AC 2012-4044: FACTORIAL DESIGN OF EXPERIMENTS FOR LABORATORIES INCORPORATING ENGINEERING MATERIALS

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Dr. Daniel Edward Ephraim
Factorial Design of Experiments for Laboratories Incorporating Engineering Materials

Abstract

Engineering laboratory experiments that involve materials and/or material properties are often designed to establish a level of specification and implementation methodology. Often these laboratory experiments are developed for well defined systems in controlled environments to take advantage of limited resources (i.e., materials, testing supplies, laboratory space, time, etc.). Material systems that incorporate a dependence on more than one parameter for processing and subsequent characterization pose a significant problem in that the experiment designer may not possess the information to identify the key parameters that influence the critical properties sought after. The ultimate goal is for the student experimental designer to predict parameters and properties based on a limited number of experiments or available data.

The proposed methodology in this paper describes a general full factorial design for experiments involving the processing of materials for characterization used in undergraduate mechanics and materials laboratories. This Factorial Design Analysis (FDA) approach facilitates a ‘between-participants’ design analysis that includes more than one independent variable, and has the advantage over a simple randomized design in that one can test the effect of more than one independent variable and the interactive effect of the various independent variables. The method is validated for the optimization of the boundary conditions that influence the material properties of electrodeposited metals. Specifically, a $2^k$ factorial statistical analysis is conducted, analyzed, and a mathematical model derived, to describe how the electrolytes’ boundary conditions influence the mechanical properties of electrodeposited nickel-iron (Ni$_{80}$Fe$_{20}$). Results include the students’ full factorial design of experiments for an upper-level undergraduate engineering materials laboratory and an assessment of the laboratory experience.

Introduction

Laboratory experiments are a critical part of the required curriculum for students seeking degrees in the science, technology, engineering and mathematics (STEM) fields. These laboratory experiments usually involve materials and/or material properties that were designed to establish an level of specification and implementation methodology. However, often these laboratory experiments were developed for well defined systems in controlled environments to take advantage of limited resources such as expensive materials and testing supplies. Material systems that incorporate a dependence on more that one parameter for processing and subsequent characterization pose a significant problem in that the experiment designer may not possess the information to identify the key parameters that influence the critical properties sought after. The ultimate goal is for the student experimental designer to predict parameters and properties based on a limited number of experiments or available data.

Electroplating fabrication processes are generally proposed as one exercise for upper-level undergraduate engineering laboratory experiments in the STEM fields, and this paper describes the methodology and implementation experienced in a specific course (Engineering Materials
Laboratory). Electroplating metals are used in a variety of devices, including high-aspect-ratio and high-density laminated magnetic cores and multi-layered windings for advanced micro-magnetic generators, as developed by Arnold, et al.\textsuperscript{1} For these types of devices, the magnetic and electrical properties of the materials are most critical for performance, however, since these devices involve micro-rotating machinery, the mechanical properties of the materials are also critical for operation and durability. For these reasons, Neodymium Iron Boron (NdFeB) is used as the permanent magnet material of choice, whereas electrodeposited nickel-iron (Ni\textsubscript{80}Fe\textsubscript{20}) is used as the back iron material as well as to fill the cavities between the silicon and the permanent magnet, and to laminate the permanent magnet inside the silicon hub of the ultra high speed heavy rotor, as depicted in Figure 1. Ni\textsubscript{80}Fe\textsubscript{20} has excellent magnetic and electrical properties, however, the mechanical properties of electrodeposited NiFe have not been studied extensively, thus providing an excellent opportunity for upper-level STEM students to engage in relevant, material’s oriented laboratory experiences.

![Electroplated NiFe, Silicon Frame, Silicon Hub, Back Iron, NdFeB PM, Silicon Spokes](image)

**Figure 1.** Heavy Rotor Schematic using NdFeB permanent magnet material laminated in the Silicon Hub with electro-deposited Ni\textsubscript{80}-Fe\textsubscript{20}.

**Electrodeposition of Permalloy Ni\textsubscript{80}Fe\textsubscript{20}**

Electrodeposition is the process used in electroplating, whereas electroplating is the process of using electrical current to reduce metal cations—an atom or group of atoms carrying a positive charge—in a solution and coat a conductive object with a thin layer of metal\textsuperscript{2}. The primary application of electroplating is to deposit layer(s) of a metal having some desired property (example, abrasion and wear resistance, corrosion protection, lubricity, improvement of aesthetic qualities, magnetic, etc.) onto a surface lacking that property. Also electroplating is used to build up thickness on undersized parts, typically the part to be plated is the cathode of the circuit and the anode is made of the metal to be plated on the part. Both components are immersed in a solution called an "Electrolyte" containing one or more dissolved metal salts as well as other ions
that permit the flow of electricity. A rectifier supplies a direct current to the cathode causing the metal ions in the electrolyte solution to lose their charge and plate onto the cathode. As the electrical current flows through the circuit, the anode slowly dissolves and replenishes the ions in the bath. Electroplating has been a process of major significance in the fabrication of thin-film recording heads, which are important components in magnetic recording hardware. The development of electroplating processes for nickel-iron alloys, such as Ni$_{80}$Fe$_{20}$, enabled thin-film recording heads to become technologically viable. With the introduction of thin-film inductive heads and, later, magneto-resistive (MR) heads, the disk-drive field has been able to sustain rapid growth. Magnetic micro-actuators and inductors such as solenoids, valves, and cantilevers are fabricated using electrodeposited Permalloy (Ni$_{81}$Fe$_{19}$).

To fabricate the microstructures by electroplating, a conductive plating base or seed layer (in our case, sputtered nickel) and a means to pattern the electro-deposit (photolithography) are needed. Typically, the electro-deposit is patterned by an additive process (selective deposition) instead of a subtractive process (etching). Since the localized electro-deposition rate is proportional to the localized current density, a uniform current density over the entire seed layer is needed to obtain an electro-deposit having a uniform thickness. To achieve selective deposition, however, portions of the seed layer are covered with an insulating masking material that makes the current density in its proximity non-uniform. The nickel plating solution is shown in Table 1.

Table 1: Nickel-Iron (Ni$_{80}$Fe$_{20}$) plating solution.

<table>
<thead>
<tr>
<th>Material</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel Sulfate</td>
<td>200 g/L</td>
</tr>
<tr>
<td>Nickel Chloride</td>
<td>5 g/L</td>
</tr>
<tr>
<td>Boric Acid</td>
<td>25 g/L</td>
</tr>
<tr>
<td>Ferrous Sulfate</td>
<td>8 g/L</td>
</tr>
<tr>
<td>Saccharin</td>
<td>3 g/L</td>
</tr>
</tbody>
</table>

The cathode and anode remained in the iron electrolyte solution (nickel iron bath) under a constant current, bath temperature, and agitation for approximately four hours. At the end of four hours, five nickel iron test specimens between 25 µm and 75 µm thick—depending on the plating parameters—are cultivated on the copper seed layers of the silicon wafer. Figure 2 shows a Si wafer containing five electrodeposited NiFe test specimens. The wafer was then prepared for chemical etching to remove the test specimens. First, the wafer is rinsed and dried using deionized water and compressed nitrogen respectively. Second, the wafer is placed in 100% acetone to remove the remaining polymer layer from the wafer; rinsed and dried again used deionized water and compress nitrogen. Third, after the polymer layer has been removed, the wafer is then placed in a blue etching solution to remove the copper seed layer from the wafer;
and repeat cleaning. The aluminum and titanium layers are then etched from the wafer with 10% HF (Hydrofluoric) acid solution—the HF etching process takes approximately five hours—the results are five nickel iron dog-bone test specimens. The five dog-bone test specimens are then rinsed and dried using deionized water and compressed nitrogen. Figure 3 shows a dog-bone specimen with dimensions that is ready for mechanical testing.

Figure 2. A Si wafer containing five electrodeposited Ni\textsubscript{80-Fe20} test specimens.

Figure 3. (Left) Actual Ni\textsubscript{80-Fe20} test specimen; and (Right) schematic drawing of a dog-bone shape Ni\textsubscript{80-Fe20} test specimen depicting dimensions (in millimeters).

Mechanical Properties and Testing

In this study, electrodeposited Ni\textsubscript{80-Fe20} test specimens were fabricated for mechanical, magnetic, and composition testing. Sharpe\textsuperscript{5} states that tensile tests have the advantage of uniform stress and strain fields, which is why they are used to determine mechanical properties at larger scales. However, they have disadvantages at smaller scales in that larger forces are required and specimen gripping may be difficult. Johnson\textsuperscript{6} compares bending and tension tests and notes that the former requires small forces and produce large displacements, whereas the latter require large forces with correspondingly small displacements. Bending, resonant, and membrane or bulge
tests are inverse methods of determining mechanical properties. A simple or sophisticated model of a test structure is constructed, and independent stimulus is applied, and a response is measured. The parameters in the model are adjusted until the predicted response is sufficiently close to the result. For example, in simple Euler bending of a cantilever beam, the displacement, \( \delta \), is given by the familiar \( \frac{PL^2}{3EI} \) where \( P \) is the applied force, \( L \) is the length of the beam, \( I \) is the area moment of inertia, and \( E \) is the desired Young’s modulus. One measures \( \delta \) for a known \( P \) and computes \( E \). There are two challenges in extracting mechanical properties of smaller specimens, such as encountered with electrodeposited Ni\(_{80}\)Fe\(_{20}\) in this manner. First, it is sometimes difficult to know the boundary conditions. Electrodeposited test specimens are likely released by an etching process, which may vary slightly among specimens. The supporting boundaries are typically of a similar material with roughly the same thickness and stiffness. Second, the stress state at the point of failure can be very complicated. This is not so much an issue in determining modulus because one can match the shape of the test structure to a simple model. However, even in simple cantilever beam test structures, most failures occur at stress concentrations. The effect of the size of the highly stressed region and stress gradient in it complicates matters, particularly for brittle materials, such as electrodeposited Ni\(_{80}\)Fe\(_{20}\).

Sharpe\(^7\) states that there are three main challenges in testing small and thin specimens: (1) specimen preparation and handling, (2) specimen gripping and pulling, and (3) strain measurement during the test. Metallic thick Ni film was tested at Johns Hopkins University for strain measured using an interferometric strain/displacement gage (ISDG)\(^9\). More experiments on materials/structures were recently reviewed by Sharp\(^9\), Srikar and Spearing\(^10\), and Xue and Veazie\(^11\) on various types of mechanical tests at the microscale—bend, resonance, nanoindentation, and tension, etc. Materials that have been characterized using the microtensile test include single crystal silicon\(^12\), polysilicon\(^13\), aluminum\(^14\), and nickel\(^15-16\). However, a complete set of experiments on Ni\(_{80}\)Fe\(_{20}\) alloy films has not been published.

The test specimens, fabricated according to methods described above, were tested to failure in tension to validate regression analysis models. The first generation tensile testing system used a 10 kN MTS test frame with digital control and computer data acquisition. Specific testing details and experimental procedures can be found in Xue and Veazie\(^11\). Tension studies were conducted of free standing electroplated Ni\(_{80}\)Fe\(_{20}\) films of 10–30 micron thick at room temperature in laboratory air. The microstructures and crystalline structures of the electroplated Ni\(_{80}\)Fe\(_{20}\) were also studied as a function of various fabrication and annealing conditions, which can be correlated to the changes in thermal-mechanical properties\(^11\). A typical stress-strain curve that resulted from tensile testing is shown in Figure 4.

Experimental Design, ANOVA and the Effects of Factors

A Factorial Design Analysis (FDA) approach was used for statistical computation and design. A FDA is a between-participants design analysis that includes more than one independent variable. This design has the advantage over the simple randomize design in that you can test the effect of more than one independent variable and the interactive effect of the various independent variables. The FDA is broken down into two significant effects: the main and interaction effects. The main effect is an outcome that is a consistent difference between levels of a factor. An interaction effect is when one factor is a function or dependent upon another factor.
The main effects are produced by the independent variable; whereas, the interaction effects occurs when the effect of one independent variable depends on the level of the other independent variables being considered. Therefore, the three specific advantages to using a FDA in experimentation rather than the classical methods are: (1) efficiency/economy—requires fewer participants and retains the same degree of accuracy, (2) comprehensiveness—in addition to analyzing the effect of a single factor, FDA enables us to analyze the effect of the interactions as well, and (3) wider inductive basis—allows for a broader interpretation of results, i.e. the conclusions are based on an experiment having many independent factors and these factors have been tested under a broader range of conditions than if only one variable had changed at a time. The objectives were to determine, statistically, how these external electrolytes boundary conditions influence the mechanical properties of electrodeposited Ni80Fe20, and to model the mechanical properties influence as a function of these external electrolytes boundary conditions.

Design of Experiments (DOE) is a powerful analysis tool for modeling and analyzing the influence of control factors on performance output, and its straightforward application was used in this upper-level laboratory exercise\textsuperscript{17-18}. The most important stage in the design of experiment lies in the selection of the control factors. Therefore, a number of factors are included so that non-significant variables can be identified at the earliest opportunity. Specifically, a $2^k$ Factorial Analysis and Design of Experiments was designed for the critical electrolyte boundary conditions which affect the mechanical properties of electrodeposited Ni$_{80}$Fe$_{20}$. A Regression Analyses was also conducted to model the mechanical properties as a function of the critical electrolytes’ boundary conditions. Three independent parameters, viz., current density ($C$), temperature ($T$), and agitation ($A$), each at two levels, high (+) or maximum value (denoted as upper bound), or low (-) or minimum value (denoted as a lower bound), are considered in this study in accordance with $2^k$ factorial orthogonal array design. Specifically, the current density used was 5 or 10 mA/cm$^2$, the temperature was 25 or 50°C, and the agitation used was 0 or 300 rpm duty cycle. The mechanical tests are carried out under operating conditions given in Table 2.
Table 2: Nickel-Iron (Ni$_{80}$-Fe$_{20}$) specimen test matrix.

<table>
<thead>
<tr>
<th>Batch</th>
<th>Current Density (mA/cm$^2$)</th>
<th>Agitation (RPM)</th>
<th>Temp (deg C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td>0</td>
<td>25</td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td>300</td>
<td>50</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

There are eight treatment combinations in this design. Figure 5 shows graphically (as a cube) the effect of the three control factors on the Young’s modulus. Thus, there are seven degrees of freedom between the eight treatment combinations in the $2^3$ factorial design analysis. Three degrees of freedom are associated with the main effect: agitation ($A$), temperature ($T$), and current density ($C$). Four degrees of freedom are associated with the interactions: agitation-temperature ($AT$); agitation-current density ($AC$); temperature-current density ($TC$); and agitation-temperature-current density ($ATC$). The measured Young modulus’ ($E$) values at the electrolytes’ boundary conditions are shown in Figure 6, and the treatment combinations are shown for the measured Young’s modulus values (in GPa) in Table 3. This is called the design matrix; where the treatment combinations are written in the following order: $abc$, $bc$, $ac$, $c$, $ab$, $b$, $a$, and $(1)$. These symbols also represent the total of all $n$ observations taken at that particular treatment combination.

Figure 5. (Left): Treatment combinations in the $2^3$ design. (Right): Treatment combinations in the $2^3$ design for Young Modulus ($E$); with measured $E$ values (in GPa).

Figure 6. The measured Young modulus’ (GPa) values at the electrolytes’ boundary conditions.
Table 3: Treatment combinations for the $2^3$ design, with measured Young’s Modulus ($E$) values.

<table>
<thead>
<tr>
<th>Treatment Combination</th>
<th>Factorial Effect</th>
<th>Factorial Effect</th>
<th>Factorial Effect</th>
<th>Factorial Effect</th>
<th>Factorial Effect</th>
<th>Factorial Effect</th>
<th>Factorial Effect</th>
<th>Factorial Effect</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>abc</td>
<td>I+</td>
<td>A+</td>
<td>T+</td>
<td>AT+</td>
<td>C+</td>
<td>AC+</td>
<td>TC+</td>
<td>ATC+</td>
<td>129.135</td>
</tr>
<tr>
<td>bc</td>
<td>I+</td>
<td>A-</td>
<td>T+</td>
<td>AT-</td>
<td>C-</td>
<td>+</td>
<td>-</td>
<td>-</td>
<td>131.880</td>
</tr>
<tr>
<td>ac</td>
<td>I+</td>
<td>A+</td>
<td>T-</td>
<td>AT+</td>
<td>C-</td>
<td>+</td>
<td>-</td>
<td>-</td>
<td>89.900</td>
</tr>
<tr>
<td>c</td>
<td>I+</td>
<td>A-</td>
<td>T-</td>
<td>AT+</td>
<td>C+</td>
<td>+</td>
<td>-</td>
<td>-</td>
<td>152.140</td>
</tr>
<tr>
<td>ab</td>
<td>I+</td>
<td>A+</td>
<td>T+</td>
<td>AT-</td>
<td>C-</td>
<td>+</td>
<td>-</td>
<td>-</td>
<td>165.785</td>
</tr>
<tr>
<td>b</td>
<td>I+</td>
<td>A-</td>
<td>T+</td>
<td>AT-</td>
<td>C+</td>
<td>+</td>
<td>-</td>
<td>-</td>
<td>127.880</td>
</tr>
<tr>
<td>a</td>
<td>I+</td>
<td>A+</td>
<td>T-</td>
<td>AT-</td>
<td>C-</td>
<td>+</td>
<td>+</td>
<td>-</td>
<td>147.340</td>
</tr>
<tr>
<td>(1)</td>
<td>I+</td>
<td>A-</td>
<td>T-</td>
<td>AT+</td>
<td>C-</td>
<td>+</td>
<td>+</td>
<td>-</td>
<td>168.200</td>
</tr>
</tbody>
</table>

The analyses are made using the popular software specifically used for design of experiment applications known as Design Expert®. Before any attempt is made to use this simple model as a predictor for the measures of performance, the possible interactions between the control factors must be considered. Thus factorial design incorporates a simple means of testing for the presence of the interaction effects. Analysis of the results leads to the conclusion that factor combination of low agitation, low current density, and low temperature gives the maximum Young’s modulus. As far as maximizing the Young’s modulus is concerned, current density has the greatest effect. Agitation speed has a significant effect, albeit not as significant as current density. Temperature has the least significant effect, and the interaction between temperature and current density shows the least significant effect on Young’s modulus. Although the temperature individually has the least contribution on the stiffness property, its interaction with agitation speed has the most significant contribution on maximizing the Young’s modulus. On the other hand, the factors of current density and agitation speed and their interaction have major contribution on the Young’s modulus.

In order to understand a concrete visualization of impact of various factors and their interactions, it is desirable to develop an analysis of variance (ANOVA) table to find out the order of significant factors as well as interactions. Table 4 shows the results of the ANOVA with the Young’s Modulus for a 95% confidence interval. Table 4 also shows that the current density ($p$-value = 0.0044) and agitation ($p$-value = 0.0097) have the greatest influence on the Young’s modulus, whereas the temperature has the least influence ($p$-value = 0.1570). The interaction of agitation speed with temperature ($p$-value = 0.0039) shows the most significant contribution on the Young’s modulus, whereas the interaction of temperature with current density ($p$-value = 0.0114) shows the least significant contribution.
Table 4: Analysis of variance (ANOVA) table for Young’s modulus [Partial sum of squares - Type III]

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F Value</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model</td>
<td>4494.69</td>
<td>6</td>
<td>749.12</td>
<td>11245.67</td>
<td>0.0072</td>
</tr>
<tr>
<td>A–Agitation</td>
<td>287.28</td>
<td>1</td>
<td>287.28</td>
<td>4312.71</td>
<td>0.0097</td>
</tr>
<tr>
<td>B–Temperature</td>
<td>1.05</td>
<td>1</td>
<td>1.05</td>
<td>15.78</td>
<td>0.1570</td>
</tr>
<tr>
<td>C–Current Density</td>
<td>1408.48</td>
<td>1</td>
<td>1408.48</td>
<td>21144.35</td>
<td>0.0044</td>
</tr>
<tr>
<td>AB</td>
<td>1748.18</td>
<td>1</td>
<td>1748.18</td>
<td>26244.00</td>
<td>0.0039</td>
</tr>
<tr>
<td>AC</td>
<td>841.12</td>
<td>1</td>
<td>841.12</td>
<td>12626.99</td>
<td>0.0057</td>
</tr>
<tr>
<td>BC</td>
<td>208.59</td>
<td>1</td>
<td>208.59</td>
<td>3131.40</td>
<td>0.0114</td>
</tr>
<tr>
<td>Residual</td>
<td>0.067</td>
<td>1</td>
<td>0.067</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cor Total</td>
<td>4494.76</td>
<td>7</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Regression Model

The regression model is the final test in the design of the experiment process. The purpose of the regression model is to validate the conclusions drawn during the analysis phase. It is performed by conducting a new set of coded values to predict the boundary condition’s influence on the Young’s modulus. The estimated boundary condition’s influence on the Young’s modulus ($E$) can be calculated with the help of following predictive equation:

$$
E = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3
$$

(1)

where the coded variables $x_1$, $x_2$, and $x_3$ are defined on a scale from -1 to +1; the low and high levels of $A$, $T$, and $C$ respectively. The terms $x_1 x_2$, $x_1 x_3$, $x_2 x_3$, and $x_1 x_2 x_3$ are $AT$, $AC$, $TC$, and $ATC$ interactions respectively. The $\beta$'s are regression coefficients and are related to the effect estimates, and $\beta_0$ is the estimated average of all eight responses, hence

$$
\beta_0 = \frac{1}{8} [129.135 + 131.880 + 89.900 + 152.140 + 165.785 + 127.880 + 147.340 + 168.200] = 139.0325
$$

(2)

The resulting model seems to be capable of predicting the boundary condition’s influence on the Young’s modulus to a reasonable accuracy. An error of 9.1% for the Young’s modulus is observed. However, the error can be further reduced if the number of experiments is increased. This validates the development of the mathematical model for predicting the measures of performance based on knowledge of the input parameters. As a result, the regression model-coded values-is given by:

$$
\text{Young modulus (E)} = +139.03 - 5.99 * A - 0.36 * T - 13.27 * C
$$

$$
+ 14.78 * A * T - 10.25 * A * C + 5.11 * T * C
$$

(3)
Similarly, the regression model-in actual physical values-is given by:

\[
\text{Young Modulus (} E \text{)} = +245.46125 - 0.13053 \times \text{Agitation} - 2.43710 \times \text{Temperature} - 7.33350 \times \text{Current Density} + 7.88400E-003 \times \text{Agitation} \times \text{Temperature} - 0.027343 \times \text{Agitation} \times \text{Current Density} + 0.16340 \times \text{Temperature} \times \text{Current Density}
\]  

Design Expert® enables the capability to construct 3D surface plots of the Young modulus as functions of agitation and temperature for inclusion in the laboratory report, as shown in Figure 7. For this case, the current density is fixed at 10.0 A/cm². Similarly, a 3D surface plot of the Young modulus as functions of agitation and temperature with current density fixed at 5.0 A/cm² is shown in Figure 8.

![Figure 7](image1)

**Figure 7.** Young modulus (GPa) 3D surface plot as function of agitation and temperature; current density is fixed at 10.0 A/cm².

![Figure 8](image2)

**Figure 8.** Young modulus (GPa) 3D surface plot as function of agitation and temperature; current density is fixed at 5.0 A/cm².
Conclusion

This paper describes the methodology and implementation experienced in a specific course (Engineering Materials Laboratory) that embodies the goals of upper-level undergraduate engineering laboratory experiments incorporating a general full factorial design for experiments for the processing of materials for characterization. This Factorial Design Analysis (FDA) approach facilitates a ‘between-participants’ design analysis that includes more than one independent variable, and has the advantage over a simple randomized design in that you can test the effect of more than one independent variable and the interactive effect of the various independent variables. The overall academic learning outcomes for the student successfully completing the Engineering Materials Laboratory course include the achievement of a basic grasp of materials processing to incorporate process control and analysis, and the demonstration of the technical competence to characterize material properties. Through the implementation of this FDA, the students were able to meet the course objectives.

The first evidence of the meeting the learning outcomes include the prediction and assessment of parameters (i.e., the critical boundary conditions of agitation, temperature, and current density) and their influence on the mechanical properties. The successful implementation is validated by inferences such as that for higher temperatures, Young modulus increases as agitation increases, and for low current density, Young modulus is directly proportional to agitation and temperature. The next evidence of achieving the learning objectives include the correlation of the predicted mechanical properties with measured values based on a limited number of experiments or available data. This project was devised to teach students the fundamentals of design of experiments for the processing of materials for characterization, and the exercise assessment was deemed satisfactory because of the students’ capability to predict the boundary condition’s influence on the Young’s modulus to a reasonable accuracy (9.1% for the Young’s modulus was observed). Overall, the upper-level undergraduate student experience for this laboratory initiative was satisfactory.

The objective of a statistical factorial analysis relative to the classical approach is to provide insight to the behavior while minimizing the number of experiments. As a result of this research, the critical boundary conditions have been identified as agitation, temperature, and current density; and their influence on the mechanical properties have been shown as \( E = E(A,T,C) \). Whereas the regression models may not yield the exact mechanical properties values, they are easily replicated and can provide insight on how these boundary conditions influence the mechanical properties and the degree of their influence. In general, the upper-level undergraduate students’ performance was satisfactory, the student overall experience as evidenced by the end-of-term evaluations were extraordinary, and the grades assessed by the professor were above average. Because of the good correlation with measured values, it is anticipated that this approach can be implemented for other courses in various areas (controls, thermal sciences, design, etc.) in the future.

References