# A CELL FOR INVESTIGATION OF THE LEIDENFROST PHENOMENON AT PRESSURES ABOVE ATMOSPHERIC

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By

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May, 1966

Submitted to the Faculty of the Graduate College of the Oklahoma State University in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE July, 1967

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# Thesis Approved:

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#### ACKNOWLEDGMENT

I am indebted to Dr. Kenneth J. Bell for serving as research adviser for this work. Together with the other members of the faculty, his guidance has made this project possible. The suggestions received from my fellow graduate students are gratefully acknowledged. I also wish to thank my wife, Rita, for her help and patience.

Financial support for my project has been appreciatively received from the United States Army Research Office, Durham, North Carolina.

Also, I wish to thank the staff of the Research and Development Laboratory for their advice on and construction of my test cell.

# TABLE OF CONTENTS

Chapter																					Р	age
I. IN	IRODUCI	CION.	•••	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	1
II. LI	TERATUR	E SURV	/EY .	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	2
III. DE	SCRIPTI	ON OF	EQUI	PME	NT	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	4
IV. EX	PERIMEN	TAL PR	ROCED	URE	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	12
V. DI	SCUSSIO	N OF F	RESUL	TS	•	•	•	•	•	•	•	•	•	•	•	•	•		•	•	•	13
VI. CO	NCLUSIO	NS ANI	REC	OMM	ENI	DA'	TI	ON	S	•	•	•	•	•	•	•	•	•	•	•	•	15
A SELECT	ED BIBL	IOGRAF	РНҮ.	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	16
APPENDIX	A - CA	LIBRAI	TONS	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	17
APPENDIX	B – DA	TA	• •	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	22

### LIST OF FIGURES

Figu	re	Page
1.	Schematic Drawing of Test System	5
2.	Test Cell	6
3.	Detailed Views of Test Cell	7
4.	Cooling System for Sample Line	9
5.	Droplet Lifetimes Vs. Temperature Difference	14

### CHAPTER I

#### INTRODUCTION

The first scientific observation and investigation of film boiling of discontinuous liquid masses was conducted by a German doctor, J. G. Leidenfrost, in 1756. Leidenfrost first noticed the phenomenon with droplets of water on a hot smooth iron spoon. He noted that the droplets were spherical in shape and took a relatively long time to evaporate. The evaporation of droplets in stable film boiling is termed the Leidenfrost Phenomenon, in his honor.

The temperature of the wall at which the minimum heat flux on the pool boiling curve occurs is called the Leidenfrost Point, also in his honor.

The object of this work was to design and construct a test cell to investigate the effect of pressure on the Leidenfrost Phenomenon for various liquids. The cell was designed to operate up to 1000 psia, which is above the critical pressure of most liquids, and simultaneously at a maximum temperature of 500°C. The entire system was tested with benzene at 1.0 and 4.4 atmospheres.

### CHAPTER II

#### LITERATURE SURVEY

Much work has been done in the area of stable film boiling of droplets since the German doctor, J. G. Leidenfrost, first recorded his observations. The entirety of this work has been at atmospheric pressure (2,3,4,5,10). However, there is in the literature some work on film pool boiling under pressure, notably Farber and Scorah (3) and Kovalev (7).

Farber and Scorah studied the pool boiling of water from a series of wire surfaces, these being nickel, tungsten, chromel A, and chromel C, over a pressure from 0 to 100 psig. Their results generally indicate that the Leidenfrost Point decreases with increasing pressure, over the range of pressures and surfaces covered. They also noted that the surface material made a large difference on the maximum and minimum fluxes.

Kovalev investigated the minimum heat fluxes in the pool boiling of water. The author made his study on polished nichrome wires 2.0-2.5 mm in diameter. The point of interest is the large pressure range of the data, 1.0-100 atmospheres. The correlation for the minimum heat flux  $(q_{min})$  for the range of pressures studied is as follows:

 $q_{min} = 18.5 \times 10^3 \times p^{0.48} \text{ kcal/m}^2 \text{hr.}$ where p is the pressure in atmospheres.

Hoffman and Gauvin (6) studied the rate of evaporation of droplets of water, methanol, benzene, pentane, and cumene from a filament 70 microns in diameter. The work was carried out in an electrically heated stainless steel sphere up to a pressure of 10 atmospheres. The atmosphere inside the sphere was in all cases the vapor of the particular liquid under study. The droplets were in the range of 0.4 to 1.4 mm in diameter and atmosphere temperatures from 100°C to 550°C.

The authors found no correlation between natural convection and the evaporation rate, since the Nusselt number had no dependence on the Grashof number. The evaporation rates were correlated successfully by the Spalding Transfer Number B.

Adadevoh (1) investigated the lifetime of droplets evaporating on a hot plate under pressure. The liquids studied were benzene, iso-octane, and n-cetane. The author was primarily interested in the temperature at which the minimum lifetime of a droplet occurred as an optimum operating temperature for an internal combustion engine.

Adadevoh made his study over a range up to 800°F, 100 psia, and a droplet diameter of approximately 2 mm. Even though in the upper range of temperatures film boiling should occur with a consequent characteristic maximum in the lifetime curve, none was observed by the author. Consequently, no information is available about the Leidenfrost Point and the effect of pressure on it.

#### CHAPTER III

### DESCRIPTION OF EQUIPMENT

A high pressure test system was designed and constructed and is shown schematically in Figure 1. The entire system may be subdivided into three subsystems: the inert gas pressurizing system, the pressure and temperature measuring system, and the sample injection system. These systems operated as auxiliaries to the test cell. The test cell was designed for conditions of 1000 psia and 500°C.

The main body and split ring of the test cell were machined from 316 stainless steel forgings. The test cell lid was fabricated from a piece of flame cut 410 stainless steel plate. Detailed drawings of these parts are presented in Figure 2.

A depression two inches in diameter with a slope of one degree was machined on the inside bottom of the main body to keep the droplet in sight. Five thermocouple wells were drilled to within one sixty-fourth of an inch from the inner surface of the bottom of the cell. Slots were milled from the thermowells to one side of the cell as seen in Figure 3. Holes were drilled and tapped in the side of the cell for the nitrogen inlet and outlet, sample line cooling jacket, and gas thermocouple. An O-ring groove was machined at the top of the main chamber to seal the lid. The O-ring was made of Parker Seal Company type 77-545 Viton-A



Figure 1. Schematic Drawing of Test System



Figure 2. Test Cell



Figure 3. Detailed Views of Test Cell

as were the O-rings in the lid. The main body was originally plated with chrome, but the chrome had to be removed in the area of the depression due to flaking.

The lid serves to hold the sight glass through which the evaporating droplets were observed. The sight glass was a fused quartz disc obtained from General Electric. The glass was pitch polished and then etched in a twenty percent solution of hydrofluoric acid. The disc was sealed in the lid by means of two Viton-A O-rings as seen in Figure 2. A steel ring was used to center the disc and to prevent the retaining nut from being screwed onto the surface of the quartz.

The split retaining ring was used to hold the lid to the main chamber. However, the O-ring must be compressed until the two halves will slide over the retaining lips. This assured a tight seal between the lid and the main chamber as long as there was no compression set in the O-rings.

In order to prevent the sample from vaporizing as it passed through the wall of the cell the sample line, which runs from the top of the samble bomb to the test cell, was incorporated in a double pipe heat exchanger. Swagelock tees and male connectors were used as connections for the jacket of the exchanger. A drawing of the exchanger may be found in Figure 4. The end of the sample line was tapered to accept standard hypodermic needles. To control the flow rate through the sample line so that a droplet would form slowly, a Whitey model 22RS4 micrometer handle needle valve was used. The valve was able to regulate the flow very precisely due to its small orifice diameter, 0.02 inches.



Figure 4. Cooling System for Sample Line

To force the liquid through the sample line, a system as seen schematically in Figure 1 was used. The mercury slug was used to keep the nitrogen, which was at 10 psi higher pressure than the cell, from diffusing into the liquid. The volume of the sample bomb was 150cc, while that of the mercury reservoir was about 350cc.

The test cell was centered on two Hevi-Duty #54-KSS flat electric heating units. The units were controlled by a 20 ampere, 120 volt Powerstat. To insulate the bottom of the heating unit three sheets of transite, separated by air gaps, were used. The top sheet had three adjustable screws for leveling the test cell.

Dry nitrogen was used to pressurize the cell. The nitrogen pressure was regulated by a Matheson regulator and recorded on two gauges, a 1000 psi ACCO Helicoid and a 100 psi Marsh. The calibration of these gauges may be found in Appendix A.

Chromel-Alumel thermocouples were used for the temperature measurements because of their stability over the temperature range, their high voltage differential per degree, and their availability. Saureisen cement was used to hold the thermocouples in the wells and slots. The bead of the thermocouple was in contact with the metal to give a faster response time. The thermocouples were calibrated at 0°C and at 100°C and deviated less than 1°C from the thermometer readings. The calibration may be found in Appendix A. The thermocouple emf was measured by a Leeds and Northrup model 8690 potentiometer in conjunction with an ice bath as a reference junction.

To observe the droplet evaporating, a projection head from a model 15710 Porta-Scribe overhead projector was used. The projection head not only served to turn the image 90° but also magnified the image. With this aid the droplet evaporation times were measured with a Meylan number 218 stopwatch. A Tensor high intensity lamp was used to illuminate the droplets during evaporation. On the safety glass protective shield a sheet of polarized plastic was used to reduce the glare from the light reflected from the bottom surface of the test cell.

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#### CHAPTER IV

#### EXPERIMENTAL PROCEDURE

Prior to heating the cell, the surface was cleaned with jeweler's rouge, then rinsed with alcohol and wiped dry with Kimwipes. This procedure was necessary due to the small residue left after a set of data had been taken.

The mercury reservoir was pressurized to about 10 psi over the pressure at which the run was made. This pressure differential was found to give the best control of the formation of the droplet.

The test cell was next assembled, and the pressurizing nitrogen then was admitted through the inlet valve. The flow of nitrogen out of the cell was regulated by a needle valve. The Powerstat was turned on, and the cooling water was also started. After a sufficient temperature was attained, a drop was formed on the end of the needle and allowed to fall. The stopwatch was then started. After the drop evaporated, the watch was stopped; and the temperature read on the potentiometer. The time and temperature were recorded, and the same procedure with the drop repeated.

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#### CHAPTER V

#### DISCUSSION OF RESULTS

The equipment performed as designed. The only malfunction was found to be with the O-rings. Due to the high temperatures in the cell, the O-rings tended to set under the compression and hence leaked when re-used several times. The solution to this problem was to discard the bad O-ring.

The sample injection system performed quite well. The micrometer handle needle valve gave the desired control over the formation of the droplets.

The Leidenfrost Point for benzene is observed to increase with increasing pressure over the very limited pressure range tested. A plot of the data may be seen in Figure 5. Farber and Scorah report the opposite effect in the pool boiling of water; that is, the Leidenfrost Point decreases with increasing pressure. A possible explanation of this is the different ranges of reduced pressure. The range of reduced pressures studied by Farber and Scorah was from 0.00458 to 0.0367; whereas, in this work the two reduced pressures studied were 0.021 and 0.0922. Intermediate values of the reduced pressure should be studied to determine the effect of pressure on the Leidenfrost Point.



Figure 5. Droplet Lifetimes Vs. Temperature Difference

### CHAPTER VI

### CONCLUSIONS AND RECOMMENDATIONS

The following conclusions may be drawn from the results of this study:

- (1) The equipment as designed will work.
- (2) The Leidenfrost Points of benzene are 188°C and 256°C for pressures of 1.0 and 4.4 atmospheres, respectively.

The following recommendations are made in light of the previous discussion:

- Another sight glass should be installed on the side of the test cell to aid photographic work.
- (2) A pressure regulator following the present one would aid in control of the chamber pressure, especially at higher pressures.

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### CALIBRATION OF THERMOCOUPLES

Five Omega and one Conax chromel-alumel thermocouples were used for temperature measurements in the test cell. The Omega thermocouples had a diameter of .015 inch, and the Conax a diameter of 1/16 inch. Chromel-alumel was selected for its relatively high thermal emf per degree and stability over the temperature range.

The thermocouples were calibrated utilizing boiling water and an ice bath. The calibration was performed on the same potentiometer as the data, with a reference junction of melting ice.

A calibrated total immersion thermometer with a range of -l° to +lOl°C in increments of .l°C was used to check the thermocouples. A correction was made for the emergent stem as specified in the <u>Handbook of Chemistry and Physics</u>.

The thermocouples from Omega are numbered from one to five, and the Conax thermocouple is numbered 6.

# TABLE I

## CALIBRATION OF THERMOCOUPLES

Thermocouple No.	Reading mV	Temperature °C
1	0.00	0.00
2	0.00	0.00
3	0.00	0.00
4	0.00	0.00
5	0.00	0.00
6	0.00	0.00

Thermometer Reading 0.00°C + .4°C

Thermocouple No.	Reading mV	Temperature °C
1	4.01	98.00
2	4.01	98.00
3	4.01	98.00
4	4.01	98.00
5	4.01	98.00
6	4.00	97.75

Thermometer Reading 97.8°C <u>+</u> .9°C

# TABLE II

## CALIBRATION OF NEEDLE

Needle size	e: #27	Tempe	rature: 25.7°C
Pressure:	l atm.	Pressure:	4.4 atm.
Liquid Mass gm .00480	Deviation From Mean percent +3.23	Liquid Mass Do gm .00465	eviation From Mean percent 2.65
.00470	+1.08	.00447	-1.32
.00426	-8.40	.00429	-5.30
.00475	+2.15	.00471	3.97
<u>.00472</u>	+1.50	.00455	44
.00465	3.27	.00453	2.74

### TABLE III

### CALIBRATION OF PRESSURE GAUGES

A Marsh and an ACCO Helicoid gauge were used for pressure measurements in the cell. The Marsh gauge had a pressure range of 0-100 psig, and the ACCO gauge had a range of 0-1000 psig.

The two gauges were calibrated on a Budenburg Dead Weight Gauge, which has an accuracy of 0.05%.

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Helicoid Gauge

### Marsh Gauge

Dead Weight	Gauge	Dead Weight	Gauge
Tester	psig	Tester	psig
20	20	20	20
40	41	40	40
50	51	50	50
60	61	60	60
100	101	100	100
200	201		
300	300		
400	400		
500	500		
600	601		
700	701		
800	802		
900	902		
1000	1004		

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# TABLE I

## DROPLET VAPORIZATION TIMES

Run No. 1 Pressure:	14.7	psia	Date: Time:	2-28- 3:00	67 pm
Time		Potentiometer Reading	Т	empera	ture
sec.		mV		°C	
18.35		7.18		176	_
20.20		7.31		179	.5
20.00		7.45		183	_
19.65		7.51		184	<b>~</b> 5
20.50		7.64		188	
19.50		7.70		189	ł
19.80		7.80		192	
19.35		7.86		193	
19.35		7.92		195	
18.60		8.02		197	
18.25		8.10		199	)
19.60		8.21		202	_
18.65		8.27		203	.5
18.70		8.32		205	
18.85		8.38		206	
18.40		8.43		207	.5
18.00		8.48		208	.5
18.00		8.53		210	1
17.90		8.60		211	.5
19.45		7.64		187	
19.15		7.91		194	
18.55		8.13		200	1
18.15		8.48		208	
17.60		8.70		214	,
16.40		8.93		220	)
16.25		9.14		225	

.

# TABLE II

# DROPLET VAPORIZATION TIMES

Run No. 2			Date:	4-29-67
Pressure:	14.7	psia	Time:	2:00 pm
Time		Potentiometer Reading	Т	emperature
sec.		mV		°C
19.80		7.82		192
18.95		8.24		203
19.35		8.44		207.5
18.85		8,58		211
17.20		9.08		223.5
15.35		9.65		237.5
14.65		9.90		244
14.85		10.05		247.5
15.00		10.29		253
13.60		10.71		263.5
13.60		10.86		267
12.90		11.02		271
12.50		11.18		275
12,45		11.30		278
11.72		11.72		288
11.40		11.86		291.5
11.80		11.92		293
10.70		12.04		296

•

# TABLE III

## DROPLET VAPORIZATION TIMES

Run No. 3 Pressure:	64.7	nsia	Date: Time:	4-29- 8:00	-67 pm
		Potentiometer	1	rempera	ature
		Reading			
sec.		mV		°C	2
10.40		10.40		256	5
9.20		10.52		259	
9.50		10.66		262	2
9.21		10.99		270	)
9.11		11.16		274	1.5
8.35		11.31		278	3
8.60		11.44		281	<u> </u>
8.40		11.57		<b>28</b> 4	1.5
8.55		11.78		289	.5
8.45		12.04		296	5
8.40		12.34		303	5
8.25		12.45		306	5
8.09		12.50		307	,
7.90		12.69		311	. • 5
7.60		12.78		314	ŀ
7.40		13.00		319	•
6.90		13.18		323	5
7,35		13.40		328	8.5
7.35		13.46		330	)
6.80		13.58		333	5
6.90		14.00		343	5
6.55		14.11		345	5.5
6.80		14.26		349	•
6.60		14.34		351	•
6.10		14.52		355	5.5
6.40		14.55		356	5

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### Thesis: A CELL FOR INVESTIGATION OF THE LEIDENFROST PHENOMENON AT PRESSURES ABOVE ATMOSPHERIC

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