Session 2464

How we learned to love the phase diagram with a Ti-Cr alloy characterization lab

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Abstract

While many students learn how to read and use a phase diagram in introductory materials courses, greater appreciation for such a tool can be garnered through the laboratory setting. A laboratory module for a "Structures of Materials" class (a "core class" for materials majors) has been developed to demonstrate the usefulness of phase diagrams, as well as, to emphasize the connections among processing, structure, and properties. Competence in determining stable phases, phase compositions, and mass fractions of phases are not the end goal, per se, but transpire since the skills are required to help solve a puzzle.

Students are given a set of Ti-Cr alloys (different compositions that have also been processed at different temperatures), however all the samples are unmarked. Given a few clues, the students must then investigate the samples through x-ray diffraction, metallography, and hardness tests to sort out and identify the samples. The lab module is open-ended in approach, and different groups arrive at the same solution in different ways. Several experimental techniques and different concepts (e.g., lattice constants, Vegard's rule, strengthening mechanisms) are brought together in a cohesive manner. Students have found the lab module to be quite challenging, yet in the end, also very satisfying.

Introduction

"Structures of Materials" is the first core class in the Materials Engineering curriculum at Cal Poly, where students delve into much more detail about crystal structures, symmetry, defects, and microstructures. These same topics are introduced in an earlier "Introduction to Materials Engineering" course that also serves as a survey course to all other engineers. Students often have varying degrees of understanding and appreciation for phase diagrams. While sometimes students can work out problems dealing with phase diagrams (e.g., mass fractions), they do not always fully understanding the concepts. This particular laboratory enables students to appreciate the utility of phase diagrams by posing questions within a context that ties together processing, structure and properties of alloys. Students typically demonstrate frustration at the beginning of the lab, but then consistently rate this lab as the most valuable learning experience of the course on surveys.

The lab is run for 2-3 weeks at the end of the term, and is presented as a puzzle where several different pieces need to be solved. Lectures can supplement the lab activities, or the concepts are reviewed during the lab. Students are given 5 different Ti-Cr alloys (3 alloy compositions processed at 2 different annealing temperatures), yet all the samples are unmarked. Given a few clues, the students must then investigate the samples through x-ray diffraction, metallography, and hardness tests to sort out and identify the samples. The students have already been introduced to all the experimental techniques.

The class is broken up into 2 groups, and smaller teams can then be formed, if desired. One group performs x-ray diffraction while the other group does the metallography and hardness measurements. The following week, the groups switch tasks and then use all the information to solve the puzzle under guidance of the instructor.

The learning objectives are as follows:

- contrast an *alloy* vs. an *intermetallic*
- perform XRD on uncharacterized samples
- identify phases and crystal structures from XRD
- distinguish between FCC and BCC structures with XRD
- distinguish between single and two-phase alloys with XRD
- compute lattice constants
- use Binary Alloy Phase Diagrams reference book and the Periodic Table
- explain features of phase diagrams: phase stability, tie-lines/isotherms, lever-rule
- determine alloy compositions of solid solutions from lattice constants
- determine placement of alloy on the composition-temperature (phase) diagram
- characterize microstructures from optical (light) microscopy
- determine volume % of multi-phase alloys (by image analysis and other techniques)
- calculate expected volume fractions of phases given an overall alloy composition
- apply ASTM standards to conduct hardness measurements
- explain solid solution strengthening
- classify phases based on hardness values
- relate ***microstructural*** features to mechanical ***properties***
- communicate findings in a lab report

Procedures

The Ti-Cr equilibrium phase diagram (Figure 1) is given to the students, and a few items are discussed before the students are charged with a problem to solve. The following in italics is part of the actual lab handout:

Congratulations, you've just gotten a job at the prestigious Acme Materials, Inc.! Since you are a recent graduate in Materials Science & Engineering, you are able to start doing work and contribute to the company right away. However, you have a peculiar challenge in front of you. A previous employee has suddenly left the company after hitting the lottery jackpot, and you have just inherited all of her samples. Unfortunately, most of the paperwork is missing, and the samples are unlabeled. (This actually happens quite frequently in real life; not winning the lotto, but having unlabeled or mismarked samples.) The pieces of information you can fully trust are:

__3__ different alloy compositions in the Ti–Cr system were cast from the foundry
__2__ different heat treatments (anneal & quench) were performed on the alloys

It is your job to figure out what the ***alloy compositions*** *are and what heat treatments were used (i.e.,* ***annealing temperatures****). In addition, you are to relate how the* ***structure*** *of these materials will affect the* ***mechanical properties****. (The annealing treatments were immediately water quenched to lock-in the phases at the high temperatures.) Luckily, you have 5 different x-ray diffraction (XRD) samples and 5 mounted metallographic samples at your disposal. (Each of these 5 samples is some combination of a given alloy composition and annealing temperature.) You immediately go to the library to get a copy of the Ti–Cr equilibrium phase diagram and associated information. You are to submit a report to your boss on the characterization and properties of the samples in two weeks.*

Metallography

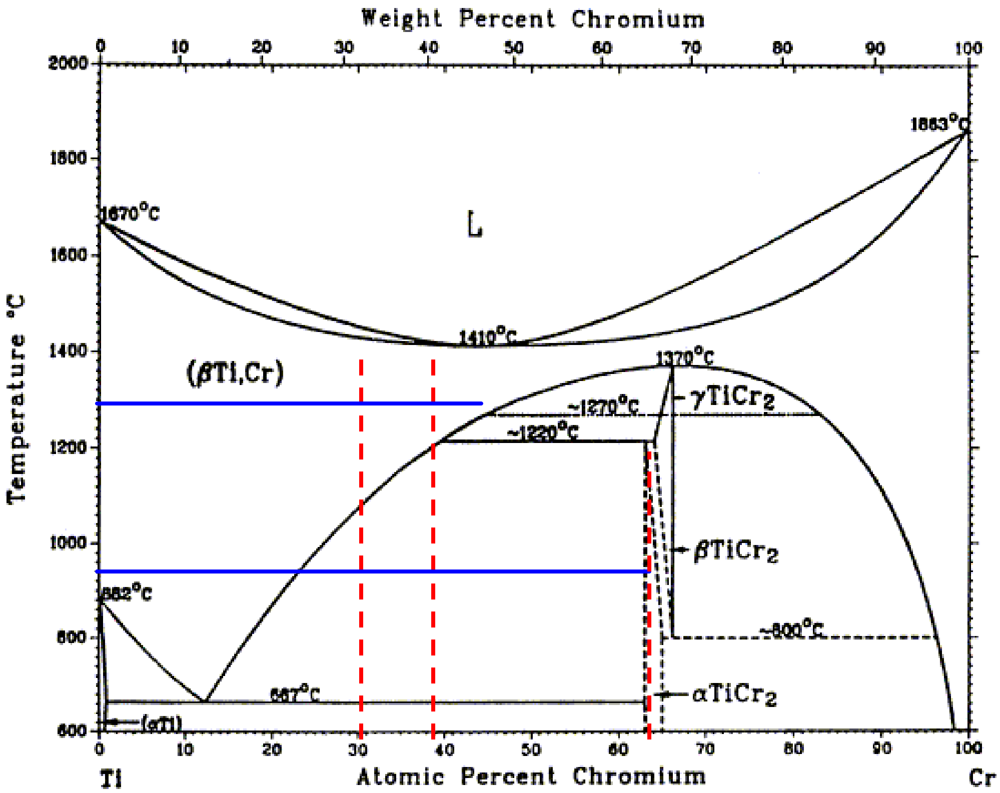
Metallographic samples are mounted, polished, and etched ahead of time, and at first glance, all the samples look similar. The students use optical, light microscopes to characterize the microstructures, and begin to note differences among the samples. A table of pertinent information to collect (Table I) is provided for the students. Explanations of each item are usually required of the instructor or teaching assistant as the students work. The "just in time" help during the lab has proved to be more effective than lecturing before the lab activities. The lab is designed to be more "inquiry-based" and the students must figure out or discover things for themselves, rather than follow cookbook type instructions.

The students determine which samples are single-phase, and which samples are two-phase alloys. Sketches and observations (such as grain size, precipitate morphology, etc.) are recorded in lab notebooks. For the two-phase samples, the volume fraction of the precipitates is determined (using point counting and/or image analysis). Depending on how much of the samples has already been characterized, actual identification of the puzzle pieces occurs at different points in time. Each group or team may arrive at the solutions in different ways, but they all eventually get to the same conclusions.

The same mounted samples are also used for microhardness tests. A copy of ASTM E-384: "Standard Test Method for Microhardness of Materials," is purposely placed by the instrument. The samples get further distinguished by their mechanical properties. The students are asked to discuss what microstructural features would affect the mechanical properties (i.e., hardness) and how they might present their collective data (Figure 2).

Table I. Results from metallographic experiments.

Sample #	Single or Two-phase	Volume %	Hardness (HV)	Phases
1	*single-phase*	100	236	β-(Ti,Cr)
2	*single-phase*	100	250	β-(Ti,Cr)
3	*two-phase*	25/75	387	β-(Ti,Cr) + $TiCr_2$
4	*two-phase*	40/60	458	β-(Ti,Cr) + $TiCr_2$
5	*single-phase*	100	872	$TiCr_2$



Special Points of the Ti-Cr System

Reaction		Composition, at.% Cr		Temperature, °C	Reaction type
L ↔ $(\beta Ti,Cr)$		44		1410 ± 5	Congruent
(βTi) ↔ $\gamma TiCr_2$		~66		1370 ± 10	Congruent
(βTi) ↔ (αTi) + $\alpha TiCr_2$	12.5 ± 0.5	0.6	~63	667 ± 10	Eutectoid
(βTi) + $\beta TiCr_2$ ↔ $\alpha TiCr_2$	39	~63	~65	~1220	Peritectoid
$\gamma TiCr_2$ ↔ $\beta TiCr_2$		~65 to 66		~1270	Unknown
$\beta TiCr_2$ ↔ (Cr) + $\alpha TiCr_2$	~65	96	~66	~800	Eutectoid
L ↔ βTi		0		1670	Melting
βTi ↔ αTi		0		882	Allotropic
L ↔ Cr		100		1863 ± 20	Melting

Ti-Cr Crystal Structure Data

Phase	Composition, at.% Cr	Pearson symbol	Space group	Strukturbericht designation	Prototype
$(\beta Ti,Cr)$	0 to 100	*cI2*	$Im\bar{3}m$	A2	W
(αTi)	0 to 0.2	*hP2*	$P6_3/mmc$	A3	Mg
$\alpha TiCr_2$	63 to 65	*cF24*	$Fd\bar{3}m$	C15	Cu_2Mg
$\beta TiCr_2$	64 to 66	*hP12*	$P6_3/mmc$	C14	$MgZn_2$
$\gamma TiCr_2$	64 to 66	*hP24*	$P6_3/mmc$	C36	$MgNi_2$
Metastable phase					
ω	...	*hP3*	$P\bar{3}m1$	...	ωCrTi

Figure 1. The equilibrium Ni-Ti phase diagram[1].

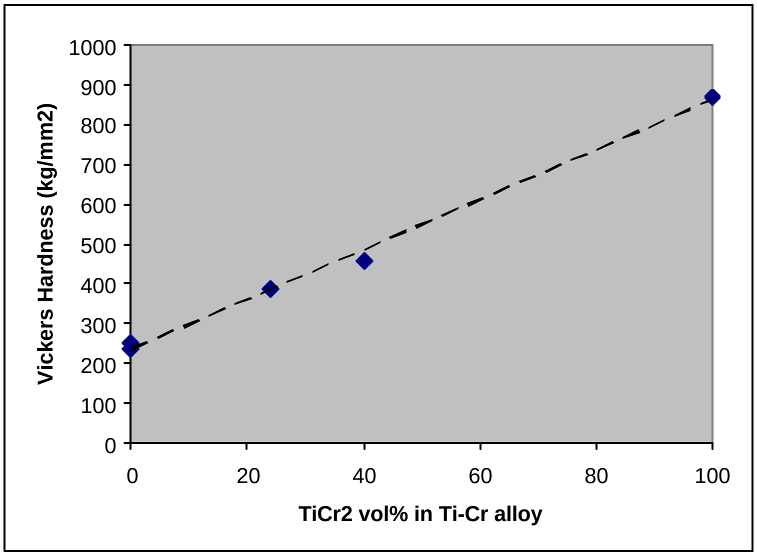


Figure 2. Vickers hardness versus the volume percentage of the $TiCr_2$ intermetallic phase in the alloy. The strength and hardness are a function of the type and amount of phases present.

Data is shared among the group members, and they brainstorm together to try to solve the puzzle. If the metallography portion of the lab is done first, the actual alloy compositions and annealing temperatures cannot be specified yet.

X-Ray Diffraction (XRD)

Students are given 5 unlabeled samples and must come up with a labeling and testing scheme to identify the different alloys. While all the samples appear similar, one sample contains some macroscopic cracks. The students are told to make note of the observation, which offers an additional, subtle clue (i.e., single phase intermetallics are very brittle!).

While the students perform XRD, discussion occurs about the possible phases in the Ti-Cr system[1]. Review of the space group notation and characteristic XRD patterns for the BCC and FCC lattices become more important and relevant to the students in this setting versus the classroom.

A number of issues appear during this portion of the lab, and questions are often prompted by the students. The superposition of XRD peaks for two phase systems and the lack of powder diffraction file (pdf) scans for alloys require the students to think on their own. They are sometimes dismayed that the answer cannot come from a quick “Search and Match” feature of the software! Comparisons among the XRD scans are required to note what is the same and what is different.

Again, a table (Table II) and lots of instructor guidance are provided for the students. Usually pairs of students are responsible for determining the lattice constant for one of the phases, and the data for all the phases are shared among the group.

Table II. Results from x-ray diffraction (XRD) experiments.

Sample Name	Phases	Crystal Structure	Lattice Constant	Phase Composition	Alloy Composition
A	β-(Ti,Cr)	BCC	3.183 Å	Ti-30 at% Cr	Ti-30 at% Cr
B	β-(Ti,Cr)	BCC	3.140 Å	Ti-40 at% Cr	Ti-40 at% Cr
C	β-(Ti,Cr) + $TiCr_2$	BCC + FCC(C15)	3.212 Å 6.942 Å	Ti-23 at% Cr Ti-66 at% Cr	Ti-30 at% Cr
D	β-(Ti,Cr) + $TiCr_2$	BCC + FCC(C15)	3.212 Å 6.942 Å	Ti-23 at% Cr Ti-66 at% Cr	Ti-40 at% Cr
E	$TiCr_2$	BCC	6.942 Å	Ti-66 at% Cr	Ti-66 at% Cr

The students discuss how lattice constant and alloy composition might be related, and eventually come up with a method to relate the two. Essentially, Vegard's Rule is followed (Figure 3) and more pieces of the puzzle are revealed as the data table entries (Table II) gets completed. The lattice constants[2] for pure, BCC Ti and Cr are used, and the BCC β-phase composition can be computed as follows:

$$\text{at\% Cr (in BCC-}\beta\text{, Ti-Cr alloy)} = \frac{3.3066 - a_b}{3.3066 - 2.8847}$$

Ultimately, the placement (i.e., composition and temperature) of the 5 samples on the phase diagram is identified (Figure 1). During the course of the activity, tie-lines and the lever rule are invoked and many students finally see how the phase diagram is used and why! The remaining piece of the puzzle is to match up the samples from the metallography portion of the lab with those from the XRD part (Table III). The structure-property connection is made, and the students are usually thrilled to see how all the pieces fit together.

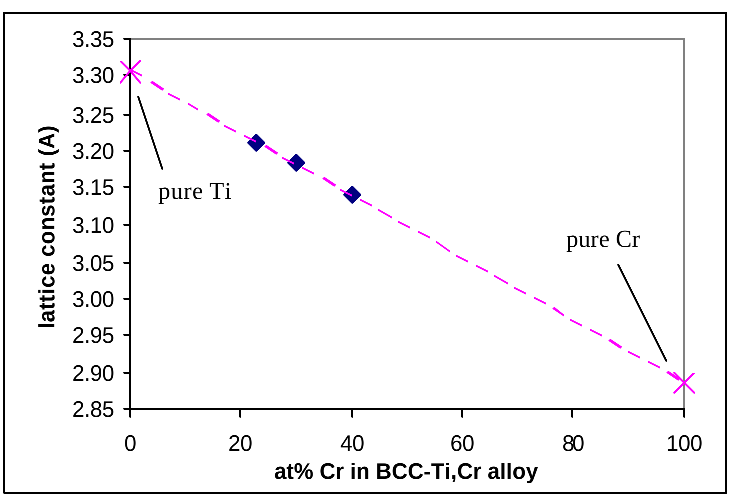


Figure 3. The β-BCC lattice constant versus Ti-Cr alloy composition. The dotted line represents Vegard's Rule and is based on the lattice constants for pure Ti and Cr[2]. Extrapolations of the experimental lattice constants to the Ti-Cr alloy composition are quite accurate.

Table III. Alloy composition and annealing temperature of the five samples.

Metallography Sample	XRD Sample	Alloy Composition	Annealing Temperature
1	A	Ti-30 at% Cr	1300ºC
2	B	Ti-40 at% Cr	1300ºC
3	C	Ti-30 at% Cr	950ºC
4	D	Ti-40 at% Cr	950ºC
5	E	Ti-66 at% Cr	950ºC

Conclusions

Although this particular laboratory requires lots of attention and guidance from the instructor and teaching assistant, the learning by the students is tremendous. One of the biggest confusions of students that gets resolved is the difference between the compositions of phases within a two-phase system (using tie lines) and the overall alloy composition.

Students respond that they finally understand the finer details of alloys and phase diagrams. They also enjoy that they worked on "real" materials and got "real" data. Although many students felt overwhelmed and struggled at first, they felt great satisfaction once all the pieces came together and that they ultimately succeeded.

Bibliography

1. *Binary Alloy Phase Diagrams*, **10**, 219 (1989).
2. B.D. Cullity, Elements of X-Ray Diffraction, 2nd edition, Addison-Wesley, 1978, p. 506-507.

Biography

KATHERINE C. CHEN is an Associate Professor in the Materials Engineering Department at Cal Poly State University, San Luis Obispo, CA. She received her bachelor degrees (in Chemistry and Materials Science & Engineering) from Michigan State University, and Ph.D. from the Massachusetts Institute of Technology. At Cal Poly, she teaches undergraduate students Structures of Materials, Kinetics of Materials, and various other courses.