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Laboratory Experiment in Engineering Materials for Upper-Level Undergraduate and Graduate Students

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Abstract

Laboratory experiments are a critical part of the required curriculum for upper-level undergraduate and graduate 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 a level of specification and implementation methodology. However, often these laboratory experiments were developed for well defined systems in controlled environments in order to take advantage of limited resources such as expensive materials, laboratory space and testing supplies. 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.

This paper focuses on the educational merits of a laboratory experiment in engineering materials, and what was learned about educating the upper-level and graduate students. The proposed methodology in this paper describes a general full factorial design for experiments involving the mechanical characterization, specifically the mechanical strength and processing parameters, of a material. 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 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 strength of electrodeposited nickel-iron ($Ni_{80}Fe_{20}$). The critical external boundary conditions examined for this material system include the current density of the electrolytic bath, the bath temperature, and the speed of agitation in the bath. Results show the ANOVA (analysis of variance) table of results for the critical factors, as well as the *F*-test on the interactions. Based on the results, regression models are developed and surface plots presented for the mechanical strength of the material system as a function of the external boundary conditions.

Introduction

Engineering laboratory experiments that involve materials and/or material properties are often designed to establish a level of specification and implementation methodology, and is usually required for students seeking degrees in the science, technology, engineering and mathematics (STEM) fields. 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 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, as well as to capture students' interest and get them excited to learn about the integration of materials and/or material properties for design.

The methodology in this paper describes an experience uniquely suited for upper-class STEM students because of their specific interests, multidisciplinary and interdisciplinary experiences, and their background in mathematics, physics, mechanics and computer programming. Upper-class courses, especially those at technical institutions, are often scheduled as a combination of lecture and laboratory (or studio) components, and usually involve multidisciplinary and interdisciplinary concepts[1]. For this specific laboratory (Engineering Materials Laboratory), an active learning environment was adopted with the students required to work on open-ended problems in small groups. Creativity was emphasized, and the students were encouraged to reflect, both in writing and orally.

The overall academic learning outcomes for the student successfully completing this Engineering Materials Laboratory course are summarized by the following:

1. Achieve a basic grasp of atomic structure and bonding incorporating crystal structure and crystal defects, including documentation and symbols.
2. Demonstrate technical competence controlling material dislocation and strengthening, including utilizing phase diagrams incorporating alloys.
3. Apply fundamental rules, laws and criteria for implementing phase transformation/heat treatment for material selection for design and manufacturing.
4. Demonstrate the ability to explain and apply fundamental mechanical properties and experimental testing, including failure.
5. Demonstrate the ability to model and analyze diffusion.
6. Apply writing, and communication skills through written reports and oral demonstrations.

The following paragraphs describe in detail how these academic learning outcomes are achieved in the laboratory course. Where applicable, specific links to the learning outcomes are made.

Electrodeposition of Permalloy $Ni_{80}Fe_{20}$

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.[2] 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, electrodeposited nickel-iron ($Ni_{80}Fe_{20}$) can be as a magnetic material of choice as well as the back iron material to fill the cavities between the other non-metals, such as silicon. $Ni_{80}Fe_{20}$ 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.

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[3]. 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[4]. Magnetic micro-actuators and inductors such as solenoids, valves, and cantilevers are fabricated using electrodeposited Permalloy ($Ni_{81}Fe_{19}$)[5].

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#5 and #6) 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 typical setup of an electroplating bath is shown in Figure 1, whereas the specific nickel plating solution and other bath and plating parameters can be found in Veazie and Ephraim[6]. The cathode and anode are placed in the iron electrolyte solution (nickel iron bath) under a constant current, bath temperature, and agitation. 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 (Left) 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. Next, 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. 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. Figure 2 (Right) shows a dog-bone specimen with dimensions that is ready for mechanical testing.

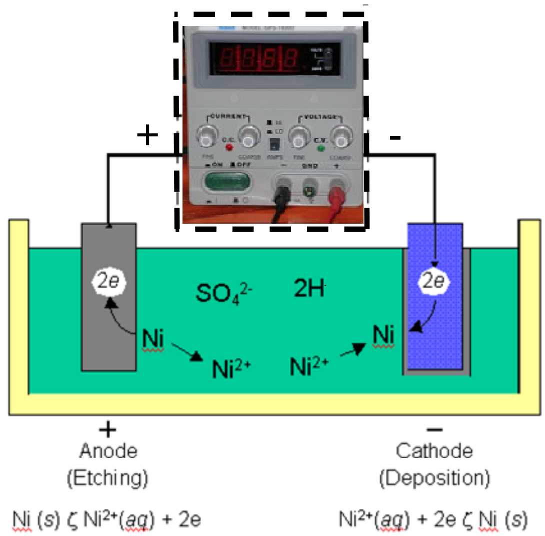

Figure 1. A Si wafer containing five electrodeposited Ni_{80}-Fe_{20} test specimens.

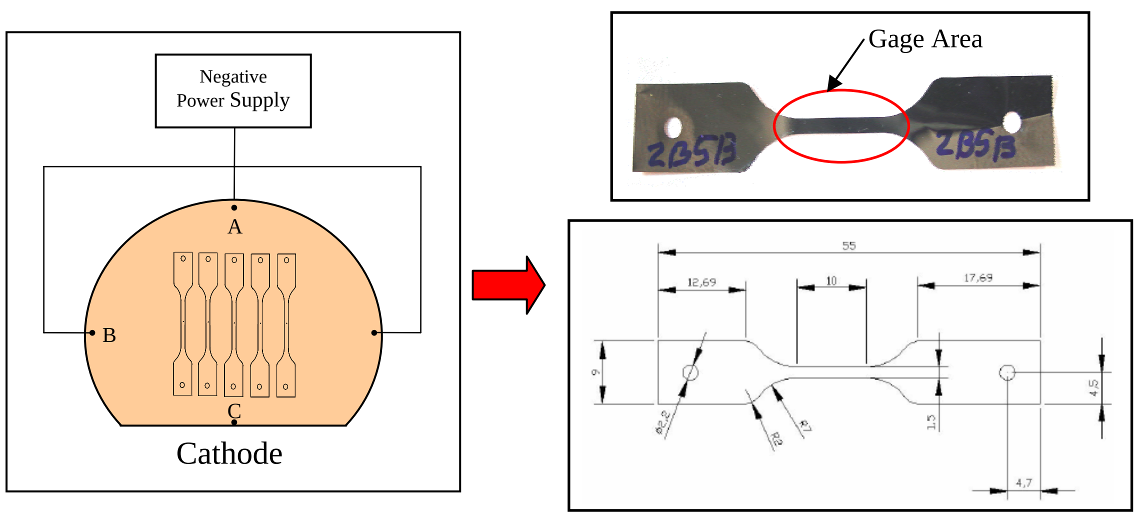

Figure 3. (Left) A Si wafer containing five electrodeposited Ni_{80}-Fe_{20} test specimens; and (Right) an actual Ni_{80}-Fe_{20} test specimen (top) with schematic drawing (bottom) of a reference ASTM dog-bone shape Ni_{80}-Fe_{20} test specimen depicting dimensions (in millimeters).

Mechanical Properties and Testing

In this study, electrodeposited $Ni_{80}Fe_{20}$ test specimens were fabricated for mechanical, magnetic, and composition testing. Sharpe[7] 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[8] 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. 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. 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)[10]. More experiments on materials/structures were recently reviewed by Sharp[11], Srikar and Spearing[12], and Xue and Veazie[13] 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[14], polysilicon[15], aluminum[16], and nickel[17-18]. 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[13]. 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[13].

Experimental Design, ANOVA and the Effects of Factors for Mechanical Strength

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 additional 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 $Ni_{80}Fe_{20}$, 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[19-20]. 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 1, and a plot of ultimate strength (σ_{ult}) as versus the specimen's thickness is shown in Figure 3.

Table 1: Nickel-Iron (Ni_{80}-Fe_{20}) specimen test matrix for mechanical strength.

	Current Density (mA/cm^2)		Agitation (RPM)		Temp (deg C)	
Batch	5	10	0	300	25	50
1	■		■			■
2	■		■		■	
3	■			■		■
4	■			■	■	
5		■	■			■
6		■	■		■	
7		■		■		■
8		■		■	■	

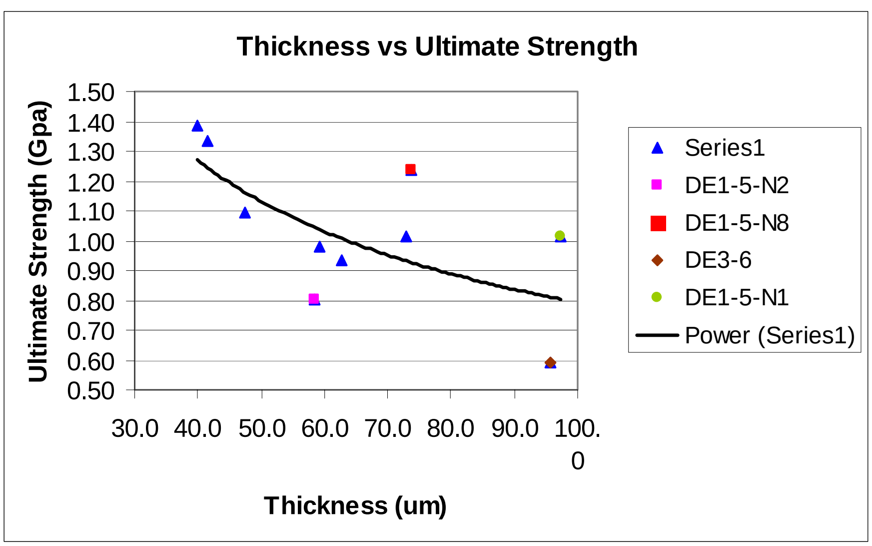

Figure 3. Plot showing ultimate strength (σ_{ult}) as a function of specimen thickness.

There are eight treatment combinations in this design, Figure 4 shows graphically (as a cube) the effect of the three control factors on the ultimate strength. 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 ultimate strength (σ_{ult}) values at the electrolytes' boundary conditions are shown in Table 2, and the treatment combinations are shown for the measured ultimate strength 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.

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[19]. Analysis of the results leads to the conclusion that factor combination of low agitation, low current density, and low temperature gives the maximum strength. As far as maximizing the ultimate strength 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 effect on the ultimate strength. Although the temperature individually has the least contribution on the strength property, its interaction with agitation speed has the most significant contribution on maximizing the strength.

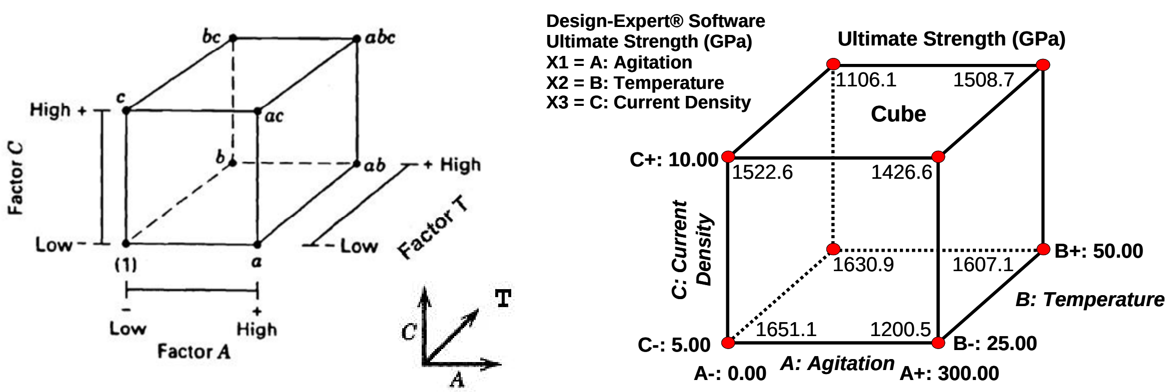

Figure 4. (Left): Treatment combinations in the 2^3 design. (Right): Treatment combinations in the 2^3 design for ultimate strength (σ_{ult}); with measured σ_{ult} values (in GPa).

Table 2: The measured ultimate strength (GPa) values at the electrolytes' boundary conditions.

Agitation (rpm)	Current Density (mA/cm^2)			
	5		10	
	Temperature (Deg C)		Temperature (Deg C)	
	25	50	25	50
0	1651.134	1630.90	1522.63	1106.13
300	1200.46	1607.07	1426.60	1508.72

Table 3: Treatment combinations for the 2^3 design, with measured ultimate strength (σ_{ult}) values.

Treatment Combination	Factorial Effect								
	I	A	T	AT	C	AC	TC	ATC	σ_{ult} (MPa)
abc	+	+	+	+	+	+	+	+	1508.72
bc	+	-	+	-	+	-	+	-	1106.13
ac	+	+	-	-	+	+	-	-	1426.60
c	+	-	-	+	+	-	-	+	1522.63
ab	+	+	+	+	-	-	-	-	1607.07
b	+	-	+	-	-	+	-	+	1630.90
a	+	+	-	-	-	-	+	+	1200.46
(1)	+	-	-	+	-	+	+	-	1651.134

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[19]. Table 4 shows the results of the ANOVA with the ultimate strength for a 95% confidence interval. Table 4 also shows that the current density (*p-value* = 0.0864) and agitation (*p-value* = 0.2571) have the greatest influence on the Young's modulus, whereas the temperature has the least influence (*p-value* = 0.6009). The interaction of agitation speed with temperature (*p-value* = 0.0493) shows the most significant contribution on the ultimate strength, whereas the interaction of temperature with current density (*p-value* = 0.0632) shows the least significant contribution.

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 ultimate strength. The estimated boundary condition's influence on the ultimate strength (σ_{ult}) can be calculated with the help of following predictive equation:

Table 4: Analysis of variance (ANOVA) table for ultimate strength [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	2.866E+005	6	47772.42	74.18	0.0886
A-Agitation	*3525.65*	*1*	*3525.65*	*5.47*	*0.2571*
B-Temperature	*337.95*	*1*	*337.95*	*0.52*	*0.6009*
C-Current Density	*34516.68*	*1*	*34516.68*	*53.60*	*0.0864*
AB	*1.071E+005*	*1*	*1.071E+005*	*166.25*	*0.0493*
AC	*76257.62*	*1*	*76257.62*	*118.42*	*0.0583*
BC	*64936.15*	*1*	*64936.15*	*100.84*	*0.0632*
Residual	643.97	1	643.97		
Cor Total	2.873E+005	7			

$$\sigma_{ult} = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3 \quad (1)$$

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_1x_2, x_1x_3, x_2x_3, and $x_1x_2x_3$ are *AT*, *AC*, *TC*, and *ATC* interactions respectively. The β's are regression coefficients and are related to the effect estimates, and β_0 is the estimated average of all eight responses, hence

$$\beta_0 = \frac{1}{8}[1508.72 + 1106.13 + 1426.6 + 1522.63 + 1607.07$$

$$+ 1630.9 + 1200.46 + 1651.13] = 1456.706 \quad (2)$$

The resulting model seems to be capable of predicting the boundary condition's influence on the ultimate strength to a reasonable accuracy. An error of 8.9% for the ultimate strength 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:

$$\text{Ultimate Strength } (\sigma_{ult}) = +1456.71 - 20.99 * A + 6.50 * B - 65.69 * C$$

$$+ 115.68 * A * B + 97.63 * A * C + - 90.09 * B * C \quad (3)$$

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

$$\text{Ultimate Strength } (\sigma_{ult}) = +1484.35400 - 4.40627 * \text{Agitation} + 12.88800 * \text{Temperature} + 42.78600 * \text{Current Density} + 0.061698 * \text{Agitation} * \text{Temperature} + 0.26035 * \text{Agitation} * \text{Current Density} - 2.88302 * \text{Temperature} * \text{Current Density} \quad (4)$$

Design Expert® enables the capability to construct 3D surface plots of the ultimate strength as functions of agitation and temperature for inclusion in the laboratory report, as shown in Figure 5. For this case, the current density is fixed at 10.0 mA/cm^2. Similarly, a 3D surface plot of the ultimate strength as functions of agitation and temperature with current density fixed at 5.0 mA/cm^2 is shown in Figure 6.

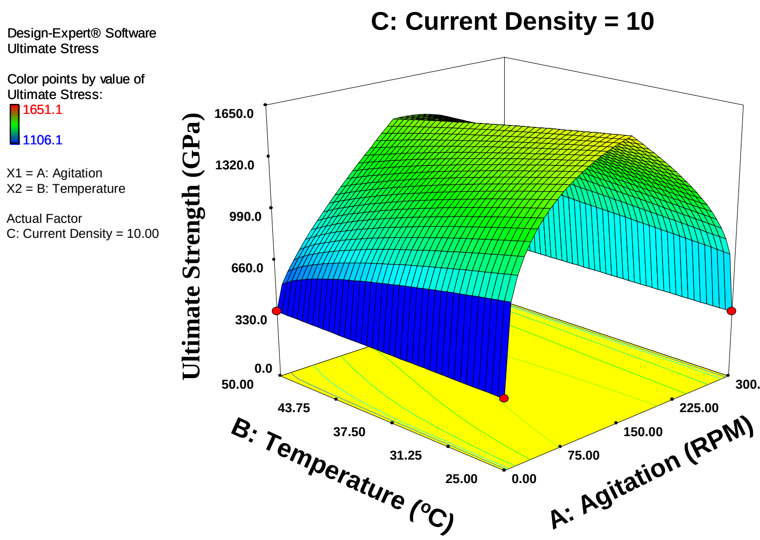

Figure 5. Young modulus (GPa) 3D surface plot as function of agitation and temperature; current density is fixed at 10.0 mA/cm^2.

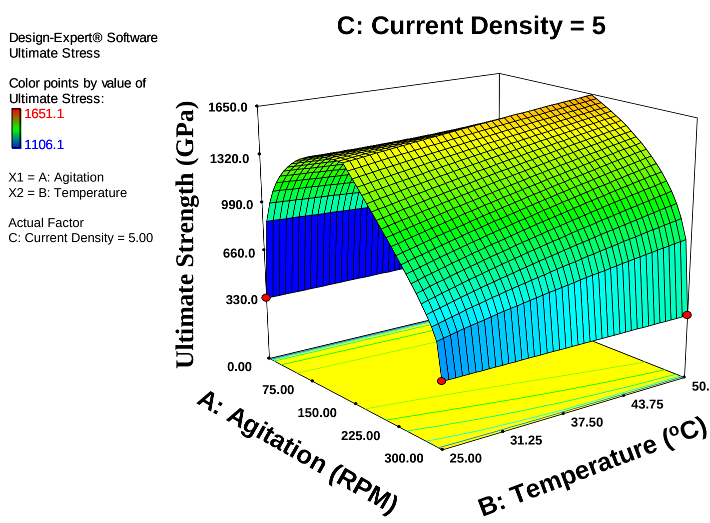

Figure 6. Young modulus (GPa) 3D surface plot as function of agitation and temperature; current density is fixed at 5.0 mA/cm^2.

Assessment Results

The first evidence of the meeting the learning outcomes includes 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, ultimate strength increases as agitation increases, and for low current density, ultimate strength is directly proportional to agitation and temperature. This is a direct correlation to the learning outcome of demonstrating the ability to explain and apply fundamental mechanical properties and experimental testing, including failure.

The next evidence of achieving the learning objectives includes 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 ultimate strength to a reasonable accuracy (8.9% for the ultimate strength was observed). Hence, the students were able to achieve the learning objective of demonstrating the ability to model and analyze material properties. Overall, the upper-level undergraduate student experience for this laboratory initiative was satisfactory.

Conclusion

This paper describes the methodology and implementation experienced in a specific course (Engineering Materials Laboratory) that embodies the goals of upper-level undergraduate and graduate 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 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 $\sigma_{ult} = \sigma_{ult}(A,T,C)$. 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. 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. In a comparison with previous laboratory courses in materials, a noticeable improvement in the achievement of the learning outcomes by the students was observed. In general, the students' performance was noticeably superior, the student evaluations were extraordinary, and the grades assessed by the professor were above average.

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