Characterization of Anisotropic Plasticity in Material Systems Using Modified Indentation-Based Techniques

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ABSTRACT

Plastic anisotropy in rolled sheets has traditionally been analyzed by conducting tensile tests on strips cut at different angles from the rolling direction and measuring the contraction ratios during testing. This method is tedious, yet sufficient for sheet metals but the application to other material systems is limited. For example, if one were to seek the properties of a coating-substrate system, such an analysis would be impractical due to the combined effects of the coating and the substrate on which it lies. Indentation-based experiments are a great candidate for evaluation of such properties in various material systems for a few, main reasons. Indentation testing machines are readily available commercially and in material characterization laboratories world-wide and are currently being used for the classification of various material properties. Second, the systems in which indentation can be used are far from limited; indentation testing is notorious for its large-scale applicability. Finally, indentation provides a means of inducing localized plastic deformation, which can ultimately serve as a great analysis tool for anisotropy in these properties for a given material.

It has been shown that examination of material flow in the contact region could serve to uniquely characterize the degree of anisotropy. Therefore, the work presented in this dissertation pertains to the development, design, and testing of a set of virtual experiments using Finite Element Modeling with the specific aim to uniquely characterize the anisotropic plastic property of a material in all normal directions given minimal material data prior to testing. Current characterization methods and yield criteria are reviewed, and results suggesting anisotropic yielding in coatings are presented. Further, indentation stress-strain behavior of plastically anisotropic materials is examined, and finally, characterization methods involving indentation-based techniques are presented.
# TABLE OF CONTENTS

**CHAPTER 1. INTRODUCTION** .................................................................................................................. 1

1.1. OVERVIEW ........................................................................................................................................... 2

1.2. FACTORS INDUCING ANISOTROPIC PLASTIC BEHAVIOR IN MATERIALS .... 5

1.3. CURRENT CRITERIA FOR CHARACTERIZATION OF ANISOTROPIC PLASTIC BEHAVIOR IN MATERIALS ......................................................................................................................... 8

1.3.1. Anisotropic Yield Criteria for Plane Stress ................................................................................. 10

1.3.2. Anisotropic Yield Criteria for General Stress States ............................................................... 14

1.4. EXPERIMENTAL TECHNIQUES ........................................................................................................... 20

1.4.1. Uniaxial Testing of Sheet Metals ............................................................................................... 20

1.4.2. Biaxial Testing of Sheet Metals ................................................................................................ 21

1.4.3. Indentation-based Characterization Techniques ......................................................................... 24

1.5. PLASTIC ANISOTROPY IN COMMERCIAL FINITE ELEMENT CODES ........... 29

1.6. PURPOSE AND SCOPE ....................................................................................................................... 32

**CHAPTER 2. EFFECTS OF PLASTIC ANISOTROPY AND HARDENING ON INDENTATION MODULUS OF THIN FILMS** ........................................................................................................... 34

2.1. INTRODUCTION ..................................................................................................................................... 35

2.2. OVERVIEW OF OLIVER-PHARR METHOD OF MODULUS EXTRACTION ...... 37

2.3. COMPUTATIONAL METHODS .............................................................................................................. 38

2.4. RESULTS AND DISCUSSION ............................................................................................................... 41

2.4.1. Hard Film on Soft Substrate - The Effect of Plastic Anisotropy .............................................. 41

2.4.2. Soft Films - The Effect of Plastic Anisotropy ............................................................................. 43

2.5. CONCLUSIONS AND IMPLICATIONS ................................................................................................. 46
CHAPTER 3. STRESS-STRAIN BEHAVIOR OF MATERIALS EXHIBITING TRANSVERSE ISOTROPIC PLASTIC PROPERTIES ................................................................. 48

3.1. INTRODUCTION .............................................................................................................. 49

3.2. COMPUTATIONAL METHODS .......................................................................................... 49

3.2.1. Simulation of Spherical Indentation and Extraction of Properties ......................... 49

3.2.2. Simulation of Conical Indentation and Extraction of Properties ............................... 52

3.2.3. Examination of the Plastic Strain Fields ................................................................. 53

3.2.4. Simulation of Scratch Tests on Materials with Transverse Isotropic Plastic Properties ........................................................................................................ 54

3.2.5. Analytical Examination of Hill (1948) Yield Criterion and Its Implications ............ 55

3.3. RESULTS .......................................................................................................................... 56

3.3.1. Spherical Indentation of a Material with Transverse Isotropic Plastic Properties . 56

3.3.2. Conical Indentation of a Material with Transverse Isotropic Plastic Properties .... 59

3.3.3. Relation Between the Plastic Strain Field Aspect Ratio and the Ratio of In-plane to Out-of-plane Yield Stresses ......................................................... 61

3.3.4. Examination of Varying Degrees of Residual Deformation Resulting from Scratch Testing ........................................................................................................ 62

3.3.5. Comparison of Analytical Solution with Simulation Results ................................. 64

3.4. DISCUSSION AND IMPLICATIONS .............................................................................. 65

3.5. CONCLUSIONS .............................................................................................................. 70

CHAPTER 4. MINIMAL-CONSTRAINT INDENTATION TESTING OF PLASTICALLY ANISOTROPIC MATERIALS .............................................................. 71

4.1. INTRODUCTION ............................................................................................................ 72

4.2. COMPUTATIONAL METHODS ....................................................................................... 74

4.2.1. Indentation Near a Single Free Surface ..................................................................... 74

4.2.2. Indentation Near a Two Free Surfaces (Corner Indentation) .................................... 77
4.3. OBSERVATIONS DURING INDENTATION NEAR A FREE EDGE ..................... 78
   4.3.1. Observations via Spherical Indentation ........................................ 78
   4.3.2. Observations via Conical Indentation ......................................... 82
4.4. OBSERVATIONS DURING INDENTATION NEAR A CORNER ..................... 87
4.5. CONCLUSION ..................................................................................... 94

CHAPTER 5. CHARACTERIZATION OF TRANSVERSE ISOTROPIC PLASTIC
BEHAVIOR OF MATERIALS USING SINGLE SPHERICAL INDENTATION .......... 95
   5.1. INTRODUCTION AND OVERVIEW .................................................... 96
   5.2. DESCRIPTION OF EXPERIMENTAL PROCEDURE ................................ 98
   5.3. COMPUTATIONAL PROCEDURE ....................................................... 100
      5.3.1. Mesh, Geometry, and Loading Conditions .................................... 100
      5.3.2. Material Input ........................................................................... 102
   5.4. RESULTS .......................................................................................... 103
   5.5. DISCUSSION AND IMPLICATIONS ................................................... 108
   5.6. CONCLUSIONS ................................................................................. 112

CHAPTER 6. CHARACTERIZATION OF IN-PLANE PLASTIC ANISOTROPY ...... 114
   6.1. OVERVIEW OF SCRATCH TEST METHODOLOGY ............................... 115
   6.2. METHODS ....................................................................................... 117
      6.2.1. Finite Element Model of Linear Scratch Test ................................. 117
      6.2.2. Finite Element Model of Circular Scratch Test .............................. 119
   6.3. RESULTS .......................................................................................... 121
      6.3.1. Linear Scratch Test on Materials with In-Plane Anisotropic Plasticity .... 121
      6.3.2. Circular Scratch Test on Materials with In-Plane Anisotropic Plasticity .... 124
      6.3.3. Circular Scratch Test on Materials with Combined In-Plane and Out-of-Plane
            Anisotropic Plasticity .................................................................... 130
<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.4. SUMMARY/CONCLUSIONS</td>
<td>134</td>
</tr>
<tr>
<td>CHAPTER 7. CONCLUSIONS &amp; FUTURE WORK</td>
<td>137</td>
</tr>
<tr>
<td>REFERENCES</td>
<td>140</td>
</tr>
<tr>
<td>PUBLICATIONS</td>
<td>147</td>
</tr>
</tbody>
</table>
# LIST OF TABLES

1-1 Summary of the experimental results obtained by Yonezu et al. [70]. Results from five indentation tests and the corresponding value of $m$ are shown with the associated error from the accepted value of 1.25 as determined through compression testing ................. 28

4-1 Assessment of the accuracy/precision of the proposed equation to relate the drop in loading curvature ($C$) and strain-hardening slope ($H$) to the anisotropic yield ratio $R_{22}/R_{11}$. The normalized $C$ value was obtained from the computational models, and the Input $R_{22}/R_{11}$ and Input $H$ were taken from the inputs used in the models ................................. 93

5-1 Material input properties used in the FEM models ................................................................. 103

6-1 Tabulated results of the reaction force tangent to the scratch path resulting from the FEM simulation of a circular scratch test. Loads are tabulated for scratch tip positions of 180° and 90° for different input yield stress ratios. Error percentage between input properties and calculated properties are also tabulated ................................................................. 127

6-2 Tabulated results of the reaction force tangent to the scratch path resulting from the FEM simulation of a circular scratch test. Loads are tabulated for scratch tip positions of 180° and 90° for different input yield stress ratios incorporating both in-plane and out-of-plane anisotropy. Error percentage between input properties and calculated properties are also tabulated ........................................................................................................................................................................ 134
# LIST OF FIGURES

1-1  A) A sheet that has been torn from deep drawing as a result of low $\overline{R}$-value and B) a sheet that has formed ears during drawing as a result of a high degree of in-plane anisotropy ................................................................. 4

1-2  Extracted from Ref. [31]. Variation of the in-plane ($\Delta r$) and normal anisotropy ($<r>$) of AA1050 and AA6016 alloys with number of ARB rolling cycles ................................. 7

1-3  Schematic representation of the quadratic (dashed-line), and non-quadratic (solid-line) yield surfaces in principal stress space ................................................................. 10

1-4  Barlat 1991 yield loci in plane stress for different values of shear stress to uniaxial yield stress ratios. Axes represent the stresses in line with the directions of anisotropy. Note that with decreasing magnitude of shear stress, the surface resembles that which is typical of polycrystalline materials .................................................. 16

1-5  Influence of $r_0$ (left) and $r_{90}$ (right) on the yield locus defined by the Hill 1948 yield criterion in principal stress space ................................................................. 18

1-6  Tensile specimen geometry used to determine the Lankford Coefficient from rolled sheet metal samples. In this case, $\alpha$ denotes the orientation of the bar with respect to the sheet rolling direction [54] ................................................................. 20

1-7  Schematic representation of the biaxial testing machine developed by Makinde et al [59] .................................................................................................................. 22

1-8  A) Schematic representation of the experimental setup designed by Kuwabara et al. [60] B) Stress-strain relation for low-carbon steel obtained experimentally with the biaxial tensile test fixture and comparison with the behavior as predicted by the Hill quadratic yield criterion and the Hosford yield criterion [62] ........................................... 23

1-9  Results extracted from ref. [67]. Contour maps of surface height near the impression region of the material for isotropic material (a) $m = 1.0$, and anisotropic materials (b) $m = 1.5$, and (c) $m = 2.0$ ............................................................................................................ 26

1-10 Extracted from Ref. [67]. Coefficients (a) $C_1$ and (b) $C_2$ with respect to $n$ for the above relation ............................................................................................................ 27

1-11 Schematic representation of the orientation of the $R$-values implemented throughout this work. The 2-direction corresponds to the indentation direction in all cases ...... 31

2-1  A) Schematic representation of single-splat indentation using a Berkovich indenter tip. B) Shows the axisymmetric mesh implemented for the computational model. Axisymmetric BCs were applied to the left face, and roller BCs to the bottom face. The film thickness was defined as $t$ and the substrate thickness was $50t$ ............... 40
Modulus decrease (input $E = 200$ GPa) for hard films on soft substrates with varying degrees of plastic anisotropic behavior. Shows that increasing the material hardness and inputting anisotropic plastic behavior forces the resolved modulus to decrease with indentation depth similar to the experimentally observed results.

$P-h$ response of the above mentioned case of a hard film on a soft substrate, as well as the $P-h$ response obtained experimentally.

$P-h$ response of the above mentioned case for a soft film on a hard substrate. Note that the shape of the curve shows no inflection during loading.

$E$ vs. $h_{max}/t$ for the above case with both isotropic and anisotropic plastic properties as inputs. The x-axis is normalized with respect to film thickness.

Contours of plastic strain for the isotropic (left) and anisotropic (right) cases compared above for maximum indentation depth of $h/t = 0.50$. Note that in both cases, plastic strain in the substrate is significantly larger than in the film. This effect is larger when anisotropy is present, in which yielding is suppressed due to substrate constraint and plastic anisotropy.

Mesh design for axisymmetric spherical indentation model. Roller boundary conditions were applied to the bottom face, while an axisymmetric boundary condition was applied to the left face. The spherical indenter tip was given a constant radius, $R$.

Schematic showing the methodology used to calculate the aspect ratio of the plastic strain field. For this study, aspect ratio is defined as the ratio between the vertical distance to the bottom of the plastic strain field to the horizontal distance to the right edge of the plastic strain field.

Shows the mesh design and coordinate system for the 3D FEM model of a mechanical scratch test.

Shows computational results of the dependence of normalized average pressure beneath a spherical indenter tip at four different values of Tabor strain ($\varepsilon = 0.2a/R$).

Load-Depth curves of different inputs of transverse isotropic plasticity beneath a spherical indenter. Depth is normalized with respect to the radius of the indenter tip and load is normalized with respect to maximum load in the case of $R_{22}/R_{11}=0.50$.

Plots of the plastic strain fields beneath the spherical indenter at 6% Tabor strain for different ratios of $R_2/R_1$. A) $R_{22}/R_{11} = 0.50$ B) $R_{22}/R_{11} = 0.60$, C) $R_{22}/R_{11} = 0.70$, D) $R_{22}/R_{11} = 0.80$, E) $R_{22}/R_{11} = 1.00$, F) $R_{22}/R_{11} = 1.50$. 
3-7 The relation between normalized indentation load and $R_{22}/R_{11}$ at an indentation depth of 500 μm. Results are normalized with respect to the maximum indentation load ($R_{22}/R_{11} = 0.50$). A clear minimum occurs at $R_{22}/R_{11} \approx 0.70$ ............................................. 59

3-8 The load-depth curves for different $R_{22}/R_{11}$ ratios in response to conical indentation. The indentation load is normalized with respect to maximum load when $R_{22}/R_{11} = 0.50$ and the depth is normalized with respect to the maximum indentation depth ... 60

3-9 Plots of the plastic strain fields beneath the conical indenter for different ratios of $R_{22}/R_{11}$. A) $R_{22}/R_{11} = 0.50$ B) $R_{22}/R_{11} = 0.60$, C) $R_{22}/R_{11} = 0.70$, D) $R_{22}/R_{11} = 0.80$, E) $R_{22}/R_{11} = 1.00$, F) $R_{22}/R_{11} = 1.50$ ............................................. 61

3-10 Shows the dependence of the aspect ratio of the plastic strain field on the value of $R_{22}/R_{11}$ for both spherical and conical indenter tip geometries. It is evident that the aspect ratio is highly sensitive to the ratio $R_{22}/R_{11}$ ................................................................. 62

3-11 Residual depth profiles resulting from the FEM of a mechanical scratch test. A) Schematic of the residual deformation following scratch and the planar-directions of the plots. B) $R_{22}/R_{11} = 0.50$, C) $R_{22}/R_{11} = 0.60$, D) $R_{22}/R_{11} = 0.70$, E) $R_{22}/R_{11} = 1.00$, F) $R_{22}/R_{11} = 1.20$, G) $R_{22}/R_{11} = 1.50$, H) $R_{22}/R_{11} = 2.00$. Axis units in B-H are in microns and each case shows the height of pile-up post-scratch. The dashed plots in B-H show the geometry of the Rockwell C tip ................................................................. 63

3-12 Plot of the analytical solution for average pressure beneath a flat, rigid die compared with the pressure results from the simulations of conical and spherical indentation, and the inverse of mechanical scratch test pile-up height. All results are normalized with the isotropic case ........................................................................... 64

3-13 Shows the material flow direction beneath the conical tip for A) $R_{22}/R_{11} = 0.50$ B) $R_{22}/R_{11} = 0.60$, C) $R_{22}/R_{11} = 0.70$, D) $R_{22}/R_{11} = 0.80$, E) $R_{22}/R_{11} = 1.00$, F) $R_{22}/R_{11} = 1.50$ ................................................................. 68

3-14 Shows the material flow direction beneath the spherical tip for A) $R_{22}/R_{11} = 0.50$ B) $R_{22}/R_{11} = 0.60$, C) $R_{22}/R_{11} = 0.70$, D) $R_{22}/R_{11} = 0.80$, E) $R_{22}/R_{11} = 1.00$, F) $R_{22}/R_{11} = 1.50$ ................................................................. 69

4-1 Schematic representation of A) edge indentation vs. semi-infinite indentation and B) corner indentation vs. semi-infinite indentation ................................................................. 73

4-2 Shows the mesh and geometry used in the finite element simulation of indentation near a free edge. The plane of symmetry, free edge, and coordinate system are presented as defined ........................................................................... 74

4-3 The mesh used for the analysis of semi-infinite indentation with a cone. An axisymmetric model was adopted and appropriate BCs were applied. The coordinate system used is also highlighted ................................................................. 76
4-4 The geometry and mesh used in the 3D analysis of indentation near the corner. The faces shown are both free, and the coordinate system is also shown ........................................ 77

4-5 Load-depth relations extracted from spherical indentation near the edge of samples with varying degrees of anisotropic plastic properties. In all cases, increased compliance was observed at low loads (1N) in cases in which the sample was indented closer the edge ................................................................. 79

4-6 The dependence of indentation depth at a controlled load (1N) on the distance from the free-edge during spherical indentation. Results are normalized with respect to the depth in the semi-infinite case for each value of $R_{22}/R_{11}$ ......................................................... 81

4-7 Load-depth relations during conical indentation at distances of $d/2$, $d$, $2d$, and semi-infinite ................................................................................................................................. 82

4-8 Load-depth relations of materials, with various degrees of anisotropic plastic behavior in the out of plane direction, resulting from conical indentation at a distance $d = h_{\text{max}}$ away from a free edge ........................................................................................................ 83

4-9 Drop in loading curvature, $C$, during conical indentation at different factors of $d$ (maximum depth) away from a free edge. The loading curvature was constant in all semi-infinite cases and all results are normalized with respect to the average semi-infinite loading curvature .................................................................................. 84

4-10 Normalized loading curvature during conical indentation at $d$ away from the free edge for different in-plane to out-of-plane yield stress ratios. Results are normalized by the average loading curvature in the semi-infinite case corresponding to each material. 85

4-11 Normalized drop in loading curvature for $\sigma_Y/E$ ratios of 1/250, 1/500, and 1/1000 . . 86

4-12 Normalized loading curvature during conical indentation at $d$ away from the corner for different in-plane to out-of-plane yield stress ratios. Results are normalized by the average loading curvature in the semi-infinite case corresponding to each material. 88

4-13 Illustrates the dependence of the normalized loading curvature on the strain-hardening rate in the case of indentation near the corner. In all cases of out-of-plane anisotropy examined, the normalized curvature increased with the hardening rate ....................... 90

4-14 A) The relation between normalized loading curvature at depth $d$ and strain-hardening rate during indentation $d$ away from the sample corner. The results are fitted with logarithmic expressions as shown. B) Intercept of the curve fits as a function of anisotropy ratio $R_{22}/R_{11}$ ........................................................................................................ 91
Flow chart of the technique developed to determine out-of-plane to in-plane yield stress ratio from a combination of semi-infinite indentation and indentation to a maximum depth \(d\) at a distance \(d\) away from the sample corner ................................. 93

(a-c) Scanning electron microscope (SEM) cross-sections of the coatings indented in the experimental study. From left, APS, CS, HVOF [95, 96]; (d-f) binary images showing out-of-plane residual deformation after 100N indentation; white areas protrude more than 500 nm [95] ................................................................. 100

A) Schematic of the bonded interface technique  B) Finite Element mesh that was employed in this study ................................................................. 101

FEM results for material 1 showing out-of-plane contours, revealing a strong dependence on plastic anisotropy ................................................................. 104

FEM results for material 1 with dashed circles inscribed. Note that when \(R_{22} = 1\), the out-of-plane deformation contour fits the circle precisely ................................. 105

FEM results for material 2, with a significantly higher yield strength than material 1, showing a similar trend with respect to \(R_{22}\) ................................................................. 106

FEM results on a plastically isotropic material \((R_{22} = 1)\) showing that other parameters such as a) elastic anisotropy, b) residual stress, c) contour selection, and d) yield strength have a negligible effect on the residual deformation profile .......... 107

Results from experiments (top) compared with virtual experiments (bottom) showing good agreement in residual displacement fields, allowing estimation of transverse plastic anisotropy ................................................................. 108

FEM results for Material 3; a material with high yield strength and perfectly plastic post-yield behavior. Contours are presented with an applied imaging threshold of 250 nm ................................................................. 110

Schematic of the ball-on-disk scratch test. The ball is pressed into the disk to a constant load \(F\), and the disk is rotated at a constant angular velocity, \(w\) ............ 116

Shows the mesh design and coordinate system for the 3D FEM model of the linear mechanical scratch test ................................................................. 118

Shows the FEM mesh implemented in the circular scratch study, the coordinate system, and the tip geometry for the scratcher ................................................................. 120

Computationally calculated residual depth profiles resulting from scratch testing of materials with different values of \(R_{33}/R_{11}\). Units are in \(\mu m\) and the dashed line represents the scratch tip geometry ................................................................. 122

Computationally calculated residual depth profiles resulting from scratch testing of materials with different values of \(R_{11}/R_{33}\). Units are in \(\mu m\) and the dashed line represents the scratch tip geometry ................................................................. 123
FEM calculation of the angular dependence of the magnitude of in-plane reaction force tangent to the circular scratch path for materials with different input values of in-plane anisotropic plastic properties. A) $R_{33}/R_{11} = 0.66$, B) $R_{33}/R_{11} = 1.20$, C) $R_{33}/R_{11} = 1.50$, D) $R_{33}/R_{11} = 1.75$, E) $R_{33}/R_{11} = 2.00$ .............................................................. 125

The displacement contours normal to the surface at scratch tip rotation angles of 90° (left) and 180° (right) for $R_{33}/R_{11} = 0.66$ (top) and $R_{33}/R_{11} = 1.75$ (bottom). Pile-up behavior is reflective of the in-plane reaction force results shown in Fig. 6-6 ....... 128

FEM calculation of the angular dependence of the magnitude of in-plane reaction force tangent to the circular scratch path for materials with different input values of combined in-plane and out-of-plane anisotropic plastic properties. The black dotted plots represent comparative cases in which no out-of-plane anisotropy is present. A) $R_{33}/R_{11} = 1.00$, B) $R_{33}/R_{11} = 0.66$, C) $R_{33}/R_{11} = 1.20$, D) $R_{33}/R_{11} = 1.50$, E) $R_{33}/R_{11} = 2.00$ ......................................................................................... 131

The displacement contours normal to the surface at scratch tip rotation angles of 60° (left), 90° (center), and 180° (right) for the case in which $R_{11} = 1.00$, $R_{22} = 0.70$, $R_{33} = 2.00$. Pile-up behavior is reflective of the in-plane reaction force results shown in Fig. 6-8E ........................................................................................................... 132

Schematic cartoon of an experimental setup for a depth-controlled circular scratch testing system. The scratch tip is lowered and held at a constant depth ($h$) and the sample beneath is rotated through one cycle at a constant angular velocity, $w$ ....... 136
1.1. OVERVIEW

Indentation tests have been used extensively to measure mechanical properties of virtually every known synthetic material, in different forms [1]. Due to the minimal necessary sample preparation involved, micro- and nano-indentation methods have been particularly useful in probing structures for which uniaxial tension, compression, or simple shear are experimentally unfeasible. Such structures include thin films [2], coatings [3], multi-layers [4, 5] or lithographic shapes [6], as well as nanotubes [7, 8], micro-volumes of polymer [9], bio-composite constituents [10], and intact organs [11]. For a number of studies, material architecture, arrangement, or constitutive response is complex enough such that measurement of indentation-specific properties, such as hardness or indentation modulus is sufficient. Nevertheless, there is a large body of literature dedicated to the limits of indentation as a substitute to extract continuum-level behavior, for example Young’s Modulus [12], yield strength and strain hardening rate [13], and residual stress [14] to provide a substitute for uniaxial tests. Other, less heavily studied scenarios include, for example, the extraction of coating properties from a coating-substrate system [15], determination of in-plane residual stresses [16], fracture toughness [17], and anisotropy [18].

Compositionally-homogeneous materials exhibit mechanical anisotropy primarily due to texture, or crystallographic orientation [19]. It has long-been established that materials have orientation-dependent elastic modulus, and this manifests itself in particular for single crystal systems. Accordingly, the effect of elastic anisotropy upon indentation results has been well-studied, and it is accepted that this effect is dulled slightly due to the multi-axial stress fields beneath an indenter [20]. Such analyses are particularly well-suited to electronic single crystal studies, and coatings with high texture. Far less studied is the effect of plastic anisotropy on
indentation results; such anisotropy exists in rolled metal sheets [21], electrodeposited films [22], and presumably in other structures with anisotropic architecture, but this has not been confirmed experimentally.

Plastic anisotropy in material systems can exist either in-plane, out-of-plane, or both; oftentimes materials possess unique, direction-specific properties in all of the orthogonal and shear directions. Sparse computational studies exist in the literature to address in-plane plastic anisotropy, and the potential for indentation to extract this behavior by examining and quantifying the degree of pile-up around the indenter [23, 24]. However, these proposed techniques are limited by experimental complexity, errors in analysis, and employ complex inverse analysis techniques to extract the degree of in-plane anisotropy from experimental data. The aforementioned techniques apply to in-plane anisotropic plasticity, which is of particular importance in cup drawing applications, as the degree of anisotropy is related to the tendency of a sheet to form “ears” during deep-drawing applications (Fig. 1-1B) [21, 25].

Perhaps equally significant is the quantification of plastic anisotropy out of the plane of the sheet, which, in conjunction with planar anisotropy, provides information on the formability of a material. Out-of-plane plastic anisotropy in sheets is typically represented by means of the Lankford Coefficient (\( \bar{R} \)), which is defined as the ratio of the plastic width-strain (in the plane of the sheet, transverse to rolling direction) to the through-thickness plastic strain [19], during a longitudinal tensile test in the rolling direction. In this manner, \( \bar{R} \) indicates the "easy direction" for transverse plastic strain and in general, a material with a higher value of \( \bar{R} \) will have a higher resistance to thinning/tearing during forming or drawing operations (Fig. 1-1A) [26], and \( \bar{R} \) greater than unity is often desirable.
Plastic anisotropy in rolled sheets has traditionally been analyzed by conducting tensile tests on strips cut at different angles from the rolling direction and measuring the contraction ratios during testing [19]. This method is tedious, yet sufficient for sheet metals but the application to other material systems is limited. For example, if one were to seek the properties of a coating-substrate system, such an analysis would be impractical due to the combined effects of the coating and the substrate on which it lies. Indentation-based experiments are a great candidate for evaluation of such properties in various material systems for a few, main reasons. Indentation testing machines are readily available commercially and in material characterization laboratories world-wide and are currently being used for the classification of various material properties. Second, the systems in which indentation can be used are far from limited; indentation tests are notorious for their large-scale applicability, as mentioned in the beginning of this section. Finally, indentation provides a means of inducing localized plastic deformation, which can ultimately serve as a great analysis tool for anisotropy in these properties for a given material.
1.2. FACTORS INDUCING ANISOTROPIC PLASTIC BEHAVIOR IN MATERIALS

The three most important causes of plastic anisotropy in materials are: (i) anisotropy of individual crystals in polycrystalline metals, (ii) directionality of the distribution of particular phases and defects in multiphase alloys (i.e. fibrous structures), and (iii) internal stresses developing as a result of oriented deformation history. The last factor becomes insignificant in metals that have been recrystallized through annealing or other material processing technologies. In such cases, the texture, or preferred crystallographic orientation, becomes the sole significant cause of plastic anisotropy in materials. The chief impact of plastic anisotropy in the process of plastic working is the role played by texture and anisotropy in the process of metal sheet drawing [27].

The effects of anisotropic plastic properties in materials generally manifest when the material undergoes forming processes through which application specific parts are manufactured. In general, it is found that when there is a high degree of anisotropy in the plane of the sheet, the orientation of the sheet with respect to the part to be formed (i.e. die) will be important; in circular parts, for example, asymmetric forming will be observed which is quite often undesirable. In the case of anisotropic plastic behavior in the normal direction (normal to the plane of the sheet), a material that shows a width strain greater than the thickness strain during a tensile test has a greater strength in the through-thickness direction and, consequently, a resistance to thinning. As previously mentioned, a high value allows for deeper parts to be drawn from the sheet. Furthermore, in shallow, smoothly-contoured parts (e.g. outer panels of an automotive) a higher normal anisotropy ratio reduces the probability of wrinkling or ripple-formation in the part.
Plastic anisotropy can therefore be seen as advantageous or disadvantageous when applied to the formability of sheet materials. While a high degree of normal anisotropy is preferred, a low degree of planar anisotropy is ideal. In a study by Lankford et al. [28], it was shown that increases in normal anisotropy has a significant impact on the deep drawability of steel sheets. This finding came in a time in which the scientific community believed that material anisotropy was synonymous with unfavorable results during high degrees of plastic working. Lankford continued to state that only in particular cases could planar anisotropy make a process more efficient. However, this type of anisotropy can be beneficial if a drawing operation is inherently asymmetrical or if the sample can be oriented in such a way that makes favorable use of the intrinsic directionality of the material properties [29].

To examine how the anisotropic plastic property (both in-plane and normal) of a material responds to working, a rolling technique that leads to a large degree of reduction in the thickness of the sheet (i.e. severe plastic deformation) is ideal. A recently established technique known as accumulative roll bonding (ARB) is a method developed in 1998 by Saito et al. [30] for the production of ultrafine grained materials. The process involves passing sheets of material through a set of rollers, cutting the sheets in half, stacking, and repeating the process through multiple cycles with each cycle leading to a 50% reduction of the original thickness. A formal explanation of the experimental protocol can be found in Ref. [30].

The effect of the ARB technique on the anisotropic plastic behavior of metal sheets was studied in a recent article by Beausir and colleagues [31]. Changes in the planar \((\Delta r)\) and normal \((<r>\) anisotropy as a function of ARB rolling cycles were analyzed for aluminum alloys AA1050 and AA6016. For ideal performance of sheet materials, \(\Delta r \approx 0\) and \(<r> \gg 1\) is desirable. Experiments showed that the average normal anisotropy \(<r>\) increases steadily with
number of ARB cycles from about 0.6 to 0.9 and the average planar anisotropy ($\Delta r$) decreases and changes sign from about 0.6 to -0.7 for AA1050 and from 0.2 to -0.7 for AA6016. Inflection in sign of $\Delta r$ indicates a shift in the earing tendency from the rolling/transverse direction to the 45° direction. The results of the experiments up through eight ARB cycles are shown in Figure 1-2 [31]. In regards to the deep drawing properties, the evolution of $\Delta r$ and $<r>$ are contradictory, and therefore, it is shown that the best conditions for deep drawing applications are satisfied through low-cycle ARB.

Figure 1-2: Extracted from Ref. [31]. Variation of the in-plane ($\Delta r$) and normal anisotropy ($<r>$) of AA1050 and AA6016 alloys with number of ARB rolling cycles.

The most applicable finding of the study by Beausir et al. is not the specific description of the anisotropy ratios of two metallic alloys. Conceivably more pertinent, is the observed relationship between the degree of working of a metal and the resulting anisotropic material properties, which emphasizes the importance and impact of the deformation history on the plastic
behavior of the material. Thus, an important point is raised; anisotropy is not a property that is unique to materials but perhaps, more strongly depends on the working history. This fact emphasizes the need for standardized testing techniques for such materials so that easy classification can be achieved on an application-specific basis. This is the point that is driving research currently in terms of characterization of anisotropic plastic behavior of material systems.

1.3. CURRENT CRITERIA FOR CHARACTERIZATION OF ANISOTROPIC PLASTIC BEHAVIOR IN MATERIALS

To facilitate the characterization of plastic anisotropy in material systems (predominantly in sheet metals), methods have been heavily sought over the course of the past few decades. Not only have various yield criterion been proposed, but a number of experimental methods have been suggested for full characterization as well. In general, most of the anisotropic models are derived mathematically, using isotropic yield criterion developed by von Mises or the generalized forms; most notably that developed by Hosford in 1972 [32]. In order to adapt the criteria to allow for anisotropic yielding, the Lankford Coefficients are used, and are measured via uniaxial tensile tests at different angles with respect to the rolling direction. The coefficient is measured using the following relation:

\[ R_\alpha = \frac{e_{\text{trans}}^p}{e_{\text{thick}}^p}, \]

where \( \alpha \) is the angle from an anisotropy-direction (i.e. the rolling direction in worked sheet samples) and \( e_{\text{trans}}^p \) and \( e_{\text{thick}}^p \) represent the plastic strain in the transverse direction (90° from \( \alpha \)) and the through thickness direction, respectively. It is well known that anisotropic materials
will show a variation in both the Lankford Coefficient and the yield stress with $\alpha$ [33], and the Lankford coefficient as a function of $\alpha$ generally shows a V-shaped dependence with maxima at $0^\circ$ or $90^\circ$, and a clear minimum at $45^\circ$ [34].

The yield criteria for anisotropic materials can take one of two forms; either the components of the stress tensor and quadratic or non-quadratic. The resulting shapes of the yield surfaces of these two mathematical formulations differ slightly; the quadratic form showing the elliptical shape representative of the von Mises yield surface and the non-quadratic form showing the characteristic corners of the Tresca yield surface. A schematic representation of these two types of yield surfaces are shown in Figure 1-3 [33]. Quadratic yield criterion are oftentimes more applicable to sheet metals and less accurate experimentally for polycrystalline materials with face-centered cubic (FCC) or body-centered cubic (BCC) crystal systems [35]. Thus, the yield-criteria of the non-quadratic form are thought to be more accurate in describing yielding of FCC or BCC materials.

Furthermore, yield criteria can be grouped even further into criteria for conditions of plane stress, and those that are applicable to general stress states. Choice of yield criteria is entirely dependent on the sample geometry and loading conditions, but to encompass all such scenarios, both types will be discussed, beginning with plane stress and followed by the more general cases.
1.3.1. Anisotropic Yield Criteria for Plane Stress

The set of yield criteria that are given for the plane stress condition are conditional; (i) they are limited in the type of anisotropy that they can describe and (ii) they are limited to systems in which the plane stress assumption can be applied. Plane stress is the special case in which the stress components in one of the orthogonal directions are zero. The most widely known case is when stresses in the z-direction (through the thickness) are zero, that is, $\sigma_{zz} = \sigma_{yz} = \sigma_{xz} = 0$. The stress matrix is therefore reduced, and can be written as:

$$[\sigma] = \begin{bmatrix} \sigma_{xx} & \sigma_{xy} \\ \sigma_{yx} & \sigma_{yy} \end{bmatrix}. \quad (1-1)$$
It is important to realize that the plane stress condition applies to three-dimensional geometry, however, due to the geometry of the problem, the stress state is two-dimensional [36]. A simple example of such a condition is a thin plate; specifically a geometry in which the cross-sectional area is significantly larger in one plane than in the others, forcing the stress components in that direction to vanish.

The criteria presented are designed to describe orthotropic plastic behavior under plane stress loading conditions. Orthotropic materials are those which fall into the category of having symmetry with respect to three mutually perpendicular (orthogonal) planes [36]. Metal sheets are a common example of a material which exhibits this type of anisotropy. As they are generally fabricated by rolling the material in a sequential process, the orthotropy directions become: (i) the rolling direction, (ii) the direction transverse to the rolling direction, and (iii) the direction perpendicular to the plate (i.e. the through-thickness direction of the plate).

Bassani (1977)

A 1977 plane stress yield criterion was proposed by Bassani [37] which was limited to conditions of planar isotropy or more specifically, materials exhibiting transverse isotropic plastic properties. This is to say, the properties in the plane of the sheet are independent of direction, but different from the properties in the direction through the thickness of the plate. The criterion is proposed to be applied to the behavior of FCC and BCC metals whose textures give rise to transverse isotropic plastic properties, and takes the following form:

\[
\phi = \left(\frac{\sigma_1 + \sigma_2}{2\sigma_b}\right)^n + \left(\frac{\sigma_1 - \sigma_2}{2\tau_0}\right)^m = 1
\]

(1-2)
where $\phi$ represents the non-dimensionalized yield function, $\sigma_1$ and $\sigma_2$ are the principal stresses in the plane of the sheet, $\sigma_b$ and $\tau_0$ are the biaxial yield stress and simple-shear yield stress in the plane of the plate, respectively, and $m$ and $n$ are two, arbitrarily chosen dimensionless parameters which can be varied to better accommodate the yield surface.

**Logan & Hosford (1980)**

Another orthotropic criterion was proposed by R.W. Logan and W.F. Hosford in 1980 [38] which was developed around the assumption that the stress state has minimal contributions from the shear stress components. The criterion states that yielding will occur if the condition:

$$\phi = g|\sigma_1|^m + f|\sigma_2|^m + h|\sigma_1 - \sigma_2|^m = \sigma_b^m$$

is satisfied. The constants $f$, $g$, and $h$ are positive in nature and must satisfy the condition $f + g = 1$. The exponent $m$ is limited to two values; $m = 6$ for the BCC crystal system, and $m = 8$ for the FCC crystal system. As evident, the yield criterion has no shear stress components which leads to a major limitation in its applications. For this yield criterion to be valid and practical, the principal directions (directions of the principal stresses) are required to lie parallel with the orthotropy directions. Therefore, the criterion cannot be applied to general stress states, limiting its general use.

**Hill (1990 & 1993)**

In addition to the yield criterion proposed by Hill which encompasses anisotropy throughout a material given a general stress state, he also derived several yield criteria for materials subjected to plane stress conditions. The first, being more restrictive in its use, was proposed to model anisosensitive behavior, a case in which the response of the material changes
when the signs of all stress components are negated [39]. The model is given by the following relation:

\[
\phi = \frac{\sigma_1^2}{\sigma_0^2} \frac{c\sigma_1\sigma_2}{\sigma_0\sigma_{90}} + \frac{\sigma_2^2}{\sigma_{90}^2} + \left\{ (p + q) - \frac{p\sigma_1 + q\sigma_2}{\sigma_b} \right\} \frac{\sigma_1\sigma_2}{\sigma_0\sigma_{90}} = 1
\]  

(1-4)

where \( c, p, \) and \( q \) are dimensionless parameters and \( \sigma_0 \) and \( \sigma_{90} \) are the yield stresses of the material in the rolling and transverse directions, respectively. Similar to the criterion proposed by Logan and Hosford, Hill’s version also requires that the principal directions lie parallel to the orthotropic directions.

To remove the restriction imposed by this yield criterion, Hill proposed another yield criterion [40] designed for use in states of general plane stress. For the purpose of generalization, he introduced the shear stress component (\( \sigma_{xy} \)) and proposed a criterion that was no longer dependent on the principle stress terms:

\[
\phi = \left| \sigma_x + \sigma_y \right|^m + \left( \frac{\sigma_b}{\tau_0} \right)^m \left| (\sigma_x - \sigma_y)^2 + 4\sigma_{xy}^2 \right|^{m/2}
\]

\[
+ \left| \sigma_x^2 + \sigma_y^2 + 2\sigma_{xy}^2 \right|^{(m/2)-1} \left\{ -2a(\sigma_x^2 - \sigma_y^2) + b(\sigma_x - \sigma_y)^2 \right\} = 2\sigma_b^m
\]

(1-5)

where \( a, b, \) and \( m \) are dimensionless parameters which can be altered to vary the convexity of the yield surface. In the case of this yield criterion however, changes to the parameter \( m \) do not accurately define a yield surface consistent with that which is determined for polycrystalline materials/metals experimentally.
1.3.2. Anisotropic Yield Criteria for General Stress States

The yield criteria designed for the characterization of anisotropic yielding of materials in the general stress state are advantageous as their use has fewer limitations than those prescribed for the plane stress condition. The most general was presented by Tsai and Wu in 1971 [41], which takes into account differences in strengths due to positive and negative stresses, and can successfully encompass alternative material symmetries, multi-dimensional space, and multi-axial stress states. The general yield criterion is of quadratic form and is given in indicial notation as:

\[ \phi = F_i \sigma_i + F_{ij} \sigma_i \sigma_j = 1 \]  \hspace{1cm} (1-6)

where \( F_i \) and \( F_{ij} \) are experimentally determined material strength parameters and the normal and shear stress components are defined according to the relation:

\[
\begin{bmatrix}
\sigma_1 \\
\sigma_2 \\
\sigma_3 \\
\sigma_4 \\
\sigma_5 \\
\sigma_6 \\
\end{bmatrix} =
\begin{bmatrix}
\sigma_{xx} \\
\sigma_{yy} \\
\sigma_{zz} \\
\sigma_{yx} \\
\sigma_{zx} \\
\sigma_{xy} \\
\end{bmatrix}
\]  \hspace{1cm} (1-7)

The presence of linear terms in the criterion take into consideration the difference between positive and negative yield stresses, and therefore make this criterion particularly attractive for applications to anisotropic composite materials and other materials exhibiting the Bauschinger effect; having different yield strengths in tension and compression. This form is not particularly suitable for practical applications, as it requires experiments in order to determine the material strength parameters on a per material basis. However, from the general equation, better known and more widely used criteria have been derived for use in more practical situations.
Hoffman (1967)

Following the formulation of Tsai and Wu, Hoffman proposed a yield criterion, valid for general states of stress for orthotropic materials showing anisotropic behavior, which takes the form [42]:

\[ \phi = C_1(\sigma_y - \sigma_x)^2 + C_2(\sigma_z - \sigma_x)^2 + C_3(\sigma_z - \sigma_y)^2 + C_4\sigma_x + C_5\sigma_y + C_6\sigma_z + C_7\tau_{yx}^2 + C_8\tau_{zx}^2 + C_9\tau_{xy}^2 = 1 \]  

The nine material parameters \((C_1-C_9)\) can be uniquely defined through derived relations to basic strength data; the uniaxial tensile yield stresses \((T_x, T_y, T_z)\), the uniaxial compressive yield stresses \((C_x, C_y, C_z)\), and the pure shear yield stresses \((S_{yx}, S_{zx}, S_{xy})\). Hoffman explicitly derives the relations between the material parameters and testing data to obtain \(C_1, C_4,\) and \(C_7\), and the remaining six constants are determined by permutation of \(x, y,\) and \(z\). The key benefit to this formulation is in the fact that differences between tensile and compressive yield strength can be defined, however for cases in which this is not the case, yield criteria designed for isosensitive materials are more applicable and desirable. Under the assumption that material behavior does not vary with the sign of the uniaxial stress terms, the linear terms of the above equation vanish to give a more simplistic formulation with less experimentally derived material input data required.

Barlat (1991)

A criterion valid for arbitrary states of stress in orthotropic materials was proposed by Barlat et al. in 1991. The criterion is non-quadratic in form and is defined as:

\[ \phi = |S_1 - S_2|^m + |S_2 - S_3|^m + |S_3 - S_1|^m = 2\sigma_0^m \]  

(1-9)
where $S_{1,2,3}$ are the components of a symmetric matrix with components defined in terms of the triaxial stress tensor as follows:

\[
S_{xx} = \frac{c(\sigma_{xx} - \sigma_{yy}) - b(\sigma_{zz} - \sigma_{xx})}{3}
\]

\[
S_{yy} = \frac{a(\sigma_{yy} - \sigma_{zz}) - c(\sigma_{xx} - \sigma_{yy})}{3}
\]

\[
S_{zz} = \frac{c(\sigma_{zz} - \sigma_{xx}) - a(\sigma_{yy} - \sigma_{zz})}{3}
\]

(1-10)

And the shear components of the tensor are simply defined as the shear stress components scaled by constants $f$, $g$, and $h$. The exponent $m$ can be used to influence the curvature of the yield surface to achieve consistency with that observed in polycrystalline metals. Figure 1-4 [33] shows the variation of the Barlat 1991 yield surface with changing shear stress to uniaxial yield stress ratio.

Figure 1-4: Barlat 1991 yield loci in plane stress for different values of shear stress to uniaxial yield stress ratios. Axes represent the stresses in line with the directions of anisotropy. Note that with decreasing magnitude of shear stress, the surface resembles that which is typical of polycrystalline materials.
Hill (1948)

Hill’s 1948 yield criterion [43] is a generalization of the von Mises criterion which expresses anisotropy in materials with three orthogonal planes of symmetry. It is expressed by a quadratic function:

$$\phi = F(\sigma_y - \sigma_z)^2 + G(\sigma_z - \sigma_x)^2 + H(\sigma_x - \sigma_y)^2 + 2L\tau_{yx}^2 + 2M\tau_{zx}^2 + 2N\tau_{xy}^2 = 1 \quad (1-11)$$

which is of the same form as Hoffman’s criterion with the linear stress terms eliminated; as the tensile and compressive yield strengths are assumed to be identical. Consistent with the quadratic nature of the yield function, the yield surface is elliptical; resembling that representative of the von Mises yield criterion for isotropic materials. Here, F, G, H, L, M and N are constants that are specific to the state of anisotropy of the material and x, y, and z are the principal axes of anisotropy. In the case of sheet metals, x is generally parallel to the rolling direction, and y and z are in the transverse and normal direction, respectively to maintain orthogonality of the coordinate system.

The constants can be expressed in terms of the uniaxial tensile yield stresses (X, Y, Z) by the following relations:

$$\frac{1}{X^2} = G + H; \quad \frac{1}{Y^2} = H + F; \quad \frac{1}{Z^2} = F + G$$

and these relationships can be further simplified to obtain individual expressions for the constants in terms of the uniaxial yield stresses:

$$2F = \frac{1}{Y^2} + \frac{1}{Z^2} - \frac{1}{X^2}; \quad 2G = \frac{1}{Z^2} + \frac{1}{X^2} - \frac{1}{Y^2}; \quad 2H = \frac{1}{X^2} + \frac{1}{Y^2} - \frac{1}{Z^2}$$
The constants $L$, $M$, and $N$ are then proportional to the inverse squares of the shear yield strengths.

When the geometry of the problem calls for a situation of plane stress, the yield criterion is reduced to:

\[
\frac{1}{x^2} \sigma_x^2 - \left( \frac{1}{x^2} + \frac{1}{y^2} - \frac{1}{z^2} \right) \sigma_x \sigma_y + \frac{1}{y^2} \sigma_y^2 + \frac{1}{z^2} \tau_{xy}^2 = 1
\]  

(1-12)

a form requiring four experimentally resolved yield strengths as opposed to the general form which requires six.

When simulating sheet metal forming processes, it is perhaps more convenient to rewrite the plane stress yield criterion in terms of the anisotropy ratios $r_0$, $r_{90}$, and $r_{45}$ [44]:

\[
\sigma_x^2 - \frac{2r_0}{1+r_0} \sigma_x \sigma_y + \frac{r_0 + r_{90}}{r_{90}(1+r_0)} \sigma_y^2 + \frac{r_0 + r_{90}}{r_{90}(1+r_0)} (2r_{45} + 1) \tau_{xy}^2 = \sigma_0^2
\]  

(1-13)

This form of the yield function allows for tracking the influence of the anisotropy coefficients on the yield surface as shown in Figure 1-5 [44].

![Figure 1-5: Influence of $r_0$ (left) and $r_{90}$ (right) on the yield locus defined by the Hill 1948 yield criterion in principal stress space.](image)
In the case of a material exhibiting only normal anisotropy \( r_0 = r_{90} = r \), it is imposed that \( \sigma_0 = \sigma_{90} \) and the yield criterion (in terms of principal stresses) takes the simplified form:

\[
\sigma^2_1 - \frac{2r}{1+r} \sigma_1 \sigma_2 + \sigma^2_2 = \sigma^2_u
\] (1-14)

where \( \sigma_u \) is the uniaxial yield stress.

For general stress states, due to its relatively simple formulation and broad applicability, the Hill-criterion is the most widely used criterion both in practice and in commercial Finite Element Analysis software. Perhaps the chief advantage is the fact that the parameters can be easily found from standard tensile tests. However, a drawback of Hill’s yield criterion is that it oftentimes cannot accurately describe the experimentally observed “rounded-off” Tresca yield surface seen in metals such as aluminum, due to its quadratic nature. Furthermore, if the Lankford coefficients \( (R_d) \) are implemented to calculate the constants, the variation of the yield stresses as a function of the orientation with respect to the principle direction of anisotropy, is overestimated [33]. The other, more recent criteria proposed by Barlat [45] and Karafillis (see reference [46] for full explanation of this criterion) are known to give better results for cases of polycrystalline materials subjected to general states of stress [33].

The mathematical formulae available for describing the anisotropic plastic properties/yielding of materials are not limited to the cases described here; researchers are constantly developing additional criteria that are best-suited for the specific materials and material systems that need to be characterized [47-53]. However, the criteria discussed here are the most general and widely implemented for the cases of plane stress and for general stress states. The real challenge that is associated with all available criteria is the experimental determination of the anisotropic parameters (constants) that are necessary for the accurate
characterization of yielding. The next section provides an overview of the methods currently applied for the determination of these parameters for the completion of the anisotropic yield criteria for specific materials.

1.4. EXPERIMENTAL TECHNIQUES

1.4.1. Uniaxial Testing of Sheet Metals

Traditionally, anisotropic plastic behavior has been generally, in rolled sheets, by the plastic strain ratios observed in response to tensile tests on strips cut at different angles with respect to the rolling-direction, in the plane of the sheet [54, 55]. The typical arrangement of these tests is highlighted in Figure 1-6. The parameter most commonly used for characterization is the Lankford Coefficient (R-value), which is defined as the ratio of the plastic strain in the width direction (90° to the tensile axis) to the plastic strain through the thickness of the sheet.

![Figure 1-6: Tensile specimen geometry used to determine the Lankford Coefficient from rolled sheet metal samples. In this case, α denotes the orientation of the bar with respect to the sheet rolling direction [54].](image-url)
In this technique, the $R$-value is generally measured after the specimen has failed, or at half the strain at fracture. More recently, standards have called for determination of the $R$-value at specific strains; for example the ASTM E517 standard suggests measurement at a strain of 20\% [56]. As there is oftentimes a dependence of $R$-value on magnitude of strain, it is often suggested that a general formulation of the dependence be developed prior to testing [57]. From the $R$-values at $0^\circ$, $45^\circ$, and $90^\circ$ averaging techniques have been used to characterize the resistance of a sample against thinning and the in-plane anisotropy in the sample by the following relations:

$$\Delta R = \frac{R_0 + R_{90} - 2R_{45}}{2}$$
$$\bar{R} = \frac{R_0 + R_{90} + 2R_{45}}{4}$$

with the former being the measure of in-plane anisotropy and the latter being for out-of-plane anisotropy. $\bar{R}$ greater than unity is an indication that the material has good formability; for example the material can be drawn into a cup with less tendency to thin to the point of tearing. To put the range of this parameter in perspective, high-quality steels manufactured for sheet forming applications have $\bar{R}$ of about 2.0, and such values are not extreme for other sheet materials with hexagonal crystal structure (HCP). However, in materials showing face centered cubic (FCC) crystal structure, such as aluminum or copper sheet, $\bar{R}$ values are rarely greater than one and oftentimes are considerably less [54].

1.4.2. Biaxial Testing of Sheet Metals:

Testing of sheet metals under conditions of multi-axial loading is becoming increasingly important as forming processes generally subject the materials to multi-axial states of stress. As a result, biaxial testing with cruciform specimens has developed into the most favorable method
as it allows for altering the proportion of load or displacement across two coordinate axes [58].

It is also important to note that uniaxial tension tests can only provide accurate experimental data for isotropic materials, and therefore in the case of anisotropic materials the results of standard uniaxial tests can be erroneous.

Makinde et al. [59] designed a biaxial testing machine for cruciform specimens that allowed for simultaneous tensile in-plane loading across two orthogonal directions. A schematic of the test fixture is shown in Figure 1-7. The device implements displacement control (strain control) and the corresponding load is measured via two load cells; one placed in line with one orthogonal direction and the other placed perpendicular to it. Various types of cruciform specimens can be placed between the grips and can be subjected to both small and large strains. Ultimately, the device is capable of determining non-uniformity of strain inside the gauge length and thus, gives an understanding of the inherent anisotropy both in the elastic response and plastic response of the material.

![Figure 1-7: Schematic representation of the biaxial testing machine developed by Makinde et al [59].](image_url)
Lowering the cost, accuracy, and effectiveness of testing apparatus, Kuwabara et al. [60] designed a new device in order to clarify, experimentally, the elastic and plastic deformation behavior of cold-rolled low-carbon steels subjected to biaxial tension. The setup consists of two pairs of opposing hydraulic cylinders, each connected in series, so that the same pressure could be applied through the opposing cylinders. The primary modification of this device, setting it apart from that of Makinde et al., was the introduction of a link mechanism which held the center of the sample in place during testing [61]. This addition served to limit lateral translation of the cruciform specimen due to miniscule variations in opposing applied pressures. As before, load cells are implemented to measure the applied stress in the sample, and the corresponding strains are measured using strain gauges placed in the center of the sample. Figure 1-8A shows a schematic representation of the test fixture. It was observed that the measured flow stresses in the low-carbon steel samples were in good agreement with those predicted by both Hill's quadratic yield criterion and Hosford's yield criterion as shown in Fig. 1-8B; the latter better representing the experimental results [62].

Figure 1-8: A) Schematic representation of the experimental setup designed by Kuwabara et al. [60] B) Stress-strain relation for low-carbon steel obtained experimentally with the biaxial tensile test fixture and comparison with the behavior as predicted by the Hill quadratic yield criterion and the Hosford yield criterion [62].

Aside from the cost and customization required for the design and development of a suitable biaxial testing, one of the most challenging aspects of such a testing system lies in the design of the test specimen [61]. Although cross-shaped geometries are generally used to perform biaxial tests on sheet metals, the design of the specimen is the main difficulty that restricts the applications of the test [63]. Modifications to the specimens have been researched extensively [64, 65], however no standard geometry exists and therefore, a barrier to comparison and collaboration between different research groups exists. The use of the Finite Element Method, and other modeling techniques, have facilitated work in the design phase and aided in the optimization process [66].

This presents a necessity for the development of alternative testing methods for specimens with anisotropic plastic properties. Although there have been quite a few advancements in the testing protocols over the past few years, there remains a demand for more universal and accurate testing methods. Recently, researchers have explored the concept of using instrumented indentation techniques for characterization of anisotropic yielding [67-69] due to the fact that it provides a means of inducing localized multi-axial stress fields, wide-scale availability of testing equipment, minimal sample preparation, and applicability across a variety of material systems.

1.4.3. Indentation-based Characterization Techniques:

In a recent study by Yonezu et al. (2009) [67], a method was devised to extract the in-plane anisotropy of a sample using a single spherical indentation and the corresponding load-depth relation. The proposed method was developed through the use of FEM and a subsequent
dimensional analysis to determine the yield stress \((\sigma_y)\), work hardening exponent \((n)\), and the ratio of the in-plane yield stresses \((m)\).

The present technique relies on the examination of the deformed geometry normal to the indented surface following unloading. Essentially, the remaining permanent deformation resulting from the indentation was found to be non-uniform; the pile-up height in the direction of lower yield stress becomes higher than that in the direction of higher yield stress. In fact, it was observed that the differences in pile-up height became larger as the value of \(m\) increased. The method as proposed involves the use of several functions relating the indentation response and impression geometry to the anisotropic plastic property of the material.

Figure 1-9 [67] shows the dependence of pile-up height on the yield stress ratio \((m)\) resulting from the FEM analysis as presented by Yonezu et al. The computed unloaded impressions for an isotropic material (a), and two anisotropic materials (b-c) are shown. For the isotropic material, the boundary at the contact area is uniform and completely circular in shape, signifying that there is no anisotropy in the in-plane plastic behavior. In contrast, different pile-up heights were observed between the x- and y- axes in the anisotropic materials; varying to a degree consistent with the anisotropy case modeled, although the yield stress and work-hardening rate are equivalent in the cases shown. When the yield stress along the x-axis is higher than along the y-axis, the height in the y-direction becomes larger than that in the x-direction. It is important to note that this is under the assumption that the work-hardening rate and yield stress in shear are isotropic in nature, however the difference between the anisotropic cases (i.e. \(m = 1.5\) and \(m = 2.0\)) may be attributed to the in-plane anisotropy of the material yield stresses.
From a parametric study, the relationship between $m$ and the pile-up height ratio, was developed of the form:

$$\frac{h_{cX}}{h_{cy}} = C_1 \times \sigma_Y^{C_2}$$

where the left-hand-side of the equation is the ratio between the pile-up heights, $\sigma_Y$ is the yield stress of the material, and $C_1$ and $C_2$ are constants that depend on both the value of $n$ and $m$. The dependence of these constants on work-hardening rate for values of $m = 1.20$, $1.50$, and $2.00$ are shown in Figure 1-10. The relations can be fit with quadratic equations, the coefficients are obtained, and through substitution of $\sigma_Y$ into the relation between the pile-up height ratio and the yield stress, a relationship between the pile-up height ratio and $m$ can be obtained.

![Figure 1-9: Results extracted from ref. [67]. Contour maps of surface height near the impression region of the material for isotropic material (a) $m = 1.0$, and anisotropic materials (b) $m = 1.5$, and (c) $m = 2.0$.](image)
Figure 1-10: Extracted from Ref. [67]. Coefficients (a) C1 and (b) C2 with respect to n for the above relation.

In further work, Yonezu et al. applied the proposed technique to characterize the in-plane anisotropic yielding of silicon-carbide whisker-reinforced aluminum alloys [70]. The authors considered this a representative anisotropic material as such metallic composite materials are known to have unique features in addition to a wide range of applications. Samples were prepared through an extrusion process; forming an Al6061 rod with SiC (20% volume fraction) whiskers oriented parallel along the longitudinal direction. Due to the aligned whiskers along a
single direction, the mechanical properties were inherently anisotropic in nature, with the anisotropic principal axes being the longitudinal and transverse directions of the rod.

Two sets of experiments were conducted; first a set of compression tests in both the longitudinal and transverse directions (up to 20% uniaxial strain) were used to determine the degree of anisotropic yielding of the samples so that the results from the indentation analysis could be compared. The yield stress in the longitudinal and transverse directions were found to be 281 and 224 MPa, respectively, giving a value of the yield stress ratio \((m)\) of approximately 1.25. Next, a set of five indentation tests were carried out using an indenter with a diamond spherical tip, and the load-displacement relationships and pile-up topographies were obtained. Through inverse analysis of the indentation data, the yield stress ratio was resolved. The results of the analysis and the error in calculation are summarized in Table 1-1. In all cases, the in-plane yield stress ratio was approximated within <10% error using the proposed algorithm. While this is a reasonable assessment, there is room for improvement in the technique so that better estimates can be made for the tested materials.

<table>
<thead>
<tr>
<th>Test #</th>
<th>Yield Stress Ratio, (m)</th>
<th>Error %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.34</td>
<td>+7.2%</td>
</tr>
<tr>
<td>2</td>
<td>1.32</td>
<td>+5.6%</td>
</tr>
<tr>
<td>3</td>
<td>1.36</td>
<td>+8.8%</td>
</tr>
<tr>
<td>4</td>
<td>1.35</td>
<td>+8.0%</td>
</tr>
<tr>
<td>5</td>
<td>1.35</td>
<td>+8.0%</td>
</tr>
<tr>
<td>Compression test</td>
<td>1.25</td>
<td></td>
</tr>
</tbody>
</table>

Table 1-1: Summary of the experimental results obtained by Yonezu et al. [70]. Results from five indentation tests and the corresponding value of \(m\) are shown with the associated error from the accepted value of 1.25 as determined through compression testing.
The above method is useful in estimating the in-plane yield stress ratio within a reasonable factor of error. However, a number of predetermined material parameters are required for implementation of the dimensional analysis, which for anisotropic materials can be oftentimes difficult to uniquely ascertain. Furthermore, determination of the pile-up heights within a reasonable accuracy is tedious; complex imaging equipment and techniques are a necessity. As a result, the analysis is subject to confounding error and unique determination of the in-plane anisotropy becomes complicated. Nevertheless, the contributions by Yonezu et al. suggest that instrumented indentation and contact based experimental techniques can be useful when the extraction of anisotropic plastic properties is desired.

1.5. PLASTIC ANISOTROPY IN COMMERCIAL FINITE ELEMENT CODES

The goal of this work is to use Finite Element Modeling to develop and test experiments to fully characterize the anisotropic plasticity in a sample using indentation-based techniques. For the purpose of this study, the Hill Yield Criterion [43] for general stress states (see Section 1.3.2.: Hill (1948)) will be employed and the anisotropy will be constrained to the normal directions (isotropic in shear).

To examine anisotropic plasticity via indentation, it is essential to define a local coordinate system with respect to which the plastic properties will be characterized. For the purpose of indentation analysis, an orthogonal coordinate system will be chosen, with the X and Z directions corresponding to the length and width of the plane, and the Y direction being the out-of-plane direction; parallel to the axis of indentation. Such a coordinate system allows for the classification of plastic anisotropy in the normal and shear directions. This is more applicable to
indentation than the Lankford Coefficients [28] that are used to characterize out-of-plane to in-plane yield stress ratios in sheet metals at different angles with respect to the rolling direction.

In order to fully describe the state of plastic anisotropy, it is necessary to implement a total of six constants (R-values), that are representative of the yield stress ratios in the normal ($R_{11}$, $R_{22}$, and $R_{33}$) and shear ($R_{12}$, $R_{13}$, and $R_{23}$) directions. It is important to note that one of these R-values is maintained at unity, while the five others are determined with respect to it.

In ABAQUS, one can input these R-values and they will be employed Hill’s yield criterion for general stress states [43] which has the form:

$$f \equiv F (\sigma_{yy} - \sigma_{zz})^2 + G (\sigma_{zz} - \sigma_{xx})^2 + H (\sigma_{xx} - \sigma_{yy})^2 + 2L\tau_{yz}^2 + 2M\tau_{zx}^2 + 2N\tau_{xy}^2 = 1,$$

where $\sigma_{ij}$ and $\tau_{ij}$ represent the normal and shear stress components, respectively, with respect to the local coordinate system. $F$, $G$, $H$, $L$, $M$, and $N$ are constants that collectively represent the state of plastic anisotropy present. The values of these constants are defined by the following relations:

$$F = \frac{1}{2} \left( \frac{1}{R_{22}^2} + \frac{1}{R_{33}^2} - \frac{1}{R_{11}^2} \right)$$
$$L = \frac{3}{2R_{23}^2}$$

$$G = \frac{1}{2} \left( \frac{1}{R_{33}^2} + \frac{1}{R_{11}^2} - \frac{1}{R_{22}^2} \right)$$
$$M = \frac{3}{2R_{13}^2}$$

$$H = \frac{1}{2} \left( \frac{1}{R_{11}^2} + \frac{1}{R_{22}^2} - \frac{1}{R_{33}^2} \right)$$
$$N = \frac{3}{2R_{12}^2}$$
There are some relevant points to keep in mind. First, only one of F, G, and H can be negative although it is unlikely due to the fact that it would require very large differences in the yield stresses in different directions [43]. Second, the yield stress ratios are decoupled unless there is symmetry in the anisotropy. For instance, in the case of indentation, one can speculate that there will be symmetry such that \( R_{11} = R_{33} \) and, according to Hill, a relationship between \( R_{22} \) and \( R_{12} \) is accordingly developed. Third, Hill’s equations do not accept extreme differences in normal anisotropy ratios. As an example, if there is a case in which \( R_{11} = R_{33} \) and the ratio between \( R_{22} \) and \( R_{11} \) is 0.5, an instability arises in Hill’s equations. Thus, other yield criteria have been shown to be more applicable for the full characterization of anisotropic plasticity, depending on the material system being characterized. In the special case of transverse isotropic plasticity, namely when \( R_{11} = R_{33} = 1 \) while \( R_{22} \) is varied, \( R_{22} \) can be related to the Lankford Coefficient (\( \bar{R} \)) through the relation: \( \bar{R} = 2R_{22}^2 - 1 \). For clarification, the coordinate system as applied in this study is highlighted schematically in Figure 1-11.

Figure 1-11: Schematic representation of the orientation of the \( R \)-values implemented throughout this work. The 2-direction corresponds to the indentation direction in all cases.
1.6. PURPOSE AND SCOPE

Examination of the broad extent of literature devoted to the characterization of anisotropic plastic materials both in the planar and normal directions suggests that there is a lack of a general method to analyze such materials. Furthermore, there is a need for a general method so that collaboration and comparison between the results of different research groups can be made possible. Currently, the most general method involves uniaxial tensile testing of samples in a number of orientations with respect to the principal anisotropy axis. Even so, this method is really only applicable to sheet materials; material systems that consist of only one component. For example, applications of these methods to coating-on-substrate systems or other multi-component systems is impractical.

The use of indentation based techniques can serve to widen the applicability of characterization techniques because they are already used for characterization of an extensive range of materials; spanning from \textit{in situ} biological organs \cite{11} to complex mechanical/material systems. The benefits in contact based methods fall mainly in the fact that localized, multiaxial stress fields can be induced in materials making observation of plastic flow and stress-strain relations quite reliable. Preliminary results have led to the understanding that the most plausible candidate for the unique characterization of materials exhibiting anisotropic plastic properties is through examination of the material behavior (i.e. flow) in the vicinity of contact. In regards to the stress-strain behavior of such materials it will be shown that unique characterization of parameters is inhibited by the fact that inflections arise when examining material hardness.

The present work is for the development, design, and testing of a set of virtual experiments using Finite Element Modeling with the specific aim to characterize the anisotropic
plastic property of a material in all normal directions given minimal material data prior to testing. With this aim, the main goal is to develop techniques using modified contact-based methods to successfully extract/characterize the anisotropic plastic property of a material sample. Models have been created and implemented with varying yield stress ratios using the ABAQUS POTENTIAL function, and plastic deformation was induced via contact with a rigid body. Details of the work done to achieve this objective will be presented in the following chapters.

Preliminary results have shown that anisotropic plasticity has a significant effect on the indentation load-displacement (P-h) relations and, in turn, the resolved Elastic Modulus from the Oliver-Pharr method [12]. This result further validated the importance of characterizing such behavior so that differences could be accounted for. It has been determined that indentation stress-strain relations are not sufficient for unique characterization of plastic anisotropy; often depending on a combination of material properties (i.e. elastic modulus, work-hardening rate, base-line yield stress, etc.). Thus, methods are sought to induce measurable differences in material response that are exclusive to anisotropic plastic behavior so that such behavior can be uniquely characterized.
CHAPTER 2. EFFECTS OF PLASTIC ANISOTROPY AND HARDENING ON INDENTATION MODULUS OF THIN FILMS


2.1. INTRODUCTION

Over the last decade, instrumented indentation has emerged as a powerful and flexible technique for the probing of mechanical properties for a wide range of materials systems. These include bulk materials, composites, thick- and thin-films on substrates, and biomaterials. Two major factors in this tool’s near-ubiquitous usage are the relative ease of specimen preparation, and the readily available algorithms to compute important quantities, such as elastic modulus, from force-depth ($P-h$) data. Focusing on the latter, the Oliver–Pharr method for modulus extraction has been incorporated into a number of commercial systems, largely due to its robustness, simplicity and ease of modification (i.e. correction factors) [1]. Nevertheless, in the literature, a number of cases, specifically the indentation of films on substrates, have been reported for which modulus values calculated via this method exhibit significant error [71, 72]. The majority of these cases concern with situations in which the indented specimen exhibits elastic heterogeneity (e.g., stiff film on compliant substrate) with or without plastic heterogeneity (e.g., soft/hard film on hard/soft substrate). However, little attention has been paid to the modulus variation of the cases in which purely plastic mismatch exists; that is to say, film and substrate have the same elastic properties but differ only in their plastic properties (e.g., yield strength).

In a study by Chollacoop et al. [73], experimental Berkovich indentation was carried out on single splat-on-substrate systems to extract load-displacement relations (loading and unloading). The term "splat" refers to a single impacted powder particle that results from a spray-coat process; a total coating is achieved when a series of splats are deposited on the target substrate. Load-displacement (loading and unloading) relations were experimentally determined for single splats with varying maximum depths between 100-1000 nm. Elastic modulus was
determined from the maximum load and slope of the unloading curve as per the Oliver-Pharr method for modulus extraction [12]. The system comprised a single Ni splat \((E \approx 200 \text{ GPa})\) on a stainless steel substrate \((E \approx 200 \text{ GPa})\) so that no elastic mismatch was inherent. Results showed that the resolved modulus shows a decrease with increasing maximum indentation depth; a result that should not be expected of a system with no elastic mismatch between the coating and substrate.

In an attempt to explain this anomalous behavior, Chollacoop and colleagues performed computational models of indentation for hard films on soft substrates, with no elastic mismatch, and showed that significant drops in indentation modulus could be obtained in such cases. The computations deviated from the experiments in two ways: first, the modulus drop observed, although significant, did not fully match experimental observations and second, the \(P-h\) behavior in the models was far too hard, and contained an inflection point during loading that was not observed experimentally.

To address this, in the present study Berkovich indentation of thin films on substrates has been modeled, with appropriate modifications to the plastic behavior to explore the possibility of achieving modulus decrease comparable to experiments without extremely hard \(P-h\) response. To that end, two possible material models have been incorporated: (i) plastic anisotropy, expected for the ultra-fine, columnar microstructure observed in rapidly quenched films [74] and (ii) low initial yield strength with high hardening slope. These parameters were explored systematically, and both additions were found to influence modulus decrease significantly.
2.2. OVERVIEW OF OLIVER-PHARR METHOD OF MODULUS EXTRACTION

For the determination of Elastic Modulus from instrumented indentation, the most significant algorithm is that which was proposed by W.C. Oliver and G.M. Pharr in 1992 [12]. The technique, as proposed, relies on extraction of specific properties from the load-displacement ($P$-$h$) relation of the sample both during loading and unloading. The three experimentally determined values are (i) the maximum depth of indentation, (ii) the maximum indentation load, and (iii) the initial slope of the unloading curve, all of which can be readily singled out from the indentation curve. The discussed parameters are then related to the Elastic Modulus ($E$) of the sample through the relation:

$$E^* = \frac{1}{\beta} \frac{dP}{dh} \left|_{h_m} \right. \frac{1}{2} \sqrt{\frac{\pi}{A_m}},$$

where $dP/dh$ is the initial unloading slope, $\beta$ is a correction factor, and $E^*$ and $A_m$ are the effective modulus of contact and the projected contact area, respectively, given by the expressions:

$$E^* = \left[ \frac{1 - v^2}{E} + \frac{1 - v_i^2}{E_i} \right]$$

$$A_m = \pi h_c^2 \tan^2 \theta.$$

In the latter expression, $h_c$ is the contact depth and can be calculated from experimental parameters and data by:

$$h_c = h_m - \frac{\varepsilon P_m}{dP/dh \mid_{h_m}}.$$
In the above relation, \( \varepsilon \) is a tip geometry factor equal to 0.72 for conical, 0.75 for spherical, and 1 for flat-punch indenters. \( P_m \) and \( h_m \) are the maximum indentation load and maximum indentation depth, respectively.

### 2.3. COMPUTATIONAL METHODS

Finite element simulations were carried out using ABAQUS/CAE. A schematic representation of the geometry of the model is shown in Figure 2-1A. Berkovich indentation was modeled using an axisymmetric conical tip of angle 70.3° from vertical, which has been shown to accurately represent 3D Berkovich indentation [75]. Figure 2-1B shows the axisymmetric mesh that was applied. A high-density mesh was used in the region underneath the indenter tip to ensure numerical accuracy and convergence of the results while eliminating contribution from applied far-field boundary conditions. A total of 4800 four-noded, bilinear axisymmetric quadrilateral elements were used throughout the study with appropriate boundary conditions (BCs) for axisymmetric analysis. Appropriate roller BCs were applied to both the left and bottom faces of the displayed mesh, while the right face was unconstrained. The mesh was partitioned to a thin top layer (film of thickness \( t \)) which was perfectly bonded to a thicker bottom layer (thickness 49\( t \)). A uniform mesh was applied to the top-left region underneath the indenter tip while a gradually coarser mesh was applied to regions that were further away.

The material properties of the modeled substrate were kept consistent with 304 Stainless Steel, with an elastic modulus (\( E \)) of 200 GPa, Poisson’s ratio (\( \nu \)) of 0.30, and yield stress (\( \sigma_y \)) of 215 MPa. For the film, \( E \) and \( \nu \) were kept constant at 200 GPa and 0.30, respectively, but different plastic property inputs were attempted. In the first set, ‘hard’ films from a previous study [73] were explored; film yield strength of 20 (20F) and 40 (40F) times substrate yield
strength, including plastic anisotropy. Plastic anisotropy was input by means of 6 R-values (as described in the Background section) using the POTENTIAL sub-option in ABAQUS, and $R_{22}$ and $R_{12}$, corresponding to anisotropy in normal and shear directions, were increased in accordance with experimentally observed values from columnar-grained materials [74].

The tip of the indenter was depicted via an axisymmetric line placed at the top left corner of the film. Loading and unloading were simulated using displacement control. Contact between the indenter and film was frictionless and the large number of elements in contact ensured convergence of $P-h$ response. The analysis was conducted using the NLGEOM option to accommodate the large deformations induced by the sharp indenter tip. Indentations were modeled to various maximum depths ($h_{\text{max}}$) up to $0.75t$ and subsequently unloaded until the specimen was fully out of contact. Load-depth relations were output from the model, and an Oliver-Pharr analysis [12] was performed on the unloading curves in order to extract the apparent elastic modulus. For the purposes of this study, measured elastic modulus is shown as a function of normalized indentation depth ($h_{\text{max}}/t$).
Figure 2-1: A) Schematic representation of single-splat indentation using a Berkovich indenter tip. B) Shows the axisymmetric mesh implemented for the computational model. Axisymmetric BCs were applied to the left face, and roller BCs to the bottom face. The film thickness was defined as $t$ and the substrate thickness was $50t$. 
2.4. RESULTS AND DISCUSSION

2.4.1. Hard Film on Soft Substrate - The Effect of Plastic Anisotropy

Figure 2-2 shows calculated modulus as a result of normalized depth, for hard films on soft substrates, with different anisotropy values, as well as experimental data. A1 and A2 in Fig. 2-2 represent different cases of input anisotropic plasticity, with the latter being more extreme than the former. Similar to the trend observed in Ref. [73], modulus decreases significantly with depth. Anisotropy significantly affects modulus values, with the lowest trend in modulus appearing for the 40F case and highest anisotropy. However, it is noteworthy that for the harder (40F) case, the extent of anisotropy does not appear to have a significant effect. This is not as apparent for case 20F, in which differences in resolved modulus (i) increase with increasing depth and (ii) exhibit significant differences. Anisotropy combined with hard film/soft substrate combination seems to approach experimental values of modulus decrease.

The modulus decrease for a hard film/soft substrate combination was attributed to a ‘plastic hinge’ in the substrate, at greater depths. That is to say, plastic deformation is nearly suppressed in the film, and primarily occurs in the substrate, placing the film into a state of bending, rather than compression typically associated with indentation. Thus, during elastic unloading, the response is far more compliant than expected. Plastic anisotropy enhances this effect (Fig. 2-2), as it further suppresses plasticity due to the primary modes of indentation, compression and shear. Finally, it is notable that whereas for the isotropic hard film/soft substrate cases, the modulus decrease does not appear until an indentation depth of $h_{max}/t = 0.25$, when anisotropy is introduced the decrease begins far earlier. However, the $P-h$ responses in these cases exhibited a strong inflection point, due to the sudden increase in compliance.
associated with substrate yielding. To address this issue, cases of soft films on harder substrates were explored.

The $P-h$ curves were matched to a certain extent when cases of extreme anisotropy in both the indentation direction and the shear direction, as well as a high rate of strain-hardening were used as inputs. The case that was best found to match was a 40F case with a hardening slope of 45% of the Elastic Modulus and the following values for $R_{11}$, $R_{22}$, $R_{33}$, $R_{12}$, $R_{13}$, and $R_{23}$: 1, 7, 1, 0.275, 1, and 1, respectively. The $P-h$ curve, with the experimental results overlaid, is highlighted in Figure 2-3. While the loading curve was matched nearly perfectly, the unloading slope was significantly higher in the computational case than in the experiments. Therefore, the resolved elastic modulus was 114.50 GPa which is significantly lower than the input 200 GPa but not quite to the point of the experimentally-observed drop.

![Figure 2-2: Modulus decrease (input $E = 200$ GPa) for hard films on soft substrates with varying degrees of plastic anisotropic behavior. Shows that increasing the material hardness and inputting anisotropic plastic behavior forces the resolved modulus to decrease with indentation depth similar to the experimentally observed results.](image-url)
2.4.2. Soft Films - The Effect of Plastic Anisotropy

Figure 2-4 shows a typical $P-h$ response for a case of a soft film on a harder substrate, in which low initial yield strength was followed by significant hardening behavior (20% of $E$). The anisotropic plasticity in this case is defined by $R_{11}$, $R_{22}$, $R_{33}$, $R_{12}$, $R_{13}$, and $R_{23}$: 1, 3, 1, 3, 1, and 1, respectively. It is apparent that any inflection point is suppressed, and loading response maintains a positive curvature. The elastic modulus in this case was calculated to be 89.28 GPa at a maximum depth of 0.60 µm, which while still higher, approaches that of the experimental value at this depth.
Figure 2-4: $P-h$ response of the above mentioned case for a soft film on a hard substrate. Note that the shape of the curve shows no inflection during loading.

Figure 2-5 shows modulus decrease with increasing indentation depth, for the isotropic and anisotropic soft film/hard substrate models. Note first that the decrease is far larger when plastic anisotropy is present, to the extent where one could state that in this case, anisotropy is necessary for any significant decrease. Second, the decrease appears for both cases almost immediately, and modulus does not exhibit a constant value for initial stages of loading, as in the hard film/soft substrate cases. Third, the modulus decrease tends toward a plateau, rather than a continuous drop, and even appears to reverse slightly at deeper depths, for the isotropic case. These characteristics are similar to those of the experimental results, even though decrease is not as significant. One other point to note, however, is that the modulus decrease begins earlier than the experimental case (it has dropped to 150 GPa at the lowest depths). This is a result of the
high hardening, and could be addressed by considering delayed hardening, that is to say, a short region of perfectly plastic behavior before an increase in stress-strain slope. Other models run with much higher hardening behavior (slope = 0.30E), exhibited further modulus decrease, but as $P-h$ curves showed inflection they were not pursued further in this study.

![Figure 2-5: $E$ vs. $h_{\text{max}}/t$ for the above case with both isotropic and anisotropic plastic properties as inputs. The x-axis is normalized with respect to film thickness.](image)

The modulus decrease with depth for these input cases were not observed for homogeneous models of the same film material input. Thus, it can be assumed that this is an artifact of the film-substrate combination. The interpretation of this effect is that the initial low yield strength of the film, in addition to the finite film thickness, strongly reduces the self-similarity of the sharp indenter problem. As the indenter approaches the substrate, the
representative shear-strain increases, as the stronger substrate resists yielding and thus constrains the material near the tip (Fig. 2-6). This increased shear strain is associated with a far higher stress, due to the high hardening slope, and thus the indentation unloading is more ‘elastic.’ The increased shear strain is a direct result of the proximity of the tip to the substrate, and therefore the modulus decrease is a function of indentation depth.

![Figure 2-6: Contours of plastic strain for the isotropic (left) and anisotropic (right) cases compared above for maximum indentation depth of h/t = 0.50. Note that in both cases, plastic strain in the substrate is significantly larger than in the film. This effect is larger when anisotropy is present, in which yielding is suppressed due to substrate constraint and plastic anisotropy.](image)

2.5 CONCLUSIONS AND IMPLICATIONS

Following this modeling study, there are further points for discussion. Perhaps paramount, the plastic behavior, within physically realistic degrees, can dramatically influence the apparent modulus that is extracted via the Oliver-Pharr method, either due to the ‘plastic hinge’ described in [73] or via constraint of a highly work-hardening material with anisotropic plastic properties, as described here. In the former case, such a decrease would perhaps be anticipated, given the strong deviation of P-h behavior from Kick’s law which describes P to be
proportional to \(h^2\). In the latter case, indentation loading behavior is more typical, and does not by itself suggest that any large artifact in modulus would occur. From a tribological standpoint, it is one aim of this work to draw attention to the fact that differences in plastic behavior, for thin film or multilayer systems, can lead to errors in elastic modulus measurement through indentation, beyond those associated with pile-up and sink-in for homogeneous materials. It should also be mentioned that in similar soft film-hard substrate models, different slopes of strain-hardening (slope \(0.05E - 0.40E\)) were attempted, which led to varying levels of modulus decrease. However, for the most part, the largest we observed was \(\approx 60\%\). However, the mechanism responsible for the decrease remained similar in all cases.

In this study, it was shown via FEM investigations, that significant artifactual decreases in indentation modulus could occur for film-substrate combinations in which no elastic mismatch was provided as input. In all cases, this decrease was exacerbated via the introduction of anisotropic plastic behavior, specifically the relative strengthening of the film in the normal and out-of-plane shear directions. The modulus drop was attributed to a more ‘elastic’ response of the film, i.e. suppression of yielding, which in turn affected the unloading slope. For the harder films on soft substrates, the suppression of yielding led to a bend load placed on the film, strongly increasing compliance. For soft films on hard substrates, a significant hardening, coupled with a depth-dependent constraint from the substrate, led to this effect.

Furthermore, the assumptions of input properties were made based on the microstructure of sprayed coatings, and the results are consistent with the coating microstructures. Perhaps the most significant finding of this study is that it can now be suggested that plastic anisotropy exists in coatings and emphasizes the importance of characterizing such properties in order to account for their manifestations in widely-used experimental protocols for material property assessment.
CHAPTER 3. STRESS-STRAIN BEHAVIOR OF MATERIALS EXHIBITING TRANSVERSE ISOTROPIC PLASTIC PROPERTIES
3.1. INTRODUCTION

The goal of this work was to determine whether information on the anisotropic plastic material properties of a material could be determined through the use of stress-strain response, as predicted by Tabor, induced by contact with a rigid indenter tip of different geometries. This work was conducted under the ideology that if the stress response of materials with different degrees of plastic anisotropy were examined at the same indentation depths (i.e. strains), there would be a predictable difference in results. As an example, if results were of a monotonic nature, prediction of anisotropic plastic behavior would be possible through examination of the stress-strain relation. Furthermore, analysis was conducted by performing virtual scratch tests on the same materials to observe whether results showed a predictable trend.

3.2. COMPUTATIONAL METHODS

3.2.1. Simulation of Spherical Indentation and Extraction of Properties

Spherical indentation was modeled by means of an analytical rigid quarter circle with radius $R$, while the dimensions of the specimen were $15R \times 15R$. The large dimensions of the specimen compared with those of the indenter assured that there were no effects due to boundary conditions. An axisymmetric BC was applied to the left point of the indenter so as to maintain consistency with the specimen BCs. Appropriate BCs were applied for axisymmetric analysis; roller BCs were applied to the left and the bottom faces of the displayed mesh, while the left face was unconstrained. Indentation loading was simulated using displacement control, and each simulation was taken to a maximum depth of $0.25R$. A coefficient of friction of 0.15 [76] was implemented between the rigid indenter and deformable body, and the large number of elements in contact ensured convergence of results. The analysis was conducted using the NLGEOM...
option to accommodate the large deformations induced by the indenter. Figure 3-1 shows the axisymmetric mesh that was applied to the specimen. A high-density mesh was applied in the vicinity of the indenter tip to ensure accuracy and convergence of results while limiting contribution from far-field boundary conditions (BCs). A mesh convergence study was conducted to further confirm numerical accuracy of results. A total of 12,100 four-node, bilinear quadrilateral elements were used throughout the analysis. A uniform mesh was applied to the top-left region under the indenter while a gradually coarser mesh was applied to regions that were further away. Models with elastic material properties were compared with Hertz contact theory \[77\] prior to analysis in order to guarantee accuracy of the results and ensure convergence of the meshing. For a spherical contact of an elastic material, Hertz contact theory gives the following relationship between indentation load \((P)\) and indentation depth \((h)\) for contact of an elastic sphere with an elastic half-space:

\[
P = \frac{4}{3} E^* R^{1/2} h^{3/2}
\]

(3-1)

where \(R\) is the radius of the spherical indenter tip, and \(E^*\) is the effective elastic modulus of the contact. \(E^*\) is given by the relation:

\[
\frac{1}{E^*} = \frac{1-v_1^2}{E_1} + \frac{1-v_2^2}{E_2}
\]

(3-2)

where \(E_1\) and \(E_2\) are the elastic modulus of the two bodies in contact, and \(v_1\) and \(v_2\) are the corresponding Poisson's ratios of the materials. When modeling indentation, or in the case of experimental indentation, the indenter tip is assumed rigid compared with the indented specimen and therefore, the condition is imposed that \(E_1 > E_2\); effectively eliminating one of the terms of
the above equation. Therefore, the effective modulus of the indented specimen need only be considered.

For this study, the Elastic Modulus ($E_0$), Poisson’s Ratio ($\nu$), and Yield Stress ($\sigma_y$) of the specimen were kept constant at 100 GPa, 0.30, and 100 MPa, respectively. Bilinear stress-strain behavior was adopted; a work-hardening rate ($n$) of $0.02E_0$ was implemented to maintain consistency with typical metals. A state of transverse isotropic plasticity was employed using the POTENTIAL sub-option in ABAQUS by maintaining all R-values at unity with the exception of $R_{22}$, corresponding to $\sigma_y$ in the indentation direction, which was varied from 0.50 to 1.50.
Stress and strain were calculated according to Tabor’s definitions [78] of average indentation pressure \( p \) and strain \( \varepsilon \):

\[
p = \frac{P}{2.8\pi a^2}
\]

\[
\varepsilon = 0.20 \frac{a}{R}
\]

where \( P \) is the indentation load, \( a \) is the contact radius, and \( R \) is the radius of the spherical indenter. Average pressure was plotted as a function of \( R_{22}/R_{11} \) at different values of strain (4\%, 6\%, 8\%, and 10\%) and was normalized with respect to maximum stress. Contributions of work hardening to Tabor’s equations were neglected due to the comparative nature of the study, as \( n \) was held constant. Load-Depth \((P-h)\) curves and plastic strain contours \((PEMAG)\) were extracted from the models. To maintain consistency in the analysis, all of the plastic strain contours were analyzed at 6\% indentation \((\text{Tabor})\) strain.

3.2.2. Simulation of Conical Indentation and Extraction of Properties

For the purpose of comparison, the spherical indenter in the above model was replaced by an axisymmetric conical indenter of angle 70.3° from vertical, which has been shown to accurately model 3D conical indentation [75]. The material properties and meshing of the specimen were left unchanged so results could be compared. The simulations were taken to the same maximum depth as in the spherical case, and similarly, \( P-h \) curves were extracted. The plastic strain contours were plotted at maximum depth. For conical indentation, the strain is completely dependent on the indentation depth due to self-similarity of the cone geometry. Namely, the primary characteristic of the cone is the apex angle, and since for experimental indentations this value is predetermined, results no longer depend on the geometry of the indenter.
3.2.3. Examination of the Plastic Strain Fields

The contour plots of the magnitude of plastic strain beneath both the spherical and conical indenter tips were quantified by the aspect ratio of the strain field. In this study, aspect ratio was defined as the vertical distance from the point of contact to the bottom of the strain field divided by the distance between the axis of indentation (Y-axis in the local coordinate system) to the right-most edge of the plastic strain field. The technique is summarized in Figure 3-2. The aspect ratios were plotted vs. $R_{22}$ for results beneath both the spherical and conical indenter tips.

![Figure 3-2: Schematic showing the methodology used to calculate the aspect ratio of the plastic strain field. For this study, aspect ratio is defined as the ratio between the vertical distance to the bottom of the plastic strain field to the horizontal distance to the right edge of the plastic strain field.](image)
3.2.4. Simulation of Scratch Tests on Materials with Transverse Isotropic Plastic Properties

A three-dimensional FEM simulation (ABAQUS/Standard) of a mechanical scratch test was employed to provide insight to the scratch response of materials with varying degrees of out-of-plane plastic anisotropy. The material used throughout this portion of the study was given constant linear-elastic properties \( (E = 300 \text{ GPa}, \nu = 0.3) \) and the input \( \sigma_Y \) was held constant at 600 MPa \( (\sigma_Y/E = 1/500) \). A linear work-hardening behavior was input as in the aforementioned models \( (n = 2\% \text{ of } E) \). Anisotropic plastic behavior was input by varying the value of \( R_{22} \) while the other values were maintained at unity.

The material was scratched with an analytical rigid Rockwell C indenter, with a tip radius of 200 \( \mu \text{m} \). The width of the sample was modified so that the 3D model could accurately represent the semi-infinite case. A mesh convergence study was conducted, and the final mesh consisted of 173,000 6-noded wedge (C3D6) elements. The mesh, dimensions, and coordinate system are highlighted in Figure 3-3. A fine mesh was used in the vicinity of the scratch and was gradually coarsened in regions sufficiently distant from the region of interest. Displacement of the bottom face was fixed in all directions, and symmetric BCs were applied to the face labeled A in Fig. 3-3, i.e. displacement was constrained only in the direction normal to face A. Frictionless contact was defined so as to simulate a lubricated scratch test. The indenter tip was first lowered into the material to a maximum depth of 5 \( \mu \text{m} \) and subsequently dragged 500 \( \mu \text{m} \) across the surface of the sample (in the 1-direction). Residual depth profiles (2-3 plane) for each case of plastic anisotropy were plotted for the same set of nodes for comparison, and the height of residual pile-up was analyzed to compare with the stress-strain behavior from the previous models.
3.2.5. Analytical Examination of Hill (1948) Yield Criterion and Its Implications

Although Hill does not directly describe the theory of three-dimensional indentation with conical and spherical indenter tips, similarities may be extracted from his theoretical analysis of plane-strain indentation of a block of metal with a flat, rigid die. This is the type of contact loading expected in, for example, deep drawing of cups from metal sheet. The average pressure beneath the die under the plane-strain assumption is given by the following set of equations [79]:

\[
p_0 = T \left( \frac{1 - c}{1 - c \sin^2 2\gamma} \right)^{\frac{1}{2}}
\]

\[
c = 1 - \frac{N(F+G)}{2(FG+GH+HF)} \quad (-\infty < c \leq 1)
\]

(3-5)

where \(c\) is a lumped constant describing the state of plastic anisotropy (F, G, and H are defined in Section 1.6.: Plastic Anisotropy In Commercial Finite Element Codes), \(p_0\) is the average pressure beneath the die, \(T\) is the yield stress in shear, and \(\gamma\) is the angle between the x-axis of
anisotropy (1-direction) and the indented surface. In this study, to remain consistent with the cases of plastic anisotropy examined, $\gamma$ was set to $0^\circ$.

Values of $N$, $F$, $G$, $H$ were chosen to represent the cases of transverse isotropic plastic properties examined throughout the other sections of the study. The corresponding values of $c$, $T$, and $p_0$ were calculated and the analytical solution for $p_0$ was plotted for different values of $R_{22}/R_{11}$ for comparison with the results from the indentation and scratch test simulations.

3.3. RESULTS

3.3.1. Spherical Indentation of a Material with Transverse Isotropic Plastic Properties

As shown in Figure 3-4, the value of the ratio of $R_{22}$ to $R_{11}$ has a significant effect on the average pressure beneath a spherical indenter tip. Four cases are shown (4%, 6%, 8%, and 10% Tabor strain) and the same trend is apparent in all cases. The average pressure is normalized with respect to the maximum pressure (i.e. when $R_{22}/R_{11} = 0.50$) at each strain. There is a clear inflection point in every case at $R_{22}/R_{11} \approx 0.70$. The curves for 6%, 8%, and 10% strain are coincident, while in the 4% case the difference between the average pressure when $R_{22}/R_{11} = 0.50$ and the other ratios is less dramatic.

Figure 3-5 shows the load-depth curves for certain cases of transverse plastic anisotropy. It is evident that variation of the $R$-value in the indentation direction has a significant effect on the indentation hardness. As expected from the results shown in Figure 7, the hardness is greatest in the case where $R_{22}/R_{11} = 0.50$ and lowest in the case where $R_{22}/R_{11} = 0.70$. It is also important to note that the overall hardness of the material also shows an inflection point when $R_{22}/R_{11} = 0.70$. 
Figure 3-4: Shows computational results of the dependence of normalized average pressure beneath a spherical indenter tip at four different values of Tabor strain ($\varepsilon = 0.2a/R$).

Figure 3-5: Load-Depth curves of different inputs of transverse isotropic plasticity beneath a spherical indenter. Depth is normalized with respect to the radius of the indenter tip and load is normalized with respect to maximum load in the case of $R_{22}/R_{11}=0.50$. 
Another characteristic of the specimen that is highly sensitive to the out-of-plane yield stress in the case of transverse plastic anisotropy is the plastic strain field beneath the indenter. Plots of the plastic strain fields beneath the indenter are shown for the same depth in Figure 3-6. Upon inspection, a definitive pattern can be observed in the shapes. When \( R_{22}/R_{11} = 0.50 \), the shape of the strain field is more rectangular while at \( R_{22}/R_{11} = 0.60 \) and 0.70, the field is more round. As the \( R_{22}/R_{11} \) ratio begins to approach and surpass the state of isotropic plasticity (i.e. \( R_{11} = R_{22} = R_{33} = 1 \)), the trend suggests that the plastic strain field is again approaching a rectangular form. Another interesting aspect of the plastic strain fields is that in the cases of Fig. 3-6A, E, and F, a large degree of straining occurs in near the right edge of the contact. These results resemble the behavior of a sample that is subjected to flat-punch indentation; in which a flat cylinder is pressed into the specimen.

Figure 3-6: Plots of the plastic strain fields beneath the spherical indenter at 6% Tabor strain for different ratios of \( R_{22}/R_{11} \). A) \( R_{22}/R_{11} = 0.50 \) B) \( R_{22}/R_{11} = 0.60 \), C) \( R_{22}/R_{11} = 0.70 \), D) \( R_{22}/R_{11} = 0.80 \), E) \( R_{22}/R_{11} = 1.00 \), F) \( R_{22}/R_{11} = 1.50 \).
3.3.2. Conical Indentation of a Material with Transverse Isotropic Plastic Properties

Figure 3-7 shows the relation between indentation load and the ratio between the out-of-plane and in-plane yield stress ratios for a conical tip. All indentation loads were taken at the same indentation depth and the indentation load was normalized with respect to the maximum indentation load, which occurred at $R_{22}/R_{11} = 0.50$. The trend in Fig. 3-7 follows the same as that shown in Fig. 3-4. However, the difference between the maximum and minimum loads during conical indentation is less than the difference between the maximum and minimum average pressure beneath a spherical indenter.

![Figure 3-7: The relation between normalized indentation load and $R_{22}/R_{11}$ at an indentation depth of 500 μm. Results are normalized with respect to the maximum indentation load ($R_{22}/R_{11} = 0.50$). A clear minimum occurs at $R_{22}/R_{11} \approx 0.70$.](image)
The $P-h$ curves resulting from conical indentation are shown in Figure 3-8 for different inputs of transverse plastic anisotropy. Reflective of the plot shown in Fig. 3-5, the hardness is maximum when $R_{22}/R_{11} = 0.50$ and minimum when $R_{22}/R_{11} = 0.70$. The models for both the conical and spherical indenter tips showed the same trends in indentation hardness. As in the spherical case, the dependence of the shape of the plastic strain field beneath the conical tip and the $R_{22}/R_{11}$ ratio was significant. The plastic strain fields for different values of $R_{22}/R_{11}$ are presented in Figure 3-9. All images were taken at the maximum depth of 500 μm.

![Figure 3-8: The load-depth curves for different $R_{22}/R_{11}$ ratios in response to conical indentation. The indentation load is normalized with respect to maximum load when $R_{22}/R_{11} = 0.50$ and the depth is normalized with respect to the maximum indentation depth.](image)
3.3.3. Relation Between the Plastic Strain Field Aspect Ratio and the Ratio of In-plane to Out-of-plane Yield Stresses

The shape of the plastic strain field was quantified through the method described in Section 3.2.3. Examination of the Plastic Strain Fields so that a relationship between the aspect ratio and yield stress ratio could be established. To do so, this method was implemented for many cases of $R_{22}/R_{11}$ and a plot of aspect ratio vs. $R_{22}/R_{11}$ for indentation loading with both a spherical and conical tip is shown in Figure 3-10. While the results for both tip geometries seem to follow the same trend, the aspect ratios are slightly greater in the spherical case for all $R_{22}/R_{11}$.
values. It is recognized that the behavior shown in the plots in Fig. 3-10 is quite consistent with those in Figs. 3-4 and 3-7.

Figure 3-10: Shows the dependence of the aspect ratio of the plastic strain field on the value of $R_{22}/R_{11}$ for both spherical and conical indenter tip geometries. It is evident that the aspect ratio is highly sensitive to the ratio $R_{22}/R_{11}$.

3.3.4. Examination of Varying Degrees of Residual Deformation Resulting from Scratch Testing

Figure 3-11 shows the residual depth profiles following scratching as extracted from the Finite Element model. Fig. 3-11A shows a schematic of the residual scratch profile as seen in the model with a deformation scale factor of 5 applied in the 2-direction to accentuate the deformation pattern. Fig. 3-11B-H show plots of coordinates for different ratios of $R_{22}/R_{11}$. There is a dependence of residual pile-up upon the yield stress ratio of the material that is
consistent with results from the other studies presented here. That is to say, the level of pile-up is maximum in the case of $R_{22}/R_{11} = 0.70$ and minimum in the case of $R_{22}/R_{11} = 0.50$.

Figure 3-11: Residual depth profiles resulting from the FEM of a mechanical scratch test. A) Schematic of the residual deformation following scratch and the planar-directions of the plots. B) $R_{22}/R_{11} = 0.50$, C) $R_{22}/R_{11} = 0.60$, D) $R_{22}/R_{11} = 0.70$, E) $R_{22}/R_{11} = 1.00$, F) $R_{22}/R_{11} = 1.20$, G) $R_{22}/R_{11} = 1.50$, H) $R_{22}/R_{11} = 2.00$. Axis units in B-H are in microns and each case shows the height of pile-up post-scratch. The dashed plots in B-H show the geometry of the Rockwell C tip.
3.3.5. Comparison of Analytical Solution with Simulation Results

The plane-strain solution to average pressure described in Section 3.2.5. *Analytical Examination of Hill (1948) Yield Criterion and Its Implications*, as well as the results from spherical and conical indentation, and the residual pile-up resulting from the mechanical scratch test are plotted in Figure 3-12. All results are normalized with respect to the isotropic case ($R_{11} = R_{22} = R_{33} = 1$). The inverse of the pile-up height was presented due to the fact that a lesser-degree shows an increased material hardness. The results from all sections of the study follow the analytical trend quite nicely, showing agreement between the results and theory.

![Figure 3-12: Plot of the analytical solution for average pressure beneath a flat, rigid die compared with the pressure results from the simulations of conical and spherical indentation, and the inverse of mechanical scratch test pile-up height. All results are normalized with the isotropic case.](image)
3.4. DISCUSSION AND IMPLICATIONS

The results presented suggest that materials with transversely isotropic plastic properties in which the in-plane yield properties are equivalent and different from the out-of-plane yield properties show interesting behavior when subjected to contact loading via a rigid body. When all results presented in this study are considered, it is clear that this behavior is consistent through instrumented indentation with a sphere, cone, and even through a mechanical scratch test.

When looking at the trends observed in the average pressure beneath a spherical indenter and the reaction load during conical indentation of materials with different out-of-plane yield properties, interesting and more importantly, consistent trends have been observed. In all cases, there is a minimum in overall "hardness" of the material when $R_{22}/R_{11} \approx 0.70$ and unpredictably, a maximum when $R_{22}/R_{11} = 0.50$, suggesting that although the yield strength in-plane is double that in the loading direction, the resistance to plastic deformation (or load-bearing capability) in the direction normal to the plane is higher. It is also important to note that this phenomenon is not depth-dependent; namely the load-displacement behavior presented in Figs. 3-5 and 3-8 show that after the material has plastically deformed, at any particular depth, the indentation hardness of the material with $R_{22}/R_{11} = 0.50$ is maximum and is minimum when $R_{22}/R_{11} \approx 0.70$.

This trend is unique to anisotropic plasticity, and is not observed when varying the degree of elastic anisotropy in the same ratios. In such cases, the stiffness of the material in response to contact loading increases consistently with the value of $E_{22}/E_{11}$, suggesting that the plastic strains in the material are the core cause of the behavior. The shapes of the plastic strain fields during loading with both a spherical and conical indenter tip shown in Figs. 3-6 and 3-9, respectively, are quite reflective of the differences between hardness of the material. When looking at the
plastic strain fields when $R_{22}/R_{11} = 0.50$, the shape is square and quite different from the round shape observed when $R_{22}/R_{11} = 0.60$ and thus, the hardness in these cases are also quite different. However, in the cases of $R_{22}/R_{11} = 0.60, 0.70, 0.80$ the shapes of the fields are quite similar and consequently, the differences observed in pressure between these cases are minute. As the ratio $R_{22}/R_{11}$ increases further beyond these values, the shape of the plastic strain field tends toward the square shape observed when the ratio is 0.50 and therefore, the indentation pressure also approaches the value obtained when $R_{22}/R_{11} = 0.50$.

This concept is visualized in Fig. 3-10 in that the aspect ratios of the plastic strain fields in both the spherical and conical loading cases follow the same dependence on $R_{22}/R_{11}$ as was seen in the average indentation pressures. However, one can see that although the contact regime is different with the different tip geometries, the magnitudes of the aspect ratios are similar and the same trend is observed. In the case of the scratch test (Fig. 3-11), the degree of residual pile-up suggests that the level of "plasticity" is highest when $R_{22}/R_{11} = 0.70$ and lowest (more elastic) when $R_{22}/R_{11} = 0.50$. In fact, the sink-in observed in the latter case can be attributed to one of two parameters: (i) a high degree of elastic behavior beneath the indenter [80], or (ii) a large degree of work-hardening occurring in response to the loading [81]. The work-hardening rate for all models throughout this study is constant; signifying that there is another underlying aspect constraining plastic deformation due to contact loading in a way which leads to material response suggestive of high work-hardening.

Figure 3-13 and 3-14 show the material flow vectors during loading with a conical and spherical indenter, respectively. The black arrows represent the nodal deformation directions in the material. When $R_{22}/R_{11} = 0.50$ (Fig. 3-13A & 3-14A), the tendency of the material is to flow downward in the region directly below the indenter tip, and the plots show that there is a
constraint on flow in the in-plane direction. As the yield stress ratio is increased toward the isotropic case, this constraint is removed, and the material can flow freely away from the contact region. Again, as $R_{22}/R_{11}$ is increased to values greater than 1, the flow becomes more and more constrained in the in-plane direction. In the case of spherical indentation, the flow fields show a tendency of the material to "curl" upwards at the edge of contact, which is the factor leading to the similarity of the plastic strain fields to that of a flat punch indentation. Turning our attention back to the conical flow fields, the behavior observed is consistent with the trends encountered when looking at average pressure, and we turn the discussion to the true meaning of the Lankford coefficient to explain this phenomenon.

The Lankford coefficient is defined in terms of plastic strains through the relation:

$$\bar{\tau} = \frac{e_{p,w}}{e_{p,t}},$$

the ratio between the plastic strains in the width direction and through-thickness directions in a material [19]. In the case of transversely isotropic plastic properties, as implemented in this study, in which the in-plane plastic properties are equivalent and differing from the through thickness plastic properties, the Lankford coefficient is related to $R_{22}$ through the relation: $\bar{\tau} = 2R_{22}^2 - 1$. From the relation, it is seen that the value of the Lankford Coefficient is 0 when $R_{22}/R_{11} \approx 0.707$, negative if $R_{22}/R_{11} < 0.707$, and positive for $R_{22}/R_{11} > 0.707$. If a Lankford coefficient is negative, it is mathematically suggested that if a positive strain is induced in the thickness direction through compressive loading, the in-plane strains will be negative; with a tendency to pin material within the contact region.
Figure 3-13: Shows the material flow direction beneath the conical tip for A) $R_{22}/R_{11} = 0.50$ B) $R_{22}/R_{11} = 0.60$, C) $R_{22}/R_{11} = 0.70$, D) $R_{22}/R_{11} = 0.80$, E) $R_{22}/R_{11} = 1.00$, F) $R_{22}/R_{11} = 1.50$. 
Figure 3-14: Shows the material flow direction beneath the spherical tip for A) $R_{22}/R_{11} = 0.50$ B) $R_{22}/R_{11} = 0.60$, C) $R_{22}/R_{11} = 0.70$, D) $R_{22}/R_{11} = 0.80$, E) $R_{22}/R_{11} = 1.00$, F) $R_{22}/R_{11} = 1.50$. 
3.5. CONCLUSIONS

The results presented in this chapter drew attention to the fact that examination of stress-strain relations of materials exhibiting transverse isotropic plastic properties, with out-of-plane yield properties differing from in-plane, are inconclusive. Therefore, tracking the load-depth or stress-strain relations will be inconclusive when attempting to characterize such materials, when applying Hill's 1948 yield criterion for general stress states. There is a strong inflection point observed in both indentation hardness and stress-strain behavior, leading to identical results for specific states which lie to the left ($R_{22}/R_{11} < 0.707$) and their images falling to the right ($R_{22}/R_{11} > 0.707$).

Upon further inspection of the problem, it is suggested that more valuable information may be acquired through examination of material flow in the vicinity of contact. Perhaps, more importantly, tests that are designed to allow free flowing of the material could be advantageous in characterizing anisotropy in the *plastic* properties. Results suggest that in cases in which anisotropic plastic properties are prescribed, there are inherent constraints in the plastic deformation/flow of the material resulting from contact, which leads to unexpected mechanical behavior. Consequently, for the remainder of this study, experiments were designed around the basis that mechanical constraints would be minimized on the sample during testing; free-flow of material is preferred and inherently established.
CHAPTER 4. MINIMAL-CONSTRAINT
INDENTATION TESTING OF
PLASTICALLY ANISOTROPIC
MATERIALS
4.1. INTRODUCTION

In the previous chapter, it was discussed that the best way to extract anisotropic plastic material properties from a sample is to focus on the constraint of material flow in the vicinity of contact. With this aim, it is crucial to design contact test protocol in which free-flow is encouraged; with minimal mechanical constraints being imposed on the sample. Perhaps the most promising candidate for such a test is indentation near a free-surface; one that is not fixed locally in any of the orthogonal directions. Such a design will allow for material to flow naturally, leaving the only constraints present those imposed intrinsically by the state of anisotropic plasticity.

The effect of indentation near a free edge was examined in a recent study by Jakes et al. entitled "The Edge Effect in Nanoindention" [82]. The aim of the study was to quantify the experimental artifacts associated with the free edge by introducing a structural compliance. Similar to the standard machine compliance that must be examined prior to using a mechanical tester, the structural compliance does not depend on the indenter geometry; it is a function of the position with respect to the edge. The results in indentation hardness and load-depth relation will begin to deviate from the semi-infinite case when the plastic zone induced by contact reaches the free surface. Conceivably, these errors are brought upon by the fact that once the plastic zone reaches the unconstrained surface, the material will have an augmented ability to escape the region of contact. Thus, a higher drop in indentation hardness will be observed with decreasing distance from the free edge. Furthermore, it was shown that in samples with a higher Poisson's ratio, the effect was amplified; validating the assumption that indentation near a free edge is an ideal candidate for allowing free flow of the material in the contact region and consequently, eliminates mechanical constraints during sample testing.
The two regimes that will be examined in this work are (i) indentation near a free edge of a sample and (ii) indentation on the corner of a sample. These two experiments are represented schematically in Figure 4-1. The ideology used here is that in case (i), the indentation will be conducted near one free surface while in case (ii) there will be two free surfaces. In other words, the sample in case (i) will be more constrained than that in case (ii). Another advantage of such an experimental protocol is that it allows for examination of the semi-infinite indentation results on the same sample. This allows for normalization of the edge and corner data so that effects of other material properties such as elastic modulus, yield stress, work-hardening rate, and Poisson's ratio can be removed from the analysis. Therefore, the effects of anisotropic plastic properties can be uniquely examined. Fig. 4-1A shows the problem of indentation near the edge and 4-1B shows indentation near the corner. The use of a conical indenter in this case eliminates the problem of indenter tip geometry as the self-similarity of a conical geometry is exploited.

Figure 4-1: Schematic representation of A) edge indentation vs. semi-infinite indentation and B) corner indentation vs. semi-infinite indentation.
4.2. COMPUTATIONAL METHODS

4.2.1. Indentation Near a Single Free Surface

To examine the effect of anisotropic plastic properties on the material response to indentation near a free edge, a half-symmetry model was developed using ABAQUS/CAE. A picture of the model in its complete form, the implemented mesh, and the coordinate system used in this study are highlighted in Figure 4-2. Due to the geometry being modeled, one plane of symmetry was implemented to conserve computational resources so that a wide-scale set of models could be analyzed. Roller boundary conditions (BCs) were applied to the plane of symmetry; displacement normal to the surface was fixed. Displacement in the 2-direction fixed for the bottom face to counteract the forces applied by the indenter tip. No BCs were prescribed for the face adjacent to the plane of symmetry as this would serve as the free edge in the model. The specimen was modeled as deformable with a bilinear stress-strain response while the indenter was modeled as a discrete rigid shell with a half angle of 70.3°.

Figure 4-2: Shows the mesh and geometry used in the finite element simulation of indentation near a free edge. The plane of symmetry, free edge, and coordinate system are presented as defined.
A total of 56,000 eight-noded, linear hexahedral elements (type C3D8R) were assigned to the elastic-plastic solid and 270 linear quadrilateral elements (type R3D4) were used for the discrete rigid indenter. The number of elements was chosen following a complete mesh convergence study to ensure accuracy of the results, however, since such a study had never been conducted, minimal data was available for comparison to results. Due to the comparative nature of this study, convergence of results with increasing element density was deemed sufficient. For the conical geometry of the indenter, a mesh was chosen to provide a smooth geometry for the rigid shell so as to accurately simulate conical indentation. As shown in Fig. 4-2, a fine mesh was applied directly beneath the indenter, and a gradually coarsened mesh was applied sufficiently far from the region of interest.

As the specimen was modeled as elastic-plastic and the effect of anisotropic plastic properties was to be examined, all other properties were kept constant. The elastic modulus (E), Poisson's ratio, base-line yield stress (σ_Y), and work-hardening slope were held at 100 GPa, 0.30, 100 MPa, and 0.02E, respectively. For completeness, however, different values of σ_Y/E (1/250 and 1/500) were also attempted. A state of transverse isotropic plasticity was employed using the POTENTIAL sub-option in ABAQUS by maintaining all R-values at unity with the exception of R_{22}, corresponding to σ_Y in the indentation direction, which was varied from 0.60 to 1.50.

In all cases, the maximum indentation depth (h_{max}) was taken as d (5 mm) and the distance from the edge was set to 0.5d, d, and 2d. A semi-infinite indentation case was also modeled for the purpose of comparison with the proximity to edge results. This case was modeled as an axisymmetric conical indentation, which has been shown to accurately depict
semi-infinite indentation. A picture of the axisymmetric mesh is shown in Figure 4-3 and the BCs applied were consistent with those described in *Section 3.2.1*.

**Figure 4-3:** The mesh used for the analysis of semi-infinite indentation with a cone. An axisymmetric model was adopted and appropriate BCs were applied. The coordinate system used is also highlighted.

In general, for conical indentation of a uniform and homogenous elastic-plastic solid, the equation for the loading curves are often assumed to be given by: $P = Ch^2$, where $P$ is the indentation load, $h$ is the instantaneous depth, and $C$ is a function of the indenter geometry and material properties and has units consistent with the units of pressure [83]. Dao et al. referred to the parameter $C$ as the "loading curvature" of the indentation curve during sharp indentation [75]. For semi-infinite indentation, the value of $C$ does not vary as a function of depth, however in the case of indentation near a free surface, it is expected that there will be a dependence as the induced strain field grows toward the edge. Therefore, results near the free edge were quantified in terms of "loading curvature", $C$, and were normalized with respect to the constant $C$ observed in the semi-infinite case.
4.2.2. Indentation Near Two Free Surfaces (Corner Indentation)

For simulation of indentation near the corner of a sample, the model described in the previous section was modified. The geometry of the rigid cone was extended to fully model a 3D cone and a sufficiently dense mesh was applied to ensure a smooth surface. The geometry and meshing of the sample were also modified to better accommodate the loading regime. The model used for this problem is shown in Figure 4-4; the sample geometry, meshing, and coordinate system are presented. A total of 39,900 linear hexahedral elements of type C3D8R were used for the elastic-plastic solid and 2242 linear quadrilateral elements of type R3D4 for the conical indenter. Symmetry was not employed for this geometry as the purpose was to examine the effects of two free surfaces in the vicinity of contact. The only BC applied was to the bottom face of the sample; constraining displacements in all normal directions.

Figure 4-4: The geometry and mesh used in the 3D analysis of indentation near the corner. The faces shown are both free, and the coordinate system is also shown.
Differing from the indentation near the edge case where the indenter was placed at different distances from the free edge, in this study the indenter tip was placed at a distance $d$ from the corner and $d$ was equivalent to the maximum indentation depth. The material properties were kept consistent with those described in Section 4.2.1, however only the cases $\sigma_Y/E = 1/1000$ and $1/500$ were considered. In addition, the work-hardening slope was also changed in order to pin-point and account for any difference this parameter may cause in the results. As in the case of edge indentation, the results were quantified in terms of the parameter $C$, and normalized with respect to the semi-infinite cases examined.

4.3. OBSERVATIONS DURING INDENTATION NEAR A FREE EDGE

4.3.1. Observations via Spherical Indentation

Initially, the described model above was used with a spherical indenter to check the dependence of results on indentation with a sphere. The distance from the free edge was gauged as a function of the fixed spherical tip radius $R$. Simulations were carried out for values of $0R$ (directly on edge), $0.5R$, $0.6R$, $0.75R$, $R$, and $10R$ (semi-infinite). Results were analyzed to determine the distance at which the results in indentation hardness no longer showed a dependence on proximity to the free edge. The $P-h$ curves were plotted (Figure 4-5) to a maximum load of 1N and the trends in hardness were observed for materials with different degrees of out-of-plane anisotropy.

For the case of spherical indentation, it was observed that proximity to the edge had a significant effect on the overall indentation hardness of the materials even at small values of normal load (1N). During elastic loading (initial portion of loading curve), indentation response is identical in all cases observed, however when plastic deformation is initiated, the tests
conducted nearer to the free edge show a more significant deviation from the semi-infinite case \((10R)\). The heightened degree of compliance due to the presence of the free edge validates the simulation’s ability to observe the changes in indentation response.

Figure 4-5: Load-depth relations extracted from spherical indentation near the edge of samples with varying degrees of anisotropic plastic properties. In all cases, increased compliance was observed at low loads (1N) in cases in which the sample was indented closer to the edge.
Furthermore, a few other significant points were made apparent in this study. Indentation hardness is a parameter that is directly proportional to the load and inversely proportional to the contact area at any instant. It is unlikely that at the loads applied (<1N), any degree of pile-up or sinking-in would be observed and therefore, the contact area can be assumed constant for a given load in the present cases. Keeping this in mind, we see that the minimal indentation hardness at 0.50R away from the edge was observed in the case of $R_{22}/R_{11} = 0.60$ and monotonically increased with the value of the anisotropy ratio $R_{22}/R_{11}$. Thus, the inflection point that appeared in the stress-strain response of the anisotropic materials at $R_{22}/R_{11} = 0.707$ (see Section 3.4) is eliminated when the sample is no longer subjected to mechanical constraints, and free-flow of material is favored.

To examine the distance from the free edge at which the observed $P-h$ behavior reverts to that in the semi-infinite case, the normalized depth (with respect to depth in the semi-infinite case) at 1N indentation load was tracked as a function of distance from the free edge. The relation is highlighted in Figure 4-6 for $R_{22}/R_{11} = 0.60, 0.70, 1.00, 1.20, \text{and} 1.50$. As a greater depth at a constant load suggests a lower hardness, the plot shows that the lowest hardness (highest indentation compliance) is observed in the case in which indentation was conducted directly on the edge. Also, as discussed previously, these results verify that a minimal hardness is observed when the anisotropy ratio is 0.60, as the depth on the edge vs. the semi-infinite indentation is nearly 3 times greater. According to the analysis, at low indentation loads, any changes in hardness are generally seen when indentation is carried out at a distance less than the radius of the spherical tip; at distance $R$ the depth typically converged to the results in the semi-infinite case.
Figure 4-6: The dependence of indentation depth at a controlled load (1N) on the distance from the free-edge during spherical indentation. Results are normalized with respect to the depth in the semi-infinite case for each value of $R_{22}/R_{11}$.

While it was shown that through spherical indentation near a free-edge, the increase in sample compliance depends on both the proximity to the edge and the state of anisotropy, management of the geometrical parameter $R$ is required. Though distances can be normalized with respect to this parameter, it would be advantageous to design the test in such a way that has no geometric dependence. The use of a conical indenter promotes just that; being that geometric similarity across size scales of cones is preserved. So as to exploit this property, the spherical tip geometry was replaced with a cone for the remainder of the simulations. Granted the apex angle of the cone remains constant, this would allow for the use of a single parameter, the indentation depth $d$, and the placement of the indenter could be set to factors of $d$ away from the free edge for consistency in analysis. With this regime, the geometry of the complex problem is simplified and the primary focus can be shifted to the anisotropic material properties.
4.3.2. Observations via Conical Indentation

The conical indenter was placed at distances of \( d/2 \), \( d \), \( 2d \), and a semi-infinite distance away from the free edge, where \( d \) represents the maximum indentation depth. The material in this case has a \( \sigma_{y}/E \) ratio of 1/1000 and the load-depth curves for these cases are shown in Figure 4-7. As expected, the minimum indentation hardness is observed when the indenter is placed nearest the free edge in all anisotropy cases examined. It is also shown that a significant increase in compliance is observed with respect to the semi-infinite results in all near-edge cases.

Figure 4-7: Load-depth relations during conical indentation at distances of \( d/2 \), \( d \), \( 2d \), and semi-infinite.
Focusing on the case in which the indenter is placed $d$ away from the free-edge, the overall hardness is a minimum when $R_{22}/R_{11} = 0.60$ and a maximum when $R_{22}/R_{11} = 1.50$, as shown in Figure 4-8. Again at this particular distance, the inflection point at $R_{22}/R_{11} = 0.707$ is suppressed and the hardness shows a monotonic dependence on anisotropy ratio. This is the preferred experimental setup as the distance from the edge is equivalent to the maximum indentation depth. Moreover it is apparent that as the indentation depth increases, the difference in hardness is accentuated; in essence there is no clear, unique difference at depths $< 1$ mm however as the depth approaches $d$ the difference becomes progressively more significant. Therefore, with these results in view, it is proposed that monitoring the change in "loading curvature", $C$, of the $P$-$h$ curves will provide a useful measure of the out-of-plane anisotropic plastic properties.

![Figure 4-8: Load-depth relations of materials, with various degrees of anisotropic plastic behavior in the out-of-plane direction, resulting from conical indentation at a distance $d = h_{max}$ away from a free edge.](image)

83
Analysis of the loading curvature ($P/h^2$) as a function of indentation depth for tests conducted at varying factors of $d$ away from the edge is presented in Figure 4-9. As expected, the loading curvature in the semi-infinite case is a constant for all cases of plastic anisotropy.

![Figure 4-9: Drop in loading curvature, $C$, during conical indentation at different factors of $d$ (maximum depth) away from a free edge. The loading curvature was constant in all semi-infinite cases and all results are normalized with respect to the average semi-infinite loading curvature.](image-url)
examined. Thus, all results are normalized with respect to the average loading curvature in the semi-infinite cases. Due to this normalization, the near edge results can be viewed in terms of percent difference from semi-infinite for all cases of anisotropy. As indentation location approached the edge, the curvature tended to decrease significantly to a somewhat plateau as the depth approached $d$.

The extent of the drop is evidently a function of the out-of-plane yield stress ratio as the percent drop observed in all cases showed a strong dependence on this ratio. This concept is visualized specifically by cross-comparing the drop in normalized loading curvature observed in the different cases of anisotropy in a single plot (Figure 4-10). The results show that the significance of the drop is completely dependent on the yield stress ratio, and that the ratio can presumably be determined by the extent of the drop.

![Figure 4-10: Normalized loading curvature during conical indentation at $d$ away from the free edge for different in-plane to out-of-plane yield stress ratios. Results are normalized by the average loading curvature in the semi-infinite case corresponding to each material.](image)
Additionally, the normalization scheme adopted in which the edge results are normalized by the results of semi-infinite indentation on the same sample should make the observed normalized drop a constant even when the $\sigma_Y/E$ ratio is varied. To validate this assumption, values of $\sigma_Y/E$ of 1/250, 1/500, and 1/1000 were compared for materials with different yield stress ratios. These results are summarized for $R_{22}/R_{11} = 0.60, 0.70, 1.00, \text{ and } 1.50$ in Figure 4-11. In general, the results seem to be consistent across all $\sigma_Y/E$ cases examined even though the variation between cases was quite broad. However, from the plots it is observed that the effect of $\sigma_Y/E$ is more pronounced at higher values of $R_{22}/R_{11}$, sometimes to the point where results for a specific case of anisotropy could be incorrectly classified due to an extremely high, or low, $\sigma_Y/E$ ratio.

Figure 4-11: Normalized drop in loading curvature for $\sigma_Y/E$ ratios of 1/250, 1/500, and 1/1000.
To lessen the minimal effect of $\sigma_Y/E$ on the loading curvature drop, a method is sought to accentuate the difference between the results for different anisotropy ratios. To do so, introduction of a second free surface is examined through modification of the experimental protocol. Conical indentation near the corner of the sample is explored in place of near the edge of a sample; the problem of two free surfaces in the vicinity of contact as opposed to a single free surface. It is presumed that the removal of an additional mechanical constraint will alleviate material flow in the contact region further, and can ultimately serve as a better characterization method for the out of plane yield stress ratio $R_{22}/R_{11}$.

4.4. OBSERVATIONS DURING INDENTATION NEAR A CORNER

As predicted, the loading curvature showed a more significant drop during indentation on the corner of the sample than in the case of indentation near the edge. The reason for this phenomenon is in a sense clear; the additional free edge in the contact region leads to a further reduction in the compliance of material. This phenomenon is illustrated in Figure 4-12 which shows the drop in loading curvature, $C$, for different values of $R_{22}/R_{11}$, for a material with $\sigma_Y/E = 1/500$ and a hardening slope equal to 0.02$E$. The results presented in Fig. 4-12 are for the same material presented in Fig. 4-10; the sole difference being that in the case of Fig. 4-10 the sample is indented at a distance $d = h_{\text{max}}$ away from the edge while in Fig. 4-12, the sample is indented a distance $d = h_{\text{max}}$ away from the corner. To clarify the placement of the indenter tip, imagine a line bisecting the corner angle of a sample (45° from each adjacent edge) and the indenter is placed at a location $d$ away from the corner along this line.
A few key points can be observed from Fig. 4-12. First, as mentioned previously, the drop in loading curvature is observed to a much greater extent in the corner indentation case than in the edge case. Second, the differences in drops due to each case of anisotropy in the corner case are more significant and therefore, more reflective of the specific case of anisotropy being tested. This feature better enables the unique determination of the out-of-plane anisotropic plastic property of the material. Furthermore, with the normalization scheme, it has been shown (from the edge indentation case) that results are consistent even when large variations in $\sigma_Y/E$ ratio are considered. Thus, the lone plastic property that needs to be considered and will presumably not be removed from the analysis through the normalization scheme is the degree of
work-hardening of the sample. By indenting the sample near the corner, the degree of plastic
deformation is heightened to a large degree and in addition, the removal of additional mechanical
constraints during testing promotes excessive plastic flow/work. Consequently, the contributions
of the work hardening rate will increase over the semi-infinite case during corner indentation,
and will not be effectively removed by the normalization forcing any derived trends to
incorporate changes in work-hardening rate.

To quantify the effect of hardening on the decrease in loading curvature in the corner
indentation case, bilinear stress-strain behavior was input with post yielding behavior described
by a line of slope $nE$ where $E$ is the slope of the elastic portion of the stress-strain curve. Models
were conducted for cases of $n = 0.02, 0.04, \text{ and } 0.10$, and a dependence of loading curvature
decrease on hardening slope was observed in all cases of anisotropy examined (Figure 4-13). The
least significant drop was observed when $n = 0.10$ and the most significant drop when $n = 0.02$. A nonlinear dependence was observed; the extent of loading curvature drop observed was
not constant as $n$ was increased. To account for the effect of hardening rate mathematically, a
logarithmic fit was applied to the results from each case of anisotropy as shown in Figure 4-14A.

Through examination of the trends observed in Fig. 4-14A, it is evident that the shape of
the curve fit for each case of anisotropy is nearly identical, however the curves do vary
significantly in the values of the intercepts. All fits were of the form $y = a \ln \left(\frac{H}{100} \right) + b$, where $H$
represents the hardening rate as a percentage of $E$ ($H = 100n$). The value of $a$ was essentially
constant across the cases of anisotropy examined, with an average of 0.03672 and a standard
deviation of 0.002024. In terms of the y-intercept of the fits however, the state of anisotropy had
a considerable, yet mathematically predictable effect. The relation between the intercepts and
the state of anisotropy are highlighted in Figure 4-14B.
Figure 4-13: Illustrates the dependence of the normalized loading curvature on the strain-hardening rate in the case of indentation near the corner. In all cases of out-of-plane anisotropy examined, the normalized curvature increased with the hardening rate.

The curve fit in Fig. 4-14A shows that the drop in normalized loading curvature \( (C) \) is related to the linear hardening slope \( (H) \) by:

\[
C = 0.03672 \ln H + b,
\]

and Fig. 4-14B shows that the intercepts \( (b) \) found in Fig. 4-14A can be related to the anisotropy by the equation:

\[
b = 0.139975 \ln \frac{R_{22}}{R_{11}} + 0.216071.
\]
Figure 4-14: A) The relation between normalized loading curvature at depth $d$ and strain-hardening rate during indentation $d$ away from the sample corner. The results are fitted with logarithmic expressions as shown. B) Intercept of the curve fits as a function of anisotropy ratio $R_{22}/R_{11}$.
As the goal of this parametric study is to mathematically describe the relation between the normalized value of loading curvature at depth $d$ and the anisotropy ratio, these two equations can be substituted into one another to obtain:

$$ C = 0.03672 \ln H + 0.139975 \ln \frac{R_{22}}{R_{11}} + 0.216071, $$

where $C$ is the normalized loading curvature (unitless) at a depth $d$, $H$ is the strain hardening slope as a percentage of elastic modulus $E$, and $R_{22}/R_{11}$ is the ratio between the in-plane and out-of-plane yield stresses. Through algebraic manipulation, this expression can be written in a more applicable form:

$$ \frac{R_{22}}{R_{11}} = \exp[7.14413C - 0.26233 \ln H - 1.54364], $$

in which the normalized loading curvature and strain-hardening rate are taken as inputs and the output is the anisotropy ratio. In order to assess the accuracy/precision at which the equation can determine the anisotropy ratio, the model inputs for the anisotropy ratio were checked against the calculated values according to the derived equation (Table 4-1).

The in-plane to out-of-plane yield stress ratios were calculated within a reasonable degree of error. Error percentages of less than 5% were observed in all calculations with the exception of Case 9. This degree of error is not regarded as problematic as the hardening slope in that case is quite high; surpassing that observed in typical materials. Thus, the proposed equation seems to be a viable tool for the analysis of the yield stress ratio in materials given the data from an indentation test conducted to a depth $d$, at a distance $d$ away from the sample corner. A flow chart of the proposed analysis technique is presented in Figure 4-15 for clarity. The proposed methodology calls for the determination of strain-hardening slope from the load-depth curve.
from the semi-infinite indentation. For an overview of this technique, the reader is directed to Ref. [75]

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Table 4-1: Assessment of the accuracy/precision of the proposed equation to relate the drop in loading curvature (C) and strain-hardening slope (H) to the anisotropic yield ratio $R_{22}/R_{11}$. The normalized C value was obtained from the computational models, and the Input $R_{22}/R_{11}$ and Input H were taken from the inputs used in the models.

Figure 4-15: Flow chart of the technique developed to determine out-of-plane to in-plane yield stress ratio from a combination of semi-infinite indentation and indentation to a maximum depth $d$ at a distance $d$ away from the sample corner.
4.5. CONCLUSION

In this chapter, methods were sought in order to extract the yield stress anisotropy present in an arbitrary sample using the load-depth relation extracted from a set of two indentation tests; one in a semi-infinite regime and the other in the vicinity of free surfaces. The problems of edge and corner indentation were explored, and through the use of the latter, a relation between the normalized loading curvature ($C$), linearized strain-hardening slope ($H$), and the out-of-plane to in-plane yield stress ratio was developed. Furthermore, it was shown that as a result of the normalization scheme, the drop observed in $C$ did not depend significantly on material hardness (i.e. $\sigma_Y/E$); results obtained were consistent through a large range of this parameter.

The technique relies on a few basic assumptions: (i) the stress-strain behavior of the material can be accurately modeled by a bilinear curve with a linear elastic portion followed by a linear strain-hardening slope post-yield, (ii) the material possesses transverse isotropic plastic properties (i.e. yield stress is constant in-plane, is different out-of-plane), and (iii) the indentation is indented to a maximum depth $d$ and is placed a distance $d$ away from the sample corner. While there are many material candidates that obey the assumptions proposed in (i) and (ii), it is accepted that the design of an experiment which follows assumption (iii) can be difficult to the point where it is impractical. Nonetheless, the studies presented in this chapter lay the theoretical groundwork for such a technique, and with the development of new testing devices and equipment, this technique can be ultimately used to characterize the anisotropic yield properties of a material given minimal information about the material prior to testing.
CHAPTER 5. CHARACTERIZATION OF TRAVSVERSE ISOTROPIC PLASTIC BEHAVIOR OF MATERIALS USING SINGLE SPHERICAL INDENTATION
5.1. INTRODUCTION AND OVERVIEW

In the previous chapter, a technique was presented to characterize the ratio between the out-of-plane and in-plane yield properties of a material that is inherently transversely isotropic in plasticity. While the theoretical framework for the methodology was proposed, there was no validation experimentally and the obtained results were purely numerical in nature. In reality, the experimental design of such a study is limited by the commercially available indentation testers. In the case of hardness testing, the attachment of an optical microscope allows for a degree of accurate prescription of the indentation location, however for instrumented indentation, aiming the indentation location is oftentimes arduous. Therefore, the technique developed in this chapter focuses on experimental feasibility of the method and proposes a practical characterization method.

In the present work, computational models were performed to simulate spherical indentation on the free edge of a material exhibiting anisotropy in plastic behavior and the extent of anisotropy was correlated to the out-of-plane sub-surface deformation fields that persisted after unloading. It was further shown that some aspects of the fields were sensitive only to this anisotropy, and insensitive to yield strain, hardening behavior, elastic anisotropy, or in-plane residual stresses, suggesting an indentation-based method of property measurement. Results from the simulations were compared to Brinell indents on bonded interface specimens of Ni-5%Al coatings deposited by various spray techniques. The coatings sprayed via air plasma spray (APS), high velocity oxy-fuel (HVOF) and cold spray (CS) were found to have normal-to-in plane yield ratios of 1.15, 1.30 and 0.60, respectively. Furthermore, micromechanical arguments are provided to describe the differences in anisotropy.
Out-of-plane plastic anisotropy in sheets is typically represented by means of the Lankford Coefficient (or $\bar{R}$-value), which is defined as the ratio of the plastic width-strain (in the plane of the sheet, transverse to rolling direction) to the through-thickness plastic strain [19] during a longitudinal tensile test in the rolling direction. In this manner, the $\bar{R}$-value indicates the ‘easy direction’ for transverse plastic strain and in general, a material with a higher $\bar{R}$-value will have a higher resistance to thinning during forming or drawing operations, and an $\bar{R}$-value greater than unity is often desirable. Although not equivalent, in transverse anisotropy, $\bar{R}$ is directly related to the ratio of through-thickness yield strength to in-plane yield strength ($R_{22}$ in our models), by the relation $\bar{R} = 2(R_{22})^2 - 1$.

With the ongoing development and demand of lightweight/low density structural materials such as magnesium and its alloys (e.g., AZ31, ZK60) to be used in forming [84], it is particularly important to quantify the $\bar{R}$-value so that the formability and performance of a material can be assessed prior to application. In addition, several strategies for forming involve high temperatures, and the effect on anisotropy must also be taken into account. As one example, reported $\bar{R}$-values for AZ31 sheet have shown significant temperature sensitivity (drop by a factor of 2 between room temperature and 150°C) [84-87]. Such behavior suggests the potential value of a simple method to extract the $\bar{R}$-value of various materials and systems under different conditions.

Determination of the $\bar{R}$-value in real materials is experimentally tedious; requiring, in the simplest formulation, measurement of the plastic strain ratios in the rolling, diagonal (45° to rolling direction), and transverse directions. It is also important to note that anisotropy can exist in several directions beyond this [88-91]. However, even in the simplest cases of transverse
plastic anisotropy, (in-plane isotropic, differing from normal) little attention has been paid to connections between out-of-plane plastic anisotropy and indentation response. In previous experiments, indentation near polished free edges of lamellar metallic coatings fabricated by melt-spray-impact caused sub-surface out-of-face deformation after unloading. The shape of the deformation field was different among coatings fabricated with different processes. Given their well-known anisotropic architecture [77, 92-94], it is hypothesized that the differences are due to varying degrees of plastic anisotropy.

In this study, virtual near-edge indentation tests on materials with varying plastic anisotropy were performed using FEM, and resulting out-of-plane deformations were compared to experimental observations. After matching experimental and computational output and eliminating the possibility of other material effects, it is concluded that the coatings exhibit significant out-of-plane plastic anisotropy. In addition, material input to FEM was expanded to encompass a range of realistic parameters, and results were analyzed in the context of a general method of using indentation to analyze out-of-plane plastic anisotropy in other systems.

5.2. DESCRIPTION OF EXPERIMENTAL PROCEDURE

As the FEM study was based on experimental observations, a brief summary of the experiments conducted by Choi et al. [95] is presented. Ni-5%Al coatings were fabricated using thermal spray and cold-spray methods. Figure 5-1(a-c) shows cross sections. Specific experiments are discussed in [95], and salient points of the process are discussed later in the paper in the context of mechanical findings. Elastic modulus, indentation stress-strain, and residual stresses have been measured and tabulated for these materials [95, 96]. Sub-surface deformation was observed via a modified 'bonded interface' (BI) technique, originally designed
for ceramics, in which coatings are cut, polished, and glued back together for spherical loading along the seam; the purpose of which is to preserve indentation-induced damage and features [97-99]. For the images shown here, spherical loading was performed on the interfaces to 100 N with a customized tip of radius $R = 1.5875$ mm, using a Mitutoyo AVK-C2 hardness tester.

Test times were specified, and preliminary tests between 15 second test (5 sec load + 10 sec dwell at max load) and 30 second test (5 sec load + 25 sec dwell) showed negligible difference in resulting imprint. In addition, standardized tests have shown that for Vickers testing on metallic materials, variation in loading rate across several orders of magnitude (0.01 N/s to 5 N/s) caused no change in hardness [100]. This lack of time-dependence is expected for this material at room temperature, as the indentation loads are several orders of magnitude higher than those (100 N vs 1-10 mN) typically associated with room temperature indentation creep [101].

Following indentation, the faces were separated and residual out-of-face deformation was measured using scanning white light interferometry (see [102] for qualitative precedent of this technique). Figure 5-1(d-f) shows binary fields, in which deformation cut-off was 500 nm; namely material that has protruded greater than 500 nm is shown as white, and material that has not is shown as black. Note that the three coatings have distinctly different field shapes. Given the magnitude of the strains under the tip, beyond the expected elastic range, it was hypothesized that the shapes could be due to plastic anisotropy of different degrees.
Figure 5-1: (a-c) Scanning electron microscope (SEM) cross-sections of the coatings indented in the experimental study. From left, APS, CS, HVOF [95, 96]; (d-f) binary images showing out-of-plane residual deformation after 100N indentation; white areas protrude more than 500 nm [95].

5.3. COMPUTATIONAL PROCEDURE

5.3.1. Mesh, Geometry, and Loading Conditions

A systematic set of finite element (FE) models were employed to explore the resulting deformation fields beneath virtual indentation, under different conditions. Three-dimensional spherical indentation was modeled using ABAQUS. Due to the symmetry of the problem, one-fourth of the experiment was modeled in order to conserve computational resources so that a complete set of material properties could be examined. Figure 5-2A shows a schematic representation of the BI technique and Figure 5-2B shows the model and meshing implemented throughout the analysis. A fine mesh is applied to the region beneath the indenter tip, while a coarser mesh is applied in the regions sufficiently distant from the contact region. A total of 57,190 linear hexahedral elements (C3D8R) are used for the elastic-plastic solid. The indenter was modeled as a discrete rigid shell with radius $R = 1.5875$ mm, and meshed with 705 linear
quadrilateral elements (R3D4). The coordinate system, as used throughout the study, is also highlighted in Fig. 5-2B. A coefficient of friction ($\mu$) of 0.15 was defined between the indenter and the specimen in all simulations [76].

![Figure 5-2: A) Schematic of the bonded interface technique B) Finite Element mesh that was employed in this study.](image)

Referring to Fig. 5-2B, roller BCs consistent with symmetry were applied to the left face (A), while the right face (B) was unconstrained. The free edge was chosen so as to maintain
consistency with the BI tests in which the two faces were adhered together with a compliant glue layer several microns thick. Load-controlled indentation was modeled to a maximum load of 100 N and subsequently unloaded to 0 N. Two sets of models were run: (i) a set of ‘virtual experiments’ in which material input and loading was matched to the available experimental data described in section 5.2. for comparison, and (ii) a more general set of models, with variation in mechanical properties and load, to begin to explore their effect on observed results.

Primary output of the models was the residual deformation in the direction normal to face B in Fig. 5-2B. Contours were plotted to show displacements greater than a specified cut-off value. Considering the magnitudes of loading that were applied, this cut-off was set to values ranging from 250 nm - 1000 nm as the deformation induced is approximately consistent with this order of magnitude.

5.3.2. Material Input

For the virtual experiments, elastic modulus ($E$), Poisson’s Ratio ($\nu$), out-of-plane yield strength ($\sigma_{Y-22}$), and hardening behavior were estimated from stress-strain curves extracted from previous literature [95, 96]. Transverse anisotropy was imposed in addition to this; following Hill’s treatment [103]. Specifically, the state of transverse plastic anisotropy was implemented by means of 6 $R$-values using the ABAQUS POTENTIAL sub-option in the plasticity module. Three $R$-values ($R_{11}$, $R_{22}$, $R_{33}$) correspond to the ratio of normal yield strength in directions 1,2,3 (see Fig. 5-2) to a given yield strength $\sigma_y$, and three other $R$ values ($R_{12}$, $R_{23}$, $R_{13}$) correspond to the ratio of shear yield strengths of $\sigma_y/\sqrt{3}$, as implemented in Hill's 1948 yield criterion [103] (see Section 1.3.2).
For the models to explore transverse anisotropy, all directional $R$-values were maintained at unity with the exception of $R_{22}$, corresponding to the ratio of yield strength in the indentation direction ($\sigma_{Y_{22}}$) to input $\sigma_Y$; this $R_{22}$ was varied from 0.50 to 2.00. This can be likened to the variation of the Lankford Coefficient ($\bar{R}$) from 0.50 to 2.00. Note that as described in Chapter 1, there are greater degrees of complexity in plastic anisotropy and anisotropic yield criteria, but given the beginning stages of work in this area the most general form was chosen. For the general set of models, different yield strength to elastic modulus ratios, and hardening behavior were implemented. In addition, elastic anisotropy and in-plane residual stresses were imposed. All properties are listed in Table 5-1.

<table>
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<th>$E_2$ (GPa)</th>
<th>$E_3$ (GPa)</th>
<th>$\sigma_Y$ (MPa)</th>
<th>$\nu$</th>
<th>Hardening Rate (% of $E_2$)</th>
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<td>110</td>
<td>368</td>
<td>0.31</td>
<td>2.57 From Ref. [96] (CS)</td>
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<td>180</td>
<td>432</td>
<td>0.31</td>
<td>1.85 From Ref. [96] (HVOF)</td>
</tr>
</tbody>
</table>

Table 5-1: Material input properties used in the FEM models.

5.4. RESULTS

Figure 5-3 shows line contours for out-of-plane residual deformation, with a cutoff of 500 nm, for Material 1 (Table 5-1). A few significant features can be seen: (i) contour shape is a strong function of plastic anisotropy, (ii) ‘sharpness’ of contour increases with increasing $R_{22}$. Following this, a potential means for characterization of contours is to inscribe a circle matching the radius at the lowest point of the contour, and observe how the circle intersects the remainder of the contour; as illustrated in Figure 5-4. Using this method, it is apparent that if $R_{22} > 1$, the
inscribed circle falls inside the contour, and if $R_{22} < 1$, it falls outside the contour. If $R_{22} = 1$, the shape of the contour is circular.

Figure 5-3: FEM results for material 1 showing out-of-plane contours, revealing a strong dependence on plastic anisotropy. Contour threshold is 500 nm.
Figure 5-4: FEM results for material 1 with dashed circles inscribed. Note that when $R_{22} = 1$, the out-of-plane deformation contour fits the circle precisely.

Figure 5-5 shows results for Material 2, in which $\sigma_Y/E$ is significantly higher than in Material 1. Note that although the trends of contour sharpness persist, the magnitudes by which they vary with $R_{22}$ are different, that is to say sensitivity decreases with increasing $\sigma_Y/E$. Nevertheless, for all cases, isotropic plasticity gives a clearly circular contour. Figure 5-6 shows effects of the other input property and measurement variations, including elastic modulus anisotropy, in-plane residual stress, hardening behavior, and out-of-plane cutoff on a plastically isotropic material. In all cases, the out-of-plane contour remains circular, and any effects of these parameters are negligible.
Figure 5-5: FEM results for material 2, with a significantly higher yield strength than material 1, showing a similar trend with respect to $R_{22}$. 

$R_{22} = 0.50$ \hspace{2cm} $R_{22} = 0.60$

$R_{22} = 0.70$ \hspace{2cm} $R_{22} = 0.80$

$R_{22} = 1.00$ \hspace{2cm} $R_{22} = 1.25$

$R_{22} = 1.50$ \hspace{2cm} $R_{22} = 2.00$
Figure 5-6: FEM results on a plastically isotropic material ($R_{22} = 1$) showing that other parameters such as a) elastic anisotropy, b) residual stress, c) contour selection, and d) yield strength have a negligible effect on the residual deformation profile.

Figure 5-7 shows results comparing experiments on coatings, and the virtual experiments. Virtual results show the best fit of $R_{22}$ to experimental data, given the literature stress-strain input. The Ni-Al coatings deposited by APS, CS and HVOF exhibit $R_{22}$ values of 1.15, 0.60, and 1.30, respectively. This implies that under the loading conditions in the experiments, APS has a
nearly 15% higher out of plane yield strength, CS has higher in-plane yield strength (40%), and HVOF has higher out-of-plane yield strength (30%).

![Experimental and virtual results comparison](image)

Figure 5-7: Results from experiments (top) compared with virtual experiments (bottom) showing good agreement in residual displacement fields, allowing estimation of transverse plastic anisotropy.

### 5.5. DISCUSSION AND IMPLICATIONS

The results from the general models show that spherical indentation on the free edge of an elastic-plastic material induces a measurable “bulge,” the shape of which is dependent upon out-of-plane plastic anisotropy. Figure 5-3 shows six different line contours of the deformation in cases with different values of $R_{22}$. From the figure, it is apparent that the results show particular trends from which valuable information regarding the plastic material properties can be obtained. As previously mentioned, when the specimen is plastically isotropic (i.e., $R_{22} = 1$), the field is perfectly circular in geometry and the inscribed circle fits nicely over the deformation.
field. However, in cases in which the yield stress is lower in the indentation direction than in the plane of the samples, the radius of curvature of the bottom portion of the residual bulges increases; effectively increasing the radius of the circle that is fit to it. Thus, the deformation field lies completely inside the circle when $R_{22}$ is less than unity. On the contrary, when the $R_{22}$ value is greater than unity (or yield stress is greater in the indentation direction), the bottom of the deformation field becomes sharper, causing the inscribed circle to fit entirely inside the field. Through the use of this algorithm, experimental determination of how the value of $R_{22}$ in a sample compares with unity becomes possible with minimal sample preparation (consistent with MBI) and a single hardness test. A chief advantage of this method is that it is highly applicable to sheets, thin-film/substrate systems, and other material systems in which determination of bulk plastic properties is oftentimes limited.

It is expected that with a wider range of models, an empirical relation could be found between aspects of the inscribed circles and plastic anisotropy, given a set of elastic and plastic properties. However, this may require a focused range of properties of interest to a particular material set, for example in several indentation papers that extract properties for ranges relevant to hardening metals [18, 104-107]. Models indicated that shape of the out-of-plane deformation is not affected by other properties reported in Fig. 5-6. This is perhaps expected, given the magnitude of the associated strains, well into the plastic region of constitutive behavior. Effects of in-plane anisotropy were not tested. Materials with perfectly plastic behavior showed decreased sensitivity of deformation field shape on plastic anisotropy, as shown in the case of Material 3 (Figure 5-8), but from the contours the difference between $R_{22} > 1$ and $R_{22} < 1$ is still evident. For some sheet metals, Hill’s treatment of plastic anisotropy has been discussed as too simplistic, and it is currently untested how more advanced constitutive models [88-91] may
affect the results shown here. Overall, this work is similar in philosophy to reference [82], in which the authors determined algorithms to correct for edge effects on extraction of material properties, such as elastic modulus. Here, we begin to show how inclusion of a free boundary condition may provide access to anisotropic behavior traditionally not inaccessible via indentation. It is sought that this observation will ultimately promote further discussion in literature, especially as 3D FEM models are now computationally feasible.

![Figure 5-8: FEM results for Material 3; a material with high yield strength and perfectly plastic post-yield behavior. Contours are presented with an applied imaging threshold of 250 nm.](image)

Through a slightly different analysis, it is also possible to estimate the value of $R_{22}$ in a sample using the proposed method. By inspection and induction (trial and error), reasonable matches were found for the three experimental sets of coating results available (Fig. 5-7). The comparisons suggest that the APS coating has an $R_{22}$ of approximately 1.15 (15% stronger in the
indent direction than in-plane), the HVOF coating has \( R_{22} = 1.30 \) (30% stronger in the indent direction than in-plane) and the CS material has an \( R_{22} = 0.60 \) (40% weaker in the indent direction than in-plane).

The above results can be explained with a discussion of the microstructure and processing of the above three materials. In spray coatings, particles of 10-50 micron diameter are accelerated in a heated gas jet towards a substrate where they impact, flatten, cool, and solidify. Successive impact of particles leads to a dense coating, characterized by a lamellar or quasi-lamellar microstructure of flattened particles or splats. In thermal spray, all but the most refractory particles are completely molten upon impact, and rapid cooling and solidification occurs, leading to a columnar microstructure within each splat. Air plasma spray (APS) and high velocity oxy-fuel (HVOF) differ primarily in impact velocity (100-200 m/s for APS and 300-500 m/s for HVOF) which imparts greater flattening and more rapid solidification in the latter process. Cold spray (CS) increases impact velocity to 700-900 m/s and decreases temperature to near room temperature. That is to say, solid particles impact, with sufficient kinetic energy for severe plastic deformation to cause localized heating and inter-particle bonding. The combination of particle impact velocity and temperature in APS typically produces a spread but wavy lamellar architecture, with fine columnar grains that are on the order of lamellar thickness [94-96, 108]. Assuming that within this range of applied indentation strain, the lamellar interfaces do not de-bond, one could expect the resulting plastic properties to be more isotropic than HVOF, as observed in the present study.

In addition, elastic and plastic anisotropy of similar APS Ni-5%Al coatings were measured in [18], using dual Berkovich nano- and micro-indenters (max load 100 mN and 3.5 N, respectively) in orthogonal directions and inverse analysis. The authors in that study found an
anisotropy size effect in that on the nano-scale (max depth 1.20 μm) effective $R_{22}$ was 1.30 but on the micro-scale (max depth 10 μm) that value dropped to 1.20. As volume of material sampled increases, $R_{22}$ approaches the values seen in the current analysis.

For HVOF, splats impact on the surface at significantly higher velocities (up to 3x APS), causing more flattening and faster cooling. As the elastic modulus of these materials is typically near bulk values, one can again assume that within this range of indentation strain, interfaces do not de-bond. The microstructure in this case consists of finer columns (several hundred nm) than the splat thickness [95, 96]. Thus, the higher yield strength out of plane is expected. The anisotropy in CS is particularly interesting, as during (supersonic) impact, particles are not molten, and do not flatten as extremely as in the melt-spray techniques. Nevertheless, they undergo a high degree of cold working [95, 96, 109], perhaps analogous to a rolling process in which the rolling direction is bi-directional and in-plane. It has been shown via laborious tests in the literature that this produces anisotropic plastic behavior, with strengthening along the ‘rolling’ direction (e.g., [95]), which is also in agreement with our results. While plastic anisotropy in the context of forming is not as critical for coatings and films as for sheet metals, it could potentially be a valuable descriptor for fundamental microstructural studies.

5.6. CONCLUSIONS

In this study a modified bonded interface technique was used in conjunction with direct FEM comparison to show anisotropic plastic behavior in three sprayed materials. It should be noted here that additionally, this analysis worked for indentation loads that were lower than the ones reported, but in some cases at higher loads the predictions did not match. At higher loads, it can be assumed that inter-particle fracture begins to occur, presumably modifying the inelastic
mechanisms in place. In its current form, these observations could be useful for bulk sheet
metals, in situations for which easy direction could be determined (i.e., $R_{22} > 1$), but the
experiments are perhaps just as laborious as conventional methods. However, for research
purposes in thick coatings or thin films, this behavior could be used to correlate plastic
anisotropy to microstructure, processing or tribological behavior, particularly in systems in
which load-depth data is not readily available. It is anticipated that supplementary information
contributing to the analysis could be derived from load-depth data, perhaps to elucidate
contributions from modulus or work hardening, but such investigations were not the main goal of
the work presented in this chapter. Finally, if the methodologies used in this work were explored
at lower loads, time-dependent effects would likely need to be taken into account [101, 110,
111].
CHAPTER 6. CHARACTERIZATION OF IN-PLANE PLASTIC ANISOTROPY
6.1. OVERVIEW OF SCRATCH TEST METHODOLOGY

In the previous chapters of the current work, characterization of the yield stress ratio out-of-plane to in-plane was the primary focus, and the yield stresses in the plane of the specimen were assumed to be equal. Namely, the cases considered were limited to transverse isotropic plastic behavior. As the goal of this study is to characterize the ratios of the yield stresses in all orthogonal directions of a sample, the study presented in this chapter will focus on materials with anisotropic yielding in-plane.

It has been shown that the ideal means of characterizing such behavior is through examination of the material flow in the vicinity of contact. Furthermore, it was shown in the findings presented in Chapter 4 that tests which limit mechanical constraints provide more favorable results due to the fact that such tests allow for analysis of the "pinning" of material that results from the inherent state of plastic anisotropy. To exploit this property in ascertaining the in-plane yield stress ratios, the use of a mechanical scratch test is proposed. This is the test of choice for two chief reasons: (i) mechanical scratch induces significant plastic deformations in the immediate vicinity of contact, (ii) information on the resistance of the material to plastic deformation is manifested in the residual deformation resulting from the scratch, and (iii) mechanical properties can be evaluated readily through measurement of the loads in the normal and transverse directions of the scratch tip via multiaxial force measurement.

Traditionally, scratch tests have been applied to evaluate the scratch resistance of a material. The idea is that through plowing and cutting the surface of a weaker material with a more rigid scratch tip, the scratch resistance of a material can be quantified (i.e. via the scratch hardness) [112]. Due to recent advancements in force and depth sensing technology, the scratch test remains a popular alternative to other material property testing methods and maintains
relevance in many fields of engineering, ranging from macro-scale applications through the nano-scale [113, 114]. It is important to note that the applicability of scratch tests to material systems is similar to indentation; as minimum sample preparation is required to scratch on a flat surface of a sample.

Typically, the scratch test is conducted using a Rockwell C tip and a scratch is carried out in a linear-path so that properties such as adhesion-strength of coatings, surface friction, overall wear characteristics, and other useful properties can be determined. Other formulations of the scratch test are the pin-on-disk and ball-on-disk wear tests. Focusing on the latter, in its simplest construction, a spherical ball is pressed to a known load into a disk which is rotating at a constant angular velocity as shown schematically in Figure 6-1 [115]. This test is primarily used to assess the wear properties of both the ball and the disk over time. As the test is continuous over a set time period (number of rotations), its application falls within the class of standard fatigue tests.

![Figure 6-1: Schematic of the ball-on-disk scratch test. The ball is pressed into the disk to a constant load F, and the disk is rotated at a constant angular velocity, w.](image-url)
Due to the multi-directionality of the test, it seems to be a prime candidate for evaluating the in-plane yield stress ratios in a given sample. More useful than the linear scratch which is typically unidirectional, by tracking the in-plane reaction force tangent to the scratch path continuously, a relation between the reaction force and the angle of tip rotation can be developed.

In this chapter, both linear and circular scratch tests will be explored via FEM so that the applicability of the tests for extracting anisotropic plastic behavior in materials can be evaluated. First, linear scratch results will be evaluated on materials with varying in-plane yield stress ratios by varying the properties both in the scratch and transverse directions. Next, the results of the ball-on-disk analysis will be explored with the goal of finding a trend in the results that is sensitive, perhaps exclusively, to the directionality in yield stress.

6.2. METHODS

6.2.1. Finite Element Model of Linear Scratch Test

A three-dimensional FEM simulation (ABAQUS/Standard) of a mechanical scratch test was employed to provide insight to the scratch response of materials with varying degrees of in-plane plastic anisotropy. The material used throughout this portion of the study was given constant linear-elastic properties \( E = 300 \) GPa, \( \nu = 0.3 \) and the input \( \sigma_Y \) was held constant at 600 MPa \( (\sigma_Y/E = 1/500) \). A linear work-hardening behavior was input with a slope equal to 2% of the Elastic Modulus slope. Anisotropic plastic behavior was input by maintaining the \( R_{11} \) (scratch-direction) and \( R_{22} \) (out-of-plane) values at unity, and varying \( R_{33} \) (in-plane, transverse to scratch path). Furthermore, analysis was conducted on samples with the opposing anisotropy; maintaining \( R_{33} \) at 1 while varying \( R_{11} \). This technique effectively varies the ratio of the yield stresses in the plane of the sample while keeping the out-of-plane ratio constant.
The material was scratched with an analytical rigid Rockwell C indenter, with a tip radius of 200 μm. The width of the sample was modified so that the 3D model could accurately represent the semi-infinite case. A mesh convergence study was conducted, and the final mesh consisted of 173,000 6-noded wedge (C3D6) elements. The mesh, dimensions, and coordinate system are highlighted in Figure 6-2. A fine mesh was used in the vicinity of the scratch and was gradually coarsened in regions sufficiently distant from the region of interest. Displacement of the bottom face was fixed in all directions, and symmetric BCs were applied to the face labeled A in Fig. 6-2, i.e. displacement was constrained only in the direction normal to face A. Frictionless contact was defined so as to simulate a lubricated scratch test. The indenter tip was first lowered into the material to a maximum depth of 5 μm and subsequently dragged 500 μm across the surface of the sample (in the 1-direction). Residual depth profiles (2-3 plane) for each case of plastic anisotropy were plotted for the same set of nodes for comparison, and the height of residual pile-up was analyzed for comparison.

Figure 6-2: Shows the mesh design and coordinate system for the 3D FEM model of the linear mechanical scratch test.
6.2.2. Finite Element Model of Circular Scratch Test

In order to characterize the plastic anisotropy in-plane, it is proposed that a novel circular scratch method be employed. Traditionally, scratch tests are designed with a linear scratch path for the purpose of extracting parameters such as coefficient of friction of the scratched surface, or adhesion between a coating and a substrate. However, for the purposes of exploring the anisotropic plasticity in the plane (normal to indentation direction) of a material, it is hypothesized that scratching in a circular path, while making multi-axial force measurements, can provide useful insight to these properties.

A computational model of a circular scratch was developed using ABAQUS. The virtual experiment is designed with a Rockwell C tip with tip radius of 200 µm acting as the scratch-tip. The tip is first lowered to a depth of 10 µm, which will induce the proper degree of plastic deformation, and subsequently rotated 360° and the reaction forces in the 1-, 2-, and 3- directions is measured at each angle. Figure 6-3 shows the mesh design of the sample, the mesh design of the Rockwell C tip, as well as the coordinate system that was be employed throughout the study. The sample is meshed with 145,800 six-node linear wedge elements (C3D6) with a fine, uniform mesh in the scratch path. The indenter tip is modeled as a discrete rigid shell attached to a lever arm. In the initial step in which the indenter tip is lowered into the sample, the arm is free to displace laterally in the 2-direction and all other displacements and rotations are prevented using appropriate BCs. In the scratch step, the displacement in the 2-direction is fixed (constant depth) and rotation about the 2-axis is prescribed to a total rotation of 2π radians in fine increments for the analysis. From the reaction forces, the magnitude is calculated from the relation: \( RF_{mag} = \sqrt{RF1^2 + RF3^2} \). If calculated at every increment throughout the scratching step, the force tangent to the circular path at any angle will be obtained.
As in Section 6.2.1., properties other than anisotropic yielding were held constant. The material used throughout this study was given constant linear-elastic properties ($E = 300$ GPa, $\nu = 0.3$) and the input $\sigma_Y$ was held constant at 600 MPa ($\sigma_Y/E = 1/500$). A linear work-hardening behavior was input with a slope equal to 2% of the Elastic Modulus. Anisotropic plastic behavior was input initially by maintaining $R_{11}$ and $R_{22}$ values at unity, and varying $R_{33}$ to values both greater than and less than 1. This would embody cases in which the yield stress was both less and greater in the principle direction of anisotropy. For completeness, the analysis was taken further to incorporate variations in $R_{22}$ as well; ensuring that any trends observed persisted with the introduction of out-of-plane anisotropy.
6.3. RESULTS

6.3.1. Linear Scratch Test on Materials with In-Plane Anisotropic Plasticity

From the linear scratch on materials with in-plane anisotropic yielding, behavior consistent with the anomalous trends described in Chapter 3 was observed. When varying the ratio between the yield stress in the 3-direction (transverse to scratch) to the yield stress in the 1-direction (scratch direction), the material hardness (resistance to plastic deformation) showed a minimum when $R_{33}/R_{11}$ was 0.70, and a maximum when 0.50. This statement is quantified through the measurement of the residual depth profile following the scratch as shown in Figure 6-4. For clarity, the indenter geometry is also presented in the figure and although the geometry of the indenter seems sharp, it should be noted that the x- and y- scales on the plot are quite different to emphasize the differences in residual pile-up. The deformed coordinates were extracted from the finite element simulation for the same set of nodes for accurate comparison.

The highest level of pile-up was observed when $R_{33}/R_{11} = 0.70$, and the minimum when $R_{33}/R_{11}$ was 0.50. It is important to note that the degree of residual pile-up in the latter case was nearly equivalent to that observed when $R_{33}/R_{11} = 2.00$, however the sharp rise in material at the edge of contact that was observed in the cases of 0.60-2.00 was absent when the ratio was 0.50. The unpredictable trend apparent here shows that the tracking of residual deformation post scratch leads to inconclusive results when attempting to determine the anisotropic yield ratio in the sample. The analysis was conducted further to see if the trend persisted when varying the yield stress in the scratch direction in place of the transverse direction.
Figure 6-4: Computationally calculated residual depth profiles resulting from scratch testing of materials with different values of $R_{33}/R_{11}$. Units are in μm and the dashed line represents the scratch tip geometry.

Figure 6-5 shows the residual depth profiles post-scratch for materials with varying degrees of anisotropic yielding in the direction of scratch. Specifically, this figure represents materials with a constant yield stress in the transverse direction ($R_{33} = 1$) and a varying yield stress in the scratch direction ($R_{11}$ varies). It is apparent that the trend in residual deformation profile does not show the inflection in hardness observed in Fig. 6-4. The maximum degree of pile-up is observed when $R_{11}/R_{33} = 2.00$ and the minimum when $R_{11}/R_{33} = 0.50$. This phenomenon can be explained by monitoring the means in which the imposed anisotropic yield behavior of the sample is being varied with respect to the 2-direction (normal to surface, $R_{22}$). In the case of $R_{11}/R_{33} = 2.00$, the ratio of the yield stress $R_{22}/R_{11}$ is a minimum, thus promoting a
heightened degree of plastic deformation in the 2-direction with respect to the 1-direction. The prescribed condition manifests in the observation of extensive pile-up normal to the surface and as the ratio $R_{11}/R_{33}$ is decreased, the tendency of the material is to demonstrate a lesser degree of pile-up. Moreover, the residual scratch depth is greatest when $R_{11}/R_{33} = 2.00$, further validating the hypothesis that due to the case of anisotropy, plastic flow of material in the normal direction is most preferred.

Although useful information was extracted through the simulation of the linear scratch, drawing conclusions regarding the anisotropic yielding of a sample from the results observed is impractical. In addition to the residual deformation profiles being inconclusive, when tracking
the normal load (2-direction) vs. the load in the scratch direction (1-direction) no predictable trend could be developed. Furthermore, the simulations described here did not include a friction coefficient for the contact between the scratch tip and the surface; assuming a perfectly lubricated scratch surface. This assumption is feeble; it is generally improbable that such an experiment can be designed as there will always be a degree of friction between two surfaces in contact. In terms of the loads and residual deformations observed, it is certain that variations of friction coefficient will have a pronounced effect. As a result of these limitations to the linear scratch technique, a modification was proposed in which the sample was subjected to a circular scratch, similar to a ball-on-disk configuration, however with a Rockwell C instead of a spherical scratch tip. The goal was to analyze the direction-dependent behavior of the sample through the use of a single test.

6.3.2. Circular Scratch Test on Materials with In-Plane Anisotropic Plasticity

Following the linear scratch analysis, simulation of a circular scratch was attempted in hopes that the degree of anisotropic plasticity in a sample could be obtained from a single test. As previously described, a Rockwell C tip was lowered to a depth at which significant plastic deformation was induced. The resulting reaction force tangent to the indenter tip was calculated at different angles of rotation from 0° (beginning of scratch) to 360° (end of scratch). A plot of the calculated reaction forces as a function of angle is shown in Figure 6-6. The results show a significant dependence on anisotropic yield stress ratio ($R_{33}/R_{11}$). For clear definition of the coordinate system employed, the reader is directed to Fig. 6-3. Cases are presented with $R_{33}/R_{11}$ both less than (Fig. 6-6A) and greater than 1 (Fig. 6-6B-E). For the isotropic case (dashed line in Fig. 6-6A), the reaction force is constant through rotation, indicating that there is no directional dependence of the in-plane yield stress of the material. The effects of varying coefficient of
friction ($\mu$) between the scratch tip and the sample surface were also examined; $\mu = 0.20$ and $0.30$ were input, as the typical range of friction coefficient is inclusively represented by these values.

Figure 6-6: FEM calculation of the angular dependence of the magnitude of in-plane reaction force tangent to the circular scratch path for materials with different input values of in-plane anisotropic plastic properties. A) $R_{33}/R_{11} = 0.66$, B) $R_{33}/R_{11} = 1.20$, C) $R_{33}/R_{11} = 1.50$, D) $R_{33}/R_{11} = 1.75$, E) $R_{33}/R_{11} = 2.00$. 

125
Two distinct shapes are observed upon careful assessment of the curves; when $R_{33}/R_{11} < 1$ (Fig. 6-6A), the maximum force occurs at 90° and the minimum at 180°, and when $R_{33}/R_{11} > 1$ the opposite is observed, maximum at 180° and minimum at 90°. This outcome is significant in itself, as clear determination of the orthogonal direction with lower yield stress is made possible. If the curve shape resembles that in Fig. 6-6A, the yield stress is lower in the direction parallel to the orientation of the scratch tip at 0° and if the shape resembles that in Fig. 6-6B-E, the yield stress is lower in the direction parallel to the orientation of the scratch tip at 90°. Though a noteworthy finding, the main goal is to determine the exact ratio of the in-plane yield stresses in the sample and thus, further analysis of the presented data is required.

The plots show that as the ratio $R_{33}/R_{11}$ increases, the difference in the maximum and minimum loads also increases. Upon more attentive investigation, it can be shown that the ratio between the loads at 180° and 90° is quite representative of the imposed in-plane yield stress ratio. These results are tabulated in Table 6-1 for the cases presented in Fig. 6-6. The ratio between the reaction force magnitudes can predict the yield stress ratio in the sample to a minimal degree of error. The results also show that the analysis is independent of the friction coefficient between the scratch tip and the sample surface; through large variations of the parameter $\mu$, the analysis is still precise. Furthermore, it should be mentioned that variations in the baseline yield strength also do not impact the results (Fig. 6-6C), likely due to the normalization scheme employed; contributions of other material properties are removed through division of the reaction force data at 180° by the force at 90° on the same sample.

To explain the observed results, the evolution of material pile-up in front of the scratch tip is examined. In the case of isotropic yielding in-plane, aside from the initial portion when the scratching of the material is evolving from the region of scratching to the region of plowing, the
Table 6-1: Tabulated results of the reaction force tangent to the scratch path resulting from the FEM simulation of a circular scratch test. Loads are tabulated for scratch tip positions of 180° and 90° for different input yield stress ratios. Error percentage between input properties and calculated properties are also tabulated.

<table>
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<th>R_{22}</th>
<th>R_{33}</th>
<th>\mu</th>
<th>RF_{Mag} @ 180°</th>
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level of pile-up is constant. Thus, there is no variation in the reaction forces sensed by the scratch tip and the calculated ratio of the loads is 1; signifying isotropic behavior in-plane. When in-plane yield anisotropy is present, however, the degree of pile-up along the circular path varies in a somewhat predictable manner. Figure 6-7 shows the normal-to-surface displacement contours on the top surface of the sample for two cases of in-plane anisotropy at angles of 90° and 180°. In the case in which R_{33}/R_{11} = 0.66, or in general, less than one, the highest amount of pile-up occurs when the scratch tip is at 90° and the minimum occurs when the tip is at 180°. This behavior is reflected in the measured reaction forces as seen in the plot in Fig. 6-6A; the maximum load is at 90° and the minimum at 180°. Conversely, when R_{33}/R_{11} = 1.75 (greater than 1), the maximum pile-up occurs at 180° and the minimum at 90°, indicative of the results shown in Fig. 6-6D which show a maximum in-plane reaction force at 180° and a minimum force at 90°. The pile-up behavior can be explained through further consideration of the material constraints that are imposed by the anisotropic in-plane yield stress ratios.
Figure 6-7: The displacement contours normal to the surface at scratch tip rotation angles of 90° (left) and 180° (right) for $R_{33}/R_{11} = 0.66$ (top) and $R_{33}/R_{11} = 1.75$ (bottom). Pile-up behavior is reflective of the in-plane reaction force results shown in Fig. 6-6.

Generally speaking, when $R_{33}/R_{11} < 1$, flow of material is more preferred in the 3-direction than in the 1-direction; the ratio indicates that the yield stress is greater in the 1-direction than in the 3-direction. As a result, when the scratch tip is positioned at 90°, the material is inherently constrained in such a way that flow is preferred in the 3- and 2-directions.
while hindered in the 1-direction. Thus, with this type of anisotropy, the maximum pile-up naturally occurs 90°; the position at which the 1-direction is perpendicular to the scratch path. By the same logic, the minimum pile-up is observed at the 180° position at which the 3-direction (direction of preferred flow) is perpendicular to the scratch direction. At this position, material can freely escape the region proceeding the scratch tip as flow perpendicular to the tip is most favored at this position, and consequently, the degree of pile-up is minimized. In cases in which $R_{33}/R_{11} > 1$, the exact opposite phenomenon is observed; the higher yield stress in the 3-direction promotes material flow in the 1- and 2- directions. Therefore, pinning of material in the 3-direction leads to a maximum pile-up when the scratch tip is at the 180° position (3-direction perpendicular to the scratch path) and a minimum pile-up when the tip is at the 90° position (3-direction parallel to the scratch path).

The proposed technique enables prediction of the in-plane anisotropic plasticity of a sample to a minimal degree of error as shown in Table 6-1. The models described in this section were limited to materials in which the yield stresses differ only in the in-plane direction; cases in which the material exhibited anisotropic yielding in all three normal directions were not explored. However, it was shown that effects of friction between the scratch tip and the sample surface did not affect the analysis. For completeness, in the following section, further analysis is conducted on materials possessing anisotropic yield properties in all normal directions so that the proposed technique can be validated with no restrictions on the anisotropies that can be accurately described.
6.3.3. Circular Scratch Test on Materials with Combined In-Plane and Out-of-Plane Anisotropic Plasticity

The cases examined in this section are designed to develop an understanding of the behavior of the material when the out-of-plane yield stress ratio is modified, given that there is also anisotropic yielding defined in the plane of the sample. Figure 6-8 shows the magnitude of the reaction force (i.e. the reaction force in the direction tangent to the circular path along which the Rockwell C tip is travelling) vs. the angle of rotation of the tip, for varying cases of anisotropic plasticity in all three orthogonal directions. As in the previous section, directions 1 and 3 refer to the planar directions, and the 2-direction refers to the out-of-plane direction. Magnitudes were obtained through the relation: \( RF_{mag} = \sqrt{RF1^2 + RF3^2} \), where RF1 and RF3 are the reaction forces in the 1 and 3 directions, respectively.

Fig. 6-8A shows three cases of isotropic in-plane yield stresses; one that is isotropic in all directions, one that is isotropic in-plane and has a higher yield stress out-of-plane \((R_{22} = 1.50)\), and one that is isotropic in-plane and has a lower yield stress out-of-plane \((R_{22} = 0.70)\). In all three cases, aside from minor fluctuations, the reaction force remains constant when the in-plane yield stress is constant. This observation signifies that in such cases, out-of-plane anisotropy has no significant effect on the observed in-plane reaction forces and furthermore, a constant in-plane reaction force signifies in-plane isotropy in the yielding behavior of the material. Fig. 6-8B represents the case in which the yield stress is lower in the 3-direction than in the 1-direction \((R_{33}/R_{11} = 0.66)\). In this particular case, a significant effect of out-of-plane anisotropy on the reaction force calculated in the FEM simulation is seen. Specifically, when compared with the dotted line in Fig. 6-8B in which the yield stress is only anisotropic in-plane, the results are significantly different. In Fig. 6-8C-E, however, aside from differences in overall reaction force
magnitude, the ratios between the reaction forces at 180° and 90° are more or less equivalent in the presence and absence of out-of-plane anisotropy.

Figure 6-8: FEM calculation of the angular dependence of the magnitude of in-plane reaction force tangent to the circular scratch path for materials with different input values of combined in-plane and out-of-plane anisotropic plastic properties. The black dotted plots represent comparative cases in which no out-of-plane anisotropy is present. A) $R_{33}/R_{11} = 1.00$, B) $R_{33}/R_{11} = 0.66$, C) $R_{33}/R_{11} = 1.20$, D) $R_{33}/R_{11} = 1.50$, E) $R_{33}/R_{11} = 2.00$. 
Of particular interest is the behavior of the plots in Fig. 6-8D-E. In both of these cases, in the proximity of 90° and 270°, there is a sharp rise in the reaction force that is not apparent in any of the other cases. The anisotropy in these cases has $R_{22} = 0.70$, meaning that out-of-plane material flow (i.e. pile-up) is the preferred deformation mechanism in the vicinity of the indenter tip. To explain this phenomenon, the discussion is turned to the pile-up behavior of the sample in these cases (Figure 6-9). Fig. 6-9 is for the case in which $R_{11} = 1.00$, $R_{22} = 0.70$, and $R_{33} = 2.00$. The contours represent displacement in the out-of-plane (2) direction. When looking at the contours at 60°, one can see that there is no material pile-up proceeding the indenter, and thus, the reaction force tangent to the path is minimum (as seen in Fig. 6-8E). At 90°, pile-up is then beginning to develop, contributing to a heightened reaction force and again, symmetrically, the reaction force drops back down due to the same material behavior shown at 60°. When the indenter has rotated 180°, there is a significant amount of pile-up in front of the tip, and hence, a high reaction force magnitude is observed.

![Figure 6-9: The displacement contours normal to the surface at scratch tip rotation angles of 60° (left), 90° (center), and 180° (right) for the case in which $R_{11} = 1.00$, $R_{22} = 0.70$, $R_{33} = 2.00$. Pile-up behavior is reflective of the in-plane reaction force results shown in Fig. 6-8E.](image)

In this case, as discussed in Section 6.3.2., the preferred deformation direction is in the 2-direction as a result of the inherent anisotropy. In reality, it is more appropriate to think of this
material as having *least constraint* in the 2-direction. At 180°, for example, the material is being loaded with the indenter and the material must be displaced somewhere. Thus, the pile-up effect is most apparent here. The material is both constrained in the 3-direction and the 1-directions at this position, and so, the material is being pushed "upward" as opposed to being pushed "away". As there is more material at this point in the path of the indenter, the reaction force observed is maximum. At 90°, the material is more readily pushed "away" because there is less of a constraint in the 1-direction than in the 3-direction. So more of the material is escaping out of the region of contact and therefore the reaction force is lower than at 180°. The interesting point is at 60° and a bit prior, where there is a significant amount of pile-up (shown in red in Fig. 6-9) between about 15°-30°. Here all of the displaced material is being pushed upward, because of the lack of constraint in the 2-direction. The material is initially piled up significantly at 0° similar to the way it is at 180°. When the material finally escapes, it escapes upward, and away from the scratch path. When the rotation reaches approximately 60°, there is no more material remaining to pile-up and the reaction force is therefore, minimized.

Now if the two cases shown in Figs. 6-8D and 6-8E are compared, we see that the peak in 6-8E at 180° is far more abrupt than the peak in 6-8D. The only difference in the anisotropy of these two cases is in the 3-direction, with the material flow in the 3-direction being more "constrained" in Fig. 6-8E. The 3-direction constraint is naturally felt most at 0° and 180° as those are the positions at which the 3-axis is perpendicular to the scratch path. Thus, the pile-up increases at a higher rate (in Fig. 6-8E) leading to a sharper increase in the reaction force, and "dissolves" at a higher rate leading to the sharper decrease.

Although the out-of-plane anisotropy of the sample has a significant impact on the pile-up behavior during testing, the reaction forces observed at the key positions (90° and 180°) are
less affected. Consequently, through calculation of the ratio between the reaction forces at these positions, the in-plane anisotropy of the sample can still be calculated within a reasonable degree of error. Table 6-2 shows tabulated results for the examined cases with anisotropic yield properties both in-plane and out-of-plane. In most of the cases, the in-plane anisotropy ratio is predicted to a minimal degree of error, with the only cases with significant error (> 5%) being those highlighted in red in Table 6-2. These outliers are not of major concern as the presented cases suggest a very large degree of anisotropy in the yield stresses in the three orthogonal directions. The technique has thus been shown as a useful characterization tool for describing anisotropy in the in-plane yield stress ratios in a sample given minimum information about the material preceding testing.

<table>
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<tr>
<th>$R_{11}$</th>
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<th>$R_{33}$</th>
<th>$\mu$</th>
<th>RF$_{Mag}$ @ 180°</th>
<th>RF$_{Mag}$ @ 90°</th>
<th>Ratio</th>
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Table 6-2: Tabulated results of the reaction force tangent to the scratch path resulting from the FEM simulation of a circular scratch test. Loads are tabulated for scratch tip positions of 180° and 90° for different input yield stress ratios incorporating both in-plane and out-of-plane anisotropy. Error percentage between input properties and calculated properties are also tabulated.

6.4. SUMMARY/CONCLUSIONS

In this chapter, a technique was presented for the characterization of in-plane plastic anisotropy in a material sample using a circular scratch experiment. Finite element simulations were implemented to design virtual experiments and to determine the validity of the proposed experiment. The experiment is simple in its formulation; a Rockwell C scratch tip is initially lowered to a maximum depth and subsequently rotated 360°, in a circular path, along the surface
of the sample. The sole factor that must be considered during testing is that the depth must be held constant, and the maximum depth must be chosen so that fully developed plasticity is achieved beneath the scratch tip. Having satisfied the above condition, through successful monitoring of the magnitude of the in-plane reaction force as a function of angle of rotation of the scratch tip, the in-plane anisotropic plastic property (anisotropic yielding) of a sample can be assessed with a high level of accuracy.

Currently, most scratch testing devices used in the field of tribology are based on load-control as opposed to displacement control. In line with ASTM standards [115], scratch tests generally fall under two distinct categories: (i) Progressive load scratch tests (PLSTs) and (ii) Constant load scratch tests (CLSTs). The former requiring an applied ramp load with a specified loading rate and a constant lateral displacement of the tip with respect to the sample, and the latter requiring a constant applied load. As variations in scratch depth would significantly affect in-plane reaction force, the experimental setup of current scratch testing apparatuses would need slight modification. Figure 6-10 shows a simple schematic representation of a proposed experimental setup.

The Rockwell C tip is initially pressed to a depth $h$ into the sample, or alternatively, the sample is raised by a level platform a distance $h$ into the completely fixed scratch tip. After the depth $h$ is achieved, the tip is completely constrained, preserving a constant depth and preventing the tip from moving laterally. The sample is then rotated with a constant angular velocity, $w$, through 1 complete rotation, and the in-plane forces on the scratch tip are measured via a multi-axial force measurement system. As of now, such an experimental apparatus does not exist, however the design and production of such equipment is not impractical given the current commercial availability of highly-capable pin-on-disk and ball-on-disk testers.
Figure 6-10: Schematic cartoon of an experimental setup for a depth-controlled circular scratch testing system. The scratch tip is lowered and held at a constant depth \( h \) and the sample beneath is rotated through one cycle at a constant angular velocity, \( w \).
CHAPTER 7. CONCLUSIONS & FUTURE WORK
In the present work, the goal was to discover a new method of characterizing anisotropic plastic behavior using modified indentation-/contact-based techniques. A series of computational models were designed using the finite element method, and presented for the purpose of characterizing anisotropic plasticity in material systems in which little material information is known before testing. In preliminary studies, it was shown that anisotropic plasticity can exist in materials with suggestive microstructures that have been subjected to a history of plastic working, e.g. thermal spray coatings, and that these properties are revealed through errors in elastic modulus measurement via indentation. As the work evolved further, it was revealed that indentation stress-strain behavior of materials with anisotropic yielding was seen to be inconclusive when characterizing the degree of anisotropic behavior. However, it was discovered, through examination of material flow in the vicinity of contact, that anisotropic properties lead to an inherent constraint in the plastic flow of material. This lead to the perception that the most plausible candidates for successfully characterizing this property would be experiments in which significant plastic flow is induced and mechanical constraints are minimized.

With these points in mind, a number of experiments were planned and tested through parametric analysis and virtual experiments using FEM. The techniques proposed in the preceding chapters covered a number of different cases: (i) indentation near a free edge, (ii) indentation near a free corner, (iii) indentation of a bonded-interface sample, (iv) linear and circular scratch tests. The latter two proved as the most useful tools for determining the out-of-plane and in-plane plastic anisotropy, respectively. While the other methods were also able to provide conclusive results, their experimental feasibility has yet to be proven. Nevertheless, the ultimate goal of the present work was achieved; techniques were developed to characterize the
anisotropic yielding in a sample using single tests which required minimal sample preparation. As for the others, the theoretical framework of the tests has been laid out, and future work would be necessary to develop their experimental practicality.

The work presented in this dissertation can be expanded by performing a systematic set of experiments, based on the present computational results, on materials with known degrees of anisotropic yielding. Unfortunately, testing on sheet metals would not be advised as the anisotropy is highly dependent on the forming history of the sheet. Thus, it is highly unlikely that two samples will have the same anisotropic properties, and the use of tensile tests would need to precede the indentation-based testing. Perhaps a better method would be to produce composite materials in a specific way to promote a specific level of anisotropic yielding. With samples in hand, the proposed techniques that have not been validated experimentally could be verified. In addition, work could be done for the design of new experimental test fixtures; both for straightforward indentation on a free edge/corner, and for constant-depth circular scratch coupled with multi-axial force measurement. Devices could then be used to verify, experimentally, the findings proposed in this dissertation and ultimately be marketed to instrumentation companies to promote commercial availability and applicability of such testing techniques.
REFERENCES


142


PUBLICATIONS


