# AC 2007-2229: INTRODUCING RESEARCH CONCEPTS TO SENIOR STUDENTS IN DESIGN OF EXPERIMENTS LABORATORY

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# Introducing Research Concepts to Senior Students in a Design of Experiments Laboratory Course

## Introduction

The country's increasing demand for engineering talent has led to introduction of several innovative programs into the K-12 outreach activities and also in the existing undergraduate curriculum. One such activity is integrating research into the undergraduate program [1-4]. Over the last several years, many undergraduate courses have been offered with a research component which has resulted in a variety of positive outcomes[1]. Research-oriented courses enable the students to get experienced with enhanced presentation skills, group experience and applying research concepts in a professional environment. In this paper, we discuss about the introduction of a research oriented laboratory experiment into a course that has traditionally used machine shop processes such as spot welding and machining to teach experimental design. Achievement of student proficiency in designing experiments and statistically analyzing the data is the principal objective of this senior mechanical engineering laboratory course. During the initial few classes, the students perform simple experiments to understand the concepts of random variables and their distribution. They correlate the results to designing experiments and their quality. Later, they perform experiments using a factorial design of experiments chart. Then, the output data from the experiments are analyzed. Based on the experiments, a term project is assigned that will allow the students to develop an experimental design chart by identifying the independent and dependent variables, collecting data by performing experiments and using statistical tools to analyze the results. We are examining a new experimental design arena - where students have the opportunity to design simple experiments that will be performed using the sophisticated infrastructure of an advanced materials research lab. Powder metallurgy and solid oxide fuel cells concepts are introduced to the students through this project. The principal motivation is to expose all students to the state of the art and stimulating more of them to favorably consider a research career.

## Course Objective

This particular course is a senior level laboratory course, MEEN 502 – Mechanical Engineering Laboratory III, the third in a series of four laboratory courses. The course is about the application of statistical methods in the engineering field. The course enables the student to design the experiment, conduct the experiment, collect data, analyze and interpret the experimental results.

## Process

Yttria stabilized zirconia (YSZ) particles constitute the most popular starting material for fabricating electrodes for solid oxide fuel cells (SOFCs). In an ongoing research project in our Center, we have been using YSZ particles of average size ~ 58 nm. During the first stage, the students work with the instructor to design an experiment to study he effect of processing parameters on the density (and hence porosity) of the compacted and sintered electrodes. Since the students did not have a prior knowledge about the compaction behavior of YSZ material, the instructor helped the students to decide the levels for the each factor. The discussion led to the design chart (Table 1) and the corresponding experimental order (Table 2).

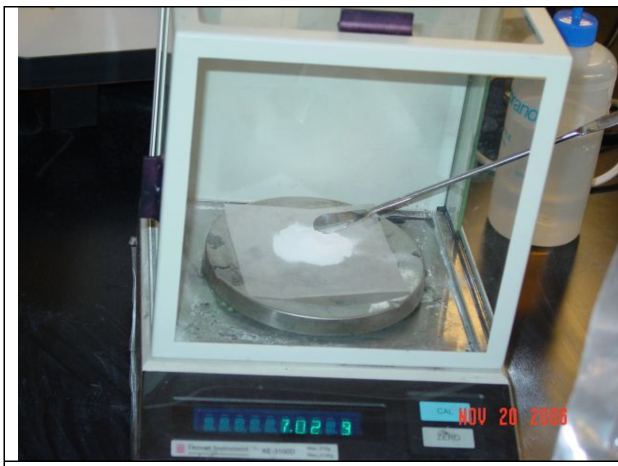
Table 1. Process variables and their treatment levels selected for the experimental design

| Level / Factor | -1 (Low) | 0 (Medium) | +1 (High) |
|---|---|---|---|
| **F - Compaction Force (lb)** | 2200 | 2400 | 2600 |
| **T - Sintering Temperature (°C)** | 1000 | 1200 | 1400 |

Table 2. Combination # and randomized order of experimentation
(2 replicates per combination = total 36 samples prepared and tested)

| Combination # | Run Order (Randomized) | F - Compaction Pressure (lb) | T - Sintering Temperature (°C) |
|---|---|---|---|
| 1 | 6 | 2200 | 1000 |
| 2 | 1 | 2200 | 1200 |
| 3 | 4 | 2200 | 1400 |
| 4 | 9 | 2400 | 1000 |
| 5 | 11 | 2400 | 1200 |
| 6 | 2 | 2400 | 1400 |
| 7 | 3 | 2600 | 1000 |
| 8 | 17 | 2600 | 1200 |
| 9 | 12 | 2600 | 1400 |
| 10 | 10 | 2200 | 1000 |
| 11 | 14 | 2200 | 1200 |
| 12 | 7 | 2200 | 1400 |
| 13 | 16 | 2400 | 1000 |
| 14 | 15 | 2400 | 1200 |
| 15 | 5 | 2400 | 1400 |
| 16 | 8 | 2600 | 1000 |
| 17 | 18 | 2600 | 1200 |
| 18 | 13 | 2600 | 1400 |

After designing the experiments, the students meet in the lab for the second stage of the project, i.e., for conducting the experiments. A 1.2 in (3 cm) steel die was used for the powder compaction. Students performed volume calculations to decide the amount (~ 7g) of YSZ powder for each sample. Powder samples for all 18 experiments were prepared at the outset using the digital weigh scale (Fig. 1a). The powder samples were moved to the die in the press in the order of experiments. For the first sample, the instructor demonstrated the procedure for cleaning the die (Fig. 1b), loading with the powder (Fig. 1c), placing the die in the compaction unit and applying the required pressure (Fig. 1d), and then removing the sample carefully without damaging the sample (Fig. 2a). The students observed the steps and every one had the opportunity to be personally involved in fabricating the green samples for compaction. After compactions, the green forms were sintered in air (Fig. 2b) at different temperatures (over night sintering for each condition). During the third stage, the students collected all the sintered samples (Fig. 2c) for the measurement. The students measured the weight (using the digital weigh scale (Fig. 2a) and the dimensions (using calipers, Fig. 2d) for the volume calculations.




(a)


(b)


(c)


(d)

Fig. 1 (a) Weighing the powder (b) steel die and its components used for the compaction (c) preparing for die for compaction and (d) powder compaction process


(a)


(b)


(c)


(d)


(e)

Fig. 2 (a) Green sample ejected from the die, (b) furnace used for sintering, (c) students measuring the weight of the sintered sample, (d) students measuring the dimensions of the sintered sample and (e) sintered samples

Table 3. Calculated volume and the porosity based on the sample measurements

| Force (lb) | Temp. (°C) | Replicate # | Wt (g) | Dia. (mm) | Thickness (mm) | $V_a$ (actual) | $V_t$ (theoretical) | $(V_a-V_t)$ ($mm^3$) | Porosity (%) |
|---|---|---|---|---|---|---|---|---|---|
| 2200 | 1400 | A | 6.85 | 25.12 | 3.26 | 1.61 | 1.04 | 0.58 | 35.73 |
| | | B | 6.85 | 24.99 | 3.08 | 1.51 | 1.04 | 0.47 | 31.26 |
| 2400 | 1400 | A | 6.70 | 24.98 | 3.15 | 1.54 | 1.02 | 0.53 | 34.21 |
| | | B | 6.83 | 25.06 | 3.11 | 1.53 | 1.03 | 0.50 | 32.50 |
| 2600 | 1400 | A | 6.91 | 25.38 | 3.21 | 1.62 | 1.05 | 0.58 | 35.50 |
| | | B | 6.72 | 24.80 | 3.08 | 1.49 | 1.02 | 0.47 | 31.53 |
| 2200 | 1200 | A | 6.78 | 29.02 | 3.70 | 2.45 | 1.03 | 1.42 | 58.00 |
| | | B | 6.86 | 29.12 | 3.89 | 2.59 | 1.04 | 1.55 | 59.86 |
| 2400 | 1200 | A | 6.90 | 27.31 | 3.36 | 1.97 | 1.05 | 0.92 | 46.86 |
| | | B | 6.86 | 27.33 | 3.58 | 2.10 | 1.04 | 1.06 | 50.48 |
| 2600 | 1200 | A | 6.90 | 29.13 | 3.55 | 2.36 | 1.05 | 1.32 | 55.79 |
| | | B | 6.92 | 28.58 | 3.68 | 2.36 | 1.05 | 1.31 | 55.57 |
| 2400 | 1000 | A | 6.75 | 30.98 | 3.83 | 2.89 | 1.02 | 1.86 | 64.56 |
| | | B | 6.80 | 30.77 | 3.85 | 2.86 | 1.03 | 1.83 | 63.99 |
| 2600 | 1000 | A | 6.85 | 31.01 | 3.71 | 2.80 | 1.04 | 1.76 | 62.94 |
| | | B | 6.72 | 31.07 | 3.71 | 2.81 | 1.02 | 1.79 | 63.78 |
| 2200 | 1000 | A | 6.73 | 30.89 | 3.79 | 2.84 | 1.02 | 1.82 | 64.08 |
| | | B | 6.73 | 30.89 | 3.79 | 2.84 | 1.02 | 1.82 | 64.08 |

## Calculations

Based on their measurements, the students calculated the actual volume and the theoretical volume of the each sample to calculate the apparent density and eventually the porosity (Table 3). The porosity values are used as the input for the final calculations. The Analysis of Variance (ANOVA) method is used as a tool for analyzing the data. Therefore the calculations are done based on the ANOVA (Table 4). The students are familiar with the ANOVA calculations from the prior experiments and they used the porosity values as the input values and performed the calculations. The F values are calculated and also obtained from the F–table for the corresponding degrees of freedom.

## Analysis

The calculated F values were compared with the table values for the data analysis. If the calculated F value is larger than the table value, the corresponding source term is deemed to have a significant effect on the output. Also, analysis is done by comparing the raw data values (porosity values) for different experimental conditions.

Table 4. ANOVA calculations

| Source of Variation | Sum of squares | Degrees of freedom | Mean Square | $F_0$ |
|---|---|---|---|---|
| Speed (A) | $SS_A$ | a-1 | $MS_A = \frac{SS_A}{a-1}$ | $F_0 = \frac{MS_A}{MS_E}$ |
| Feed Rate (B) | $SS_B$ | b-1 | $MS_B = \frac{SS_B}{b-1}$ | $F_0 = \frac{MS_B}{MS_E}$ |
| Interaction (AXB) | $SS_{AB}$ | (a-1)(b-1) | $MS_{AB} = \frac{SS_{AB}}{(a-1)(b-1)}$ | $F_0 = \frac{MS_{AB}}{MS_E}$ |
| Error | $SS_E$ | ab(n-1) | $MS_E = \frac{SS_E}{ab(n-1)}$ | |
| Total | $SS_T$ | abn-1 | | |

(a = no. of levels for source A and b = no. of levels for source B)

**Discussion**

The instructor introduced the design of experiments and the ANOVA concepts in the class room two weeks before the project. The students performed the entire calculation part analytically, to have a better understanding about the complete process. The students also performed a simple $2^2$ factorial design of experiments, and had earlier in the semester performed experiments in the machine shop using the milling machine and used ANOVA for the data analysis. When the students got ready for the project, they had a general idea about the design of experiments but not much about powder metallurgy and SOFC concepts. Once the students got to the research lab, the instructor took the students for a quick guided tour of the different equipment that the students will be using for their work. Then the students started measuring the YSZ powder. Once after the powder preparation, the students moved on to powder compaction. The instructor demonstrated the green pellet fabrication process for one sample and the students followed the procedure for all the remaining samples. Initially, the students struggled in each stage of the fabrication, like handling the powder, placing the plugs properly on top of the powder and pumping the pressure to the required level. The most challenging job for any student was to eject the green sample from the die and move it to the table without forming any crack on the surface of the pellet. It took more than one attempt for some of the students to do these steps successfully. After fabricating all the required green samples, the students loaded the sample into the furnace for the sintering. Since each sintering cycle takes about 9 hours, it took 3 days to complete the whole sintering process for all the samples. The students gathered again to collect the final set of data for further calculation and analysis. They measured the weight and size of the sintered samples. The instructor showed them the calculation procedure to find out the porosity volume of the each sample based on the volume calculation. After collecting all the data, the students were required to write a technical report including a detailed literature review of powder metallurgy concepts, YSZ and its application in different field including the SOFCs. All the in-lab work was done as a group experience while the calculation and the report writing were assigned as individual work. This enables each and every student to collect their own information and perform their own calculation which will help in better understanding of the overall process.

Every student in the class made sincere efforts to gather relevant information about YSZ material and its application. Though the students did not design the complete experimental process on their own, they made valid comments about the selection of the process variables and their levels. The students could perform all the required calculations without any difficulty. Performed all the calculation, compared the F values and drew conclusions about the effect of each factor on the output variable. The students gained confidence in analyzing the calculated porosity values, since the figures clearly show significant influence of the respective process variables. However, the students found it difficult to draw graphs for further analysis and interpretation of the results. The students also provided a lot of feedback comments in their report, like the use of design of experiments and how it can be used more effectively by including/increasing different factors and levels, the necessity to spend more time in the research lab for a more thorough understanding of the process, necessity to spend more time in the class room to discuss about this particular experimental concept and also design of experiments in general. This is only a one-credit course, but activities like this help spark student interest in graduate studies as being more practical and realizable that they had thought before. It definitely shows that students are always willing to explore and learn new concepts.

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