

X-Ray Diffraction Lab

Diffractometer/PE Degree of Crystallinity/Phase Analysis

[Polyethylene Trace](#)

[Close to Amorphous PE Trace](#)

[Metallocene Low Crystallinity](#)

Objective: To become familiar with use of the diffractometer for quantitative analysis of phases in semi-crystalline polymers or phase mixtures in metals (students choose on of these two parallel areas).

Background: Chapter 14 (Phase Analysis), Chapter 7 (Diffractometer), pp. 139 section 4-12 and equation 4-21 for Diffractometer.

The integrated intensity from a diffraction peak is proportional to the volume fraction of that material in a sample. This means that for a material with multiple phases, crystalline/amorphous or several crystalline phases, XRD can be used to determine the volume fraction of these phases. For a semicrystalline polymer such as polyethylene this results in a determination of the degree of crystallinity of the sample which is a critical measurement for determination of the properties of the polymer. For a multi-phase metal this corresponds to a quantitative analysis of the phase composition. For example, this is a common technique for determination of martensite/austenite content in steel.

There are alternative approaches to determination of phase composition including density measurements, thermal analysis and spectroscopy, but XRD remains the most robust and simplest technique so it is of dominant importance.

The intensity from a diffractometer in reflection geometry must be corrected for the angular dependence of absorption/beam profile and for the Lorentz Polarization factor which is also angularly dependent. In addition, account must be made for the multiplicity of planes in a crystal, variation in thermal broadening with angle and some other factors, see equation 14-1 in pp. 408.

Phase Composition for Steel:

As Cullity described on pp. 408 to 409, equation 14-1 can be simplified for comparison of two reflections which correspond to two different phases in a metal sample, such as martensite and austenite in steel. Equation 14-11 on pp. 411 is used for such a case:

$$I_{\alpha} = \frac{K R_{\alpha}^2 c_{\alpha}}{2 \mu_m}$$

K is a constant which will cancel in intensity ratios, c_{α} is the concentration of the alpha component, R_{α} is an angular dependent term which is defined in equation 14-10 on pp. 411, μ_m is the absorption coefficient for the mixture. For two peaks, α and β , from two phases the concentration of the two phases can be determined by ratios of equation 14-11,

$$\frac{I_{\alpha}}{I_{\beta}} = \frac{R_{\alpha}^2 c_{\alpha}}{R_{\beta}^2 c_{\beta}} \text{ and } c_{\alpha} + c_{\beta} = 1$$

For steel containing austenite and martensite the diffraction patterns are indexed in Figure 14-3 on pp. 413. The 110 peak of martensite can be compared with the 111 peak of austenite to determine the relative contributions of the two components.

