Teach Phase Equilibria to Students of Sn-Bi alloys Using a Differential Scanning Calorimeter and X-ray Diffraction (XRD)

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Abstract

In the past, lead and tin was used as the primary material in soldering due to its low melting point. However, as medical research revealed the hazards of lead to humans a search began for a replacement. One such material is an alloy of bismuth and tin. The objective of this study is to obtain familiarity with using both DSC and XRD, as well as generate data to analyze and learn more about Bismuth and Tin mixtures. DSC and XRD are essential to the future of materials engineering in the field of materials characterization and identification. As an aid for teaching phase equilibria to undergraduate students, we have designed a laboratory exercise that allows them to create a phase diagram from data produced by DSC and hands-on insight by an exposure to modern analytical instrumentation and practice quantitative skills such as calculation (e.g., peak temperatures with % of Bi-Ti alloys, phases, graphing etc.). The samples is also analyzed through the use of a DSC in order to learn at what temperature different compositions of this material undergo phase changes and to create an experimental phase diagram and 57wt% Bi would be the best fit for soldering as an replacement for traditional lead-tin solder.

Key words

primary soldering material, Sn-Bi alloys, Bismuth and Tin mixtures DSC, powder XRD, replacement for lead

1. Introduction

In the past, lead was used as the primary material in soldering due to its low melting point. However as medical research revealed the hazards of lead to humans a search began for a replacement. One such material is an alloy of bismuth and tin. Through the use of X-Ray Diffraction (XRD), samples of tin, bismuth, and alloys of different compositions of these two materials will be analyzed to determine what elements and crystalline structures are present. The samples will also be analyzed through the use of a Differential Scanning Calorimeter (DSC) in order to learn at what temperature different compositions of this material undergo phase changes and to create an experimental phase diagram. Finally, an unknown metal will be identified by manually indexing an XRD diagram to discover its crystalline structure and atomic radius.

1.1 Collaborative Approach

A leadership responsibility for lab work was taken by making sure the proper procedures are being followed and that all data is being collected. Student #1 in each group would take leadership responsibility for analyzing data and writing the report for one of the two main processes in each report. Student #2 would take the lead on other assignments such as the teamwork questionnaire, making graphs from data, and making sure everyone has access to the right documents. Student #3 took leadership responsibility for proofreading and editing reports along with making sure everyone is staying in communication. All group members will be responsible for communicating with each other and checking each other's work so fewer mistakes are made.

Through a collaborative group messaging system, the team will communicate the various roles and designations for given assignments. Meaning, different team members will focus on specific parts of a

group project so that everyone is included. This ensures that no person feels excluded by not being allowed to participate in the project. It will be the duty of the remaining team members to review and approve of each individual team member's work. This allows for a system of checks and balances making sure that no team member falls behind and allowing for multiple team members to work together if one person gets stuck. If one person is unable to complete a designated task the team will come together and work as a collective to finish the task.

2. Overview of study

2.1 Purpose of the study:

Any phase diagrams may be developed by melting samples of crushed metals containing different proportions of elements like Pb-Sn or Sn-Bi etc. Once equilibrium is established, the samples are quenched and then analyzed for the relative proportions of crystallized minerals and vitrified melt stable under the experimental conditions. Usually two or more metal components alloys are bonded or joined by solders which are used extensively in the electronics industry to physically hold assemblies together; they must allow expansion and contraction of the various components, transmit electrical signals, and dissipate any heat that is generated. Melting the solder materials is accomplished by bonding action and allowing it to flow among and make contact with the components which do not melt to be joined; finally, upon solidification, it forms a physical bond with all of these components.

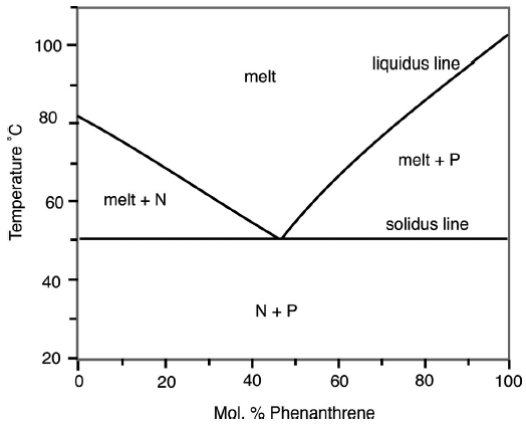

Figure 1: Binary phase diagram for the system naphthalene-phenanthrene (after Gallus et al., 2001).

Depending on composition, several different types of microstructures are possible for the slow cooling of alloys belonging to binary eutectic systems. These possibilities will be considered in terms of the lead–tin phase diagram, Figure 1 (Callister and Rethwisch, 2014). The first case is for compositions ranging between a pure component and the maximum solid solubility for that component at room temperature.

A group of analytical techniques thermal analysis (TA) is that measure properties or property changes of materials as a function of temperature. Changes in materials properties with temperature are considered as thermal events. The properties that can change include dimension, mass, phase and mechanical behavior. Thermal analysis methods are relatively simple because changing the temperature of a sample is less complicated than probing a sample using high-energy X-ray, electron or ion beams in techniques of spectroscopy. Many TA methods have been developed for a variety of examination purposes and one of the most commonly used thermal analysis techniques for materials characterization is differential scanning calorimetry (DSC) and widely used for examining the phase changes of materials. This technique has the same objective: to examine thermal events in a sample by heating or cooling without mass exchange with its surroundings. The thermal events examined by DSC include solid phase transformation, glass transition, crystallization and melting. 'Differential' emphasizes that analysis is based on differences between sample material and a reference material in which the examined thermal events do not occur.

Differential Scanning Calorimetry

A DSC instrument is designed to measure the heat flow difference between sample and reference. There are two widely used DSC systems: the heat flux DSC 'quantitative DTA' and the power-compensated DSC. It measures the temperature difference directly and then converts it to heat flow difference as illustrated in Figure 2a. This conversion is accomplished by an algorithm in computer software installed in the system. In power-compensated DSC, there are two separate chambers to contain the sample and reference. Each chamber has its own individual heat element to control temperature (Figure 2b). When a thermal event occurs in the sample, the power to the heating element has to change in order to maintain the sample temperature the same as that of the reference. An endothermic event causes an increase in power to heat the sample; an exothermic event causes a reduction in power to cool the sample. The amount of power change should equal the energy of heat flow to compensate for the heat release or gain of the sample.

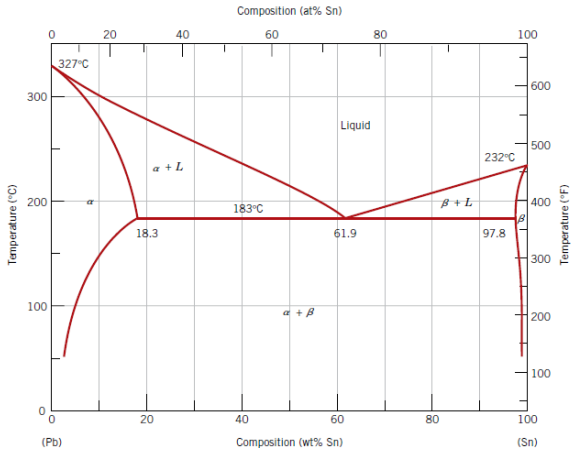

Figure 2: The lead–tin phase diagram. [Adapted from Binary Alloy Phase Diagrams, 2nd edition, Vol. 3, T. B. Massalski (Editor-in-Chief), 1990. Reprinted by permission of ASM International, Materials Park, OH.]

A DSC curve is illustrated in Figure 3 in which heat flow is plotted versus temperature. The heat flow has a unit of energy per unit time per unit mass. Commonly, heat flow into a sample is indicated as an upward feature of the DSC curve. The DSC curves are commonly recorded over a temperature range by heating or cooling a sample with a constant rate.

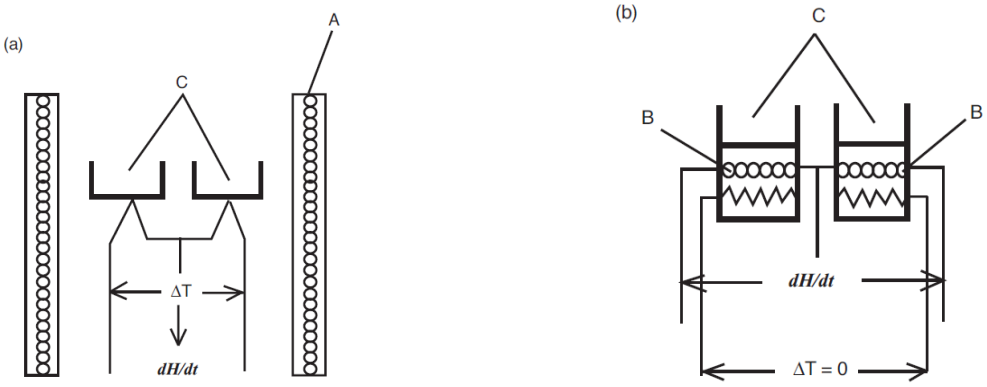

Figure 3: Differential scanning calorimetry (DSC) instrumentation design: (a) heat flux DSC; and (b) power compensation DSC. A, furnace; B, separate heaters; and C, sample and reference holders. (Reproduced with permission from E.L. Charsley and S.B. Warrington, Thermal Analysis: Techniques and Applications, Royal Society of Chemistry, Cambridge, UK. Ⓒ 1992 Royal Society of Chemistry.)

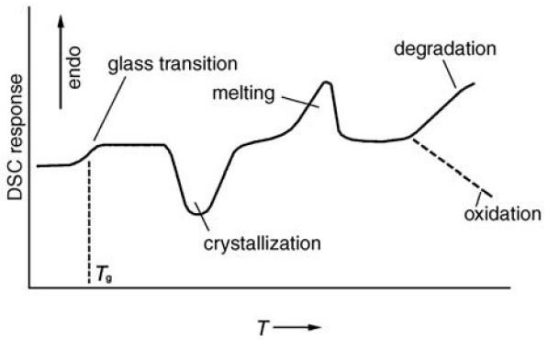

Figure 4: Schematic DSC curves for a polymeric sample. Tg, glass transition temperature. (Reproduced with kind permission of Springer Science and Business Media from M. Brown, Introduction to Thermal Analysis, Kluwer Academic Publishers, Dordrecht. © 2001 Springer Science.)

2.2 Statement of Problem and its significance

Currently a lower temperature melting point lead-tin solders, a shiny finish, which makes it easier to notice under oxidation [pcboardrework (2020)] allowing the components to be less damaged by the process are widely used because it solidifies faster. However, a lead solder also has some dangerous disadvantages; hazardous for humans because it can cause asthma or eye/upper respiratory tract irritation [Blink.ucsd.edu. (2020)]. Under the above circumstances there are lead-free solder are benchmarked such as bismuth and tin. The characteristics of solder elements: bismuth has an even lower melting point than lead (~138° C) and tin has a corrosive property. By using the DSC, the different melting temperatures of Sn-Bi alloys can be explored. The DSC (Differential Scanning Calorimeter) measures the material changes with respect to temperature and time, this can be used to find melting points and any chemical reactions that occur. Using the XRD (X-ray Powder Diffraction), the unknown materials can be indexed by calculating the lattice parameter and the atomic radius to discover the unknown material. By these two types of characterization tests, one can find the best combination of Sn-Bi alloy for soldering to replace Pb-Sn.

3. Literature Review

This lab is a chance for a hands-on experience with Materials Engineering practices, processes, documentation, and instruments. It will introduce you to the science and practice of: X-Ray Diffraction (XRD) and Differential Scanning Calorimetry (DSC). First, we will work on safety procedures. Safety is always of prime importance in any Cal Poly lab. We also emphasize professional practices and written communications including lab notebooks, lab reports, and other important forms of written communication and documentation. There will be opportunities for you to gain professional practice and enjoy learning new information and gain new skills. Professional engineering performance begins with accurate and complete record keeping in lab. The proper use of Lab Notebooks is an important part of engineering practice to follow the guidelines. It is possible that these guidelines are somewhat different from what you have done in the past.

In DSC, the common exercises involve pursuing the paths of melting or crystallization for a given composition as temperature changes (Glazner, 2003) or constructing a phase diagram using a supplied data set (Smith, 2003). It is evident that students can explore the systematics of a diagram and respond correctly to specific questions while these exercises are in effect.

This paper tells phase diagrams may also be constructed via thermal analysis techniques like DSC. It postulates that heat is absorbed (enthalpy of the reaction or latent heat of melting) when a given substance is heated and starts to melt. In contrast releases heat (latent heat of crystallization) by the formation of crystals during solidification. Thus, identification of temperature as a sample is warmed or cooled from solid–liquid phase transitions are significant in DSC. Sharp changes in the slope (dT/dt) of the temperature

curve signal melting or crystallization and can be used to map the liquidus and solidus curves. The eutectic composition can be estimated by extrapolating the liquidus curves down toward the eutectic temperature by analyzing a number of sample mixtures for solidus and liquidus lines. As a result, delineating the phase boundaries in the N–P system by plotting all of the student-produced data after three administrations of this lab exercise illustrates the effectiveness of the technique for (Fig. 5).

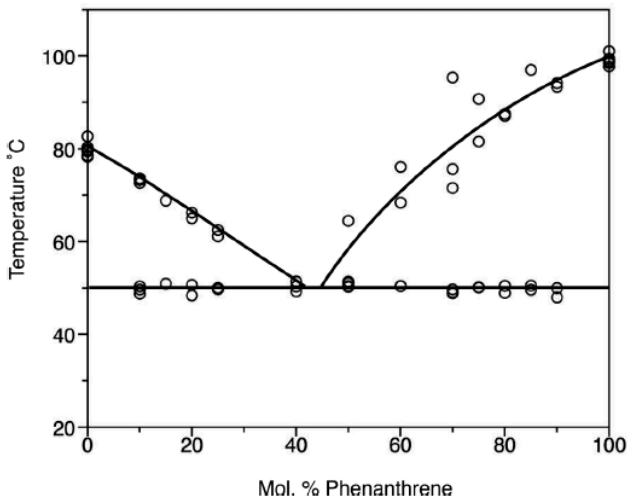

FIGURE 5: N–P [naphthalene (N) – phenanthrene (P)] phase diagram based on data collected by students in 2007, 2009, and 2010. (Anton et al., 2011)

4. Methodology

4.1 X-Ray Diffraction

The X-ray diffraction spectra of as-received silica fume were taken using an X-ray diffractometer [Phillips Powder Diffractometer; Cu X-ray tube. Copper K_α radiation with the wavelength of 1.540560 Å was used as a monochromatic X-ray source to bombard the sample. The operating voltage and current were 25 - 40 KV and 40 mA respectively. The scans were conducted over a 2θ angular range of 10° - 100° with a speed of 1 degree per minute. The XRD patterns obtained were used for both qualitative and quantitative determination of the existing phases within the sample. Important advantages and uses of powder diffraction:

i. The need to grow crystals is eliminated.
ii. A powder diffraction pattern can be recorded very rapidly and the technique is non-destructive.
iii. With special equipment very small samples may be used (1-2 mg.)
iv. A powder diffraction pattern may be used as a fingerprint. It is often superior to an infrared spectrum in this respect.
v. It can be used for the qualitative, and often the quantitative, determination of the *crystalline components* of a powder mixture.
vi. Powder diffractometry provides an easy and fast method for the detection of crystal polymorphs. Powder patterns are provided when a drug is being registered with the FDA. (Polymorphs are different crystal forms of the same substance.)

4.2 Differential Scanning Calorimetry (DSC)

Samples for DSC should be in the form of dense powder or small discs. Film, sheets and membranes are often cut into discs fitting into the sample pans. Large shear forces during cutting samples should be avoided because shear-force may induce plastic deformation in a sample and that can affect DSC curves. Low-mass samples are also preferred because a low-mass sample can quickly reach temperature equilibrium

throughout its volume. A large-mass sample will have undesirable internal temperature gradient and this will affect the accuracy of the curve. Commercial DSC instruments are able to measure phase change in microgram samples. In practice, the lower limit of sample size relates to the nature of sample materials. For composites and polymer blends, large sample size of about 10 mg may be required. DSC is usually operated in a low temperature range (<500° C), and thus aluminum pans are commonly used as sample and reference holders. The pans often need to be sealed to avoid sample mass change due to evaporation. A special press can be used to mechanically weld a lid and a pan together.

Separating the reversing and non-reversing heat flow components and can distinguish overlapping thermal events. Figure 6 illustrates the examination of a poly (ethylene terephthalate)–poly(acrylonitrile–butaliene–styrene) (PET–ABS) polymer blend. Figure 6a shows conventional DSC curves of the polymer blend and reveals the PET glass transition at 340 K, the PET cold crystallization at 394 K and PET fusion at 508K (not shown). The ABS glass transition can only be revealed by a second heating after the first heating and cooling as first heating curve overlapped with the PET cold crystallization peak. However, it clearly shows both PET and ABS glass transitions in its reversing curve (Figure 6b).

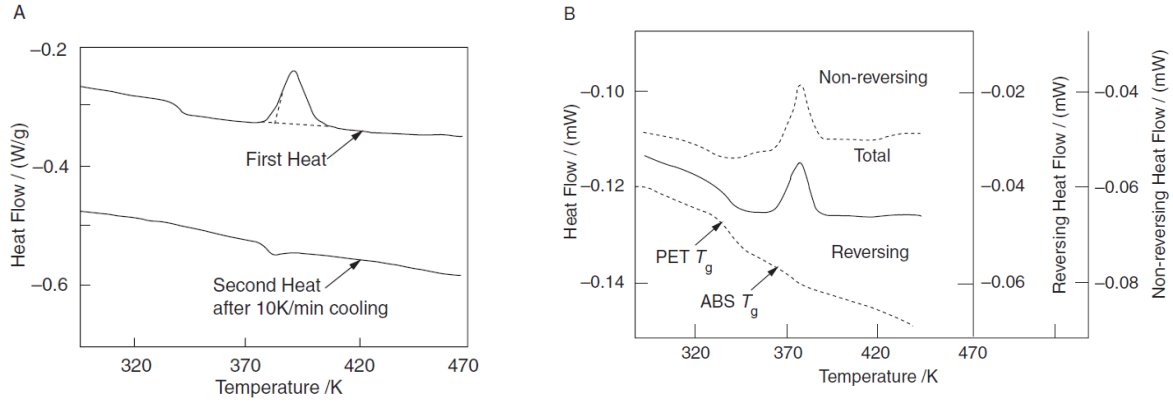

Figure 6: DSC curves of poly (ethylene terephthalate)–poly(acrylonitrile–butaliene-styrene) (PET–ABS) blends: (a) conventional DSC first and second heating curves with heating and cooling rate of 10Kmin^{-1}; and (b) temperature modulated DSC (TMDSC) first heating curves with β = 2 Kmin^{-1}, p = 60 s and B = ±1K. T_g, glass transition temperature. (Reproduced with permission from T. Hatakeyama and F.X. Quinn, *Thermal Analysis: Fundamentals and Applications to Polymer Science*, 2nd ed., John Wiley & Sons Ltd, Chichester. Ⓒ 1999 John Wiley & Sons Ltd.)

To show proficiency in identifying unknown materials through XRD analysis, manual indexing will be used to determine what an unknown sample material is. Starting with the mathematical analysis, integer values are found for $3\sin^2(\theta)/\sin^2(\theta_{min})$. This gives us values of $h^2 + k^2 + l^2$ that imply Miller indices of 111, 211, 220, 311, 222, and 400 which indicates a Face Centered Cubic (FCC) crystalline structure. Using λ = 1.5405 Å both the interatomic spacing and atomic radii can be calculated for each peak. When these are averaged the result is an atomic radius of 1.245 Å which, when also considering the FCC crystalline structure, indicates the unknown element is Nickel.

For assurance, the unknown element is Nickel the same data was also examined through analytical analysis. Integers ranging from one to ten were used in $\sin^2(\theta)/x$ in order to find the lowest common quotient which our data provided as K = 0.04786. Once again, the values found by $\sin^2(\theta)/K$ indicate Miller indices of 111, 211, 220, 311, 222, and 400 again implying a FCC crystalline structure. The interatomic spacing was calculated using $a = \lambda/(2(K)^{1/2})$ and then the atomic radius was calculated using $r = a(2)^{½}/4$ in order to get an average atomic radius of 1.245 Å once again. The FCC crystalline structure and calculated atomic radius indicates Nickel which also has a FCC crystalline structure and a similar atomic radius of 1.246 Å.

Considering the results are consistent across both forms of manual indexing it is safe to conclude that the unknown material is nickel.

5. Results and Discussion

5.1 X-Ray Diffraction (XRD)

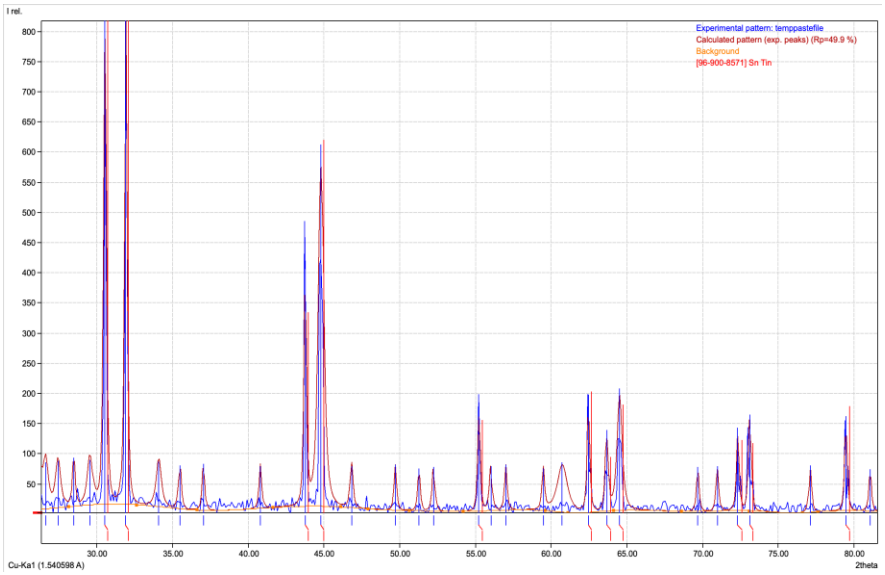

Figure 7. Pure Sn, blue spectrum, graphed with the Sn reference pattern, red.

XRD analysis software (Match) enabled to determine that the pure Sn data was consistent with pure Sn reference patterns. The capability of being able to do this by comparing our data's peaks with the reference pattern's peaks. All of the peaks from the experimental pattern are accounted for when comparing to a pure Sn reference pattern. Then repeated this for all compositions of the Sn-Bi alloys. When looking at the Sn Bi phase diagram there should be proeutectic β-Sn and Bi present in all samples (Figure 7).

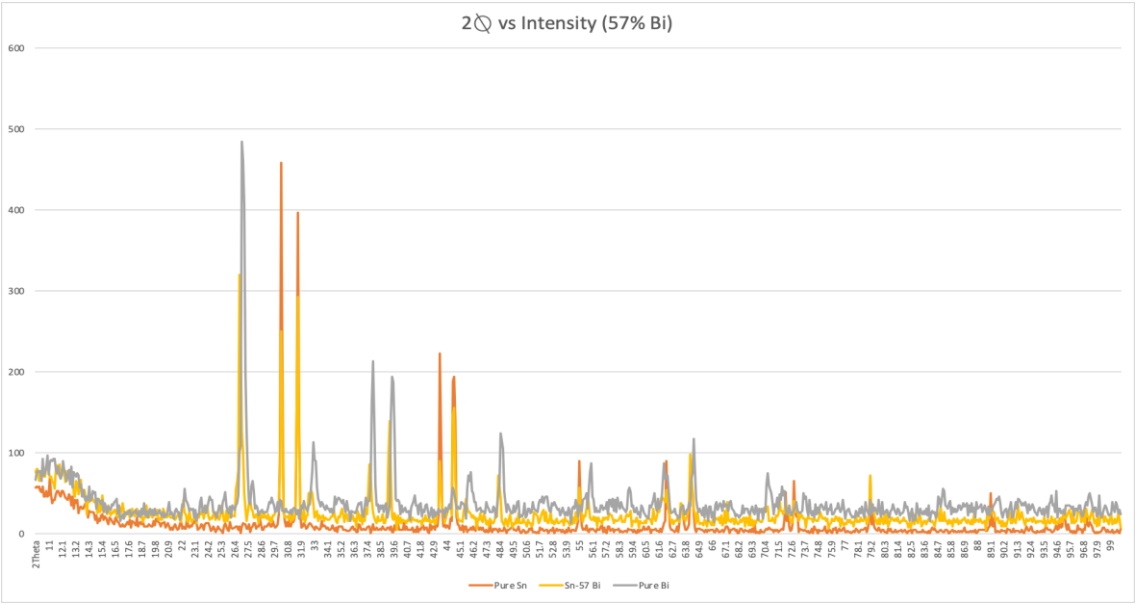

Figure 8. A Sn-Bi compound with 57wt%, in yellow, is graphed with Pure Sn and Pure Bi, in orange and grey respectively.

When analyzing this graph, it is evident that both the experimental data and Sn reference pattern match up in but are not completely lined up, this deviation confirms the presence of another element in the compound. In contrast, the large experimental peaks line up with the Sn reference pattern while the smaller experimental peaks are lined up with Bismuths' reference pattern, so we can conclude that this compound is comprised of Sn and Bi. However, analysis software did not find any matches with Sn or Bi for the 57wt% Bi Sn-Bi compound so we then plotted the experimental data along with the Pure Sn and Pure Bi data, which was already confirmed to be those elements (single large peak, present in the Bi spectrum and two large peaks for Sn are also present in this composition which means it is comprised of both these elements (Figure 8).

5.2 Differential Scanning Calorimetry (DSC)

- For the Pure Sn sample the glass transition temperature was observed at 224° C. On the heating curve the sample was melting from around 230 to 260° C . On the Cooling Curve the sample was solidifying from 215 to 195° C.
- In the Sn70-Bi30 The glass transition temperature could not be determined from the data available. The melting temperatures were from 235 to 246° C during the heating phase. The sample was solidified from 218 to 200° C during the cooling phase.
- The Sn43-Bi57 sample had a glass transition that could not be determined from the data collected. The sample had melting temperatures from 142 to 155° C during the DSC's heating process. The sample was solidified from 128 to 112° C during the cooling process.
- For the Sn10-Bi90 the glass transition temperature was observed to be 140° C. The sample was melting from 230 to 250° C during the heating phase. The sample was resolved on the cooling phase from 165 to 135° C.
- Pure Bi was found to have a glass transition temperature at 231° C.
- During the heating portion of the trial it was observed to be melting from 273 to 277° C. While the sample was in its cooling phase it solidified from 199 to 153° C.

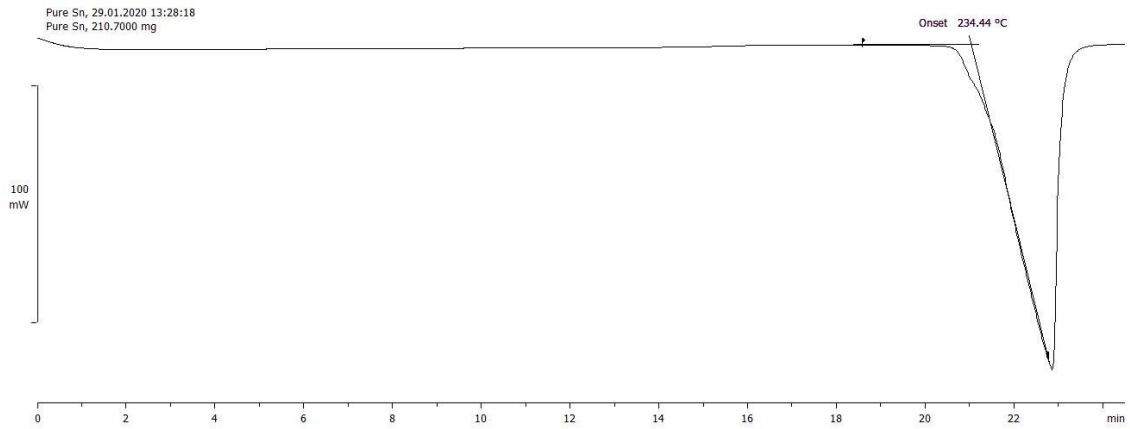

Figure 8. Pure Sn, the peak occurs at 250°C. This is high when compared to a phase diagram but the onset occurring at 234°C is consistent indicating that the wider, less accurate peak is the result of some contaminants in the sample. The phase change that occurs on cooling is only apparent from the collected data and occurs at 221.1°C. When analyzing this data it shows the onset temperature, at which a phase change occurs, is 234.44°C. This is consistent with the Sn-Bi phase diagram where β-Sn turns to liquid at 232°C. The slight deviation indicates that there may be some contaminants in the sample.

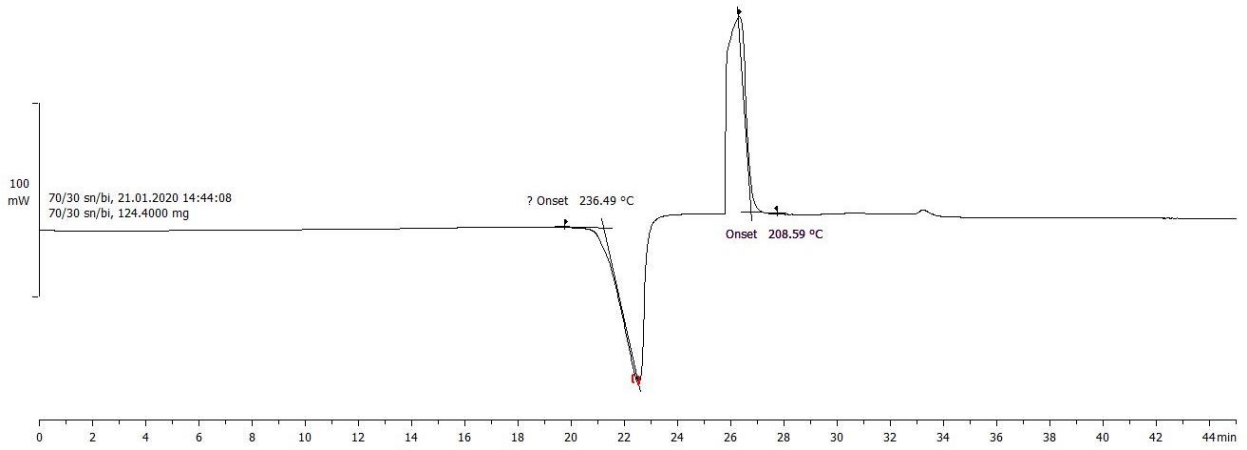

Figure 9. 30wt% Bi, the first peak occurs at 249°C while the second occurs at 213.7°C. It seems like the melting point occurs at a higher temperature than expected but the phase change of returning to a solid match what was expected.

The onset temperature for the heating process is 236.49℃ which is inconsistent with the phase diagram. The second onset temp for the cooling process is 208.59°C which is consistent with the phase diagram for passing through the liquidus line (Figure 9).

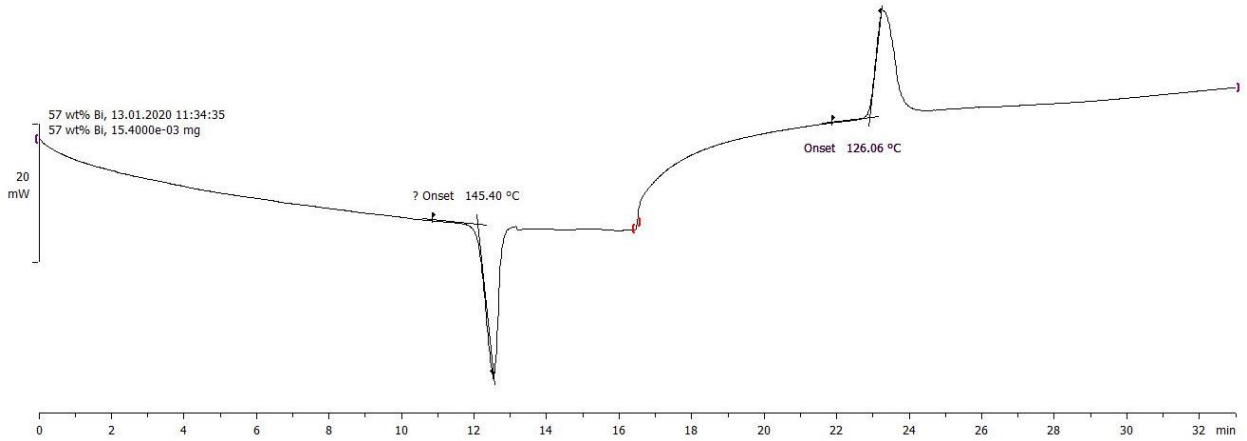

Figure 10. 57wt% Bi, first peak occurs at 148.3°C, second peak occurs at 121.7°C. Here the melting point occurs at the expected temperature while the transition back to a solid occurs at a slightly lower temperature than expected.

The onset temperature, 145.40℃, is significantly lower than the previous compositions indicating that 57wt% Bi is the eutectic composition and that this is the eutectic temperature. With this in mind, there should not be a second peak, indicating this peak may be insignificant noise. This would be the ideal composition for soldering use as it has the lowest melting temperature and does not go through the two-phase region (Figure 10).

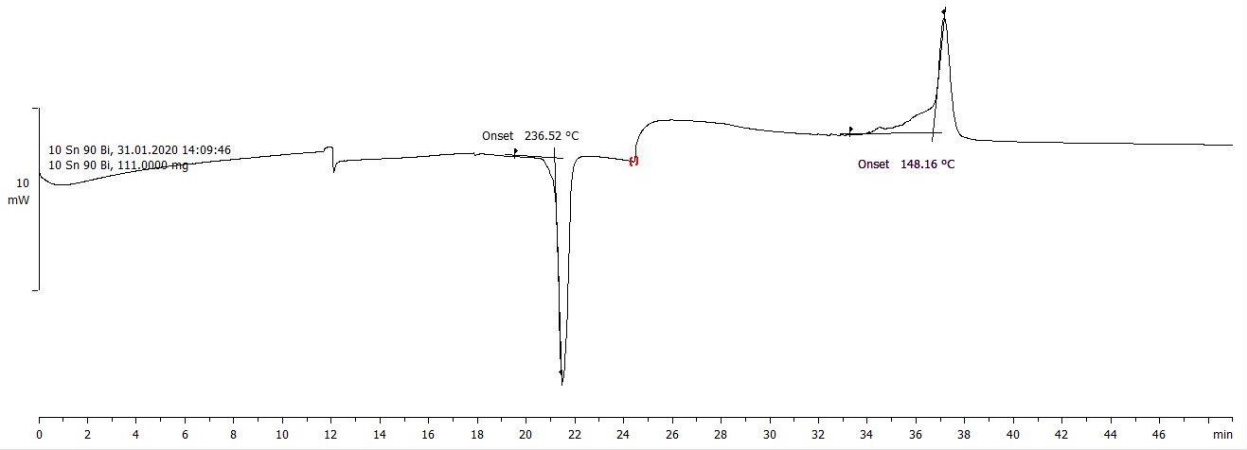

Figure 11. 90wt% Bi, first peak occurs at 239.3℃, second peak occurs at 145.2℃. Here both phase changes occur as expected.

The first onset temperature 236.3℃ is consistent with passing through the liquidus line, the second onset temperature of 148.16℃ matches the phase change going through the eutectic temperature, Bi+L to Bi+Sn (Figure 11).

The first onset temperature is 274.72℃, which is consistent with the phase change going through the liquidus line, from L to Bi + L. However, the second onset temperature is inconsistent with the Sn-Bi phase diagram and is smaller than all previous significant peaks. Because of these reasons we decided to throw out this peak and write it off as insignificant noise on the graph. The third onset temperature is representative of the eutectic temperature, but it is slightly higher than the established 139℃ from the Sn Bi phase diagram indicating there may be some contamination in the sample (Figure 12).

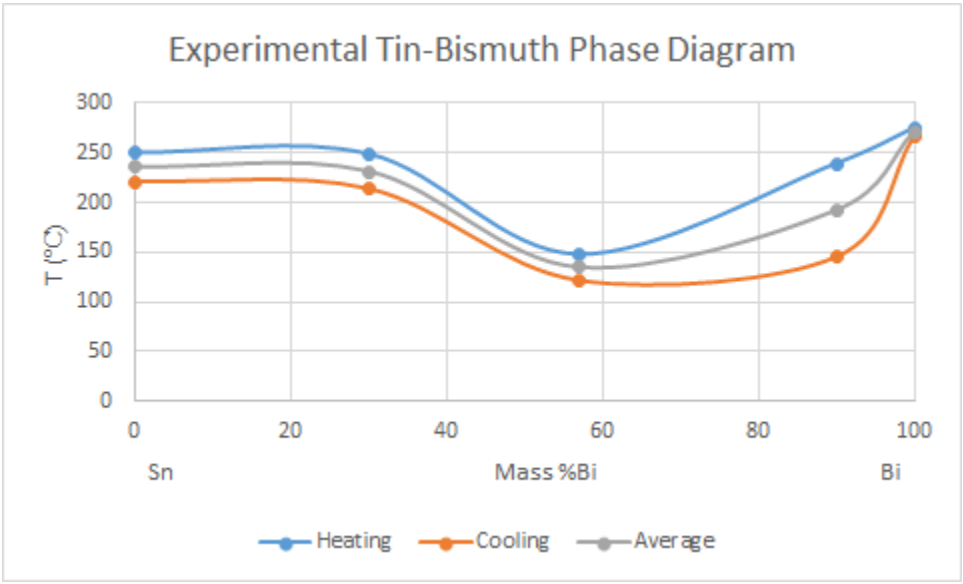

Figure 12. Experimental Phase Diagram created using the phase transition temperatures through DSC.

Using the graphs and data gained from the DSC an experimental phase diagram can be created and compared to existing phase diagrams. The average of the temperature at which the material melted and solidified was used in order to plot the liquidus line of the phase diagram (Figure 12). In a standard tin-

bismuth phase diagram the liquidus line starts at 232℃ for pure tin, drops down to 131℃ at the eutectic point around 57wt% Bi, and then climbs up to 271℃ when it is pure bismuth. While our experimental values aren't exactly equivalent to the points on a proven diagram, ours still comes close and follows the same trends indicating that while not exact, our phase diagram is still accurate.

6. Conclusion

It is concluded that the element in the indexing exercise was nickel by finding that it was a face centered cubic structure with an atomic radius of 1.245 Å. Analysis (Match) confirmed that pure Sn and our Bi data matched up almost identically with the reference data. When overlaid the data for the different mixes of Sn and Bi with the reference of pure Bi and Sn, it is evident that the mix would mostly represent the pure XRD data of the element with the higher composition. The crystalline structure to be close to simple cubic and confirmed Match! data using manual indexing of the XRD data. DSC data seemed to be consistent with the Sn-Bi phase diagram when excluding certain peaks that seemed to just be mistakes from the experiment that could possibly indicate some contamination in the sample. Using the information gathered from the DSC it is recommended that 57wt% Bi would be the best fit for soldering because it has the lowest melting temperature of the compounds making it a good replacement for traditional lead-tin solder.

Acknowledgements

The completion of any disciplinary project depends upon co-ordination, cooperation and combine efforts of serval sources of knowledge. We take this momentous opportunity to express our heartful ineptness and regards to **Trevor S. Harding, Ph.D.**, Chair and Professor, Materials Engineering professor **Sarder Sadique, Ph.D.** in Materials Engineering, California Polytechnic State University for his even willingness to give us valuable advice and direction whenever we needed. We are thankful for him for providing his valuable time and interest towards the project.

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Biography / Biographies

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