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An Investigation of the Formability of ZEK100 Mg Alloy Using Pneumatic Bulge Formability Testing Methods

John Briou Bourgeois

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An investigation of the formability of ZEK100 Mg alloy using pneumatic bulge formability testing methods.

By

John Briou Bourgeois

A Thesis
Submitted to the Faculty of Mississippi State University in Partial Fulfillment of the Requirements for the Degree of Master of Science in Mechanical Engineering in the Department of Mechanical Engineering

Mississippi State, Mississippi
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John Briou Bourgeois

2016
An investigation of the formability of ZEK100 Mg alloy using pneumatic bulge formability testing methods.

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The current study investigates the formability of ZEK100, a rare-earth containing magnesium alloy, using an in-house developed technique of pneumatic bulge forming. The thesis pursued innovation of sample preparation, testing, and experimental data analysis in order to create several forming limit diagrams (FLDs) of critical importance for determining a methodology for Mg formability. Samples were bulged through elliptical and circular dies at room temperature, 150 C, and 250 C, in two orientations, rolling direction (RD) and transverse direction (TD), in order to determine temperature dependence and orientation characteristics. The current research concluded ZEK100 is not a suitable alloy for room temperature forming processes used in automotive industries. Little difference between safe and marginal, as well as marginal and failure strain ratios was seen for RD orientation testing, while greater resolution is evident for TD orientation testing. ZEK100 exhibits a temperature dependence in relation to limiting strain between RD and TD.
DEDICATION

This work is dedicated to my family, and specifically to my wife Chelsea Rae Bourgeois. Without the support and inspiration you have given me throughout graduate school, I would not be where I am today. Thank you for your loving care and support.
ACKNOWLEDGEMENTS

I would like to thank the Center for Advanced Vehicular Systems (CAVS) at Mississippi State University for providing me with the opportunity to study as a graduate research assistant in the pursuit of my graduate degree.

Effort sponsored by the Engineer Research & Development Center under Cooperative Agreement number W912HZ-15-2-0004. The views and conclusions contained herein are those of the authors and should not be interpreted as necessarily representing the official policies or endorsements, either expressed or implied, of the Engineer Research & Development Center or the U.S. Government.
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CHAPTER I

INTRODUCTION

Background

As the demand for more fuel efficient vehicles grows, automobile manufacturers look to lightweight materials in order to reduce vehicle mass, in turn increasing fuel efficiency. One such lightweight material, magnesium, is the subject of a large amount of research [1] [2] [3]. Different magnesium alloys have been investigated as potential materials for use in automotive body panels[1] [2] [4] [5] [6] [7] [8]. The focus of these investigations has been the formability of the alloy in question. Due to certain material characteristics, discussed below, magnesium alloys typically exhibit less than desirable formability characteristics at room temperature, as compared to current steel and aluminum materials [1]. It has been seen that at higher temperatures, common magnesium alloys, such as AZ31 and others, exhibit excellent formability characteristics [1] [2].

In order for a magnesium alloy to be considered for use in the manufacture of automotive body panels, it must be formable at room temperature or slightly above room temperature. Magnesium alloys which exhibit good forming characteristics only at highly elevated temperatures are not an economical option for automobile manufacturers due to the increased cost of hot forming operations. A limited number of deformation modes, only basal slip and twinning, are active at room temperature, which limits the
ductility and thus formability of Mg alloys\[1\] \[9\] \[10\] \[11\]. At elevated temperatures, up to 400°C, it has been seen that the formability of magnesium alloys is significantly increased due to the fact that the five deformation modes including basal slip, prismatic slip, pyramidal slip, tensile twinning and compressive twinning are activated at these elevated temperatures \[1\] \[2\] \[8\] \[11\] \[12\] \[13\]. Strain rate as well as sheet orientation are also important factors in the formability of magnesium alloy sheets. It has been seen in previous studies that as the strain rate is decreased, the formability of magnesium sheet alloys generally increases \[1\] \[8\] \[11\] \[14\]. It has also been seen that as forming temperature increases, Mg alloys exhibit an increased strain rate sensitivity during forming processes, with higher strain rates producing less deformation prior to failure \[1\] \[14\].

The orientation of the sample in relation to the rolling direction of the sheet plays a role in formability as well. This orientation dependence is due to the anisotropic characteristics of magnesium alloys, as described in literature \[15\] \[16\]. Abu-Farha et al. \[1\] noted that greater strains can be reached prior to failure when the major axis of deformation is in line with the rolling direction of the magnesium alloy sheet.

A majority of formability studies performed have used what is known as a limiting dome height (LDH) test \[17\]. In this test a hemispherical punch is used to deform several material samples of different widths. As the widths of the samples change, the major to minor strain ratio changes as well. This change in strain ratio provides different major and minor strain values needed to generate a forming limit diagram (FLD). The punch is generally used in a load frame in order to control and monitor punch force as well as strain rate. The test samples are securely clamped as to
allow no material slip during an LDH test. The standardized LDH test procedure can be carried out in a wide range of load frames making it an attractive test process to many researchers.

Though the LDH test is standardized and widely accepted there are some drawbacks. One problem, as reported by Karthik et al. [18], is the inherent laboratory-to-laboratory scatter. Karthik et al. [18] goes on to explain issues caused by the boundary conditions at the edges of the sample being poorly controlled and characterized. Abu-Farha et al. [1] [2] describes an important problem with the LDH test to be frictional interactions between the sample and the punch as well as between the sample and the die. The interactions between the sample and the punch and/or die have been known to cause failure away from the center of the sample [1]. These failures do not depict the true formability of the material and thus are not used in constructing forming limit diagrams. In an effort to reduce these frictional effects, lubrication is applied, however consistent lubrication from test to test is difficult to achieve [19]. Furthermore, the failure criterion for the LDH test is not clearly defined [19]. The lack of a clear definition of failure leads back to the large laboratory-to-laboratory scatter as described above.

Work has been done to develop a new test apparatus in order to mitigate some of the inherent limitations associated with the standard hemispherical punch LDH test [1] [2] [8]. Abu-Farha et al. [1] [2] addressed several of these issues in previous studies. The most significant issue addressed by the new apparatus is the effect of friction during a test. Unlike the LDH test, the new test, referred to here as Pneumatic Bulge Forming (PBF), does not use a punch to deform the material samples. Each sample is placed in a bulge chamber, into which high pressure gas, in most cases argon, is flowed. The
pressure of the forming gas forces the sample to bulge through a die stretching the material bi-axially. A series of elliptical dies is employed, each with a different major-minor axis ratio. The differing major-minor axis ratios provide a greater range of strain ratios to more fully develop the forming limit diagram. A second advantage of this system is the use of samples with a single geometry. The samples for this type of testing are all the same size and shape regardless of the die geometry being used. This single geometry sample reduces the possibility of errors in machining different sized samples, as with LDH testing. It is thought that by eliminating frictional effects, and using a single geometry for samples, the laboratory-to-laboratory scatter will be decreased [1] [19].

As of yet, few drawback have been found in using the PBF testing apparatus and method, but they do exist. One such drawback is the fact that the PBF test does not produce any negative minor strain. Due to this lack of negative minor strain, standard dog bone style tensile tests are needed to produce the negative strain values seen on the left hand side of the FLD. Furthermore, strain-rate is not easily controlled unless a closed-loop pressure control system is used. Though closed-loop pressure control systems are commercially available, they may not be used in every laboratory. Without a closed-loop pressure control system, strain-rate must be calculated once the test is completed.

**Current Work**

The current study investigates the formability of a magnesium alloy known as ZEK100, using a pneumatic bulge forming process similar to that used by Abu-Farha et al. [1] [2]. The ZEK100 supplied to Center for Advanced Vehicular Systems (CAVS) at
Mississippi State University has a typical composition, shown below in Table 1, as found in literature [20].

Table 1   Table of the typical composition of ZEK100 as found in literature [20].

<table>
<thead>
<tr>
<th></th>
<th>Zn (wt.%)</th>
<th>Zr (wt.%)</th>
<th>Nd (wt.%)</th>
<th>Ce (wt.%)</th>
<th>La (wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bal</td>
<td>1.34</td>
<td>0.230</td>
<td>0.182</td>
<td>0.008</td>
<td>0.001</td>
</tr>
</tbody>
</table>

The more randomized textured of the ZEK100 alloy, created by the rare earth (RE) alloying, shows promise for an improved forming characteristic, allowing for greater deformation at room temperature prior to failure. Though the texture of the ZEK100 is more randomized, it has been shown that it still exhibits strong in-plane anisotropy, meaning that there are changes in ductility, yield strength, and hardening rates depending on sheet orientation in relation to the rolling direction (RD) [9].

Samples taken from ZEK100 sheets were prepared, tested, and analyzed in order to determine the formability characteristics of the alloy. Testing was carried out at room temperature as well as at elevated temperatures. The purpose of the elevated temperature testing was to investigate the temperature dependence characteristics of the ZEK100.
CHAPTER II
EXPERIMENTAL METHODOLOGY

Bulge Die Apparatus

The bulge die experimental apparatus was designed based on the previous work of Abu-Farah [2] [1]. The complete apparatus, seen in Figure 1, consists of four major parts: the die base, the die cap, the dies, and the pneumatic control system. Machining of the die base and die cap took place in-house at the CAVS facility at Mississippi State University. A 4” diameter cylindrical 4140 steel billet was used as the starting material. The center of the die base was first drilled out using a drill bit with a diameter of 1”. Following drilling, a series of boring bars was used to produce a final inside diameter of 3.25”. The bottom of the die base was turned down to fit into a section of pipe to be used as the die base stand-off. The die cap was turned down in a similar manner in order for a second section of pipe to be used as the die cap stand-off. The die base and die cap stand-offs allow the entire apparatus to be fitted to an Instron 5882 load frame. The apparatus was designed and built to fit within an environmental chamber used in conjunction with the Instron 5882 load frame. This environmental chamber is capable of reaching temperatures up to 600°C, giving researchers the ability to test materials in a wide range of temperatures.
The system consists of four parts: the pneumatic control system, the die cap, the dies, and the die base.

After the die cap was turned down, a hole was drilled in its center and tapped to accept a 0.25” NPT threaded pipe. This pipe is used to plumb the forming gas from the source to the upper die. Every piece of plumbing hardware selected has a rating of 10,000 psi or greater in order to ensure the safety of the test operator as well as nearby researchers and equipment. Alignment holes were drilled in both the die base and die cap, and alignment pins inserted. These alignment pins ensure the die base, die cap, and dies, as well as the sample to be tested, all align properly during testing.

Figure 1  Photograph of the bulge die formability apparatus
The dies used in the apparatus, seen in Figure 2, were machined off site by Tombigbee Tooling Inc. [21]. The out sourcing of the dies was done to obtain accuracy not possible with current in-house methods of machining. Four pairs of dies were made, each with an opening of specific elliptical geometry with aspect ratios of 1.0, 0.8, 0.6, and 0.4. The entry of each die had a 0.25” radius fillet machined into it and finely polished. It is crucial for the entry of each die to be polished and clean of any debris to prevent failure of the test specimen at this point. During testing, one die was placed below and above the specimen in order to promote uniform clamping force. One die from each pair was further machined in-house with a series of grooves in order to promote a better seal between the sample and the upper die. These grooves act similar to a draw bead, commonly found on the more standard LDH formability test.

![Figure 2](image.png)

Figure 2  
The four elliptical die sets, including an upper and lower die, used in testing have major-minor axis ratios of 1.0, 0.8, 0.6, and 0.4.

A relatively simple pneumatic system was employed to serve as the forming pressure control. The system consisted of a high pressure argon tank capable of pressures
up to 6000 psi, a pressure regulator, a flow rate control valve, and miscellaneous piping and fittings. The pressure at which the forming gas enters the upper die is controlled by the pressure regulator. The flow rate control valve is used primarily as a safety shut off in case of a leak or any unexpected problem.

Sample Preparation

The ZEK100 material was received in 12” x 12” x 0.079” sheets. These sheets were cut into 4” x 4” squares. These squares were then sent out to be precision ground to a thickness of 0.039”, roughly 1 mm. Material was ground from both the top and bottom sides of the sheet in order to ensure the center section of the sheet thickness was tested. Due to the fact that the samples were ground on both the top and bottom of the sheet, the markings used to determine orientation with relation to the rolling direction were lost. The loss of the orientation marks meant each sample needed to be analyzed via optical microscopy (OM) in order to determine the rolling direction of the sheet. For the OM analysis one corner of each sample was cut off and mounted in an acrylic mount. Each mounted sample was then polished and etched following standard preparation techniques. The etchant used was composed of 2.5 grams of picric acid, 100 mL of ethanol, 25 mL of acetic acid, and 25 mL of water. This etchant was designed to highlight the individual grains in the Mg samples. Each prepared sample was observed using an Axiovert OM, with grain elongation being the focus of the observation. The rolling direction was determined by observing the direction in which the grains were elongated. Once the rolling direction of each sample was established, four alignment holes were drilled in each sample with the same geometry as the alignment pins in the bulge die system. After
machining, scratches were removed from each sample using SiC paper, starting at 220 grit and working up to 2000 grit.

A grid pattern was then applied to each sample using a system from Lectroetch [22], seen here in Figure 3. This system uses a roller to pass current through a grid stencil of 0.1 in diameter circles in order to etch the circles onto each sample. Lectroetch LNC4 was the electrolyte used in this etching process. The grid is used later to measure local strain after the samples have been deformed in a test. After the grid application, each sample was examined prior to testing to ensure the circles in the grid were consistent and accurate. Figure 4 shows an example of a fully prepared sample awaiting testing. Ten grid circles from each sample were measured prior to testing in order to have an average initial grid size for each sample.

Figure 3  The Lectroetch grid marking system was used to apply a grid of circles having a nominal diameter of 0.1” on each sample prior to testing [22].
Figure 4  An example of a ZEK100 bulge die testing sample which has been cut, ground, polished, machined, and grid marked, and is ready for testing.

**Experimental Procedure**

After sample preparation was completed and initial grid size measurements were taken, the samples were ready for testing. For each round of testing, a test matrix was employed, which prescribe the test conditions for each sample. An example of one such test matrix is seen here in Table 2. In each round of testing, 24 samples are tested. Six samples are tested for each die ratio with orientation and temperature varying between them. A constant strain rate, with a target of $10^{-2} \frac{1}{s}$, was maintained to the best of the ability of the pneumatic pressure control system. In the test matrix shown in Table 2, three temperatures were used: RT (approximately 23°C), 150°C, and 250°C. These three temperatures were chosen in order to study the formability of the material in question in conditions closer to that of real world manufacturing. For elevated temperature testing, the test apparatus was brought up to temperature over the course of 3.5 hours and allowed
to soak for one additional hour once the final temperature was reached. Each sample was allowed to reach thermal equilibrium by allowing it to sit at temperature for 10 minutes prior to forming. A soak time of 30 minutes was used when new dies were inserted into the testing apparatus in order to ensure the entire test system is up to temperature. A thermocouple fixed to the test apparatus adjacent to the sample location was used to ensure an accurate temperature reading.
Table 2 Table depicting the test matrix used for the bulge die formability testing of ZEK100 in both the rolling direction (RD) and transverse direction (TD) orientations at three temperatures.

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Die Ratio</th>
<th>Orientation</th>
<th>Temperature</th>
<th>Sample #</th>
</tr>
</thead>
<tbody>
<tr>
<td>TD</td>
<td>RD</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>1*</td>
<td></td>
<td>Room Temp</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>1*</td>
<td></td>
<td>T2 150°C</td>
<td>3</td>
</tr>
<tr>
<td>3</td>
<td>1*</td>
<td></td>
<td>T3 250°C</td>
<td>5</td>
</tr>
<tr>
<td>4</td>
<td>1</td>
<td>*</td>
<td>*</td>
<td>2</td>
</tr>
<tr>
<td>5</td>
<td>1</td>
<td>*</td>
<td>*</td>
<td>4</td>
</tr>
<tr>
<td>6</td>
<td>1</td>
<td>*</td>
<td>*</td>
<td>6</td>
</tr>
<tr>
<td>7</td>
<td>0.8*</td>
<td></td>
<td></td>
<td>7</td>
</tr>
<tr>
<td>8</td>
<td>0.8*</td>
<td></td>
<td>*</td>
<td>20</td>
</tr>
<tr>
<td>9</td>
<td>0.8*</td>
<td></td>
<td>*</td>
<td>18</td>
</tr>
<tr>
<td>10</td>
<td>0.8</td>
<td>*</td>
<td>*</td>
<td>8</td>
</tr>
<tr>
<td>11</td>
<td>0.8</td>
<td>*</td>
<td>*</td>
<td>21</td>
</tr>
<tr>
<td>12</td>
<td>0.8</td>
<td>*</td>
<td>*</td>
<td>22</td>
</tr>
<tr>
<td>13</td>
<td>0.6*</td>
<td></td>
<td></td>
<td>9</td>
</tr>
<tr>
<td>14</td>
<td>0.6*</td>
<td></td>
<td></td>
<td>17</td>
</tr>
<tr>
<td>15</td>
<td>0.6*</td>
<td></td>
<td>*</td>
<td>24</td>
</tr>
<tr>
<td>16</td>
<td>0.6</td>
<td>*</td>
<td>*</td>
<td>10</td>
</tr>
<tr>
<td>17</td>
<td>0.6</td>
<td>*</td>
<td>*</td>
<td>19</td>
</tr>
<tr>
<td>18</td>
<td>0.6</td>
<td>*</td>
<td>*</td>
<td>25</td>
</tr>
<tr>
<td>19</td>
<td>0.4*</td>
<td></td>
<td></td>
<td>11</td>
</tr>
<tr>
<td>20</td>
<td>0.4*</td>
<td></td>
<td>*</td>
<td>15</td>
</tr>
<tr>
<td>21</td>
<td>0.4*</td>
<td></td>
<td>*</td>
<td>26</td>
</tr>
<tr>
<td>22</td>
<td>0.4</td>
<td>*</td>
<td>*</td>
<td>12</td>
</tr>
<tr>
<td>23</td>
<td>0.4</td>
<td>*</td>
<td>*</td>
<td>16</td>
</tr>
<tr>
<td>24</td>
<td>0.4</td>
<td>*</td>
<td>*</td>
<td>27</td>
</tr>
</tbody>
</table>

To begin testing, a sample was placed in the die and the environmental chamber was closed around the entire apparatus. The sample was allowed to reach thermal equilibrium, as described above, and a clamping force was applied using the load frame. It should be noted that the alignment pins discussed in the experimental apparatus section are used to ensure proper sample orientation, whether it be in the rolling direction (RD).
or transverse direction (TD). A specific loading method was developed using the Instron Bluehill 2 software [23], which applies a compressive load between 65 kN and 85 kN chosen depending on material, temperature, and die geometry, followed by a compressive displacement of 0.02 mm. The compressive displacement is then held in order to maintain proper clamping load once the forming gas is introduced to the system. Upon reaching the proper clamping load, forming gas was introduced into the upper die, forcing the sample to deform through the lower die. As described in the experimental apparatus section of this report a simple hand-controlled pressure control system is used to deform each sample. In the current research, the temperature of the forming gas is not monitored. The amount of argon pressure used to deform the sample was controlled by adjusting the pressure control knob on the pressure regulator. Forming gas pressure was steadily increased until the sample failed releasing pressure into the die base. This method does not produce a perfectly constant strain-rate, described by Abu-Farha et al. [1][2], though without a closed-loop control system capable of producing a constant strain rate, this method was sufficient for current research. Sample failure was easily detected by a sudden drop in pressure seen at the pressure gauge located on the pressure regulator.

Once the sample failed, the forming gas pressure was released into the atmosphere through the pressure regulator. Next, the environmental chamber was opened and removed from the apparatus. The clamp load was then removed from the die system allowing the die cap and upper die to be removed, followed by the sample. Any pieces of the sample which became detached during testing were retrieved from the die base and set aside with the sample. From this point a new sample was loaded and the system reset.
for another test. For elevated temperature testing, personal protective equipment was used in order to avoid any personal injury or equipment damage.

After testing, the samples were analyzed in order to recover the strain data needed for construction of the FLD. If no fracture or tear was present in the sample, the thickness of the sample was measured using a micrometer. Several thickness measurements were taken throughout the bulged area of the sample. The center of the bulge was generally the area that exhibited the most thinning through the thickness. The thickness measurements were used to determine if the sample had been thinned enough to be considered necked. After the thickness of the sample is measured, specific grid circles of interest were marked for further analysis. The grid circles chosen for further analysis were marked as “non-failed” data points. In cases where a fracture or tear was present in center of the sample, the grid circles touching the fracture were marked as “failure,” the grid circles adjacent to the “failure” grid circles were marked as “marginal,” and finally the grid circles adjacent to the “marginal” circles were marked as “safe”. In cases where the failure did not occur in the center of the sample, but occurred along the die shoulder, that sample was considered as exhibiting “shoulder failure”. The differences between “non-failed,” “failure,” “marginal,” “safe,” and “shoulder failure” zones are discussed in the results and discussion section below.

Once the grid was marked appropriately, strain measurements were taken. In the current research, a handheld digital microscope was used, commercially available from Dino-Lite [24] shown here in Figure 5. The microscope was calibrated using a calibration standard provided by the manufacturer prior to use. The microscope was
calibrated at the magnification that was to be used during strain measurement, in order to obtain the most accurate measurements.

Figure 5  A ZEK100 bulge die sample after testing which has designated failure, marginal, and safe regions marked in preparation for strain measurement with the hand held digital microscope pictured behind the sample.

Prior to bulge testing, ten grid circles from each sample were measured to provide an average non-deformed grid size to be used in strain calculation. The grid circles marked as “non-failed,” “failure,” “marginal,” and “safe,” were inspected individually using the handheld microscope. Images of each specified grid mark were captured for measurement. Using the still images, measurements were taken in both the major and minor axis, using the DinoCapture2.0 [24] software. To measure each grid mark, a line
was drawn across the inside diameter of the grid mark along the major axis, as well as the minor axis, where the minor axis is defined as being perpendicular to the major axis. Figure 6 below, shows an image taken of a single grid mark after the major and minor axis have been measured. The length of these diameters are used along with the initial grid mark diameter to calculate strain in accordance with ASTM E2218-15 [17]. The equation used can be seen below.

\[
\text{Major Strain} = \frac{L_f - L_o}{L_o} \times 100 \\
\text{Minor Strain} = \frac{W_f - W_o}{W_o} \times 100
\] 

where \( L_o \) is original length, \( L_f \) is final length, \( W_o \) is original width, and \( W_f \) is final width.

Once the major and minor strains were calculated for each selected grid circle, the data points were plotted according to their designations. Further discussion of the data plots can be found in the results and discussion section of this report.
Results and Discussion

The calculated major and minor strains were plotted in accordance with their previously determined designation (failure, marginal, safe, and non-failure). Failure points were plotted as red squares, marginal as blue triangles, safe as green dots, non-failure as purple diamonds. In the current study, six plots were created. Two plots were created for each of the three temperatures tested; one for samples aligned in the RD and one for samples aligned in the TD. In the following section, comparisons are made
between the RD and TD plots for each temperature, as well as between the plots of one temperature to another.

Room temperature testing of the ZEK100 proved to be challenging. In room temperature testing, samples failed primarily at the shoulder of the die entry, regardless of rolling direction or aspect ratio. Though a wide array of clamping forces were used, room temperature samples did not fail in the center of the deformed area. As seen in the two left most columns of samples in Figure 7, and in greater detail in Figure 8, all room temperature samples show failure at the shoulder of the die entry. This failure is thought to be the result of a combination of the biaxial stress components, stretching in the x and y directions as well as a bending stress in the z direction creating a stress triaxiality. The bending stress is experienced through the thickness of the sample, causing a shear driven failure.

Given the results of this testing the conclusion can be made that ZEK100 exhibits poor room temperature formability similar to that of other magnesium alloys [1] [2] [3] [4]. The data obtained from room temperature tests is believed to be unreliable due to the fact that it does not capture the strain associated with triaxial stress state, which caused the shoulder failure. Because of this lack of reliable data, the FLDs for room temperature tests have not be included in this report.
Figure 7 The 24 ZEK100 samples used in developing forming limit diagrams for room temperature, 150°C, and 250°C, with the major die axis aligning in both the rolling direction (RD) and the transverse direction (TD).
The eight samples tested at room temperature all exhibited failure at the entry shoulder of the die. This failure is not classified as a successful test, therefore the strain results of the room temperature test are not reported here.

Elevated temperature testing produced much more useful results for FLD construction than those results produced from room temperature testing. The vast majority of samples tested at both 150°C and 250°C failed in the center of the deformed area. The data extracted from the elevated temperature testing is much more reliable and meaningful than that from the room temperature testing. The primary focus of this report, therefore, is the resulting data from testing performed at 150°C and 250°C.

The resulting data from the current study provides data only for the right hand side of the FLD. Figures 9 and 10, seen here, show the data taken from the testing done at 150°C. In both plots, four types of data points are plotted: failure, marginal, safe, and
non-failed sample. The failure, marginal, and safe data points were chosen and plotted as stated above. The non-failed sample data points were picked at random from a sample that did not fail during testing. The non-failed sample did not rupture due to a leak in the pneumatic system caused by a lack of clamping load. Instead of removing the sample completely, it was decided to include the data as it provides further insight into the formability of the material, unlike the shoulder failure data points. The non-failed sample data is interpreted as safe data points because strains were not high enough to cause a failure, including necking, in the sample.

Figure 9  Graph depicting strain data taken from samples tested at 150°C with the major die axis aligned with the rolling direction.
In Figure 9 the lines between safe and marginal, as well as marginal and failure zones are not well-defined. Marginal data points exist in both the failure region as well as in the safe region. The failure and safe regions of the plot are so closely spaced it is almost as if a specifically marginal region does not exist at 150°C in the RD orientation. In comparison Figure 10 shows a more distinctive marginal region. The failure region is distinctively separated from the safe region in this FLD with the marginal region separating the two. It is also important to note that though there is a better separation between safe and failure zones in the TD orientation, the failure strains experienced in this orientation are much lower than those experienced in the RD orientation. It is understood, that ZEK100 at 150°C exhibits greater formability when the major die axis is aligned with RD of the material even though the marginal region is small in comparison.
to that of the TD orientation. In the RD orientation safe major strains reach nearly 40% with minor strains ranging up to roughly 35%, where as in the TD orientation safe major strains only reach 15% with minor strains extending no further than 12%. This difference in safe zone strain between the two orientations show that a distinct in-plane anisotropy exists at 150°C even with the weaker, randomized texture created by the rare earth alloying elements in the ZEK100.

The FLDs resulting from the 250°C tests show similarities to those from 150°C testing. Shown here in Figures 11 and 12 are the FLDs for the 250°C testing. Similar to the results from the 150°C testing in the RD orientation, the lines between the safe and marginal zones, as well as between the marginal and failure zones are nearly indistinguishable. As seen in Figure 11 there are failure data points intermixed throughout what would be called a marginal region. When examining the formability of ZEK100 it is important to keep these lower strain failure points in mind. While the intermixed failure points may not represent the actual forming limit, they must be kept in mind when developing forming processes.
Figure 11  Graph depicting strain data taken from samples tested at 250°C with the major die axis aligned with the rolling direction.
Figure 12 shows the FLD for tests run at 250°C with the samples aligned in the TD orientation. In this orientation, like the results of the 150°C TD test, the failure, safe, and marginal regions are more easily discerned. There is significantly less intermingling of failure and marginal data points. There does exist, however, a great deal of intermixing of safe and marginal zone data points. The lack of a distinctive marginal zone poses as a challenge when determining the forming limit of this material. Unlike the 150°C testing, there is little difference in the strain levels reached by samples in the TD orientation when compared to those reached by samples in the RD orientation. This decrease in orientation dependence at 250°C shows that the in-plane anisotropy of ZEK100 is temperature sensitive and decreases as temperature is increased.
In all plots, failure data points are seen intermixed with both the safe and marginal regions. These outlying failure points are likely caused by flaws in the original sheet material, such as voids or contaminate particles. These failure points could also be taken from a secondary tear in the sample caused after the initial crack spread. Given this uncertainty in the interpretation of these outlying failure points, in the current study they are believed to have little effect on the overall average FLD result, but must be considered nonetheless. At this time no meaningful correlation can be made between these outlying failure points and testing temperature or orientation.

**Conclusions**

The current research has led to the following conclusions: ZEK100 does not exhibit acceptable formability characteristics at room temperature for use in the automotive industry. In instances when the major die axis is aligned with the rolling direction of the sheet there is little difference between safe and marginal strain ratios, and likewise with marginal and failure strain ratios. A greater distinction between safe, marginal, and failure strain ratios is evident when the major die axis is aligned with the transverse direction of the sheet. ZEK100 exhibits a temperature dependence in relation to limiting strain, as temperature increases the difference in limiting strain between RD and TD alignment decreases.
CHAPTER III
FUTURE WORK

As research progresses a wide variety of materials can become the subject of bulge die formability testing. The current system is not limited to any one specific metal alloy. Future work investigating a family of third generation advanced high strength steel alloys is likely. In order for steel alloys to be studied though, greater clamping load is needed during testing. The current bulge die apparatus can easily be modified to be used in conjunction with a larger load frame, capable of greater clamping load than that of the load frame used in the current study.

The greatest improvement to be made to the experimental system would be to implement a closed-loop electro-pneumatic control system. The implementation of a closed-loop control system would vastly increase the precision as well as accuracy of strain rate control during testing. With a digitally controlled pneumatic system, the closed form solution for bulging through an elliptical die, developed by Banibic et. al, [25] can be employed to control the forming pressure. Using this closed form solution, shown below, in conjunction with the digital control system allows forming pressure-time profiles to be created and followed resulting in a constant or near constant prescribed strain rate [1] [25]. Having a prescribed and constant strain rate would be an improvement over the current hand-controlled strain rate which is calculated after each test has been completed.
\[ P(t) = \frac{2s_0\bar{\sigma}1+\alpha k^2}{b_0 a_0} \frac{1}{\rho} e^{-3\bar{\varepsilon}t/2\rho} e^{(2-\alpha)/2\rho} \left( e^{(2-\alpha)/2\rho} \bar{\varepsilon}t - 1 \right) \]  

(3)

\[ k = \frac{b_0}{a_0}, \quad \alpha = \frac{1}{2} (1 + e^{1 - (1/k)}), \quad \rho = \sqrt{1 - \alpha + \alpha^2} \]  

(4)

where \( P \) is the forming pressure, \( t \) is the forming time, \( \bar{\sigma} \) is the effective stress, \( \bar{\varepsilon} \) is the effective strain rate, \( s_0 \) is the initial sheet thickness, \( a_0 \) and \( b_0 \) are the major and minor half axis of the elliptical die [1] [25].

An additional improvement which could be made to the system would be the incorporation of a digital image correlation (DIC) system. Having the ability to track certain measurements during a bulge test, via a remote camera, would allow for real time strain data acquisition. This real time strain data could then be used to preform interrupted test, allowing greater material characterization throughout the forming process. Having the ability to stop a test at certain strain levels and analyze the material could shed new light on the deformation mechanics of the material.
REFERENCES


