Lab 5

Qualitative Phase Analysis

This experiment concerns the use of x-rays for phase identifications. The first procedure is demonstrated with a single-phase. Then you will be required to do a similar analysis for a more complex (multiphase) sample.

5.1. Qualitative Chemical Analysis

X-ray diffraction can be used to carry out a simple qualitative chemical analysis. The approach is based on the fact that a given substance always for a particular substance is accomplished by the identification of the pattern of that substance.

Ad advantage of diffraction analysis is that it discloses the presence of a substance in the form that it actually exist in the sample. The substance is identified in terms of crystal structure. This is different as compared to usual chemical analysis, which normally gives elemental analysis.

The advantage of this analysis can be illustrated as follows. An alloy such as Fe-15%Ni can be either martensitic (BCC) or austenitic (FCC) depending on the heat or mechanical treatment applied. The usual chemical analysis would give the same elemental analysis, i.e. Fe 85% and Ni:15% regardless of whether the alloy is austenitic or martensitic. With x-ray diffraction, however, the alloy is identified with its crystal structure and hence this information about the state of the substance can easily be obtained.

Chemical analysis with x-ray diffraction requires a collection of diffraction data of all substances present in nature. Then the approach is to compare the data of a given sample with that collection until a matching pattern is found.

5.2. Procedure

Measurement for chemical analysis starts with obtaining the diffractograms of a substance. Then diffractogram is evaluated by calculating the d-spacing corresponding to each peak together with their integrated intensities. The integrated intensity values are normalized and expressed in terms of I/I_1 are tabulated, the unknown substance can be identified by the following procedure

1. Select three strongest lines from the diffractogram, i.e., d_1, d_2, d_3
2. Use the Hanawalt Numerical Search Method and find the appropriate section of this manual containing d_1. This manual contains three column. d_1 appears in the first and d_2 appears in the second column(with decreasing order). Search the second column of d-values to find the closet match with d_2.
3. After d_2 is matched, repeat the same for d_3.
4. Compare the experimental intensities with those tabulated in the manual.
5. If they agree, look at the card number of the matching substance

5.3.Experiments

The experiment consists of two parts:

1. Part I: Single Phase
   In the first part, intensities of diffected beam of a single-phase sample will be evaluated. The aim is to illustrate the producedure of chemical analysis with x-ray differection. For the purpose, the diffractogramme of an unknown substance will be given in the lab session.
   -follow the steps (given in the previous page) from 1 to 6, and
   -verify that you can correctly identify the sample.

2. Part II: Multi-phase (mixture of elemental powders X_A and X_B)

   In this part, the diffractogramme of a mixtrue of elemental powders X_A and X_B (contains a mixture of phases) will be given. The approach is similar to that given in the previous page. Therefore, d_1, d_2 and d_3 can be selected ,n the same way but one cannot be sure that all these three lines belong the same phase. Therefore,

   -select two "d" values out of three and use numerical search manual to find possible candidates for one of the phases, then

   -use minor peaks to find the actual phase present in the sample.

   -if no results are obtained, select the other two "d" values and repeat the sample steps.

   -when one of the phases is identified, disregard the "d" values corresponding to the phase and repeat the usual procedure in order to find the remaining phase(s).

Classwork: Qualitative Phase Analysis

$\lambda_{Co\,(Ka)}$=1,79026 Å $\lambda=2d \sin\theta$ $d=\lambda/2 \sin\theta$

PART I:

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2θ	d(Å)	Integrated Intensity	Normalized Intensity	Phase	(hkl)
43.2		2.6			
50.3		30.125			
73.8		14.3			
89.5		2.08			
94.7		6.05			

Self Study: Qualitative Phase Analysis

$\lambda_{Co\ (Ka)}$=1,79026 Å $\lambda=2d\sin\theta$ $d=\lambda/2\sin\theta$

2θ	**d(Å)**	**Integrated Intensity**	**Normalized Intensity**	**Phase**	**(hkl)**
29,8		2,745			
41,1		4,095			
44,2		1,710			
45,0		10,575			
50,8		4,455			
52,5		4,770			
61,9		2,025			
68,0		4,000			
70,7		0,160			
72,5		0,600			
77,4		2,925			
79,2		1,750			
81,4		2,350			
92,7		1,445			
94,4		3,750			
97,4		0,550			
100		1,365			
102,5		0,390			
105,4		0,595			
109,0		0,585			
112,2		1,080			