A MANUAL FOR THE PREPARATION OF **GRADUATE THESE** EIGHTH REVISED EDITION

REVISED AUGUST 30, 2017





THESIS | DISSERTATION OFFICE YOUNG HALL B-80 765.494.3231

TABLE OF CONTENTS

CHAPTER 1. UNIVERSITY FORMAT REQUIREMENTS	1
University Format Requirements	1
Paper Requirement	1
Font and Quality	1
Line Spacing	2
Margins	2
Pagination	3
Chapter Titles	3
Additional Departmental Style Requirements	4
CHAPTER 2. ORDER OF THESIS DISSERTATION PAGES	5
Preliminary Pages	5
Title Page	5
Statement of Committee Approval Page	5
Dedication Page	5
Acknowledgements Page	6
Preface	6
Table of Contents	6
List of Tables	7
List of Figures	7
List of Symbols, Abbreviations, Nomenclature, or Glossary	8
Abstract	8
Main Body Pages	9
Parts	9
Introduction	9
Body	. 10
Summary and Conclusions	. 10
Recommendations	. 10
Back Matter Pages	. 10

References	
Appendix/Appendices	11
Notes and Footnotes	11
Vita	
Publication(s)	
CHAPTER 3. GENERAL INFORMATION	13
Thesis Delay of Publication	
Thesis Confidentiality	
Foreign Language Theses	14
APPENDIX A. THE DEPOSIT PROCESS	15
APPENDIX B. ORDER OF PAGES	17
APPENDIX C. STYLE HEADINGS	
APPENDIX D. CAPTIONS FOR LARGE FIGURES	
APPENDIX E. UNIVERSITY COPYRIGHT INFORMATION	
APPENDIX F. SAMPLE PAGES	

CHAPTER 1. UNIVERSITY FORMAT REQUIREMENTS

The copy of a thesis submitted to the Thesis/Dissertation Office is called the *Deposit* copy. The *Deposit* copy of a thesis cannot be altered or edited after acceptance by the Thesis/Dissertation Office except with the express approval of the Graduate School. Accordingly, special care must be taken in producing this copy. The *Deposit* copy cannot be produced until after you have orally defended your research, made any required revisions, and have had them approved. The *Deposit* copy must conform to all University format specifications described below.

University Format Requirements

Paper Requirement

- Letter 8 $\frac{1}{2}$ x 11" paper sizing must be used for the *Deposit* copy.
- Oversize pages up to 11 x 17" are acceptable. Oversize pages should not be used unless necessary.

Font and Quality

- Times New Roman 12 point font will be the only accepted font.
- Major headings and chapter titles should be typed using 14pt bolded font.
- All text and page numbers must be in the same font. Table numbers, figure numbers, captions, references, and footnotes should be no smaller than 10pt and no greater than 12pt font. For general text, type size should neither be less nor greater than 12 points. Font and font size may be varied for symbols or emphasis when appropriate (i.e., for scientific or mathematical terms).
- Well-crafted text usually does not require font variations for emphasis. Avoid underlining, bolding, or italicizing text purely for emphasis only. If you use them, do so sparingly and consistently. However, use of these variations for special symbols or words with special meaning is acceptable.

Line Spacing

- Vertical spacing of all text, including bibliographic references, should be 1.5 lines. Doublespacing is also acceptable. Long quotations, heading, footnotes, and captions may be single-spaced. You may use a writing style that is most commonly used within your discipline.
- The line spacing in chapters should match the line spacing in Front Matter and Back Matter pages.

After Space Setting	Where to Use It
12 (equal to single line spacing)	After lowest level subheadings
18 (equal to1.5 line spacing)	After higher level subheadings
24 (equal to double spacing)	After figure captions or table captions
36 (equal to 3 single spaced	After title page blocks, major headings and
blank lines; equal to 1 inch)	chapter headings

Table 1. Suggested Line Spacing

Margins

• Page margins should be set using the guidelines in Table 2.

Left	1"
Right	1"
Bottom	1"
Тор	1" (1.5" margins are required on pages i and ii ONLY)

Avoid ending pages with one-line paragraphs or with only the first line of text of a paragraph continuing on the following page (these are commonly called "orphans").
 Paragraphs ending pages of text must contain at least two lines of text or be moved to the top of the following page. This rule also applies to "hanging" subheadings.

- New pages of text must not begin with the last line of paragraphs carried over from previous pages (these are commonly called "widows"). Instead extend the bottom margin slightly on the previous page to accommodate the remainder of the paragraph, footnote, or figure caption.
- All tables and figures, including their captions, must conform to margin requirements. If figures or table and their captions cannot be placed on the same page, the table or figure caption should go on the page before the table or figure. See Appendix D for example.

Pagination

- Except for the title page, all pages of theses containing text or figures/tables are numbered. Page numbers must be placed on each page of the manuscript.
- Preliminary pages (i.e., dedication through ABSTRACT) are numbered consecutively in lowercase Roman numerals. Text and all reference pages, including appendices, are number consecutively in Arabic numerals beginning with 1 on the first page of text (usually CHAPTER 1).
- Pagination should be placed in the upper right corner with ½" below the top edge of the page. The last digit of the page number is even with the right margin.
- Page numbers must be in the same font and font size as your text.
- Title pages are technically the first numbered pages (Roman numeral "i"). However, the numeral "i" does not appear on the title page. The first page following the title page (Statement of Approval page) is numbered "ii". See Appendix E for example.
- The word "page" never accompanies the number. Pages numbered with a suffix (i.e., 2.b) are not permitted.

Chapter Titles

- The first page of a section or chapter must have a heading.
- Headings must be centered on the page and typed in all caps.
- Include 3 single spaced blank lines between heading and first line of text (or 36pt. spacing after the title).

Additional Departmental Style Requirements

Your department/major professor can provide guidance on what style is preferred in your discipline. Common style manuals include the MLA Handbook for Writers of Research Papers, and the Publication Manual of the American Psychological Association, and Chicago/Turabian.

CHAPTER 2. ORDER OF THESIS|DISSERTATION PAGES

This section describes the format specifications in *A Manual for the Preparation of Graduate Theses*. The presentation order of preliminary material, text, appendices, and publications must apply to all theses. Consistency and simplicity are key things to remember!

Preliminary Pages

Title Page

- Title pages are not numbered, but are counted as page "i" of the preliminaries.
- This page has a 1.5" top margin.
- The title page in the Purdue University Thesis Template MUST be used. This page has specific formatting requirements including the official University Seal that cannot be altered in any way.
- Type your full name as it appears on University records.
- Degree title for all doctoral candidates is "Doctor of Philosophy".
- Specific degree names for master's candidates appear on a candidates' Plan of Study.
- Title page date is the month and year your degree is actually awarded (not your defense or deposit date even if you are "Exam/Degree Only").
- Ensure you include the correct location of the degree-awarding campus (West Lafayette, Hammond, Fort Wayne, or Indianapolis).

Statement of Committee Approval Page

- This page is REQUIRED. The page should be numbered page "ii" and should NOT be listed in your TABLE OF CONTENTS.
- This page has a 1.5" top margin.

Dedication Page

- This page is OPTIONAL. They should be brief and centered on the page. No heading is used. Number this page "iii". Your dedication is not listed in the TABLE OF CONTENTS.
- You may choose or use a stylish font type as long as it looks professional.

Acknowledgements Page

- An Acknowledgements page is optional; however, most theses include brief statements of appreciation or recognition of special assistance. This page is not listed in your TABLE OF CONTENTS.
- The heading ACKNOWLEDGEMENTS, in capital letters, is centered between the left and right margins, without punctuation or underlining, one (1) inch from the top of the page, with the text beginning 3 single-space lines below.
- Line spacing of the text must be the same as in the rest of your thesis (i.e., 1.5 lines or double spacing).
- Pagination is in lowercase Roman numerals.
- This page begins 1" top margins that should be used in the remainder of the manuscript.

Preface

- This is optional. Appropriate examples of prefaces can be found in various style manuals. Prefaces are not listed in the TABLE OF CONTENTS.
- The heading PREFACE, in capital letters, is centered between the left and right margins, without punctuation or underlining, one (1) inch from the top of the page, with the text beginning 3 single-space lines below.
- Line spacing of the text must be the same as in the rest of your thesis (i.e., 1.5 lines or double spacing).
- Pagination is in lowercase Roman numerals.

Table of Contents

- This section is required.
- The heading TABLE OF CONTENTS, in capital letters, is centered between the left and right margins, without punctuation or underlining, one (1) inch from the top of the page, with the text beginning 3 single-space lines below.
- Line spacing of the TABLE OF CONTENTS should be 1.5 lines.
- All sections following the TABLE OF CONTENTS are listed. No preceding sections are listed.

- Headings of parts, sections, chapters, and their principal subdivisions are listed in the TABLE OF CONTENTS and must be worded exactly as they appear in the body of your thesis. When listing the subdivisions, list the same levels of headings and subheadings consistently with each chapter.
- Dotted leaders are required between headings and page numbers.
- Pagination for this section is in lowercase Roman numerals.

List of Tables

- This section is required if tables are represented in the thesis. The format for the LIST OF TABLES is generally the same for the TABLE OF CONTENTS.
- The heading LIST OF TABLES, in capital letters, is centered between the left and right margins, without punctuation or underlining, one (1) inch from the top of the page, with the text beginning 3 single-space lines below.
- Line spacing of the text should be 1.5 lines.
- Dotted leaders are required between captions and page numbers.
- This section contains exactly the same numbers and captions appearing above the tables in the text and appendices.
- Subsequent lines of captions should be indented.
- Pagination is in lowercase Roman numerals.

List of Figures

- This section is required if figures are represented in the thesis. The format for the LIST OF FIGURES is generally the same for the TABLE OF CONTENTS.
- The heading LIST OF FIGURES, in capital letters, is centered between the left and right margins, without punctuation or underlining, one (1) inch from the top of the page, with the text beginning 3 single-space lines below.
- Line spacing of the text should be 1.5 lines.
- Dotted leaders are required between captions and page numbers.
- This section contains exactly the same numbers and captions appearing above the tables in the text and appendices.
- Subsequent lines of captions should be indented.

• Pagination is in lowercase Roman numerals.

List of Symbols, Abbreviations, Nomenclature, or Glossary

- Lists of symbols, lists of abbreviations, nomenclature, or glossary may be appropriate for some theses. If needed, these appear after lists of tables and figures.
- Follow a form acceptable in your field of study.
- Pagination is lowercase roman numerals.
- Line spacing of the text should be 1.5 lines and dual columns can be used.

Abstract

- Abstracts are required.
- The heading ABSTRACT, in capital letters is centered between the left and right margins, without punctuation or underlining, one (1) inch from the top of the page. The first paragraph begins three single-space lines below the heading, is single-spaced, and begins at the left margin.
- The first paragraph must contain your name as it appears on the title page but with the last name first, the abbreviation of the degree title, the name of the institution granting the degree, the month and year the degree is awarded, the title of the thesis, and the name(s) of your major professor(s).
- Follow the first paragraph with a statement of the thesis problem, a brief exposition of the research and a condensed summary of your findings. There is no maximum length your summary must be.
- Line spacing of the text should be 1.5 lines.
- Mathematical formulas, diagrams, and other illustrative materials are not recommended for your abstract.
- Paginate abstract pages with lowercase Roman numerals.

Main Body Pages

The text of your thesis follows the preliminaries. A standard and consistent, organizational scheme must be adopted and used throughout your thesis.

Parts

For theses divided into parts, there are special instructions:

- If several chapters are grouped to form a part, the heading (ex: PART ONE, PART TWO, etc.) and the title are writing in capital letters and are centered on a separate divider sheet. The divider sheets are numbered and counted.
- The names of the parts are listed in the TABLE OF CONTENTS as major headings with their page numbers.
- Immediately following each divider sheet is the first page of the first chapter of each part. This page must carry the chapter number and title printed in capital letters, centered, and placed one (1) inch from the top of the page.

Introduction

- Introductions may precede the first chapters or major divisions of theses.
- In these cases, the heading INTRODUCTION, in capital letters, is centered without punctuation or underlining, one (1) inch from the top of the page. Text begins three (3) single-space lines below the heading.
- Begin pagination with Arabic numeral 1 on the first page of text.
- Introductions may also be first chapters of theses, in which case INTRODUCTION is the title of the first chapter or major division, and its placement is consistent with other chapter titles.
- If your thesis is divided into parts or sections, and if the introduction serves to introduce the entire work, it precedes the divider sheet for PART ONE.
- If each part has its own introduction, the INTRODUCTION section should follow the separate part divider sheet.

Body

• The body of the thesis is the substance of your dissertation, the comprehensive statement of year research.

Summary and Conclusions

• The summary and/or conclusions are often the last major division(s) of your text.

Recommendations

• You may include recommendations as a major division if your subject matter and research dictate.

Back Matter Pages

References

- The references/bibliography/works cited/list of references contains sources consulted during the course of your research.
- The heading REFERENCES, BIBLIOGRAPHY, WORKS CITED, or LIST OF REFERENCES, in capital letters is centered without punctuation or underlining, one (1) inch from the top of the page, and the first reference should be listed 3 single spaces below the title.
- Line spacing of this section should be consistent. Within entries, line spacing is the same required for the publishing style used in your discipline.
- Reference entries must not be divided between pages. To preclude this, shift the entire entry to the top of the next page and leave the bottom of the previous page blank.
- In some departments, theses are composed of separate and distinct parts. Each part, or chapter, has a references section that is placed at the end of the part. If this method is used, the references will follow the chapter with a heading typed in Title Case (like other subheadings) and will be listed in the TABLE OF CONTENTS as a subheading of a part, or chapter.
- If you are not using the above method, your references section will be listed in the TABLE OF CONTENTS as a major heading.

• Standards for presentation of references are set forth in style manuals or will be prescribed by your major professor.

Appendix/Appendices

- Appendices are not necessarily part of every thesis. Appendices are used for supplementary illustrative material, original data, computer programs, and other material not necessarily appropriate for inclusion within the text of your thesis.
- Appendices must meet the left margin requirement and the top margin, but not necessarily the bottom or right margin requirements.
- If there is only one appendix, it will be treated as a major division in the TABLE OF CONTENTS. If there are multiple appendices, each appendix will be treated as a major division in the TABLE OF CONTENTS.
- Appendix tables and figures MUST be included in the LIST OF TABLES and LIST OF FIGURES.
- See the sample thesis at the end of this manual for examples of appendix pages.

Notes and Footnotes

- The main requirement for notes and footnotes is consistency.
- When notes are placed at the end of chapters, they are treated as first order subdivisions and begin one (1) inch from the top of the page. The heading 'Notes' is listed in the TABLE OF CONTENTS.
- When notes are deferred to the end of your text, treat them as a major division and include the major division heading NOTES in the TABLE OF CONTENTS.
- When footnotes are used, begin and end them on the same page. If necessary, you can slightly reduce the font size (no smaller than 10 point font) and use single-spacing to accommodate the entire note on one page.

Vita

- This section is OPTIONAL for all candidates.
- The heading VITA, in capital letters, is centered on the page without punctuation or underlining, centered, one (1) inch from the top of the page. Do not use "VITAE" or "CURRICULUM VITAE".

- Your vita is the last major division of your TABLE OF CONTENTS and in your thesis unless followed by a publication.
- Do not use different font colors, graphics, photographs, figures, tables, etc. in your vita. Format should follow guidelines mentioned above. Usually, a few paragraphs are sufficient. Some departments may require a "curriculum vitae"; if so, these should generally follow University format.
- The font, font size, and line spacing should be consistently used throughout your entire thesis.
- Avoid using personal information in this section as it will be published and available throughout the internet.

Publication(s)

- Some departments require that a publishable paper, based on your thesis, be included. Your major professor will direct your preparation of this paper.
- Incorporation of publications should be placed as the last section of your thesis.
- The heading PUBLICATION, in capital letters is centered between the left and right margins, without punctuation or underlining, one (1) inch from the top of the page.
- Articles should be listed in the TABLE OF CONTENTS as PUBLICATION (or, if more than one, PUBLICATIONS).
- Journal article pages must conform to margin requirements stated for the rest of your thesis. Ensure article pages are fully legible, especially if they are scanned onto pages.

CHAPTER 3. GENERAL INFORMATION

Submission of your thesis to the Thesis/Dissertation Office usually satisfies the final prerequisite for receiving a graduate degree. Theses, as a rule, may not be altered or edited after their acceptance by the Thesis/Dissertation Office. Theses should not be uploaded to ProQuest until after all revisions, recommended by your committee and department, have been accomplished and departmental format approval has been secured.

There is more to a thesis than satisfying the University and departmental format and procedural requirements described in the previous sections of this manual. Your major professor as well as your departmental faculty and staff will help you meet the tangible requirements of an advanced degree. They will also help you with the intangible basics of conducting and communicating the results of your original research in your specialized area. The information in this chapter focuses on other, but no less important, the aspects of the research process including copyright protection and infringement, additional publication of your thesis, and use of unusual materials in your thesis.

Thesis Delay of Publication

Delay of Publication is a ProQuest sponsored program. Unlike Confidentiality, your thesis will be delivered to ProQuest as soon as your degree has been cleared by the records office. Your Abstract and References section will be published, but your content will remain unpublished until your Delay of Publication has expired, after which, your content will become available. It is possible to extend or shorten a Delay of Publication. If you have any questions about this program <u>contact our office</u>.

Thesis Confidentiality

Special circumstances, contractual obligations, patent or proprietary rights, or other factors may require that publication and distribution of theses be delayed. In such cases, you should choose the appropriate Confidentiality period on your Electronic Thesis Acceptance Forms. Withholding or delaying publication of research findings in a thesis is a serious matter and should be considered

carefully by you and your major professor. Initial requests for confidentiality are normally granted for one (1) year.

If you have any questions about this program, contact our office.

Once your thesis has been released from confidentiality by the Graduate School, your thesis will be delivered to ProQuest for publishing.

Foreign Language Theses

Occasionally, thesis texts are written in foreign languages. In these cases, title pages and abstracts must be in English. However, thesis titles may appear on the title page and in the first paragraph of the abstract in the same language as the text. English translations of titles may be included in parentheses immediately following titles in thesis abstracts (but not titles on title pages).

APPENDIX A. THE DEPOSIT PROCESS

[1] Schedule Your Appointment

- <u>Click here</u> to schedule an online appointment. Please, carefully review your Appointment Confirmation email.
- We recommend scheduling your appointment as far in advance as possible, especially if you are planning to deposit during the week before the deadline, as 75% of all deposit appointments are scheduled for the 14 days leading up to the deadline.

[2] Initiate Electronic Thesis Acceptance Form [ETAF] and Complete Survey(s)

• In addition to ETAF, **Master's candidates** will need to complete the Graduate School Exit Questionnaire and **Doctoral candidates** will need to complete the Graduate School Exit Questionnaire and the Survey of Earned Doctorates. Certificates of Completion will automatically be sent to the Thesis|Dissertation Office. Be sure to save a copy of the certificate for your personal records.

[3] Submit Electronic Thesis Deposit [ETD]

- Once your ETAF has been signed through the Thesis Form Head, refer back to your Plan of Study portal. A new link containing ProQuest information will appear and you may continue with submission. Provide all requested information to ProQuest. Carefully read all items, as some of ProQuest's optional services have fees attached. Buying into these optional services is not required.
- Check your thesis carefully to avoid formatting errors.

[4] Check Your Email

For requested formatting changes by the Thesis|Dissertation Staff.

[5] Be Available for your Online Appointment

A staff member from the Thesis Dissertation Office (TDO) will contact you via email during your appointment time with any questions that they may have. If they do not have any questions, they will simply email you a Deposit Receipt. If you complete all required steps before your deposit appointment time, your appointment may be moved to an earlier date by the TDO staff.

[6] Pay the Deposit Fee

- Master's Thesis Fee: \$90.00
- Ph.D. Dissertation Fee: \$125.00

West Lafayette Campus

West Lafayette candidates will pay the deposit fee through their myPurdue accounts. The Deposit Fee should appear in a candidate's student account approximately 2-3 days after their thesis deposit appointment.

Regional Campuses

Purdue Northwest and *Fort Wayne* candidates will pay their fees to their local Bursar's office as part of the clearance process by their regional campus format advisors.

IUPUI candidates will receive a paper bill following their successful deposit.

APPENDIX B. ORDER OF PAGES

Front Matter	Main Body	Back Matter
ABSTRACT (Required)	CHAPTER 4, etc.	PUBLICATIONS (Optional)
LIST OF TABLES, Figures, etc.	CHAPTER 3	VITA (Optional)
(Required – if using figures/tables in thesis)	CHAPTER 1	REFERENCES (Required – either after each
TABLE OF CONTENTS (Required)		chapter or a separate section at the end of the thesis)
PREFACE (Optional)		(Optional)
ACKNOWLEDGMENTS (Optional)		
Dedication (Optional)		
STATEMENT OF THESIS/DISSERTATION APPROVAL (Required)		
TITLE PAGE (Required) Page Number Not Typed		

APPENDIX C. STYLE HEADINGS

The following tables present a number of Styles that are common throughout multiple disciplines that have been modified for Purdue University Graduate School formatting requirements. You may use whatever style that is most common in your discipline, as long as your headings and subheadings are consistent throughout your thesis.

	APA HEADINGS
	(MODIFIED FOR GRADUATE SCHOOL ACCEPTANCE)
Level	Format
1	INTRODUCTION
2	Subheading 1
3	Subheading 2.
4	Subheading 3.
5	Subheading 4.

MLA HEADINGS		
	(MODIFIED FOR GRADUATE SCHOOL ACCEPTANCE)	
Level	Format	
1	CHAPTER 1. INTRODUCTION	
2	1.1 Subheading 1	
3	1.1.1. Subheading 2	
4	1.1.1.1 Subheading 3	
5	1.1.1.1 Subheading 4	

This style is represented in the sample pages in Appendix F

	CHICAGO/TURABIAN HEADINGS
	(MODIFIED FOR GRADUATE SCHOOL ACCEPTANCE)
Level	Format
1	CHAPTER 1. INTRODUCTION
2	1.1 Subheading 1
3	1.1.1 Subheading 2.
4	1.1.1.1 Subheading 3
5	1.1.1.1 This is subheading 4.

INSTI	TUTE OF ELECTRICAL AND ELECTRONICS ENGINEERS (IEEE)
	(MODIFIED FOR GRADUATE SCHOOL ACCEPTANCE)
Level	Format
1	1. INTRODUCTION
2	1.1 Subheading 1
3	1.1.1 Subheading 2
4	1.1.1.1 Subheading 3
5	1.1.1.1 This is subheading 4

APPENDIX D. CAPTIONS FOR LARGE FIGURES



Sample A



Sample B

APPENDIX E. UNIVERSITY COPYRIGHT INFORMATION

WHAT IS COPYRIGHT?

Copyright protects creative works and is a federal law. It allows authors to control the use of their works for a limited period of time. Once that time period has expired, the public is allowed to freely use the works without paying royalties and/or obtaining permission from the copyright holder.

#1 What are the requirements for a work to be copyrighted?

The work must be an original work that is fixed in a tangible medium of expression. The word "copyright" or \mathbb{O} is no longer required for works to be considered copyrighted. A work is protected from the moment of fixation.

#2 What are the copyright holder's exclusive rights?

- To publish or distribute the work
- To reproduce the work
- To create derivative works

- To perform or display the work publicly
- To authorize others to exercise the above rights

#3 How long does copyright protection last?

Works created on or after January 1, 1978 are protected for a term of the life of the author plus 70 years. If the work is a product of a corporate author, then the protection is for the shorter of 95 years from publication or 120 years from creation.

#4 What can be copyrighted?

Eight categories of works are copyrightable:

- Literary, musical, and dramatic works
- Pantomimes and choreographic works
- Pictorial, graphic, and sculptural works
- Sound recordings

#5 What cannot be copyrighted?

- Ideas, procedures, methods, systems, and processes
- Titles, names, short phrases, and slogans

- Motion pictures and other AV works
- Computer programs
- Compilations and derivative works
- Architectural works
- Facts, news, and research
- Works produced by US government employees or agencies

#6 What is the public domain?

The public domain consists of all works that never had copyright protection, such as US government works, and works on which has expired. Works published in the US prior to 1923 are in the public domain. All works in the public domain are free for the public to use.

FAIR USE EXCEPTION

The purpose of the fair use exception is to allow the public to use copyrighted works under certain circumstances without having to pay royalties or obtain permission. Fair use is determined by applying a four factor analysis that balances the rights of the copyright holder with that of the public. The four questions are designed to help a user decide if the planned use of the work is fair and under the law.

Factor #1 What is the purpose and character of the use?

Favors Fair Use:

- Non Profit
- Educational
- Personal
- Teaching
- Criticism/Comment
- Scholarship/Research
- News Reporting

Factor #2 What is the nature of the work to be used?

Favors Fair Use

Published

Small amount

Favors Permission

Fact

•

•

- Creative • Unpublished

Factor #3 How much of the work will be used?

Favors Fair Use

Favors Permission

- Large amount
 - Heart of the work

- **Favors Permission**
 - Commercial
 - For Profit
 - Entertainment

Factor #4 What is the market effect of the use of the work?

Favors Fair Use

Favors Permission

- Small amount
- No effect
- Licensing/permission unavailable
- Large amount
- Major effect
- Work is made available to the world

If the balance of the four factors weighs in favor of fair use, then the work can be used without obtaining permission from the copyright owner. However, if the balance weighs against fair use and other exceptions do not apply, then permission to use the work must be obtained. The fair use analysis must be applied to each use of every work. Fair use is technologically neutral so the analysis may be used for any medium.

Please be aware that not all educational use is automatically fair use. It is the responsibility of each member of the Purdue University community to apply all the factors and to make a reasonable and good faith determination as to whether or not the use can be considered fair.

UNDERSTANDING YOUR RIGHTS

Almost everyone is an author in some way. You generally own the copyright to your works such as self-created websites, photographs, emails, and home videos, as well as the works produced in your roles as students and researchers. It is important to understand your rights as an author, as well as the ways in which you can better protect your intellectual property.

For further information on the management of intellectual property at Purdue, review *Purdue Policy I.A.1: Intellectual Property*, <u>www.purdue.edu/policies/academic-research-affairs/ia1.html</u>

The information in this appendix has been reproduced with the permission of Donna L. Ferullo, J.D. – University Copyright Office.

Ferullo, Donna. (2016). *University Copyright Office – A Guide to Copyright* [Brochure]. N.p.

APPENDIX F. SAMPLE PAGES

These sample pages follow the MLA formatting style. For examples including IEEE, APA, and Chicago/Turabian visit the templates section on the <u>TDO's Formatting and Templates</u>

page.

THE DYNAMIC STRESS-STRAIN RESPONSE OF HIGH-ENERGY BALL MILLED ALUMINUM POWDER

by

Andrew W. Justice

A Thesis

Submitted to the Faculty of Purdue University In Partial Fulfillment of the Requirements for the degree of

Master of Science in Mechanical Engineering



Department of Mechanical Engineering West Lafayette, Indiana May 2017

THE PURDUE UNIVERSITY GRADUATE SCHOOL STATEMENT OF COMMITTEE APPROVAL

Dr. Steven F. Son, Chair

Department of Mechanical Engineering

Dr. Weinong Chen

Department of Aeronautics and Astronautics

Dr. Ibrahim E. Gunduz

Department of Mechanical Engineering

Approved by:

Dr. Jay P. Gore

Head of the Graduate Program

In Dedication to Irina

ACKNOWLEDGMENTS

The author would like to thank M.T. Beason, H. Liao, B.H. Lim, and C.D. Kirk for their experimental support with the split-Hopkinson pressure bar. The author would also like to recognize Dr. Steven Son, Dr. Ibrahim Gunduz, and Dr. Weinong Chen for their support and guidance throughout this project. This work was supported by the Department of Energy, National Nuclear Security Administration, under the award number DE-NA0002377 as part of the Predictive Science Academic Alliance Program II.

TABLE OF CONTENTS

LIST OF TABLES	vi
LIST OF FIGURES	vii
ABSTRACT	viii
CHAPTER 1: INTRODUCTION	1
1.1 Background on the Split-Hopkinson Pressure Bar	2
1.2 SHPB Theory	
CHAPTER 2: EXPERIMENTAL METHODS	6
2.1 Material Preparation	6
2.2 Passive Confinement	6
2.3 The Split-Hopkinson Pressure Bar	
2.4 SHPB Data Analysis	9
2.5 Validation of Passive Confinement	9
2.6 P-α modelling	11
CHAPTER 3: RESULTS AND DISCUSSION	14
3.1 Stress-Strain Response of Aluminum and HEBM Aluminum Powders	14
3.2 P-α Comparison	16
CHAPTER 4: CONCLUSIONS	19
REFERENCES	20

LIST OF TABLES

Table 3.1: Aluminum and HEBM powder P-α parameters 10	6	,
--	---	---

LIST OF FIGURES

Figure 2.1: Section	on a) and b) depict the powder morphology of the HEBM and pure
alum	inum respectively
Figure 2.2: The a	luminum pellet's polycarbonate confinement with S7 tool steel
comp	pression pins
Figure 2.3: The s	plit-Hopkinson pressure bar in a compression configuration9
Figure 2.4: High-	speed imaging of confining jacket deformation11
Figure 3.1: Section	on a) and c) provide the calculated stress-strain curves for pure and
HEB	M aluminum powders at various strain-rates. Section b) and d) present
the a	verage stress and relative density curves for the two powders and their
respe	ective P- α models. All plots include error bars of two standard
devia	ations above and below the mean
Figure 3.2: A con	nparison of strain hardening for solid 1100 series aluminum and
alum	inum powder P- α models with the shaded region representing two
stand	lard deviations above and below the mean17

ABSTRACT

Author: Justice, Andrew, W. MSME Institution: Purdue University Degree Received: May 2017 Title: The Dynamic Stress-Strain Response of High-Energy Ball Milled Aluminum Powder Major Professor: Steven Son

High-energy ball milling (HEBM) is a bulk powder manufacturing process used in the creation of dispersion strengthened and nanolaminate materials. Fundamentally, these materials have not been dynamically characterized in a green state prior to hot consolidation. This study incorporates porosity with these HEBM compacts to investigate the effect of high strain-rate on void collapse and particle interaction which has broad applications in development of predictive models for impact events of porous metallic structures that may be employed as energy absorbers, reactive structures, and intermetallic materials. Here, pure and HEBM aluminum powders have been characterized under dynamic compression using the split-Hopkinson pressure bar (SHPB) in a passive confinement configuration. The plastic deformation of the powder and crush up were shown to be strain-rate insensitive; and as a result, were modelled adequately with a second order $P-\alpha$ model. The pure aluminum and HEBM aluminum powders appear to have the same strain-hardening coefficient and strength index as solid aluminum after yielding. The powders' respective stress-strain responses follow the same trend but differ only in strength as result of porosity and pre-strain experienced prior to dynamic compression. The HEMB powder was found to be twice as strong as the untreated pure aluminum powder.

CHAPTER 1. INTRODUCTION

High-energy ball milling (HEBM) is a manufacturing technique used to reduce, mix, and alloy particles in the ceramic and powder processing industries [1]. HEBM was originally created as a bulk powder process to create dispersion strengthened superalloys by the hot consolidation of ball milled powders [2]. As a result, there has been extensive work done to characterize and model the quasi-static and dynamic mechanical behaviors of these dispersion strengthened alloys and nanocrystalline composites [3,4]. Since then, HEBM has also been used to create nano-lamellar intermetallic composites resulting in intermetallics with increased reactivity [5–7]. Additionally it has been used to entrain propellant additives in aluminum based fuel systems [8–10]. However, these powder systems have not been studied under high strain-rate compression without first being sintered and annealed.

To date, it is not well understood how porosity effects the dynamic mechanical response of metallic powder systems. Dynamic characterization of HEBM metallic powders has broad implications for the understanding and creation of energetic structural members and energy absorbers under impact. Experimental parameters gathered from this work will aid in the modeling and testing of such impact events on aluminum powder based structures.

This work aims to characterize the dynamic response of porous HEBM aluminum powder compacts. This was done using a split-Hopkinson pressure bar (SHPB) with the specimen under passive confinement; which was adapted from the study of porous geomaterials like concrete and sand [11,12]. This allows for porous materials to be tested in the SHPB by applying weak radial stress. As a result, HEBM aluminum compacts of 70-90% theoretical maximum density (%TMD) were characterized with the SHPB in a biaxial stress condition.

1.1 Background on the Split-Hopkinson Pressure Bar

The split-Hopkinson Pressure bar in the compression configuration was originally designed by Herbert Kolsky in 1949 [13]. For this reason, the SHPB is also know by many as the Kolsky bar; however, it retains the name 'Hopkinson' in the memory of John and Bertram Hopkinson. Bertram continued his father's work on stress wave propagation by devising a method of measuring pressure waves imparted by high velocity projectiles or high explosives [14]. The SHPB established by Herbert Kolsky, consisted of three sections, an incident bar, small test sample, and transmission bar [15]. It used a cathode ray oscilloscope in combination with electrical condenser units to record the pressure waves' propagation through the bars. The technique of measuring the wave propagation through the SHPB has been changed and improved over the years allowing for more precise measurements with the advent of strain gauges and high speed oscilloscopes. This experimental method enabled the measurement of the dynamic stress-strain behavior of materials.

The SHPB works on the same principles outlined by Bertram Hopkinson. It is assumed that the incident and reflected waves propagated through the bar without appreciable augmentation; Kolsky clarified this assumption, and broke it into three aspects [16]. The wave propagation through the bars is assumed to be one dimensional in nature and described by one-dimensional wave propagation theory. In the test sample, there are axially uniform stress and strain fields; meaning the test sample is in stress equilibrium in the axial direction. Additionally, the frictional and inertial effects of the sample are assumed negligible.

1.2 SHPB Theory

The succeeding analysis follows from Lindholm [17]. Strain measurements taken from the incident and transmission bars allow for the calculation of stress, strain, and forces exerted on the test specimen. Based on one-dimensional theory of elastic wave propagation, the displacement u is given by,

$$u(t) = c_0 \int_0^t \varepsilon(t) dt', \tag{1}$$

with time, *t*, and the elastic wave speed, c_0 , and strain, $\varepsilon(t)$. The displacement of the face of the incident bar, $u_1(t)$, is produced by incident strain pulse, $\varepsilon_l(t)$, traveling towards the test specimen and the reflected strain pulse, $\varepsilon_R(t)$, and is given by

$$u_1(t) = c_0 \int_0^t \varepsilon_I(t) dt' + (-c) \int_0^t \varepsilon_R(t) dt' = c_0 \int_0^t (\varepsilon_I(t) - \varepsilon_R(t)) dt' \quad . \tag{2}$$

Similarly, the displacement of the front face of the transmission bar is given by integration the transmitted pulse,

$$u_2(t) = c_0 \int_0^t c_T(t) dt'.$$
 (3)

Taking the change of displacement between the two bar faces and dividing by the initial length of the specimen, L_s , yields the engineering strain experienced by the specimen, $\varepsilon_s(t)$, and is given by,

$$\varepsilon_s(t) = \frac{u_1(t) - u_2(t)}{L_s} = \frac{\varepsilon_0}{L_s} \int_0^t (\varepsilon_I(t) - \varepsilon_R(t) - \varepsilon_T(t)) dt' \quad . \tag{4}$$

Assuming that the stress found across the short test specimen is uniform yields

$$\varepsilon_R(t) = \varepsilon_T(t) - \varepsilon_I(t). \tag{5}$$

Substituting Equation (5) into Equation (4), reduces the base equation for the test specimen's engineering strain to

$$\varepsilon_s(t) = -\frac{2c_0}{L_s} \int_0^t \varepsilon_R(t) \, dt' \tag{6}$$

The applied loads $P_1(t)$ and $P_2(t)$ on each face of the specimen are given by,

$$P_1(t) = EA(\varepsilon_l(t) + \varepsilon_R(t)), \tag{7}$$

and

$$P_2(t) = EA\varepsilon_I(t), \tag{8}$$

with, A being the cross-sectional area of the bar and, E being the elastic modulus of the bar material. Given the loads imparted on the faces of the test specimen, the stress found within the sample is

$$\sigma_{s}(t) = \frac{P_{1}(t) + P_{2}(t)}{2A_{s} 2 A_{s}} = \frac{1}{E} \left(\frac{A}{2}\right) \left(\varepsilon_{I}(t) + \varepsilon_{R}(t) + \varepsilon_{T}(t)\right).$$
(9)

Equation (9) can be simplified with Equation (4) reducing to the following,

$$\sigma_{s}(t) = E\left(\frac{A}{A_{s}}\right)\varepsilon_{T}(t).$$
(10)

These derivations all assume that the incident, reflected, and transmission strain measurements have been shifted in time so that they coincide at the time the wave has initially reached the test specimen's front face. The forces and velocities of the bar faces are calculated with

$$F_{input}(t) = AE[\varepsilon_I(t) + \varepsilon_T(t)], \qquad (11)$$

$$F_{output}(t) = AE[\varepsilon_I(t) + \varepsilon_T(t)], \qquad (12)$$

$$V_{input}(t) = c_0[\varepsilon_I(t) + \varepsilon_T(t)], \qquad (13)$$

and

$$V_{output}(t) = c_0 \varepsilon_T(t). \tag{14}$$

An important element in this analysis is that a SHPB experiment must not significantly violate the four guiding assumptions. The most difficult assumption to maintain is the axial force equilibrium postulation. To produce a pulse that allows for a test specimen to deform in quasi-stress equilibrium, the initial pulse must be conditioned. Therefore, the incident pulse is formed to closely resemble the transmitted response produced by the sample by placing a pulse shaper on the face of the incident bar face that is struck by the striker bar.

CHAPTER 2. EXPERIMENTAL METHODS

2.1 Material Preparation

The aluminum and HEBM aluminum powders were sized to 106-355 μ m using sieves in a Ro-Tap Motorized Sieve Shaker RX-29 for a duration of 24 hours. The pure aluminum powder (Alfa Aesar, 99.98% purity) had an average particle size of 44-420 μ m and the HEBM aluminum originated from a particle size of 7-15 μ m. The 7-15 μ m aluminum was milled with a planetary ball mill (Retsch GmbH, model PM100, Germany) in a 250-ml stainless steel jar with 175 g of 9.5 mm stainless-steel media. A wet milling condition was used to achieve the desired particle size, 20 ml of hexane was added to the 10:1 mass ratio of media to the aluminum powder mixture.



Figure 2.1: Section a) and b) depict the powder morphology of the HEBM and pure aluminum respectively.

The jar was pressurized with high purity argon (99.998%) to 275 kPa, creating an inert milling environment; the jar was purged four times prior to final pressurization. The

aluminum was milled at 650 revolutions per minute (rpm) for five minutes, inactive for fifteen minutes, then rotated in the reverse direction for an additional five minutes at 650 rpm and left to dry under vacuum at 293 K prior to sieving. These powders are shown in Figure 2.1a and Figure 2.1b.

The powders were cold pressed to a height of 4 mm in a 6-mm diameter cylindrical easy-retrieve die with a die press (Across International). The compression was halted by a mechanical stop at a height of 4 mm. Three different pellet densities were pressed from the pure aluminum powder: 70, 80, and 90 %TMD. For the HEBM powder, the 70 %TMD samples were hand-packed into the confining structure and the 90 %TMD samples were cold pressed. The HEBM aluminum could not be reliably hand-packed beyond 70 %TMD, and the cold pressed powders would break apart below 90 %TMD.

To compare the aluminum powder composites with solid aluminum dynamic compaction; disks of 1100 series aluminum were tested in the SHPB. These disks were cut from a cold rolled 1100 series aluminum rod, with a thickness of 2.74 ± 0.01 mm and a diameter of 12.70 ± 0.01 mm.

2.2 Passive Confinement

Pellets were passively confined [11] in 12.70 mm lengths of polycarbonate tubing as depicted in Figure 2.2. Each tube had an inner diameter of 6.35 mm and a wall thickness of 1.59 mm. After retrieval, the pellet had a diameter between 6.10-6.15 mm, resulting in a slip fit within the tube. The pellet was secured between two heat treated S7 tool steel pins with a 6.35 mm diameter and height. These steel pins were heat treated to a hardness of 2200 MPa to prevent deformation during dynamic loading.



Figure 2.2: The aluminum pellet's polycarbonate confinement with S7 tool steel compression pins.

2.3 The Split-Hopkinson Pressure Bar

Dynamic material testing was performed using a Split-Hopkinson pressure bar, also commonly referred to as a Kolsky bar. The SHPB was configured in a traditional compression arrangement [15], as seen in Figure 2.3. A gas gun was used to achieve strainrates from 1000-2100 s⁻¹. The cylindrical striker had a length of 47.5 ± 0.10 cm and diameter of 1.89 ± 0.01 cm which matched the diameter of the incident and transmission bars. Circular pulse shapers of various diameter were punched from a flat copper plate with a thickness of 1.90 ± 0.01 mm. The diameter of the pulse shaper was augmented for each material and strain-rate to produce the desired incident pulse shape. The striker, incident, transmission, and momentum bars were made of Vasco Max 350 stainless steel. The incident and transmission bar were 4.16 ± 0.01 m and 1.37 ± 0.01 m long. The incident and transmission pulses were measured using two strain gauges mounted in the center of both bars. Each gauge pair was oriented longitudinally and placed a half-circumferential length from one another on opposing sides of the bar. The pairs were powered and connected to independent half Wheatstone Bridges and amplified by Tektronix ADA400A Differential Preamplifiers. The conditioned voltage signals were recorded with a Tektronix DPO4034 Digital Phosphorus Oscilloscope.



Figure 2.3: The split-Hopkinson pressure bar in a compression configuration.

2.4 SHPB Data Analysis

Strain measurements taken from the incident and transmission bars allowed for the calculation of stress, strain-rate, and strain exerted on the test specimen using

$$\sigma_s(t) = E\left(\frac{A_b}{A_s}\right) \varepsilon_T(t),\tag{15}$$

$$\dot{\varepsilon}_{s}(t) = \frac{2c_{b}}{L_{s}} \varepsilon_{R}(t), \tag{16}$$

and

$$\varepsilon_s(t) = -\frac{2c_h}{L_s} \int_0^t \varepsilon_R(t) \, dt' \quad , \tag{17}$$

with strain ε , sound speed of the bar c_b , sample length L_s , young's modulus of the bar E, and area A. Subscript *s* and *b* refer to specimen and bar; *T*, *R*, and *I* are in reference to the transmitted, reflected, and incident strain measurements. The SHPB analysis is derived in detail by Lindholm [17].

2.5 Validation of Passive Confinement

To perform classical SHPB data analysis, the confinement modifications made to the apparatus must not invalidate the fundamental assumptions made; for example, it is assumed that the stress waves propagating through the SHPB and test specimen can be described by one-dimensional wave propagation theory [16]. Additionally, the axial

stress and strain fields are assumed uniform and the inertial and frictional effects on the faces of the sample are deemed negligible. If the radial strain is comparable with the axial strain during the test, then it cannot be accurately assumed that the stress wave propagating through the specimen is one-dimensional. A high-speed black and white camera (Phantom v7.3, Vision Research) capturing at 40,000 frames per second and 100 μ m per pixel spatial resolution was used to image the dynamic compression of the powdered aluminum. Figure 2.4 shows the compression of an initial 80 %TMD powdered aluminum sample. The approximate density is calculated with,

%TMD (t) =
$$\frac{m_i}{\rho_{al} \left(\frac{\pi h_l d_l^2 (1 - \varepsilon_s(t))}{2} \right)}$$
 (18)

with initial pellet mass m_i , diameter d_i , height h_i , and the density of aluminum ρ_{al} . At 75 µs, the sample has reached 95% of its theoretical solid density in the assumption that the pellet's cross-sectional area remained constant during compression event. This assumption is justified during this portion of the test. The radial strain was measured to be approximately 1.5% using the imaging's spatial resolution. When the radial strain is compared to the axial strain of 20%, the radial strain can be deemed negligible [12]. Therefore, the test is reasonably one-dimensional until the sample approaches full density (passes 95 %TMD). Consequently, the assumptions made for these tests become less appropriate as the test goes beyond 5, 15, 25% strain in the axial direction for the 90, 80, and 70 %TMD samples respectively.



Figure 2.4: High-speed imaging of confining jacket deformation.

In the passive confinement configuration, the aluminum is in a bi-axial stress state. We use,

$$\sigma_r = \frac{E}{\frac{2R_2}{2}} (R_1^2 - (19))$$

to calculate the radial stress, σ_r , given the radial strain, ε_r , assuming a thick-walled cylinder [12], with *E* being the polycarbonate's Young's modulus (2.0 GPa) and R_1 , R_2 being the outer and inner radius respectively. Therefore, the radial stress on the sample at 95 %TMD was approximately 18.75 MPa given a radial strain of 1.5%. Consequently, the radial stress on the sample was lower than 20 MPa for the initial compaction of the aluminum compact.

2.6 P-α modelling

The P- α model is a phenomenological constitutive equation created by Hermann [18] to describe the irreversible compaction of porous materials. The model relies on the assumption that the macroscopic shear strength of the parent material is negligible in

comparison to the irreversible compaction of the pores. At low pressures, the porous structure deforms elastically and at a certain point yielding occurs as the pressure exceeds the elastic limit. This yielding is attributed to the inelastic compaction of the structure resulting from the rearrangement and plastic deformation of the parent particles [19]. The partially compacted material is described by the specific volume, v, entropy, η , and porosity defined as,

$$\alpha = \frac{\nu_p}{\nu_p}^{\nu} \tag{20}$$

with the subscript p representing the specific volume of the parent solid at the thermodynamic state of interest. This leads to the second underlining principle of the P- α model, the conversion between the parent solid to porous material can be done without altering its internal energy. Therefore, the equation of state of the parent and porous material is given by

$$e = e(\nu, \eta, \alpha) = e_p(\nu_p, \eta), \qquad (21)$$

with *e* representing internal energy.

Carroll and Holt [20,21] later drew the correlation between the pressure in the parent solid and the porous material as a function of porosity. Instead of assuming that the pressure in the parent solid and porous material were the same, Carroll and Holt suggested that $p = p_p/\alpha$. Therefore, the pressure state is given by

$$p(\nu, \eta, \alpha) = \frac{1}{\varrho} p_p(\nu_p, \eta).$$
⁽²²⁾

To fit a P- α model to the stress-strain data collected from the SHPB experiments, yield points were approximated from each experimental stress-strain curve by calculating the intersection of the elastic and plastic regimes. Using elastic data given on the pure aluminum powder investigated by Salvadori et al. [22]; logarithmic curve fits were generated for each initial porosity of the elastic regimes and intersected with linear fits for the plastic regimes of each SHPB stress-strain curve. The intersection of these curves resulted in the calculated yield point for each experimental stress-strain curve. A second order P- α model of the form,

$$\alpha = 1 + (\alpha_e - 1)(\frac{p_s - p}{p_s - p_e})^2,$$
(23)

was used to model the data. The parameters, p_e and p_s represent the pressure at the yielding and full density conditions respectively, and α_e as the porosity at the yield point. These parameters were determined with a least-squared analysis of the second order P- α with the approximated yield points. Therefore, Equation (23) gives porosity as a function of the pressure applied to the powder matrix.

CHAPTER 3. RESULTS AND DISCUSSION

3.1 Stress-Strain Response of Aluminum and HEBM Aluminum Powders

Figure 3.1 provides the generated stress-strain responses for both the pure and HEBM aluminum. Figure 3.1a and Figure 3.1c give the stress-strain curves for the two materials at the various initial densities and strain-rates. Each curve is an average of five SHPB tests at a given initial density and strain-rate taken at room temperature (25°C). It is clearly seen that the HEBM strengthens the powder significantly; the HEBM aluminum is shown to be approximately twice as strong as the pure aluminum powder.



Figure 3.1: Section a) and c) provide the calculated stress-strain curves for pure and HEBM aluminum powders at various strain-rates. Section b) and d) present the average stress and relative density curves for the two powders and their respective P- α models. All plots include error bars of two standard deviations above and below the mean.

This increase in strength can be attributed to work hardening of the aluminum during the milling process. High-energy ball milling cold welds and reduces powder particles leading to nanocrystalline grains. It has been found that grain size decreases with milling time asymptoting to a constant value that scales with melting point for faced-centered cubic (FCC) metals. [23] The mechanism of forming nanometer sized grains during milling mirrors that of dynamic recrystallization; the minimal grain size is attributed to the balance of dislocation accumulation and dynamic recovery in the form of new grains and subgrain boundaries [24]. Aluminum with ultrafine grain (UFG) structure's strength has been found to hold true to a Hall-Petch relationship [25]; which states that strength increases with decreasing grain size [26,27].

Additionally, it should be noted that both aluminum powders' stress-strain responses appear to be relatively strain-rate insensitive. This is unlike what has been observed in the testing of solid nanocrystalline aluminum in dynamic and quasi-static loading conditions [4]. This may be attributed to the introduction of porosity in this experiment. The variation of the stress-strain curves for each initial density are within experimental error of one another demonstrating that there is negligible strain-rate effect on the dynamic compaction of the aluminum powder systems.

3.2 P-a Comparison

	ae	p _s (MPa)	p _e (MPa)
PURE AI	1.55	159	6.97
HEBM AI	1.94	259	7.24

Table 3.1: Aluminum and HEBM powder P-α parameters.

Since the aluminum powders exhibit limited strain-rate dependency in their stressstrength responses, the curves for each strain-rate were averaged to create a composite curve in Figure 3.1b and Figure 3.1d to be compared with their corresponding P- α model. The P- α models appear to represent the data well with the following parameters in Table 3.1. The model trends closely with experimental data from 70-95 %TMD in Figure 3.1b and Figure 3.1d; after which, the slope of the model quickly steepens towards full compaction. As mentioned previously, the data and model diverges at these higher densities as the result of radial expansion of the passive confinement. Overall, the experimental data correlates well with the P- α model calibrated from the initial yield points for the aluminum powder suggesting that the assumptions made with the P- α model holds true for aluminum powder since its compaction is relatively strain-rate insensitive.



Figure 3.2: A comparison of strain hardening for solid 1100 series aluminum and aluminum powder P- α models with the shaded region representing two standard deviations above and below the mean.

To compare the effect of ball milling on the aluminum powder's deformation and crush-up; the P- α models for both powders were converted to equivalent parent solids using Equations (21) and (22) beginning at 70 %TMD. These converted curves were then compared with experimental data taken on disks of 1100 series aluminum. The rod stock aluminum was tested with the SHPB at strain-rates between 1000 – 6000 s⁻¹. After yielding, the disks deformed plastically in a linear fashion. Figure 3.2 illustrates the linear trend of the HEBM and pure aluminum equivalent parent solids in comparison to the solid 1100 series aluminum rod after yielding. All three forms of aluminum appear to maintain the same strength index and follow the Ludwik's equation [28] for strain-hardening,

$$\sigma = \sigma_o + K(\varepsilon)^n,\tag{24}$$

K is the strength index, σ_o is yield strength and n is the strain-hardening exponent. This region was well correlated to a linear fit; therefore stress-strain responses were approximated in the first order with a strain-hardening exponent of one. This approximation revealed that the strain-hardening exponent and strength index of the aluminum powders were not altered because of high-energy ball milling, rate of strain, or porosity.

Since the strain hardening exponent and strength index remained constant for these three forms of aluminum; the strength differences seen in these materials can simply be expressed as a function of pre-strain or work hardening prior to dynamic compression in the SHPB. The equation for strain-hardening can be expressed as a function of strain and pre-strain, ε_o , in swift's equation [28],

$$\sigma = K(\varepsilon + \varepsilon_o)^n. \tag{25}$$

Therefore, the strength seen in the parent solid material in the powders and the solid aluminum rod only differ in the amount of work-hardening and underlining grain structure and dislocations in the material.

CHAPTER 4. CONCLUSIONS

The dynamic compaction of pure and high-energy ball milled aluminum powders have been characterized in a passive confinement configuration using the SHPB. A second order P- α model was used to model the irreversible compaction of the porous aluminum. The dynamic responses of the powders were found to be largely insensitive to the effect of strainrate and their strengths appear to be a function of porosity and pretreatment. High-energy ball milling and porosity was found to not alter the strain hardening exponent or strength index of the aluminum powder for this milling condition. The milling process strengthened the aluminum powder; however, no increased sensitivity to strain-rate was observed as seen in nanocrystalline aluminum solids. The reduction of void space in the aluminum compact appears to be a strain-rate independent process.

Passive confinement provides an effective means to test porous aluminum in the SHPB. This technique may be expanded with other metallic powders to provide dynamic material parameters to assist in the calibration and validation of dynamic compression models on porous metallic structures. Consequently, this work will assist in the understanding and creation of energetic structural members and energy absorbers exposed to impact.

REFERENCES

- Koch, E., Ammonium Nitrate Explosives for Civil Applications Handbook of Combustion Organic Chemistry of Explosives High Energy Materials n.d. [2] Benjamin, J.S., Dispersion strengthened superalloys by mechanical alloying. Metall Trans 1970;1:2943–51. doi:10.1007/BF03037835.
- Khan, H., Zhu, Z., Estimates of the sticky-information phillips curve for the United States. J Money, Credit Bank 2006;38:195–207. doi:10.1016/j.ijplas.2004.07.008. [4] Farrokh, B., Khan, A.S., Grain size, strain rate, and temperature dependence of flow stress in ultra-fine grained and nanocrystalline Cu and Al: Synthesis, experiment, and constitutive modeling. Int J Plast 2009;25:715–32. doi:10.1016/j.ijplas.2008.08.001.
- [5] Schwarz, R.B., Srinivasan, S., Desch, P.B., Synthesis of Metastable Aluminum-Based Intermetallics by Mechanical Alloying. Mater Sci Forum 1992;88–90:595– 602. doi:10.4028/www.scientific.net/MSF.88-90.595.
- [6] Gunduz, I.E., Kyriakou, A., Vlachos, N., Kyratsi, T., Doumanidis, C.C., Son, S., et al., Spark ignitable Ni-Al ball-milled powders for bonding applications. Surf Coatings Technol 2014;260:396–400. doi:10.1016/j.surfcoat.2014.06.068.
- [7] Reeves, R.V., Mukasyan, A.S., Son, S.F., Thermal and impact reaction initiation in Ni/Al heterogeneous reactive systems. J Phys Chem C 2010;114:14772–80. doi:10.1021/jp104686z.
- [8] Sippel, T.R., Son, S.F., Groven, L.J., Altering reactivity of aluminum with selective inclusion of polytetrafluoroethylene through mechanical activation.
 Propellants, Explos Pyrotech 2013;38:286–95. doi:10.1002/prep.201200102.
- [9] Terry, B.C., Sippel, T.R., Pfeil, M.A., Gunduz, I.E., Son, S.F., Removing hydrochloric acid exhaust products from high performance solid rocket propellant using aluminum-lithium alloy. J Hazard Mater 2016;317:259–66. doi:10.1016/j.jhazmat.2016.05.067.

- [10] Rubio, M.A., Gunduz, I.E., Groven, L.J., Sippel, T.R., Han, C.W., Unocic, R.R., et al., Microexplosions and ignition dynamics in engineered aluminum/polymer fuel particles. Combust Flame 2017;176:162–71. doi:10.1016/j.combustflame.2016.10.008.
- [11] Gong, J.C., Malvern, L.E., Passively confined tests of axial dynamic compressive strength of concrete. Exp Mech 1990;30:55–9. doi:10.1007/BF02322703.
- Bragov, A.M., Lomunov, A.K., Sergeichev, I.V., Tsembelis, K., Proud, W.G.,
 Determination of physicomechanical properties of soft soils from medium to high strain rates. Int J Impact Eng 2008;35:967–76. doi:10.1016/j.ijimpeng.2007.07.004.
- [13] Chen, W.W., Song, B., Split Hopkinson (Kolsky) Bar: Design, Testing and Applications. 2013. doi:10.1007/978-1-4419-7982-7.
- [14] Hopkinson, B., A Method of Measuring the Pressure Produced in the Detonation of High Explosives or by the Impact of Bullets. Philos Trans R Soc London Ser A, Contain Pap a Math or Phys Character 1914;213:437–56.
- [15] Kolsky, H., An investigation of the mechanical properties of materials at very high rates of loading. Proc Phys Soc Lond B 1949;62:676–700.
- [16] Zhao, H., Gary, G., On the use of SHPB techniques to determine the dynamic behavior of materials in the range of small strains. Int J Solids Struct 1996;33:3363–75. doi:10.1016/0020-7683(95)00186-7.
- [17] Lindholm, U.S., Some experiments with the split hopkinson pressure bar*. J Mech Phys Solids 1964;12:317–35. doi:10.1016/0022-5096(64)90028-6.
- [18] Herrmann, W., Constitutive equation for the dynamic compaction of ductile porous materials. J Appl Phys 1969;40:2490–9. doi:10.1063/1.1658021.
- [19] Davison, L., Fundamentals of Shock Wave Propagation in Solids. 2008. doi:10.1007/978-3-540-74569-3.
- [20] Carroll, M.M., Holt, A.C., Static and dynamic pore-collapse relations for ductile porous materials. J Appl Phys 1972;43:1626–36. doi:10.1063/1.1661372.
- [21] Holt, W.H., Mock, W., Zerilli, F.J., Clark, J.B., Experimental and computational study of the impact deformation of titanium Taylor cylinder specimens. Mech Mater 1994;17:195–201. doi:10.1016/0167-6636(94)90059-0.

- [22] Salvadori, A., Lee, S., Gillman, A., Matou^{*}, K., Salvadori, A., Lee, S., et al., Numerical and Experimental Analysis of the Young 's Modulus of Cold Compacted Powder Materials . Mech Mater 2017 (Accepted)
- [23] Koch, C.C., The synthesis and structure of nanocrystalline materials produced by mechanical attrition: A review. Nanostructured Mater 1993;2:109–29. doi:10.1016/0965-9773(93)90016-5.
- [24] Zhang, D.L., Processing of advanced materials using high-energy mechanical milling. Prog Mater Sci 2004;49:537–60. doi:10.1016/S0079-6425(03)00034-3.
- [25] Tsuji, N., Ito, Y., Saito, Y., Minamino, Y., Strength and ductility of ultrafine grained aluminum and iron produced by ARB and annealing. Scr Mater 2002;47:893–9. doi:10.1016/S1359-6462(02)00282-8.
- [26] Nieh, T.G., Wadsworth, J., Hall-petch relation in nanocrystalline solids. Scr Metall Mater 1991;25:955–8. doi:10.1016/0956-716X(91)90256-Z.
- [27] Meyers, M.A., Mishra, A., Benson, D.J., Mechanical properties of nanocrystalline materials. Prog Mater Sci 2006;51:427–556. doi:10.1016/j.pmatsci.2005.08.003.
- [28] Kleemola, H.J., Nieminen, M.A., On the strain-hardening parameters of metals. Metall Trans 1974;5:1863–6. doi:10.1007/BF02644152.