

MTE 481
Final Laboratory Project
Phase Identification and Microstructural Characterization of an Unknown Material

Instructional Objectives

Upon completion of this module, the student will be able to accurately identify an unknown material using x-ray diffraction, the ICDD search manuals, ICDD search-match software, light optical microscopy, scanning electron microscopy, and energy dispersive x-ray spectroscopy.

Required Materials

1. Bulk unknown specimen
2. Metallographic mounting compound
3. Metallography supplies (i.e., grinding papers, polishing cloths, polishing compound, etc.).
4. Sample crusher and hammer
5. High vacuum grease
6. Mortar and pestle
7. 325 mesh sieve
8. Spatulas
9. Fused silica slides
10. Files
11. Razor blades
12. Acetone and alcohol

Reading Materials

1. Course notes (available on course web site) and appropriate sections in the required texts.
2. Phase diagrams, ASM Handbooks, etc.

Other Useful Reading Materials

- ASM Handbook on Materials Characterization (available in the MTE Department and at the Rodgers library).

Tasks for Lab Project

Each group will be responsible for full characterization and identification of an unknown specimen. Required tasks include:

Imaging

- Light optical microscopy (LOM)
- Scanning electron microscopy (SEM)
 - secondary electron imaging
 - backscattered electron imaging

Phase Identification and Chemistry Determination

- Scanning electron microscopy (SEM)
 - energy dispersive x-ray spectroscopy (EDS) in the SEM
- X-ray diffraction (XRD) analysis
 - Phase identification
 - Lattice parameter determination
 - Quantification of phase amounts

Sample Preparation

Each group should section a piece of their respective unknowns for metallographic sample preparation. These specimens will be used for both LOM and SEM/EDS analysis. Powder XRD specimens should be prepared in accord with the techniques used in Laboratory Exercises #1 and 2 (i.e., indexing of cubic and non-cubic x-ray diffraction patterns).

Data Collection

Each group should schedule time with the instructor to do their SEM/EDS analysis. SEM/EDS must be done with the instructor. This will allow you to ask him and him to ask you questions. Your LOM results should consist of properly annotated images showing the representative microstructure of your unknown material.

Your SEM/EDS data should include representative micrographs of the overall microstructure and the various microstructural features in your unknown material. You should also collect EDS spectra for the (i) overall composition and (ii) individual phase compositions where possible.

Your XRD results should consist of an XRD pattern. Once you have collected that pattern you are to: (i) identify all XRD peaks within the pattern using the Hanawalt search method; (ii) conduct the same ID using the ICDD Search/Match software (you should get the same results as you did in (i)); (iii) fully index the diffraction peaks using the methods learned in laboratory Exercise #2; and (iv) prepare an individual final written report and a 15 minute group oral report to be presented during the final week of class. All group members will contribute in the oral report.

In your final report you are to include a section on possible safety concerns in materials preparation and data collection.

Comments

Do not copy from any source. This would constitute plagiarism. You can be expelled for it. Everything should be written you your own words. References should be cited in the proper fashion. Any figures taken from books, articles or other published media must be properly referenced.

Additional Required Information

1. Properly identify unknowns using the Hanawalt Search manual and match to the appropriate ICDD cards. Your report must include copies of the appropriate cards. You must also reference the proper pages that your phases are located on in the Hanawalt Search manual.
2. Compare calculated results with reference data on ICDD cards and relevant reference literature (i.e., journal papers, handbooks, phase diagrams, etc.). Explain any differences.

Your final reports **must** include the following:

- Tabulated results
- Photocopies of appropriate page(s) from Hanawalt Search Manual illustrating your match.
- Photocopies or printouts of the proper ICDD card(s).
- Tables comparing experimental and ICDD database results.
- Discussion of possible sources for observed differences between experimental and reference results.
- Proper identification and labeling of all peaks in the experimental XRD pattern(s).

Additional Considerations

As good scientists and engineers you are expected to consider the underlying causes for differences between your tabulated data and the standards provided by the ICDD.

It might be helpful to look at the appropriate phase diagram(s).

Things such as alloy composition and the influence of alloy composition on lattice parameters should be a consideration. Pickup of impurities during processing is also another potential consideration. **In your report, I will expect you to use your common sense and materials engineers to explain any discrepancies.** Feel free to talk about it with classmates. Feel free to consult other professors within the Department. Also feel free to consult alternative reading material (e.g., Physical Metallurgy texts, Materials Handbooks, etc...).