Australian Synchrotron

X-ray Absorption Spectroscopy Beamline 12ID

Transmission Poster No1: A Perfect Sample

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➢What is "A Perfect Sample"?

Many considerations need to be taken into account in order to determine whether a sample is perfect (or suited) for a transmission experiment. Such considerations are discussed on our webpage [1]. In this poster series (see also "Transmission Poster No2: An Imperfect Sample") we will highlight the importance of sample "homogeneity" and its effect upon data quality. By homogeneity we refer to how evenly the element of interest is distributed throughout the volume of the sample. Examples of homogeneous samples are shown in Figure 1, and in Figure 2 a transmission flux map is shown of a typical "perfect sample", homogeneous to within ~ 2 %.

➢Why is homogeneity important?

In a transmission experiment, as in Figure 3, the ratio of the transmitted and incident photons is used to calculate an EXAFS spectrum. We measure the bulk properties of the sample and "thickness effects" become important. Thickness effects refer to leakage of incident photons through pinholes or areas with less sample than others. This is likely to be the main source for noise in our transmission spectra. Additionally, and depending on the beamline configuration, if the beam moves (it does by a few μm) or if the sample moves while measuring (e.g. caused by vibrations in the cryostat), you effectively irradiate different parts of the sample throughout a measurement. If that sample is not homogeneous this can be detrimental to data quality.

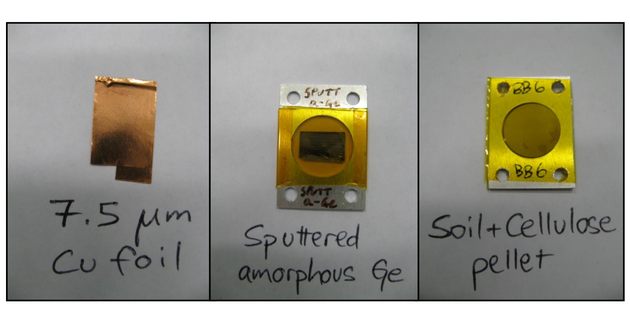


Figure 1

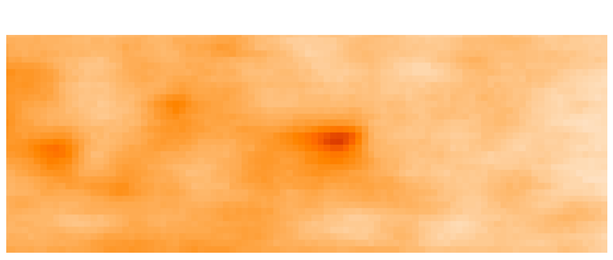
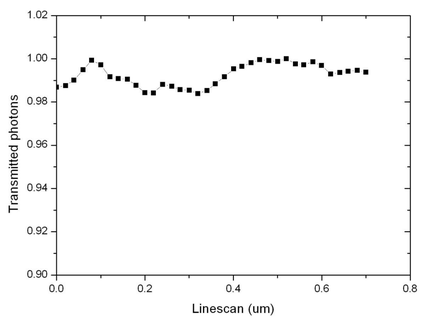


Figure 2



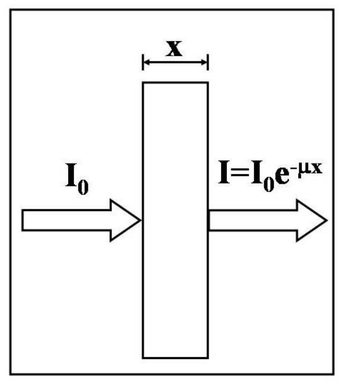


Figure 3

➢What is the nature of the statistics our beamline can deliver?

As an example we measured a standard foil, ideally suited for transmission experiments, from the set of references commonly used for energy calibrations (EXAFS Materials Inc, USA). This sample is perfectly homogenous. We measured it at room temperature and in the cryostat as a function of temperature. Figure 4 shows 12 repeat scans at room temperature to 24 $Å^{-1}$. Any beam movement and/or sample vibration is not reflected in the data quality given the homogenous nature of the sample. Figure 5 shows a neat temperature dependent study for this sample. This is the type of statistics our beamline can deliver when supplied with a perfect sample.

Please note that not all foils are perfectly homogenous and not all homogeneous samples need to be foils, etc (Figure 1). It really comes down sample preparation (grinding, mixing, evaporation, etc), trial, error and hopefully, in the end, success.

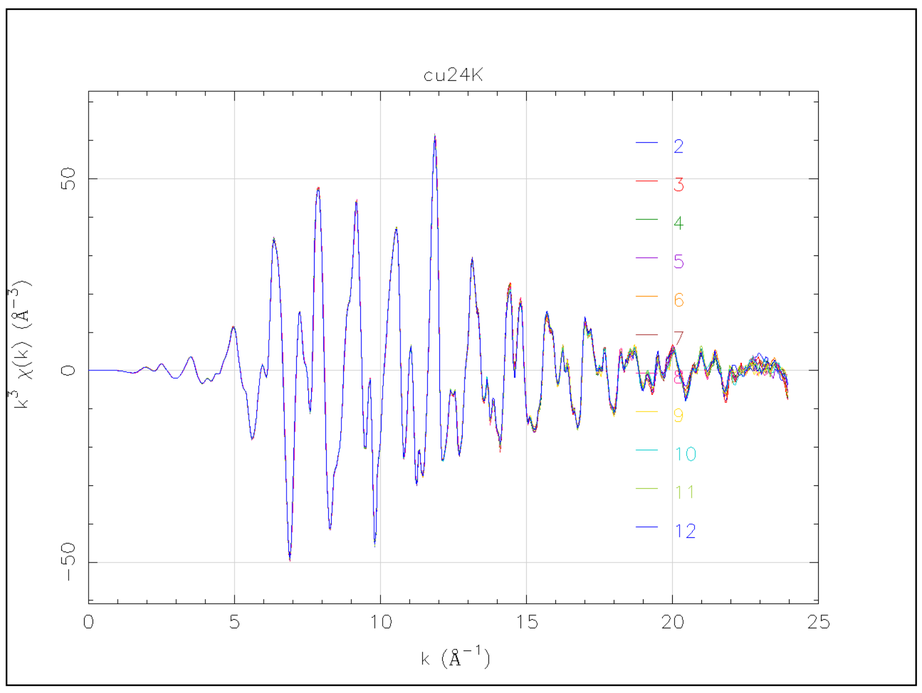


Figure 4

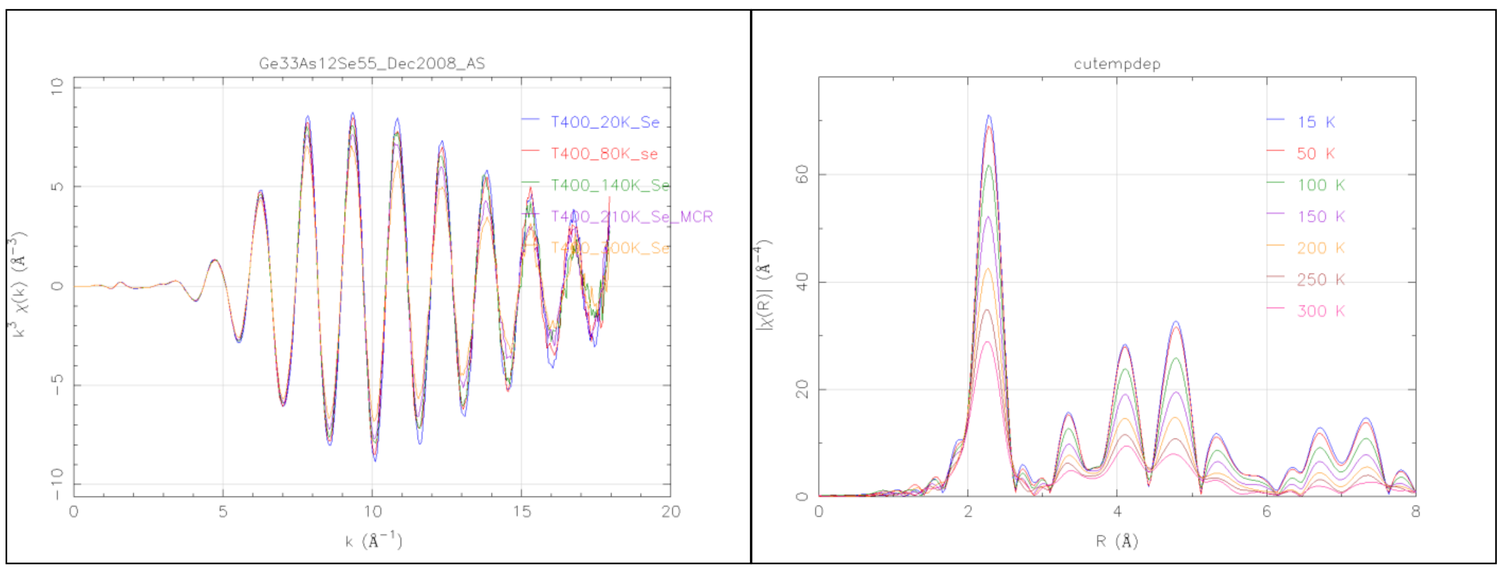


Figure 5