

Active learning of eutectic phase diagram in engineering courses

Abordagem ativa para o estudo de diagrama de fases eutético nos cursos de engenharia

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Resumo

O presente trabalho apresenta um modo de tornar o assunto de diagrama de fases menos teórico e mais desafiante para alunos do curso de engenharia mecânica. Ele é baseado na aprendizagem ativa, ou seja, os alunos são instados a conhecer o diagrama de fases eutético por meio de dois experimentos: a difração de raios-X e a caracterização por metalografia. No primeiro, os alunos devem obter um difratograma e utilizar a análise de Rietveld por meio de software específico para a determinação da fração mássica de três diferentes ligas eutéticas. Em seguida, eles devem comparar esses resultados com aqueles apresentados pelo diagrama de fases teórico após cálculo da regra da alavanca. No segundo, os alunos devem usar um software de análise de imagens para a determinação da fração em área das fases presentes e comparar os resultados com o diagrama de fases teórico, levando em conta as densidades das fases presentes para que a comparação seja possível. Os alunos foram entrevistados ao final das aulas e apreciaram o fato de os experimentos, acrescido do uso dos softwares, terem permitido o entendimento do diagrama de fases de um modo no qual eles são os principais atores de seu próprio aprendizado.

Palavras-chave: Diagrama de fases eutético. Difração de raios-X. Metalografia.

Abstract

The present article shows a new strategy used to cover phase diagrams for undergraduate mechanical engineering students. It is based on active learning in which students are required to investigate experimentally three different eutectic alloys by using X-ray diffraction and metallography. The first technique allows students to use Rietveld analysis to obtain the mass fraction of the present phases and they must compare their results with those obtained theoretically whilst the second one is used to characterize those phases and measured their area fractions. From these results, they can also compare them with those obtained theoretically, but considering the density of different phases. The results have shown that students understood more clearly concepts such as the lever rule, and the very interpretation of phase diagram: the liquidus and solidus curve and finally the eutectic point.

Keywords: Eutectic phase diagram. X-ray diffraction. Metallography.

1. Introduction

It is well recognized nowadays that active learning is far better for our present students as they learn in different styles (TAYO, OLUWAKEMI, 2015) and teachers must be aware of not only these styles but also allow them to choose their strategies. This is one of the so many reasons teachers are preparing their students for the future (GOODHEW, BULLOUGH, TAKTAK, 2010) considering their attitudes and expectations.

One important aspect in undergraduate engineering students is that theory must be present along with experiments. On the one hand, the theory provides the necessary background to understand nature, more specifically those concepts related to materials science. On the other hand, experiments do present their problems and limitations for they must be carefully prepared so students may grasp both theoretical concepts and the phenomena that make sense of what they have learned.

Metallic Materials, a discipline taught at the University Center of FEI in São Bernardo do Campo, Brazil, is for undergraduate mechanical engineering students. They have previously studied materials science and mechanical properties, so they are now prepared to study metals and their alloys, i.e., concepts about phase transformations, their typical microstructures, heat treatment, and thermomechanical treatment.

Some of those already mentioned attitudes are the rise of computing and the internet, which has changed dramatically both the availability and accessibility of information (GOODHEW, BULLOUGH, TAKTAK, 2010) although in Brazil there is a major difficulty for our students since English is required by industries, in a general sense, and our elementary schools do not allow them to read, write, speak and listen fluently. For these reasons, teachers now tend to avoid the talk-and-chalk strategy towards active learning in which students play a central role in class.

The first two classes are about phase diagrams in both laboratory and theoretical classes. The present paper presents our strategy to explore concepts related to the eutectic phase diagram by making our students use two different techniques to characterize the phases present in one specific metallic alloy and compare their results with those from the phase diagram. The purpose is to make them aware of the strategy used to characterize those phases, work in groups and explore their skills and competencies in software to acquire the mass fraction of the phases present in three different alloys: hypoeutectic, eutectic, and hypereutectic alloys.

2. The need to change materials science subject in undergraduate engineering courses

Goodhew, Bullough, and Taktak (2010) have published a thorough investigation about how materials education has changed in the United Kingdom and have shown, among other findings, that students thought that materials subject areas such as characterization of composition and microstructure, mechanical behavior, processing, and manufacture are useful for their careers since graduation. They also mentioned that these studies gave them the competencies, skills, and attributes needed when employed, and believed that laboratory skills are important.

It is also important to mention that the availability and accessibility of information and free software have made it easier for teachers to implement their use in their classes provided they allow students to understand the concepts for a particular topic without having difficulties learning how to use them.

Engineering courses have changed slowly in Brazil for the past forty years. Governmental guidelines from the Ministry of Education were published in 2001 (MINISTRY OF EDUCATION, 2001), but its main concern was the time spent on the course and the subjects were the same previously published in 1977 (MINISTRY OF EDUCATION AND CULTURE, 1977). However, a major change took place in these guidelines in 2019, in which the main concern was the development of competencies, skills, and attitudes rather than being focused on subjects that were important in previous ones. Some curriculum flexibility has also been made possible in the present guidelines although financial restraints may be present in private institutions.

The University Centre of FEI adopted its new curriculum in 2019. The main concern was therefore to develop competencies, skills, and attitudes in our students, so they are prepared for this ever-rapid changing market and technology. Kemper and Sanders (2001) have highlighted that the main reasons for these changes are, among other aspects, the intelligent technology offers greater creative opportunity and the ability to work smarter, the global workplace demands multicultural skills; expanding social infrastructure needs talent for complexity. It is now well recognized that not only our undergraduate engineering students have to know what they are supposed to know but mainly to do what they are required to do based on what they know, the last one being related to skills rather than just knowledge.

Considering the aforementioned aspects, a teacher must be conscious of when to seize every opportunity to infuse technology in class. As mentioned by Tomei (2017), the key to success is knowing what works best in particular classroom situations. Prof. Diran Apelian from the University of California said in 2020, in the last Materials Symposium from Granta Edupack[®], that students have to be on “fire”, meaning have the activities involved with students have to fun, inspiring, relevant, and most of all, they must empower our students.

Phase equilibria and phase transformations in metals and alloys are quite difficult to undergraduate mechanical engineering students at first because their previous subjects did not explore thermodynamical concepts typically explored in materials science. For this reason, theoretical classes tended to investigate Gibbs’ phase rule without being concerned with the interpretation and analysis of phase diagrams, the last ones being much more important for them when employed daily in industries.

Bloom et al (1956) proposed a taxonomy of educational objectives for many different purposes, one of them to help a teacher gain a perspective on the emphasis given to certain behaviors by a particular set of educational plans. Although they are divided into three main categories, cognitive, affective, and psychomotor ones, the first one has been used widely. The present paper sees the first one as the way classes were organized, i.e., the goal was to make students learn to know, comprehend, interpret, apply, compare, analyze and evaluate their findings in both laboratory and theoretical classes.

3. Strategy in class

Students were asked to read their textbook beforehand concerning phase diagrams. Callister (2015) was used for this purpose. In class, students used Ansys Granta Edupack[®] software to analyze different microstructures obtained when different eutectic alloys were cooled: hypoeutectic, eutectic and hypereutectic ones. They were asked to spot the differences obtained in these microstructures and had to calculate the mass fractions of both phases at room temperature. They were then presented for the first time the lever rule.

Although the first part was conducted theoretically, the purpose was to provoke students for the lever rule and how counterintuitive it was. The use of different colors present in that software was particularly helpful for students, but only after the laboratory class was this clearer for our students.

4. The diffraction of Pb-Sn alloys and Rietveld analysis

4.1 Eutectic Phase Diagram

Phase diagram is a type of thermodynamic map that shows the phase or phases in equilibrium for a given temperature and chemical composition at a constant pressure. In the case of eutectic binary Pb-Sn alloys, the object of this work, there is a point at which three phases are in equilibrium, L, the α phase, and the β phase that occurs at a temperature of 183 °C and composition equals to 61.9% Sn, the remainder being Pb. Thus, it can be seen in Figure 1 the binary eutectic phase diagram of a lead-tin system. This was obtained from Thermocalc[®], Database: TCBIN: TC Binary Solutions V1.1.

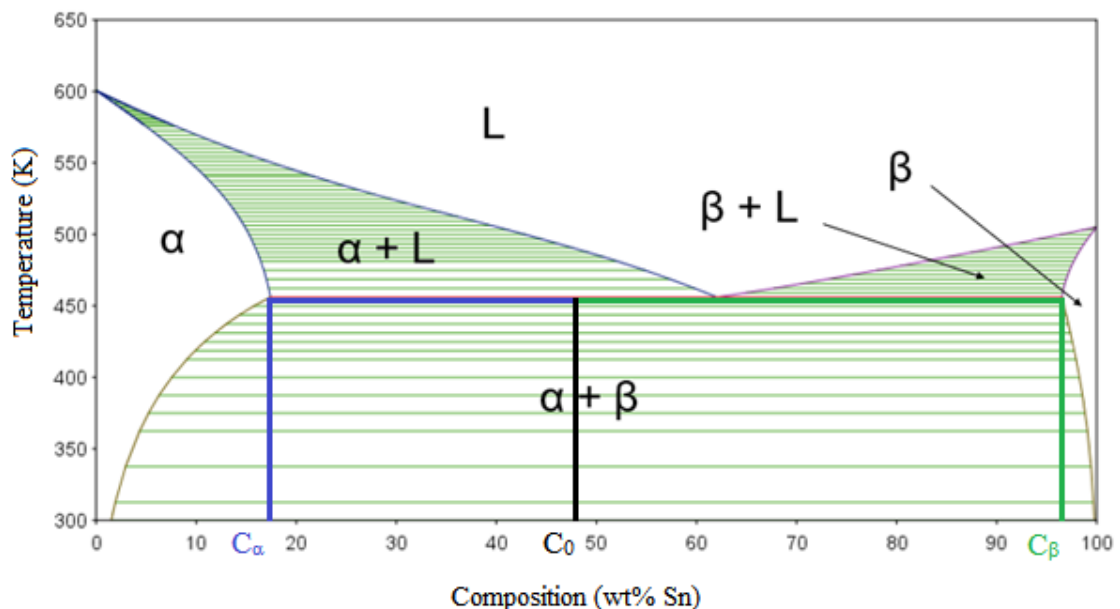


Figure 1 – Eutectic phase diagram of the Pb-Sn system.

To calculate the mass fraction of each phase, first, it is necessary to determine the temperature (at constant pressure) at which the analysis will be made and then apply the so-called lever rule. To apply this rule, it is necessary to know the mass composition of the alloy phase under study (named C_0). Having this information, it is easy to consult the eutectic phase diagram to obtain the maximum

concentration of each phase at a given temperature, named C_α and C_β , respectively, for phases α and β . Assuming the chosen temperature for the calculations is just below the eutectic one (Figure 1), i.e., 182.9 °C, to obtain the mass fraction of the present phases α and β , one just has to use the corresponding relationships to Equation 1 and Equation 2, shown below.

$$f_\alpha = \frac{c_\beta - c_0}{c_\beta - c_\alpha} \quad (1)$$

$$f_\beta = \frac{c_0 - c_\alpha}{c_\beta - c_\alpha} \quad (2)$$

4.2 The Rietveld Method

Developed by the Dutch scientist Hugo Rietveld (1932-2016), the Rietveld method (RIETVELD, 1966; 1968) is used either in the refinement of crystalline structures or in the quantitative analysis of present phases in the microstructure of a certain material using X-ray or neutron diffraction. To do both quantitative and qualitative analysis, the Rietveld method calculates the intensity at each point of the diffractogram and compares this calculated value with the experimentally measured intensity, point to point, trying to minimize the squared differences between them. The method was first released in 1966 (RIETVELD, 1966); however, his work did not have much visibility at first. Only in 1969 the Rietveld method was more accepted by the academic society after it was suggested to apply his method with X-ray diffraction and not by neutrons.

To do both quantitative and qualitative analysis using the Rietveld method, it is necessary to calculate the intensity value of each diffraction peak, since each phase has its peak values. However, it is necessary to do a more detailed analysis in the calculation of these intensities because they are due to factors that can both influence and modify their value, such as the structure factor, the atomic scattering factor, Lorentz-Polarization factor, Debye-Waller, and the multiplicity factor. More details on these factors can be found on Hehnes and Dutra (2020).

4.3 Sample Preparation

The samples used in this work were synthesized at the Mechanical Laboratories Center, poured into the Sand Laboratory of the University Center FEI, presented in the form of ingots. The synthesis of these ingots is the result of a didactic initiation investigation carried out by Feliciano and Dutra (2019). The three ingots used to obtain the investigated samples were a hypoeutectic alloy (Pb-30% Sn), a eutectic alloy (Pb-61.9% Sn), and a hypereutectic alloy (Pb-80% Sn).

To obtain the samples, it was necessary to cut the studied alloy twice so the section with the largest surface area to have a greater incidence of X-rays on the sample, as shown in Figure 2.

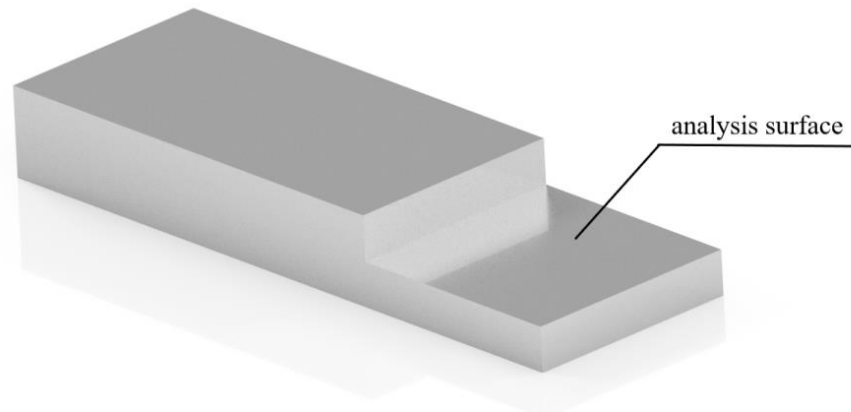


Figure 2 - Analysis surface prepared for the present investigation.

After cutting the sample, the preparation consists of cutting a smaller piece of the alloy, and a metallography resin case was used for mechanical polishing. Mechanical polishing was conducted with silica sandpaper mesh 600, 1000, and 1200, in this order. Polishing cloths with diamond paste 6 μm , 1 μm , and 0.25 μm were used in this order and constant lubrication with alcohol. After obtaining a surface free of roughness and impurities, as shown in Figure 3, it was necessary to remove the sample from the specimen mounting so that the resin would not interfere in the analysis of peaks formed by X-ray diffraction. Figure 3 shows a sample prepared for X-ray diffraction analysis. A Shimadzu[®] XRD-700 was used for this purpose with a Cu-target at 40 kV voltage, with an interval angle from 30 to 100 $^{\circ}$, velocity 1 $^{\circ}$ /min, and step 0.01 $^{\circ}$. The whole experiment took 70 minutes.

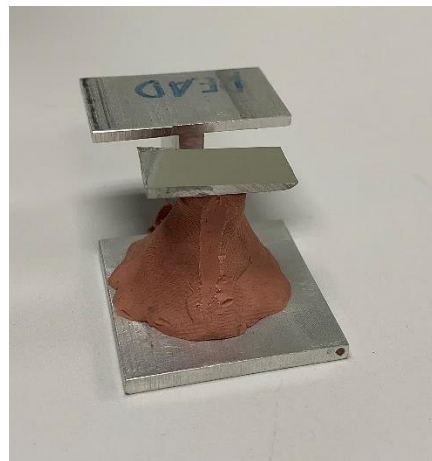


Figure 3 - Sample prepared for analysis.

44 The Rietveld Method Using the Software Maud[®]

The application of the Rietveld method to do both the quantitative and qualitative phase analysis can be done with the aid of software, which makes the necessary iterations to characterize the existing phases by comparisons between the observed diffraction pattern and a calculated pattern, called crystallographic parameters. The software Maud[®] was chosen for this purpose.

To analyze the diffraction patterns obtained from these tests, it is necessary to open the Maud[®] software and open the desired diffraction pattern, in “txt.” format, via the “Load Datafile” command in the “File” menu. It is important to note that this file must be opened in the “Datasets” tab. Once the file is obtained, it is then necessary to open the CIF file (s) for comparison and the determination of phases. CIF files must be opened in the “Phases” tab. For tin, the CIF file code is 9008570, and for lead, 9013418, which can be obtained on the site crystallography.net (DAY, 2020).

To start the analysis, it is necessary to search in the menu for a function named “Analysis” and after opening it, one must click on the “Wizard” option so it is possible to select the parameter to be analyzed; in this case, the phases present in the sample from the option "Quantitative Analysis". The result of this analysis for a eutectic alloy is shown in Figure 4, which shows the existing phases in the CIF files overlapping with the diffraction pattern found experimentally, as well as the indication of the phases present in each peak.

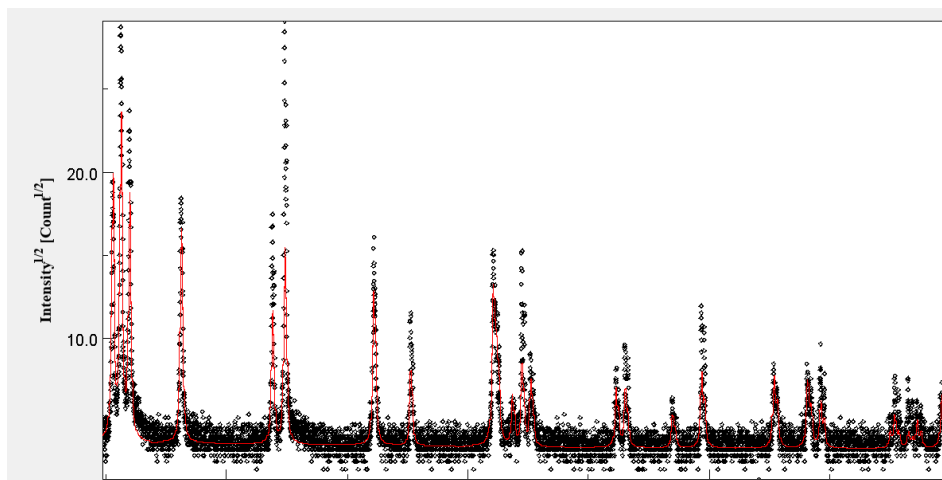


Figure 4 - Results of quantitative phase analysis of the eutectic alloy.

To refine the results and make them more accurate, it is possible to make the texture analysis before doing the quantitative one. This analysis can be done because in some diffraction peaks, both the experimental and the theoretical results can present differences, making one peak higher than the other and one of the main causes of this inaccuracy may be due to texture. The results after the texture analysis are shown in Figure 5.

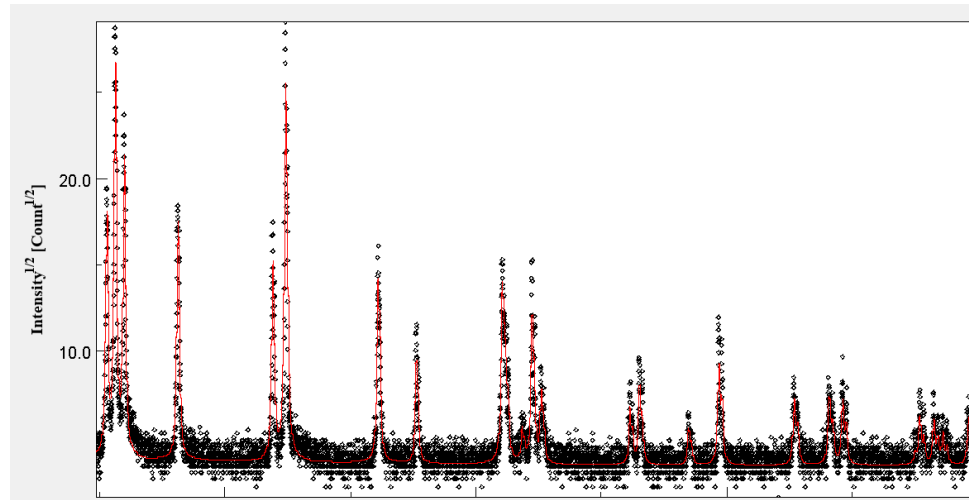


Figure 5 - Results of quantitative phase analysis of the eutectic alloy with texture analysis.

4.5 Results and Study Application

Table 1 shows the results found for the α phase (rich in lead) and the percentage of error, when compared to the result obtained using the phase diagram.

Table 1 - Test results with the comparison between theoretical results and experimental.

Type	Composition	Eutectic (Pb-61.9%Sn)	Hypoeutectic (Pb-30%Sn)	Hypereutectic (Pb-80%Sn)
	C_0	61.9	30	80
	C_α	2	2	2
	C_β	99	99	99
Diagram	f_α	0.3825	0.7113	0.1959
Maud	f_α	0.3925	0.6823	0.2052
	$\Delta\%$	2.9%	-4.1%	4.8%

Part of this work was put into practice in the second half of 2020, using the Maud[®] software to compare the results of mass fractions, on the students' computer for the classes MRD220 – Metallic Materials from the Mechanical Engineering course of the University Center of FEL.

Videos were recorded to show the X-ray diffraction exam and made available on the Youtube[®] channel, as well as videos were recorded explaining the use of the Maud[®] software. After conducting this class in which students had the opportunity to learn about the software and compare the results obtained theoretically and experimentally, a survey was carried out in the form of a questionnaire through the website mentimeter.com[®] to assess whether the use of the Maud[®] software for determining the mass fraction of the elements of the lead-tin alloys contributed positively to the understanding of the concepts related to phase diagrams, pointing out that 95% of the students judged the use of the software useful for the understanding of a eutectic phase diagram.

5. Characterization of Pb-Sn microstructures

Metallography is a technique for the characterization of metals and their alloys that comprehend the microstructure observation and the consequent analysis of the morphology,

distribution, orientation and, quantity of the features present. Metallography characterization is used in materials specification, process control, quality control, and quality assurance.

This technique consists of sectioning, embedding, grinding, polishing, and finally etching a metallic sample. In this project, the investigated alloys were used from another study made by Feliciano and Dutra (2019). Three samples of different chemical compositions were selected, one hypoeutectic with Pb-30%Sn, the second was a eutectic alloy with Pb-61.9%Sn, and the last one a hypereutectic with Pb-80%Sn. These samples were first sectioned in three different directions. A total of 9 samples were studied. In this paper, only the sectioned plan B of the eutectic alloy will be presented, because this was the sample that presented the best results in the experiment, as shown in Figure 6.

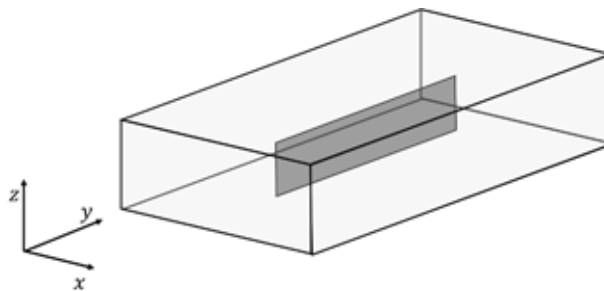


Figure 6 - Schematic picture, showing the cutting plane denominated “B”, in which the transversal section is perpendicular form x axis, which means that is the $yz \perp x$ plane.

After sectioning, the units were placed in a specific silicone vessel to embed with cold-curing resin for 24 hours. The product used was a mixture of resin and catalyst in a proportion of $\frac{1}{2}$ of catalyst for one part of resin.

For the grinding process, as tin-lead alloys are so soft, several sandpapers were used to promote a better finishing of the sample, each of them with the mesh size of 240, 400, 600, and 1000, in this order. During the process, water was used as a lubricant. Then, polishing was conducted with three types of cloth, all of them with diamond paste and alcohol as a lubricant. The procedure started with a $6\text{-}\mu\text{m}$ diamond paste, used to remove the scratches caused by the grinding stage, followed by a $1\text{-}\mu\text{m}$ diamond paste, where it stayed for 15 minutes, under a small pressure and with circular movements; finally, a $0.25\text{-}\mu\text{m}$ diamond paste was used the same way as the step before, but now for 20 minutes. It is important that the used cloth must not contain any dirt or rips.

At the end of every cycle, the samples were observed on the microscope to verify if the polishing was effective on the removal of scratches. Before etching, all samples were analyzed on a microscope. To use it, it was necessary to fasten the sample with a lump of modeling clay in a metallic plate. Then a leveler was used, to keep the surface of the sample parallel with the objective lens of the microscope. After that point, the images were captured by the Stream Essentials[®] software for future comparisons.

The best etchant for these alloys was found to be the one with the following composition: 10 mL of silver nitrate and 90 mL of distilled water, the etching was done by swabbing a piece of cotton soaked into the solution on the sample for 1 minute. After that, 90 images in total, being 10 of each sectioned plane were captured by Stream Essentials[®] software.

A typical microstructure of the eutectic alloy can be seen in Figure 7. It is possible to see a rich tin microstructure, represented by the β -brighter phase while the α phase is characterized by the dark regions.

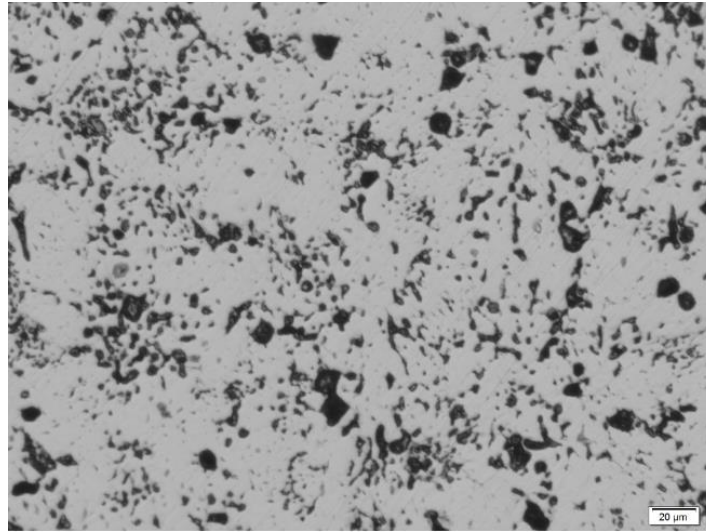


Figure 7 - Light Micrograph of the eutectic alloy Pb-61.9%Sn, section B.

5.1 Measurement of Fraction in Area

To make this analysis, it was necessary the use ImageJ[®] software, which can be downloaded from this site <https://imagej.nih.gov/ij/download.html>. To use it, it is necessary to duplicate the image so that the original one does not change. To start measuring, one needs to set the scale to the micrograph and then adjusted for a black and white filter.

The threshold command is responsible for making the area measurement of the image, an important part of this stage is that, as the picture can have a third color, more grayish, sometimes the software can make a wrong reading, counting this color as part of one of the phases, so then, it is up to the software manipulator, to control what is being read in the measurement of the area fraction.

After having the are percentages of each phase, it is possible to convert them into area fractions to compare with the expected area fraction of each phase from the alloy based on its phase diagram. This conversion is important to show the precision of the study. By calculating the mass fraction of each phase with the lever rule from the Pb-Sn diagram at room temperature for each alloy, it was possible to obtain the expected volume fraction. The theoretical density values were obtained using the ideal values from Callister and Rethwisch (2015). They were then compared to the experimental ones.

5.2 Analysis of Variance

The analysis of variance is the main and most important technique used to solve problems, and it was initially created as an instrument for analysis for agricultural equipment by the statistic R. A. Fischer (COSTA NETO,2002).

It is a powerful technique used to compare the means of three or more variables and thus, to identify if those means are different from one another, in other words, what this method does is to

verify the variation between groups and within groups. This method allows various groups to be compared at the same time. Some important characteristics of this analysis are that the sampling should be random, the variables must be independent, and the data must obey a normal distribution.

In this study, an analysis was made for each alloy, in each sectioned plane A, B, and C, adopting a significance level of 0.05 (5%). The most practical method of doing this analysis was with Excel[®], with a function named Data Analysis in which one only needs to choose the option Anova: unique factor, select an entrance gap, then the grouping type, in other words, select if the data analyzed are organized in columns or lines, then determine the significance level, in this case, it was 5%, which means that the certainty of the results is about 95%, and last, one must choose the exit interval, in which a table will be presented.

5.3 Variance Analysis for Eutectic Alloys Pb-61.9%Sn

Table 2 shows the mean and variance of area fractions from the three different sections. This analysis was made concerning the cutting sections to note any discrepancies between them and between the other alloys. The chosen significance level was 5% because it was the most precise. The results of the analysis of variance can be observed in Table 3.

Table 2 – Summary of part of the calculations of the analysis of variance made in Excel[®] for eutectic composition, Pb-61.9% Sn. Values are expressed as a percentage.

Section	Count	Mean	Variance
A	10	72.27	1.27
B	10	73.49	18.58
C	10	70.50	3.39

Table 3 – Results of the analysis of variance in Excel[®] for the eutectic alloy Pb-61.9% Sn.

Source of Variation	Sum of Squares	Degrees of freedom	Medium Squares	F Statistic	P-Value	F Critic
Between Groups	45.23	2	22.62	2.92	0.071	3.35
Inside Groups	209.16	27	7.75	-	-	-
Total	253.39	29	-	-	-	-

The sum of squares measures the data variation, i.e., it measures the variation of the area fraction from the different micrographs referring to the cutting sections and between sections, the degrees of freedom are calculated with the number of analyzed images and the medium square is the ratio of the sum of squares to the degrees of freedom. The F statistic from Fisher Snedecor is calculated by the ratio of the mean square between the groups and the mean square within the groups; last, the P-Value is the measure of the probability of the difference observed between the groups being caused by random reasons and not because of the studied factors. About the F critic, this is obtained from a table of distribution for a 5% significance level.

To conclude the analysis and to verify if there is any difference between a measure distribution between three or more groups, one must have two hypotheses:

H_0 = known as a null hypothesis, where all means are equals, which means that there are no significant statistical differences between the means of groups;

H_1 = known as alternative hypotheses, where all the population means are different, i.e., at least one of the means is different from the others.

In this case, as the P-Value is 0.071 it means that by repeating the experiment 100 times, there is a 92.9% of chance of similar results being found. With this analysis, it is possible to conclude that, as the Statistic F value is lower than the F critical value, there are no significant differences between the means of the groups. This also can be proved with a P-Value that had a result greater than the level of significance, once again validating the null hypothesis.

5.4 The Study Application

For the students, the only replicable part would be the microscope analysis and the software manipulation, mainly due to the Covid-19 pandemic. There is also a problem related to mechanical polishing which is painstaking and difficult to be successful.

This study was carried out in the school environment in the second half of 2020. The three first classes of MRD220 – Metallic Materials from the Mechanical Engineering course of the University Center of FEI studied phase diagrams and all the important concepts for their interpretation.

Initially, a video was made that is now available on social media Youtube[®], showing how to download the ImageJ[®] software, how to install it, and finally how to use it to measure are fractions. The students did not show any difficulty in this part, while the use of the software was taught by the professor during the class. The purpose of this was to divide the class into three groups and asked them do the characterization of the phases involved on the Webex[®] platform.

After the class, the students were asked about the use of the ImageJ[®] software, if that helped them to understand phase diagram concepts. Only 22 of the 80 students answered. The results showed that 59% of the interviewees believed that the use of the software helped them to have a better comprehension of the phase diagram and the concepts related to it.

6 Conclusions

The present paper presents an example of active learning concerning phase diagrams, more specifically eutectic phase diagrams. Students were asked to use different types of software to obtain the mass fraction of phases present in different Pb-Sn alloys and compare them to those obtained experimentally from X-ray diffraction and quantitative stereology.

The results show that important concepts related to phase diagrams are better understood by the students when confronted with experimental results, their limitations, and by using different types of software to characterize the phases involved. It is certainly this practical approach that is likely to improve the competencies of our students when it comes to articulate both concrete and abstract aspects typically required in engineering problems.

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