# POLYCAPILLARY OPTICS BASED NEUTRON FOCUSING FOR SMALL SAMPLE NEUTRON CRYSTALLOGRAPHY

**W.M. Gibson[a], H.H. Chen-Mayer[b], D.F.R. Mildner[b], H.J. Prask[b], A.J. Schultz[c], R. Youngman[a], T. Gnäupel-Herold[b], M.E. Miller[c], and R. Vitt[c]**

*a) X-Ray Optical Systems, Inc., Albany, NY, b) National Institute of Standards and Technology, Gaithersburg, MD, c) Argonne National Laboratory, Argonne, IL*

**ABSTRACT**

This work presents preliminary measurements designed to explore a new approach to neutron diffraction that is somewhat analogous to the pseudo-Laue technique, except that instead of using a broad energy (wavelength) bandwidth it uses a broad angular bandwidth. We have used a polycapillary focusing optic to focus neutrons from a monochromatic beam (using the BT-8 spectrometer on the NIST research reactor) and from a polychromatic beam at a pulsed spallation source (the Intense Pulsed Neutron Source, IPNS at Argonne National Laboratory) into a small, intense spot and have carried out preliminary diffraction measurements. Using the single crystal diffraction (SCD) facility on IPNS, diffraction of a $3^o$ convergent beam from an alpha quartz crystal showed six diffraction beams in the 1-5 Å wavelength bandwidth transmitted by the optic. The diffraction spots showed an intensity gain of 5.8±0.9 compared to a direct beam diffracting from the same sample volume as that illuminated by the convergent beam.

## 1. INTRODUCTION

Rapid, accurate, high-resolution structural analysis of proteins is central to the success of rational drug design and other medical and scientific applications. Neutron diffraction has powerful and unique potential for determination of the structure and dynamics of proteins including direct determination of hydrogen positions, solvent association, etc. (Timmins, 1995). However, the low intensity, broad energy (wavelength) bandwidth and large angular spread of neutron beams and the limited availability of neutron sources have kept this potential from being realized. A particularly serious limitation arises from the small size of the crystals available for medically important proteins.

Other cases that involve the use of small crystals or small sampling areas include strain or phase distributions in metals (for example, at welds), and measurement of the structure and phase changes of materials at high pressure or low temperature. Also, mapping of composition in lateral directions by prompt neutron activation analysis requires small size, high intensity neutron beams.

Another important feature of neutron diffraction arises from the lower energy of neutrons compared to that of X-rays of the same wavelength. For example a 0.034 eV neutron and an 8.03 keV X-ray both have a wavelength of 1.54 Å. This leads to reduced radiation damage during neutron irradiation and therefore longer crystal lifetime during diffraction measurements which, among other things, reduces the need to cool the crystal during the measurement. Also the penetration (or scattering mean-free-path) of neutrons is much greater than for x rays and the lower attenuation reduces anisotropic absorption effects for plate-like or rod-like crystals that often leads to errors in x-ray analysis which are sometimes difficult to eliminate. In addition, because neutrons scatter from the nuclei, the scattering power for neutrons does not fall off dramatically with scattering angle as does X-rays. This means that higher resolution data should be easier to obtain with neutron diffraction.

The "pseudo-Laue" method has been used to permit a wide wavelength bandwidth to be used with either a reactor-based source (Wilkinson, 1991) or a pulsed spallation source (Schoenborn, 1992a). In addition to providing a larger usable neutron intensity, this technique samples a larger volume of reciprocal space on each exposure, therefore, requiring fewer exposures to cover the angular space necessary for a structure determination.