g, was on top of the test section. A photograph of the ZREX-25a debris is shown in Fig. D.33. A sieve analysis of the debris was conducted. The results are summarized in TableD.1.

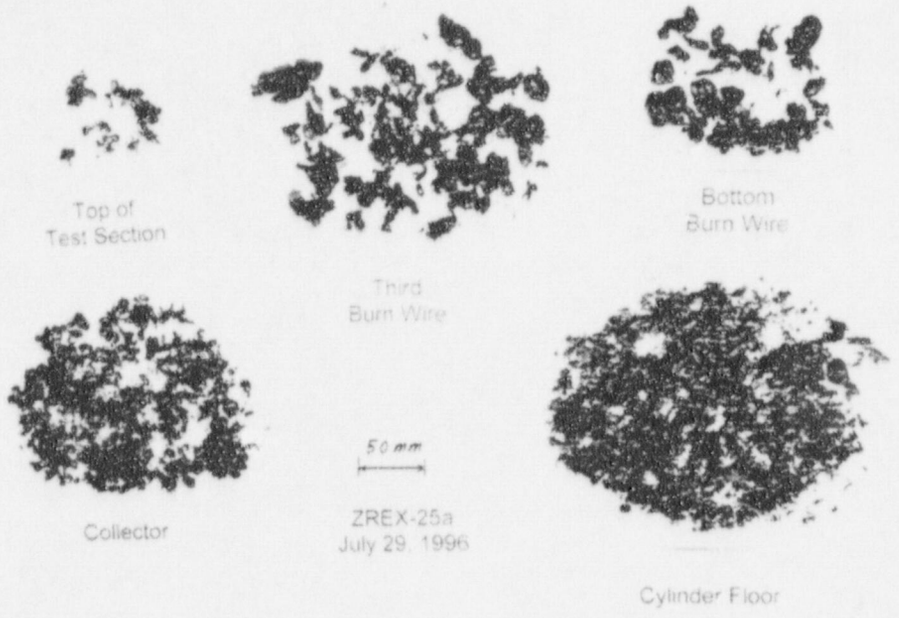


Figure D.33. ZREX-25a Debris.

Table D.1 Sieve analysis of ZREX-25a debris

Size Range (mm)	Amount (%)
>8.00	41.0
8.00 - 5.60	18.7
5.60 - 4.00	14.8
4.00 - 2.80	10.3
2.80 - 2.00	7.4
2.00 - 1.40	3.7
1.40 - 1.00	2.1
1.00 - 0.71	1.1
0.71 - 0.50	0.5
0.50 - 0.355	0.3
<0.355	0.2

8 ZREX-27

ZREX-27 was run on October 10, 1996. This experiment was an un-triggered drop of one-kg melt of 10 w/o ZrO_2/90 w/o Zr mixture into water near saturation. The material was charged into a standard dropping crucible as mixture of ZrO_2 and Zr powders contained in four polyethylene bags. The crucible was heated above the liquidus temperature of the mixture and held for a length of time. The temperature was then raised above the highest holding temperature and the drop mechanism actuated. The water was nearly saturated. A set of three burnwire frames suspended in the test section beneath the water. The frames were placed at 5.0, 41.0, and 77.0 cm deep.

8.1 Operation

A dropping crucible was loaded with 900 g of Zr powder and 100 g of ZrO_2 powder in four polyethylene bags, wrapped with new insulation and placed in the apparatus. Heating was started at 90 A (Ampere) (62% current level) giving 30% power. This gave a very slow heating rate so the current was increased to 120 A after about 100 s. When the crucible temperature reached 2073 K, the current was reduced to 95 A followed by several current adjustments until the temperature reached 2373 K. Minor adjustments in the current level from 110 to 115 A were made to hold the temperature around 2373 K for the next fifteen minutes. At the end of the fifteen-minute holding time, the water from the reservoir (preheated to 365 K) was pumped into the test section (preheated to ~373 K), and the power to the crucible was increased to 130 A. When the crucible temperature reached 2473 K, the dropping mechanism was actuated (see the pyrometer record shown in Fig. D.34). The video cameras showed that the crucible valve opened and melt fell into the test section. There was no explosion.

8.2 Observations

The three burnwires opened. The second opened 0.3 s after the first and the third 0.47 s later. This gives a drop velocity of 1.2 m/s between the top two wires and the 0.75 m/s between the bottom two. The top vessel temperature jumped from 383 K to 398 K, while the bottom jumped from 338 K to 371 K. The vessel pressure changed from 110 kPa to 205 kPa (see Fig. D.35).

Post-test examination of the apparatus showed that complete melting of the ingot had occurred and all had fallen out of the crucible. Approximately one-half of the water was gone from the test section. Molten melt (181 g) had impacted on the bottom of the crucible support plate (aluminum), sticking to it and partially melting it. A small amount (79 g) of melt had splashed onto the plate covering the top of the test section. A major part of the melt (338 g) had fallen through the water to the catch cup at the bottom of the test section. Some had frozen as spherical particles while some was still molten and melted through the bottom of the cup. An additional 372 g of debris was collected from the containment vessel floor. Some 5 g of powder remained in the crucible leaving about 25 g that was welded to various parts of the apparatus. A photograph of the debris is shown in Fig. D.36. Table D.2 summarizes the results of a sieve analysis of the debris.

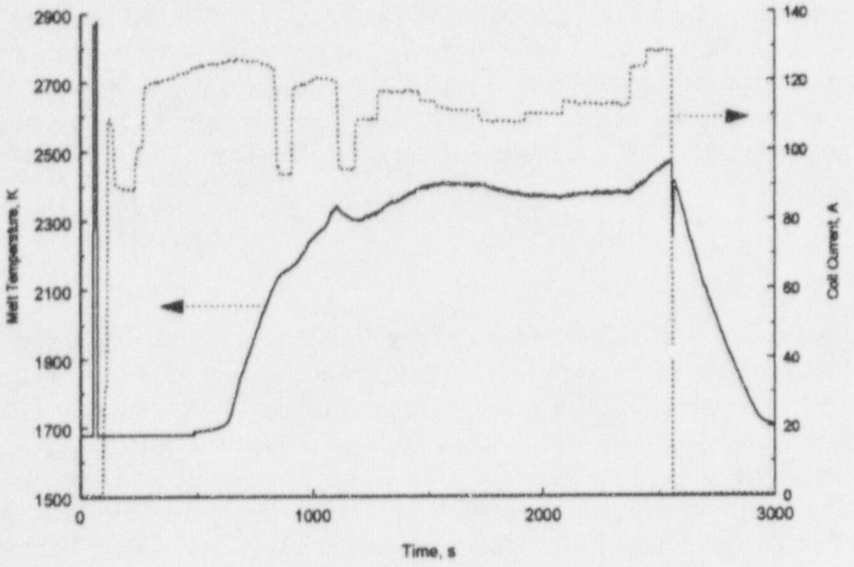


Figure D.34. ZREX-27 Melt Heating Record.

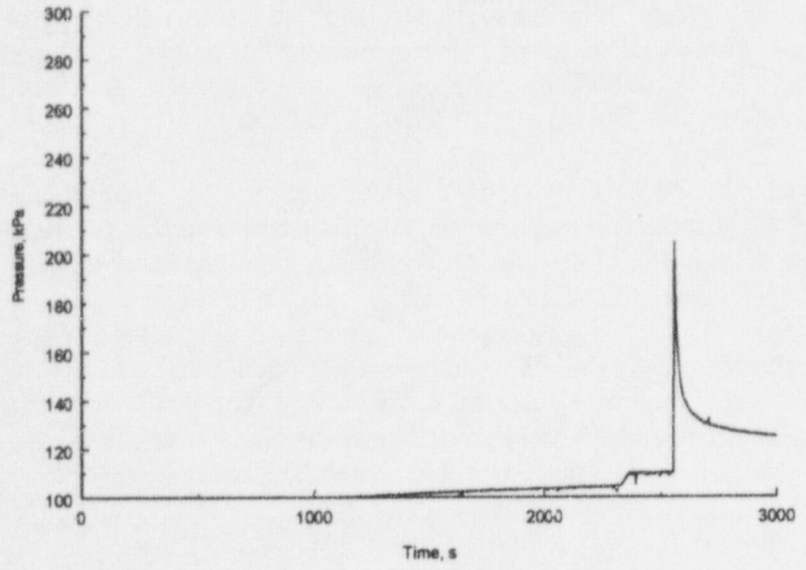


Figure D.35. ZREX-27 Containment Pressure.

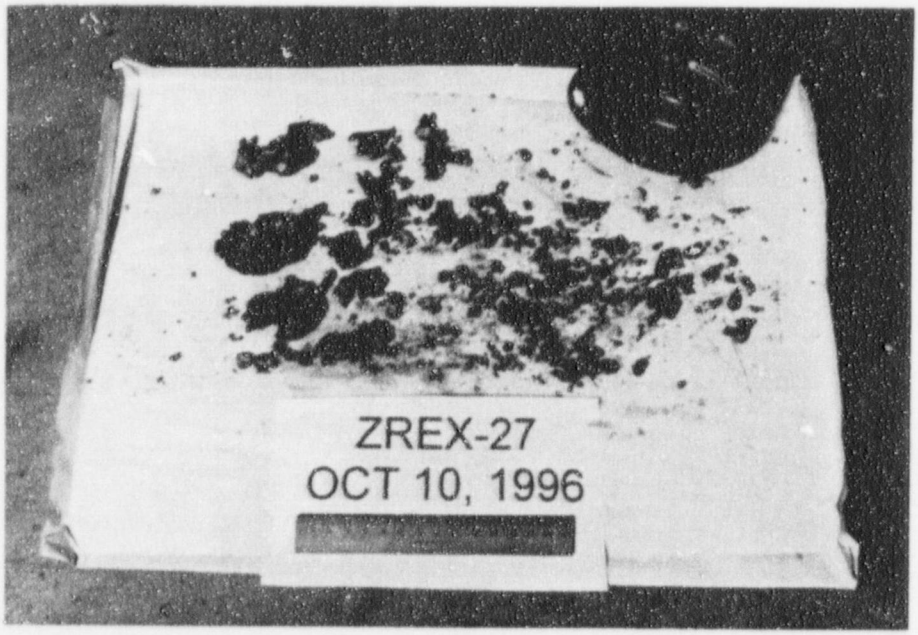


Figure D.36. ZREX-27 Debris.

Table D.2 Sieve analysis for ZREX-27 debris

Size Range (mm)	Amount (%)
>8.00	23.1
8.00 - 5.60	17.3
5.60 - 4.00	13.7
4.00 - 2.80	12.0
2.80 - 2.00	10.3
2.00 - 1.40	8.4
1.40 - 1.00	5.6
1.00 - 0.71	4.2
0.71 - 0.50	2.2
0.50 - 0.355	1.4
<0.355	1.7

9 ZREX-28

ZREX-28 was run on October 17, 1996. This experiment was a triggered drop of one-kg melt of 10 w/o ZrO_2/90 w/o Zr mixture into preheated water near saturation. A set of three burnwire frames was suspended in the test section beneath the water. The frames were placed at 5, 60 and 61 cm deep. The lower two burnwire grids (i.e, 60 and 61 cm deep) were attached to a circuit that would fire the trigger if either grid opened.

9.1 Operation

A dropping crucible was loaded with 900 g of Zr powder and 100 g of ZrO_2 powder in four polyethylene bags, wrapped with new insulation and placed in the apparatus. Heating was started at 135 A (95% current level) giving 69% power. When the crucible temperature reached 2373 K, the current was reduced to 113 A followed by several current adjustments to 106 A when the temperature stabilized at 2403 K. The temperature was held at around 2403 K for the next fifteen minutes. At the end of the fifteen-minute holding time, the water from the reservoir (367 K) was pumped into the test section (374 K), and the power to the crucible was increased to 130 A. When the crucible temperature reached 2470 K (see the pyrometer record shown in Fig. D.37), the dropping mechanism was actuated (at this time the water temperature in the test section was 363 K). Initially, the video cameras showed no melt drop. After about 5 s, the valve opened and a thin stream of melt fell into the test section, resulting in an explosion. Bubbles in the coil cooling lines indicated that the coil had been ruptured by the explosion, so the coil cooling solenoid valves were closed.

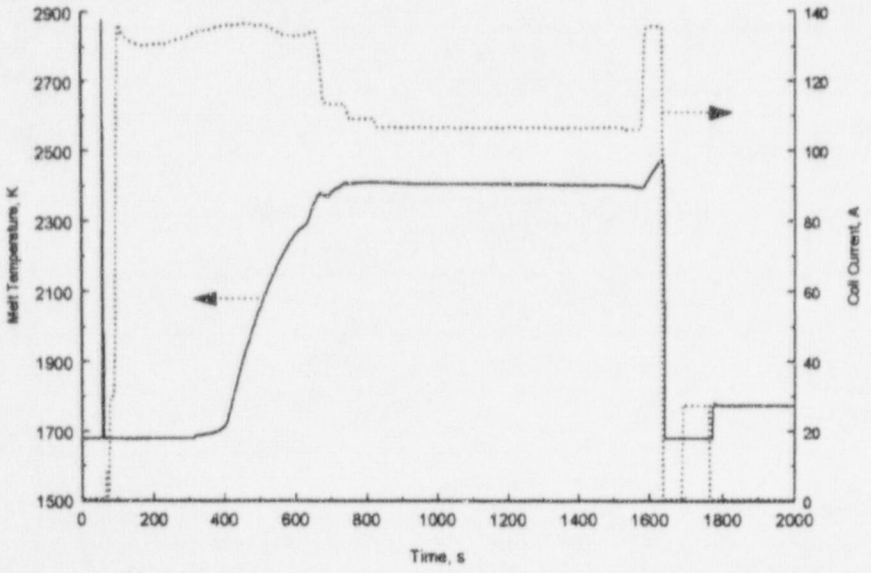


Figure D.37. ZREX-28 Melt Heating Record.

9.2 Observations

The three burnwires opened. However, the top wire had many circuit openings and closings giving ambiguous timing data. The top vessel temperature jumped from 398 K to 408 K, while the bottom jumped from 303 K to 455 K (probably due to hot debris hitting the thermocouple). The vessel pressure showed a

spike from 110 kPa to 430 kPa (see Fig. D.38).

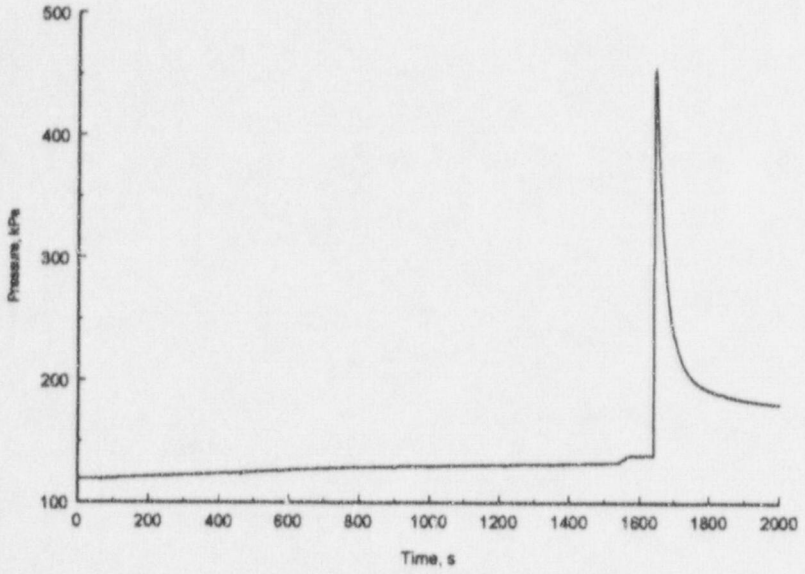


Figure D.38. ZREX-28 Containment Pressure.

Post-test examination of the apparatus showed that much damage had been done. The damage done to the internals of the containment chamber was similar to those observed in previous triggered experiments. In addition, it was found that the bottom crucible support plate was bowed, concave down, leading to the conjecture that an explosion had occurred above the plate, probably in the crucible when the water slug expelled by the explosion in the test section was driven into the crucible.

All but one of the fast transducers produced reasonable signals. The bottom transducer, p-2, 76 cm deep, showed erratic swings from the positive to negative limits of the record. The deepest operating transducer, p-3 at 61 cm deep (i.e., the level of the burnwire triggering the explosion), showed a peak pressure of about 20 MPa with a duration of about 4 ms. The next deepest, p-5 at 45 cm deep, showed a slightly smaller peak of about 15 MPa. The two shallowest, p-6 and p-7 both at 30 cm deep, both showed peaks of about 20 MPa. These pressure traces are shown in Figs. D.39-42. The reaction force signal had a peak of 300 kN and a duration of about 4 ms (see Fig. D.43). The gas shock pressure at the wall of the containment chamber peaked at 230 kPa (see Fig. D.44).

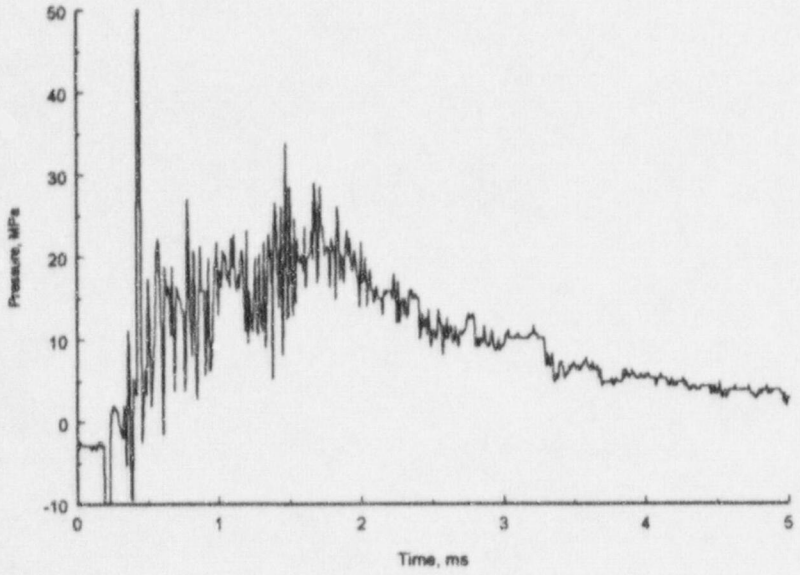


Figure D.39. ZREX-28 P-3 Pressure Record.

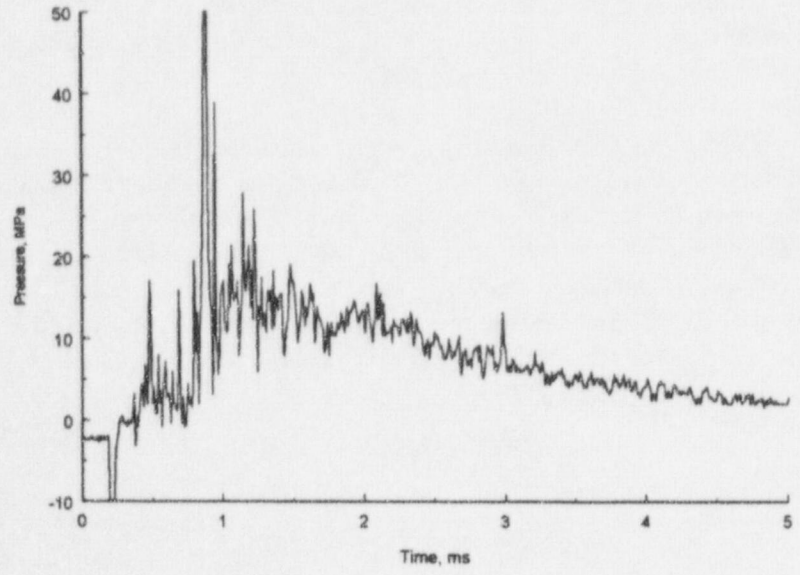


Figure D.40. ZREX-28 P-5 Pressure Record.

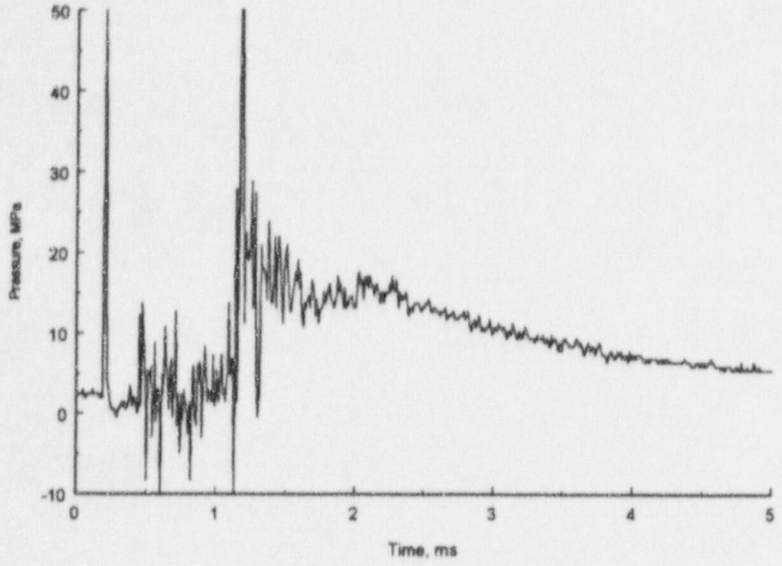


Figure D.41. ZREX-28 P-6 Pressure Record.

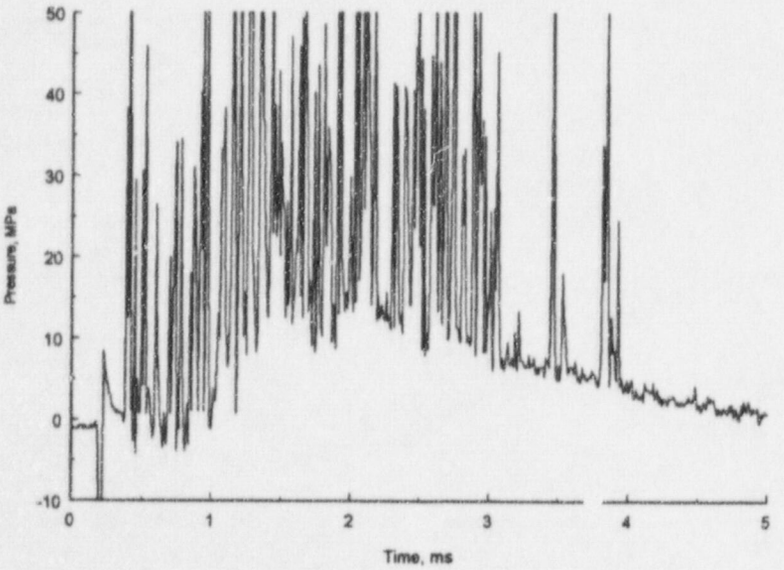


Figure D.42. ZREX-28 P-7 Pressure Record.

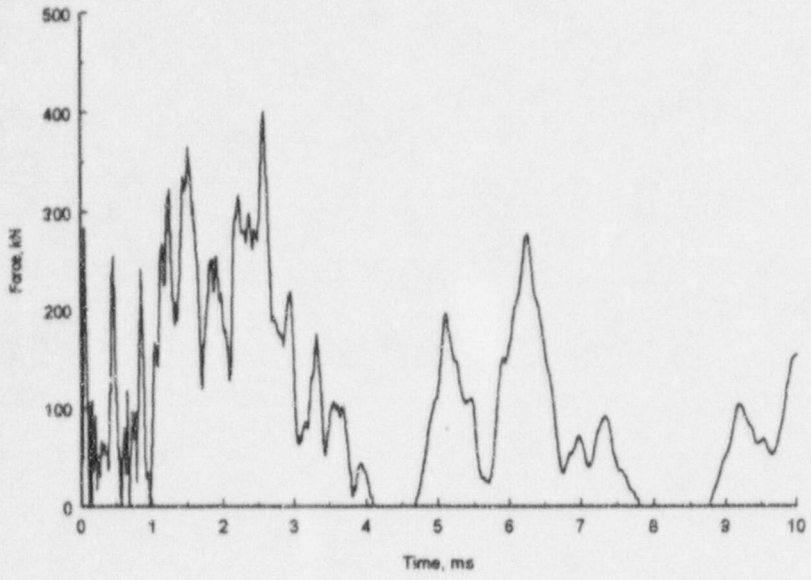


Figure D.43. ZREX-28 Force Record.

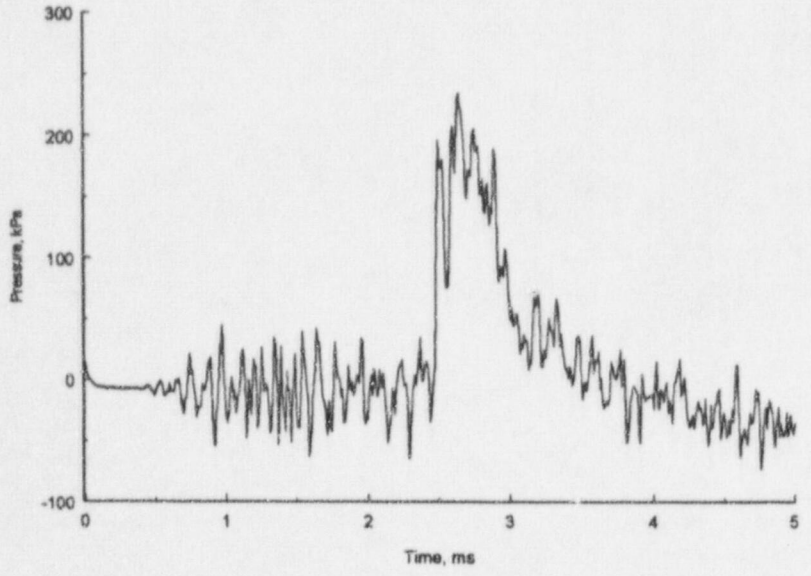


Figure D.44. ZREX-28 Fast Response Containment Pressure.

10 ZREX-34

ZREX-34 was run on January 3, 1997. This experiment was an attempt to drop about one kg of 40 w/o ZrO_2/Zr into room temperature water and trigger an explosion. The material was broken and sawed pieces of the ingot from ZREX-33. A set of three burn-wire frames suspended in the test section beneath the water. The frames were placed at 5.0, 30.0, and 31.0 cm deep. Either of the bottom two burn-wires opening would trigger the detonator.

10.1 Operation

An attempt was made to prevent the crucible parts from sticking together when the melt contained zirconia by coating the valve parts with yttria. As yttria is reduced by graphite at the operating temperature, a layer of TiC was applied to reduce the diffusion of carbon to the yttria. A standard dropping crucible with cylindrical valve seat was prepared by washing both the valve plug and seat with two applications of a water-based suspension of TiC with air drying after each coat. This was followed by a spray application of a yttrium oxide aerosol refractory coating. The crucible parts were then dried in an oven for two hours at 533 K. The crucible was loaded with 834 g of the ingot from ZREX-33 (40 w/o ZrO_2, 10.4 w/o O, 39.8 a/o O) wrapped with insulation and placed in the apparatus

The containment vessel was evacuated and backfilled with argon. Heating was started at 125 A (90% current level) giving 67% power. The water pump was started as soon as the crucible had heated to a temperature where the self-luminosity was enough to allow a final check of the pyrometer alignment. The reservoir temperature was 291 K and the test section temperature 293 K. When the crucible temperature reached 2573 K (see the pyrometer record shown in Fig. D.45), the dropping mechanism was actuated. The video cameras showed that the crucible valve did not open. After about 4 s, however, the valve opened and the melt fell into the test section.

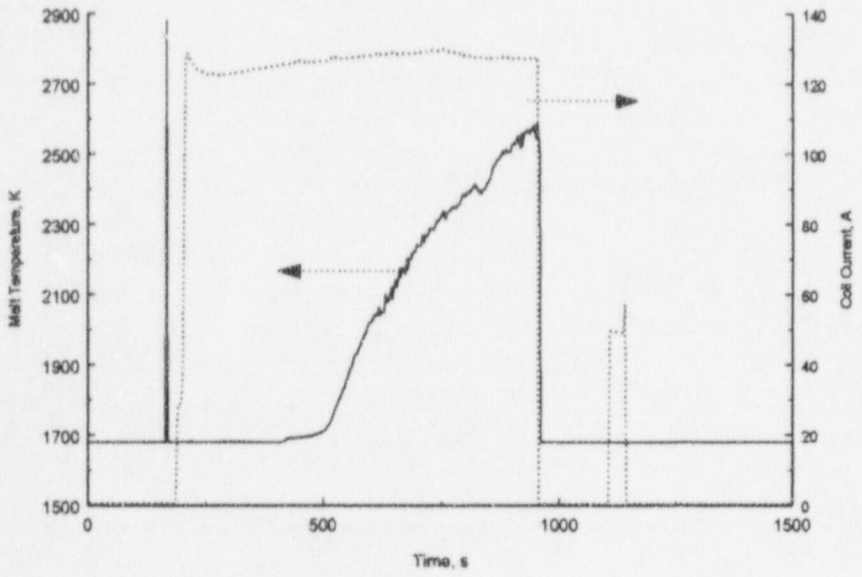


Figure D.45. ZREX-34 Melt Heating Record.

10.2 Observations

All of the burn wires opened. The one at 31 cm opened 220 ms after the top wire followed by the one at 30 cm 50 ms later. The top vessel temperature reached 382 K while the bottom reached 494 K. The vessel pressure jumped from 122 kPa to 409 kPa (see Fig D.46). Post test examination of the apparatus showed that much damage had been done, typical of the previous triggered runs.

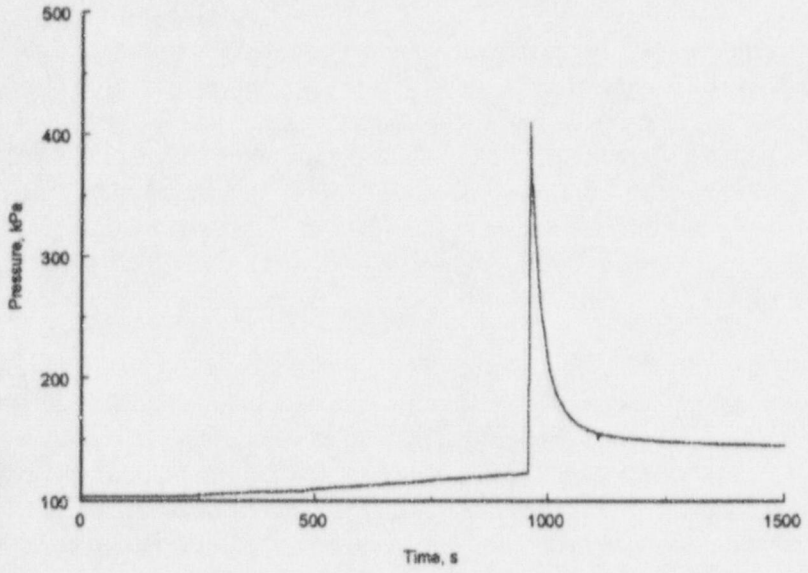


Figure D.46. ZREX-34 Containment Pressure.

The wire break signal was complex with several makes and breaks over a period of 14 ms. It appears that the two data capture PCS started at different times, about when the trigger voltage reached 1.0 V. One PC, recording force link, breakwire, vessel pressure, and one test section pressure transducer, started 0.33 ms before the other PC, recording the remaining test section pressure transducers.

All but one of the fast transducers produced reasonable signals. A low transducer, p-5 at 46 cm below the surface, showed erratic swings from the positive to negative limits of the record, a typical symptom of a damaged cable. Another transducer, p-3 at 61 cm below the surface, showed a blocky, digitized appearing signal, although the pulse width and peak pressure agreed well with the other transducers. The two transducers at 30 cm deep, p-6 and p-7, showed similar peaks of about 60 MPa (see Fig. D.47 and D.48). The deepest, p-2 at 76 cm deep, showed a pulse with shorter duration and pressure peaking at 100 MPa (see Fig. D.49). Forces measured by the force transducer over-ranged at 500 kN (see Fig. D.50). The chamber pressure at the wall of the containment peaked at 100 kPa (see Fig D.51).

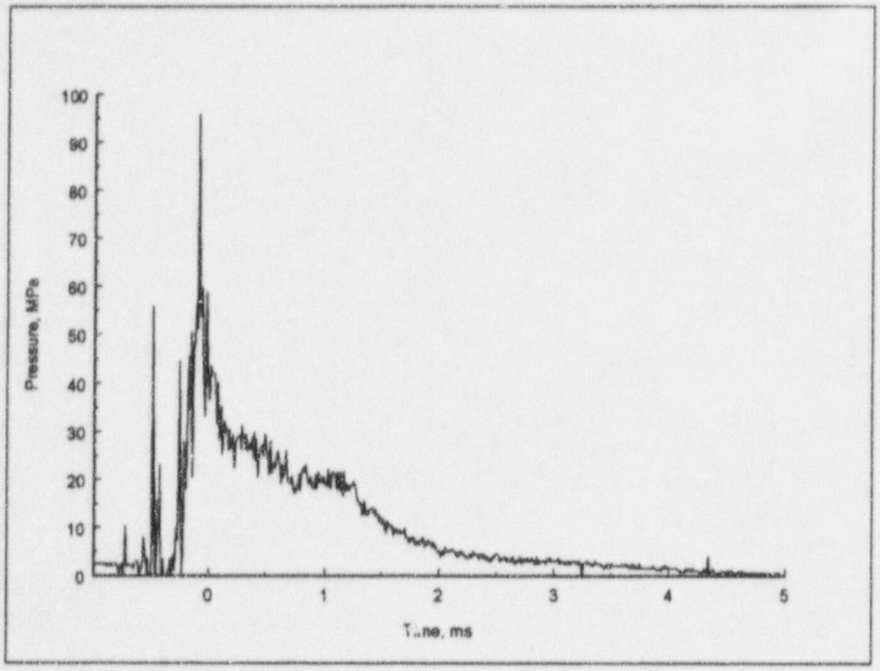


Figure D.47. ZREX-34 P-6 Pressure Record.

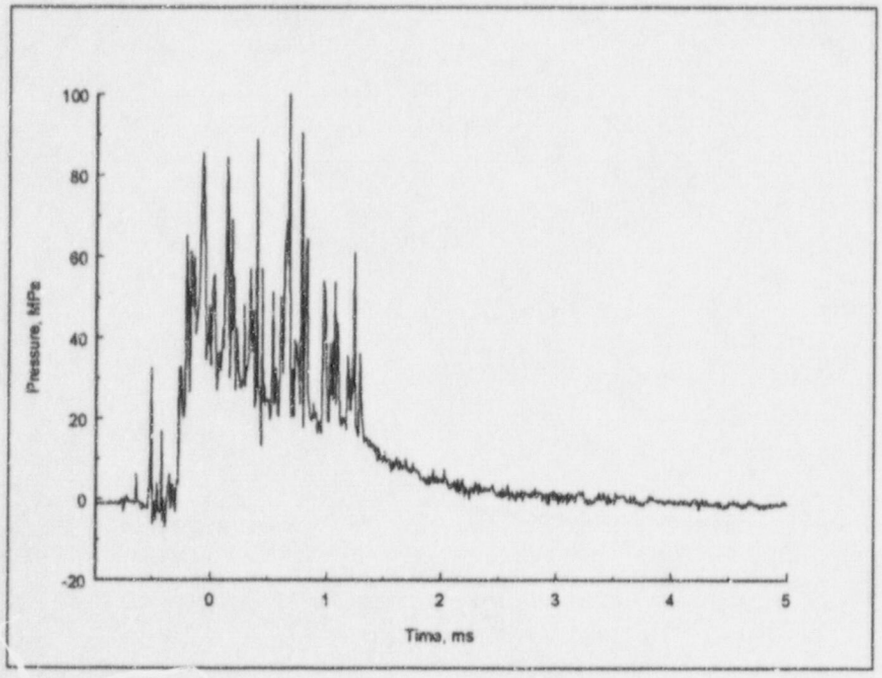


Figure D.48 ZREX-34 P-7 Pressure Record.

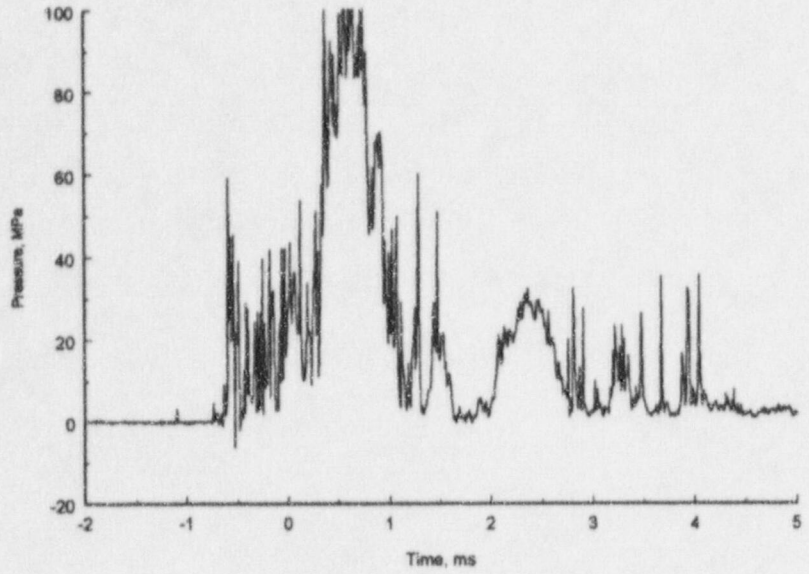


Figure D.49. ZREX-34 P-2 Pressure Record.

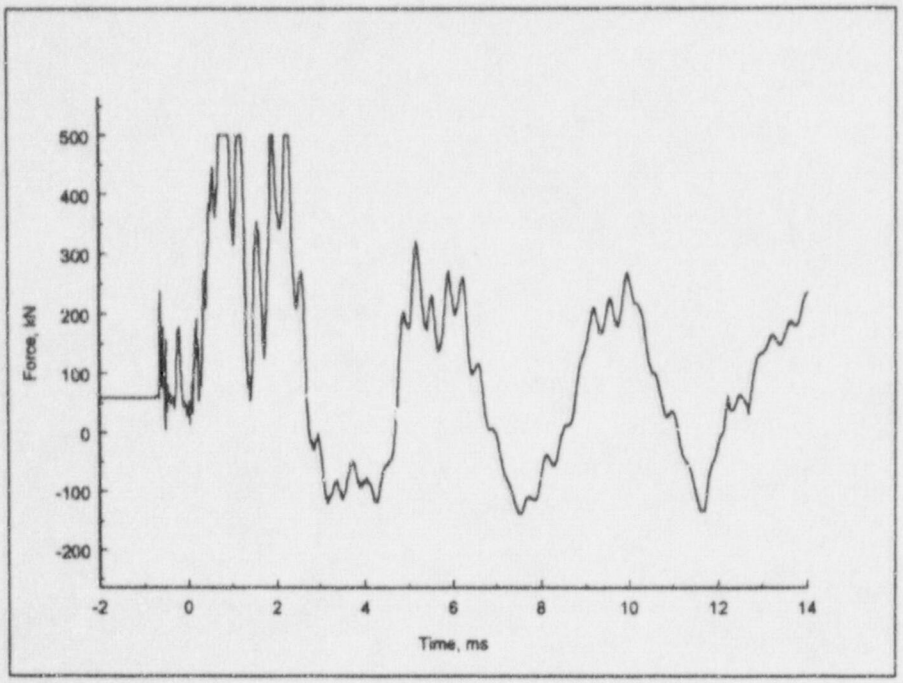


Figure D.50. ZREX-34 Force Record.

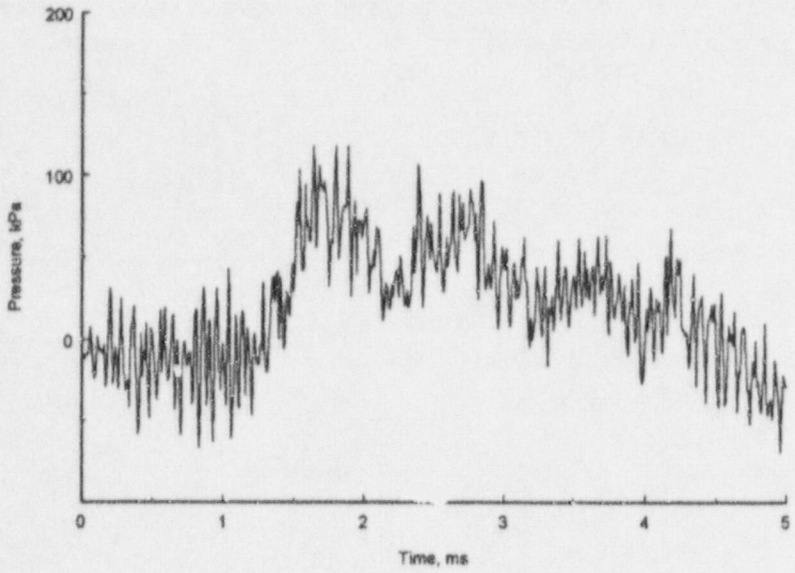


Figure D.51. ZREX-34 Fast Response Containment Pressure.

11 ZREX-36

ZREX-36 was run on February 4, 1997. This experiment was an attempt to drop about one kg of 20 w/o ZrO_2/Zr into room-temperature water and trigger an explosion. The crucible and load were that used in ZREX-35. The load was broken and sawed pieces of the ingots from ZREX-29a and ZREX-31. The valve parts had been coated with a wash of TiC followed by Y_2O_3 as in ZREX-33. With no delay, the valve opened, dropping the melt into the water where a triggered explosion occurred.

11.1 Operation

The crucible and load from ZREX-35 were used for this experiment. As the failure of ZREX-35 involved only a broken actuator cable, this was replaced. The setup for ZREX-35 was somewhat different from normal. Two circles, 33 mm in diameter were formed from 1.3 mm Zr wire and placed around the valve plug (see Fig. C.12). Six g of Zr powder was then placed around the Zr wire. The crucible load was made up of 974 g of ingot fragments from ZREX-29a and ZREX-31 (20 w.o ZrO_2, 5.2 w/o O, 19.9 a/o O) that had been mounted in epoxy for metallographic sectioning. The epoxy was removed by heating the fragments in air at 670 K for two hours.

One modification was made to the dropping apparatus for this experiment. An insulating plug made of zirconia board was placed in the hole in the insulation supporting the crucible. This piece was a loose fit held in place by a swinging flap that was pulled aside by the dump cylinder. When the dump mechanism was actuated, the flap moved allowing the zirconia plug to fall out of the hole into the test section and at the same time the crucible valve was opened. The plug probably fell onto the top burn wire and was retained there until the molten Zr mixture melted through the wire grid.

The containment vessel was evacuated and backfilled with argon. Heating was started at 130 A (94% current

level) giving 71% power. The water pump was started as soon as the crucible had heated to a temperature where the self-luminosity was enough to allow a final check of the pyrometer alignment. The reservoir temperature was 290 K and the test section temperature 291 K. When the crucible temperature reached 2620 K (see the pyrometer record shown in Fig. D.52), the dropping mechanism was actuated. The valve opened without delay and the melt fell into the test section. The coil cooling solenoid valves were closed.

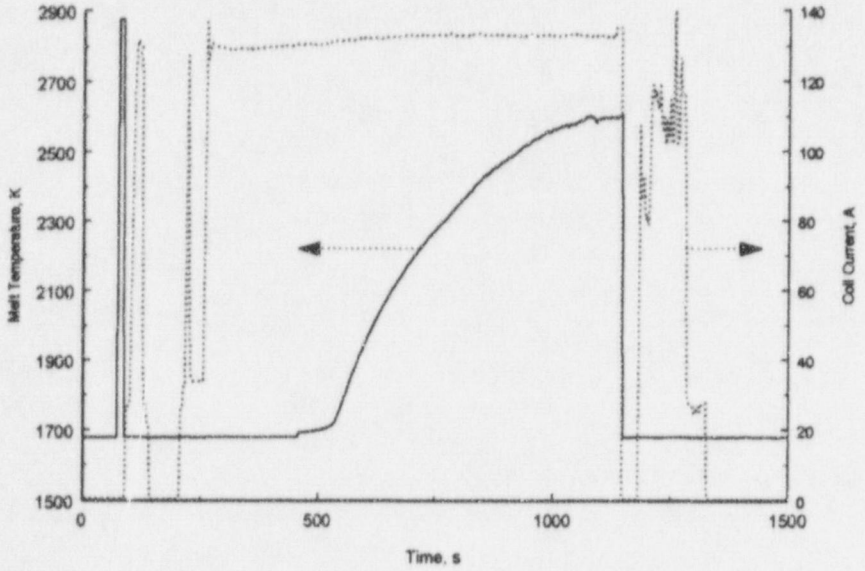


Figure D.52. ZREXX-36 Melt Heating Record.

11.2 Observations

All of the burn wires opened, the top one (at 5 cm) opened 50 ms after the two middle ones (at 30 and 31 cm). The top vessel temperature reached 406 K while the bottom reached 398 K. The containment vessel pressure jumped from 120 kPa to 450 kPa (see Fig. D.53).

The wire break signal was complex with several peaks and valleys over a period of 4 ms. It appears that the two data capture PCS started at about the same time.

Two transducers produced questionable signals. A low transducer, p-5 at 46 cm below the surface, showed erratic swings from the positive to negative limits of the record, a typical symptom of a damaged cable. This transducer had exhibited the same behavior during ZREX-34. The cable was replaced after ZREX-34 and a blank squib run was made (ZREX-35tc) which showed a good signal. Another transducer, p-3 at 61 cm below the surface, showed a reasonable signal superimposed on a signal drifting negative over about a 4 ms period. The two transducers at 30 cm deep, p-6 and p-7, showed similar records (see Figs. D.54 and D.55). They had initial spikes greater than 50 MPa, probably from the trigger explosion, followed in about 0.6 ms by the explosion signal. This signal had a peak pressure of about 40 MPa and a duration of about 3 ms. The deepest, p-2 at 76 cm deep, showed a pulse with similar duration and pressure peaking at 50 MPa (see Fig. D.56). Forces measured by the force transducer over-ranged at 500 kN (see Fig. D.57). The gas shock pressure at the wall of the containment peaked at 830 kPa (see Fig. D.58).

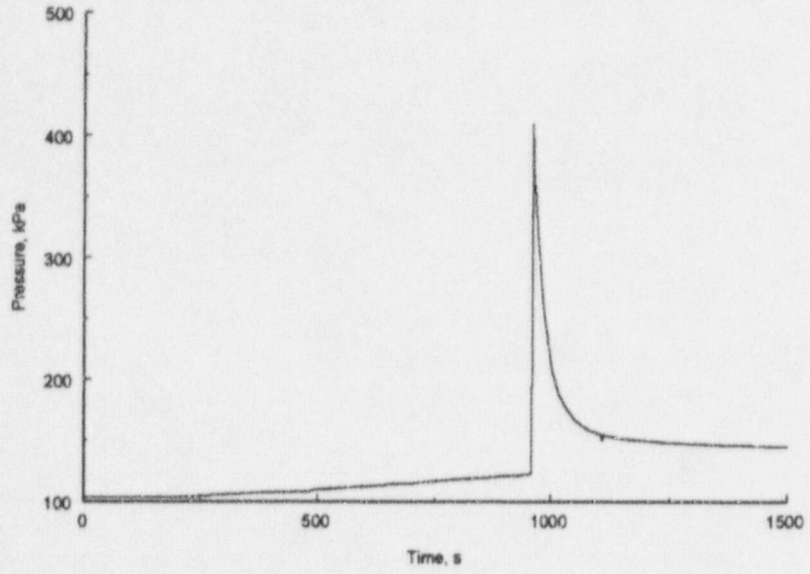


Figure D.53. ZREX-36 Containment Pressure.

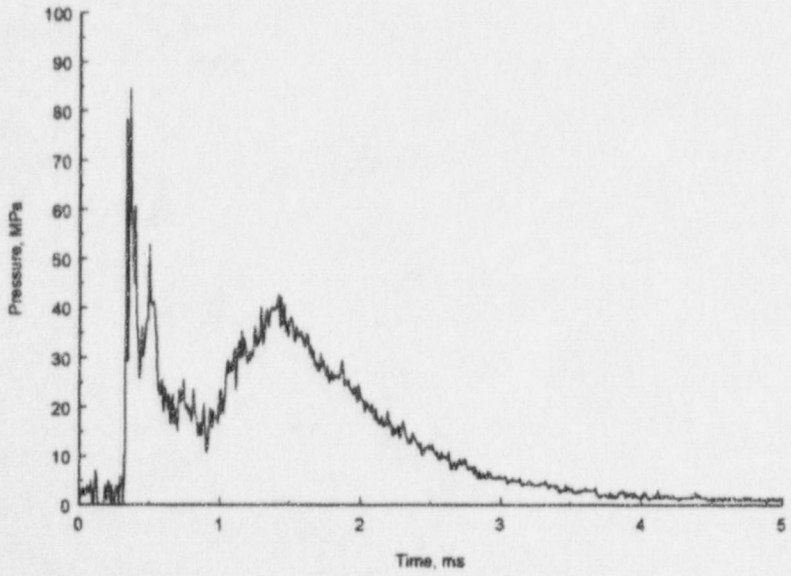


Figure D.54. ZREX-36 P-6 Pressure Record.

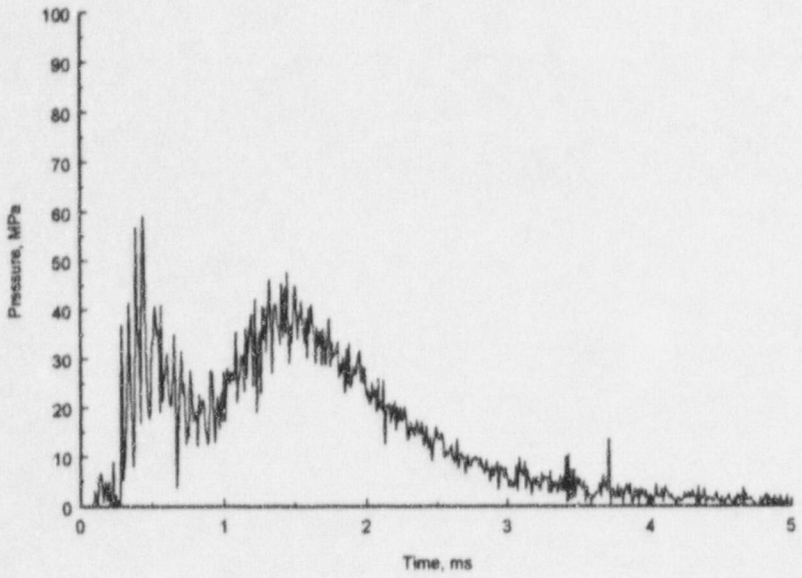


Figure D.55. ZREX-36 P-7 Pressure Record.

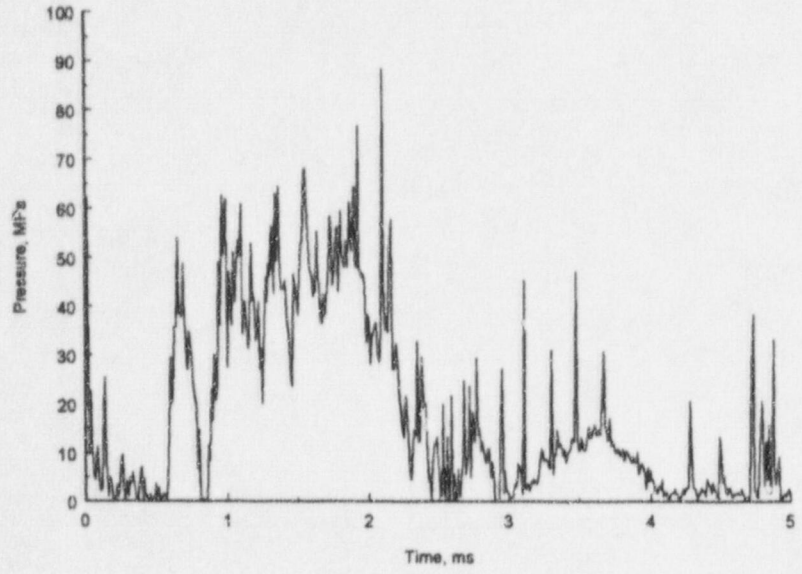


Figure D.56. ZREX-36 P-2 Pressure Record.

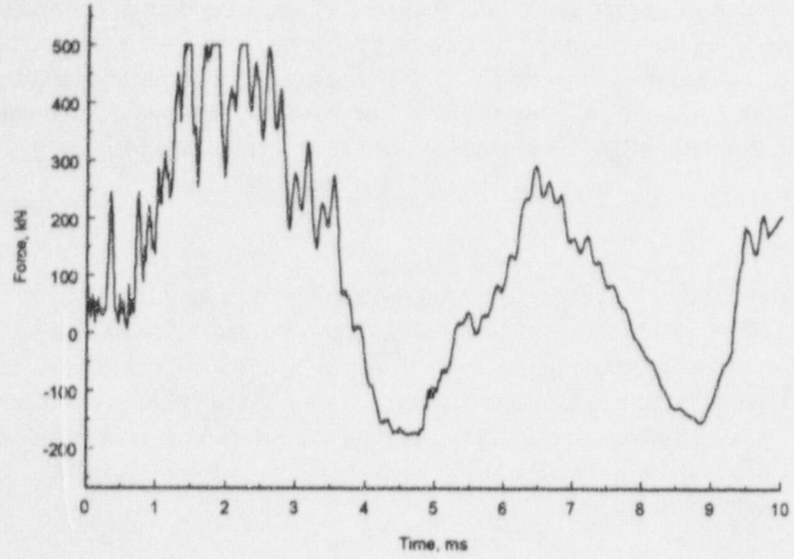


Figure D.57. ZREX-36 Force Record.

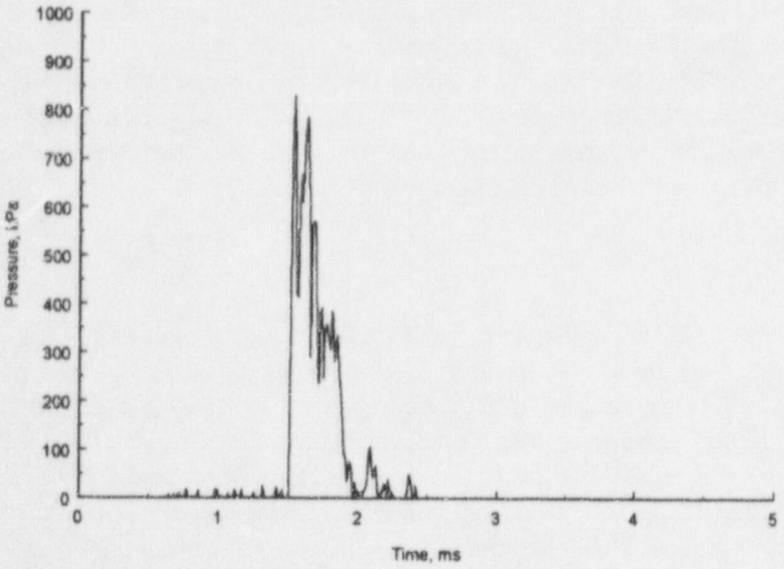


Figure D.58. ZREX-36 Fast Response Containment Pressure.

12 ZREX-41

ZREX-41 was run on March 21, 1997. This experiment was an attempt to drop about one kg of 10 w/o ZrO_2/Zr into room temperature water and trigger an explosion. The material was charged into a dropping crucible as mixture of ZrO_2 and Zr powders contained in four polyethylene bags. Two layers of zirconia felt covered the hole through the bottom insulation. The crucible was heated above the liquidus temperature of the mixture and held for a length of time. The temperature was then raised to a 150 K superheat and the drop mechanism actuated. Melt fell from the crucible into the test section and an explosion was triggered.

12.1 Operation

A standard dropping crucible with a cylindrical valve seat was used for this experiment. The valve parts were not coated. The crucible load was made up of a mixture of 100 g of ZrO_2 powder and 900 g of Zr powder (10 w/o ZrO_2, 2.6 w/o O, 13.2 a/o O) in four polyethylene bags. Two additional layers of zirconia felt, extending over the dropping hole, were placed beneath the crucible, one between the zirconia and alumina slabs and the other between the alumina slab and the aluminum support plate. Each of these felt pieces had a series of radial cuts through the felt in the hole location such that the felt in the hole was a group of triangular segments attached only at the circumference of the hole.

Three Ta wire nails, at 120°, were driven through all four pieces of bottom insulation, near the outer edge. The crucible valve plug was connected to an external pneumatic cylinder attached to the vessel lid. A Type-K thermocouple was placed between the alumina and zirconia bottom insulation, extending slightly into the drop hole.

The containment vessel was evacuated and backfilled with argon. Heating was started at 130 A (92% current level) giving 65% power then reduced to 105 A. As the temperature approached 2373 K the power was reduced to about 78 A. The crucible temperature overshot to 2393 K, then gradually dropped to 2323 K. After about 500 s, the power was raised to 80 A and held for the remainder of the soak time. Fifteen minutes after first reaching 2323 K, water was added to the test section and the detonator fire circuit was armed. After a total time above 2323 K of 1200 s, the power was increased to 122 A. The temperature increased. When it reached 2473 K (see the pyrometer record shown in Fig. D.59), the drop mechanism was actuated. Melt fell from the crucible and an explosion occurred. The water temperature was 293 K.

12.2 Observations

All of the burn wires opened. The delay between the first and second (0.22 s) translates to an entrance velocity of 1.4 m/s. Normally the second and third wires break simultaneously as the breaking of one triggers the explosion which breaks the other. However, the delay of 0.05 s between the breaking of these two wires may be due to either the third break triggering the explosion or a delay in the breaking of the third by the explosion. The top containment vessel temperature reached 453 K while the bottom reached 468 K. The containment vessel pressure jumped from 120 to 550 kPa (see Fig. D.60). The insulation thermocouple showed an abrupt jump from 1573 K to burnout at the drop time. The video showed a clean drop of most of the melt.

A new trigger circuit assured that the two data capture PCs start at the same time. Two transducers produced questionable signals. A low transducer, P-5 at 46 cm below the surface, showed erratic swings from large positive to negative. Another transducer, P-7 at 30 cm below the surface, showed a reasonable signal although with more noise than the other transducers. A separate plot was made of this data filtered through a 5 kHz low-pass filter (see Fig. D.61). P-6, also 30 cm deep, had an amplitude about half of the other transducers (see Fig. D.62). These two transducers at 30 cm deep, P-6 and P-7, otherwise showed similar

records. They had initial narrow spikes greater than 30 MPa at 0.3 ms, probably from the trigger explosion, followed in about 0.6 ms by the explosion signal. This signal had a peak pressure of about 60 (P-6) or 120 (P-7) MPa and a duration of about 3 ms. The two deepest transducers, P-2 at 76 cm deep and P-3 at 61 cm deep , showed pulses with shorter duration (~1 ms) and pressures peaking at 100 to 120 MPa (see Figs. D.63 and D.64). Forces measured by the force transducer over-ranged at 500 kN (see Fig. D.65). The containment gas shock pressure at the wall of the containment peaked at 500 kPa (see Fig. D.66).

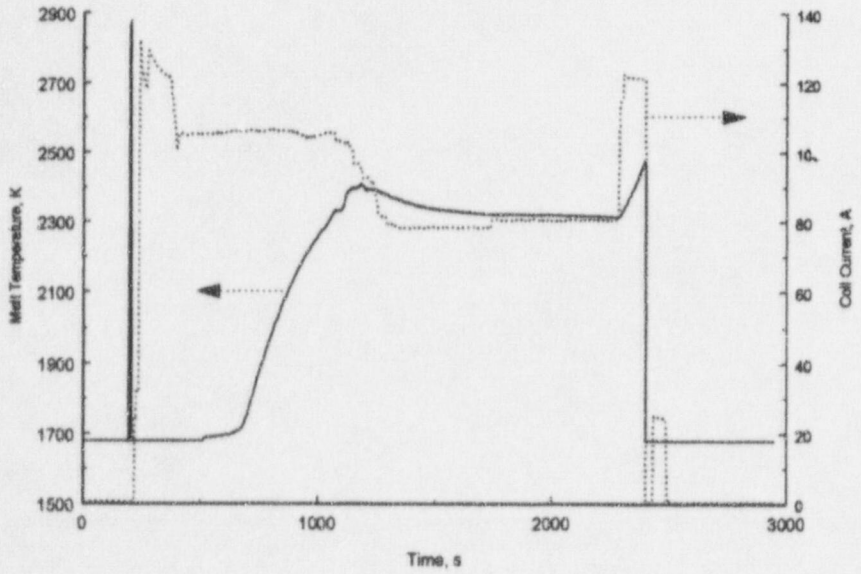


Figure D.59. ZREX-41 Melt Heating Record.

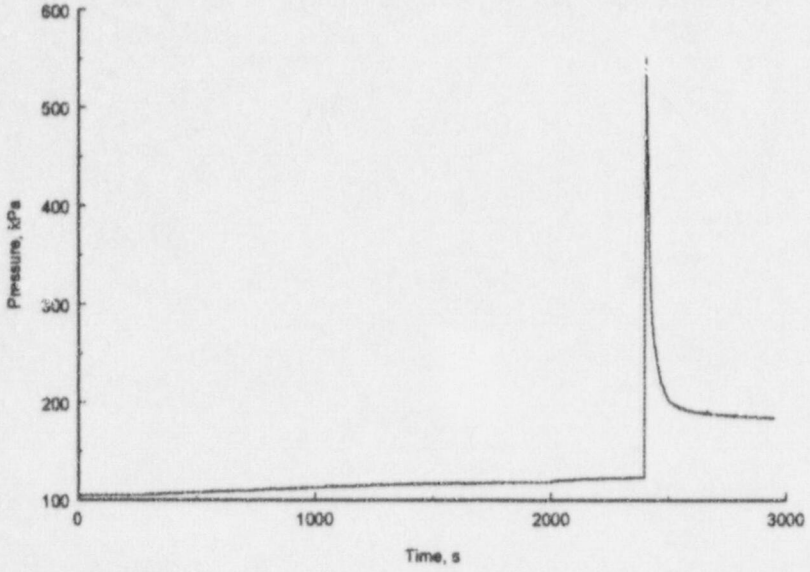


Figure D.60. ZREX-41 Containment Pressure.

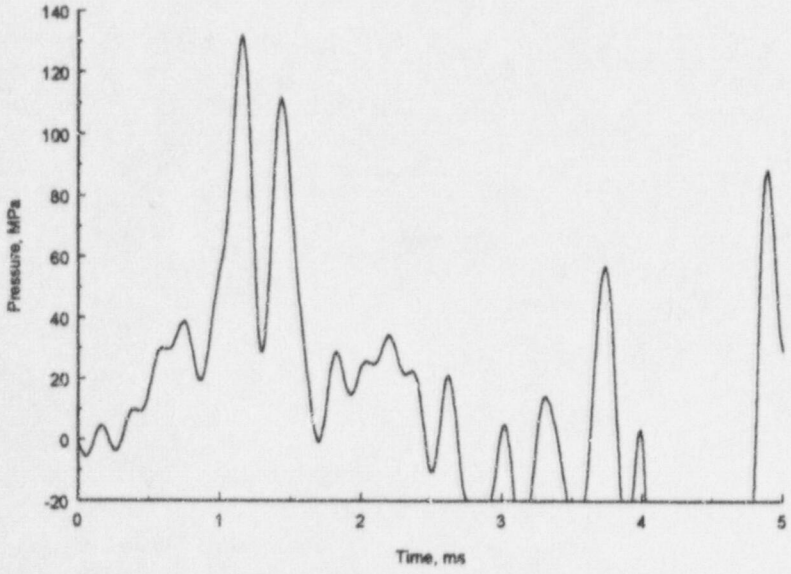


Figure D.61. ZREX-41 P-7 Pressure Record.

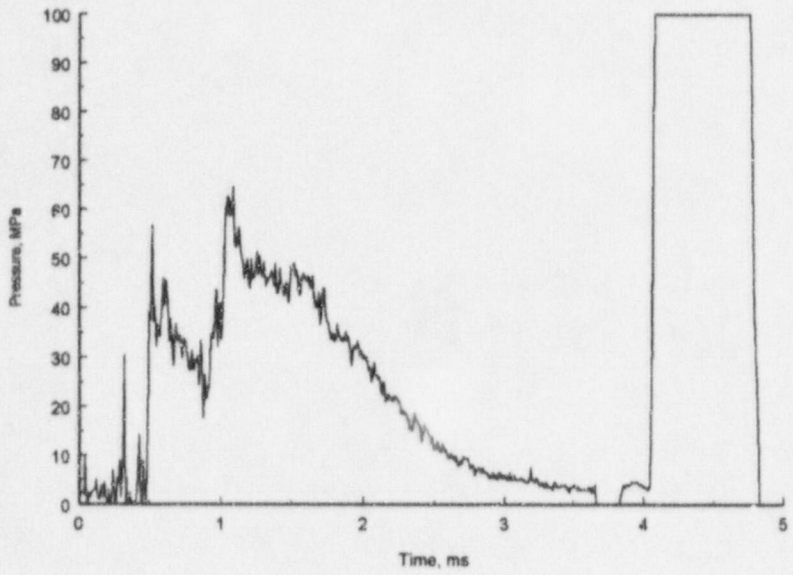


Figure D.62. ZREX-41 P-6 Pressure Record.

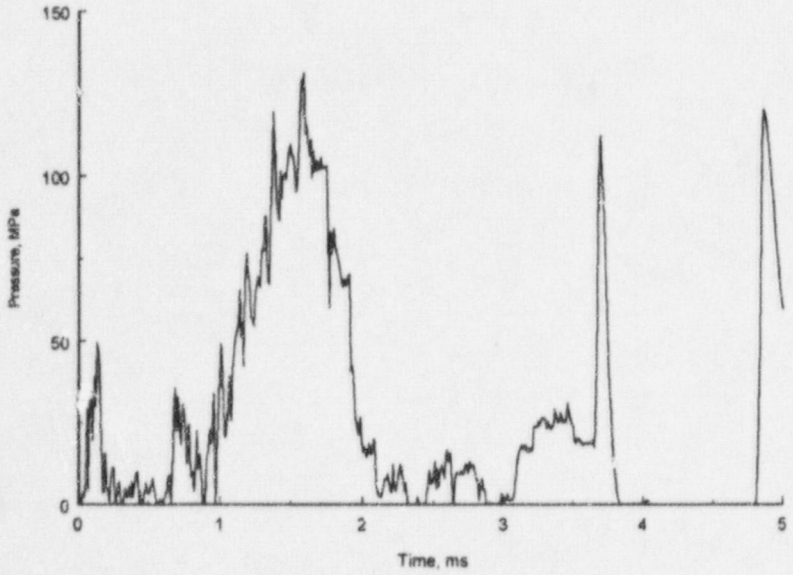


Figure D.63. ZREX-41 P-2 Pressure Record.

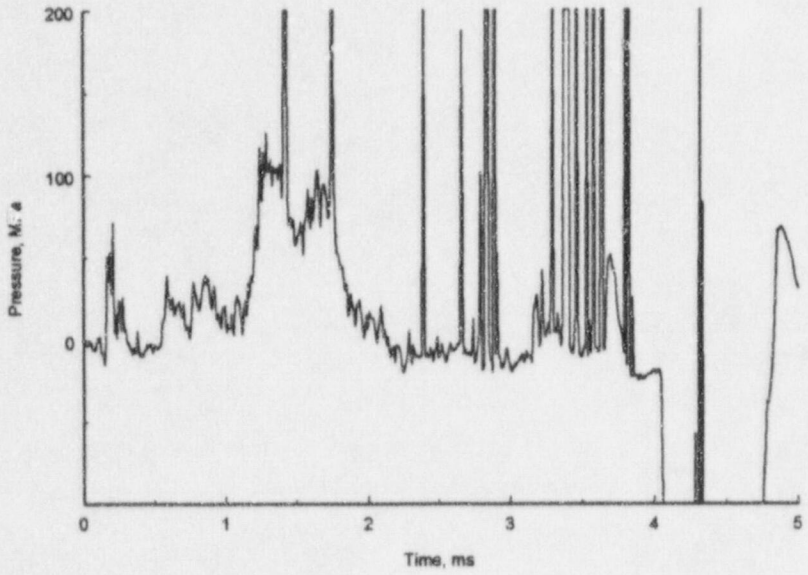


Figure D.64. ZREX-41 P-3 Pressure Record.

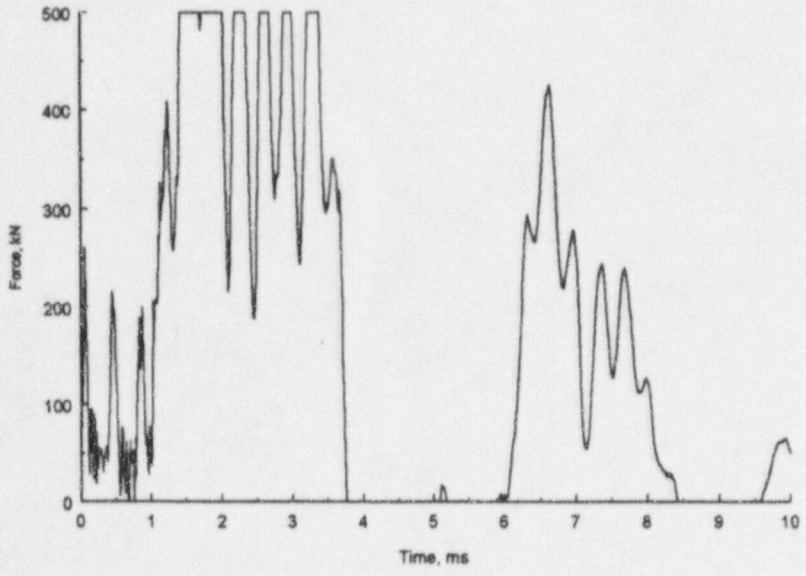


Figure D.65. ZREX-41 Force Record.

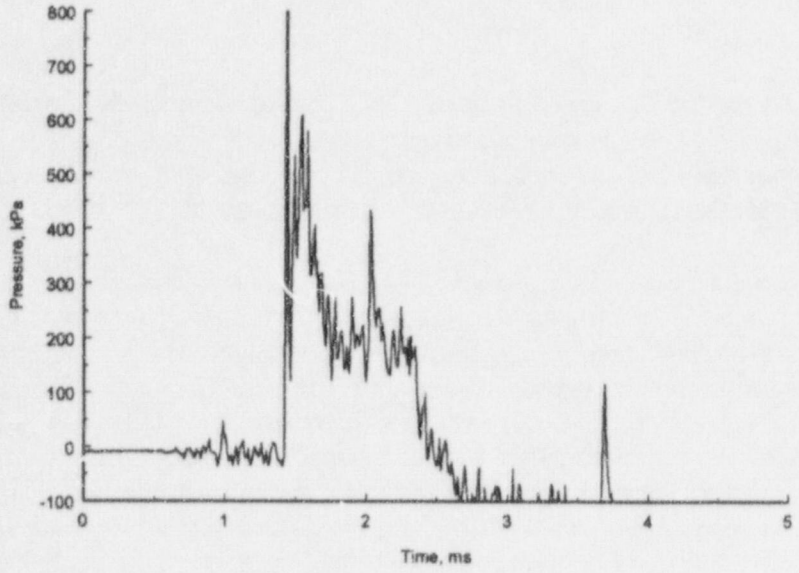


Figure D.66. ZREX-41 Fast Response Containment Pressure.

13 ZREX-47

ZREX-47 was run on July 10, 1997. This experiment was an attempt to drop one kg of 100 w/o Zr into room temperature water and trigger an explosion. It was a repeat of ZREX-46 or of ZREX-20 with the trigger burn wires set at 30 and 31 cm below the water surface. ZREX-20 had the trigger level set at 20 and 21 cm. The bottom insulating scheme used in ZREX-20 was used and the valve mechanism was not coated. A lower heat-up rate than used in ZREX-46 was used for this test. The drop mechanism was actuated and melt fell from the crucible. There was an explosion.

13.1 Operation

A standard dropping crucible with a 2 mm chamfer on a cylindrical valve seat was used for this experiment. The crucible load was 1003 g of Zr rod.

The containment vessel was evacuated and backfilled with argon. Heating was started at 118 A (83% current level) giving 59% power. The water pump was started, adding two liters of water to the test section as soon as the crucible had heated to a temperature where the self-luminosity was enough to allow a final check of the pyrometer alignment. The remaining 7.7 l of water was added when the crucible temperature reached 2123 K. This procedure was followed to prevent leaking melt from welding to the bottom of the test section. This had occurred in some earlier ZRSS runs. The solidified melt could not be removed so a new primacord hole was drilled above the frozen melt level. Heating rates for ZREX-46 and ZREX-47 were about 4 and 1 K/s respectively. When the melt temperature reached 2373 K (see the pyrometer record shown in Fig. D.67), the dropping mechanism was actuated. The valve rod moved immediately, a uniform appearing stream fell from the crucible and an explosion occurred. Subjectively, this was the largest explosion of the ZREX series. A 5 cm long segment of the top containment vessel gasket (1 mm Durlon) was blown out, allowing the vessel atmosphere to escape before a sample could be taken. The gasket gap was temporarily plugged with putty

and argon was let into the vessel at 6 l/min. This caused a reasonable flow of gas through the sample bottles. After about 2 minutes a gas sample was taken.

13.2 Observations

All of the burn wires opened with a delay of 0.11 s between top and next. The containment vessel pressure jumped from 116 to 540 kPa (see Fig. D.68). Water in the test section was at 298 K. Containment temperatures reached 508 K for the top and 458 K for the bottom thermocouple. Post-test examination of the apparatus showed that more damage had been done than in any previous experiment.

Two transducers produced questionable signals. A low transducer, p-2 at 76 cm below the surface, dropped to a negative value immediately (indicative of failure) after the trigger fired. Although it later responded to pressures from the explosion, it was probably failing. P-6, at 30 cm deep gave values about half those of p-7, also 30 cm deep. These two transducers otherwise showed similar records (see Figs. D.69 and D.70). They had an abrupt rise to a peak at 0.4 ms (after the initial electrical noise spike at zero time), followed by a fairly flat plateau lasting about 1.2 ms, then a decay back to zero in another 2 ms. The two deeper transducers, p-3 at 61 cm deep and p-5 at 46 cm deep, showed more sharply peaked pulses with similar durations (~3 ms) and pressures peaking at 100 to 180 MPa (see Figs. D.71 and D.72). Forces measured by the force transducer over-ranged at 500 kN (see Fig. D.73). The gas shock pressure at the wall of the containment peaked at 600 kPa (see Fig. D.74).

Some estimates of the energy of the explosion can be made from the force and pressure signals. The force over-ranged to a large extent and the force transducer was loose after the experiment. As no tension signals were received after the first impulse, it is assumed that the loosening occurred as a result of the explosion.

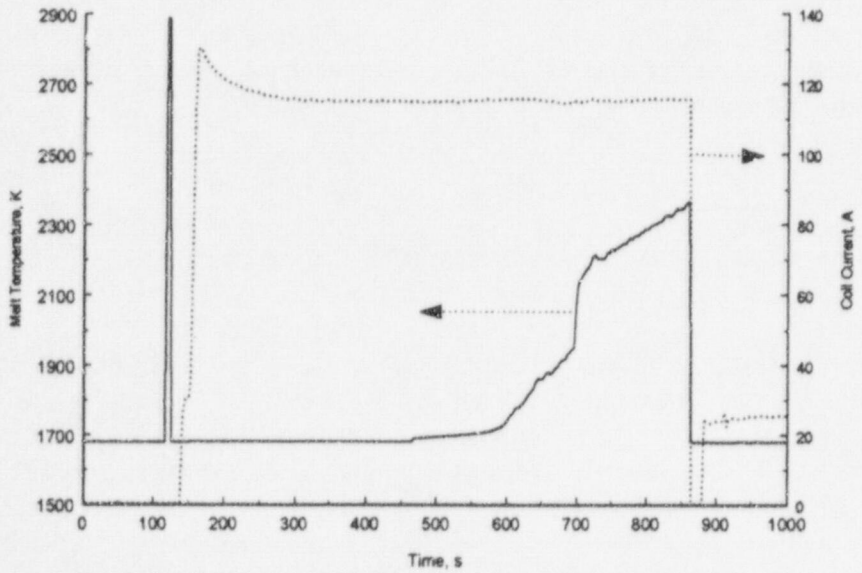


Figure D.67. ZREX-47 Melt Heating Record.

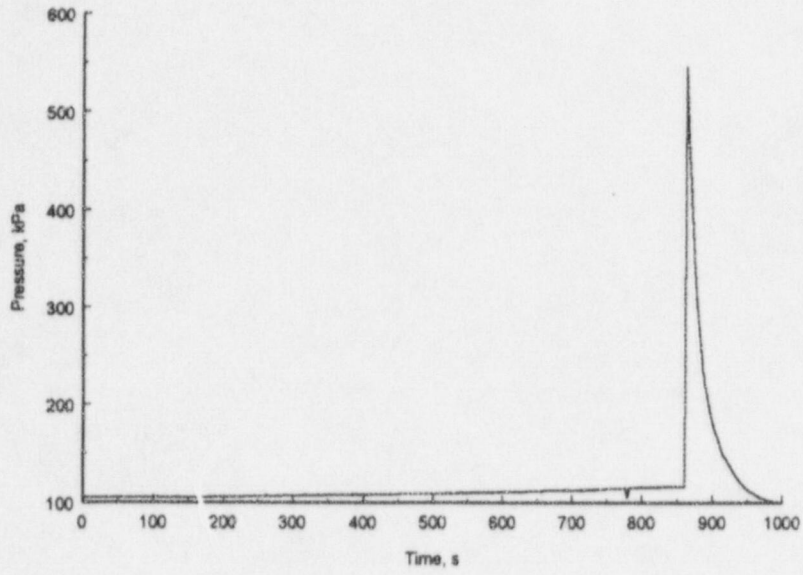


Figure D.68. ZREX47 Containment Pressure.

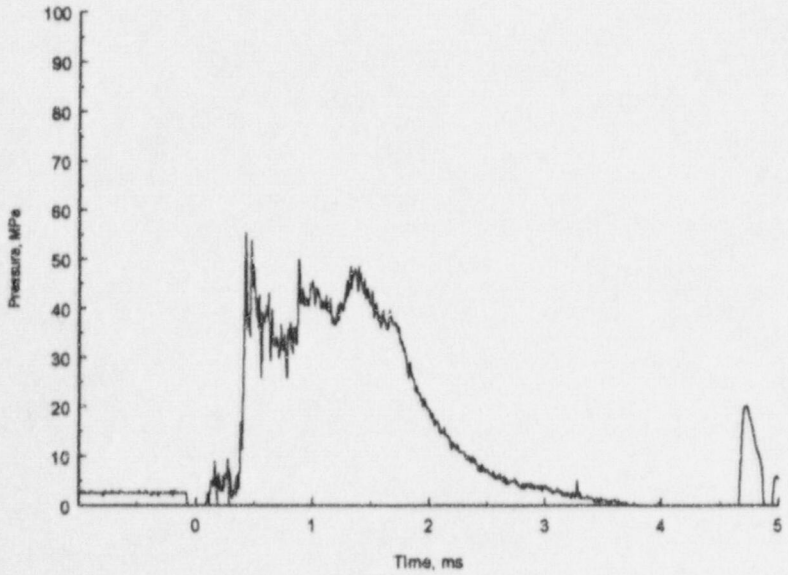


Figure D.69. ZREX-47 P-6 Pressure Record.

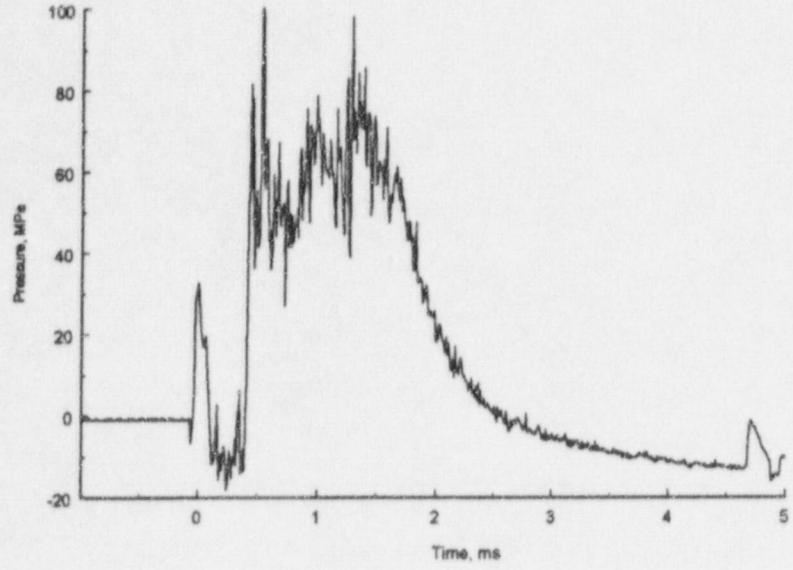


Figure D.70. ZREX-47 P-7 Pressure Record.

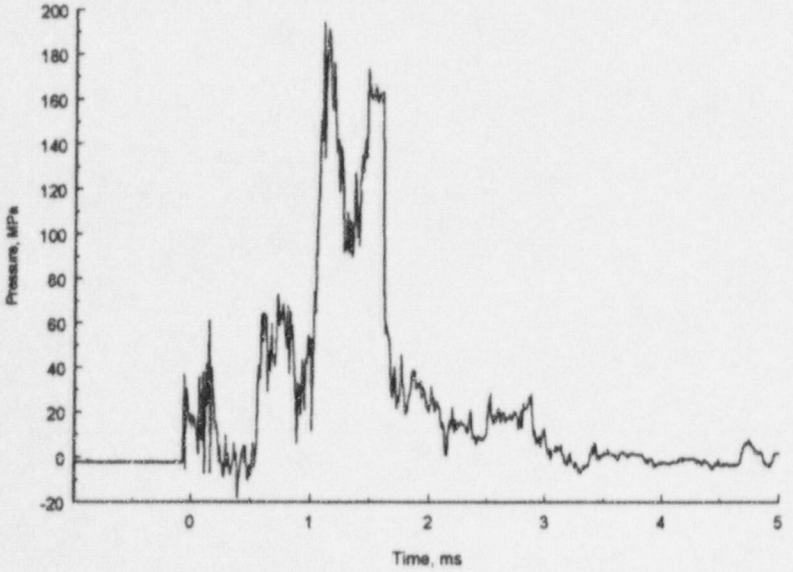


Figure D.71. ZREX-47 P-3 Pressure Record.

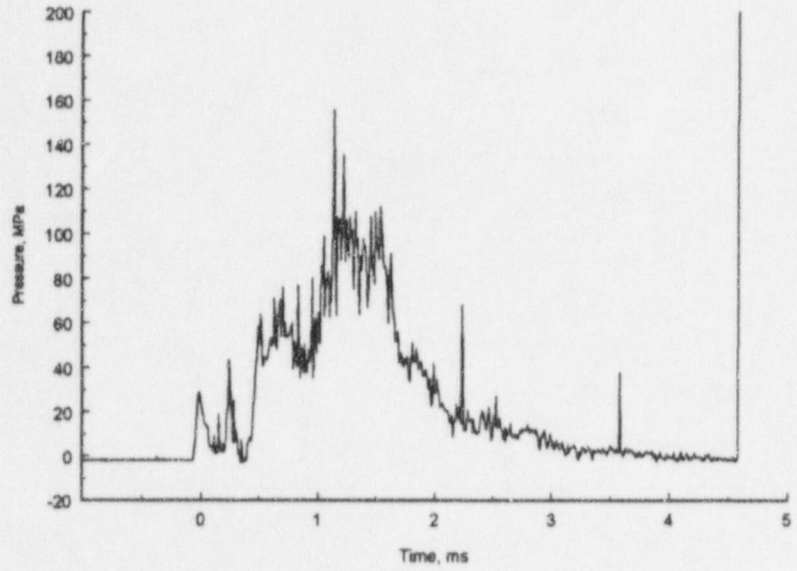


Figure D.72. ZREX-47 P-5 Pressure Record.

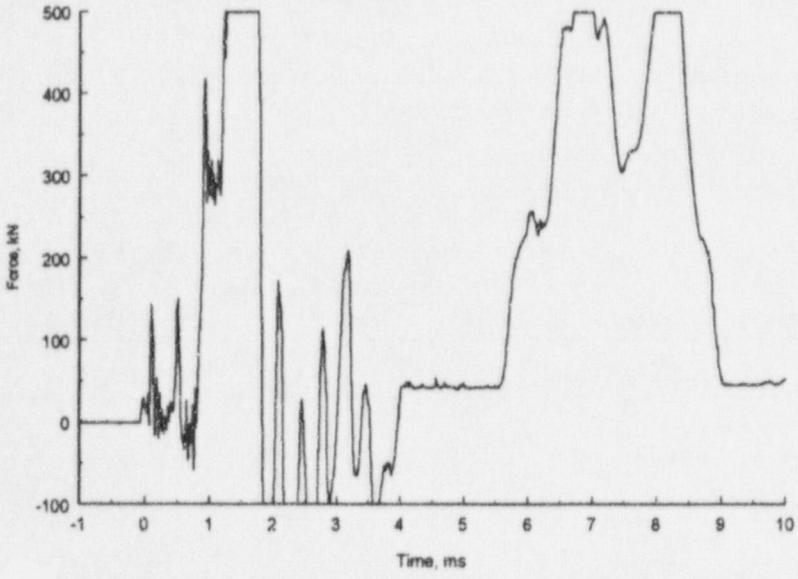


Figure D.73. ZREX-47 Force Record.

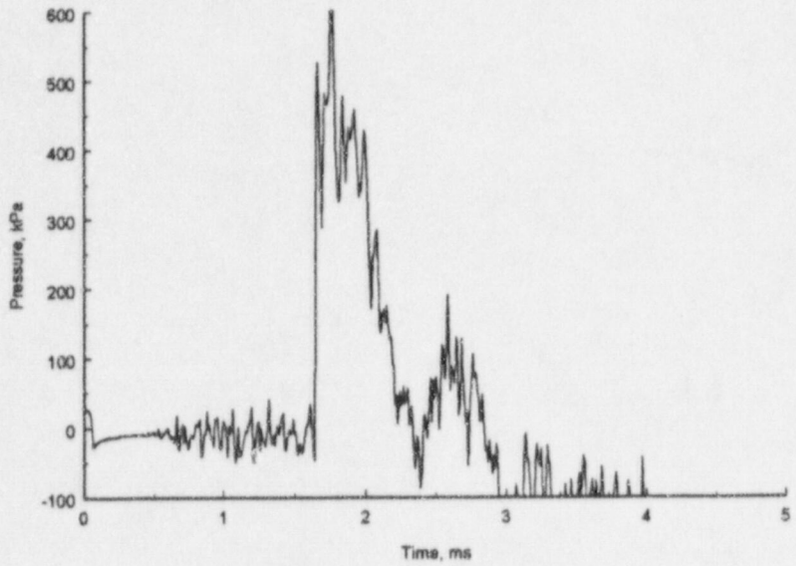


Figure D.74. ZREX-47 Fast Response Containment Pressure.

The force signal showed three pulse signals which are assumed to be the original event and two rebounds (the rebounds would be the positive side of a decaying sinusoidal signal expected from the spring-mass system made up of the test section and force transducer). All of the signals showed clipping at a 500 kN limit. Ignoring the clipping, the integrated area of the first pulse gave an impulse of 410 N-s, the second 1197 N-s, and the third 1291 N-s. These impulses can be converted to mechanical energies if it is assumed that the impulse is the reaction to ejecting a mass m of material from the test section.

$$E = I^2/(2m)$$

Assuming that all of the zirconium melt was in the test section, displacing only its own volume of water, and that all of the material above the triggering burn wire was ejected, this mass is equal to 3.23 kg. The resulting mechanical energy for each of the impulses is listed below.

Transducer	Impulse, N-s	Energy, kJ
Force link	410	30.0
Force link	1197	222
Force link	1291	258

The second value was chosen to represent the impulse developed by this explosion.

14 ZREX-50

ZREX-50 was run on August 4, 1997. This experiment was an attempt to drop one kg of 100 w/o Zr into room temperature water and trigger an explosion. It was a repeat of ZREX-47 with double the trigger energy or 2 g PETN. The bottom insulating scheme and heat-up rate used in ZREX-47 was used and the valve mechanism was not coated. When the drop mechanism was actuated, melt fell from the crucible. There was an explosion.

14.1 Operation

A standard dropping crucible with a 2 mm chamfer on a cylindrical valve seat was used for this experiment. The crucible load was 1000 g of Zr rod .

The containment vessel was evacuated and backfilled with argon. Heating was started at 115 A (86% current level) giving 89% power. The water pump was started, adding two liters of water to the test section as soon as the crucible had heated to a temperature where the self-luminosity was enough to allow a final check of the pyrometer alignment. The remaining 7.7 l of water was added when the crucible temperature reached 2073 K. The heating rate was about 1 K/s. When the crucible temperature reached 2373 K (see the pyrometer record shown in Fig. D.75), the dropping mechanism was actuated. The valve rod moved immediately, a uniform appearing stream fell from the crucible and an explosion occurred. This explosion appeared to be as large as ZREX-47. A 5 cm long segment of the top vessel gasket (1 mm Durlon) was blown out, allowing the vessel atmosphere to escape before a sample could be taken. The gasket gap was temporarily plugged with putty and a gas sample was pulled through the sample bottles with a vacuum pump.

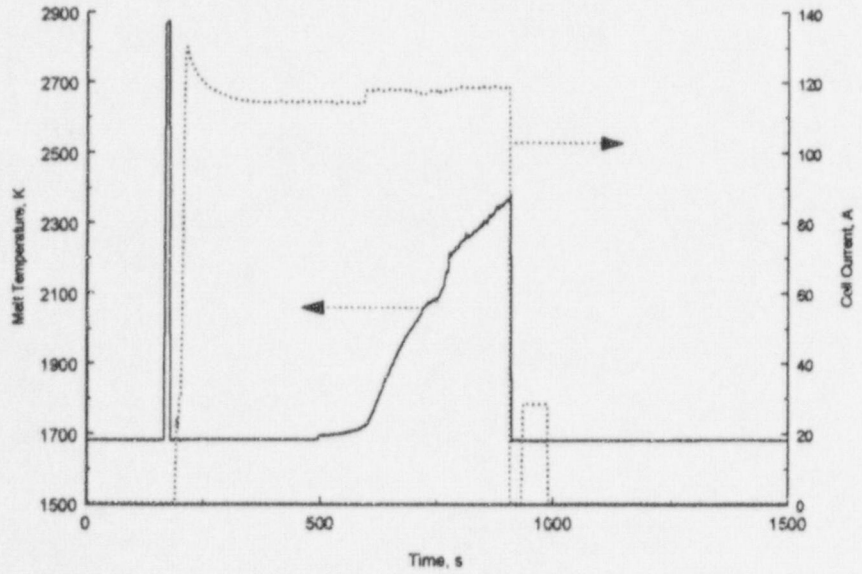


Figure D.75. ZREX-50 Melt Heating Record.

14.2 Observations

All of the burn wires opened with a delay of 0.21 s between top and next. There was an apparent delay of 0.06 s between the opening of the second burn wire and the trigger although the trigger should have occurred coincidentally with the opening of the second burn wire. The containment vessel pressure jumped from 114 to 580 kPa (see Fig. D.76). Water in the test section was at 299 K. Containment vessel temperatures reached 558 K for the top and 481 K for the bottom thermocouple.

One transducer produced a questionable signal. P-6, at 30 cm below the surface, dropped to a low value after about two ms of normal response. All of the transducers showed a coincidental noise signal at 5.2 ms. The test section pressure transducers had similar signals, first a sharp peak from the trigger followed by a fairly abrupt rise to a peak then a dip, recovery and decay back to zero (see Figs. D.77, D.78, D.79, and D.80). Forces measured by the force transducer over-ranged at 500 kN (see Fig. D.81). The gas shock pressure at the wall of the containment peaked at 600 kPa (see Fig. D.82).

Post test examination of the apparatus showed that the damage done was similar to that of ZREX-47.

The gas analysis for this run gave anomolous results. There was an excess of nitrogen unaccompanied by oxygen in both the before and after samples. This was ignored and the producer gas reaction, which was small on the basis of the carbon monoxide and carbon dioxide contents, was also ignored. Due to the gasket blow out a final gas pressure could not be measured. The total amount of hydrogen produced in the interaction was estimated by assuming that the gas composition did not change during the leak, i.e., the ratio of hydrogen to argon remained constant. Then the hydrogen amount could be calculated because the initial amount of argon was known from the before analysis.

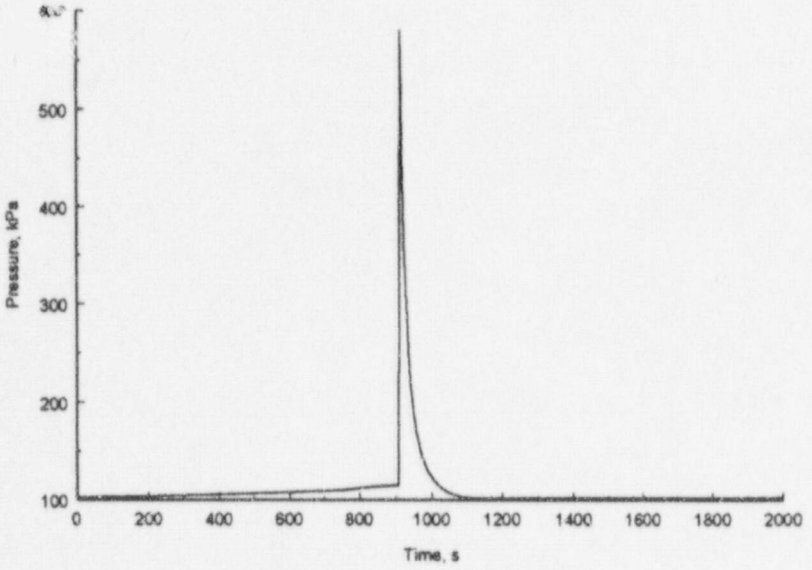


Figure D.76. ZREX-50 Containment Pressure.

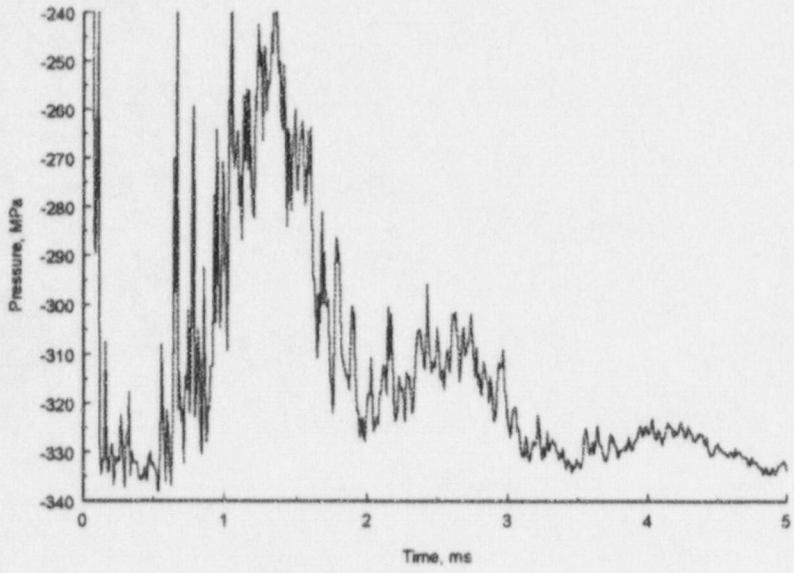


Figure D.77. ZREX-50 P-2 Pressure Record.

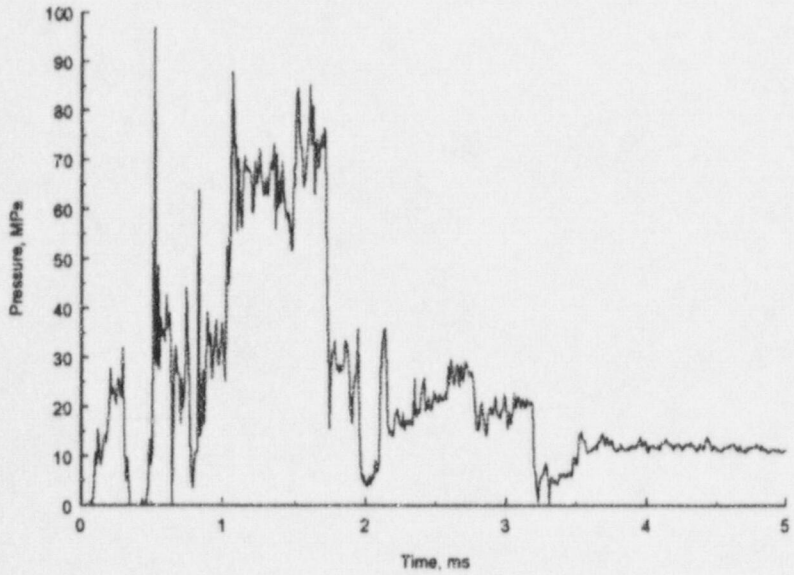


Figure D.78. ZREX-50 P-3 Pressure Record.

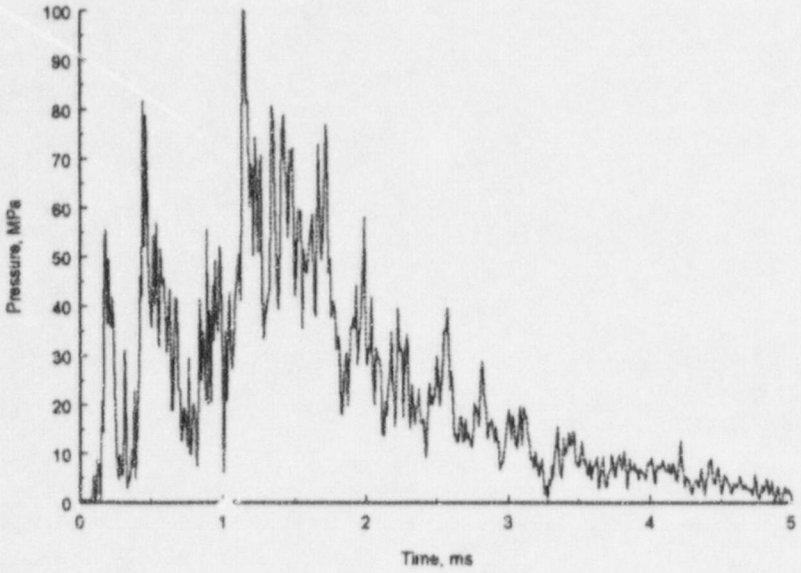


Figure D.79. ZREX-50 P-5 Pressure Record.

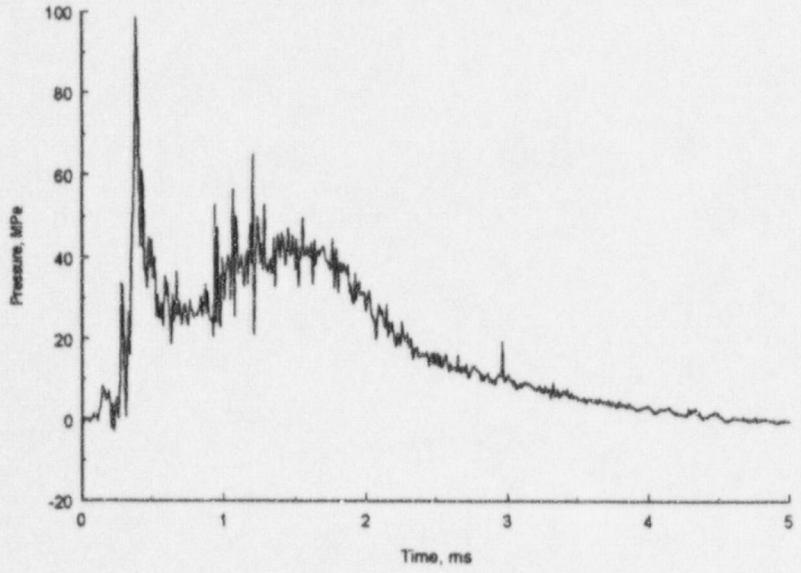


Figure D.80. ZREX-50 P-7 Pressure Record

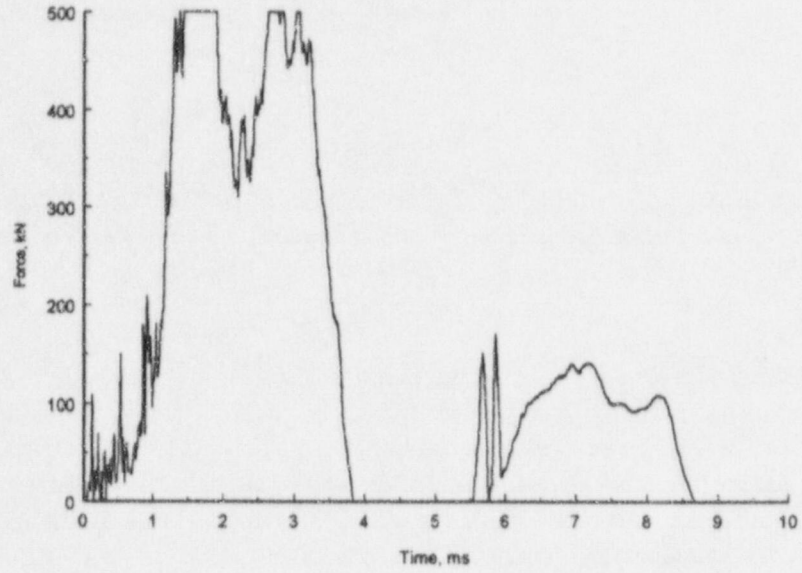


Figure D.81. ZREX-50 Force Record

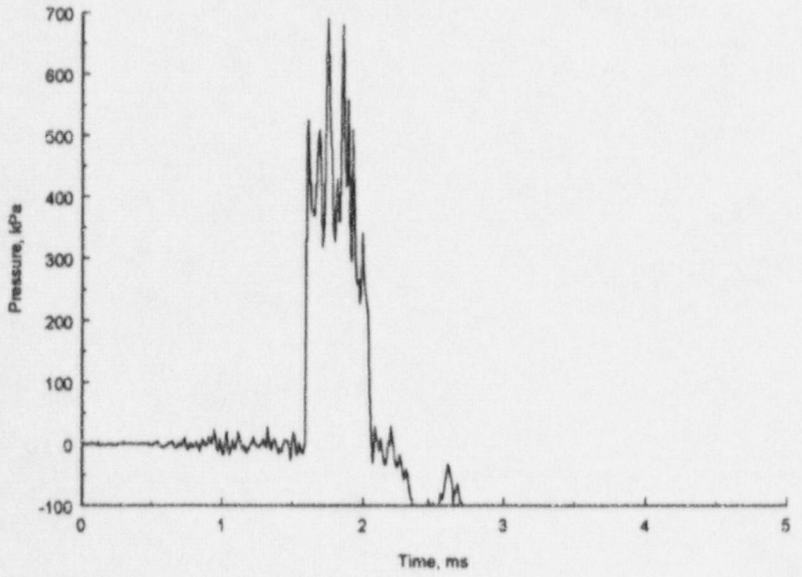


Figure D.82. ZREX-50 Fast Response Containment Pressure.

15 ZRSS-4

ZRSS-4 was run on May 15, 1997. This experiment was an un-triggered drop of 1200 g of mixture of 64 w/o stainless steel and 36 w/o zirconium into room-temperature water. There was no explosion.

15.1 Operation

A mixture of 432 g of Zr powder and 768 g of stainless steel (3 mm Type-304 shot) was put in four polyethylene bags and placed in a graphite dropping crucible. The crucible and valve were coated with two layers of yttrium oxide aerosol spray coating baked for 10 minutes at 873 K after each coat. A set of four burnwire frames was suspended in the test section beneath the water. They were placed at 1.0, 25.5, 50.0, and 74.5 cm deep.

The vessel was evacuated and backfilled with argon. The water pump was started and the test section filled with water as soon as the crucible had heated to a temperature where the self-luminosity was enough to allow a final check of the pyrometer alignment Heating was started at 115 A and held there until the crucible temperature approached 2123 K when the power was turned down to 78 A. The crucible temperature stabilized at about 2133 K and was held there for 5 minutes. At the end of this period, the power was increased to 110 A and the crucible temperature began to climb. As it approached 2153 K (see the pyrometer record shown in Fig. D.83), the dump mechanism was actuated. Melt fell from the crucible into the test section. The top burn wire opened and the PC recorders triggered.

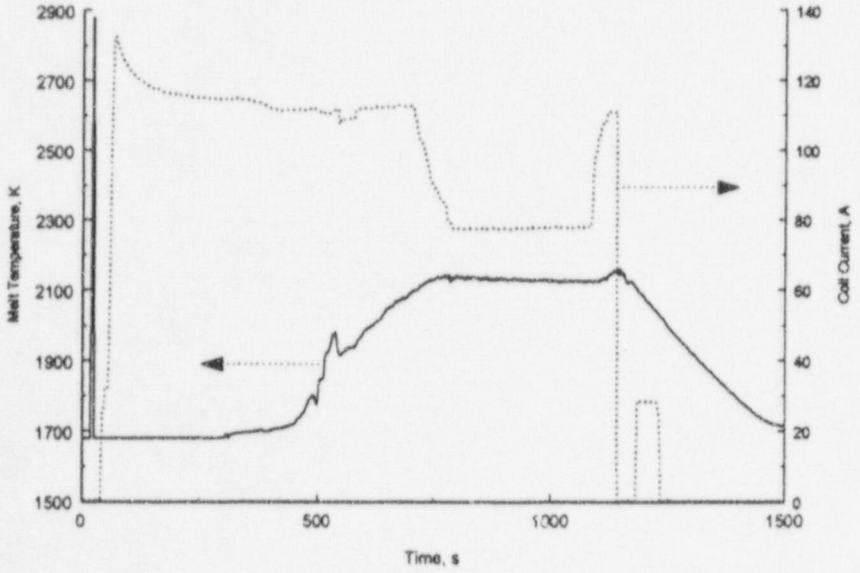


Figure D.83. ZRSS-4 Melt Heating Record.

15.2 Observations

The top three burn wires opened at an interval of 0.15 s between the top two and 0.33 s between the next two. No discernable pressures were detected. The test section water temperature thermocouple gave erroneous results, indicating temperatures above the boiling point. Experience gained from previous runs suggests that

the test section temperature was actually the same as that of the containment vessel at the start of the run which was 293 K. Examination of the crucible showed that most of the load had fallen from the crucible (199 g remained in the crucible) into the test section (972 g, 29 g missed the test section). There was little attack on the crucible. The melt had fragmented into lumps, sheets, and sharp rods. A photograph of the debris is shown in Fig. D.84. Table D.3 summarizes the results of a sieve analysis of the debris.

The containment vessel pressure jumped from 124 to 132 kPa (see Fig. D.85).

Figure D.84. ZRSS-4 Debris.

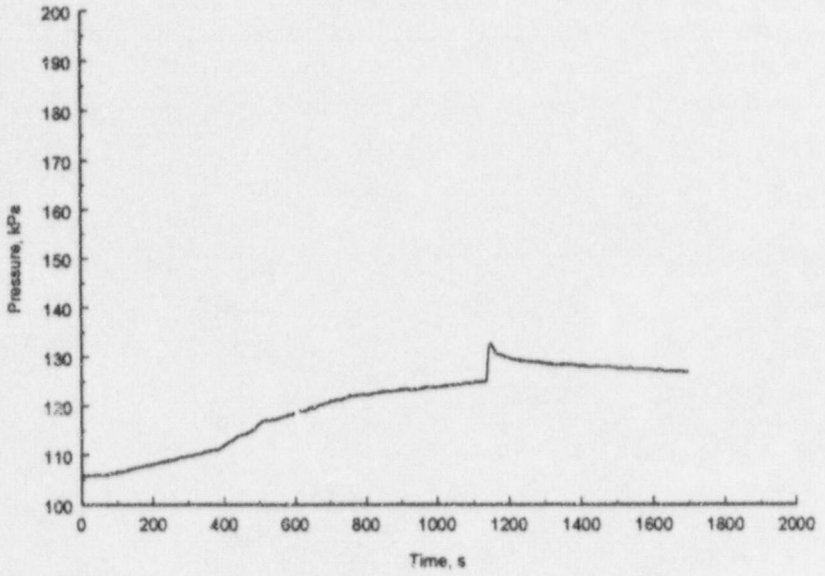


Figure D.85. ZRSS-4 Containment Pressure.

Table D.3 Sieve analysis for ZRSS-4 debris

Size range (mm)	Amount (%)
>8.00	60.9
8.00 - 5.60	16.6
5.60 - 4.00	10.3
4.00 - 2.80	6.4
2.80 - 2.00	3.4
2.00 - 1.40	1.1
1.40 - 1.00	0.51
1.00 - 0.71	0.19
0.71 - 0.50	0.15
0.50 - 0.355	0.12
<0.355	0.35

16 ZRSS-5

ZRSS-5 was run on May 27, 1997. This experiment was a triggered drop of 1200 g of mixture of 64 w/o stainless steel and 36 w/o zirconium into room-temperature. There was an explosion.

16.1 Operation

A mixture of 432 g of Zr powder and 768 g of stainless steel (3 mm Type-304 shot) was put in four polyethylene bags and placed in a graphite dropping crucible. The crucible and valve were coated with two layers of yttrium oxide aerosol spray coating baked for 10 minutes at 873 K after each coat. A set of three burnwire frames was suspended in the test section beneath the water. The frames were placed at 5.0, 30.0, and 31.0 cm deep. Opening of either of the bottom two burnwires would fire the detonator.

The vessel was evacuated and backfilled with argon. The water pump was started and the test section filled with water as soon as the crucible had heated to a temperature where the self-luminosity was enough to allow a final check of the pyrometer alignment Heating was started at 115 A and held there until the crucible temperature approached 2123 K when the power was turned down to 78 A. The crucible temperature stabilized at about 2133 K and was held there for five minutes. At the end of this period, the power was increased to 104 A and the crucible temperature began to climb. As it approached 2163 K (see the pyrometer record shown in Fig. D.86), the dump mechanism was actuated. Melt fell from the crucible into the test section and an explosion ensued.

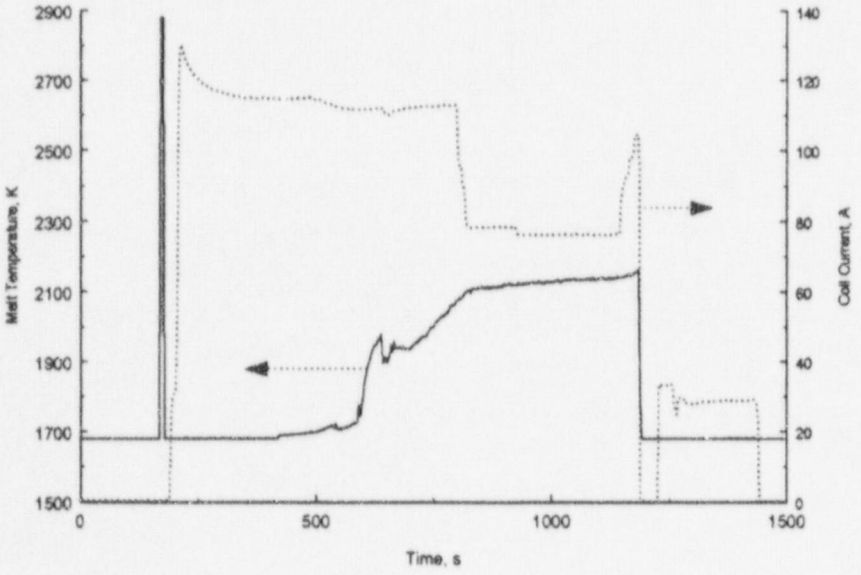


Figure D.86. ZRSS-5 Melt Heating Record.

16.2 Observations

The three burn wires opened at an interval of 0.16 s between the 5 and 31 cm wire and an additional 0.05 s before the 30 cm wire opened. The quasi-static containment vessel pressure jumped from 120 to 285 kPa (see Fig. D.87), while containment vessel temperatures reached 365 and 373 K for the top and bottom

thermocouples. The test section water temperature thermocouple indicated 293 K at the time of the explosion. The video showed a uniform drop of the melt starting in the center of the test section but shifting to the edge by the time of the explosion.

Post test examination of the apparatus showed that much less damage had been done than was normal in an explosion test with Zr or a Zr/ZrO_2 mixture.. The mechanism hanging from the vessel lid was bent but not destroyed. The crucible was mostly intact. The induction coil, and crucible support were bent. The bottom of the vessel was filled with debris and coated with mud made up of water from the experiment and grey dust along with small pieces of apparatus (crucible, burnwire, and lamp fragments, etc.) and fibrous particles from the insulation that had surrounded the crucible and the test section heaters. Only a few pieces identifiable as metal from the melt were found.

One transducer produced a questionable signal. P-3 at 61 cm below the surface, showed erratic swings from large positive to negative. The other test section transducers, p-2, p-5, p-6 and p-7, showed similar records (see Figs. D.88 - D.91). They had initial narrow spikes, probably from the trigger explosion, followed by the explosion signal. This signal had a peak pressure of about 30 MPa and a duration of about 0.5 ms. Forces measured by the force transducer peaked at something above 400 kN (see Fig. D.92). The dynamic (gas shock) pressure at the wall of the containment peaked at 200 kPa (see Fig. D.93).

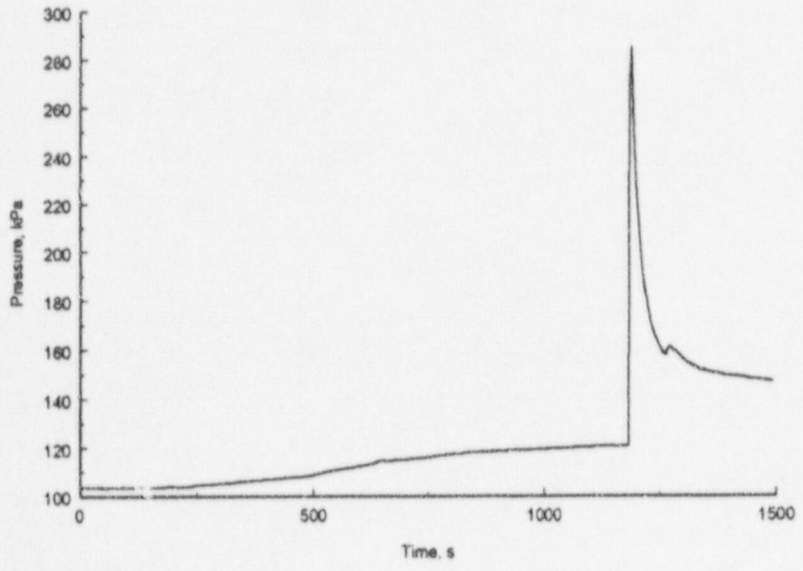


Figure D.87. ZRSS-5 Containment Pressure.

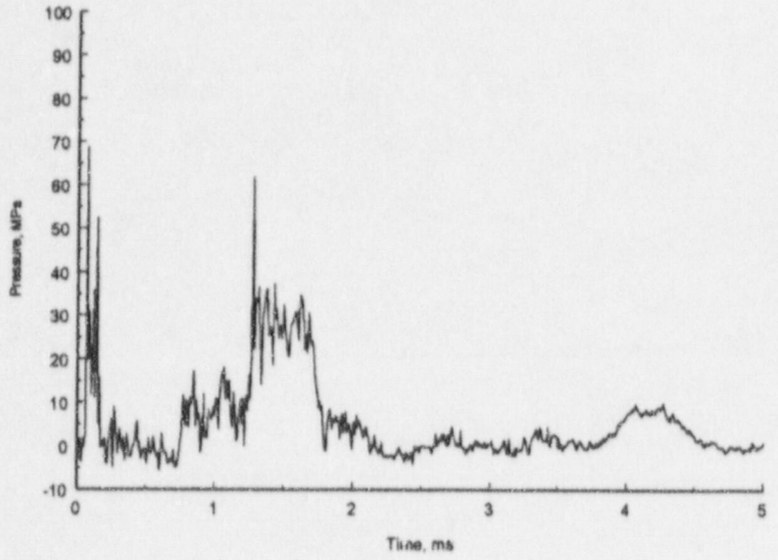


Figure D.88. ZRSS-5 P-2 Pressure Record.

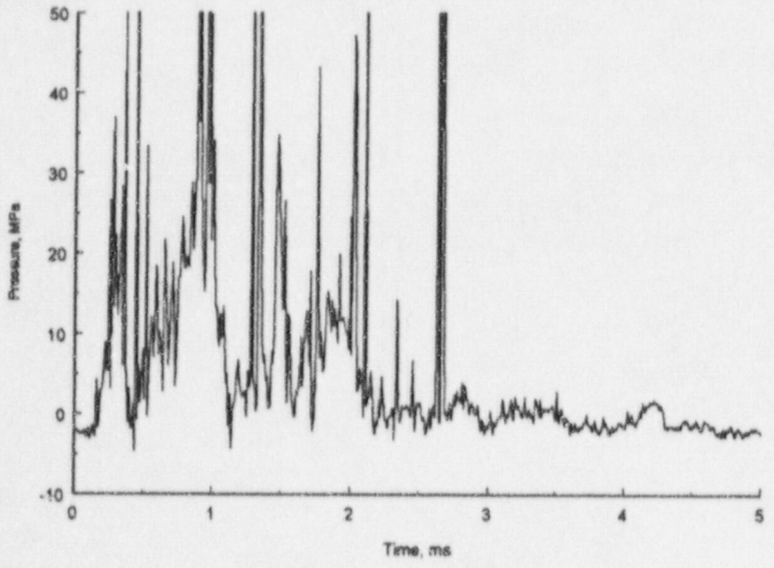


Figure D.89. ZRSS-5 P-5 Pressure Record.

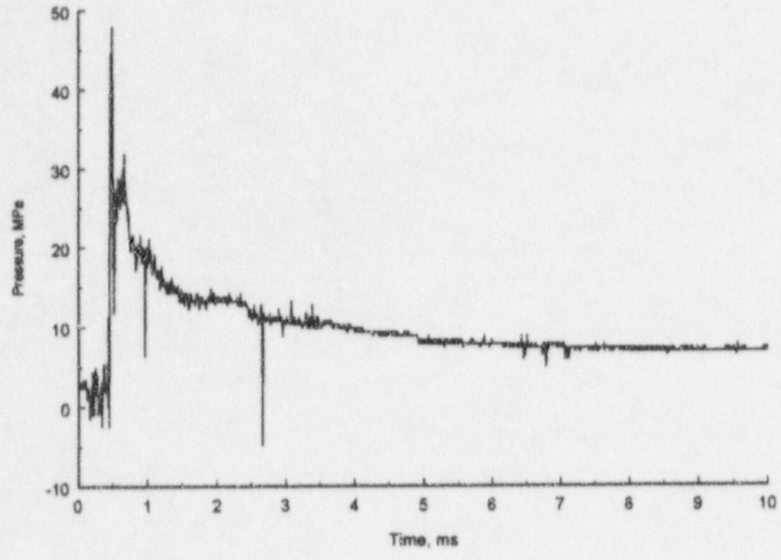


Figure D.90. ZRSS-5 P-6 Pressure Record.

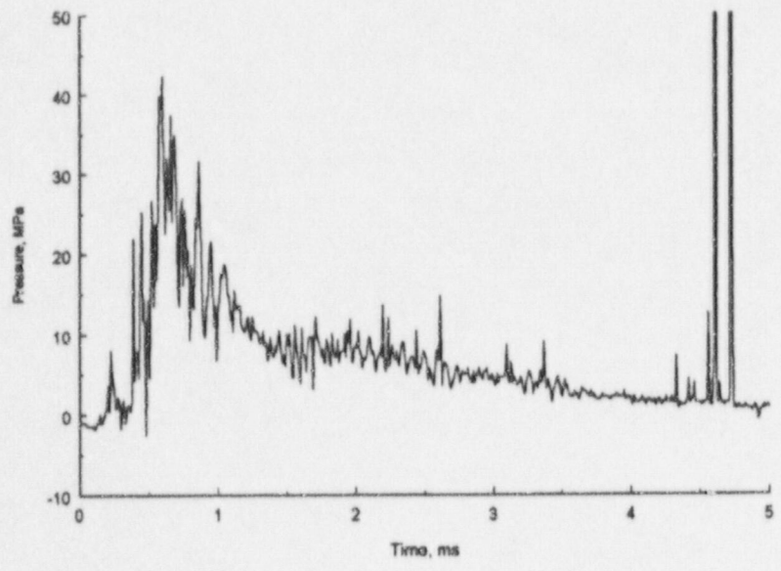


Figure D.91. ZRSS-5 P-7 Pressure Record.

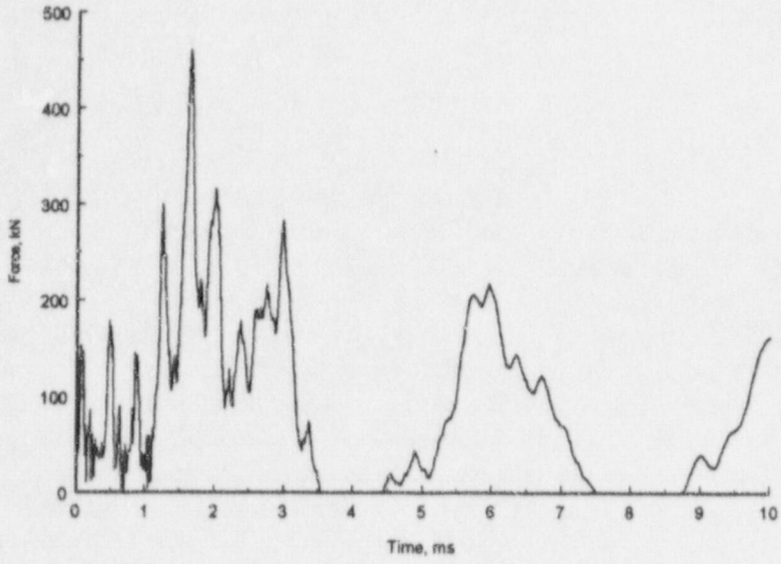


Figure D.92. ZRSS-5 Force Record.

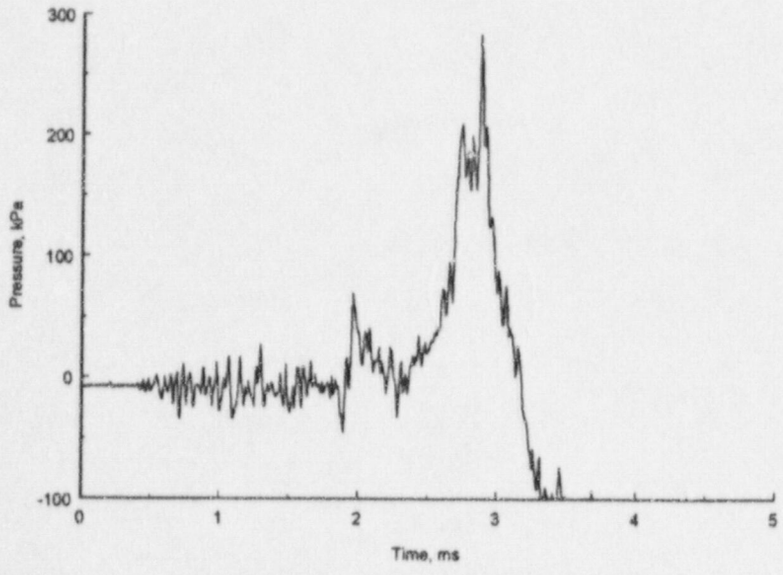


Figure D.93. ZRSS-5 Fast Response Containment Pressure.

17 ZRSS-6 and ZRSS-7

ZRSS-6 was run on June 2, 1997. This experiment was an un-triggered drop of 1200 g of 100 w/o stainless steel melt into room-temperature water. There was no explosion. ZRSS-7 was a duplicate of ZRSS-6 and was run on June 6, 1997.

17.1 Operation (ZRSS-6)

A load of 1200 g of stainless steel (3 mm shot, 304) was put in four polyethylene bags and placed in a graphite dropping crucible. The crucible and valve were coated with two coats of yttrium oxide aerosol spray coating baked for 10 minutes at 873 K after each coat

The vessel was evacuated and backfilled with argon. The water pump was started and the test section filled with water as soon as the crucible had heated to a temperature where the self-luminosity was enough to allow a final check of the pyrometer alignment. Heating was started at 118 A and held there until the crucible temperature approached 2020 K when the power was turned down to 78 A. The crucible temperature began to drop and the power was increased to 96 A when the temperature began to slowly increase with the evolution of considerable smoke and what appeared to be flake moving rapidly in the field of the pyrometer. The power was held at this level for 5 minutes while the temperature increased to 2123 K. At the end of this period, the power was increased to 110 A and the crucible temperature began to climb. As it approached 2153 K (see the pyrometer record shown in Fig. D.94), the dump mechanism was actuated. Melt fell from the crucible into the test section. The top burn wire opened and the PC recorders triggered.

ZRSS-7 was a repeat of ZRSS-6 (see the pyrometer record shown in Fig. D.95).

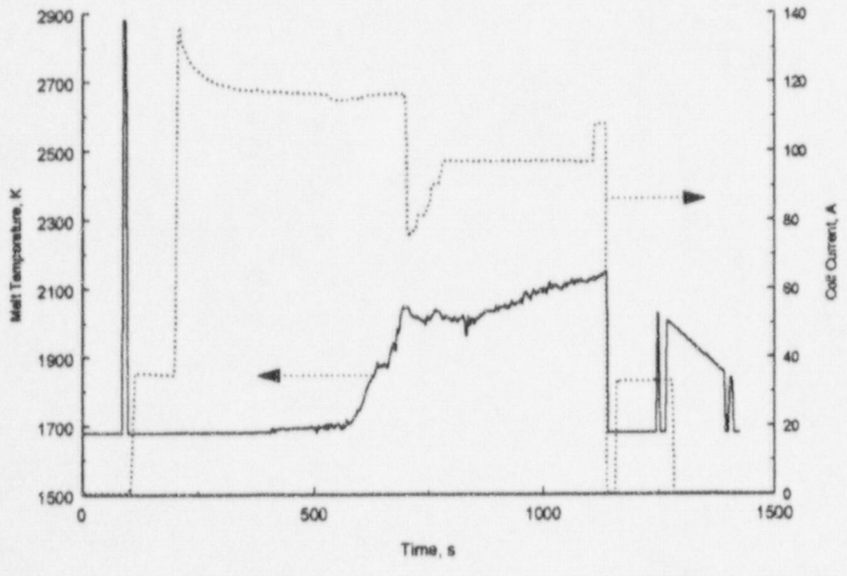


Figure D.94. ZRSS-6 Melt Heating Record.

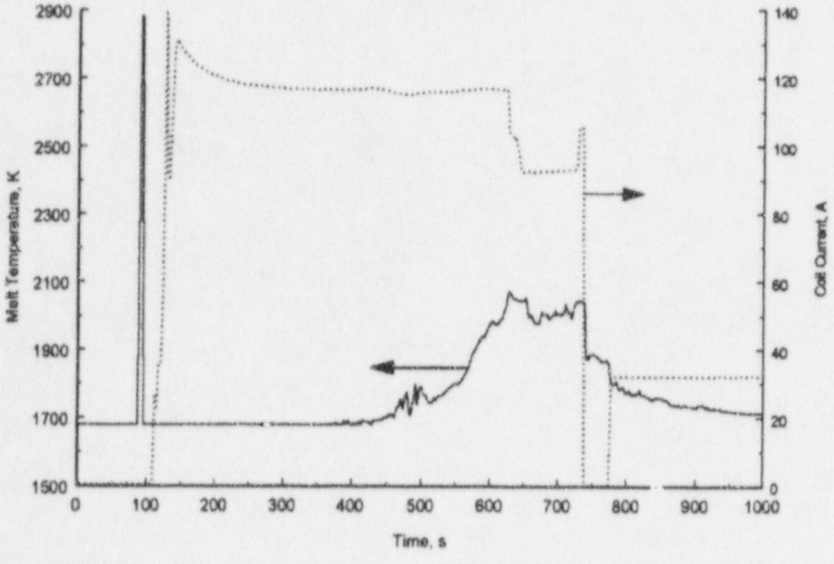


Figure D.95. ZRSS-7 Melt Heating Record.

17.2 Observations

The burn wires all opened. However, the file recorded on the PC was damaged and timing intervals could not be read. No discernable pressures were detected. The quasi-static containment vessel pressure jumped from 128 to 130 kPa (see Fig. D.96) for ZRSS-6 and from 119 to 122 kPa (see Fig D.97) for ZRSS-7.

Examination of the crucible showed that most of the load had fallen from the crucible into the test section. There was little attack on the crucible. Apparently the melt had remained molten throughout its fall and pooled in the catch cup.

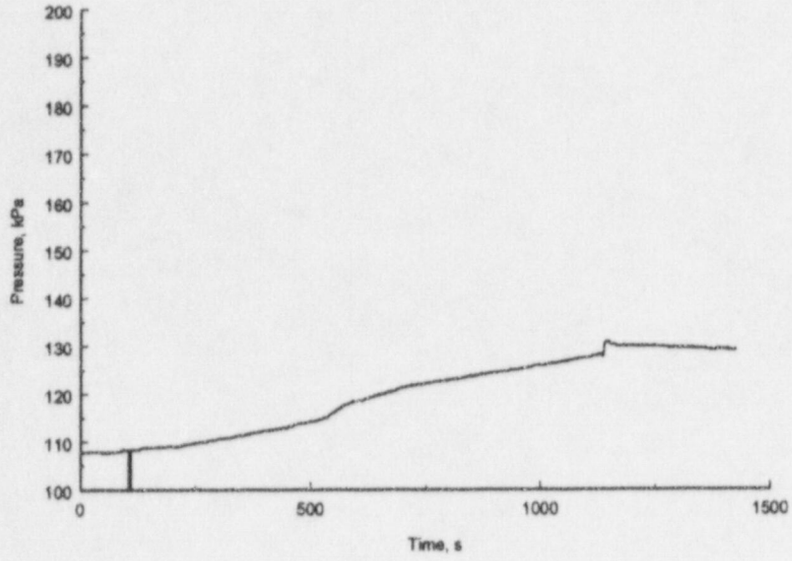


Figure D.96. ZRSS-6 Containment Pressure.

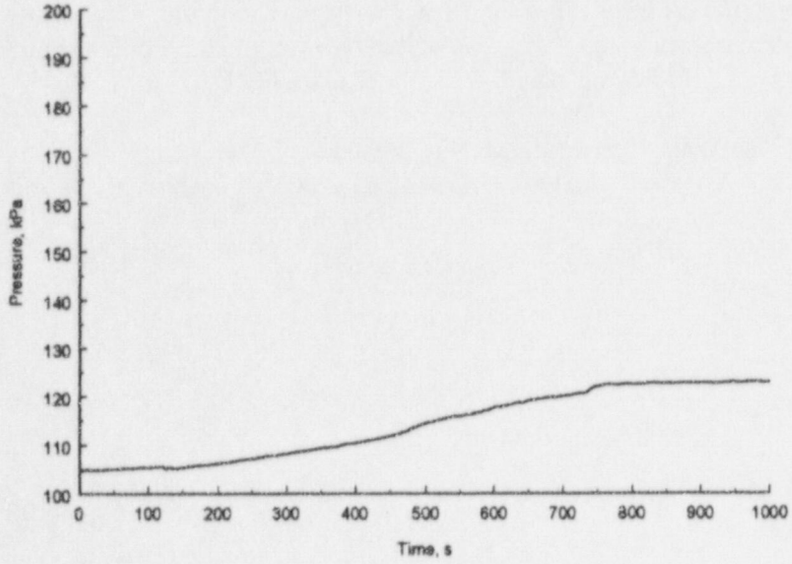


Figure D.97. ZRSS-7 Containment Pressure.

18 ZRSS-8

ZRSS-8 was run on June 11, 1997. This experiment was a triggered drop of 1200 g of stainless steel melt into room-temperature water. There was an explosion.

18.1 Operation

A load of 1200 g of stainless steel (3 mm shot, 304) was put in four polyethylene bags and placed in a graphite dropping crucible. The crucible and valve were coated with two coats of yttrium oxide aerosol spray coating baked for 10 minutes at 873 K after each coat.

The vessel was evacuated and backfilled with argon. The water pump was started and the test section filled with water as soon as the crucible had heated to a temperature where the self-luminosity was enough to allow a final check of the pyrometer alignment. Heating was started at 117 A and held there until the crucible temperature approached 2023 K when the power was turned down to 98 A. The crucible temperature slowly increased from about 2053 K to 2133 K during a five minute period. At the end of this period, the power was increased to 104 A and the crucible temperature began to climb. As it approached 2153 K (see the pyrometer record shown in Fig. D.98), the dump mechanism was actuated. Melt fell from the crucible into the test section and an explosion ensued.

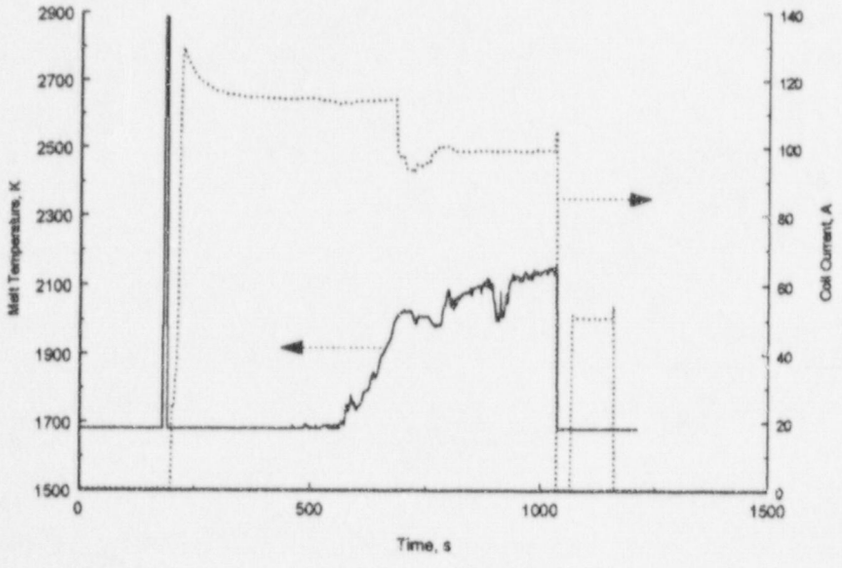


Figure D.98. ZRSS-8 Melt Heating Record.

Post explosion exhaust through the sample bottles was very slow. Three hours of flow time was allowed to ensure an adequate gas sample.

18.2 Observations

The three burn wires opened at an interval of 0.10 s between the 5 cm wire and the 30 cm wire. The quasi-static containment vessel pressure jumped from 125 to 354 kPa (see Fig. D.99), while containment vessel

temperatures reached 361 and 360 K for the top and bottom thermocouples. The test section water temperature thermocouple indicated 297 K at the time of the explosion. The video showed a uniform drop of the melt in the center of the test section. Signals from the force link were at the negative bus.

Post test examination of the apparatus showed that the damage done was similar to that of ZRSS-5, which was much less damage than was normal in an explosion test with Zr or a Zr/ZrO_2 mixture. The mechanism hanging from the vessel lid was bent but not destroyed. The crucible was intact. However, there was considerable attack on the walls of the crucible. About 28 cc of graphite were gone. There was no discernable attack on the bottom of the crucible. The valve stem and top of the valve plug were also severely eroded. The induction coil, and crucible support were bent. The bottom of the vessel was filled with debris and coated with mud made up of water from the experiment and grey dust along with small pieces of apparatus (burnwire, lamp, and connector fragments, etc.) and fibrous particles from the insulation that had surrounded the crucible and the test section heaters. The debris appeared to be made up of globules of metal ranging from cm to sub-mm size. About 60 g of fragments that appeared to be solidified melt were found on the vessel floor, and an additional 10 g as small droplets in the catch cup. An orange-brown glaze covered part of the crucible lid.

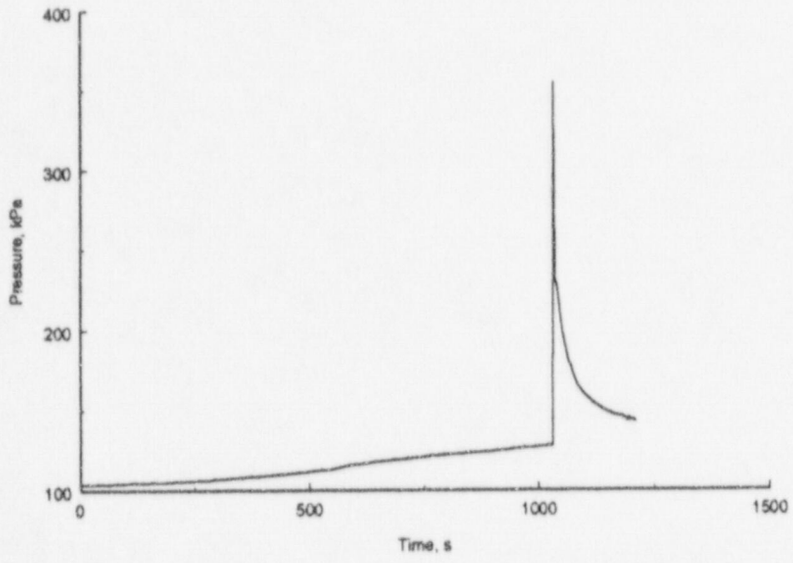


Figure D.99. ZRSS-8 Containment Pressure.

The slow exhaust rate was due to a damaged solenoid valve. The seat material, a nitrile elastomer, appeared to have melted and charred. The failure of the force link was due to a drifting charge amplifier that drifted to the negative bus over a several hour period.

The force link failed. P-2 and P-3 at 72 and 61 cm below the surface, showed similar pressures, peaking at 30 MPa with a width of about 1/2 ms (see Figs. D.100 and D.101). The other test section transducers P-5, P-6 and P-7, showed similar records, but with reduced peaks and broader widths (see Figs. D.102, D.103, and D.104). They all had initial narrow spikes, probably from the trigger explosion, followed by the explosion signal. The dynamic pressure (gas shock) at the wall of the containment peaked at 25 kPa but was very noisy

(see Fig. D.105).

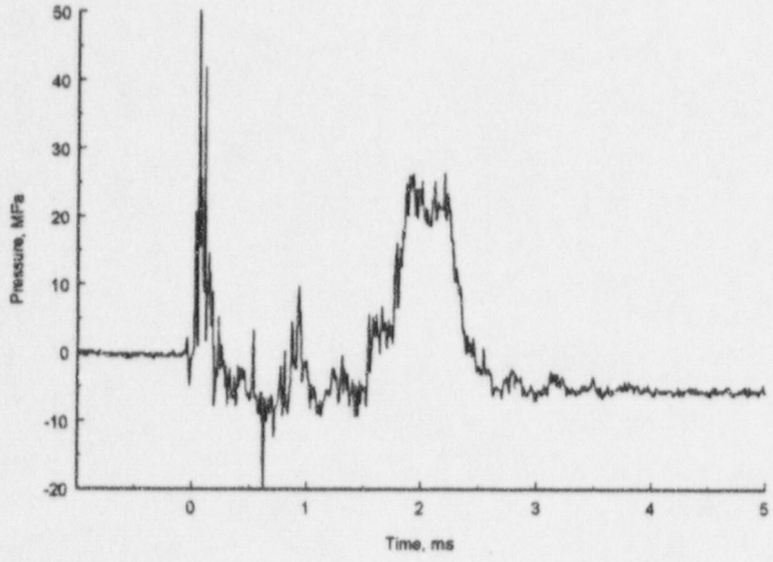


Figure D.100. ZRSS-8 P-2 Pressure Record.

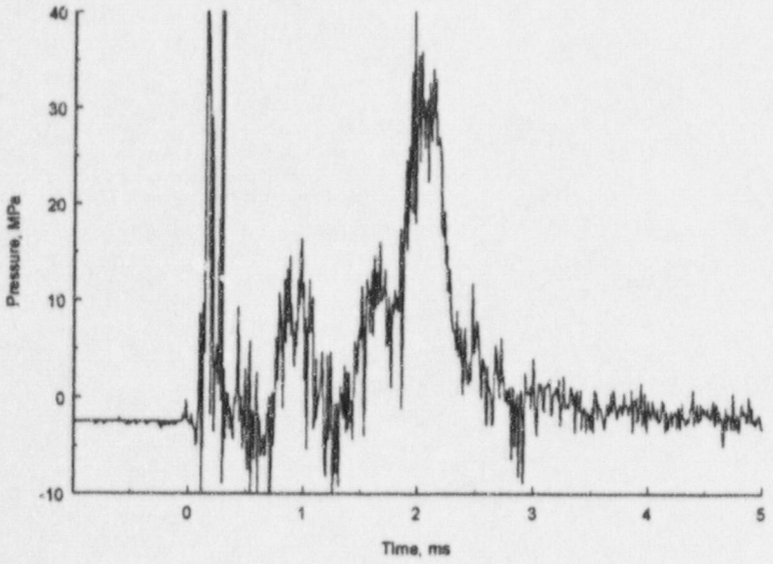


Figure D.101. ZRSS-8 P-3 Pressure Record.

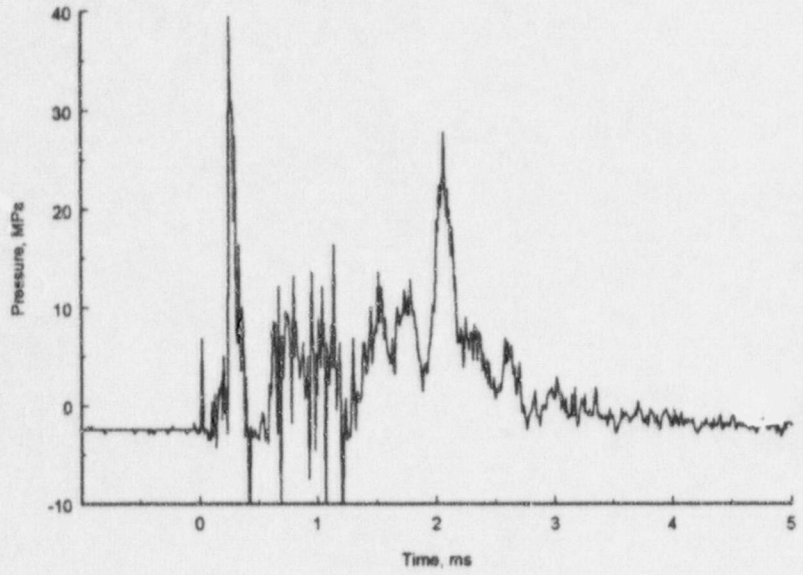


Figure D.102. ZRSS-8 P-5 Pressure Record.

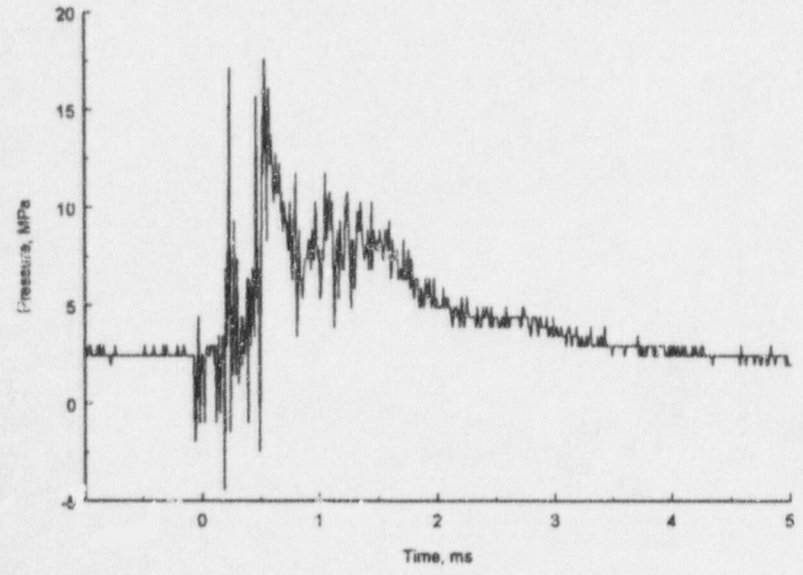


Figure D.103. ZRSS-8 P-6 Pressure Record.

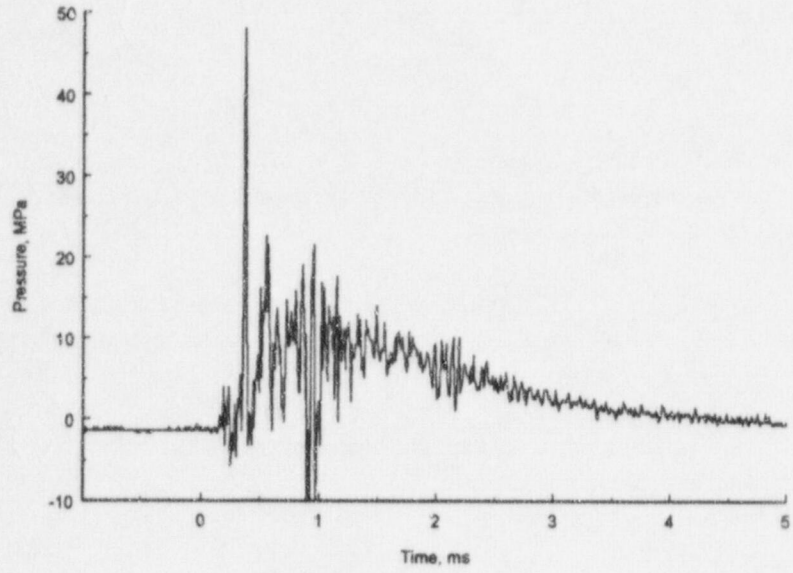


Figure D.104. ZRSS-8 P-7 Pressure Record.

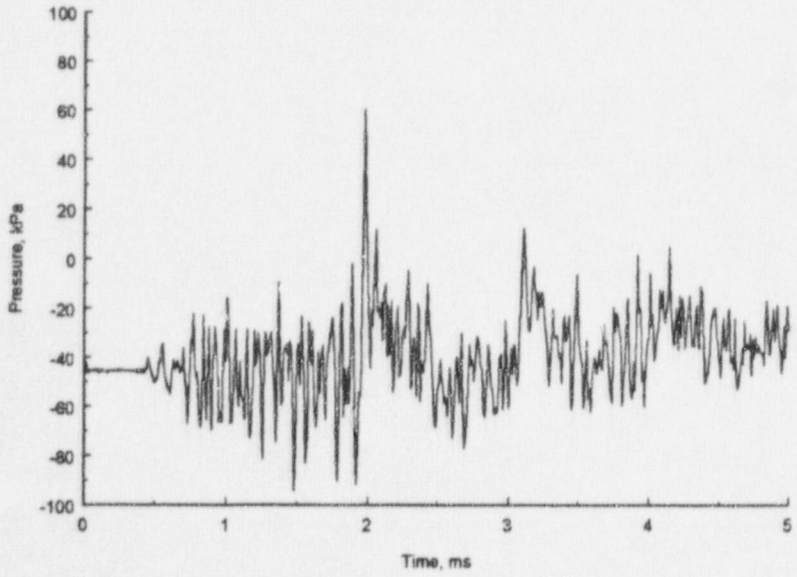


Figure D.105. ZRSS-8 Fast Response Containment Pressure.

19 ZRSS-9

ZRSS-9 was run on June 19, 1997. This experiment was a triggered drop of 1200 g of stainless steel melt into room-temperature water (a repeat of ZRSS-8). There was an explosion.

19.1 Operation

A load of 1200 g of stainless steel (3 mm shot, 304) was put in four polyethylene bags and placed in a graphite dropping crucible. The crucible and valve were coated with two coats of yttrium oxide aerosol spray coating baked for 10 minutes at 873 K after each coat.

The vessel was evacuated and backfilled with argon. The water pump was started and the test section filled with water as soon as the crucible had heated to a temperature where the self-luminosity was enough to allow a final check of the pyrometer alignment Heating was started at 118 A and held there for the duration of the run. The crucible temperature slowly increased to 2123 K when it began to fluctuate about ±50 K (see the pyrometer record shown in Fig. D.106). One minute later the dump mechanism was actuated. Melt fell from the crucible into the test section and an explosion ensued.

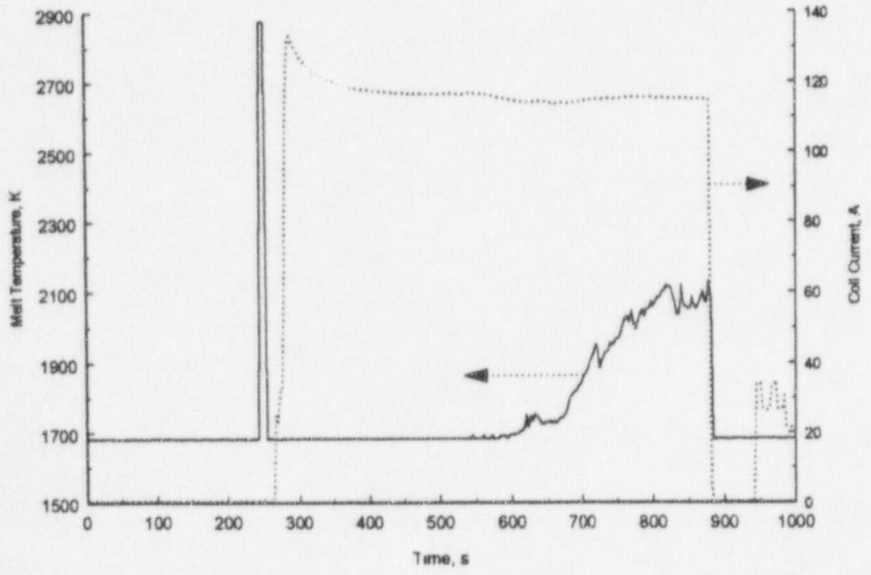


Figure D.106. ZRSS-9 Melt Heating Record.

19.2 Observations

The three burn wires opened at an interval of 0.11 s between the 5 cm wire and the 30 and 31 cm wires. The quasi-static containment vessel pressure jumped from 125 to 210 kPa (see Fig. D.107), while containment vessel temperatures reached 398 and 348 K for the top and bottom thermocouples. The test section water temperature thermocouple indicated 300 K at the time of the explosion. The video showed a uniform drop of the melt in the center of the test section.

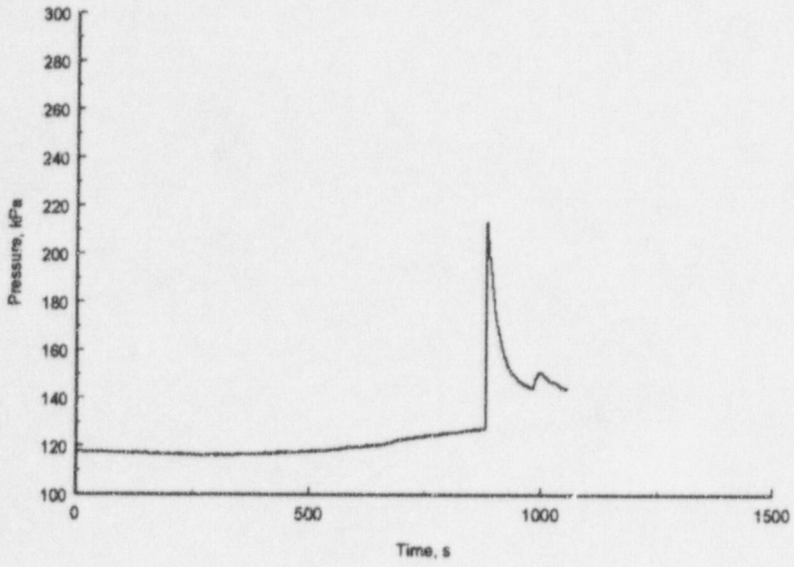


Figure D.107. ZRSS-9 Containment Pressure.

Post test examination of the apparatus showed that the damage done was very similar to that of ZRSS-8. The debris appeared to be similar to ZRSS-8, also.

The force link failed again. P-2 and P-3 at 76 and 61 cm below the surface, showed similar pressures with double peaks, the first, a narrow one peaking at 15 MPa with a width of about 1/4 ms (see Fig. D.108 and D.109). This was followed by a second peak about 1 ms later peaking at about 25 MPa, with a width of about 1/2 ms. Two other test section transducers P-6 and P-7, at 30 cm deep, did not show double peaks (see Figs. D.110 and D.111). They both showed a single broad peak corresponding to the first peak of the other transducers. This may have been due to a second, deep explosion that was insulated from transducers P-6 and P-7 by gas generated by the first explosion. The 46 cm deep transducer, P-5, failed. The dynamic pressure (gas shock) at the wall of the containment peaked at 30 kPa but was very noisy (see Fig. D.112).

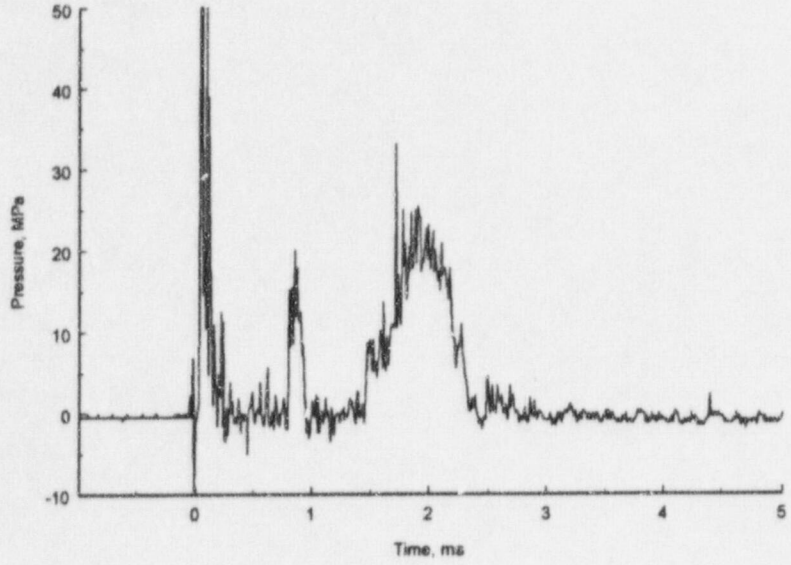


Figure D.108. ZRSS-9 P-2 Pressure Record.

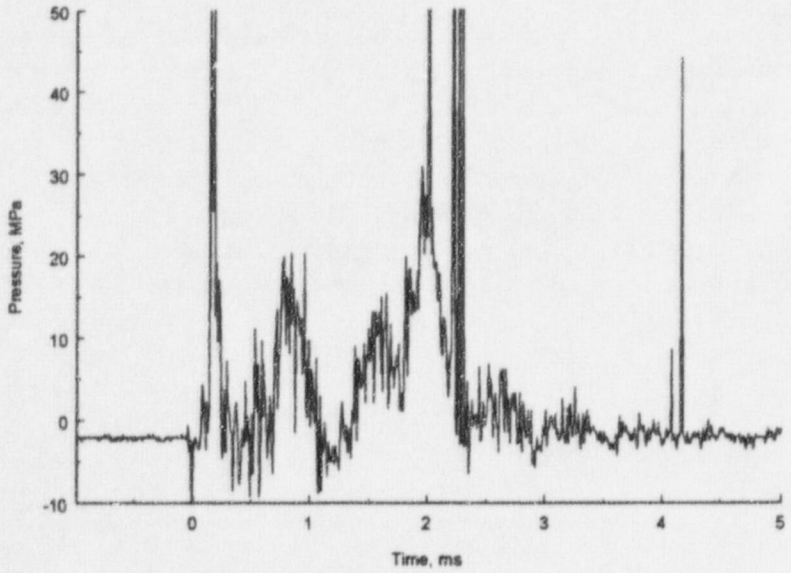


Figure D.109. ZRSS-9 P-3 Pressure Record.

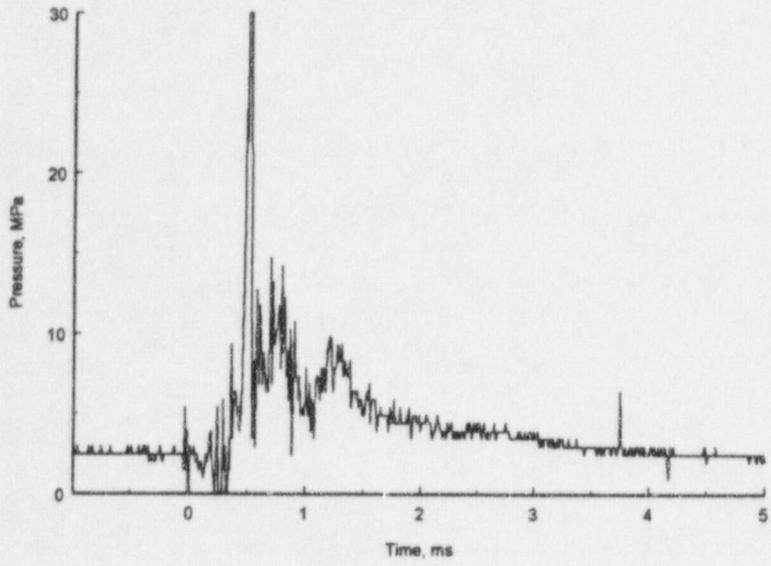


Figure D.110. ZRSS-9 P-6 Pressure Record.

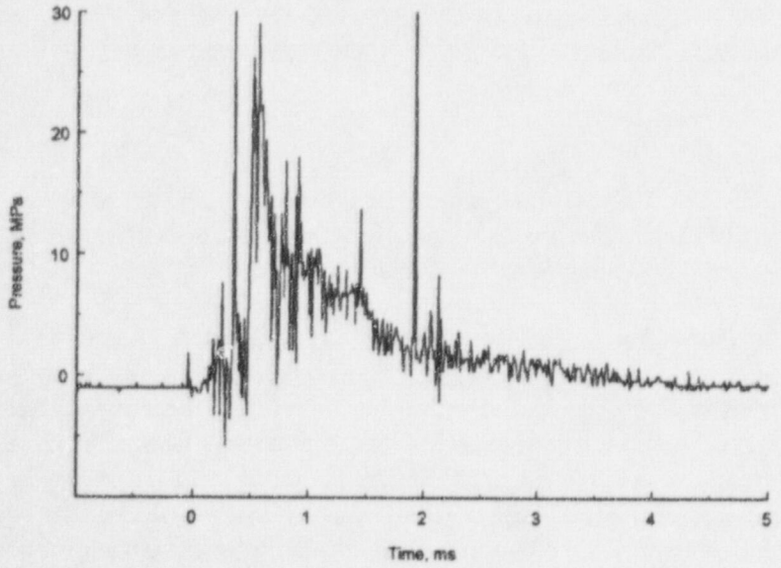


Figure D.111. ZRSS-9 P-7 Pressure Record.

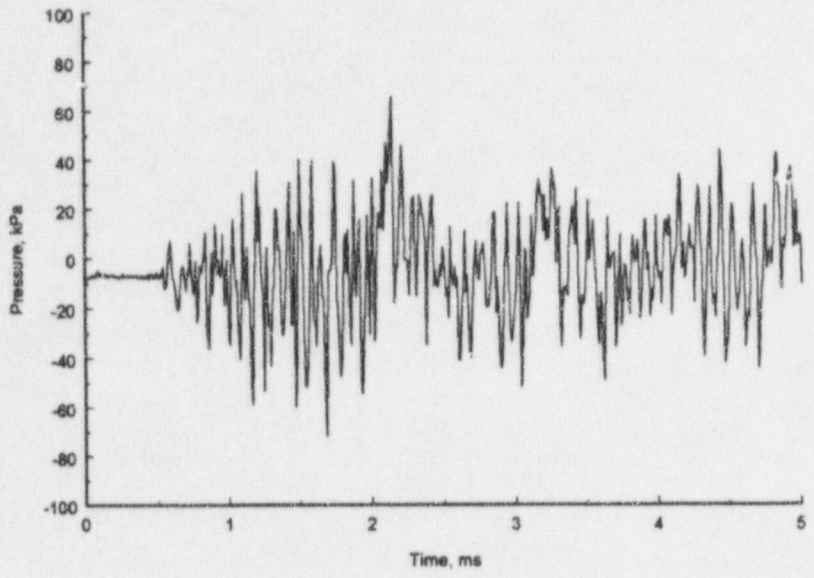


Figure D.112. ZRSS-9 Fast Response Containment Pressure.

20 ZRSS-10

ZRSS-10 was run on August 12, 1997. This experiment was a triggered drop of 1200 g of 40 w/o stainless steel - 60 w/o zirconium mixture melt into room-temperature water. There was an explosion.

20.1 Operation

A load of 480 g of stainless steel (3 mm shot, 304) and 720 g zirconium powder was put in four polyethylene bags and placed in a graphite dropping crucible. The crucible and valve were coated with two coats of yttrium oxide aerosol spray coating baked for 10 minutes at 873 K after each coat.

The vessel was evacuated and backfilled with argon. The water pump was started and ~2 liters of water was added to the test section as soon as the crucible had heated to a temperature where the self-luminosity was sufficient to allow a final check of the pyrometer alignment. Heating was started at 116 A and held there until the melt temperature reached 2073 K , when the power was lowered in an attempt to hold the melt temperature at 2123 K. The temperature overshot, reaching 2203 K, then dropping to 2123 K when the power was increased to 83 A. The temperature slowly began to climb. When it reached 2133 K, the water pump was started and the test section filled with water. About one minute later, when the temperature reached 2173 K (see the pyrometer record shown in Fig. D.113), the dump mechanism was actuated. Melt fell from the crucible into the test section and an explosion ensued. The melt was above 2123 K for about 400 seconds.

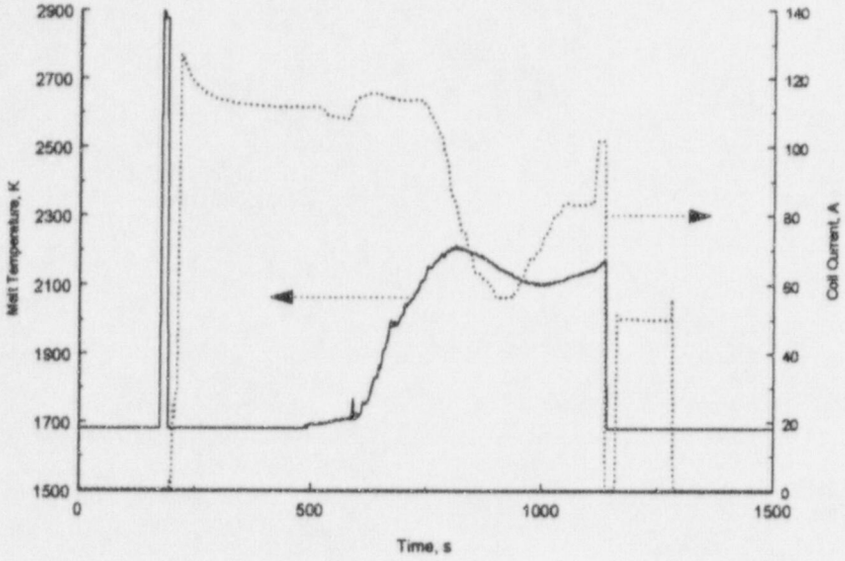


Figure D.113. ZRSS-10 Melt Heating Record.

20.2 Observations

The three burn wires opened at an interval of 0.20 s between the 5 cm wire and the 30 and 31 cm wires. The quasi-static vessel pressure jumped from 112 to 472 kPa (see Fig. D.114), while vessel temperatures reached 407 and 395 K for the top and bottom thermocouples. The test section water temperature thermocouple indicated 301 K at the time of the explosion. The video showed a uniform drop of the melt in the center of the test section.

Post test examination of the apparatus showed the damage done was similar to that of the more vigorous ZREX explosions such as ZREX-50. The entire mechanism hanging from the vessel lid was destroyed. The crucible was shattered. The induction coil, crucible support, valve opening gear, circulation fan, and sight tube were bent or torn. The bottom of the vessel was filled with debris and coated with a mud made up of water from the experiment and dark grey dust (the dust was of a darker hue than usual in ZREX explosions) along with small pieces of apparatus and fibrous particles from the insulation that had surrounded the crucible and the test section heaters.

P-2 and P-3 at 76 and 61 cm below the surface, were both very noisy. The three remaining transducers all showed an initial large, short duration trigger pulse whose delay from zero time correlated well with the distance of the transducer from the trigger source. P-5 showed a double peak, the first, a narrow one peaking at 50 MPa with a width of about 1/4 ms. This was followed by a second peak about 3/4 ms later peaking at about 40 MPa, with a width of about 1/2 ms (see Fig. D.115). Two other test section transducers P-6 and P-7, at 30 cm deep, also showed double peaks (see Figs. D.116 and D.117). They both showed an initial sharp peak followed by a broad peak. Forces measured by the force transducer peaked at about 500 kN (see Fig. D.118). The dynamic pressure (gas shock) at the wall of the containment peaked at 700 kPa then decayed below the initial zero (this may have been a thermal effect as it happens often) (see Fig. D.119).

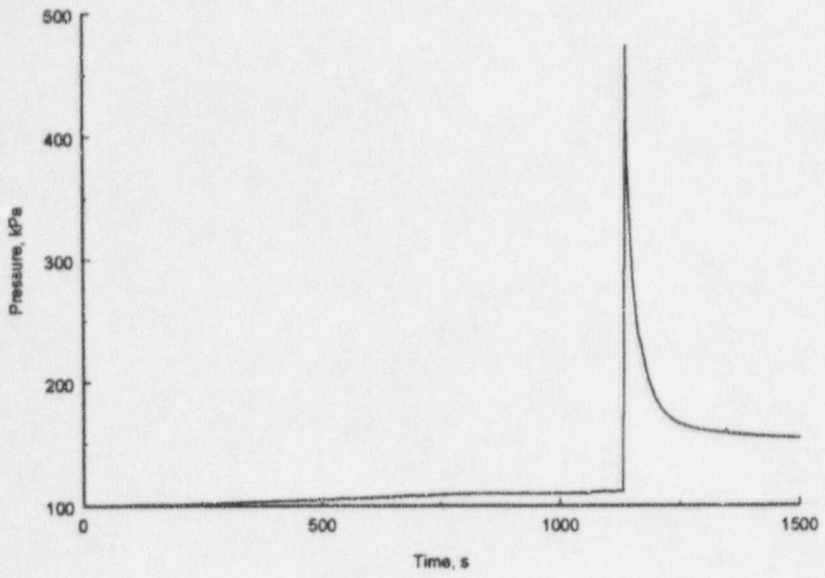


Figure D.114. ZRSS-10 Containment Pressure.

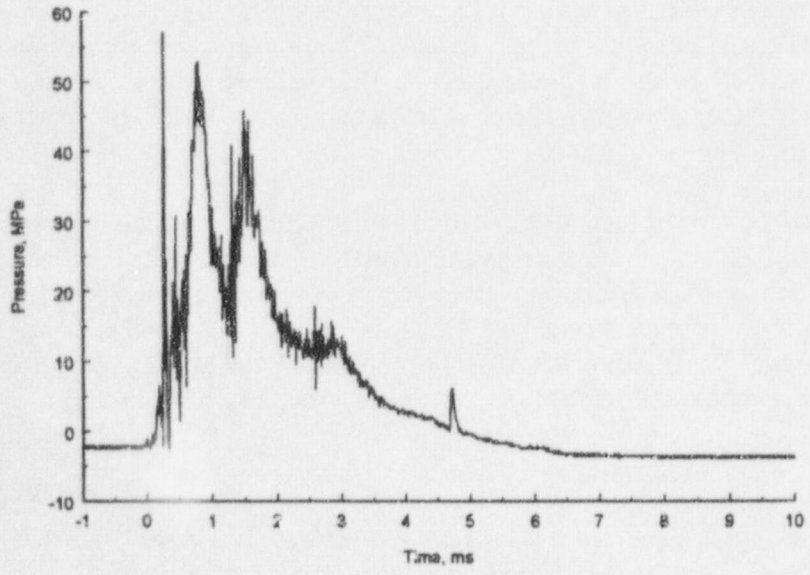


Figure D.115. ZRSS-10 P-5 Pressure Record.

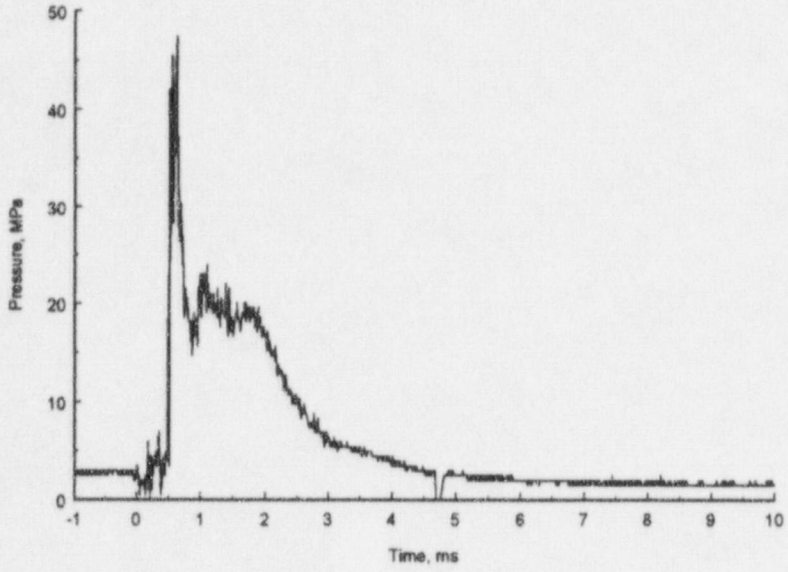


Figure D.116. ZRSS-10 P-6 Pressure Record.

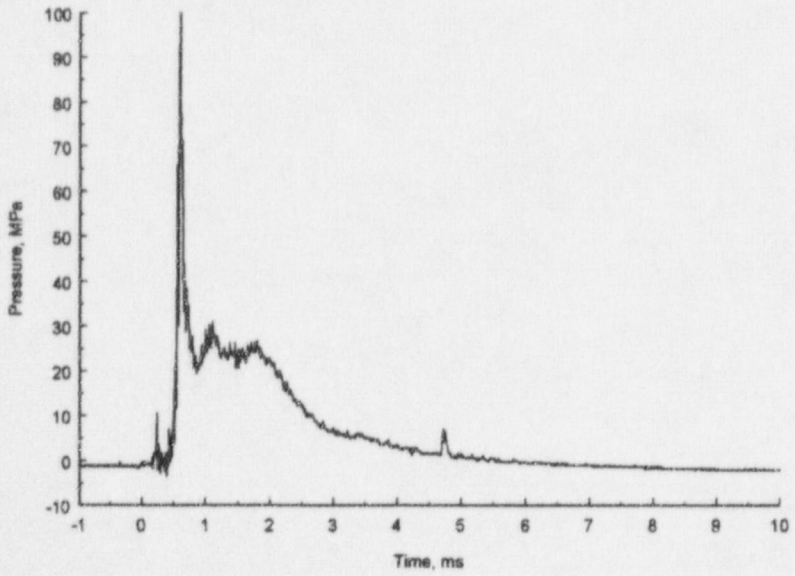


Figure D.117. ZRSS-10 P-7 Pressure Record

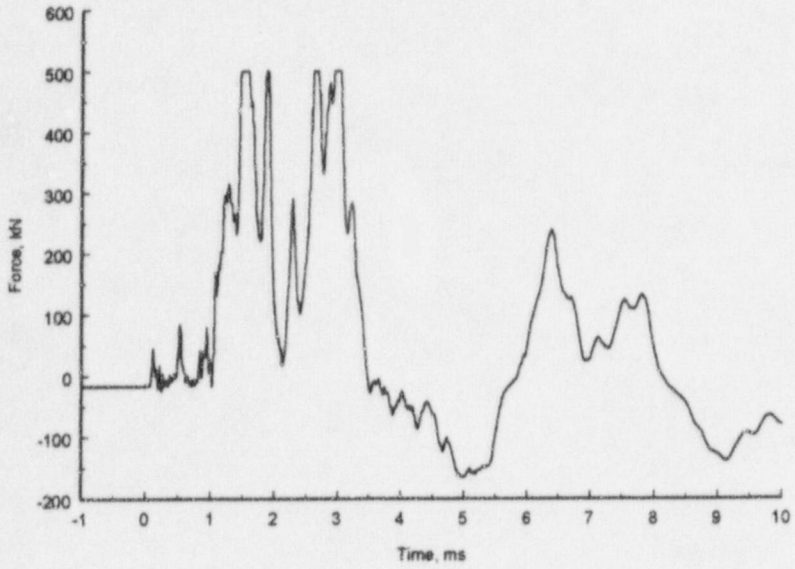


Figure D.118. ZRSS-10 Force Record.

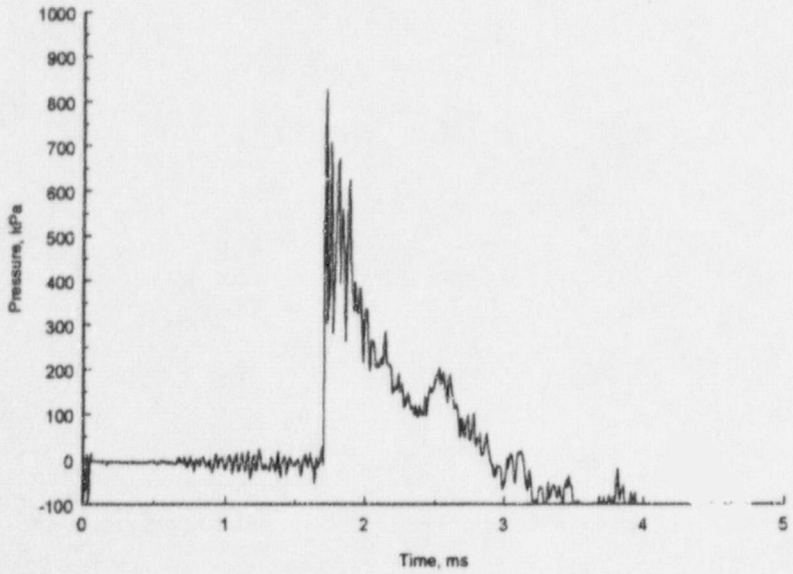


Figure D.119. ZRSS-10 Fast Response Containment Pressure.

21 ZRSS-11

ZRSS-11 was run on August 27, 1997. This experiment was a triggered drop of 1200 g of 20 w/o stainless steel - 80 w/o zirconium mixture melt into room-temperature water. There was an explosion.

21.1 Operation

A load of 240 g of stainless steel (3 mm shot, 304) and 960 g zirconium powder was put in four polyethylene bags and placed in a graphite dropping crucible. The crucible and valve were coated with two layers of yttrium oxide aerosol spray coating baked for 10 minutes at 873 K after each coat.

The vessel was evacuated and backfilled with argon. The water pump was started and all of the water was added to the test section as soon as the crucible had heated to a temperature where the self-luminosity was sufficient to allow a final check of the pyrometer alignment. The pyrometer view appeared to show melting at about 1773 K. From that point on, the surface appeared to be molten. Heating was started at 120 A and held there until the melt temperature reached 2023 K , when the power was lowered to 90 A in an attempt to hold the melt temperature at 2123 K. The temperature stabilized at 2053 K. Power was raised to 100 A without a significant change in the temperature. Power was raised again to 120 A causing the temperature to increase to 2123 K. The temperature readings were erratic, varying by ±25 K (see the pyrometer record shown in Fig. D.120). Power was increased to 128 A, and after a short delay, the dump mechanism was actuated. Melt fell from the crucible into the test section and an explosion ensued. The melt had a molten appearance for about 300 seconds.

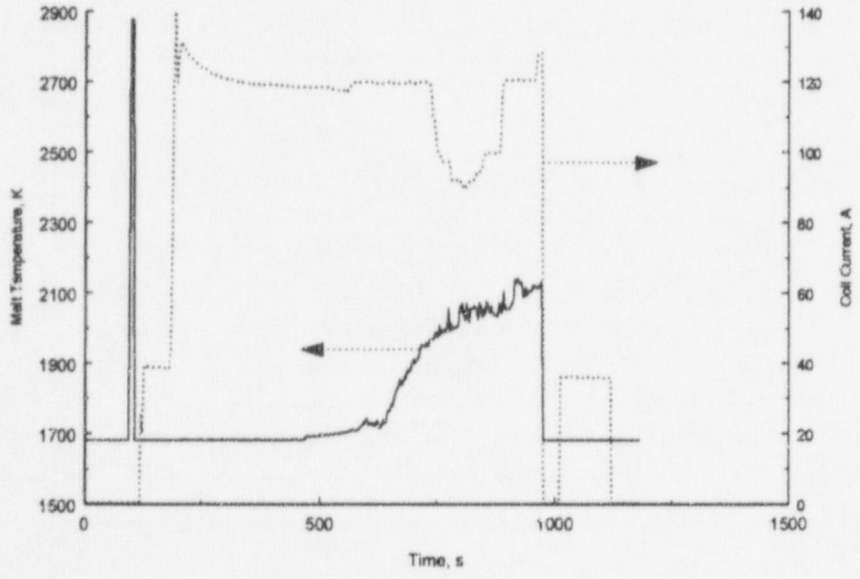


Figure D.120. ZRSS-11 Melt Heating Record.

There was a power failure on one circuit immediately after the explosion. The containment vessel static pressure transducer and drop view camera stopped operating as a result. A single pressure data point and a single TV frame were recorded after the explosion.

21.2 Observations

The three burn wires opened at an interval of 0.06 s between the 5 cm wire and the 30 and 31 cm wires. This is a shorter time than that of a drop free falling in air, thus the wires were probably melted by separate drops. The quasi-static containment vessel pressure peaked at 451 kPa (this value is suspect because of the recording problem mentioned above) (see Fig. D.121), while containment vessel temperatures reached 847 and 462 K for the top and bottom thermocouples. The test section water temperature thermocouple indicated 304 K at the time of the explosion. The video showed a uniform drop of the melt in the center of the test section.

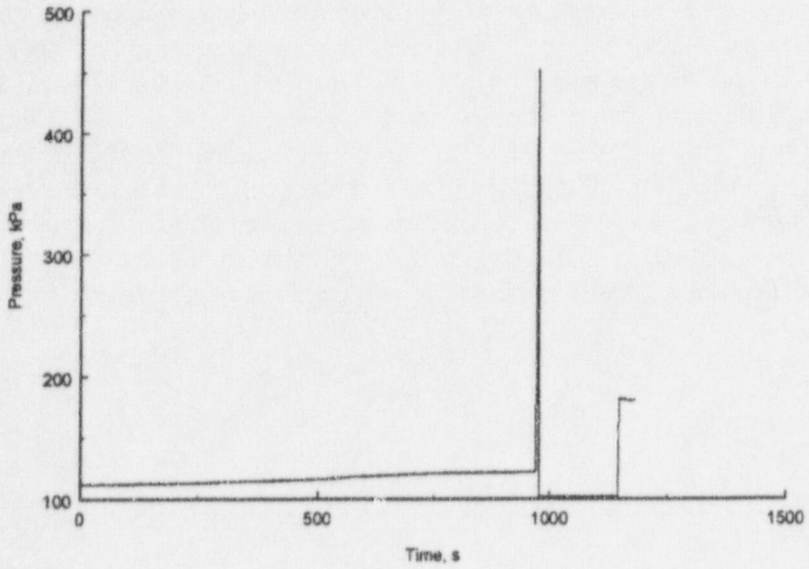


Figure D.121. ZRSS-11 Containment Pressure.

The damage done to the apparatus was very similar to that of ZRSS-10. P-2 and P-3 at 76 and 61 cm below the surface, were both very noisy. P-5 showed a single peak of about 75 MPa with a half-peak width of about one ms (see Fig. D.122). Two other test section transducers P-6 and P-7, at 30 cm deep, also showed single peaks of about 8 MPa for P-6 (see Fig. D.123) and 70 MPa for P-7 (see Fig. D.124). The 8 MPa peak for P-6 is suspiciously low compared to P-7 and P-5, and appears to involve a calibration error. Forces measured by the force transducer over ranged at 500 kN (see Fig. D.125). The dynamic pressure (gas shock) at the wall of the containment peaked at 900 kPa then decayed below the initial zero (the decay may have been a thermal effect again)(see Fig. D.126).

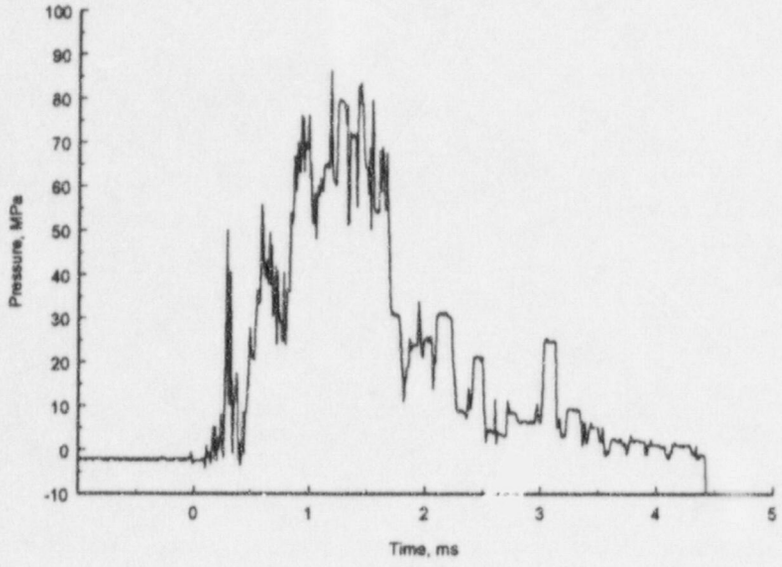


Figure D.122. ZRSS-11 P-5 Pressure Record.

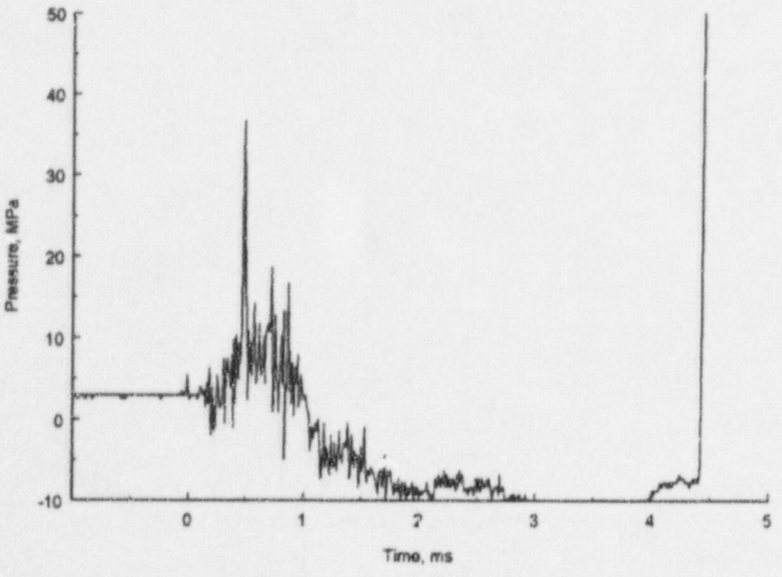


Figure D.123. ZRSS-11 P-6 Pressure Record.

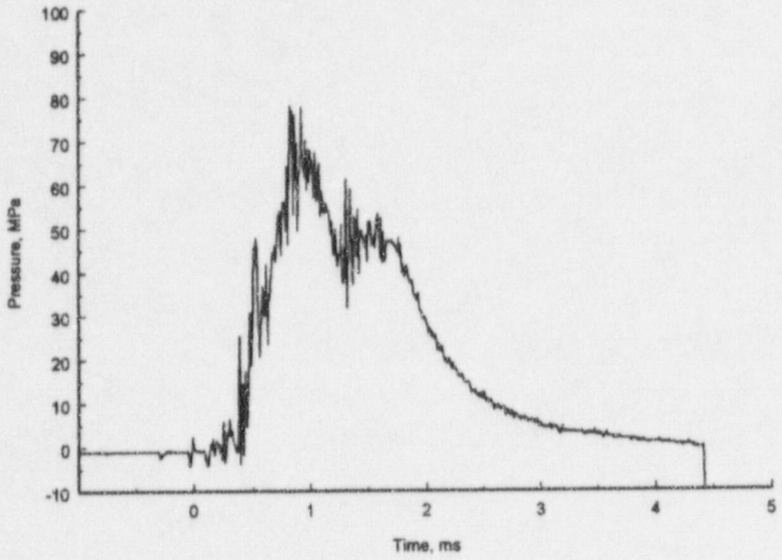


Figure D.124. ZRSS-11 P-7 Pressure Record.

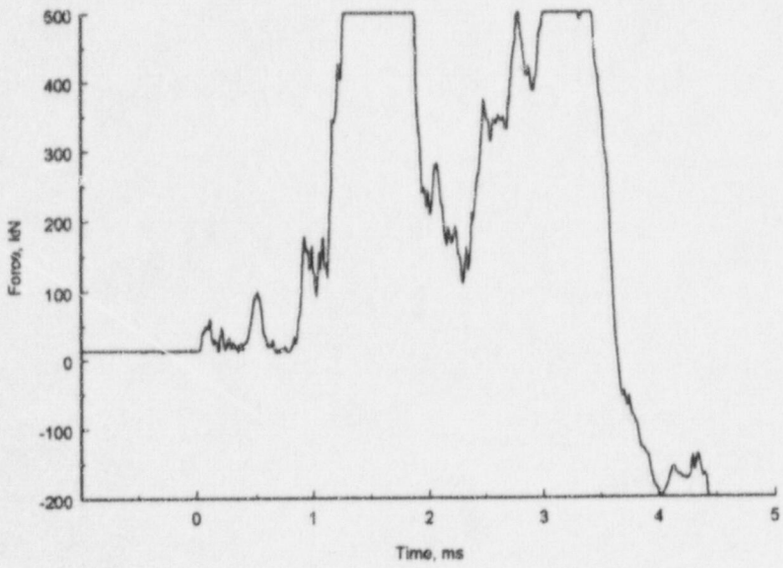


Figure D.125. ZRSS-11 Force Record.

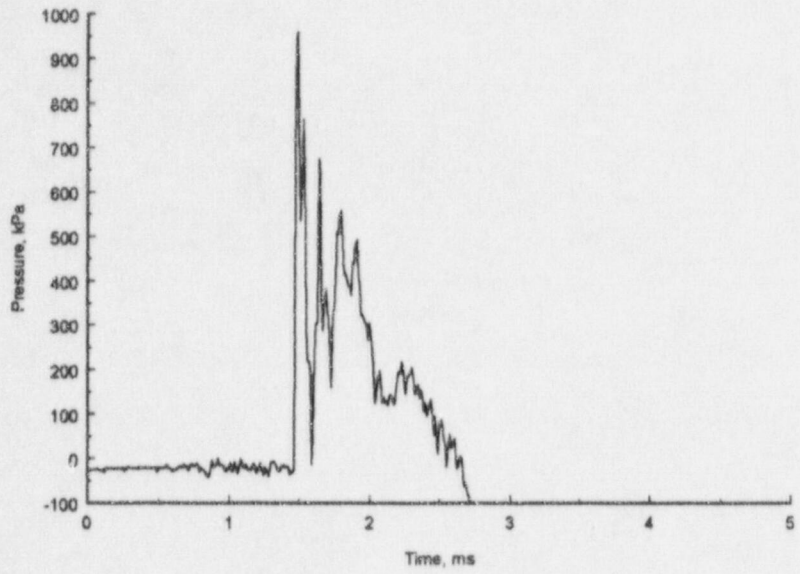


Figure D.126. ZRSS-11 Fast Response Containment Record.

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S. Basu, NRC Project Manager

11. ABSTRACT *(200 words or less)*

The results of two series of experiments on explosive interactions between zirconium-containing melt and water are described. The first series of experiments involved dropping 1 kg batches of zirconium/zirconium dioxide (Zr/ZrO2) mixture melt into a column of water while the second series employed 1.2 kg batches of zirconium/stainless steel (Zr/SS) mixture melt. A total of 14 successful tests (nine triggered and five untriggered) were conducted with Zr/ZrO2 mixtures and a total of 8 tests (five triggered and three untriggered) with Zr/SS mixtures. Explosions took place only in those tests which were externally triggered. The test results indicate that the explosion energetics were augmented by the zirconium-water reaction, the extent of augmentation being dependent on the zirconium content in the melt. The explosion energies for tests involving high zirconium contents were mostly in the range of 2-3% of the combined thermal and chemical energy available, while the energy conversion ratios for Zr/SS mixtures of low zirconium content were considerably lower.

12. KEY WORDS/DESCRIPTORS *(List words or phrases that will assist researchers in locating the report.)*

fuel-coolant interactions (FCI), steam explosions, vapor explosions, combined chemical-steam explosions, chemical augmentation, zirconium-containing melt, zirconium-zirconium dioxide mixture, zirconium-stainless steel mixture, zirconium-water reaction, hydrogen production, severe accident, nuclear reactor safety

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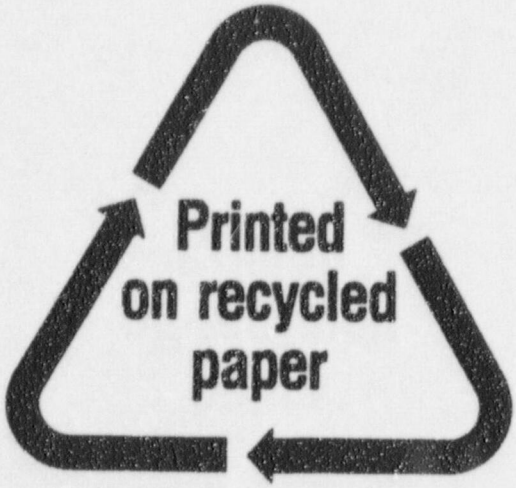

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