Course 12.141: Electron Microprobe Analysis – Problem Set 3

Problem 3.1: Quantitative analysis of Fe and Ni in Fe-Ni alloys through wavelength dispersive spectrometry (WDS). For FeKα ($\lambda_{K\alpha}$=1.937Å) and NiKα ($\lambda_{K\alpha}$=1.658Å), use Bragg's law to decide which diffracting crystal (TAP: 2d=25.757Å; PET: 2d=8.742Å; LIF 2d=4.027Å) would be the best to use to measure the X-ray intensities. The optimal range of 2θ for each crystal is between 30^o and 130^o and that the best peak shape is achieved near the middle of the 2θ range of the spectrometer.

Perform WDS analysis to determine the Fe and Ni contents of the five Fe-Ni alloy polished samples provided. Analyze five points in each sample using the Kα X-rays of Fe and Ni, an accelerating voltage of 15 kV and a beam current of 10 nA. The nominal composition of the samples are given below:

Sample	Fe (wt%)	Ni(wt%)
FN-Ol-6	98.12	1.82
FN-Ol-11	88.07	11.01
FN-Ol-12	51.95	48.06
FN-Ol-8	25.1	73.95
MA8.50-003	14.38	87.01

Problem 3.2: In the Ti-V system, the VKα peak overlaps the TiKβ peak. Analyze the steel sample for Ti and V using their Kα X-rays, an accelerating voltage of 15 kV, a beam current of 10 nA and applying peak overlap corrections. The L-values of characteristic X-rays for Ti and V are given below:

	Analyzing Crystal	
X-ray	**PET**	**LIF**
TiKα	88.03	191.12
TiKβ	80.52	174.81
VKα	80.19	174.09
VKβ	73.17	158.85

The L-value differences (peak separations) between VKα and TiKβ are 0.33 with PET and 0.72 with LIF. Since the difference is larger with LIF, it should be the preferred crystal for analyzing Ti-V compounds. In this exercise, use both crystals for the measurements. The sample contains minor amounts of Ti and V. Set the counting times such that 5-10% standard deviation of counts is achieved. Compare and explain the results obtained with the PET and the LIF crystals.

N. Chatterjee: January 2012, MIT, Cambridge, MA, USA

12.141 Electron Microprobe Analysis

January (IAP) 2012