Winter 2014

BIAXIAL STRESS TESTING OF SS-304L MICROTUBES BY AXIAL LOAD AND INTERNAL PRESSURE

Peter William Ripley
University of New Hampshire, Durham

Follow this and additional works at: https://scholars.unh.edu/thesis

Recommended Citation

This Thesis is brought to you for free and open access by the Student Scholarship at University of New Hampshire Scholars' Repository. It has been accepted for inclusion in Master's Theses and Capstones by an authorized administrator of University of New Hampshire Scholars' Repository. For more information, please contact Scholarly.Communication@unh.edu.
BIAXIAL STRESS TESTING OF SS-304L MICROTUBES

BY AXIAL LOAD AND INTERNAL PRESSURE

BY

PETER WILLIAM RIPLEY

B.S. Kinesiology, The Pennsylvania State University, 2006

THESIS

Submitted to the University of New Hampshire

in Partial Fulfillment of

the Requirements for the Degree of

Master of Science

in

Mechanical Engineering

December, 2014
This thesis has been examined and approved in partial fulfillment of the requirements for the degree of Master of Science in Mechanical Engineering by:

Thesis Director, Ioannis Korkolis, Assistant Professor of Mechanical Engineering
Todd Stuart Gross, Professor of Mechanical Engineering
Yaning Li, Assistant Professor of Mechanical Engineering

On December 5th, 2014

Original approval signatures are on file with the University of New Hampshire Graduate School.
# Table of Contents

LIST OF TABLES ............................................................................................................... v 

ABSTRACT .................................................................................................................... xi 

CHAPTER 1 INTRODUCTION .......................................................................................... 1  
  1.1 Motivation ............................................................................................................... 1  
  1.2 Applications ......................................................................................................... 3  
  1.3 Material Selection ................................................................................................. 4  
  1.4 Research goals ..................................................................................................... 4  
  1.5 Material Properties ............................................................................................. 5  
  1.6 Experimental Setup Design .................................................................................. 5  
  1.7 Rate- & Temperature-Dependent Material Characterization ............................... 6  
  1.8 Biaxial Experiments ............................................................................................. 7  
    1.8.1 Radial Stress Paths ....................................................................................... 7  
    1.8.2 Corner Stress Paths ..................................................................................... 7  

CHAPTER 2 MATERIAL PROPERTIES ....................................................................... 9  
  2.1 Overview .............................................................................................................. 9  
  2.2 Tube Eccentricity ................................................................................................. 10  
  2.3 Grain Structure of the microtubes ....................................................................... 14  
  2.4 Microhardness of the microtubes ........................................................................ 17  
  2.5 X-Ray Diffraction & Martensitic Transformation ............................................. 20  

CHAPTER 3 EXPERIMENTAL SETUP DESIGN AND TESTING .............................. 24  
  3.1 Tensile Stage ....................................................................................................... 24  
  3.2 Pressurization System .......................................................................................... 26  
    3.2.1 General Description ...................................................................................... 26  
    3.2.2 Response under Oscillating Pressure ......................................................... 28  
  3.3 Grips ..................................................................................................................... 38  
    3.3.1 Psylotech µTS Pin Grips ............................................................................. 38  
    3.3.2 Grips for Uniaxial Testing .......................................................................... 39  
    3.3.3 Grips for Isothermal Tension Tests .............................................................. 43  
    3.3.4 Grips for Biaxial Loading ............................................................................ 44
<table>
<thead>
<tr>
<th>Section</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.3.5</td>
<td>Plane-Strain Inflation</td>
<td>46</td>
</tr>
<tr>
<td>3.4</td>
<td>Hydraulic System Components</td>
<td>47</td>
</tr>
<tr>
<td>3.5</td>
<td>Biaxial Controller</td>
<td>50</td>
</tr>
<tr>
<td>3.5.1</td>
<td>Overview of OOP &amp; Actor Framework</td>
<td>50</td>
</tr>
<tr>
<td>3.5.2</td>
<td>Data Acquisition</td>
<td>57</td>
</tr>
<tr>
<td>3.5.3</td>
<td>Sensor Follower Control Type</td>
<td>63</td>
</tr>
<tr>
<td>3.5.4</td>
<td>Threshold Monitoring</td>
<td>68</td>
</tr>
<tr>
<td>3.6</td>
<td>Strain Measurement Systems</td>
<td>77</td>
</tr>
<tr>
<td>3.7</td>
<td>Infrared Temperature Measurement System</td>
<td>78</td>
</tr>
<tr>
<td>3.8</td>
<td>Thermo Scientific Neslab RTE 740 Refrigerated Bath</td>
<td>79</td>
</tr>
<tr>
<td>CHAPTER 4</td>
<td>UNIAXIAL EXPERIMENTS</td>
<td>80</td>
</tr>
<tr>
<td>4.1</td>
<td>Introduction</td>
<td>80</td>
</tr>
<tr>
<td>4.2</td>
<td>Uniaxial Tension of Tubes</td>
<td>80</td>
</tr>
<tr>
<td>4.2.1</td>
<td>Test Methods</td>
<td>80</td>
</tr>
<tr>
<td>4.2.2</td>
<td>Stress and Strain reductions</td>
<td>81</td>
</tr>
<tr>
<td>4.2.1</td>
<td>Results</td>
<td>85</td>
</tr>
<tr>
<td>4.3</td>
<td>Strip Uniaxial Tension</td>
<td>92</td>
</tr>
<tr>
<td>4.4</td>
<td>Isothermal Uniaxial Tension</td>
<td>96</td>
</tr>
<tr>
<td>4.5</td>
<td>Isothermal Tests for Strain-Rate-Sensitivity</td>
<td>107</td>
</tr>
<tr>
<td>CHAPTER 5</td>
<td>BIAXIAL EXPERIMENTS</td>
<td>116</td>
</tr>
<tr>
<td>5.1</td>
<td>Introduction</td>
<td>116</td>
</tr>
<tr>
<td>5.2</td>
<td>Experimental Setup and Procedure</td>
<td>116</td>
</tr>
<tr>
<td>5.2.1</td>
<td>Test Methods</td>
<td>116</td>
</tr>
<tr>
<td>5.2.2</td>
<td>Control Methods</td>
<td>117</td>
</tr>
<tr>
<td>5.2.3</td>
<td>Stress and Strain Reductions</td>
<td>123</td>
</tr>
<tr>
<td>5.2.4</td>
<td>Specimen Preparation</td>
<td>125</td>
</tr>
<tr>
<td>5.3</td>
<td>Results - Radial Paths</td>
<td>128</td>
</tr>
<tr>
<td>5.3.1</td>
<td>Yield Function Fitting</td>
<td>137</td>
</tr>
<tr>
<td>5.4</td>
<td>Results - Corner Paths</td>
<td>147</td>
</tr>
<tr>
<td>CONCLUSIONS</td>
<td></td>
<td>155</td>
</tr>
<tr>
<td>LIST OF REFERENCES</td>
<td></td>
<td>160</td>
</tr>
<tr>
<td>APPENDIX A</td>
<td></td>
<td>163</td>
</tr>
</tbody>
</table>
LIST OF TABLES

Table 2.1: Chemical Composition of SS-304L from Material Certification sheet in Appendix A. ........................................................................................................................................10
Table 2.2 Microhardness Testing Statistics. ..................................................................................................................................................20
Table 3.1: Psylotech µTS meso-scale tensile stage specifications........................................................25
Table 3.2: Teledyne Isco 65D Syringe Pump Specifications [37]. .......................................................28
Table 3.3: Testing parameters and results from Teledyne frequency testing.................................33
Table 3.4: Mechanical Properties of Biaxial Grip Material EOS Stainless Steel PH1 [41]. ........................................................................................................................................46
Table 5.1: %Err for von Mises & Yld2000-2D yld function models at 75MPa plastic work. .............................................................................................................................................143
Table 5.2: %Error for von Mises & Yld2004-3D yield function models at 75MPa plastic work. .............................................................................................................................................145
LIST OF FIGURES

Figure 1.1 Microforming challenges ([6],[7]).................................................................................. 2
Figure 1.2: Tube hydroforming process (Courtesy of Vojtech Kubec)........................................... 3
Figure 1.3 Hydroformed microcomponents from stainless steel SS-304 [9]............................... 4
Figure 2.1: Tube drawing over a floating mandrel [31]..................................................................10
Figure 2.2: Micrograph of tube wall thickness with digital caliper measurement.......................11
Figure 2.3: Tube wall thickness versus angular position for four different tube specimens. An average tube thickness is identified at the dashed red line..............................................12
Figure 2.4: Wall thickness as a percent difference from the average..........................................13
Figure 2.5: Optical microscopic image of grain structure of SS-304L microtubes in the radial-axial (R-Z) plane........................................................................................................15
Figure 2.6: Optical Micrograph of grain structure of SS-304L microtubes in the radial-hoop (R-θ) plane..........................................................................................................................16
Figure 2.7: Grain size determination in radial-axial (R-Z) plane by ASTM E112 circle intercept procedure............................................................................................................................17
Figure 2.8: Microhardness indentation with 50 gf indentation force...........................................18
Figure 2.9: Microhardness indentations with 300 gf indentation force........................................18
Figure 2.10: Vickers Hardness around the circumference of the tube..........................................20
Figure 2.11: Crystal structures of two allotropes, Austenite (FCC) and Martensite (BCC), found in 304 & 304L Grade Stainless Steels [35]...........................................................................21
Figure 2.12: X-Ray diffraction results of a fractured uniaxial tension specimen from SS-304L pulled to 60% axial strain. Below the main plot are two rows of markers which indicate expected spikes of intensity corresponding to the presence of martensite (top row) or austenite (bot. row). Only austenite is observed..................................................22
Figure 3.1: µTS, A meso-scale electromechanical tensile stage purchased from Psylotech..........................24
Figure 3.2: (a) Teledyne Isco Model 65D Syringe Pump and controller [37]. (b) Internal schematic of pump operation [38]. ......................................................................................................27
Figure 3.3: Oscillation of internal pressure in pulsating hydroforming of tube from Mori, 2007 [39]. ..........................................................................................................................................27
Figure 3.4: Teledyne frequency testing for 0.1Hz and 10 +/-3ksi pressure signal at 65ml initial volume (a). Zoom at 1st peak (b). ...........................................................................................................35
Figure 3.5: Teledyne frequency testing for .2Hz 10 +/-3ksi pressure signal at 65ml Initial Volume. Pressure Signal (a). Flow Rate (b). ..................................................................................................36
Figure 3.6: Teledyne frequency testing for 2Hz 10 +/-3ksi pressure signal at 65ml Initial Volume. ........................................................................................................................................36
Figure 3.7: Teledyne Isco 65D Syringe Pump Frequency Testing Analysis.................................37
Figure 3.8: Psylotech µTS pin grips and features..........................................................38
Figure 3.9: Uniaxial Tension Grip fabricated from spherical ball joint and other parts. ...40
Figure 3.10: Spherical ball joints purchased from McMaster-Carr modified to be used as
uniaxial tension grips on µTS..........................................................................................41
Figure 3.11: Custom gland with sketch of 3/32” Taper Seal© HiP female opening Details.
Identical details were machined into the thread rod insert.................................................42
Figure 3.12: 3-56 Male Pipe x 1/16” ID Barb Tube fitting added to uniaxial grips to create
an Isothermal Uniaxial Tension Grip.................................................................................44
Figure 3.13: Isometric view of biaxial grips (a). Side view cross section
showing plunger pin holes and hydraulic port details (b)..................................................45
Figure 3.14: Plane strain test fixture and specimen. ..........................................................47
Figure 3.15: Plane-strain testing hydraulic schematic.........................................................48
Figure 3.16: Pressurization system components. .................................................................49
Figure 3.17: Parent method of Actor Core.vi.................................................................54
Figure 3.18: Move Message class (a) and private data control (b). .................................55
Figure 3.19: Send.vi method of Move Msg class. ..............................................................56
Figure 3.20: Do.vi method of Move Msg class. .................................................................57
Figure 3.21: Raw Sensor configuration in systems.ini file after modifications.........58
Figure 3.22: Text added to system.ini file for pressure user sensor addition.............59
Figure 3.23: Psylotest project tree showing the additions of Hoop Stress and Axial Stress
classes to the User Sensor types (a) and their private data (b) which is identical for both
classes. ....................................................................................................................60
Figure 3.24: Axial Stress and Hoop Stress additions to the systems.ini file. ..............60
Figure 3.25: Hoop Stress Parse Setup Strain override.vi...................................................61
Figure 3.26: Sensors Pre-Converted Raw.vi from the hoop stress user sensor class. ...62
Figure 3.27: Sensors Pre-Converted Raw.vi from the axial stress user sensor class....63
Figure 3.28: Sensor Follower class shown in project tree with override vi’s (a) and
private data (b). ..............................................................................................................64
Figure 3.29: Init by Ref.vi for the Sensor Follower class. ................................................65
Figure 3.30: Return Move String.vi of the Sensor Follower class. .................................66
Figure 3.31: Update Parameter Reference.vi of the Sensor Follower class. ...............68
Figure 3.32: Setpoint.vi of the Sensor Follower class. ....................................................68
Figure 3.33: Project tree showing addition of Threshold class to the Non-Actor Classes
directory as well as the Threshold child classes in the Threshold Types directory (a).
Private data cluster for the parent Threshold class (b). ..................................................69
Figure 3.34: Threshold creation section of test profile tab on user interface.............70
Figure 3.35: Move Array class private data with array of thresholds added. ...............71
Figure 3.36: Start Test Motion.vi in Test Handler...........................................................72
Figure 3.37: Controller Loop.vi showing “New Threshold” case. ..................................................72
Figure 3.38: Threshold class added to systems class private data. ................................................73
Figure 3.39 No internal state message case structure of Controller Loop.vi. ..........................73
Figure 3.40: Process Threshold.vi checks the threshold sensor against the threshold...73
Figure 3.41: 3rd and 4th case structures in Process Threshold.vi. False (a) & True (b)...74
Figure 3.42: “Threshold Reached” state in controller loop.vi of Motion Controller. .........75
Figure 3.43: Threshold Reached.vi on Drive Comm Actor. .....................................................76
Figure 3.44: Process Motion State.vi for Drive Comm Actor. ..................................................77
Figure 3.45: Point Grey Research GRAS 20S4MC 2.0 Megapixel digital camera [42] (a). and Schneider–Kreuznach Xenoplan 17mm lenses [43] (b). .................................................78
Figure 3.46: FLIR SC645 Infrared camera [44]. .................................................................78
Figure 3.47: Thermo Scientific Neslab RTE 740 Refrigerated Bath [45]. ...............................79
Figure 4.1: Virtual extensometers measuring axial and hoop strain (a). Diagram of virtual extensometer measuring hoop strain by change in chord length (b). ..............................82
Figure 4.2 Nominal stress vs strain for uniaxial tension experiments. .................................85
Figure 4.3: Strain-rates vs strain for uniaxial tension tests.....................................................86
Figure 4.4: Uniaxial tension true stress vs strain.................................................................87
Figure 4.5: Evolution of diameter (Top). 3D DIC Line Plot (Bot). .........................................88
Figure 4.6: Evolution of axial strain vs. normalized gauge length during uniaxial tension test. .........................................................................................................................89
Figure 4.7: Evolution of tube radius vs normalized axial position during uniaxial tension test. .......................................................................................................................90
Figure 4.8: Multiple bifurcations instability during uniaxial testing (a). Box tool extractions from 3D DIC data (b). .........................................................................................91
Figure 4.9: Mock-up of the strip specimen slitting process....................................................93
Figure 4.10: Strip specimen after and before test. .................................................................94
Figure 4.11: Fractured strip specimen in μTS pin grips..........................................................94
Figure 4.12: Premature failure of uniaxial strip tensile specimens........................................96
Figure 4.13: Infrared images for monotonic uniaxial tension test at $10^2$ s$^{-1}$ ...............98
Figure 4.14: Isothermal nominal stress vs strain at different temperatures. ......................100
Figure 4.15: Temperature at location of the neck vs normalized time during isothermal tension testing. ........................................................................................................101
Figure 4.16: Temperature profile of tube immediately before fracturing during isothermal testing....................................................................................................................102
Figure 4.17: Strain-rate vs strain for isothermal testing.........................................................103
Figure 4.18: Zoomed-in strain-rate vs strain for isothermal testing.................................104
Figure 4.19: Flow stress vs temperature at different levels of true strain.........................105
Figure 4.20: Ultimate strength vs temperature for uniaxial tension specimens................106
Figure 4.21: Uniform strain vs. temperature for uniaxial tension specimens. ...............106
Figure 4.22: Nominal stress vs strain at different strain-rates and a constant temperature of 25 °C .........................................................................................................................108
Figure 4.23: Logarithmic strain-rate vs. strain for 25 °C isothermal rate testing. .........108
Figure 4.24: Comparison of 25 °C Isothermal and monotonic nominal stress vs strain response. .........................................................................................................................110
Figure 4.25: Isothermal and Monotonic uniaxial tension specimens (1.5 x 10^{-2} s^{-1}).....110
Figure 4.26: Temperature profile of tube at different levels of axial strain for the monotonic (a) and isothermal (b) uniaxial tension tests. .................................................111
Figure 4.27: Temperature vs. normalized gauge length at different strain-rates for 25 °C isothermal testing. ........................................................................................................113
Figure 4.28: Temperature at neck vs. normalized time for 25 °C isothermal testing. .....114
Figure 4.29: Flow stress vs logarithmic strain-rate at different levels of axial strain for isothermal rate testing. ........................................................................................................115
Figure 4.30: Ultimate strength vs logarithmic strain-rate for isothermal rate testing. ....115
Figure 5.1: Control schematic of biaxial experimental setup. ......................................118
Figure 5.2: Comparison of biaxial stress testing control methods. .............................120
Figure 5.3: Biaxial experiment stress paths. .................................................................122
Figure 5.4: Biaxial test specimen in μTS immediately prior to testing.........................127
Figure 5.5: Nominal hoop vs axial stress paths...........................................................129
Figure 5.6: True hoop vs axial stress path. .................................................................130
Figure 5.7: Nominal hoop strain vs. axial strain. .........................................................131
Figure 5.8: Failure modes of biaxial stress paths. 1:4 CW to 1:0 (a), 0:1 CCW to -1:5(b). .................................................................................................................................133
Figure 5.9: Nominal axial stress vs. strain. .................................................................134
Figure 5.10: Nominal Axial Stress vs. Strain zoomed in near the yield point and truncated thereafter. ........................................................................................................135
Figure 5.11: True axial stress vs. strain. .................................................................135
Figure 5.12: Nominal hoop stress vs. strain. ...............................................................136
Figure 5.13: Nominal hoop stress vs. strain zoomed in near the yield point and truncated thereafter. ........................................................................................................137
Figure 5.14: True hoop stress vs strain......137
Figure 5.15: Plastic work contours and von Mises yield function.................................139
Figure 5.16: Differential work hardening with Von Mises...........................................140
Figure 5.17: Direction of plastic strain vs loading direction and Von Mises. ...............141
Figure 5.18: Normalized plastic work contours with Von Mises and optimized...........143
Figure 5.19: Normalized plastic work contours with Von Mises & optimized .........145
Figure 5.20: Normalized plastic work contours with optimized Yield 2000-2D & Yield 2004-3D. ..........................................................146
Figure 5.21: Nominal hoop vs axial stress. .................................................................148
Figure 5.22: True hoop vs. axial stress. .................................................................148
Figure 5.23: Nominal hoop strain vs axial strain. ..................................................150
Figure 5.24: Nominal stress vs strain.................................................................151
Figure 5.25: True axial stress vs strain.................................................................152
Figure 5.26: Nominal hoop stress vs. strain.........................................................153
Figure 5.27: True hoop stress vs. strain.................................................................154
ABSTRACT

BIAXIAL STRESS TESTING OF SS-304L MICROTUBES UNDER AXIAL LOAD AND INTERNAL PRESSURE

by

Peter William Ripley

University of New Hampshire, December, 2014

The mechanical behavior and material properties of a Stainless Steel SS-304L microtube, with an OD of 2.40 mm and wall thickness of 160 µm, was investigated through uniaxial, isothermal, biaxial, and metallographic testing. The grain structure, microhardness, and tube eccentricity were investigated using optical microscopy. The rate- and temperature-dependence of the material was characterized by isothermal uniaxial tension experiments. A biaxial experimental setup, consisting of a 2 kN electromechanical tensile stage and a 1.4 kbar hydraulic pump, was created to internally pressurize and axially load the microtube in biaxial stress states. Fourteen radial nominal stress path tests were conducted to determine the formability, failure mode, and anisotropy during biaxial stress states. The Yld2000-2D and Yld2004-3D yield functions were fit to the data at the initial yield surface and higher levels of plastic work. The path-dependence of failure stresses and strains was investigated by comparing radial path results to corner paths.
CHAPTER 1
INTRODUCTION

1.1 Motivation

As devices shrink in size, the demand for microscale components has increased in recent years. Many of the microforming techniques used to manufacture these parts are simply scaled down versions of conventional macroscale processes, such as extrusion, stamping, and hydroforming ([1],[2],[3],[4]). In addition to the challenges outlined schematically in Figure 1.1, problems arise in these manufacturing processes when the magnitude of the part dimensions shrink to that of its microstructural length scale (e.g., grain size) and surface topography. Both of these often remain unchanged, or scale at a slower rate, when the overall dimensions shrink. At a dimension-to-grain-size ratio less than 10, we suspect the material behavior of these parts to be dominated by the individual, highly anisotropic grains. Furthermore, surface grains flow plastically at lower stress levels than internal, fully constrained ones [5]. As the proportion of these surface grains increases relative to the total population, the deformation of a microcomponent becomes more inhomogeneous than that of its macroscale relative.

Together these problems justify adopting a modeling approach that is microstructurally-informed rather than based on a homogeneous continuum assumption. They can also lead to a reduction in formability due to deformation-induced surface roughening and the resulting increased workpiece-die friction. Some commercially available materials such as stainless steel SS-304 and SS-304L can be heat treated to circumvent these problems by achieving a finer grain size, but this drastically increases time and production costs. Furthermore, other materials (e.g., platinum alloy microtubes for biomedical applications) are less receptive to grain refinement by similar means.
Hence understanding the inhomogeneous deformation in micro-scale components and including it in microforming process modeling is an important ongoing research effort in the community, which requires microstructurally-informed material models.

Figure 1.1 Microforming challenges ([6],[7]).

The research presented in this thesis serves as the beginning stages to a larger project with the objective to, 1) experimentally investigate the behavior of 304 stainless steel and CuZn30 brass when the dimension to grain size ratio is less than 10 under simple, well-controlled biaxial stress states, as well as during microforming processes (microtube bending and hydroforming), and 2) establish microstructurally-informed material models coupled to finite element models, to simulate the material behavior and predict failure.
1.2 Applications

In the microtube hydroforming (µTHF) process a microtube is placed inside a die and inflated with hydraulic pressure to expand and conform to the shape of the surrounding die as shown in Figure 1.2. The µTHF process is used in the production of components for medical devices (needles, catheters, microtubes for drug delivery, micropipettes), microfluidics (cooling channels for microchips, micro heat exchangers, fuel cell bipolar plates, fuel injectors), micromechatronics (shafts and components for micro-actuators and cameras) and telecommunications (sheaths for optical fiber cables) [8]. Three microtube components are shown below in Figure 1.3, which were hydroformed from tubes and show the complex geometrical features capable of this technique. Presently there is very limited understanding of the µTHF process with virtually no mapping of their forming limits and possible failure mechanisms.

Figure 1.2: Tube hydroforming process (Courtesy of Vojtech Kubec)
1.3 Material Selection

Stainless steel SS-304L, a lower carbon content variation of SS-304, was chosen as the material to be investigated for this research. This material has superior corrosion resistance, biocompatibility, ease of cleaning, and high strength and toughness which make it an excellent candidate for biomedical applications. Furthermore, the material has high ductility, 50-70%, in the fully annealed state, making it an excellent choice for any type of forming process. Seamless, fully annealed stainless steel SS-304L tubes with nominal outside diameter and wall thickness dimensions of 2.38 mm (3/32 in.) and 150 µm (.006 in.) was purchased from Microgroup, Inc. (Medway, MA) for this research.

1.4 Research goals

The primary research objectives of this thesis were to 1) Design and develop an experimental setup capable of conducting well-controlled biaxial stress experiments on the stainless steel SS-304L microtube, 2) characterize the material properties of the microtube in its as-received state, and 3) experimentally investigate the mechanical behavior of the microtube under uniaxial and biaxial stress states.

Figure 1.3 Hydroformed microcomponents from stainless steel SS-304 [9].
1.5 Material Properties

The microtube was purchased from a commercial supplier of biomedical tubes and the vast majority of the material properties were not known a priori; therefore in Chapter 2 we investigated the grain orientation and size, hardness, strain-induced martensitic transformation, and the tube geometry by optical microscopy and other metallographical methods. The tube geometry was required to accurately calculate the stress states of the tube for the uniaxial and biaxial testing in Chapters 4 and 5. Furthermore, understanding the material properties was essential to interpret and understand the results of the experimental testing. It was discovered that the tubes supplied where not oligocrystals (few grains), but had approximately 10-12 grains through the thickness. This was a welcome finding, since the experiments could be simulated with continuum material models and hence provide a link to earlier work in our group [10]. In the future, the tubes will be heat-treated to grow the grains to only a few through the thickness, and this work will be expanded to examine this oligocrystalline material.

1.6 Experimental Setup Design

In Chapter 3 the design and fabrication of an experimental setup capable of uniaxial, isothermal, plane-strain, and biaxial stress tests was documented. The setup mainly consisted of a meso-scale 2kN tensile stage (Psylotech, Inc., Evanston, IL) used to load the microtube in axial tension or compression, and a hydraulic pump (Teledyne ISCO, Lincoln, NE) that internally pressurized the microtube with fluid for biaxial experiments. The object-oriented LabVIEW control software for the tensile stage provided by its manufacturer was modified to capture and control the axial and hoop stress in the microtube for biaxial stress testing. The deformation of the tube was captured with a 2D/3D Digital Image Correlation (DIC) system from Correlated Solutions, Inc (Columbia,
SC). Other equipment, such as custom grips, a refrigerated bath (Neslab Inc., Newington, NH) used to circulate fluid through the tube for isothermal testing, and custom hydraulic parts and connections were also described.

1.7 Rate- & Temperature-Dependent Material Characterization

Stainless steel SS-304L is known to be rate-dependent and prone to deformation-induced heating [11], as well as strain-induced martensitic transformation [12]. These material properties need to be captured in order to build accurate numerical (FEA) models. In Chapter 4 isothermal uniaxial tension tests were conducted to decouple and capture the rate and temperature effects on the response of the microtube. Uniaxial tension tests were conducted at a constant strain-rate of $1.5 \times 10^{-3} \text{ s}^{-1}$ while the tube was held at constant uniform temperatures of 6, 25, 50, 76, 100, & 142 °C. Conversely, uniaxial experiments were also conducted at a constant temperature of 25 °C for a range of strain-rates from $10^{-5}$ to $10^{-1} \text{ s}^{-1}$. Relationships between flow stress, uniform strain, ultimate strength, and total elongation to temperature and strain-rate were established.

Cullen, et al. [13] executed a similar testing scheme at the macroscale on ASTM E-8 standard specimens made of stainless steel SS-304 to capture the aforementioned phenomena. The material dependencies were implemented into thermo-mechanical numerical models in Abaqus and were able to produce the same response as the experimental results. Similar numerical models will be built in the future to simulate the microtube uniaxial isothermal experiments and verify their accuracy, and that the rate- and temperature-dependent properties of the microtube have been captured correctly.
1.8 Biaxial Experiments

1.8.1 Radial Stress Paths

In addition to characterizing the basic material properties, experimental data under multi-axial stress states are needed to calibrate constitutive models. These material models make it possible to develop numerical simulations capable of predicting failure of the microtube during actual forming processes. This type of work has been carried out on macro scale Aluminum Al-6260-T4 tube with an outside diameter of 60mm (2.36 in.) and wall thickness of 2mm (0.080 in.) by Korkolis and Kyriakides [14]. In this research, the tube was subjected to radial (i.e., proportional) paths in the axial-hoop nominal stress plane, to establish the plastic anisotropy, the failure modes and the forming limits. In Chapter 5 similar nominal stress radial paths were prescribed to our microtube through a combination of axial loading and internal pressure. Fourteen different experimental paths ranging from 1:0 to -1:5 (axial:hoop nominal stress) were conducted, which populated the first and some of the second quadrant of the plane-stress space. Two failure modes were identified that agreed with the two modes determined by Korkolis and Kyriakides ([10],[14]), and the forming limits of the SS-304L microtubes were established. The Yld-2000-2D and Yld-2004-3D [15], anisotropic yield functions were optimized to fit different levels of plastic work contours from the biaxial experimental data. In the future, numerical models will incorporate these yield functions to predict failure and to simulate the biaxial experiments and μTHF.

1.8.2 Corner Stress Paths

Experimental research has proven that failure limits based on strain are highly path-dependent, therefore the forming limit diagrams generated from these failure limits are limited in scope to the forming processes with similar paths ([16],[17],[18],[19]). More
recent research has confirmed and augmented these original discoveries ([20],[21],[22],[23]). In an effort to develop more effective predictors of forming limits, it has been postulated that the failure stresses are not path dependent ([24],[25],[26],[27]). Yoshida et al. have shown experimental research that supports this notion [28], but also research that suggests it has shortcomings ([29],[30]). Korkolis and Kyriakides [10] have shown by comparing corner and radial stress paths that, when the pre-strain is minimal, the failure stress of Al-6260-T4 is significantly less path-dependent than the failure strain, and therefore can be used as a better metric to predict the forming limits. In Chapter 5 we investigated the path dependence of the microtube formability by examining corner paths through the failure stresses of four corresponding radial paths. Corner path tests were conducted for the 10:9, 1:1, 5:4, and 4:3 (axial:hoop nominal stress) nominal stress radial paths. The 10:9 and 1:1 paths began along the axial stress axis before turning at the corresponding radial path failure stress, and increasing the hoop stress until failure. The 5:4 and 4:3 corner stress paths began along the hoop stress axis before turning at the corresponding radial path failure stress, and increasing the axial stress until failure. Future numerical FEA models will simulate these corner path experiments as a benchmark for accuracy.
CHAPTER 2
MATERIAL PROPERTIES

2.1 Overview

Stainless steel SS-304L was chosen as the material for this research because of its widespread availability in the desired dimensions and its beneficial properties, such as biocompatibility, high formability, high strength and toughness, and superior corrosion resistance, to the applications aforementioned in the introduction. A comprehensive evaluation of potential tube vendors and availability was conducted, whereby geometry and bursting pressure were the foremost sought specifications. A tube of outside diameter between 2 and 3 mm was desired. Limitations of commercially available low flow, high pressure hydraulic systems above 1,069 bar (20 ksi) constrained the possibilities to a fully annealed tube with minimal thickness to outside diameter ratio, i.e., as thin-walled as possible. The commercially available low-flow pressurization systems would not be capable of bursting thick-walled tubes that had been work-hardened and sold in the "Hard" state.

After careful consideration, 15.24 m (50 ft.) of 304F10093X006SL seamless, fractional, fully annealed stainless steel 304L tube was purchased from Microgroup. The tubes have outside diameter and wall thickness nominal dimensions of 2.38 mm (3/32") and 0.15 mm (0.006") respectively. The tubes are manufactured through a combination of extrusion and drawing processes, whereby in the final deformation step they are drawn through a die over a mandrel as shown in Figure 2.1. This manufacturing process does not ensure precise tolerances on tube geometry, at least at the sizes considered. Indeed, the tolerances on the outside diameter and wall thickness are given by MicroGroup as +/- 0.127mm and +/- 15%, respectively.
The chemical composition of our material was provided as a material certification, APPENDIX A., of our batch of tube by the manufacturer and is shown below in Table 2.1. Stainless steel SS-304L is a variation of the SS-304 grade with a carbon content less than .03% that eliminates carbide precipitation due to welding and slightly lowers the strength [32]. This variation has no impact on the research that was performed.

<table>
<thead>
<tr>
<th>C%</th>
<th>Mn%</th>
<th>P%</th>
<th>S%</th>
<th>Si%</th>
<th>Ni%</th>
<th>Cr%</th>
<th>Al%</th>
<th>Fe%</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.015</td>
<td>1.43</td>
<td>0.029</td>
<td>0.008</td>
<td>0.4</td>
<td>10.85</td>
<td>18.67</td>
<td>0.003</td>
<td>Balanc</td>
</tr>
</tbody>
</table>

### 2.2 Tube Eccentricity

The variation in wall thickness of the tubes was investigated using digital calipers on optical micrographs. Four pieces in total were cut from four of the ten tube sections received, and mounted in a steel puck in order to prepare the specimens for optical microscopy. The specimens were polished with 180, 240, 320, 400, 600 and 1200 grit SiC paper and 0.3 and 0.05 µm aluminum oxide powder solutions. A Nikon Epiphot
inverted microscope configured with a digital camera was used to view the specimens and capture images. Using the digital calipers tool in the microscope software toolbox the wall thickness was measured around the circumference of each specimen. An example of this measurement is shown below in Figure 2.2.

![Figure 2.2: Micrograph of tube wall thickness with digital caliper measurement.](image)

Between 19 and 39 measurements were taken around the circumference of each of the four specimens. This data is shown below in Figure 2.3. A sinusoidal-like variation of the wall thickness is observed in the tubes. This is believed to be a remnant of the initial tube-making process for which the finishing processing step, drawing-over-mandrel, has not been able to completely erase. An average wall thickness of 161 µm was calculated from the total data set, and used in all future nominal stress calculations. This wall thickness is well within the tolerances specified by MicroGroup.
Figure 2.3: Tube wall thickness versus angular position for four different tube specimens. An average tube thickness is identified at the dashed red line.

The eccentricity of the tube as a percent difference from the average wall thickness is shown in Figure 2.4. The deviation from average wall thickness is less than 6% around the circumference of the tube which is well within the +/-15% tolerance specified by MicroGroup.
Figure 2.4: Wall thickness as a percent difference from the average.

The outside diameter of the tube was measured for all delivered tube sections to be 2.40 mm using Mitutoyo digital micrometers (No. 293-340 IP65). This measurement is well within the specification by MicroGroup and found to be consistent around the circumference of the tube and for all ten delivered tube sections. This suggests that the inside diameter of the tube varies with the wall thickness.
2.3 Grain Structure of the microtubes

The grain size and the number of grains through the wall thickness of the tubes are important aspects to this research project. Ultimately, the goal is to investigate and characterize the mechanical behavior of microtubes when there are only a few grains through the wall thickness. This direction is beyond the scope of the present thesis.

The grain structure was captured along both the R-θ (radial-circumferential) and R-Z (radial-axial) planes, as a first attempt to examine anisotropy in the crystallographic texture of the supplied tubes. Specimens were mounted in epoxy and polished using sequentially finer grits of SiC paper as well as alumina oxide solutions as described previously in section 2.2. The superior corrosion resistance of stainless steel necessitates aggressive acid etching solutions to reveal the grain structure. Electroetching is an alternative technique which uses significantly less potent acid solutions while a direct current is passed through the etching surface. For our micrographs a 10% Oxalic Acid solution was used as the electrolyte, and 0.217 A of current at 6 VDC were passed through the specimens for 90 seconds. Immediately after electroetching the prepared surfaces were rinsed with water followed by acetone, and finally a hot air gun was used to evaporate the lingering solvents. Micrographs of the two planes are shown in Figure 2.5 and Figure 2.6 below, which reveal the grain structure of the tube material.
The tube extrusion manufacturing process tends to elongate grains in the longitudinal direction. The elongated grains can lead to anisotropic mechanical behavior of the tube, whereby the stress-strain response is different in the axial versus the hoop direction. The micrographs show that the grains are not elongated, presumably since the tube was fully annealed which allows the grains to recrystallize after the extrusion process. The average grain size diameter was determined using the ASTM E112 circle intercept procedure, Figure 2.7, to be 14 µm, which yields 12 grains through the thickness. At this number of grains the use of a continuum approach to model the mechanical behavior of the tube in its as-received state is warranted. Future work will focus on growing the grains so there are only a few through the thickness of the tube.
and comparing the mechanical behavior of the resulting oligocrystal to that of the original state.

Figure 2.6: Optical Micrograph of grain structure of SS-304L microtubes in the radial-hoop (R-θ) plane.
2.4 Microhardness of the microtubes

In addition to variations in eccentricity and grain structure, it is common for the hardness of the tubes to vary around the circumference due to the manufacturing process, where parts of the tube are work-hardened more than others. The tube hardness was probed by microindentations on the radial–hoop (R–θ) surface using a Buehler microhardness tester (model number 1600-6306). The specimen used to evaluate the hardness is one of the same specimens used to measure the wall thickness, therefore see section 2.2 for surface preparation. A Vickers hardness indenter was used with both 50 gf and 300 gf indentation forces. Each indentation force was used for half the circumference of the tube. Examples of these indentations are shown below in Figure 2.8 and Figure 2.9. Recall that the average grain size was determined in the previous section to be 14 µm.
Figure 2.8: Microhardness indentation with 50 gf indentation force.

Figure 2.9: Microhardness indentations with 300 gf indentation force.
The 50 gf indentation force was adopted because it was thought that the larger indentations would report softer values due to influence from the tube wall edges and previous indentation measurements nearby. Care was taken to ensure that the microindentations lied approximately at the mid-line of the microtube cross-section, i.e., they were equidistant from the two free edges of the tube wall. A Vickers hardness value was calculated from the measurement of each indentation’s major and minor dimension [33] and the results around the circumference of the tube are shown below in Figure 2.10.

No hardness pattern was recognizable around the circumference of the tube and there appeared to be no difference between the results of the 50 gf and 300 gf indentations. An average hardness of 207 HV was found for both indentation forces. The standard deviation was slightly higher for the 50 gf indentation results and can be seen in Table 2.2 below along with average, maximum and minimum statistics. The full annealing of the tube after manufacturing most likely resolved any variations in microhardness that may have been introduced by the extrusion and drawing process.
Figure 2.10: Vickers Hardness around the circumference of the tube.

Table 2.2 Microhardness Testing Statistics.

<table>
<thead>
<tr>
<th>Metric</th>
<th>50gf</th>
<th>300gf</th>
<th>Combined</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average</td>
<td>207</td>
<td>207</td>
<td>207</td>
</tr>
<tr>
<td>St. Dev</td>
<td>12.63</td>
<td>12.05</td>
<td>12.05</td>
</tr>
<tr>
<td>Max</td>
<td>224</td>
<td>225</td>
<td>225</td>
</tr>
<tr>
<td>Min</td>
<td>182</td>
<td>167</td>
<td>167</td>
</tr>
</tbody>
</table>

2.5 X-Ray Diffraction & Martensitic Transformation

Stainless steel SS-304L is an austenitic stainless steel which, prior to any work-hardening, contains a primarily Face Centered Cubic (FCC) crystal lattice structure. After work hardening it has been shown by Lichtenfeld et al. [34], among others, that austenite in SS-304L can transform to martensite, which has a Body Centered Cubic (BCC) crystal lattice structure. Strain-induced martensitic transformation has also been shown in
stainless steel SS-304 by Moser et al. [12]. These crystal structures are show in Figure 2.11 below.

![Crystal Structures of Austenite (FCC) and Martensite (BCC)](image)

Figure 2.11: Crystal structures of two allotropes, Austenite (FCC) and Martensite (BCC), found in 304 & 304L Grade Stainless Steels [35].

The transformation of austenite to martensite happens progressively as the material is continuously strained to higher values. This is referred as strain-induced Martensite, where the deformation of the austenitic matrix generates defects that accommodate the formation and growth of martensitic embryos. As shown in De et al. [36], during deformation the $\gamma$-austenitic matrix (FCC) transforms to two forms of martensite: BCC/BCT-martensite ($\alpha'$) and HCP-martensite ($\epsilon$). However, as shown by De, the HCP-martensite progressively transforms to BCC/BCT-martensite, so that in a specimen deformed to failure only the latter is expected to be found. This observation by De is consistent with the measurements of Moser.

We expected that our tubes would be initially comprised solely of austenite, since the annealing would have allowed the grains to fully recrystallize after the forming of the tube by the extrusion and drawing process. However, we expected to be able to reintroduce this transformation by pulling our tube in uniaxial tension.
A microtube specimen was pulled in uniaxial tension until fracture (elongation-to-fracture of approx. 60%). A portion of the test-section of the deformed specimen was scanned using X-Ray diffraction to determine the crystal structures present. A Shimadzu XRD 6100 X-Ray Diffractometer using Cu $K_{\alpha}$ radiation was used to perform the measurement. The divergence slit, scattering slit, and receiving slit of the diffractometer were set to 2°, 2°, and 0.3 mm respectively. The specimen was scanned between $2\theta$ angles of 30° and 100° at a rate of 2°/min using steps of 0.05°. The results are presented below in Figure 2.12.

![Image of X-Ray diffraction results]

Figure 2.12: X-Ray diffraction results of a fractured uniaxial tension specimen from SS-304L pulled to 60% axial strain. Below the main plot are two rows of markers which indicate expected spikes of intensity corresponding to the presence of martensite (top row) or austenite (bot. row). Only austenite is observed.
In Figure 2.12 above, the intensity of diffraction peaks is plotted versus the diffraction angle. We expect to find intensity spikes at specific angles which correspond to the presence of austenite, martensite, as well as any other phase in the material. These angles are marked by lines below the plot in the two rectangular boxes labeled Iron-Fe (Martensite) and Iron-FCC-Fe (Austenite). The plot shows there are intensity spikes at 44°, 51°, 74°, 90° and 95°, which correspond to the presence of austenite. No diffracted peaks were found that indicated the presence of martensite. Furthermore, the specimen shows no signs of magnetism in the deformed state. Both results were not expected given prior research of Lichtenfeld et al. [34] on martensitic transformation in SS-304L. While an interesting finding, this direction was not pursued further as it deviated significantly from the primary focus of the research reported in this thesis.
CHAPTER 3
EXPERIMENTAL SETUP DESIGN AND TESTING

3.1 Tensile Stage

A meso-scale tensile stage was purchased from Psylotech in Evanston, IL and is shown below in Figure 3.1. The Under-microscope Test System (µTS) is equipped with a tension-compression, capacitive-based load cell with +/- 2000N capacity, 10 mN resolution and up to 1mN resolution when operating in a “Window” control mode. A complete list of specifications is shown in Table 3.1. The moving crosshead

Figure 3.1: µTS, A meso-scale electromechanical tensile stage purchased from Psylotech
translates on a high precision ball screw driven directly by a Kollmorgen AKD-P00306-NAEC000 servomotor and drive, and is capable of speeds ranging from 2nm to 100mm per second. On the back of the moving crosshead is a capacitive-based position sensor to measure displacement and velocity of the moving crosshead directly. The motor can be controlled serially through the servo drive in a motor encoder feedback control loop, or by an external analog signal.

The user interfaces with the drive and sensors through a LabVIEW-based control system called Psylotest, and a manual control pendant. A user can build multistage testing programs with any combination of ramp, hold, sine, etc. functions for displacement, velocity or force. In Psylotest, the user can select to control the motor servo-drive through either serial commands or an analog signal. The former case is the default one for controlling a servo-motor and uses the built-in encoder on the motor. However, for material testing applications, it can be advantageous to control the motor based on the actual displacement, velocity or force that is induced on the specimen, e.g., to compensate for the compliance of the load-train. For this purpose, Psylotest includes a closed-loop PID controller which issues an analog signal to the servo-drive.

Table 3.1: Psylotech µTS meso-scale tensile stage specifications.

<table>
<thead>
<tr>
<th>Specifications</th>
<th>Value</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Force Capacity</td>
<td>2000</td>
<td>N</td>
</tr>
<tr>
<td>Load Resolution (“Full Scale Mode”)</td>
<td>10</td>
<td>mN</td>
</tr>
<tr>
<td>Load Resolution (“Window Mode”)</td>
<td>1</td>
<td>mN</td>
</tr>
<tr>
<td>Stroke</td>
<td>50</td>
<td>mm</td>
</tr>
<tr>
<td>Full Scale Resolution (“Window Mode”)</td>
<td>250</td>
<td>nm</td>
</tr>
<tr>
<td>Displacement Resolution (“Window Mode”)</td>
<td>25</td>
<td>nm</td>
</tr>
<tr>
<td>Minimum Displacement Rate</td>
<td>1</td>
<td>nm/s</td>
</tr>
<tr>
<td>Maximum Displacement Rate</td>
<td>100</td>
<td>mm/s</td>
</tr>
<tr>
<td>Peak Acceleration</td>
<td>10</td>
<td>m/s²</td>
</tr>
<tr>
<td>Analog Sensor Outputs</td>
<td>BNC</td>
<td></td>
</tr>
<tr>
<td>Footprint</td>
<td>400 X 200 X 75</td>
<td>mm</td>
</tr>
<tr>
<td>Control Loop</td>
<td>500</td>
<td>Hz</td>
</tr>
<tr>
<td>Power Requirements</td>
<td>120/240, 60/50</td>
<td>V, Hz</td>
</tr>
</tbody>
</table>

25
3.2 Pressurization System

3.2.1 General Description

A low flow high pressure hydraulic pump was required to burst the tubes in a well-controlled manner. A survey of the commercially available pumps capable of low flow \((2.5\times10^{-3}\text{ ml/min})\), high pressure \((1,390\text{ bar – 20 ksi})\) revealed a very short list of prospects. “High Pressure Generators” from HiP and Kistler had ideal mechanical designs, but would require time consuming modifications to integrate a motor and feedback control system to drive these manually operated pumps. The Teledyne Isco syringe pumps offered a similar cylinder & plunger design as the HiP and Kistler pressure generators, but also included automation of the pump with a motor and controller. The Teledyne Isco 65D syringe Pump, controller, and internal schematic are shown below in Figure 3.2. The electric motor of the pump moves a piston connected to a hollow rod (termed “push tube”) through a ball screw and a gear train. The piston moves along the cylinder to generate flow by decreasing the system volume. An optical encoder tracks the motor position and provides feedback to the controller for volume and flow rate. A Honeywell TJE pressure transducer, with +/- 1.4 bar (20 psi) accuracy, provides feedback to the controller for pressure-control.

The pump has a large range of flow rates from .01 µl/min up to 25 ml/min at 1,390 bar (20 ksi). The total capacity of the pump is 67 ml, but it can be operated at lower volumes to reduce the effective compressibility of the system and increase its overall stiffness. The pump has analog outputs for pressure and an optional circuit board on the controller can
output analog signals for flow rate and volume. The controller accepts analog signals and DASNET serial commands for pressure and flow rate control. Teledyne Isco supplies LabVIEW sub-VI’s (“Virtual Instruments”) allowing the user to create their own LabVIEW control program to send commands to the pump serially without having to write the DASNET serial communication code. Table 3.2 below contains a full list of pump specifications [37].
Table 3.2: Teledyne Isco 65D Syringe Pump Specifications [37].

<table>
<thead>
<tr>
<th>Specification</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Capacity:</td>
<td>67 ml</td>
</tr>
<tr>
<td>Flow Range:</td>
<td>0.01 µl/min to 25 ml/min</td>
</tr>
<tr>
<td>Flow Accuracy:</td>
<td>±0.3% of setpoint</td>
</tr>
<tr>
<td>Displacement Resolution:</td>
<td>2.5 nl/step</td>
</tr>
<tr>
<td>Motor Stability:</td>
<td>± 0.001% per year</td>
</tr>
<tr>
<td>Pressure Range:</td>
<td>1,390 bar (20,000 psi)</td>
</tr>
<tr>
<td>Pressure Accuracy:</td>
<td>1.4 bar (20 psi)</td>
</tr>
<tr>
<td>Wetted Materials (standard):</td>
<td>Nitronic 50, PTFE, Hastelloy C-276</td>
</tr>
<tr>
<td>Plumbing Ports:</td>
<td>1/4&quot;, F250</td>
</tr>
<tr>
<td>Operating Temperature:</td>
<td>5 - 40° C Ambient</td>
</tr>
<tr>
<td>Power required:</td>
<td>100 Vac, 117 Vac, 234 Vac, 50/60 Hz (specify)</td>
</tr>
<tr>
<td>Dimensions (HxWxD, cm):</td>
<td>103 x 27 x 45</td>
</tr>
<tr>
<td>Weight:</td>
<td>Pump module - 33 kg; controller - 3 kg</td>
</tr>
<tr>
<td>Standards conformity:</td>
<td>UL</td>
</tr>
</tbody>
</table>

### 3.2.2 Response under Oscillating Pressure

In addition to the low flow, high pressure requirements of the pump for biaxial stress testing, the capacity to generate an oscillating pressure was desired. In 2007, a paper published by Mori and coworkers [39] demonstrated that the formability of a tube can be increased for tube hydroforming by oscillating the internal pressure. Figure 3.3 below shows the internal pressure history prescribed during free tube inflation by Mori and coworkers. One of the future goals of this project is to explore this approach. Teledyne Isco volunteered to perform frequency testing on the pump prior to purchase, in order to determine what amplitude and frequency sinusoidal pressure signals the pump was capable of delivering.
There are a number of parameters that determine the capability of the pump to deliver a sinusoidal pressure signal. Several of the parameters such as the maximum flow rate (25 ml/min), maximum acceleration rate (152 ml*s/min), and pressure control loop rate (40 Hz) cannot be controlled since they are determined solely by the pump design. On the other hand the compressibility of the system, which is affected by the fluid compressibility, initial fluid volume, and dissolved air in the fluid, can be chosen or controlled to a degree.

The desired sinusoidal pressure signal (past an initial ramp to a desired offset pressure level $P_o$ is given in equation 3.1

$$P(t) = P_o + A \sin(\omega t)$$  \hspace{1cm} (3.1)
where $P_o$ is the offset pressure, $A$ is the amplitude, and $\omega$ is the frequency. The offset pressure was chosen to be 690 bar (10 ksi), which is proportionally related to the expected burst pressure of the tube by the equivalent ratio of offset and burst pressure from Mori’s paper.

The change in pressure of a fluid, $dp$, is related to the initial volume, $V_0$, the change in volume, $dv$, and the fluid compressibility, $b$ (inverse of Bulk Modulus) as shown below in equation 3.2

$$dP = \frac{dv}{V_0 b}$$

By reducing the initial volume and compressibility of the fluid, a change in pressure is maximized for a change in volume prescribed by the pump. Equations 3.1 and 3.2 can both be differentiated with respect to time to relate the flow rate, $q$, to the initial volume, fluid compressibility, amplitude and frequency. The derivations and relationship, equation 3.3, are shown below.

$$\dot{P} = A\omega \cos(\omega t)$$

$$\dot{P} = \frac{q}{V_0 b} = A\omega \cos(\omega t)$$

$$q = V_0 b A\omega \cos(\omega t)$$

For any given flow-rate, equation 3.3 shows that fluid compressibility and initial volume should be minimized in order to maximize the potential amplitude and frequency of the sinusoidal pressure signal the pump can produce. Furthermore the coefficient of the trigonometric function in equation 3.3, $V_0 b A\omega$, was calculated for the tests conducted by Teledyne and used to predict the system’s capability of producing a sinusoidal pressure signal for a given volume of fluid with known compressibility.
Using the same methods as before, equations 3.1 and 3.2 can be differentiated a second time to establish a relationship between the acceleration of the pump, $\ddot{q}$, and the other parameters as shown in equation 3.4.

$$\ddot{P} = -A\omega^2 \sin(\omega t)$$

$$\ddot{P} = \frac{\dot{q}}{V_o b} = -A\omega^2 \sin(\omega t)$$

$$\dot{q} = -V_o b A\omega^2 \sin(\omega t) \quad (3.4)$$

The acceleration of the pump is limited by the hardware capabilities, therefore equation 3.4 shows that initial volume and compressibility should again be minimized to maximize the amplitude and frequency of the pressure signals the pump is capable of producing. Furthermore, it should be noted that the acceleration is related to the frequency squared, whereas it is proportional to the other terms. This suggests frequency may be a more limiting factor than amplitude. Similar to the flow rate relationship in equation 3.3, the trigonometric coefficient in equation 3.4, $V_o b A\omega^2$, was calculated for the tests conducted by Teledyne and used to predict the systems capability of producing a sinusoidal pressure signal for a given volume of fluid with known compressibility.

Various tests were conducted by Teledyne Isco at different frequencies, amplitudes and volumes. Deionized water with 5% Isopropanol was used as the pressurizing fluid. The small addition of alcohol deters the growth of bacteria. Water was the ideal choice for a fluid because of its significantly lower compressibility than oil. A standard mineral based hydraulic fluid has a bulk modulus of 1.8 GPa (at 20 ºC & 69MPa), while a water-glycol (2:1 ratio) fluid is 3.4MPa, or almost twice [40]. There is no data for the amount of
dissolved air in the fluid or system compressibility for these tests, therefore repeated tests may differ from the presented results.

Table 3.3 shows the different tests, parameters, and results from the Teledyne frequency testing. The ratio of output-to-input amplitude were used as a way to quantitatively judge the success of each test. The flow rate and acceleration trigonometric coefficients are included in the table and used to compare each test. The fluid compressibility parameter was removed from each coefficient since the same fluid was used for each test. This term would need to be considered if a different fluid was chosen.

Please note that since these tests are meant to establish the performance envelope of the system and could potentially lead to hardware damage or unsafe testing, we have relied on the manufacturer’s trials, rather than try to repeat them ourselves. Figure 3.4 shows the external control input signal and the pressure signal output for a test conducted with an initial volume of 65ml. The output signal matches very well with the input signal, with only an 8% difference in amplitude. The reference signal data was not collected during the experiment and instead was generated in MATLAB, therefore it is likely the input signal had less than a 3 ksi amplitude and matches the output signal perfectly.

The test in Figure 3.5 shows an increase in frequency from .1 to .2 Hz. The amplitude of the output signal is 25% less than the input signal, though the frequency is still maintained. The plot on the right shows that the flow rate is saturated periodically throughout the test resulting in the attenuation of the input pressure signal amplitude.
Table 3.3: Testing parameters and results from Teledyne frequency testing.

<table>
<thead>
<tr>
<th>Test</th>
<th>Vo (ml)</th>
<th>Ain (psi)</th>
<th>ω (Hz)</th>
<th>VAω</th>
<th>VAω^2</th>
<th>Aout</th>
<th>Aout/Ain</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>65</td>
<td>3,000</td>
<td>0.1</td>
<td>845</td>
<td>531</td>
<td>2750</td>
<td>0.92</td>
</tr>
<tr>
<td>2</td>
<td>65</td>
<td>3,000</td>
<td>0.2</td>
<td>1,690</td>
<td>2,123</td>
<td>2245</td>
<td>0.75</td>
</tr>
<tr>
<td>3</td>
<td>65</td>
<td>3,000</td>
<td>1</td>
<td>8,448</td>
<td>53,080</td>
<td>225</td>
<td>0.08</td>
</tr>
<tr>
<td>4</td>
<td>65</td>
<td>1,500</td>
<td>2</td>
<td>8,448</td>
<td>106,159</td>
<td>32</td>
<td>0.02</td>
</tr>
<tr>
<td>5</td>
<td>65</td>
<td>1,500</td>
<td>0.5</td>
<td>2,112</td>
<td>6,635</td>
<td>70</td>
<td>0.53</td>
</tr>
<tr>
<td>6</td>
<td>65</td>
<td>1,500</td>
<td>1</td>
<td>4,224</td>
<td>26,540</td>
<td>213</td>
<td>0.14</td>
</tr>
<tr>
<td>7</td>
<td>65</td>
<td>750</td>
<td>1</td>
<td>2,112</td>
<td>13,270</td>
<td>235</td>
<td>0.31</td>
</tr>
<tr>
<td>8</td>
<td>65</td>
<td>750</td>
<td>2</td>
<td>4,224</td>
<td>53,080</td>
<td>30</td>
<td>0.04</td>
</tr>
<tr>
<td>9</td>
<td>15</td>
<td>1,500</td>
<td>0.5</td>
<td>487</td>
<td>1,531</td>
<td>1330</td>
<td>0.89</td>
</tr>
<tr>
<td>10</td>
<td>15</td>
<td>1,500</td>
<td>1</td>
<td>975</td>
<td>6,125</td>
<td>600</td>
<td>0.40</td>
</tr>
<tr>
<td>11</td>
<td>15</td>
<td>750</td>
<td>1</td>
<td>487</td>
<td>3,062</td>
<td>645</td>
<td>0.86</td>
</tr>
<tr>
<td>12</td>
<td>15</td>
<td>750</td>
<td>1.5</td>
<td>731</td>
<td>6,890</td>
<td>470</td>
<td>0.63</td>
</tr>
<tr>
<td>13</td>
<td>15</td>
<td>750</td>
<td>2</td>
<td>975</td>
<td>12,249</td>
<td>200</td>
<td>0.27</td>
</tr>
</tbody>
</table>
Figure 3.4: Teledyne frequency testing for 0.1Hz and 10 +/- 3ksi pressure signal at 65ml initial volume (a). Zoom at 1st peak (b).
Figure 3.5: Teledyne frequency testing for .2Hz 10 +/- 3ksi pressure signal at 65ml Initial Volume. Pressure Signal (a). Flow Rate (b).

The data in Figure 3.6 shows another increase in the frequency to 2Hz. The pressure output signal is no longer sinusoidal and the system limit has been exceeded by increasing the frequency.

![Graph showing Teledyne Testing](image)

Figure 3.6: Teledyne frequency testing for 2Hz 10 +/- 3ksi pressure signal at 65ml Initial Volume.

Data from other tests listed in Table 3.3 suggests that the initial volume and input signal amplitude also have a significant effect on the system performance, which agrees with the aforementioned theoretical relationships. The two trigonometric coefficients are
calculated for each test and plotted versus the ratio of output to input pressure signal amplitude in a semi-log plot shown in Figure 3.7 below.

![Graph showing linear fits for each data set with strong correlation in the fit for the $V_A\omega^2$ parameter. These two coefficients can be used to estimate the feasibility of running a test with any combination of the individual parameters.](image)

Figure 3.7: Teledyne Isco 65D Syringe Pump Frequency Testing Analysis.
3.3 Grips

3.3.1 Psylotech µTS Pin Grips

A set of pin grips, Figure 3.8, were provided with the Psylotech µTS. The grips have a female dovetail which enables mounting onto the corresponding male dovetail that is machined on each crosshead of the µTS. The grip’s position is secured onto the dovetails by plunger pins which perform the same function as a set screw, except that the tips are spring loaded nylon spheres that don’t damage the surface of the crosshead.

![Figure 3.8: Psylotech µTS pin grips and features.](image)

Tensile specimens can be hung from a 4 mm dia. pin that spans the two identical parts that make up the grips. This also ensures precise alignment of the specimen with the load cell and drive train. Furthermore, the grips can be tightened against the specimen using 10-32 bolts to increase the holding power or for specimens that cannot be hung from the pin. The pins were used to hang the grips for tube uniaxial and isothermal tests.
3.3.2 Grips for Uniaxial Testing

The grips described above allow general-purpose testing of flat specimens, or of specimens that can be held between pins. As such, they are not sufficient for the tube-like specimens that are part of this research. A set of grips were designed to enable uniaxial tension tests of the SS-304L tubes on the μTS. The concept behind gripping the tubes was to replicate the typical connection that is used in the piping of high-pressure hydraulic systems. While each manufacturer uses a different trademark and the dimensions are not standardized, the concept is this: a male cone is attached at the end of the tube; the cone is pressed against a female cone on the 2nd component of the connection; usually, this pressing is achieved by a threaded gland that is hand-tightened with a wrench; by the elastic deformation of the two cones, a pressure-tight metal-to-metal connection is formed. Of course, while in high-pressure hydraulics the function of this connection is to seal the pressure, here the connection would have to transmit a tensile load without failure of the tube or relative slipping of any of the components. The design process that was based on this concept included several iterations. An overview of the exact design that we ultimately settled on is shown in Figure 3.9 and is detailed in the next few pages.
Figure 3.9: Uniaxial Tension Grip fabricated from spherical ball joint and other parts.

The connection contains a series of components and interfaces with the existing Psylotech grips described above (this way machining of the precise dovetail could be avoided). Immediately interfacing with the Psylotech grips is the component shown in Figure 3.10. This was made by modifying a spherical ball joint purchased from McMaster-Carr (MMC #60645K91). A spherical bearing permits angular rotation of the ball joint, up to certain limitations, as it is hung from the pin grips which mount on the dovetails of the crossheads of the μTS. The spherical bearing ensures that the tube is in pure tension and no bending moments have been imposed due to misalignment. This is especially critical here since the small size of the specimens can lead to significant prestraining during tightening of the specimen and can result in a meaningless experiment. This was indeed the case with earlier versions of the tube grips.
Figure 3.10: Spherical ball joints purchased from McMaster-Carr modified to be used as uniaxial tension grips on μTS.

The shank of the spherical ball joint contains a ¼-28 UNF female thread which was used, along with a custom gland, plug, ¼-28 threaded rod insert and two 3/32” Taper Seal© sleeves from High Pressure Equipment (HiP), to create a custom Taper Seal© fitting which is shown in Figure 3.9. The two-sleeve Taper Seal© fitting serves two purposes. First, to seal the tube so that fluid can be either passed through the tube for cooling purposes (e.g., isothermal testing) or to pressurize the tube in the case of biaxial stress testing. The second purpose is to grip the tube so an axial load can be prescribed by the μTS. Upon receiving the spherical ball joints, .312” were milled off the end of the shank, and the ¼-28 UNF female thread was drilled and tapped a half inch down from the newly cut end. A ¼” length piece of ¼-28 UNF threaded rod was screwed down to the bottom of the female thread in the shank and sealed with Loctite. Initially the
threaded rod was larger than ¼” and a slot was machined into it in order to screw the insert down into the female opening. Once in place, the insert was machined down to ¼” height and the Taper Seal© 3/32” female opening details, shown in Figure 3.11 per the HiP design, were machined. Identical dimensions were machined into the custom glands, to accommodate the second Taper Seal© sleeve.

Figure 3.11: Custom gland with sketch of 3/32” Taper Seal© HiP female opening Details. Identical details were machined into the thread rod insert.

Grade 8 High Strength Steel Cap Screws were (MMC #91286A134) machined to be used in place of 3/32” Stainless Steel SS-316 Taper Seal© Glands purchased from HiP. The stainless steel glands from HiP were found to deform and crack while under load, therefore a stronger and harder material was adopted.

The uniaxial grip works by tightening down the gland and forcing the sleeves to slide against the tapered surfaces of the threaded rod insert and gland. As the sleeves move
along these surfaces, they plastically deform, and are compressed and tightened around the tube. A stiff plug inserted into the end of the tube, which may be hollow or solid, acts to support the tube internally and prevent it from collapsing on itself as the sleeve is compressed around the outside. A rod of W1 Tool Steel with .081" outside diameter used for making drills (MMC #8890K125) was used as the plug for the uniaxial tension tests.

### 3.3.3 Grips for Isothermal Tension Tests

The isothermal grips, Figure 3.12, are modified versions of the uniaxial tension grips which allow fluid to pass through a tube specimen during uniaxial tension testing on the µTS. The fluid is circulated by a Neslab RTE 740 Refrigerated bath, which is described in section 3.8, and maintains the test specimen at a constant temperature throughout the test.

A 3-56 threaded hole was machined into the shank of the uniaxial tension grips described above in section 3.3.1. A 1/16” Barbed Tube x 3-56 Male Pipe fitting (MMC #5454K74) was secured into the threaded hole and sealed using plumber’s liquid Teflon thread sealant. A 1/16” ID Viton (MMC #5119K78), with a temperature range of -26 to +204 °C (-15 to +400 °F), connects the uniaxial tension grips to the circulation pump supply and return connection ports on the back side of the refrigerated bath. This connection is made with ¼” Male Pipe x 1/16” ID Aluminum Barbed Tube Fittings (MMC #5058K41).
While the Isothermal grips were sufficient to circulate fluid through the tube at low pressure, another set of grips were designed to handle the high pressure that would need to be generated in order to burst the tube specimens and follow biaxial stress paths. The high pressure fittings that interface the pump with the grips were significantly larger than those of the isothermal grips, therefore the biaxial grip would be designed to mount directly to the dovetails on each crosshead instead of hanging from the pin grips in order to save space. This allows for the full stroke to be utilized when testing high-elongation materials. Furthermore, mounting directly to the dovetails allows a compressive load to be applied to the tube which is necessary for biaxial stress paths counter-clockwise of plane-strain inflation (stress-ratio 2:1) in the hoop-axial stress.
plane. The same Taper Seal© fitting design used in the uniaxial tension grips would be adopted for these grips, but they would need be much thicker in order to handle the significant stress from the hydraulic pressure. The design for the biaxial grips is shown below in Figure 3.13.

![Figure 3.13](image)

**Figure 3.13**: Isometric view of biaxial grips (a). Side view cross section showing plunger pin holes and hydraulic port details (b).

The grip mounts onto the crosshead of the µTS with the dovetail feature and is secured in place by tightening down four plunger pins. The plunger pins were borrowed from the µTS pin grips. Two hydraulic ports, one in the front and one opposite the side with the dovetail, interface with the hydraulic fluid connection or the tube specimen, using the HiP Taper Seal© design. While the tube specimen is gripped using the two-sleeve Taper Seal© connection, only a single sleeve is required for the hydraulic fluid connections since no additional axial stress is prescribed.

Due to the tight tolerances on the dovetail, initially it was thought that the biaxial grips would require wire EDM machining, which would be a costly investment for an unproven design. Fortunately, an opportunity came about to have the grips 3-D printed on an EOS m 270 laser sintering system out of EOS Stainless Steel PH1 material. Normally parts
are hardened by heat treatment after being sintered, but for this application the material was already harder than what was required, therefore the heat treatment step was skipped. The mechanical properties of EOS Stainless Steel PH1 are found below in Table 3.4.

<table>
<thead>
<tr>
<th>Mechanical Properties</th>
<th>Before Heat Treatment</th>
<th>After Heat Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Yield Strength</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Horizontal (XY)</td>
<td>1150 +/- 50 MPa</td>
<td>min 1310 MPa typically 1450 +/- 100MPa</td>
</tr>
<tr>
<td>Vertical (Z)</td>
<td>1050 +/- 50 MPa</td>
<td>min 1310 MPa typically 1450 +/- 100MPa</td>
</tr>
<tr>
<td><strong>Ultimate Strength</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Horizontal (XY)</td>
<td>1050 +/- 50 MPa</td>
<td>min 1170 MPa typically 1300 +/- 100MPa</td>
</tr>
<tr>
<td>Vertical (Z)</td>
<td>1000 +/- 50 MPa</td>
<td>min 1170 MPa typically 1300 +/- 100MPa</td>
</tr>
<tr>
<td><strong>Elongation at Break</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Horizontal (XY)</td>
<td>16% +/- 4%</td>
<td>min 10% typically 12% +/- 2%</td>
</tr>
<tr>
<td>Vertical (Z)</td>
<td>17% +/- 4%</td>
<td>min 10% typically 12% +/- 2%</td>
</tr>
<tr>
<td><strong>Hardness</strong></td>
<td>30-35 HRC-</td>
<td>min 40HRC</td>
</tr>
</tbody>
</table>

### 3.3.5 Plane-Strain Inflation

In addition to the four grips that were designed to be used with the Psylotech tensile stage as described above, a standalone Plane-Strain test fixture was designed for three purposes: 1. Generate biaxial stress data before the biaxial controller was finished, 2. Test the functionality of the hydraulic system and pump, and 3. Test the effectiveness of the Taper Seal© grip design for a biaxial stress path. A simple block of steel with two Taper Seal© hydraulic ports was designed. One of the ports is for gripping the specimen and the second port is a connection to the hydraulic pump system. The design is similar to the biaxial testing grips except the plane-strain grips cannot interface to a tensile stage, which makes them significantly easier to machine.

The grips were manufactured in the UNH machine shop from A2 tool steel. Four holes allow 3/16” threaded rods to secure the blocks at a fixed distance from each other.
and prevent any axial strain in the tube. The test can also be conducted without the use of the threaded rod, and yields an identical strain path. The plane-strain inflation grips and test specimen are shown in Figure 3.14. Notice that the left grip has the ports at 90º to each other, so that it can be used for venting the air from the pressurization system. Furthermore, that grip has an additional hole drilled to it (bottom left of the picture) to secure the grip onto the testing breadboard. Of course, the grip assembly as designed is self-balancing and no net force appears during its operation. The hydraulic ports of the right grip are in-line, and is where the assembly is connected to the Teledyne pump.

![Figure 3.14: Plane strain test fixture and specimen.](image)

3.4 Hydraulic System Components

The hydraulic system encompasses everything from the Teledyne Pump to the plane-strain inflation and biaxial testing grips. Since the grips and pump have already been covered in detail, this section will focus on the rest of the system. Figure 3.15 and
Figure 3.16 show the components and connections in the hydraulic system. All of the high pressure components were purchased from HiP, while the non-pressure components were purchased from McMaster-Carr.

Coming out of the Teledyne pump are three connections: 1. The pressure transducer which was described in section 3.2, 2. A fill port, 3. A fluid pathway to the testing fixture. In order to fill the system a funnel is mounted on top of the pump. In between the funnel and the pump is a ball valve which is used to close the system during the tests. The funnel also has a stainless steel mesh screen to filter any particles in the hydraulic fluid added to the system. Care is taken so that foreign particles cannot enter the pump.

A pressure relief valve (HIP-20RV) sits in between the pump and the test valve. The purpose of the relief valve is to prevent the pump, which is capable of generating pressures up to 1,380 bar (20 ksi), from pressurizing the system more than the 1,034 bar (15 ksi) limit of the Taper Seal© fittings used downstream of the test valve. The pressure relief valve is field-adjustable between 690 and 1,380 bar (10 and 20 ksi). In the present set-up it was set at 1,034 bar (15 ksi). All of the connections, such as glands, nipples
and sleeves, leading up to the test valve are rated to 4,137 bar (60 ksi) max pressure. The ball valves themselves are rated to 1,380 bar (20ksi). Downstream of the test valve, 1.59 mm OD x 0.763 mm ID (1/16” x 0.03”) Taper Seal© tubing (HiP 15-9A1-030) and fittings are used to connect the test and purge valves to the plane strain and biaxial test grips. The 1/16” OD Taper Seal© tubing can be bent to a very tight radius (<1”). The connection between the test valve and lower biaxial test grip is sufficiently long, such that when the grip moves on the lower crosshead of the tensile stage during a biaxial test the tubing does not generate a significant load on the tensile stage.

Figure 3.16: Pressurization system components.
3.5 Biaxial Controller

The $\mu$TS LabVIEW control program, Psylotest, was modified to add additional features required to perform the biaxial stress testing experiments. The Psylotest program was written using an object-oriented programming paradigm in an actor framework architecture. Three separate modifications over the standard Psylotest software needed to be implemented in order to execute the radial and corner biaxial stress path tests. These three modifications were: 1. the addition of three user sensors, 2. a sensor-follower control type, and lastly, 3. threshold monitoring. Each task is described in more detail but first a brief overview of OOP and Actor Framework is provided below.

3.5.1 Overview of OOP & Actor Framework

Object-Oriented Programming (OOP) is a programming paradigm utilizing several techniques which promote code that maximizes reuse, minimizes debugging time, and facilitates maintenance and future code modifications. OOP also allows different parts of a code to be developed in parallel by different programmers and then seamlessly interfaced, though this isn’t relevant to this work. The coding techniques that are essential to OOP, i.e., classes and objects, encapsulation, inheritance, and polymorphism, are discussed below. This list is by no means exhaustive, but describes the features that were utilized in the software development for the biaxial testing controller.

Classes, Objects & Encapsulation

A class is a group of data which represents an abstraction, and the methods that act on that data. An object is an instance of a class in the software. The concept of grouping data and methods together is called encapsulation. By restricting the access and
manipulation of data to the code it is closely coupled to, the code becomes significantly easier to maintain and debug. Programmers begin with an idea of what their code will do and begin to identify classes and methods that need to exist in the code. Typically classes and methods can be identified by nouns and verbs used to explain the software.

Specifically in LabVIEW, classes contain a private data control which is a cluster of different data types that represent the class data. Virtual instruments contained in the class are methods used to access and modify this data. Objects, instances of classes, move along a wire in the same way as any other data type, except that the object data can only be modified by calling methods contained in the class. An example of class methods and data from the Psylotest software is shown in Figure 3.28

**Inheritance and Polymorphism**

Two other important features of OOP are inheritance and polymorphism. Inheritance is when a class receives data and methods from another class. This is useful when creating new classes which are more specific types of a higher level class. By creating different levels of abstraction, code can be written once and then easily extended to more specific instances without having to rewrite code. This practice maximizes code reuse and simplifies modifications and additions. The original class is commonly referred to as the parent class, and the inheriting class is called the child class. A child class may also be a parent class to an even more specific instance.

An example of inheritance is a vehicle, truck, and a car. In this example a truck and a car, which are both vehicles, have similarities and differences. Both have an engine and a transmission, but a truck has a bed and a car does not. The truck and car classes could be defined individually, but the similar traits would have to be defined twice. Instead, a parent vehicle class can be defined, and then the two child classes will both inherit these similar attributes. In this way the similar code is written only once.
A second concept of inheritance is the override feature. In LabVIEW a child class can override a method in its parent class. The override method can perform a separate function in addition to the parent class method, or completely ignore the parent class method all together. Going back to our vehicle class example. A transmission could be defined as a class, and be added to the private data of the car or truck class. Child classes of a transmission could be a manual and an automatic. In the parent class definition of a transmission we could describe gears, input and output shafts, fluid, all of which are common to both a manual and automatic transmission. In the child classes we could override a `shift gears.vi` method in the parent class, because changing gears is different in an automatic and manual transmission. In the automatic transmission `shift gears.vi` method, hydraulic solenoid valves open and close moving fluid through different ports, while in the manual transmission `shift gears.vi` method the driver activates a clutch and manual gear shift. There could be common functionality between the two transmissions which could be described in the parent class `shift gears.vi` method and called by each child class override method, or it is possible that both child classes implement completely different code.

Calling a specific override method of a parent class at runtime is called polymorphism or dynamic dispatching. Imagine the vehicle and transmission classes have been defined so that a user can create and drive a vehicle. The user can create any type of car or truck with either type of transmission, and then he wants to drive the vehicle using a `drive.vi` method. In the software a drive method would have to call a `shift gears.vi` method. With dynamic dispatching the programmer can write a single piece of code that will implement the correct override version of the `shift gears.vi` method at runtime, depending on whether the transmission class was defined as a manual or automatic. This eliminates the programmer’s obligation of writing code to determine
which type of class is passed into a section of code and choosing the appropriate code to be executed.

**Actor Framework Architecture**

The Psylotest software is built in an actor framework architecture. During startup different aspects of the software are spawned and run in parallel as actors. These actors can send messages back and forth to each other in order to perform a specific task or pass data. The actor framework architecture implemented in LabVIEW is meant to replace a common software pattern called the Queue Drive State Machine (QDSM). A QDSM is a case structure which executes different cases based on inputs from a queue. The queue is filled with states from other pieces of code in the software. The QDSM is powerful but has two common flaws, which are timing/race conditions, and minimal code reuse. Since the actor framework architecture is programmed using OOP, and has been rigidly tested and debugged, these flaws can be mitigated.

In the Psylotest program there are twelve different types of actors, although some of them are not relevant to this system and others remain completely unmodified for the addition of biaxial test function. The most notable actors are bolded and will be frequently referred to in the details of the software modifications below.

1. Psylotest Launcher
2. Psylotest
3. **Motion Controller Actor**
   a. **Drive Actor**
   b. *Output Actor*
   c. **DAQ Actor**
4. File IO Actor
5. **System Actor**
6. Window Controller Actor
7. Temperature Actor
8. Digital IO Controller
9. **Test Handler**

The System Actor contains a virtual instrument (vi) called the *Front Panel.vi* which acts as the user interface for the control system. In LabVIEW each vi contains a
connector pane, front panel, and block diagram so it is important to establish that there is a *Front Panel.vi*, which itself contains a front panel, connector pane, and block diagram. While all of the other virtual instruments in the software program also have front panels they are never used or seen by the user, therefore henceforth the use of the words *user interface* will refer to the front panel of the *Front Panel.vi*.

Each actor inherits from the LabVIEW actor class, which contains an *Actor Core.vi* method. The *Actor Core.vi* can be overridden by the child class, but it must still call the parent method. The *Actor Core.vi*, Figure 3.17, resembles a QDSM, where other actors place messages inside the queue to call different methods. Inside the error case structure of the vi, the message queue is passed into a while loop. The while loop pulls from the queue and receives each message calling the appropriate method.

![Figure 3.17: Parent method of Actor Core.vi](image)

LabVIEW users can create messages using the Actor Framework Message Maker tool in the tools menu. All of the methods in the actors are listed in a menu and can be chosen to create a message. The message is a class consisting of a private data control, a *Send.vi* method, and a *Do.vi* method. In the private data control is the data being sent between the actors which is the required input(s) for the chosen method the message was created for. The actor sending the message calls the *Send.vi* method,
which has input terminals for the data in the private data control. The message is then added to the queue in the *Actor Core.vi* of the receiving actor. When the queued message is processed the *Actor Core.vi* calls the *Do.vi* method. In the *Do.vi* method the data from the message is unbundled and passed into the input terminals of the method the message was created for.

An example of a message is provided for the *Move.vi*. The *Move.vi* is a method in the Motion Controller, and performs the function of adding a move to the controller state queue. A move is for one of the drives and could be a step, jog, sine wave, ramp or any other type of move. In the *Controller Loop.vi* of the Motion Controller Actor, the controller state queue is processed and a move is sent to the corresponding Drive Comm Actor. The Drive Comm Actor executes the move by controlling the drive through serial communication or an external analog signal. Since the Test Handler needs to be able to send moves to the Motion Controller Actor during a test, a message for the *Move.vi* method is created using the Actor Framework Message Maker. The *Move Msg* class and private data are shown in Figure 3.18 below.

![Figure 3.18: Move Message class (a) and private data control (b).](image)
The private data of the Move Msg class consists of only a move class since this is the only input to the Move.vi. In the Send.vi, shown in Figure 3.19, a move is bundled into the private data of the Move Msg object and the object is placed in the Motion Controller Core.vi message queue.

![Figure 3.19: Send.vi method of Move Msg class.](image)

The Actor Core.vi of the Motion Controller receives the queued message and calls the Do.vi shown in Figure 3.20. The move is unbundled from the Move Msg object private data and passed into the Move.vi method to be executed.
3.5.2 Data Acquisition

In order to control the biaxial stress path, the pressure, hoop stress and axial stress were added to the list of user sensors in the program. A user sensor can be a direct class, whereby the raw signal is simply filtered or averaged, or it could be part of its own unique class. In our case the pressure would be added to the direct user sensor class that had already existed, but new user sensor classes for the axial stress and hoop stress would be created.

The pressure user sensor addition is the simplest, therefore it will be described first. A systems.ini folder is located in the data directory under the project folder. This file contains all of the information for the analog input and output channels, user sensors, and other data that is specific to the system. Psylotech creates different testing systems therefore instead of creating different versions of the Psylotest software, the software remains identical for each system and reads the systems.ini file in order to determine how it will be uniquely configured. Since only one version of the software exists, the task of adding new features or modifying the code is significantly easier.
First the new analog channels for pressure, flow and volume need to be configured in the *systems.ini* file. Below the “[Raw Sensors]” heading exists a list of the analog channels available on the DAQ board shown in Figure 3.21. Channels 08, 09, and 10 are described as “NC” and are therefore unoccupied. By changing “NC” to “PRESSURE_FS”, “FLOW_FS”, and “VOLUME_FS”, for channels 08, 09, and 10 respectively, the raw analog signals have been configured. Since the raw sensor class has already been defined in Psylotest, no more work is required. The software will scan the analog channels for definitions, and find the newly created channels, calling them by the names that have been assigned.

```
[Raw Sensors]
Ch00 = LC_FS
Ch01 = LC_W
Ch02 = NC
Ch03 = NC
Ch04 = DISP_FS
Ch05 = DISP_W
Ch06 = NC
Ch07 = NC
Ch08 = PRESSURE_FS
Ch09 = FLOW_FS
Ch10 = VOLUME_FS
Ch11 = NC
Ch12 = NC
Ch13 = NC
Ch14 = Encoder_FS
Ch15 = NC
```

Figure 3.21: Raw Sensor configuration in *systems.ini* file after modifications.

Now that the pressure analog signal is added to the list of raw sensors, the user sensor can be created. The text in Figure 3.22 was added between user sensor 04 and what was previously user sensor 05.
Figure 3.22: Text added to system.ini file for pressure user sensor addition.

The type of user sensor is defined as Direct, and the pressure analog signal is assigned to the user sensor by writing in the name we gave to the pressure (PRESSURE_FS) signal in the raw sensors section. The numbers of the user sensors listed below pressure will need to be modified and the list of user sensors at the top of systems.ini file will need to be corrected to reflect the addition of another user sensor. Since the direct class already exists, no more work is required and the pressure user sensor addition is complete.

In order to add the axial stress and hoop stress, new classes need to be created which are shown in Figure 3.23 below.
Both classes are child classes inheriting from the User Sensor class and share identical private data which includes numerical controls for the center wall radius and thickness of the tube. In the same way the `systems.ini` file was modified for the addition of the pressure user sensor, axial stress and hoop stress user sensors are added to the file and appear as shown below in Figure 3.24.

```
[User Sensor02]
Name = Axial Stress Full Scale
PropertyName = AXSTRESS
Units = Pascals
ShortName = PA
Type = Axial Stress: PRESSURE_FS, LC_FS, Center Wall Radius .00223, Wall Thickness .00007
LimitHigh = 1000000
LimitLow = -1000000
MotionCtrl = Full
Controller Methods = Analog Velocity
TempCtrl = False
PGain = 0
IGain = 0
DGain = 0
MaxControlError=1k

[User Sensor06]
Name = Hoop Stress
PropertyName = HSTRESS
Units = Pascal
ShortName = Pa
Type = Hoop Stress: PRESSURE_FS, Center Wall Radius .00223, Wall Thickness .00007
LimitHigh = 10G
LimitLow = 0
MotionCtrl = Drive 1
Controller Methods = serial Pressure, Analog Pressure
TempCtrl = False
PGain = 0
IGain = 0
DGain = 0
MaxControlError=10W
```

Figure 3.24: Axial Stress and Hoop Stress additions to the `systems.ini` file.

The type for both user sensors is now assigned to their own unique class. It should be noted that the axial stress is the only user sensor which calls two raw sensors, and this is because it is calculated from both the pressure and axial load on the tube. Furthermore, tube geometry is defined which will be used by each class to calculate stress.
For both the Axial and Hoop stress classes, the *Parse Setup String.vi* and *Sensors Pre-Converted Raw.vi*, which belong to the User Sensor parent class, were overridden. The *Parse Setup String.vi* is identical for the Axial and Hoop Stress classes and performs the same functions as the User Sensor parent class. Additionally it parses the “Type” definition in the *systems.ini* file for the center wall radius and wall thickness data. Once the strings have been parsed they are converted to numerical data and stored in the private data of the Axial and Hoop Stress user sensor classes. The *Parse Setup String.vi* is shown below in Figure 3.25.

![Diagram](image)

**Figure 3.25:** Hoop Stress Parse Setup Strain override.vi.

In the *Sensors Pre-Converted Raw.vi* of the Axial and Hoop Stress classes, the raw sensor data configured for each user sensor is used to calculate the nominal axial and hoop stress from equations 3.5 and 3.6

\[
\sigma_\theta = \frac{PR}{t} \quad (3.5)
\]

\[
\sigma_x = \frac{F+PR^2\pi}{2\pi Rt} \quad (3.6)
\]
where $F$ is the axial load, $P$ is the pressure, $t$ is the undeformed wall thickness and $R$ is the undeformed mid-radius. The raw sensor is accessed from the hoop stress private data using the Read Raw Sensors.vi. The raw sensors array is indexed for the first and only raw sensor (pressure) associated with the hoop stress user sensor. The center wall radius and thickness data are unbundled from the private data. Using the center wall radius, thickness, and pressure data, the hoop stress is calculated and then written to the private data and passed to an output terminal.

In the axial stress Sensors Pre-Converted Raw.vi two raw sensors, load and pressure, are indexed and used along with the center wall radius and thickness from the private data to calculate the axial stress. The axial stress is then written to the private data and passed to an output terminal in the same way as the hoop stress. Figure 3.26 and Figure 3.27 below show the LabVIEW block diagrams for the Sensors Pre-Converted Raw.vi for the hoop and axial stress classes.

![Block Diagram](image)

Figure 3.26: Sensors Pre-Converted Raw.vi from the hoop stress user sensor class.
It should be noted that equations 3.5 and 3.6 are in a sense “hard-wired” into the vi’s above. Hence, if the user wants to implement a different equation, e.g., replace the current axial stress with the meridional stress of a non-circular-cylindrical shell, these vi’s have to be updated accordingly.

Furthermore, if it is desired to control the true, rather than the nominal stresses, several steps need to be taken. First the analog sensors required for axial and hoop strain measurements (mechanical extensometer and a LVDT) need to be added as raw sensors. This step is identical to what has been previously described for the addition of pressure, volume, and flow. Secondly, the user sensor definitions in the systems.ini file would need to be modified to include the additional mechanical extensometer and LVDT signals. Lastly, in the Sensors Pre-Converted Raw.vi the current geometry of the tube would be calculated using the mechanical extensometer and LVDT raw sensor signals. The true stress is then calculated using equations 3.5 and 3.6, but substituting the current values for the initial ones.

3.5.3 Sensor Follower Control Type

This feature enables a drive to be controlled in a way that maintains one user sensor proportional to a second user sensor. In the case of our biaxial stress testing this means...
that either the ball screw drive or the pump could be controlled in a way so the axial and hoop stress are maintained at a predetermined proportional value.

In order to accomplish this task the Sensor Follower class was created. This class is part of the Non-Actor classes under the Move Types directory, and inherits from the move class. It’s location in the project tree, override vi’s and private data are shown in Figure 3.28.

![Figure 3.28: Sensor Follower class shown in project tree with override vi’s (a) and private data (b).](image)

In the cluster of private data are the polynomial function, following sensor, and following time. The polynomial function is an array of numerical controls which indicate the scaling of the following sensor to the sensor being followed. The following sensor is an instance of the user sensor class, and the follow time is a numerical control that represents the length of time the following sensor will be controlled.

In total there are five virtual instruments which are overridden in the sensor follower class, but the scale move for 2nd actuator.vi simply calls the parent method. The first vi of the move class that is overridden in the sensor follower class is the Init by Ref.vi which is
shown in Figure 3.29 below. This vi is called by the *Front Panel.vi* when an user adds a stage to the test program. The different parameters that define a sensor follower test stage (amplitude, offset, following time, and following sensor) are wired as reference inputs to the vi terminal. Inside the vi, numerical values from the 3 parameter references are called and passed to the follow time and polynomial function controls in the private data. The polynomial function includes two terms. The first term is an offset and the second term is an amplitude. The user sensor from the input terminal is passed to the following sensor in the private data as well. Outside of the case statement, drive, system, a 4th parameter reference, and the sensor follower object are passed to the parent method of the *Init by Reference.vi*. Now that the sensor follower move has by initialized, it is passed to the *Add Stage.vi* in the *Front Panel.vi*.

![Diagram](image)

*Figure 3.29: Init by Ref.vi for the Sensor Follower class.*

The second vi that is overridden is the *Return Move String.vi* which is called when the user adds a stage to the stage list. In the *front panel.vi* a dynamic user event causes event structure #44 to execute the *update stage display.vi*, which in turn calls the *create stage list.vi*, which finally calls the *return move string.vi*. The *return move string.vi*, shown
in Figure 3.30, outputs a string to be displayed on the user interface that shows what stages have been added to the test stage list. A single string is concatenated from a set of strings which describe the type of stage and the stage parameters. This string is passed to the parent method, which appends more stage details that are common to any type of test stage (sampling rate, filter cutoff).

![Diagram](image)

**Figure 3.30: Return Move String.vi of the Sensor Follower class.**

The 3rd override vi is the *Update Parameter Reference.vi* and is called when the user changes either the stage or control type in the test profile tab of the user interface. The function of this vi is to modify the stage parameters listed on the user interface according to the control and stage type selected by the user. There are a number of numerical controls used to represent the test parameters for all of the different control and stage possibilities, therefore the text captions are modified each time a new control or stage type is selected. Furthermore different stage types require different number of
parameters, therefore numerical controls are made visible or hidden on the user interface.

Upon changing the stage or control type, the event structure #9 in the block diagram of the *Front Panel.vi* is executed which calls the *Update Stage Parameter Display.vi*. This vi calls the *Update Parameter Reference.vi* after initializing the potential move and passing it to the input terminal. In the sensor follower override vi, three of the numerical controls are referenced and their attributes are modified for the sensor follower stage type. These parameters are the following time, offset, and scaling factor. After modifying these numerical controls another set of references of numerical controls are passed to the parent method.
The last override vi is the Setpoint.vi and is called in the drive actor Process Motion State.vi under the “Moving” case structure. The setpoint.vi generates a set point for the PID control loop running on the drive. In the sensor follower Setpoint.vi, shown in Figure 3.32, the following sensor, following time, and polynomial function are unbundled from the private data. The polynomial function (offset and gain) is applied to the following sensor value after it is read, and then passed to the set point and set point holder out output terminals. The following time is checked and the Boolean output is passed to the done output terminal.

3.5.4 Threshold Monitoring

Threshold monitoring enables a testing stage to end when a user sensor reaches a defined value, while not affecting the normal termination of the stage. For example, a 5 mm ramp stage at 10 μm/s is prescribed in drive control. Additionally an upper threshold is assigned to the stage for an axial stress of 500 MPa. The stage could end either by reaching the end of the 5 mm ramp that was prescribed, or at any point during the stage
if the axial stress exceeds 500 MPa. Several different types of thresholds were defined and their implementation into the software is described.

A Threshold class was created and added to the Non Actor Classes directory. Six child classes, Above Threshold, Below Threshold, Within Range, Negative Transition, Positive Transition, and Outside Range, were also added in a newly created Threshold Types directory shown in Figure 3.33.

Figure 3.33: Project tree showing addition of Threshold class to the Non-Actor Classes directory as well as the Threshold child classes in the Threshold Types directory (a). Private data cluster for the parent Threshold class (b).
The above threshold class acts as an upper limit to a user sensor value, while the lower threshold class has a lower limit. Note that threshold monitoring does not begin until the threshold sensor is within the threshold limit. The outside range and within range threshold classes have both upper and lower limits but work in the opposite manner. When the threshold sensor goes inside the upper and lower limits of the within range child class, the threshold is active. The opposite is true for the outside range threshold class. Lastly negative and positive sensor transition thresholds were created, whereby if the threshold sensor value suddenly increases or decreases in a specified amount of time by a specified amplitude, the threshold is reached and the test stage will end. Note that reaching a threshold only terminates the current stage while the remaining stages of a test would still be executed.

A threshold list can be created in the test profile tab of the user interface in a similar way to how a stage list created. The numbers on the stage list correspond to the threshold list numbering. A screenshot of the threshold list and controls is shown in Figure 3.34. Before creating a threshold list a stage must be added to either the axial ball screw or pump drive.

![Threshold List Image]

Figure 3.34: Threshold creation section of test profile tab on user interface.
After the threshold list has been created it is written to the move array type test class and sent in a message from the system actor to the Test Handler. An array of thresholds was added to the move array class, which is a child of the test class. This addition is shown in Figure 3.35 below.

Figure 3.35: Move Array class private data with array of thresholds added.

When the start test button on the user interface is called a message is sent from the System Actor to the Motion Controller to run the Start Standard Test.vi. The Motion Controller then sends a message to the Test Handler to run the Start Test.vi and after checking a few things calls the Start Test Motion.vi. The Start Test Motion.vi, Figure 3.36, calls the Next Move.vi which pulls an individual move and threshold from the arrays in the test private data. The threshold and move are then sent in messages from the
Test Handler to the Motion Controller. Upon receiving the threshold from the Test Handler, the Motion Controller writes the threshold into the system private data in its own private data. This happens in the “New Threshold” case structure in the controller loop.vi in the Motion Controller, which is shown in Figure 3.37 below.

The system private data was modified to include a threshold class as shown in Figure 3.38.
Figure 3.38: Threshold class added to systems class private data.

Now that the threshold has been added to the systems class on the Motion Controller the threshold sensor value will be checked against the threshold limits during the test stage. This process is performed in the Controller Loop.vi in Figure 3.39 below.

Figure 3.39 No internal state message case structure of Controller Loop.vi.

The controller loop executes the Process Threshold.vi which checks the threshold sensor against the defined threshold. The Process Threshold.vi is shown in Figure 3.40.

Figure 3.40: Process Threshold.vi checks the threshold sensor against the threshold.
Inside the *Process Threshold.vi* the systems are read from the motion controller and auto indexed into a for-loop. The threshold is read from the systems private data and the on/off Boolean is unbundled and controls the first case structure. If the on/off is false nothing happens, but if it is true, the reached Boolean is read from the private data. If true then a “Threshold Reached” state is passed to the controller state queue. If the reached Boolean is false the threshold is checked. The threshold sensor is unbundled along with the user sensors and the value is read from the systems data stream and passed to the *Check Threshold.vi*. The sensor value is checked against the threshold limits in the *Check Threshold.vi* and the Boolean output is passed into the 3rd case structure.

![Diagram](image)

(a)  (b)

Figure 3.41: 3rd and 4th case structures in *Process Threshold.vi*. False (a) & True (b).

The 3rd case structure is controlled by the Boolean in the threshold triggered private data. If the triggered Boolean is false, the reached Boolean is passed through a not gate and written to the triggered private data. Once the sensor value is within the limits, the threshold monitoring is activated. If the sensor value is initially outside of the limits, the threshold monitoring is not active. In the true case structure the Boolean from the check threshold output controls the 4th case structure. If the fourth case structure is false,
threshold not reached, nothing is done. If the threshold is reached a “Threshold Reached” state is placed in the controller state queue.

The Controller Loop.vi recognizes the “Threshold Reached” state in its queue, and executes the “Threshold Reached” case structure which is shown in Figure 3.42. In the “Threshold Reached” controller state the systems are read from the Motion Controller and a system is indexed. The Threshold Reached.vi is called for all drives in the system. An empty threshold is written to the systems private data. The systems are then written back into the Motion Controller.

Figure 3.42: “Threshold Reached” state in controller loop.vi of Motion Controller.

The Threshold Reached.vi, Figure 3.43, is called for each drive and writes a true boolean to the Threshold Reached Control State type def in the Drive Comm Actor private data. The control state is evaluated in the process motion state.vi for each drive.
Figure 3.43: Threshold Reached.vi on Drive Comm Actor.

The process motion state.vi, Figure 3.44, executes the moves sent to each drive. In the “Moving” motion state when the threshold reached Boolean is true the motion state is changed to “Stop Move” and the current move is dequeued. Since the move is completed the test stage is ended and the Test Handler will send the next move and threshold to the Motion Controller starting everything over again.
3.6 Strain Measurement Systems

Strain measurements of the deforming tube were obtained primarily by a Digital Image Correlation (DIC) system and for a few uniaxial experiments by an axial extensometer. The DIC system was purchased from Correlated Solutions Inc. In the case of uniaxial tension, the 2D-DIC technique was used since only axial strain was desired. The 3D-DIC technique was used for all biaxial stress experiments to capture both the axial and hoop strain. Images were obtained using 2.0 Megapixel digital cameras (Point Grey Research GRAS-20S4MC) with Schneider–Kreuznach Xenoplan 35mm lenses. VIC-Snap software was used to acquire the images, and VIC-2D 2009 and VIC-3D 2012 were used to post process the test images for 2D and 3D respectively.
3.7 Infrared Temperature Measurement System

A FLIR SC645 infrared camera, Figure 3.46, was used to measure temperature of the tube specimens during isothermal testing. The black and white speckled pattern required for the DIC measurements provides a high emissivity that does not induce any artificial variation in the measured temperature, allowing accurate infrared measurements. This has been demonstrated by Cullen and Korkolis [13]. This camera has a temperature accuracy of +/- 2 °C (3.6 °F) or +/- 2% of reading, range from -20 to +650 °C (-4 to 1202 °F), and a spatial resolution of 640 x 480 pixels.

Figure 3.46: FLIR SC645 Infrared camera [44].
3.8 Thermo Scientific Neslab RTE 740 Refrigerated Bath

A Thermo Scientific Neslab RTE 740 Refrigerated Bath, Figure 3.47, was used to circulate fluid through the tube to maintain the temperature constant during uniaxial tension testing. For isothermal experiments with temperatures less than 100 °C, deionized water was used as the heat transfer medium for its beneficial characteristics such as low viscosity, high specific heat capacity, high thermal conductivity and convenience. For temperatures above 100 °C, white mineral oil (AniMed®) had to be used for its high boiling point. The temperature bath has a range of -40 to +200 °C (-40 to +392 °F), and a stability of +/- .01 °C. The unit contains an air cooled non-cfc refrigeration system for cooling, 800 watt electric heaters and a circulation pump.

Figure 3.47: Thermo Scientific Neslab RTE 740 Refrigerated Bath [45].
4.1 Introduction

The purpose of this chapter is to establish the mechanical response of the 304L stainless steel microtubes under uniaxial tension. This material is known to be rate-dependent, prone to deformation-induced heating [13], and martensitic transformation [12]. Uniaxial tension experiments were performed directly on the tubes, as well as on strips that were extracted from the tubes. Furthermore, isothermal tension tests were performed at different strain-rates.

4.2 Uniaxial Tension of Tubes

4.2.1 Test Methods

Uniaxial tension specimens were prepared by cutting 64 mm long pieces of tube from the five feet (1.524 m) delivered sections, using a metallurgical diamond cutoff circular saw of 100 mm diameter. A cutting fluid was used to minimize heating of the tube during this process. The ends of the specimen were deburred on a deburring wheel. The tube surface was cleaned from grease and oil using acetone or isopropyl alcohol. A black and white speckle pattern was then painted on the tube, for DIC strain measurements, using black and white Rust-Oleum© High Heat Specialty paints. Plugs were inserted into the ends of the tube to prevent the tube from collapsing on itself while being gripped for testing. The plugs were prepared from a 2.057 mm (0.081”) W1 Tool Steel drill rod, cut into 17 mm long pieces, which resulted in an approximately 30 mm
testing section of the tube specimen. The end of the plug that was inserted into the tube was rounded with a deburring wheel. The other end was not deburred. This prevented the plug from slipping down into the middle of the tube during assembly, before the sleeves were tightened onto the tube. Next, the glands and sleeves were careful slid onto the tube so not to disturb the painted surface. Lastly, the glands were threaded down into the shank of the spherical ball joint and tightened with a wrench. After both glands were tightened, one of the two was adjusted so that the grips were in alignment with each other. The assembled specimen and two grips were then placed in the µTS and attached to the machine’s grips using pins. The tension experiments were performed under constant velocity control at a rate of 50 µm/s resulting in a constant strain-rate of approximately 1.5 x 10⁻³ s⁻¹.

4.2.2 Stress and Strain reductions

Nominal axial and hoop strain measurements were obtained using 3D DIC measurement techniques and a virtual extensometer tool. Figure 4.1 shows a biaxial test specimen (along the nominal 0:1 axial:hoop stress path) and the full field hoop strain along with axial and hoop virtual extensometers. In the case of axial strain measurement this technique is analogous to a mechanical extensometer where the nominal axial strain is calculated by equation 4.1

\[ e_x = \frac{\Delta L}{L_0} \]  \hspace{1cm} (4.1)

where \( \Delta L \) is the change in length of the virtual extensometer and \( L_0 \) is the original length. In the case of uniaxial tension the nominal axial strain is noted as \( \varepsilon_n \) and the true axial strain, \( \varepsilon \), is calculated from equation 4.2

\[ \varepsilon = \ln(1 + \varepsilon_n) \]  \hspace{1cm} (4.2)
In the case of hoop strain, the virtual extensometer does not behave similar to a chain-type, circumferential mechanical extensometer. The virtual extensometer tracks the distance between two points in 3D space, therefore our hoop strain virtual extensometer is measuring the change in length of a chord, not the change in length of an arc on the tube surface. The same could be said for the axial strain virtual extensometer measurements since the hoop strain is not entirely uniform along a tube generator and a small radius of curvature in the meridional direction can be present. This effect was investigated and found to be insignificant for most paths since the radius of curvature is sufficiently large. The error is accentuated around the pure hoop tension paths.

Figure 4.1: Virtual extensometers measuring axial and hoop strain (a). Diagram of virtual extensometer measuring hoop strain by change in chord length (b).
In Figure 4.1, the black circle and chord, $b$, represents the original tube, while the green circle and chord represent the geometry of the tube after an arbitrary amount of uniform expansion. Assuming that the tube expands uniformly and the angle $\theta$ remains constant, it can be shown that the change in length of the tube radius is proportional to a change in length of a chord. Since the relationship is proportional and strain is a unit-less quantity, tracking the change in length of the chord is an accurate way to measure the hoop strain.

The derivation below proves the aforementioned hoop strain measurement methodology. The first line shows the relationship between the radius and hoop strain for a thin-walled tube or some other axisymmetric body deforming axisymmetrically. The second line shows the relationship for any chord on the tube and the radii that meet the ends of the chord. Substituting the radii in the first equation for chord and angle, using the relationship for the second line, we reach the third line. By multiplying the third line by the proportional constant common to both the numerator and denominator, our final result, equation 4.3 is derived. This equation shows that the hoop strain is equal to the change in length of a chord on the tube, which is measured by our hoop strain virtual extensometer.

\[ e_\theta = \frac{\Delta r}{r_o} = \frac{r - r_o}{r_o} \]

\[ r = \frac{b}{2 \sin \frac{\theta}{2}} \]
The true axial and hoop strains are calculated using the nominal strain measurements along with equations 4.4 and 4.5.

\[ \varepsilon_x = \ln\left(1 + e_x\right) \]  

\[ \varepsilon_\theta = \ln\left(1 + e_\theta\right) \]  

The nominal axial stress, \( \sigma_n \), is calculated using load cell readings and geometrical measurements of the tube described in section 2.2. Assuming a thin walled-tube under an external axial load leads to the equilibrium equation 4.6

\[ \sigma_n = \frac{F}{2\pi R_o t_o} \]  

where \( F \) is the external load, and \( R_o \) and \( t_o \) are the initial radius and wall thickness.

The true stress was calculated using equation 4.7.

\[ \sigma = (1 + \varepsilon_n)\sigma_n \]
4.2.1 Results

The response of the tube in uniaxial tension is shown in Figure 4.2 below (3 repeats). The tube yield strength was determined to be 452 MPa using the 0.2% offset method, and has an ultimate stress of 660 MPa at 45% nominal strain. The DIC digital extensometer strain measurements do not have the accuracy required to determine the elastic modulus of the material, but literature suggests it is 193 GPa for SS-304L [32].

![Figure 4.2 Nominal stress vs strain for uniaxial tension experiments.](image)

The strain-rates from the three experiments are shown below in Figure 4.3. The tests conducted on the µTS show a sinusoidal oscillation caused by a misalignment of the ball-screw and a high sensitivity of the displacement sensor to the oscillation. Disregarding the oscillations the nominal strain-rate is constant throughout each test, and the same for all tests.
Figure 4.3: Strain-rates vs strain for uniaxial tension tests.

The true stress and strain were only plotted up to the ultimate strength and are shown in Figure 4.4. Beyond this point the response is non-uniform and is not representative of the material properties of the stainless steel SS-304L tube, nor is equation 4.1 valid. The plastic region of the true stress vs strain curve shows almost linear strain-hardening.
For uniaxial tension the plug determines the testing gauge-length of the tube since the tube experiences radial contraction as it is strained axially. At approximately 2% elongation, the tube will conform to the shape of the plug. The radial pressure of this contraction in the gripped region and the associated friction, prevents the tube from further deformation in that region, while the portion of the specimen between the plugs remains in uniaxial tension and continues to plastically deform.

The axial strain and radius of the tube were tracked using a line plot tool with 3D-DIC strain analysis. The line plot tool, shown on the bottom in Figure 4.5, captures 100 points of data evenly spaced along the line for any image selected. In the top of the same figure is a series of side views of a 3D model of the tube at 0, 30, and 60% elongation. The radius of the tube decreases uniformly and then non-uniformly, and eventually forms a neck.
Figure 4.5: Evolution of diameter (Top). 3D DIC Line Plot (Bot).

Figure 4.6 shows line plots of the nominal axial strain along the normalized gauge length of the tube at different levels of overall strain (i.e., readings of the virtual extensometer). The axial strain grows uniformly at the center of the gauge length before necking in the center. At the ends of the normalized gauge length, where the ends of the two plugs sit, the strain rises to about 3% and remains constant at higher levels. The plot shows the typical behavior expected of a localization problem: beyond the end-effect due to the presence of the plugs, the axial strain grows uniformly in the test section. Furthermore, the local values of the strain are identical to the overall reading of the virtual extensometer, as expected. However, as plastic deformation accumulates, the strain distribution becomes non-uniform. At some point, in this case between 44% and 55%, the deformation localizes and a diffuse neck forms approximately at the center of the specimen (also see Figure 4.5). The growth of strain is then very rapid inside the diffuse neck, leading to ductile fracture.
Figure 4.6: Evolution of axial strain vs. normalized gauge length during uniaxial tension test.

Line plots of the tube radius at the same levels of plastic strain from above is shown in Figure 4.7 below. The events in this figure mirror those of Figure 4.6. The radius decreases uniformly in the test section of the tube and is limited at the plugged ends. After reaching 44% elongation the radius becomes non-uniform in the test section and a neck forms before fracturing.
Figure 4.7: Evolution of tube radius vs normalized axial position during uniaxial tension test.

The radius was further probed at specific points along the gauge length of the tube using the DIC box tool, and is plotted as a function of axial strain in Figure 4.8. The box tool captures an average value of the field data inside the border of the box, and changes size with the specimen as it deforms (Lagrangian flow field). At $2x/\text{GL} > 1.24$ (i.e., outside of the uniform deformation region and close to the gripped end) there is very little change in the radius of the tube for all levels of axial strain, which is expected because of the presence of the plug at that location. As we move closer to the neck of the test specimen, the radius of each box decreases relatively uniformly up until 30% elongation, when the first instability is observed as unloading at $2x/\text{GL} = 0.84$ and 0.68, while the remainder of the tube (i.e., $2x/\text{GL} \leq 0.52$) continues to shrink in diameter. The
overall strain where this instability event occurs is, of course, the same as recorded in Figure 4.6. But the data of Figure 4.8 reveal that a second instability occurs at 55% elongation, where the $2x/GL=0.047$ location is seen to stop growing, while the $2x/GL=-0.13$ location continues to do so. Necking and fracture is observed at the $2x/GL=-0.13$ point. The use of the full-field DIC measurements sheds light in the two instabilities. These are not believed to be simply diffuse and localized necking but rather two types of diffuse necking. The first instability relates to a change in diameter, while the second instability relates to a change in the wall-thickness. Unfortunately, the DIC resolution and the features of this problem do not allow a direct observation of the final, localized necking and the ductile fracture event that terminates the experiment.

![Figure 4.8: Multiple bifurcation instability during uniaxial testing (a). Box tool extractions from 3D DIC data (b).](image-url)
4.3 Strip Uniaxial Tension

In order to investigate the response of the material in uniaxial tension without the influence of the tube geometry, tests were conducted on strip specimens which were extracted from the axial direction of the tube. Cutting small arc lengths of the tube was a difficult process due to their relative size. The same drill rod that was used in the uniaxial tension tests as plugs was inserted into the tube and used to support it during machining. The tube and rod were then passed through a low tolerance hole drilled into a steel block where a .012" (.305 mm) thick embedded slitting saw cut the side of the tube. The steel block prevented the tube and saw from bending during the slitting process. After slitting on one side, the tube it was rotated by 180° and slit on the opposite side, thus creating two halves. Quarters or eighths were preferred, but could not be produced using the process described. Figure 4.9 is a mockup while the real process was performed on a milling machine; the steel block was secured in a vise on a 3 axis milling table and the slitting saw was mounted in the spindle.
Due to the small size of the specimens and the fact that they were curved, it was not possible to machine a shoulder region, hence the strips were tested directly. The experiments were performed in the μTS at a strain-rate of $1.5 \times 10^{-3}$ s$^{-1}$, same as the experiments reported in the previous section. Each specimen was prepared for DIC measurement in the same way described in section 4.1.

The strip specimen before and after testing is shown in Figure 4.10. The specimens were gripped during the test by tightening the μTS pin grips together using bolts and the nuts of Figure 4.10. This caused the ends of the strip specimen to flatten, while the center of the test specimen remained close to a half circle. The failed specimen in the grips is shown in Figure 4.11.
Figure 4.10: Strip specimen after and before test.

Figure 4.11: Fractured strip specimen in µTS pin grips
It was observed during testing that the central part of the strip specimen was curling inwards, i.e., its curvature in the plane perpendicular to the loading axis was increasing (initially, 1/"original radius"). This phenomenon is visible in Figures. 4.9 and 4.10 and can be explained by the flattening of the gripped ends. There is no way to guarantee that the plane of the flattened end will be coincident with the centroid of the curved central section. Hence, during loading, the central section experiences not only tension but bending, as well. In response to this parasitic bending moment, the tube develops anticlastic curvature, which causes the curling observed. Apparently, this is not a desirable situation to occur during testing. It could perhaps be avoided by testing smaller arcs (quarters or eights), rather than halves. And/or it could be reduced by preparing a special set of grips that would not flatten the gripped end. This was deemed more complicating than beneficial for the present study.

The nominal response of the strips are shown below in Figure 4.12. The response is the same for both the tube and strip specimens, but the strips fail much earlier. The premature failure is caused by the curling of the test section, as well as by imperfections introduced into the strip during the machining process, which created a slightly uneven lateral surface on the strip specimen. However, obtaining an identical response from both testing methods suggest that both methods are able to capture the material response and are not influenced by the specimen geometry.
4.4 Isothermal Uniaxial Tension

Isothermal uniaxial tension tests were conducted to determine the temperature- and rate-dependence of the material. Stainless steel SS-304L is typically strongly dependent on these parameters, therefore these relationships need to be captured in order to determine the appropriate material properties for future numerical (FEA) models. While in earlier work it was deemed necessary to design a heat exchanger to achieve isothermal testing [13], the fact that in this case the specimen is in tube form makes it easier to perform isothermal experiments. All that is needed is to circulate fluid through the tube at the desired temperature.
The plastic work induced in the tube during uniaxial tension generates heat. Since the tube is held at the ends by grips that remain at room temperature, heat is transferred (conducted) from the specimen towards the cold grips. (At the same time, heat also escapes through convection and radiation, but to a negligible effect in comparison to conduction [11]). Stainless steel SS-304L has low thermal conductivity and low specific heat capacity, therefore the deformation-induced heating and subsequent conductive heat transfer typically causes a rise in the temperature of the tube. Furthermore, the temperature rise is non-uniform, causing a temperature gradient in the tube. The center of the specimen has the highest temperatures and the ends of the specimen remain close to ambient. The temperature and gradient increase as plastic strain increases, as well as with higher strain-rates. At strain-rates less than $10^{-4} \text{s}^{-1}$ no rise in temperature is observed because the rate of heat generation is equivalent or less than the rate of conduction to the grips, i.e., sufficient time is provided for the heat to dissipate.

During the uniaxial tension experiments of the SS 304L tubes, a FLIR SC-645 infrared (IR) camera was used to assess the temperature fields that developed. (The specifications of the IR camera were presented in section 3.7) Figure 4.12 shows a specimen pulled at a strain-rate of $10^{-2} \text{s}^{-1}$ and the evolution of the temperature field during the experiment. A temperature gradient can be observed along the specimen.
Isothermal tests were conducted at a constant strain-rate of $1.5 \times 10^{-3}$ s$^{-1}$ for temperatures of 6, 25, 50, 76, 100, and 142 °Celsius. The specimens were prepared and secured in isothermal uniaxial tension grips in the same manner as the non-isothermal uniaxial tension tests described in section 4.1, with the exception of the plugs. For the isothermal experiments hollow plugs were required to circulate fluid through the tube. The 2.057 mm (0.081 in) drill rod plug was replaced with stainless steel SS-304 14.5 gauge hypodermic tube purchased from MicroGroup (304H14.5). The hypodermic tube was welded and drawn, and heat treated to a full hard temper which resulted in a minimum tensile strength of 965 MPa (140kpsi). The tube has an outside diameter of 1.98 mm (.078 in) which allows it to fit inside the 2.08 mm (.082 in) inside diameter of the tube specimen. The tube plug is prepared in the same manner as the solid drill rod plug described in section 4.1. After the specimen is secured in the grips they are attached to the pin grips in the μTS.
The constant temperature bath described in section 3.8 was connected to the isothermal grips using Viton rubber tubing. The bath was set to the desired temperature and fluid was circulated through the tube by the bath pump. Deionized water was used for experiments less than 100 °C while white mineral oil (Animed®) was used in experiments with temperatures above 100 °C for its higher boiling point. The infrared camera monitored the temperature of the tube as the bath modulated its temperature to reach the set point. The set point of the bath was adjusted using the infrared tube temperature data to accommodate a steady state temperature difference between the bath and tube.

The capacitive based load cell on the µTS is sensitive to changes in temperature and the reading was affected by the heat transfer between the specimen, grips and crosshead of the µTS. This effect was more prominent as the isothermal temperature deviated further from ambient conditions. In order to circumvent this problem the system was allowed to reach steady state before starting the test (typically, after 30 min.). The load cell reading at steady state was taken as the zero stress load.

The nominal stress versus strain data at different isothermal temperatures for a constant strain-rate of 1.5 x 10^{-3} s^{-1} is shown in Figure 4.14 below. The yield stress, flow stress, ultimate strength, uniform strain and total elongation all increase as the tube temperature decreases. These trends were also observed in isothermal tests conducted on stainless steel SS-304 specimens by Cullen et al. [13]
A point measurement of the tube temperature at the location where the neck will eventually appear is extracted from the infrared images and plotted versus a normalized time for each isothermal experiment in Figure 4.15 below. In a monotonic conventional (i.e., non-isothermal) tension test the temperature at the location where the neck will form is higher than anywhere else in the tube and will also be the point of the highest plastic strain in the failed specimen. The figure below shows that the temperature at the location of the neck is constant throughout these isothermal experiments. These results show that the tube was held under isothermal conditions for the duration of the experiment and accurately captured the response of the tube to at a constant nominal strain-rate.
Furthermore, a line plot is utilized to capture the temperature profile of the tube immediately before fracture for each isothermal experiment. The maximum temperature gradients are observed at this point in the experiment. Figure 4.16 shows that the temperature is constant across the gauge length tube for each experiment, adding greater validity to the isothermal conditions of each test. Interestingly, necking in the isothermal experiments did not occur at or close to the center of the gauge-length, as in the conventional tension test, but were randomly distributed along the specimen. Furthermore, the necks in the isothermal experiments appeared to be more localized than in the conventional one.
The associated strain-rates versus strain are shown in Figure 4.17. In order to capture the temperature-dependence of the material, the rate effects must be decoupled by performing each test at identical constant strain-rates. As described in section 4.1, mechanical problems experienced with the μTS resulted in a sinusoidal pattern of the strain-rates. Furthermore, a step in the strain-rate was observed at approximately 10% elongation for the isothermal experiments. Overall, the same sinusoidal pattern, step and strain-rate magnitude are observed for each test, which confirms that the rate- and temperature-dependence of the material response have been successfully decoupled.

It should be noted that the temperature-dependence has only been observed at a single strain-rate, but it may be appropriate to extend these relationships to other strain-
rates. Cullen et al. [13] performed isothermal tests on stainless steel SS-304 ASTM-E8 standard specimens and found a similar trend for the ultimate strength and total elongation at $10^{-2}, 10^{-3}, 4 \times 10^{-4},$ and $10^{-4}$ strain-rates.

The jump in the strain-rate between 10% and 20% strain is zoomed-in in Figure 4.18. The initial strain-rate is oscillating around $10^{-3}$ s$^{-1}$ before increasing to $1.7 \times 10^{-3}$ s$^{-1}$. Averaging the strain-rate over the strain results in an average strain-rate of $1.5 \times 10^{-3}$ s$^{-1}$. The sinusoidal oscillations differ in amplitude for each test, which is most likely caused by subtle differences in specimen alignment with the load train, but have a maximum peak-to-peak amplitude of $0.6 \times 10^{-3}$ s$^{-1}$. While this material has a rate-sensitivity as we will establish in section 4.5, this small variation in strain-rate doesn’t significantly affect
the shape of the flow curves. Upon close inspection, there is a sinusoidal oscillation in
the flow stress, but this is deemed trivial.

![Figure 4.18: Zoomed-in strain-rate vs strain for isothermal testing.](image)

The true stress and strain were calculated for each isothermal test. The flow stress
was plotted as a function of temperature at different levels of true strain and is shown in
Figure 4.19 below. The flow stress decreases monotonically as the temperature
increases for each level of plastic strain. Between 25 and 100 °C the rate of decrease in
flow stress is relatively constant. Above 100 °C the decrease is less and appears to be
saturating. Below 25° C the opposite effect is observed, the slope is higher. Data at
higher levels of true strain is only available at lower temperatures since the uniform
strain decreases as the testing temperature increases.
The ultimate strength (maximum nominal stress), is plotted as a function of temperature in Figure 4.20 below. The UTS decreases with increasing temperature and a polynomial function was able to fit the data very well with a strong coefficient of determination, $R^2$, value of 0.993. Below 100 °C the relationship appears linear, but above this temperature the UTS begins to saturate.

The nominal uniform strain is plotted as a function of temperature in Figure 4.21. The uniform strain decreases linearly as temperature increases. A linear function was fit to the data points and has a strong $R^2$ value of 0.97.
Figure 4.20: Ultimate strength vs temperature for uniaxial tension specimens.

Figure 4.21: Uniform strain vs. temperature for uniaxial tension specimens.
4.5 Isothermal Tests for Strain-Rate-Sensitivity

In order to establish the rate-sensitivity of the stainless steel SS-304L tube, uniaxial tension tests were performed under isothermal 25 °C conditions. The isothermal conditions decoupled the temperature- and rate-dependent effects. The nominal response of the tube at strain-rates ranging from $10^{-1}$ to $10^{-5}$ s$^{-1}$ are shown in Figure 4.22 below. As the strain-rate increases, the yield stress, flow stress, ultimate strength, and uniform strain all increase. This relationship is the opposite of the temperature-dependence discussed in section 4.4. Each test has a similar hardening rate at all levels of strain.

The strain-rates for each test are plotted versus strain in Figure 4.23 and exhibit the same sinusoidal oscillation and step phenomenon previously discussed in section 4.4. The vertical axis of the plot is a logarithmic scale. A large range of strain-rates were captured in these tests.

The data for the fastest and slowest tests contain more noise than the other rates. In the case of the fastest rate, the oversampling of the load cell by the DIC software had to be reduced from 1024 to 20 data points per image in order to increase the camera frames per second (fps). At an oversampling rate of 1024 data points per image the maximum fps of the cameras is 5. This sampling rate would result in less than 25 data points to capture the stress-strain response of the tube. By reducing the oversampling to 20, the cameras can be operated at 30 fps which provides a more reasonable number of data points, but increases noise in the load cell readings. The slowest test lasted over eleven hours long. We suspect that changes in environmental conditions introduced more noise into the load cell readings and DIC strain measurements over this large length of time.
Figure 4.22: Nominal stress vs strain at different strain-rates and a constant temperature of 25 °C

Figure 4.23: Logarithmic Strain-rate vs. strain for 25 °C isothermal rate testing.
The difference between an isothermal and monotonic test at a strain-rate of $1.5\times10^{-2}$ s$^{-1}$ is shown in Figure 4.24. The yield stress and the work-hardening rate are identical in both cases, until 45% elongation when the monotonic curve begins to soften as the material temperature rises due to deformation induced heating. The temperature gradients of the two tests are shown in Figure 4.27. For the monotonic specimen, (a), the center of the specimen rises in temperature from 25 to 32 °C at 48% nominal axial strain, which corresponds to the onset of necking. Thereafter, the temperature of the necking region continues to rise dramatically until fracture, and reaches a peak temperature of 58 °C. Conversely, the isothermal specimen, (b), maintains a constant temperature profile continuously throughout the entire test. The necking and fracture are triggered earlier in the monotonic test by the temperature gradient. Furthermore, the neck appears to be more localized in the isothermal test when compared to the monotonic. This can be seen in the images of the test specimens shown in Figure 4.25.
Figure 4.24: Comparison of 25 °C Isothermal and monotonic nominal stress vs strain response.

Figure 4.25: Isothermal and Monotonic uniaxial tension specimens (1.5 x 10^{-2} s^{-1}).
Figure 4.26: Temperature profile of tube at different levels of axial strain for the monotonic (a) and isothermal (b) uniaxial tension tests.
A significantly larger elongation-to-fracture was obtained for several of the strain-rates, but not obtained for the fastest and slowest rates. The isothermal conditions minimize the temperature gradient, which in turn triggers localized necking. The highest strain-rate, $1.39 \times 10^{-1}\ \text{s}^{-1}$, failed at a nominal elongation of 55% because the tube wasn’t held at isothermal conditions during the test. The convective cooling provided to the tube by the constant flow of fluid from the refrigerated bath was not sufficient to counter the heat generated from deformation-induced heating of the tube at this high of a strain-rate, causing a small increase in temperature during the test. Though the rise in temperature is small it is sufficient to trigger the localized necking.

Infrared images of the tube during deformation captured the temperature of the tube to verify the 25 °C isothermal conditions. The temperature profile of the tube immediately before fracture was captured from the infrared data using a line tool and is shown in Figure 4.27 below. For all of the strain-rates, except the fastest, the profile is a constant 25 °C. The rate of heat generation from deformation-induced heating in the fastest strain-rate was greater than the convective cooling of fluid flow through the tube, as described above, causing a rise in temperature. The temperature spike is shifted from the center of the tube towards the downstream end of the fluid circulation (left side of the figure). At the upstream end, the fluid enters the tube at a temperature of 25 °C. As heat is transferred to the fluid from the tube the difference in temperature between the fluid and tube decreases, causing a decrease in the heat transfer rate as the fluid moves further down the tube. At the downstream end the tube, the rate of heat generation exceeds the convective cooling of the fluid flow causing the tubes temperature to rise.
A temporal profile of the temperature at the location where the neck will form was obtained by tracking a point in the infrared images and is shown in Figure 4.28. The neck temperatures are a constant 25 °C for all of the tests except for the fastest strain-rate. For the fastest strain-rate, the tube temperature slowly increases throughout the test and then it rapidly increases with the high levels of localized plastic strain experienced during necking. Prior to necking the rise in temperature is only 4 °C, which given Figure 4.19 should only reduce the flow stress by 3 MPa, but could contribute to triggering the necking instability.
Figure 4.28: Temperature at neck vs. normalized time for 25 °C isothermal testing.

The flow stress at different levels of axial strain is plotted versus the logarithmic strain-rate in Figure 4.29. The flow stress monotonically increases with the strain-rate for all levels of axial strain, and a step is observed between $10^{-4}$ and $10^{-3}$ s$^{-1}$. Similarly, the ultimate strength is plotted versus the logarithmic strain-rate in Figure 4.30. The ultimate strength increases with the strain-rate. A simple material model such as the power-law or the Cowper-Symmonds model [46] could not be accurately fit to the data because of the step in the data points between $10^{-4}$ and $10^{-3}$ s$^{-1}$. 

114
Figure 4.29: Flow stress vs logarithmic strain-rate at different levels of axial strain for isothermal rate testing.

Figure 4.30: Ultimate strength vs logarithmic strain-rate for isothermal rate testing.
CHAPTER 5
BIAXIAL EXPERIMENTS

5.1 Introduction

The purpose of this chapter is to establish the mechanical response of the Stainless Steel SS-304L microtube under biaxial stress states that are generated by a combination of internal pressure and external axial loading. Both radial (aka proportional) and corner (i.e., non-proportional) paths were implemented in the two-dimensional nominal axial-hoop-stress space, to investigate the plastic flow and formability of the tube. Anisotropic yield functions were fit to the experimental data at different levels of plastic work in order to capture the mechanical behavior for future numerical (FEA) models of the experiments. The path-dependence of the stresses and strains at failure was investigated by comparing the experimental results from the radial and corner paths.

5.2 Experimental Setup and Procedure

5.2.1 Test Methods

Biaxial experiments were conducted with the Psylotech µTS and Teledyne 65D Syringe Pump, which were described in detail in sections 3.1 & 3.2, whereby tension/compression and internal pressure were simultaneously applied to the stainless steel SS-304L microtube. Radial path tests with nominal stress ratios, \( \alpha = \sigma_r : \sigma_\theta \), of \{6, 3, 2, 1.33, 1.25, 1.1, 1, 0.8, 0.5, 0.25, -0.1, -0.2\} were conducted, in addition to uniaxial and pure hoop tension. The tube was inflated by two different methods for the radial paths. In the first method, referred to as Force-Volume control, the tube was
inflated under volume control while the axial load was controlled by the $\mu$TS to maintain a constant stress ratio. In the second method, referred to as Displacement-Pressure control, a displacement ramp was prescribed to the end of the tube by the $\mu$TS while the pump maintained the stress ratio by controlling the pressure in proportion to the induced axial load. The limit-load instability was observed for both control methods by prescribing strains and allowing the stresses to develop freely along the radial path.

Four corner paths were strategically positioned through the failure stress of radial paths. Two of the corner paths began along the pure hoop tension axis ($\theta \rightarrow x$), and the other two began along the uniaxial tension axis of the biaxial stress plane ($x \rightarrow \theta$). The $x \rightarrow \theta$ paths began under axial stress control until the corner stress was reached and then the hoop stress was increased under volume control while the axial stress was controlled to remain constant, which enabled identification of the limit load instability and failure mode. For the $\theta \rightarrow x$ paths, the axial stress was held constant as the tube was inflated under pressure control until the corner stress was reached. After reaching the corner stress a displacement ramp was prescribed while the pump held the pressure constant. Since the pressure and displacement were being controlled, the limit-load instability and failure mode could be properly identified.

5.2.2 Control Methods

Data Acquisition and Equipment

During the tests, axial load and crosshead position, and fluid pressure, flow, and volume were fed back to the Psylotest program in order to create a control signal for the actuators, and modulate the biaxial stress state of the tube. Closed-loop PID control of the axial load, position, and axial stress takes place in the Psylotest program, while closed-loop control for pressure and volume take place on the pump controller. The Psylotest program can send a 0-10V analog signal to the pump controller as a set-point.
for flow rate or pressure. The Correlated Solutions 3D DIC system, section 3.6, was used to collect strain data during the test. Pressure and axial load data were also collected by the DIC computer in order to synchronize the stress and strain measurements. A schematic of the data acquisition and control is shown in Figure 5.1.

Figure 5.1: Control schematic of biaxial experimental setup.

**Radial Path Control Methods**

A Force-Volume control biaxial test begins by the operator configuring a sensor follower stage in the Psylotest program, with the axial stress being the \( \mu \)TS drive control feedback and the hoop stress is the sensor being followed. A stress ratio amplitude and stage time length are set. The operator starts the stage in the Psylotest program and then starts the pump in a constant flow-rate mode. The pump plunger moves at a
constant velocity causing a decrease in system volume and an increase in pressure in the tube. Pressure transducer data is acquired by the Psylotest program and used to calculate a hoop stress using equation 3.5. The set-point in the axial stress PID controller running in the Psylotest program is calculated using equation 5.1 along with the stress ratio set by the operator and the calculated hoop stress. The controller modifies the lower crosshead position to control the axial stress to the set-point, thereby maintaining a constant stress ratio.

\[ \sigma_x = \alpha \sigma_\theta \]  

(5.1)

The Displ-Press control method differs from the Force-Volume method by switching which device is driving the test and which is following. A displacement ramp stage and stress ratio are configured by the operator in Psylotest. The axial stress generated in the tube by the displacement ramp is scaled by the stress ratio and equation 5.1 to determine a hoop stress and pressure set-point. The pressure set-point is sent to the pump as a 0-10 volt analog signal from the LabVIEW DAQ board. The pump controller operates in external analog control mode for pressure. In this mode, a 0-10 volt signal can be read by the controller and acts as the pressure set-point of the control loop running on the pump controller. The pump maintains the pressure at this set-point to keep the stress ratio maintained as an axial stress is induced by the displacement ramp.

There is no difference in the average uniform stress-strain response between both control methods since the resultant biaxial stress paths are identical. With that in mind, the second method is only effective for axial–stress-dominant paths. For hoop-stress-dominant paths a marginally stable control system is observed and the pressure oscillates during the plasticity portion of the test. The magnitude of oscillation increases as the stress path becomes more hoop–dominant, rendering the path non-monotonically increasing, even though it remains proportional. The nominal hoop stress-strain
response of the 1:4 biaxial stress path is shown using both control methods in Figure 5.2. The Displ-Press control method obtains an identical average response as the Force-Volume control method, but the oscillations which result from a marginally stable control system are observed.

An advantage of the Displ-Press control method is its ability to capture the stress-strain response farther past the limit load instability. In Figure 5.2 the Force-Volume control method stops at 0.27 hoop strain whereas the Displ-Press method continues to 0.34 hoop strain and captures a greater reduction in stress. When the tube begins to fail, large deformations lead to a reduction in axial load and pressure.

A significantly greater reduction is observed in the axial load rather than the pressure, because the μTS load train is relatively stiffer than the pressurizing fluid. When the pressure control is determined by the axial load, as it is for the Displ-Press control
method, the biaxial stress is reduced beyond the limit load instability which promotes continuation of the test and higher final strains. In the Force-Volume control method the axial load is controlled based on the pressure signature. Since the pressure remains constant, relative to the drop in axial load, the biaxial stress remains high and discourages higher strains.

The plane-strain inflation (1:2 biaxial stress path) tests were conducted using the grips described in section 3.3.5 along with the Teledyne Syringe Pump. The μTS is not required for the plane-strain inflation tests because pressurizing the tube internally results in the 1:2 biaxial stress path without any additional external loading. The tube was inflated under volume control by prescribing a constant flow rate to the pump until the tube bursts. By prescribing strain and not stress, the limit load instability can be captured, albeit the compressibility of the fluid limits the observability of this effect.

**Corner Path Control Methods**

The control scheme for the corner paths were slightly different than for the radial paths. For the $x \rightarrow \theta$ paths in the nominal stress space, shown schematically as the red path in Figure 5.3, a test profile is created in the Psylotest program with two stages. The first stage is reminiscent of a uniaxial tension test, whereby a ramp is prescribed in either force or displacement control, except for this test case the μTS control feedback type is axial stress. The second stage is a hold for a specified period of time for axial stress. The operator starts the test and the axial load is ramped to the desired corner stress of the path. In the experiments reported later in this chapter, this stress was set to the failure stress of the radial path that the specific $x \rightarrow \theta$ corner path was selected to correspond to. However, generally speaking, there is no limitation to what this stress should be, provided of course that it is below the UTS of the material. Once the corner stress is reached and the axial stress hold stage begins, the operator runs the pump in
volume control mode, increasing the hoop stress under constant axial stress, until the tube fails. The μTS is able to hold the axial stress constant by decreasing the external axial load on the tube as the axial force that is induced by the pressure increases during the second stage. Because the driving mechanism of the second stage of this test is volume control, the limit load instability and failure mode can be observed.

For the $\theta \rightarrow x$ paths, shown schematically as the green path in Figure 5.3, the stage orders are reversed. A two stage test profile is defined in the Psylotest program where the first stage is a hold for axial stress, and the second stage is an axial stress ramp.

Figure 5.3: Biaxial experiment stress paths.
The pump is operated in a pressure gradient control mode during these paths. In the first stage the pump ramps the pressure until the corner hoop stress, which in the specific experiments reported here is equal to the failure stress of the corresponding radial path, is reached. Meanwhile the μTS applies an apparent compressive load which balances exactly the force applied by the increasing pressure to the load cell, so that the axial stress on the tube is kept to zero. During the second stage the pump maintains a constant pressure keeping the hoop stress constant. For the 4:3 \( \theta \rightarrow x \) corner path the μTS prescribes a displacement ramp that induces an axial stress in the tube until failure. By prescribing a displacement the limit load instability and failure mode can be identified. For the 5:4 \( \theta \rightarrow x \) corner path the μTS ramped the axial stress until the tube failed. Since the driving mechanism of the second part of this test is force-control, the post limit load stress-strain path and failure mode have greater uncertainty. The 5:4 \( \theta \rightarrow x \) corner path test was performed prior to realizing that the μTS control mode could be switched from axial stress to displacement control during the test without any adverse effects.

### 5.2.3 Stress and Strain Reductions

Axial and hoop strain measurements were obtained in the biaxial experiments using 3D DIC measurement techniques with the virtual extensometer tool described in section 4.2.2.

The nominal axial and hoop stress were calculated by considering the equilibrium of the deforming shell, adopting the thin-walled approximation and assuming membrane-only deformations. This yielded equations 5.2 and 5.3 (which are identical to equations 3.5 & 3.6 that were implemented in the Psylotest software):

\[
\sigma_x = \frac{PR_o}{2t_o} + \frac{F}{2\pi R_o t_o} \tag{5.2}
\]

123
\[ \sigma_\theta = \frac{PR_0}{t_o} \]  

(5.3)

where \( P \) is the measured pressure in the tube, \( F \) is the measured \( \mu \)TS load cell reading, \( R_o \) is the initial center wall radius of the tube, and \( t_o \) is the initial average wall thickness.

The axial stress of the tube has two terms, of which one comes for the fluid pressure and one from the \( \mu \)TS tensile stage. The fluid pressure creates a force on the tube and grip assembly in the same sense that an axial force is induced to a pressurized closed vessel by the pressure that is acting on the end-caps of the tube. The tube supports this load which, for the case of the pressurized close vessel, results in a tensile axial stress equal to half of the hoop one, using the thin-walled assumption. The second term of the axial stress comes from the external load which is applied by the \( \mu \)TS and is distributed over the cross-sectional area of the tube. The hoop stress is simply taken from equilibrium considerations, as in the usual thin-walled pressure vessel equations.

The true stresses are calculated using the same equations as above, except the current tube geometry is extracted from the 3D DIC data is updated in the equations. The tube center wall radius, \( R \), was calculated using the true hoop strain data and equation 5.4:

\[ R = R_o \times \exp(\varepsilon_\theta) \]  

(5.4)

Assuming incompressibility and ignoring elastic strains, the 3\(^{rd}\) principal strain, \( \varepsilon_r \), can be calculated from the axial and hoop strain using equation 5.5

\[ \varepsilon_r = 0 - \varepsilon_x - \varepsilon_\theta \]  

(5.5)

The 3\(^{rd}\) principal strain is used to calculate the tube wall thickness, \( t \), with equation 5.6

\[ t = t_o \times \exp(0 - \varepsilon_\theta - \varepsilon_x) = t_o \times \exp(\varepsilon_r) \]  

(5.6)
The tube wall thickness and center wall radius are calculated for each data point in the experiment and are substituted into equations 5.2 & 5.3 for the initial values to determine the true stress.

5.2.4 Specimen Preparation

Biaxial test specimens were prepared in the same manner as described in section 4.4 except the tube, plug and resultant test gauge length varied depending on the stress path. Counterclockwise (CCW) of the 1:2 (axial:hoop stress), or plane-strain path in the biaxial stress plane, a compressive load is applied to the tube potentially causing failure by buckling. Decreasing the length of the tube decreases the slenderness ratio and increases the critical load at which the pressurized tube will buckle. (Note that while in the elastic case the presence of internal pressure stiffens the tube by reducing the geometric imperfections and delaying buckling, in the elasto-plastic case at hand the internal pressure induces a hoop stress which increases the equivalent stress, so that the tangent stiffness of the tube is reduced.) The 0:1 path tube lengths were reduced from 64mm (uniaxial tension) to 53mm and the -1:10 & -1:5 specimens were reduced even further to 48mm. The length of tube for the corner stress paths was determined by the initial path of the test. For the \( x \rightarrow \theta \) tests the initial path is identical to a uniaxial tension test, therefore the tube specimens were cut to 64mm length. For the \( \theta \rightarrow x \) the initial path is identical to the 0:1 radial path, therefore a 48mm tube specimen length was adopted. The corresponding gage-sections are 30mm and 14mm, respectively.

As described in section 4.2, during uniaxial tension tests, the tube diameter decreases causing the tube to form around the plug, thereby determining the effective test gauge length of the specimen. In biaxial stress paths the addition of the hoop stress counters the negative hoop strain. For all of the paths CCW of 2:1 the hoop strain is positive and the tube no longer forms around the plug, therefore the plug no longer
determines the test gauge length of the tube. For these stress paths the diameter of the hole in the gland, which surrounds the tube, restricts the positive hoop strain that develops. Therefore the gauge-length of the tube is taken as the distance between the glands at each end. This distance was prescribed to be 30mm for all of the biaxial tests including and clockwise (CW) of 1:4, 15mm for 0:1 and 10mm for -1:10 & -1:5 tests.

For the biaxial tests including and CCW of 1:2, a standard 3/32" HiP gland with a single 3/32" HiP sleeve was used in place of the custom gland and two sleeve design described in section 3.3.2. The axial load is sufficiently low, so that the two sleeve design is no longer required.

The tube may fail by bursting in a biaxial path, therefore it is important to orient the thinnest tube wall in the direction of the DIC cameras to capture the failure and maximum strain. After the tube has been cut and the ends deburred, the wall thickness is observed under an optical microscope and the thinnest section is determined and marked. After the tube has been painted for DIC measurement, the plugs are inserted into the ends of the tube and the glands and sleeves are slid onto the ends of the tube. One end of the tube is tightened down into a biaxial grip. Next the other end is secured to the other biaxial grip. One of the ends must be adjusted until the two biaxial grips are properly aligned and can be easily slid onto the biaxial tester without imposing any plastic strain on the tube. After setting the load cell to -3.5 N (weight of a single biaxial grip), the grip and tube assembly is inserted into the µTS. The lower crosshead is adjusted during this process so that the assembly can be slid on easily without any effort. The grips are centered in the load train by adjusting the distance from the front of each crosshead to the front of each biaxial grip to 4.87mm and locked in place by engaging the plunger pins of each grip in a star pattern. The lower crosshead position is manually adjusted when tightening the plunger pins to maintain a zero load. Once the biaxial grips are secured on the crossheads, the 1/16" stainless steel SS-316 hydraulic
tubing is attached to the biaxial grips. To remove any air present in the hydraulic system the pump is run in a constant flow-rate mode at 2 ml/min until 10 ml of fluid have been purged from the system leaving the volume of the fluid available for pressurization at less than 5 ml. The hydraulic tubing on the upper crosshead biaxial grip is removed and replaced with a plug to close the system. Figure 5.4 shows a test specimen secured in the biaxial grips and mounted in the µTS with both hydraulic connections attached.

Figure 5.4: Biaxial test specimen in µTS immediately prior to testing.

An optically clear acrylic shield is placed over the µTS or plane-strain grips to protect the equipment and operators from the jet of fluid created when the tube bursts. The operator configures a test program, strain measurement, and data collection, and starts the test.
5.3 Results - Radial Paths

Fourteen radial biaxial paths in the axial-hoop nominal stress space were tested in order to capture a significant number of experimental data in both the biaxial stress and strain planes. The nominal biaxial stress paths, shown in Figure 5.5, were performed as described in section 5.2.1 above. The paths as seen to be well-controlled and very linear. The nominal failure stress is marked for each path and naturally shows a little drop in stress after the limit-load instability is attained. Tracking the response past that instability has been possible since the tubes were inflated under volume-control. The uniaxial tension test is the only path which shows a significant drop in stress after the maximum load.
The test system maintained a constant ratio between the nominal stresses. The change in tube geometry was not considered, therefore the resultant true stress paths, Figure 5.6, are non-linear. This non-linearity is different for each path and mostly insignificant for paths CW of 0:1. CCW of 1:4, the non-linearity becomes more significant as shown by the -1:5, -1:10 and 0:1 stress paths.

Figure 5.5: Nominal hoop vs axial stress paths.
The induced nominal strain paths and uniform strains are shown in Figure 5.7 below. The strain paths are seen to be close to linear. To the first degree, the formability of the tube is represented in different strain paths by the uniform strain limit. This is identified as the strains that correspond to the pressure maximum that was recorded in each biaxial response [14]. Surrounding the hoop strain axis, the uniform strain forms a noticeable “v” which is typical of a formability plot (aka Forming Limit Diagram, or FLD). The plane-strain (1:2) path has the lowest formability, as is expected [46]. The -1:5, -1:10, & 0:1 paths show that compressing the tube increases the hoop formability during inflation. We expect to see a second “v” pattern surrounding the axial strain axis. However, while one side of the “v” is observed below the axial strain axis, the uniform
strains continue to decrease even above that axis. This can be explained by considering the failure modes of the burst tubes, described next.

Figure 5.7: Nominal hoop strain vs. axial strain.

The failure modes of the tube, shown in Figure 5.8, vary depending upon the biaxial stress path. In both pictures the untested tube is shown at the original gauge length. For axially dominated paths, the tube necks in the center of the tube and fails circumferentially in a similar manner to a uniaxial tension test. For hoop dominated stress paths, the tube initially expands uniformly, maintaining its circular-cylindrical shape except of course at the gripped ends. Soon after the pressure maximum, the tube forms an axisymmetric bulge that, depending on the loading path, can deform in a stable fashion, even under descending pressure. The bulge evolves into a non-axisymmetric one and almost immediately after the tube bursts along a tube generator [14]. The
opening left behind from the rupture of the tube becomes smaller as the path becomes more hoop stress dominant. Both of these failure modes are identical to the failure modes found by Korkolis and Kyriakides [14] on 60 mm (2.36 in) OD Al-6260-T4 tubes. In picture (b) the hoop strain is noticeably non-uniform and forms a barreled shape. This is caused by the small test gauge length required to prevent buckling of the tube under the compressive axial load.
The nominal axial stress-strain response for each biaxial test is shown in Figure 5.9 and Figure 5.10 below. Starting at the uniaxial tension (1:0) and moving CCW in the biaxial stress plane towards the -1:5 path, the axial stress increases until 3:1 and then decreases thereafter while the total elongation steadily decreases. This stacking of the curves is demonstrated in Figure 5.10 which shows the axial stress-strain response zoomed in near the yield point. The axial strain becomes negative CCW of the axial plane-strain stress path. This is expected, given the curvature of the yield locus.

The true axial stress-strain response for each biaxial test is shown in Figure 5.11 below. The same characteristics described above for the nominal response are observed in the true response. The paths CCW of 1:2 take a noticeably strange path which is a result of both controlling the stress paths using nominal calculations, the large deformations and the omission of considering the axial curvature that the tubes develop in these paths. Also, notice that no consideration was given to the post-uniform
response, which was converted to true using the same equations as for the uniform regime.

Figure 5.9: Nominal axial stress vs. strain.
Figure 5.10: Nominal Axial Stress vs. Strain zoomed in near the yield point and truncated thereafter.

![Diagram of stress-strain relationship for SS-304L material](image)

Figure 5.11: True axial stress vs. strain.

The hoop stress-strain responses for each biaxial stress path are shown below in Figure 5.12. Beginning from the uniaxial tension (1:0) stress path and moving CCW in the biaxial stress plane we observe an increase in flow stress and hoop strain until reaching a peak for the axial plane-strain (1:2) stress path. Past the plane strain test the hoop strain continues to increase whereas the flow stress decreases. Figure 5.13 shows the hoop stress-strain response zoomed in near yielding and the stacking of the curves for each stress path can be observed. The true hoop stress-strain responses for each biaxial stress path are shown in Figure 5.14. The true response shows the same trends as described above for the nominal curves.
Figure 5.12: Nominal hoop stress vs. strain.
5.3.1 Yield Function Fitting

Yield functions were fit to the experimental biaxial data at different levels of plastic work, which was calculated using equation 5.7

\[ dW_p = \tau_{ij} d\varepsilon_{ij}^p = \tau_x d\varepsilon_x^p + \tau_\theta d\varepsilon_\theta^p \]  

(5.7)

where \( \tau_x \) & \( \tau_\theta \) are the true axial and hoop stress, and \( d\varepsilon_x^p \) & \( d\varepsilon_\theta^p \) are the true incremental axial and hoop plastic strain. The first yield function that was fit to the experimental data
was the isotropic von Mises model. Equation 5.8 is a reduced form of the model for principal plane-stress states, which is the state of stress for the biaxial experiments.

\[
\sigma_{vM} = \sqrt{\sigma_x^2 - \sigma_x\sigma_\theta + \sigma_\theta^2}
\]  
(5.8)

The true biaxial stress is shown for each biaxial path at 5, 25, 50 & 75 MPa of plastic work along with the von Mises model in Figure 5.15 below. The plastic work levels chosen correspond to 1%, 5%, 10% and 15% nominal axial strain in a uniaxial test in the axial direction, respectively. At a plastic work of 5 MPa the von Mises model accurately captures the experimental data, suggesting that the material is initially isotropic. As the plastic work is increased the von Mises model progressively deviates and is no longer representative overall. The equibiaxial data and nearby paths maintain fairly good accuracy with the model, but the hoop and axially dominated paths fall short of the model stress predictions. The symmetrical shape of the von Mises model is helpful to visualize the anisotropy of the material. While initially isotropic, it is evident by the drastic mismatch between the model and experimental data, particularly for the hoop dominate stress paths (Figure 5.15) that the material becomes anisotropic at higher levels of plastic work.
This anisotropy is captured in Figure 5.16, which shows the differential work hardening of the tube for different biaxial stress paths. The true hoop and axial stresses have been normalized by the true uniaxial flow stress for each level of plastic work. The hoop-dominated paths have less work hardening and therefore migrate inwards more aggressively than the axially dominate paths at higher levels of plastic work. The anisotropy of the material and shape of the experimental data suggest adopting a different model that can capture these effects.
As a further verification of the performance of the von Mises material model, the direction of plastic strain is plotted versus the loading direction for each biaxial stress path at increasing levels of plastic work in Figure 5.17, along with the von Mises model. The plot shows that even at low levels of plastic work the von Mises model does not accurately capture the experimental data, except around the uniaxial and equibiaxial regions, and warrants adopting more complex models.
The Yld2000-2D anisotropic yield function model proposed by Barlat et al. [15] was adopted to better represent the biaxial data. This model is based on the non-quadratic, isotropic Hershey-Hosford yield function [46] written in terms of the stress deviator. Two linear transformations of the stress tensor inject anisotropy while retaining the convexity. There are 8 parameters from the two transformations which can be used to alter the anisotropy of the model to conform to the shape of the experimental data. This model is very flexible and has been utilized before ([47],[48]) to capture the anisotropic effects in other FCC-austenite based metal alloys, with high accuracy.

The experimental data was input to a Matlab code which modifies the 8 anisotropic alpha (\(\alpha\)) parameters to adjust the shape of the model and fit it to the data. The model
exponent was set to 8, which is appropriate for the FCC-Austenite grain structure of the microtube ([15],[49]). The alpha parameters were all set at initial values of 1 and the resultant, non-quadratic but isotropic yield surface is generated. Distances between the surface and the experimental data are calculated and used to modify the parameters to change the shape and reduce the distance. This process is repeated until a minimum distance between the surface and experimental data is achieved within a range.

The optimized Yld2000-2D model, von Mises model, and experimental data are shown in the normalized biaxial stress plane in Figure 5.18. Note that the -1:5 to 0:1 stress paths are shown but were not used to optimize the shape of each model because of their aforementioned non-linearity. With that in mind, at low levels of plastic work, the models fit these stress paths since they are still mostly linear and the material is isotropic. At higher levels of plastic work these paths don’t agree with the model. Looking at the other biaxial stress paths the Yld2000-2D model is able to significantly improve upon von Mises. Overall the shape of the optimized model can be characterized by a more pronounced curvature in the direction of the equibiaxial stress path and flattened regions to their sides. Table 5.1 shows the distance error for the optimized Yld2000-2D and von Mises model. Points with high error are shown in red. The Yld2000-2D model shows significantly less error for all of the experimental data points, but most noticeably for the 3:1, 2:1, 4:5, 1:2, & 1:4 stress paths. On these paths the shape of the Yld2000-2D is better suited to capture the flattened portions of the plastic work contour than the von Mises model.
Figure 5.18: Normalized plastic work contours with Von Mises and optimized Yld2000-2D

Table 5.1: %Err for von Mises & Yld2000-2D yld function models at 75MPa plastic work.

<table>
<thead>
<tr>
<th>Axial to Hoop</th>
<th>von Mises</th>
<th>Yld 2000-2D</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 to 0</td>
<td>0.00%</td>
<td>0.00%</td>
</tr>
<tr>
<td>6 to 1</td>
<td>1.09%</td>
<td>0.82%</td>
</tr>
<tr>
<td>3 to 1</td>
<td>3.14%</td>
<td>1.17%</td>
</tr>
<tr>
<td>2 to 1</td>
<td>4.97%</td>
<td>1.03%</td>
</tr>
<tr>
<td>4 to 3</td>
<td>0.34%</td>
<td>0.28%</td>
</tr>
<tr>
<td>5 to 4</td>
<td>1.67%</td>
<td>0.49%</td>
</tr>
<tr>
<td>10 to 9</td>
<td>1.40%</td>
<td>3.29%</td>
</tr>
<tr>
<td>1 to 1</td>
<td>1.66%</td>
<td>1.30%</td>
</tr>
<tr>
<td>4 to 5</td>
<td>5.95%</td>
<td>1.37%</td>
</tr>
<tr>
<td>1 to 2</td>
<td>7.21%</td>
<td>1.67%</td>
</tr>
<tr>
<td>1 to 4</td>
<td>6.79%</td>
<td>0.83%</td>
</tr>
</tbody>
</table>
The Yld2000-2D model can be implemented in a UMAT to improve numerical (FEA) simulations as shown by Korkolis, et al. [49] and Dick [48]. Typically a von Mises isotropic model would be used in a numerical (FEA) model, but this would result in significant error in the simulations since this model doesn’t accurately predict the behavior of this material as previously shown. Because the Yld2000-2D is only a 2D model, its use is limited to 2D simulations with plane stress or shell elements, and cannot be used with 3D solid elements.

In order to conduct finite element simulations with 3D solid elements, the 18 parameter Yld2004-3D model [15] was also fit to the experimental data. The addition of 10 adjustable parameters further enhances the model’s capacity to match the shape of experimental data, albeit adding significant complications in calibrating the model. The model was implemented in the same Matlab code with the same exponent and iterative technique previously discussed for the Yld2000-2D model. Figure 5.19 shows the optimized Yld2004-3D & von Mises models, along with the experimental data in the true biaxial stress plane. The model is a significant improvement upon the von Mises. The shape is characterized by a more pronounced curvature near the equibiaxial stress path and flattened regions on both sides.

The distance % error at 75 MPa of plastic work is compared for the Yld2004-3D & von Mises model in Table 5.2. High errors are noted by the red text. The Yld2004-3D model has less error for the majority of the stress paths and significantly less error for the same paths that were found in the case of the Yld2000-2D model (3:1, 2:1, 4:5, 1:2, 1:4).
Figure 5.19: Normalized plastic work contours with Von Mises & optimized Yld2004-3D.

Table 5.2: %Error for von Mises & Yld2004-3D yield function models at 75MPa plastic work.

<table>
<thead>
<tr>
<th>Axial to Hoop</th>
<th>von Mises</th>
<th>Yld 2004-3D</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 to 0</td>
<td>0.00%</td>
<td>0.01%</td>
</tr>
<tr>
<td>6 to 1</td>
<td>1.09%</td>
<td>1.20%</td>
</tr>
<tr>
<td>3 to 1</td>
<td>3.14%</td>
<td>1.36%</td>
</tr>
<tr>
<td>2 to 1</td>
<td>4.97%</td>
<td>1.49%</td>
</tr>
<tr>
<td>4 to 3</td>
<td>0.34%</td>
<td>0.39%</td>
</tr>
<tr>
<td>5 to 4</td>
<td>1.67%</td>
<td>0.69%</td>
</tr>
<tr>
<td>10 to 9</td>
<td>1.40%</td>
<td>4.35%</td>
</tr>
<tr>
<td>1 to 1</td>
<td>1.66%</td>
<td>2.04%</td>
</tr>
<tr>
<td>4 to 5</td>
<td>5.95%</td>
<td>2.28%</td>
</tr>
<tr>
<td>1 to 2</td>
<td>7.21%</td>
<td>1.49%</td>
</tr>
<tr>
<td>1 to 4</td>
<td>6.79%</td>
<td>1.30%</td>
</tr>
</tbody>
</table>
Since the Yld2000-2D & Yld2004-3D optimized models are characterizing the same material the shape of each model should be the same. Furthermore, since the models will be implemented in finite element simulations with different element types, their shape should be the same in order to get consistent results. Figure 5.20 shows each model and the experimental data in the biaxial stress plane. Overall the models have nearly identical shapes which is optimal for getting consistent results from FEA simulations.

Figure 5.20: Normalized plastic work contours with optimized Yield 2000-2D & Yield 2004-3D.
5.4 Results - Corner Paths

It is well-accepted that plastic deformations are path-dependent. As a result, the failure limits that were determined in section 5.3 are also expected to depend on the loading path that was used to determine them. On the other hand, it has been suggested [46] that the stresses that correspond to failure are less dependent, or are even independent of the loading path. The path-dependent failure of our stainless steel SS-304L microtube was evaluated by taking corner paths to the failure stresses determined for several radial paths and continuing to increase the stress until failure. If the tubes failed under the corner paths at the same stress levels as they did in the radial ones, this would clearly indicate that the failure stresses are path-independent for this material. The opposite conclusion would be drawn if the tubes failed at different stress levels.

The yield and ultimate stress of the 1:0 and 0:1 uniaxial tension tests limited which paths were potential candidates for these tests. The corner stress of the path must be greater than yield stress of the initial path and less than the failure stress. Due to these restrictions only the equibiaxial (1:1) and 10:9 radial paths were conducted for the $x \rightarrow \theta$ paths, and the 4:3 and 5:4 radial paths were tested for the $\theta \rightarrow x$ paths. These paths are shown along with the corresponding radial paths in Figure 5.21. While three of the corner paths reach the failure stresses of their radial path counterparts and are able to continue, the 5:4 $\theta \rightarrow x$ test never reaches the failure stress of the original path. Three of the tests (1:1, 10:9, & 5:4) show that the failure stress of the tube is path-dependent, while the 4:3 path fail at a nearly identical biaxial stress state. In the same way that the true stress paths are non-linear for the radial tests, the corner path non-linearity is shown in Figure 5.22.
Figure 5.21: Nominal hoop vs axial stress.

Figure 5.22: True hoop vs. axial stress.
The nominal strain paths are shown for both radial and corner paths in Figure 5.23. The corner paths initially trace either the uniaxial hoop or axial tension strain path before changing direction, which corresponds to the change in direction of the stress path. Each test revealed the path-dependency of the failure strains, as expected. In every case, the failure strains of the corner paths were significantly different from those of the corresponding radial paths.

The 10:9 corner path ended shortly after yielding during the second leg of the corner path test. This test was repeated several times with the same result. The tube failed by bursting at the end of the test gauge length near the plug and gland rather than at the center of the specimen, which was the case for all of the other biaxial stress tests. Extra care was taken to remove sharp edges and smooth the plug and gland surfaces which interacted with the tube because of the suspicious failure location. This approach had no impact on the failure. Furthermore, the plug length was modified to extend beyond the gland for one test and well within the gland on another test, also to no avail. Since the specimen didn’t fail in the test section, we cannot draw any conclusion regarding the failure strain path dependence or failure mode. Regarding the remaining three paths (5:4, 1:1, & 4:3), all of them failed by bursting along a tube generator, which was the same failure mode for their corresponding radial paths, except for the 4:3 path. The 4:3 radial path failed by circumferential rupture and can be seen in Figure 5.8. The change in failure mode was also observed by Korkolis & Kyriakides [10].
The nominal and true axial stress-strain responses for the corner paths are shown in Figure 5.24 and Figure 5.25. The 10:9 and 1:1 \( x \rightarrow \theta \) stress-strain response initially follow the uniaxial tension response quite nicely. After the corner stress is reached the axial stress is held constant, but the axial strain continues to increase as the hoop stress is increased. This indicates that the subsequent yield surface (which has not been explicitly determined in this work) has a normal with a small inclination to the right, with respect to the vertical. In the true stress-strain response we observe a drop in the 1:1 \( x \rightarrow \theta \) path. This is a result of controlling the nominal stress and ignoring the true stress on the tube. The 4:3 and 5:4 \( \theta \rightarrow x \) stress-strain response begins with negative axial strain from the radial contraction as the hoop stress increases.
After the corner stress is reached and the axial stress begins to increase, we notice a very linear region before yielding at a higher stress than the radial paths. This represents the material deforming elastically: after turning the corner, the stress state is inside the yield surface, since the 1st branch was uniaxial. The significant increase in yield stress suggests there is significant isotropic hardening (i.e., expansion of the yield surface) in addition to kinematic hardening, before the material re-yields and the response becomes non-linear. The 5:4 corner path hardens very little before failing, but the 4:3 path shows significant hardening which was not seen in prior research [10]. The 4:3 path stress drops significantly after the limit load is reached. This effect could be captured because the 2nd branch of the test was conducted under displacement control. The post limit load stress doesn’t drop in the 5:4 path, because the test was conducted under axial stress.
control. This test could be repeated in order to determine the correct response of the tube after the limit load. Another interesting feature of these tests is that performing a pure hoop tension test is not as obvious as a poor axial tension one. The user needs to decide beforehand if what will remain zero is the nominal or the true stress, both of them equal to zero being impossible given equation 5.2: since the current radius and thickness are different from the original ones, the force over pressure

![Figure 5.25: True axial stress vs strain.](image)

ratio for zero axial stress will depend on which configuration of the body is considered. In our current set-up, only the nominal axial stress can be controlled to remain zero, which implies that the true axial stress will not. Of course, in the case of axial tension, both the nominal and the true hoop stresses are zero throughout the test, since they are both
proportional to the pressure, which is kept to zero. This peculiarity of the pure hoop tension experiment is shown in both Figure 5.25 and Figure 5.11.

The nominal and true hoop stress-strain responses are shown in Figure 5.26 and Figure 5.27, and are very much similar to the results of the axial stress-strain responses. Initially the 5:4 and 4:3 $\theta \rightarrow x$ corner paths follow the uniaxial hoop tension curve. After the corner stress is reached the paths remain at a constant state of stress while the strain continues to increase due to the increase in axial stress and the attendant radial contraction effect. The 10:9 and 1:1 $x \rightarrow \theta$ stress-strain responses begin with a negative hoop strain caused by the radial contraction effect and axial stress. After the corner stress

![Figure 5.26: Nominal hoop stress vs. strain.](image-url)
is reached and the hoop stress begins to increase, we notice a very linear region before yielding at a higher stress than the radial paths. The significant increase in yield stress confirms the axial stress findings that there is significant isotropic hardening in addition to kinematic hardening. After yielding, both paths exhibit very little hardening before failing, which agrees with prior research [10]. The 10:9 fails abruptly after yielding, while the 1:1 path continues for a considerable amount of strain.

Figure 5.27: True hoop stress vs. strain.
CONCLUSIONS

The mechanical and material behavior of stainless steel SS-304L microtube was investigated by metallographic, uniaxial, isothermal and biaxial testing. Optical microscopy techniques were utilized in Chapter 2 to determine the tube dimensions, microhardness and grain structure. Though the nominal dimensions of the tube were given by the manufacturer as an outside diameter of 2.38 mm (0.0935 in) and a wall thickness of 150 µm (0.00589 in), the actual dimensions were found to be 2.4 mm (0.0944 in) and 161 µm (0.00633 in), respectively. The variation in wall thickness (tube eccentricity) was captured in Figure 2.4.

The grain structure of the tube was revealed using electroetching techniques with a 10% Oxalic acid electrolyte solution for both the R-θ (Figure 2.5) and R-Z planes (Figure 2.6). The grains showed no elongation effects from the tube extrusion manufacturing processes, presumably because it was fully annealed post-manufacturing, allowing the grains to fully recrystallize. The average grain size was determined to be 14 µm in diameter which results in 12 grains through the thickness of the tube, justifying the adoption of a continuum approach to model the behavior of the material.

The average hardness of the tube was determined to be 207 HV (Figure 2.10) by making both 50 gf and 300 gf indentations at evenly spaced locations around the R-θ plane with a microhardness tester. The tube hardness had no discernable dependence on the angular position in the R-θ plane, which reinforces the idea that the grains were full recrystallized during the annealing process.

Stainless steel 304L, as well as it’s higher carbon content variation SS-304 [12], is prone to strain-induced martensitic transformation [36], which increases with lower carbon content and at higher levels of plastic strain. This phenomenon was investigated by performing X-ray diffraction measurements of uniaxial tension specimens after
deformation, but no sign of BCC/BCT-martensite could be found. The results showed a presence of only FCC-austenite grain structure.

In Chapter 3 uniaxial tension tests were conducted on the tube itself, and axial strips taken from the tube, to capture the material response. Custom grips (Figure 3.9) were fabricated from spherical ball joints to ensure a pure uniaxial tension test without any bending moments. A custom taper seal fitting was designed to grip the tube so it could be axially loaded in tension (Figure 3.10) as well as inflated by pressure through hydraulic fluid for biaxial testing, though the latter was done with a separate set of grips.

Tube specimens were pulled on a meso-scale tensile stage, referred to as the µTS, at a strain-rate of $1.5 \times 10^{-3} \text{s}^{-1}$ while strain data was collected with the 2D/3D correlated solutions DIC system. A sinusoidal oscillation (Figure 4.3) with a peak to peak amplitude of $0.6 \times 10^{-3} \text{s}^{-1}$ was discovered in the strain-rate data and was found to be a result of a misalignment of the ball-screw and a high sensitivity of the displacement sensor to the oscillation. In addition to the oscillations, a step of $0.5 \times 10^{-3} \text{s}^{-1}$ in the strain-rate was also observed. This step is caused by a reduction of the effective gauge length of the tube between 10 and 15% elongation. The tube was found to have a yield strength of 452 MPa, an ultimate strength of 660 MPa, uniform strain of 45%, and an elongation of 55% at fracture (Figure 4.2).

Full field 3D strain measurements were used to capture the geometry and axial and hoop strain evolution throughout the experiment. Two types of diffuse necking were observed (Figure 4.8), the first of which is related to a reduction in diameter of the tube and the latter to a reduction in thickness. Tension tests on axial strips cut from the tube wall showed a similar response to the tube, albeit failing prematurely from imperfections introduced during the machining process (Figure 4.12).

The rate- and temperature-dependence of the microtube were decoupled and characterized through isothermal uniaxial tension tests. Barbed hose fittings were
attached to the uniaxial tension grips so that a heat transfer medium (water or white mineral oil) could be passed through the microtube during testing (Figure 3.12). A refrigerated bath was used to circulate the heat transfer fluid through the tube to maintain a constant temperature. Tests were conducted at a constant strain-rate of $10^{-3}$ s$^{-1}$ for temperatures of 6, 25, 50, 75, 100, & 142 °Celsius and strain data was collected using the 2D correlated solutions DIC strain measurement system. The temperature of the tube was monitored with a FLIR SC-645 infrared camera. The yield stress, flow stress, ultimate strength, uniform strain and elongation at fracture all decreased as the temperature increased (Figure 4.14), which agrees with previous research on Stainless Steel 304 material [13].

Tests were also conducted at a constant temperature of 25 °C for strain-rates ranging from $10^{-5}$ to $10^{-1}$ s$^{-1}$ (Figure 4.22). The isothermal conditions increased the elongation at fracture by eliminating temperature gradients, caused by deformation induced heating, which trigger the necking instability. The yield stress, flow stress, and ultimate strength all increased with the strain-rate. The results of the isothermal tests will be essential in the future to develop accurate thermo-mechanical numerical (FEA) models to simulate these experiments and forming processes such as hydroforming.

An experimental setup was designed to perform biaxial stress tests on the tube through a combination of internal pressure and axial loading. The setup consisted of the μTS, servo-driven hydraulic pump (Teledyne Isco Syringe Pump), 2D/3D DIC strain measurement system, and custom biaxial testing grips. The μTS LabVIEW control software was modified to collect pressure data, and calculate and control both hoop and axial stress. The setup and its controller are detailed in Chapter 4 of this thesis.

Two types of biaxial stress tests were conducted in Chapter 5. The first test involved radial stress paths where the axial and hoop stress were maintained in a constant proportion to each other until failure. Fourteen radial paths were conducted (Figure 5.5)
and the forming limits of the microtube (Figure 5.8), given by the biaxial strain paths, were established. Two failure modes of the tube were identified (Figure 5.8) and agreed with previous tube inflation research performed on macros scale aluminum tubes [14].

Constructing the plastic work contours of the microtube revealed that the material is initially isotropic, but found to experience differential work-hardening (Figure 5.16), leading to anisotropy at higher levels of plastic work. In order to capture these effects three yield functions, von Mises, Yld2000-2D, and Yld2004-3D were optimized and fit to the biaxial experimental data points at multiple levels of plastic work (Figures 5.19-5.21). The von Mises model was successful at capturing the shape of the initial isotropic yield surface but deviated from the data at higher levels of plastic work. The anisotropic yield functions, Yld2000-2D, and Yld2004-3D, were able to capture the shape of the data at all levels of plastic work and were significantly more accurate than the von Mises model.

The second set of biaxial stress tests involved taking corner paths through the failure stresses of several radial paths to investigate the path-dependence of the failure stresses and strains. Four corner paths were traced, two along a $x \rightarrow \theta$ path and two along a $\theta \rightarrow x$ path (Figure 5.21). All four of the corner paths demonstrated that the failure stresses and strains are path-dependent, but the former being relatively less so. The yield stress of the tube was found to be significantly higher on the second segment of the corner path which suggests the tube experienced isotropic hardening after yielding during the first segment (Figures 5.25 & 5.27). Furthermore, after yielding during the second segment there was very little hardening experienced for 3 of the 4 corner paths, which is consistent with previous research [10]. Conversely, the 4:3 $x \rightarrow \theta$ path underwent significant hardening after yielding which had not previously been seen.

The work reported in this thesis indicates that a custom experimental apparatus for applied well-controlled biaxial stress paths on microtubes has been created. Experimental results on stainless steel 304L are reported, including anisotropic yield
functions calibrated to the data. The future steps of this research are: a) grow the grains of the tubes, investigate the response of the resulting oligocrystalline material and contrast it with the present results, b) study the deformation-induced roughening of the oligocrystalline material, and c) use the custom apparatus to investigate the anisotropic plastic flow of other materials, as the need arises.
LIST OF REFERENCES


[38] http://www.isco.com/WebProductFiles/Applications/105/Video s_and_Animations/Pump_Overview.swf


[45] Thermo Scientific NESLAB RTE Series Refrigerated Bath Thermo Scientific Manual P/N U00694 Rev. 03/06/07


APPENDIX A.

Certification of Compliance / Material

MicroGroup Inc.
7 Industrial Park Road
Medway, MA 02053-1732
Ph: 508-533-4925 / 1-800-ALL-TUBE
Fax: 508-533-1593

Customer: UNIVERSITY OF NEW HAMPSHIRE
53 AC-DAGMAC WAY
RM 101
DURHAM, NH 03824
UNITED STATES

Certification No.: 60056
Certification Group:
Cost P/N: 5E20R 91312
MG Order: 300000062956
Cost Desc/PN: Tubing/Fractional
MG Item: 304100097X006SL

Material Characteristics

Physical Properties:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>Stainless Steel 304L</td>
</tr>
<tr>
<td>Gauge</td>
<td>0.0182</td>
</tr>
<tr>
<td>Tensile Min</td>
<td>10,800</td>
</tr>
<tr>
<td>Tensile Max</td>
<td>11,500</td>
</tr>
<tr>
<td>Yield Min</td>
<td>10,800</td>
</tr>
<tr>
<td>Yield Max</td>
<td>11,500</td>
</tr>
<tr>
<td>Elongation</td>
<td>10%</td>
</tr>
</tbody>
</table>

Chemical Properties:

<table>
<thead>
<tr>
<th>Element</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.0118</td>
</tr>
<tr>
<td>Si</td>
<td>0.0009</td>
</tr>
<tr>
<td>Mn</td>
<td>0.54</td>
</tr>
<tr>
<td>P</td>
<td>0.0013</td>
</tr>
<tr>
<td>S</td>
<td>0.006</td>
</tr>
</tbody>
</table>

Mechanical Properties:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile Strength</td>
<td>10,800</td>
</tr>
<tr>
<td>Yield Strength</td>
<td>11,500</td>
</tr>
<tr>
<td>Elongation</td>
<td>10%</td>
</tr>
</tbody>
</table>

Compliance and Specifications:

- ASTM A669 Less Hydro & Phase DFAE Std BS

Additional Comments:

12: BAI QAPR & ROS Compliant

Country of Origin:

- Manufacturer: UNITED STATES
- Main Source: CANADA

We hereby certify that all the items in the shipment have been produced, inspected and found to be in compliance with the applicable drawings, military specifications and/or standards, and purchase order requirements. All documents submitted with this invoice are in the name of the contractor to be purchased by the buyer. Subcontracting records are on file and subject to review upon request.

Where applicable, MicroGroup also certifies that the lot numbers and any detailed items are correct as contained in the records of the contractor. Because the MicroGroup has no control over the subsequent processing of product specification, the MicroGroup hereby disclaims any and all responsibility, warranty or liability, other than the warranty herein set forth below. Such disclaimer includes without limitation warranty, or fitness for particular purpose and warranty of merchantability.

Authorized Signature: [Signature]

Date of Approval: 09/14/2012

Page 1 of