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**EXPERIMENTAL INVESTIGATION AND PLASTICITY MODELING OF
SS316L MICROTUBES UNDER VARYING DEFORMATION PATHS**

BY

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THESIS

Submitted to the University of New Hampshire
in Partial Fulfillment of
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ABSTRACT

EXPERIMENTAL INVESTIGATION AND PLASTICITY MODELING OF SS316L MICROTUBES UNDER VARYING DEFORMATION PATHS

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Elizabeth M. Mamros

University of New Hampshire, December, 2020

Results for SS316L microtube experiments under combined inflation and axial loading for single and multi-loading segment deformation paths are presented along with a plasticity model to predict the associated stress and strain paths. The microtube inflation/tension machine, utilized for these experiments, creates biaxial stress states by applying axial tension or compression and internal pressure simultaneously. Two types of loading paths are considered in this paper, proportional (where a single loading path with a given axial:hoop stress ratio is followed) and corner (where an initial pure loading segment, i.e., axial or hoop, is followed by a secondary loading segment in the transverse direction, i.e., either hoop or axial, respectively). The experiments are designed to produce the same final strain state under different deformation paths, resulting in different final stress states. This difference in stress state can affect the material properties of the final part, which can be varied for the intended application, e.g., biomedical hardware, while maintaining the desired geometry. The experiments are replicated in a reasonable way by a material model that combines the Hill 1948 anisotropic yield function and the Hockett-Sherby hardening law. Discussion of the

grain size effects during microforming impacting the ability to achieve consistent deformation path results is included.

JOURNAL PUBLICATION DETAILS

This thesis is based on the following publication in the "Advances in Micro and Nano-Manufacturing" special issue of the American Society of Mechanical Engineer's Journal of Micro and Nano- Manufacturing published in December 2020:

Mamros, E. M., Ha, J., Korkolis, Y. P., and Kinsey, B. L., 2020, "Experimental Investigation and Plasticity Modeling of SS316L Microtubes under Varying Deformation Paths," JMNM, 8(4).

As the first and only student author, I performed nearly all of the work for this publication. I conducted all experiments in the laboratory, completed all data post-processing, coded and executed the plasticity model to generate data, created all figures, and wrote the initial draft of the manuscript text. My advisors, Dr. Ha and Dr. Kinsey along with our collaborator from the Ohio State University, Dr. Korkolis, contributed to the work by editing the manuscript and providing their expert perspectives on the research.

INTRODUCTION

Over the past two decades, with the growing trend of miniaturization, research focused on micromanufacturing has been building momentum to establish a link between macroscale manufacturing and nanomanufacturing and to create state-of-the-art microscale components. The latest findings include applications-driven processes such as micro-machining, micro-EDM, micro-electrochemical machining, and micro-powder injection molding [1]. In terms of microforming, several processes have been scaled-down and studied, such as single point incremental forming [2] and microtube bending [3]. As at the macroscale, microforming is a high production rate process with exceptional material utilization. In order to achieve high precision and product quality, a fundamental understanding of the processes in addition to accurate prediction models and simulations are needed.

With the decrease in length scale, several new challenges arrive for manufacturers. Assumptions neglecting certain effects in macroscale manufacturing are no longer valid. Previous studies have shown that material behavior varies significantly at smaller length scales [4-6]. In forming, grain size effects occur when a part feature contains ≤ 10 grains through the cross-section, which implies that empirical and analytical knowledge derived from conventional processes is not necessarily applicable at the microscale [7]. Grain size effects can influence the deformation behavior and resulting defects, e.g., springback [3], surface finish [8], and eccentricity of microtubes.

Other factors that may contribute to the difficulty of rescaling macroscale manufacturing processes to create smaller components include process-material interactions, morphology, mechanical compatibility, equipment resolution, and heat transfer [9]. In addition, stress

superposition [10,11], which is an emerging area in forming processes where applied stress components are intentionally superimposed during the deformation, has the ability to improve the final part properties, e.g., formability, strength, and geometry. Optimizing and controlling forming parameters according to the intended part application is desired, but methods to accomplish these goals remain to be determined even at the macroscale.

One example of stress superposition is microtubes subjected to multiple biaxial loading segments, i.e., combined axial tension and inflation [12-14]. In these studies, two types of paths were investigated for SS304L: proportional (where a single stress ratio of axial to hoop stress is held constant) and corner (where a pure axial or pure hoop loading segment is executed and then followed by a biaxial stress segment as loading is increased in the transverse direction). A model capable of predicting these deformation paths based on desired final stress and strain states, which will directly affect the material properties, is required to eliminate trial and error approaches.

In this thesis, microtube experimental results and plasticity modeling are presented for single and multi-loading segment deformation paths reaching the same final strain state, but different final stress states. This variability can influence the properties, e.g., strength, heterogeneity in microstructure, and phase fractions, of materials susceptible to plasticity-induced phase transformations. This includes the material used in this study, SS316L, which transforms austenite into martensite with deformation. Plasticity modeling is also presented and shown to reasonably predict the experimental results provided that the anisotropy of the material is captured in the analyses. Therefore, this model can be utilized to optimize the stress paths during forming, which is important for enhancing martensitic transformation, while achieving the same final strain state.

CHAPTER 1

EXPERIMENTS

1.1 Experimental Setup and Methods

The experimental methods for this research include the machine used, the unique measurement systems incorporated into the process, and the material investigated. Biocompatible materials are of interest due to the potential applications of this work in the healthcare industry. Previous studies were completed using SS304L microtubes [12-14], but this current investigation utilizes a different low-carbon stainless steel, SS316L, due to enhanced biocompatibility. Similar tube failure experiments were also performed on aluminum 6260 and 6061 [15-18], although this is not considered to be a biocompatible material, and the research was conducted on the macroscale.

Figure 1.1: Microtube inflation/tension testing machine, a) entire system and b) close-up of tensile stage with microtube assembly inserted

1.1.1 Microtube Inflation/Tension Machine

The experiments were conducted on the custom microtube inflation/tension machine shown in Fig. 1.1 at UNH [12]. This testing machine consists of two major components, a microscale ball screw-driven, tensile stage from Psylotech and a Teledyne ISCO hydraulic pump, which are coupled via a leader-follower relationship in LabVIEW. That is, the axial force follows the internal pressure based on the desired ratio of the loading. The load cell capacity is 2000 N with a resolution of 1 mN, and the maximum displacement is 50 mm with a linear variable displacement transducer (LVDT) resolution of 25 nm. The pump can reach a maximum flow rate of 25 ml/min and a maximum pressure of 1380 bar (20 ksi) with resolutions of 3% of set point and 138 kPa (20 psi), respectively.

The 316L microtube specimens used in the experiments were cut from stock tubing, using a Dremel tool, into lengths varying from 45 to 60 mm (1.77 to 2.36 in), dependent upon the loading path of the given test. The shorter lengths are necessary to prevent buckling during inflation, where an axial compressive force is applied to maintain a pure hoop loading condition. To prevent the microtube from crimping while tightening the grips, a hollow, metallic insert 12 mm (0.47 in) in length was placed in each end creating an 11 to 30 mm (0.43 to 1.18 in) gage section. Next, the microtube was inserted into customized glands machined on a lathe out of grade 8 high strength steel to have a through hole and tapered opening at the bottom of the threaded end (see Fig. 1.2). Then, 4 conical sleeves (HiP Company) were placed (2 on each end of the specimen) opposite to one another as shown in Fig. 1.2. Lastly, this assembly was inserted into custom, 3D printed metallic grips, shown physically in Fig.1.1b and schematically in Fig. 1.2, and tightened. As the custom gland was tightened, the conical sleeves deformed around the microtube securing the specimen and preventing slippage and leakage. Two sleeves per end were found necessary, to be able to apply the axial tensile forces needed in these experiments without the tube slipping out of the assembly of Fig. 1.2. The stainless steel fluid inlet line was then attached to the bottom grip, and after purging any air from the system, a plug was inserted into the top

grip creating a closed system. This specimen preparation procedure follows the methodology used by Ripley and Korkolis [12].

Figure 1.2: Schematic of microtube specimen assembly inside of grip (modified from [13])

The microtube inflation/tension machine operates from a customized LabVIEW program. As mentioned previously, this program maintains a user-specified engineering stress-ratio (i.e., axial:hoop or $\sigma_x : \sigma_\theta$) via a leader-follower relationship. The tubes were inflated under volume control. The pressure sensor, acting as the leader in the system, records a measurement and sends this data to the program. The follower component is the tensile stage, which then receives a signal from the program to apply a force in reaction to the pressure reading, thus maintaining the specified stress ratio. This loading scheme was selected to allow the tubes to continue to deform after the anticipated maximum in the internal pressure (and follower axial force) is attained. In this way, the tubes were taken to failure. The program also features an option to hold the axial stress at a constant value while the pump is increasing the pressure. This allowed for the execution of corner paths, i.e., axial to hoop or hoop to axial (where an initial pure hoop or axial loading segment is followed by a loading segment in the transverse direction).

1.1.2 Digital Image Correlation and Thermal Imaging

Three dimensional Digital Image Correlation (DIC) was utilized to measure the strain field in-situ. Two FLIR 8.9 megapixel cameras were incorporated into the experimental setup (shown in Fig. 1.1a) with 50 mm Schneider Xenoplan compact lenses. Specimens were spray-painted to have a white background with a black speckle pattern. As deformation-induced heating is known to occur when deforming stainless steels [19-20], a FLIR SC-645 infrared camera (temperature range of -20°C to 650°C with a resolution of 0.05°C) was also incorporated into the experimental setup (shown in Fig. 1.1a) to obtain in-situ temperature measurements. A surface emissivity value of 0.98 was used for the thermal imaging based on calibrations with a thermocouple.

All three cameras were coupled using VIC Snap (Correlated Solutions, Inc.) to capture synchronously at 2 Hz. Post-processing of the DIC images was completed in VIC-3D (Correlated Solutions, Inc). The area of interest included the entire gauge length with two virtual extensometers, which calculate the engineering strain based on the displacement of the virtual extensometer end locations, placed in the center along the axial and transverse, i.e., hoop, directions, to extract strain measurements. The DIC parameters include a subset size of 27, a step size of 9, and a filter size of 5 pixels to analyze the strain data. See Fig. 1.3 for example strain and thermal fields from the DIC software a few frames prior to failure, showing the localization of strain in a $\sigma_x : \sigma_{\theta} = 5:4$ proportional loading test. Note that deformation induced heating is not significant in these experiments, i.e., only $\Delta T \approx +10^{\circ}\text{C}$ compared to the initial temperature, due in part to heat transfer through the inflation fluid in the experiments. Therefore, further temperature results are not presented here. The strain rate for all experiments was approximately $10^{-3} - 10^{-5} \text{ s}^{-1}$.

1.1.3 Material

Seamless, fully annealed SS316L microtubes with an outer diameter of 2.38 mm (0.094 in) and a wall thickness of 150 μm (0.006 in) were purchased from MicroGroup, part of TE

Figure 1.3: Experimental 3D DIC true a) axial and b) hoop strains and c) thermal image from a $\sigma_x : \sigma_\theta = 5:4$ proportional loading test

Connectivity [21]. Table 1.1 shows the chemical properties reported by the manufacturer. The microstructure of an annealed sample of the material is shown in Fig. 1.4. The optical microscope image was taken from the hoop-thickness ($\theta - t$) plane after etching the sample in a 10 mL nitric acid (HNO_3), 20 mL hydrochloric acid (HCl), and 30 mL water solution. There are approximately 6-8 grains through the thickness in this material, which will directly affect the mechanical behavior due to size effects (i.e., $< \sim 10$ grains through the thickness during microforming) [3,7].

C	Mn	P	S	Si	N	Ni	Cr	Mo	Fe
0.020	1.640	0.031	0.010	0.380	0.043	13.100	17.350	2.640	Bal.

Table 1.1: Chemical composition of SS316L in mass percent % [21]

For material characterization purposes, pure axial tension and pure hoop experiments were conducted. The resulting true stress-strain curves are shown in Fig. 1.5. Explicitly, the “Pure Axial” tension curve is true axial stress versus true plastic axial strain, and the “Pure Hoop” curve is true hoop stress versus true plastic hoop strain, respectively. As is evident, the material is softer in the pure hoop direction compared to axial tension, and

Figure 1.4: Optical microscope image of as-received SS316L microtube grains in the hoop-thickness ($\theta - t$) plane

the relative flow stress ratio changes from 0.86 to 0.80 as the plastic work increases from 0.5 to 150 MJ/m³. This indicates that plastic anisotropy in the flow stress is observable in this material, and furthermore, it evolves as the plastic deformation increases. In addition to the anisotropy in flow stress, the strain anisotropy was characterized by the strain ratio for pure axial tension and pure hoop inflation experiments. The strain ratio was assessed in a similar way to Lankford coefficients, i.e., r -values, for uniaxial tension of sheet materials, which are plastic strain ratios in the transverse to the loading and thickness directions. For the microtube specimens, the ratios for pure axial (r_x) and pure hoop (r_θ) experiments were calculated by the hoop-to-thickness and axial-to-thickness plastic strains, respectively. The values are $r_x = 0.908$ and $r_\theta = 0.820$ and were used to calibrate material parameters in the plasticity modeling.

1.1.4 Inflation/Tension Experiments

Two types of loading paths are considered in this paper, proportional (where a single loading path with a given axial:hoop stress ratio is followed) and corner (where an initial pure axial

Figure 1.5: True stress-strain curves for pure hoop and pure axial loading

loading segment is followed by a secondary loading segment in the hoop direction). Note that the corner engineering stress value is prescribed during these tests, but for consistency purposes with the model, all results will be presented in terms of true stresses. These experiments demonstrate that significantly different final stress states are required to achieve the same final strain state when the deformation path is altered. As mentioned, this difference in stress state may affect the microstructure, i.e., transformation kinetics of martensite, in this material and alter the final properties. An explanation of concerns related to microforming grain size effects for the alternative loading case, i.e., an initial pure hoop loading segment followed by a secondary axial loading segment, and thus why experimental data is not presented for this loading case, is provided in the Discussion section.

1.2 Experimental Results

To demonstrate that varying final stress states are required to achieve the same final strain state and to validate the plasticity model, experimental stress and strain data were collected for various prescribed single loading segment paths with constant stress ratios, i.e., proportional paths, as well as corner paths, e.g., axial to hoop loading, until failure. Note that for corner paths, the second loading segment has a varying stress ratio as the initial loading

segment's stress value at the path transition is held constant while loading in the other direction is increased. Experiments were conducted for the following proportional paths $\sigma_x : \sigma_\theta = 1:1$, $5:4$, and $1:0$ (pure axial). Additionally, three axial to hoop paths and one hoop to axial path (denoted by the true axial or hoop stress value, respectively, at the transition between loading segments in figures) were investigated. The corresponding true stress paths and induced true strain paths are shown in Fig. 1.6a and 1.6b, respectively. The loading paths shown are truncated to the desired final strain state value in most of the figures. The dominant stress dictates the failure mode of the microtube, e.g., if axial stress or hoop stress is greater, the microtube will fail circumferentially or axially respectively [14]. See Fig. A.1 for engineering stress and strain paths including additional experiments.

Figure 1.6: Experimental true a) stress and b) strain for SS316L deformation paths

As is evident in Fig. 1.6, the final stress state is significantly different, despite achieving the same final strain state, for the various deformation paths. This difference increases as the plastic deformation increases. That is, the final stress states for the $(\epsilon_x, \epsilon_\theta) = (0.03, 0.03)$ final strain state are relatively close (red square markers) compared to the $(\epsilon_x, \epsilon_\theta) = (0.14, -0.01)$ final strain state (light blue circle markers), where the specimens have undergone significantly more deformation. Achieving larger strain values will increase the austenite to martensite phase transformations of interest for this material, which will also be affected by the deformation path followed. The phase transformation results will be studied in future research. Note that the pronounced non-vertical change in slope towards the end of the $\sigma_x = 785$ MPa transition true stress case (i.e., light blue path in Fig. 1.6b) is due to strain localization.

1.3 Discussion

The corner loading paths investigated were all axial to hoop except for one hoop to axial path that was not loaded beyond the elastic limit of the material, which is $\sigma_\theta^y = 370$ MPa for a pure hoop loading. Additional hoop to axial paths are not presented in this research due to significant specimen to specimen variability and grain size effects present during microforming when $< \sim 10$ grains exist through the deformation direction. With only 6-8 grains through the thickness, variability occurred during initial pure hoop loading segments depending on the orientation of grains through the thickness in a given area of the microtube. Figure 1.7 shows how the strain path varied depending on the axial location investigated during a single test to a corner engineering hoop stress of 550 MPa. Note that all transverse virtual extensometers shown were plotted against a single axial virtual extensometer to allow for comparison based on the axial placement. The hoop strain values from the different transverse virtual extensometers exhibited deviations of approximately $\pm 13 - 20\%$ from the average hoop strain values for the portion of the strain path corresponding to the axial loading segment. Such variability was exacerbated when data from multiple specimens was considered. Often, physical differences due to grain size effect are visibly observed during

microforming, e.g., during springback [7]. Due to the small deformation induced, this is not the case during these microtube experiments. However, these grain size effects exist and require DIC analyses for observation. See Fig. A.2 for true axial and hoop strain contour images from DIC analyses of the experiment in Fig. 1.7.

Figure 1.7: Variation in strain paths depending on axial location considered due to grain size effects for a hoop to axial corner path test

In contrast to an experiment with an initial pure hoop path as in Fig. 1.7, with some application of axial tensile loading, whether following a proportional path or an axial to hoop corner path, the variability in strain path curves is not present. When the force is applied in the axial direction, there are several grains through the axial length preventing grain size effects during this axial loading. Even during an axial to hoop corner path, simply applying some axial force improves the repeatability of the tests (see Fig. 1.8 despite loading in the hoop direction during the second segment. Again, this is a clear indication that grain size effects exist during these microtube forming experiments. Note in Figure 1.8 that the true strain paths are shown to failure, but the red square indicates the approximate truncation location for figures where the same final strain state is presented.

Figure 1.8: Repeatability of axial to hoop experiment with $\sigma_x = 505 \text{ MPa}$ transition true stress

CHAPTER 2

PLASTICITY MODELING

Plasticity models coded in MATLAB for yield criterion and strain hardening were used to predict true strain paths based on user-input stress paths with single or multiple loading segments, i.e., proportional or corner paths. In the current study, the plasticity modeling is presented only as a validation between experiments and model predictions. Later, the model will be used to optimize deformation paths producing the most beneficial microstructure for strength purposes, e.g., martensite transformation, while keeping the same final strain state related to the geometry of the product, e.g., shape and thickness. See Appendix A for the MATLAB code used to perform these calculations.

2.1 Anisotropic Yield Criterion

As shown in the experiment (see Fig. 1.5), SS316L clearly presents anisotropic behavior in both flow stress and strain during plastic deformation, which creates the necessity of an anisotropic yield criterion for the plasticity modeling. For this study, one of the simple anisotropic yield criteria, i.e., Hill 48 [22], was adopted to depict the material anisotropy due to the small number of experiments required to calibrate the material parameters of the yield criterion. For a plane stress state, Hill 48 can be simplified as

$$\phi = F(\sigma_{yy})^2 + G(\sigma_{xx})^2 + H(\sigma_{xx} - \sigma_{yy})^2 + 2N(\sigma_{xy})^2 = \bar{\sigma}^2 \quad (2.1)$$

where F , G , and H are material parameters determined by plastic anisotropy. If $F = G = H = \frac{N}{3} = \frac{1}{2}$, the equation reduces to von Mises isotropic yield criterion for plane stress. The

subscripts x, y, and z represent three material orientations in general, thus, for the microtube specimens used in the study, they are assumed to be axial (x), hoop (θ), and thickness (t) directions. For the special case of zero shear stress, i.e., $\sigma_{xy} = 0$, Eq. (2.1) reduces to:

$$\phi = (G + H)\sigma_x^2 + (F + H)\sigma_\theta^2 - 2H\sigma_x\sigma_\theta = \bar{\sigma}^2 \quad (2.2)$$

The material parameters, i.e., F, G, and H, can be identified based on either stress or strain ratios, e.g., normalized stresses or r-values from uniaxial tension of sheets. In this study, only the approach based on strain ratios was considered, and the parameters were obtained as:

$$F = \frac{r_x}{r_\theta(1 + r_x)} \quad (2.3)$$

$$G = \frac{1}{1 + r_x} \quad (2.4)$$

$$H = \frac{r_x}{1 + r_x} \quad (2.5)$$

Here, the two strain ratios, r_x and r_θ , were calculated from the pure axial tension and pure hoop experiments, respectively. The identified parameters are summarized in Table 2. The yield locus predicted by Hill 48 in the $\sigma_x - \sigma_\theta$ plane is presented in Fig. 2.1, along with the von Mises isotropic yield locus for comparison. Experiments of normalized flow stresses from axial tension, pure hoop, and proportional biaxial tension experiments are also plotted together with both yield loci. Note that the flow stresses are overpredicted for pure hoop and proportional biaxial tension cases. This indicates potential concerns with being able to accurately predict stress and strain paths well by both yield criteria at those stress states.

F	G	H
0.572	0.524	0.476

Table 2.1: Anisotropic material parameters for Hill 48 yield criterion

Figure 2.1: Yield loci of Hill 48 anisotropic yield criterion calibrated based on strain ratios and von Mises isotropic yield criterion

2.2 Strain Hardening Law

With the Hill 48 anisotropic yield criterion, the strain hardening behavior assuming isotropy is modeled using Hockett-Sherby hardening law:

$$\bar{\sigma} = \sigma_0 - (\sigma_0 - A) \cdot \exp(-N\bar{\epsilon})^p \quad (2.6)$$

where σ_0 is yield stress, $\bar{\sigma}$ is equivalent stress, and $\bar{\epsilon}$ is equivalent strain. The parameters of the equation, i.e., A , N , and p , are calibrated using the uniaxial tension stress-strain curve in the uniform deformation region, i.e., up to $\epsilon = 0.25$. The calibrated result is shown in Fig. 1.8, and the parameters are summarized in Table 2.2.

σ_0 [MPa]	A [MPa]	N	p
463.00	1828.10	1.73	0.85

Table 2.2: Strain hardening parameters of Hockett-Sherby law

Figure 2.2: Hockett-Sherby law fitted to SS16L uniaxial experimental data

2.3 Model Implementation

The model is capable of predicting deformation paths for the three loading types described in this work: proportional, axial to hoop corner, and hoop to axial corner paths. Figure 2.3 presents a flow chart to calculate the true strain paths from the user-defined true stress paths. The code was programmed in MATLAB incorporating an isotropic strain hardening law, i.e., Hockett-Sherby, and an anisotropic yield criterion, i.e., Hill 48, for the material plasticity. Inputs for the calculations include material parameters for the plasticity models, transition stress and stress increments for the axial and the hoop directions to define the stress path, and the final strain state for the terminating condition. Stress and strain states are initialized to zero prior to entering the first loop to determine the plastic strain increment.

For the corner paths, i.e., axial to hoop and hoop to axial, a for loop is implemented for the first, user-defined stress path, i.e., pure axial or pure hoop. Within the for loop, the effective stress and strain are calculated based on the yield criterion and hardening law

Figure 2.3: Flow diagram of model to predict strain paths from stress paths

following the stress path, and the plastic strain increment is calculated by the associated flow rule. The strain state is updated accordingly, and this iterative process continues until the specified transition stress state is reached.

For proportional paths, the for loop is bypassed, and the code directly enters the while loop. For the corner paths, the while loop is used to continue the calculations, from the for loop, for the second path segment. The calculation is terminated once a tolerance, defined by the minimum difference of the strain state at the end of the iteration against the desired final strain state, is satisfied. A break statement is imposed should the desired final strain state not be achieved with the given stress path.

2.4 Model Validation

Figure 2.4 shows a comparison of the predicted corner strain path for a single test, i.e., axial to hoop with $\sigma_0 = 635$ MPa transition true stress, using von Mises versus Hill 48 yield criterion in the model with the experimental result. The Hill 48 prediction more accurately captures the corner strain value and the curvature of both the axial and hoop loading segments due

to the consideration of anisotropy with the strain ratios. Therefore, only Hill 48 is presented for all subsequent predictions.

Figure 2.4: Comparison of von Mises and Hill 48 strain path predictions with experimental data

To validate the model, the same corner and proportional paths as the experiments, i.e., the same axial to hoop transition stresses for corner paths and the same stress ratios for the proportional paths, were input into the model. The three axial to hoop transition true stresses were $(\sigma_x : \sigma_\theta) = (505, 0)$, $(635, 0)$, and $(785, 0)$ MPa which correspond to engineering stress values, i.e., machine inputs, of approximately $(500, 0)$, $(600, 0)$, and $(700, 0)$ MPa, respectively. The true stress and true strain path predictions are shown in Figs. 2.5a and 2.5b, respectively, with the experimental results for comparison. The path pairs targeting the same final strain state are color-coordinated, i.e., red, purple, and blue, for clarity, and markers are used to designate the final stress or strain state for each path pair.

Figure 2.5: Comparison of predicted, i.e., model, and experimental true a) stress and b) strain for SS316L deformation paths

2.5 Discussion

As is evident in the experimental results, the model also predicts that multiple deformation paths achieving the same final strain state will result in differing final stress states. For the three axial to hoop corner paths shown in Fig. 2.5, the model is able to accurately predict the corner strain state with the closest to the experimental results being the $\sigma_x = 505$ MPa transition true stress case (red in Fig. 2.5). For the axial to hoop corner paths, the model for the $\sigma_x = 785$ MPa transition true stress case (light blue in Fig. 2.5) overpredicts the final true hoop stress up to approximately 220 MPa. This overprediction corresponds to the overprediction in flow stress by Hill 48 during biaxial loading (see Fig. 2.1). It also highly affects the strain paths as the tangent of the yield locus is associated with the incremental

strain ratio for continued plastic deformation. As a result, some of the strain paths near the biaxial stress state show erroneous predictions. For example, the model prediction of the strain path for the 5:4 proportional loading case shows a comparatively higher slope than the experiment. This can be attributed to the higher slope along the normal direction of the yield locus predicted by Hill 48, i.e., $\frac{\epsilon_\theta}{\epsilon_x}$, near the 5:4 proportional loading case as seen in Fig. 2.1. This would create significant sensitivity to the strain paths if the yield criterion is not accurately represented in the model. Finally, as shown in Fig. 2.2, Hockett-Sherby predicts the overall strain hardening behavior well but does not capture the slight curvature variations visible (see insert) in the experimental data near the axial strain values from the corner paths, i.e., 0.05 – 0.1. This would cause a slightly over or underpredicted flow stress depending on the range, which results in slight under or overestimated strain respectively in the plasticity model. Note that the stress paths for the hoop loading segments deviate from vertical near their ends for all axial to hoop corner paths due to the localization of the deformation, with this being most pronounced in the corner path case with the $\sigma_x = 785$ MPa transition true stress case. The engineering to true stress-strain conversion is only valid during uniform deformation, which causes concerns for the ends of these curves.

Overall, the model predictions show reasonable agreement with the experimental results, but limitations of the current model are considered to account for the error in stress and strain path predictions (see Fig. 2.5). Future work will include modifying the model to account for these variations and improving the stress and strain predictions with an advanced anisotropic yield criterion, e.g., Yld2000-2D [23].

CHAPTER 3

CONCLUSIONS

Experimental results and plasticity models for SS316L microtubes subjected to various combinations of deformation, i.e., proportional and corner paths, to achieve the same final strain state are presented. The plasticity model prediction shows better agreement with the experimental results for linear and axial to hoop corner paths when utilizing the Hill 48 yield criterion compared to von Mises, as material anisotropy is captured. When predicting the final true stress state for axial to hoop corner paths with large (> 500 MPa) axial stress values, the model overpredicts the final hoop stress, and the corresponding strain paths deviate from the experimental results. However, these results are deemed reasonable considering the complicated biaxial deformation of the microtube inflated under axial force and internal pressure. Hoop to axial corner paths show significant specimen to specimen variation and even discrepancies based on axial location for a given specimen due to grain size effects. These differing strain paths for a specific loading case demonstrate the need to account for such process size effect considerations during microforming.

CHAPTER 4

FUTURE WORK

Future work will include simulating the microtube inflation/tension proportional and corner paths in a finite element software that will be validated by experimental results. Additionally, this austenitic stainless steel exhibits martensitic phase transformations that alter the microstructure and are of interest for creating heterogeneous biomaterials. Thus, samples will be removed from the deformed microtubes and subjected to electron backscatter diffraction (EBSD) to characterize the resulting phase fractions. As mentioned in Chapter 2, the models will also be modified to improve the stress and strain predictions using an advanced anisotropic yield criterion.

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APPENDIX A

SUPPLEMENTAL FIGURES

Figure A.1: Engineering a) stress and b) strain paths from corner (solid) and proportional (dashed) loading path experiments

Figure A.2: Experimental 3D DIC true a) axial and b) hoop strains from a hoop to axial corner loading test just prior to failure

APPENDIX B

MATLAB CODE

This section contains the MATLAB code for the plasticity model described in chapter 3. A hoop to axial corner path input was implemented as an example.

```
%% PLASTICITY MODEL
```

```
clear all
```

```
clc
```

```
%INPUT PARAMETERS
```

```
%STRESS PATHS
```

```
%for axial to hoop: sig_Hc = 0, dsig_H1 = 0, and dsig_X2 = 0
```

```
%for hoop to axial: sig_Xc = 0, dsig_X1 = 0, and dsig_H2 = 0
```

```
%corner stress value in [MPa]
```

```
sig_Hc = 300;
```

```
sig_Xc = 0;
```

```
%stress increments in [MPa]
```

```
dsig_H1 = 10;
```

```
dsig_X1 = 0;
```

```
dsig_H2 = 0;
```

```
dsig_X2 = 10;
```

```
%power law
```

```
pl = 2; %enter 1 for Hollomon-Ludwik or 2 for Hockett-Sherby
```

```
%Hollomon-Ludwik
```

```
K = 1562; %[MPa]
```

```
n_HL = 0.34;
```

```
%Hockett-Sherby
```

```
sig_inf = 1828.10; %[MPa]
```

```
n_HS = 1.73;
```

```

p = 0.85;
sig_y = 463; %[MPa]

%select yield criterion
yc = 2; %enter 1 for von Mises or 2 for Hill 1948

%anisotropy
R0 = 0.908;
R90 = 0.820;
Ra = R0*(1 + R90)/(R90*(1 + R0));
Rb = 2*R0/(1+R0);

%STRAIN PATHS
%final strain state
eps_Xf = 0.044;
eps_Hf = 0.07;

%INITIAL
%start at zero stress and strain states
eps_H1 = zeros(1,10);
eps_X1 = zeros(1,10);
epsb_1 = zeros(1,10);
depsb_1 = zeros(1,10);
deps_X1 = zeros(1,10);
deps_H1 = zeros(1,10);
sigma_X1 = zeros(1,10);
sigma_H1 = zeros(1,10);
sigb_1 = zeros(1,10);

%% PATH 1: Stress to strain conversion methodology

%determine stress increment for first path
%and ending at corner stress
if sig_Xc > sig_Hc
    end_path = sig_Xc;
    inc = dsig_X1;
else
    end_path = sig_Hc;
    inc = dsig_H1;
end

i = 2; %index
for j = 1:inc:end_path

```

```

%update stress state
sigma_X1(i) = sigma_X1(i-1) + dsig_X1;
sigma_H1(i) = sigma_H1(i-1) + dsig_H1;
sigma_3 = 0; %thin-walled assumption

%von Mises
if yc == 1 %von Mises
    %calculate effective stress from yield function
    sigb_1(i) = sqrt((sigma_X1(i) - sigma_H1(i))^2...
        + (sigma_H1(i) - sigma_3)^2 + (sigma_3 - sigma_X1(i))^2)/2);
else
    %calculate effective stress from yield function
    sigb_1(i) = sqrt(sigma_X1(i)^2 + Ra*sigma_H1(i)^2...
        - Rb*sigma_X1(i)*sigma_H1(i));
end

%increment until reaching yield stress
if sigma_H1(i) < sig_y
    epsb_1(i) = epsb_1(i-1);
    depsb_1(i) = depsb_1(i-1);
    deps_X1(i) = 0.0;
    deps_H1(i) = 0.0;
else
    %calculate effective strain from hardening law
    if pl == 1 %Hollomon-Ludwik
        epsb_1(i) = (sigb_1(i) / K)^(1 / n_HL);
    else %Hockett-Sherby
        epsb_1(i) = (log((sig_inf - sig_y)/...
            (sig_inf - sigb_1(i)))/(n_HS))^(1/p);
    end

    %calculate change in effective strain
    depsb_1(i) = epsb_1(i) - epsb_1(i-1);

    if yc == 1
        %calculate plastic strain increment from associated flow rule
        deps_X1(i) = depsb_1(i)*(sigma_X1(i)...
            - (sigma_H1(i) + sigma_3)/2)/sigb_1(i);
        deps_H1(i) = depsb_1(i)*(sigma_H1(i)...
            - (sigma_3 + sigma_X1(i))/2)/sigb_1(i);
    else
        %calculate plastic strain increment from associated flow rule
        deps_X1(i) = depsb_1(i)*((2*Ra*sigma_X1(i)...
            - Rb*sigma_H1(i))/2)/sigb_1(i);
        deps_H1(i) = depsb_1(i)*((2*Ra*sigma_H1(i)...

```

```

        - Rb*sigma_X1(i))/2)/sigb_1(i);
    end
end

%update strain state
eps_X1(i) = eps_X1(i-1) + deps_X1(i);
eps_H1(i) = eps_H1(i-1) + deps_H1(i);

i = i + 1; %increase index for next iteration
end

%% PATH 2: Stress to strain conversion methodology

%start at corner stress and strain states
eps_H2 = repelem(eps_H1(end),10);
eps_X2 = repelem(eps_X1(end),10);
epsb_2 = repelem(epsb_1(end),10);
depsb_2 = repelem(depsb_1(end),10);
deps_X2 = repelem(deps_X1(end),10);
deps_H2 = repelem(deps_H1(end),10);
sigb_2 = repelem(sigb_1(end),10);
sigma_X2 = repelem(sigma_X1(end),10);
sigma_H2 = repelem(sigma_H1(end),10);

%determine stress increment for second path
if sig_Xc > sig_Hc
    inc2 = dsig_H2;
else
    inc2 = dsig_X2;
end

i = 2; %index
dist = 0.1; %set initial distance value to enter loop
while dist > 0.005

%update stress state
sigma_X2(i) = sigma_X2(i-1) + dsig_X2;
sigma_H2(i) = sigma_H2(i-1) + dsig_H2;
sigma_3 = 0;

%calculate effective stress from yield function
if yc == 1 %von Mises
    sigb_2(i) = sqrt(((sigma_X2(i) - sigma_H2(i))^2...
        + (sigma_H2(i) - sigma_3)^2 + (sigma_3 - sigma_X2(i))^2)/2);

```

```

else
    sigb_2(i) = sqrt(sigma_X2(i)^2 + Ra*sigma_H2(i)^2...
    - Rb*sigma_X2(i)*sigma_H2(i));
end

%calculate effective strain from hardening law
if pl == 1 %Hollomon-Ludwik
    epsb_2(i) = (sigb_2(i) / K)^(1 / n_HL);
else %Hockett-Sherby
    epsb_2(i) = (log((sig_inf - sig_y)...
    /(sig_inf - sigb_2(i)))/(n_HS))^(1/p);
end

%calculate change in effective strain
depsb_2(i) = epsb_2(i) - epsb_2(i-1);

if depsb_2(i)<0.0
    sigb_2(i)=sigb_2(i-1);
    epsb_2(i)=epsb_2(i-1);
    depsb_2(i)=depsb_2(i-1);
    deps_X2(i)=0.0;
    deps_H2(i)=0.0;
else
    %calculate plastic strain increment from associated flow rule
    if yc == 1
        deps_X2(i) = depsb_2(i)*(sigma_X2(i)...
        - (sigma_H2(i) + sigma_3)/2)/sigb_2(i);
        deps_H2(i) = depsb_2(i)*(sigma_H2(i)...
        - (sigma_3 + sigma_X2(i))/2)/sigb_2(i);
    else
        deps_X2(i) = depsb_2(i)*((2*Ra*sigma_X2(i)...
        - Rb*sigma_H2(i))/2)/sigb_2(i);
        deps_H2(i) = depsb_2(i)*((2*Ra*sigma_H2(i)...
        - Rb*sigma_X2(i))/2)/sigb_2(i);
    end
end

%update strain state
eps_X2(i) = eps_X2(i-1) + deps_X2(i);
eps_H2(i) = eps_H2(i-1) + deps_H2(i);

%check distance to desired strain state
dist = sqrt((eps_Xf - eps_X2(i))^2 + (eps_Hf - eps_H2(i))^2);

i = i + 1; %increase index for next iteration

```

```

%if strain path will not intersect desired final strain state
%then corner stress state needs adjusted
if dist > 0.2
    break
end

end

%% plots

%properties
set(gca, 'linewidth', 1.5) %axes line thickness
set(findall(gcf, '-property', 'FontSize'), 'FontSize', 22) %font size

% PATHS 1&2
    plot(sigma_X1, sigma_H1, sigma_X2, sigma_H2);
    %plot(sigma_X1, sigma_H1);
    xlabel( 'True_Axial_Stress_(MPa)' );
    ylabel( 'True_Hoop_Stress_(MPa)' );

    figure(2)
    plot(eps_X1, eps_H1, eps_X2, eps_H2);
    %plot(eps_X1, eps_H1);
    xlabel( 'True_Axial_Strain' );
    ylabel( 'True_Hoop_Strain' );
    xlim([-0.1 0.25])
    ylim([-0.1 0.25])
    %legend('von Mises', 'von Mises', 'Hill 48', 'Hill 48');

if yc == 1
    legend( 'von_Mises', 'von_Mises' );
else
    legend( 'Hill_48', 'Hill_48' );
end

```