Analysis of Different Plastics for use as a Sample Container for X-Ray Diffraction Data Collection

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Introduction

Mounting samples for crystal quality screening and data collection can often be a cumbersome job for crystallographers. The most common method for mounting samples is to transfer a crystal that was grown inside a hanging or sitting drop of mother liquor to a mount comprised of a nylon loop or kapton surface that is attached to a metal sample pin. This sample is then cryo-cooled in liquid nitrogen or a cold nitrogen stream.

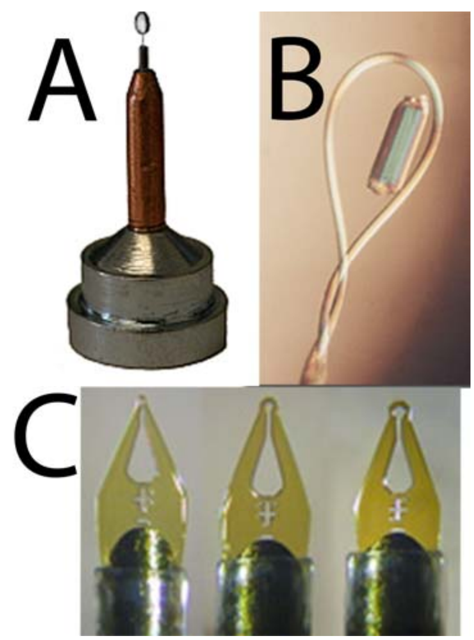

Figure 1. Two common mounts used for affixing crystals on sample pins (A) are nylon loops (B) or kapton micro mounts (C).

This process requires manual manipulation of the crystal to place it properly inside the mount. This is a time consuming process prone to human error. Crystals are commonly damaged both by physical contact and exposure to air. In collaboration with researchers at the Hautpman

Woodward Medical Research Institute, a small plastic capillary has been developed at SSRL which can be used both for crystal growth and x-ray diffraction quality screening. The capillary may be used as a pipette tip with both manual pipettors and automated liquid handling robots enabling solutions for crystal growth to be easily drawn inside.

Figure 2. Image of a plastic capillary mounted a magnetic pin.

The plastic capillary is attached to a magnetic pin that is compatible with most sample goniometers at synchrotron beam lines (shown in figure 2). The magnetic pin is also compatible with the uni-puck and cassette sample containers for robotic sample mounting at the synchrotron beamline. A schematic of the capillary inside a uni-puck is shown in figure 3. This system is very useful for crystal quality screening at room temperature and at cryogenic temperatures for samples grown in cryo-conditions.

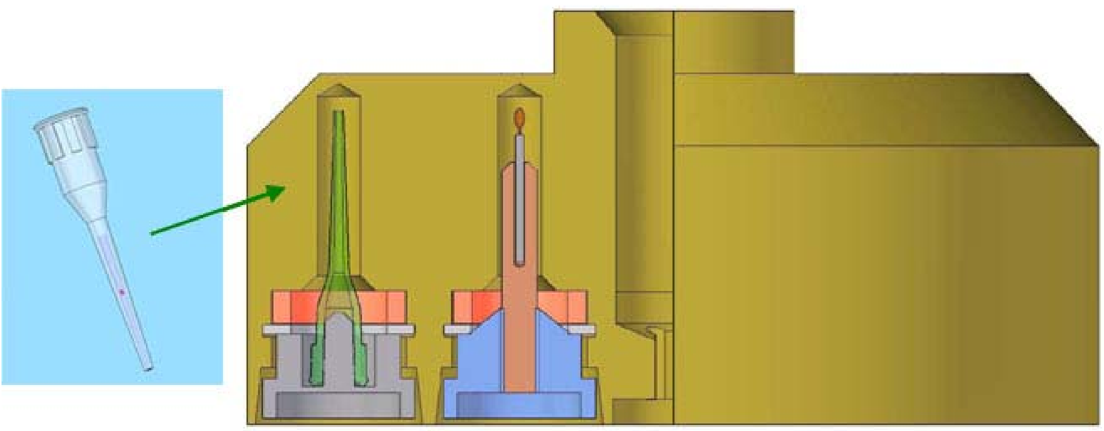

Figure 3. The capillary mount is compatible with the uni-puck storage container used with robotic sample storage systems at many synchrotrons. A comparison between the capillary mount and a loop mount on a sample pin is shown in a cutway diagram of the uni-puck storage container.

The objective of this project was to test several plastics to determine which produce the smallest x-ray background and are best for optical visualization of the sample during data collection.

Experimental

Clear grades of six polymers were injection molded into a plastic capillary. These polymers were:

- Acrylonitrile butadiene styrene (ABS)
- Polymethyl methacrylate (Acrylic)
- Polyamide (PA)
- High Impact Polystyrene (HIP)
- Polycarbonate (PC)
- Low Density Polyethylene (LPDE)

Data was collected on SSRL BL7-1. Each sample was exposed to 0.976 Å x-rays for about 30 seconds at a temperature of 100 K. The incident intensity was monitored and the exposure time varied to ensure each sample received an equivalent x-ray dose. Care was taken to expose the same volume of plastic from each sample tested. Each plastic was rotated five degrees during the exposure. The scattered intensity was measured by a Q315 detector located at a distance of 251 mm from the sample giving a maximum resolution of 1.37 Å. A diffraction pattern was measured from silicon standard placed at the sample position. In order to ensure that a given plastic had an identical diffraction independent of sample orientation, ABS and Acrylic were tested at two different angles 90 degrees apart.

The data was analyzed using FIT2D. The sample to detector distance, wavelength, beam center, and angle and orientation of detector tilt were refined and calibrated based the silicon standard data. For each plastic diffraction image, the average pixel value for each 2θ range was calculated and the data was then extracted to a text file. The same analysis was done for an air scatter image, collected with no sample at the same x-ray dose. The data was input into Microsoft Excel for graphing. The average overall scattered intensity on the plate was determined excluding the beamstop shadow and excluding the average air scatter intensity for each 2θ range.

The optical clarity under room temperature and 100 K was visually observed for each plastic. An approximately 100 micron diameter dark brown hair was placed inside each capillary and viewed with the beam line camera. Video images of the capillaries were saved at each temperature. Images of each plastic were rated relative to one another based on how 1) the clarity of the capillary and 2) how undistorted the hair looked inside each capillary. Four people rated the images independently for these two parameters.

Results

X-Ray Diffraction

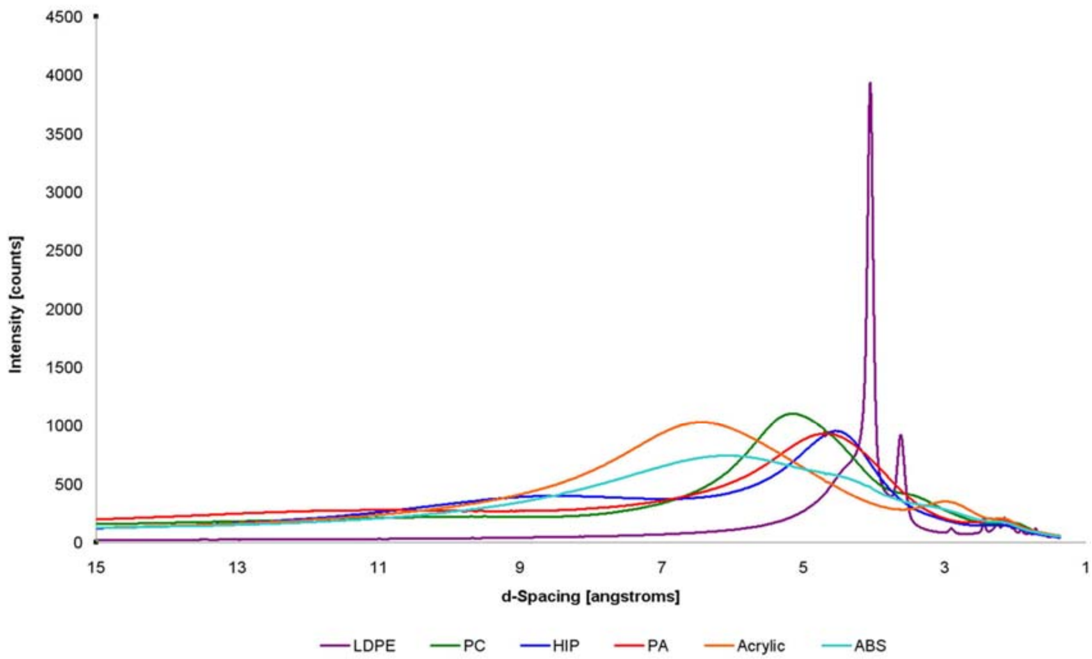

Figure 4. Average intensity versus d-spacing for various plastics.

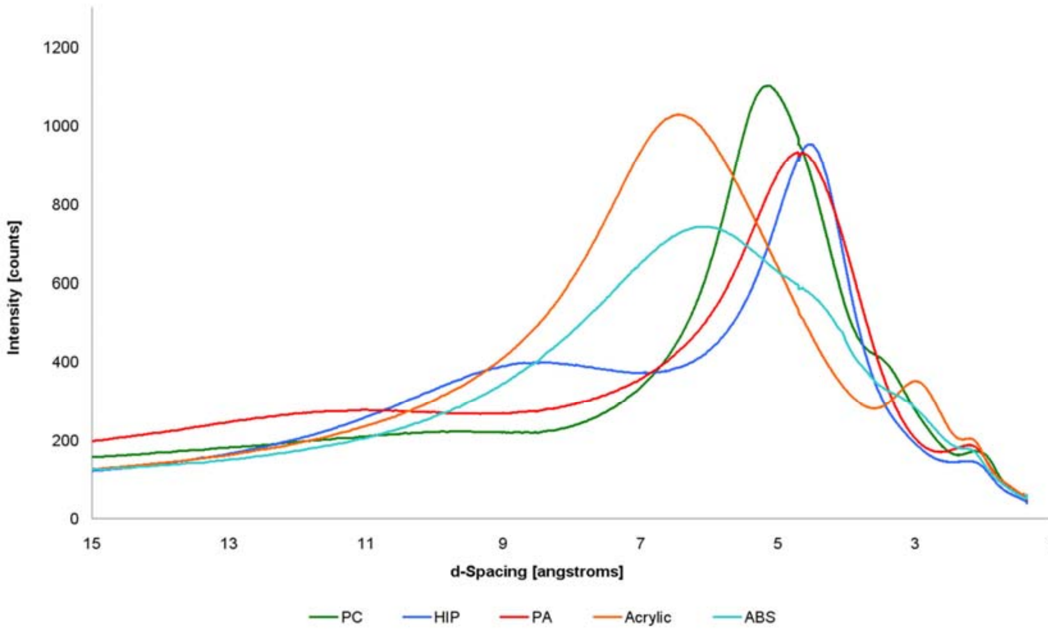

Figure 5. Average intensity versus d-spacing for various plastics. LDPE has been removed from the graph to zoom in on the other five plastics.

The average scattered intensity for each plastic is plotted verses d-spacing in Figure 4. Figure 6 shows the average scattered intensity with standard deviation as measured on the image plate with the beamstop shadow subtracted. LDPE has an intense peak at 4.05 Ǻ and several smaller but sharp peaks at lower d-spacing. Although it has the lowest overall average intensity, it has the highest standard deviation among all the plastics as shown in Figure 5. The intense scattering around 4 Ǻ makes resolving diffraction spots from a crystallization sample contained in this plastic difficult at this resolution range. Therefore this plastic is problematic to use as a container for crystallography experiments. All five other plastics performed similarly as seen in Figure 5. The scattered intensity was low and varied smoothly with resolution. ABS plastic had the lowest maximum intensity verses resolution and the broadest scattering curve out of all the plastics. PC, PA, and HIP show relatively narrower peaks than ABS and Acrylic.

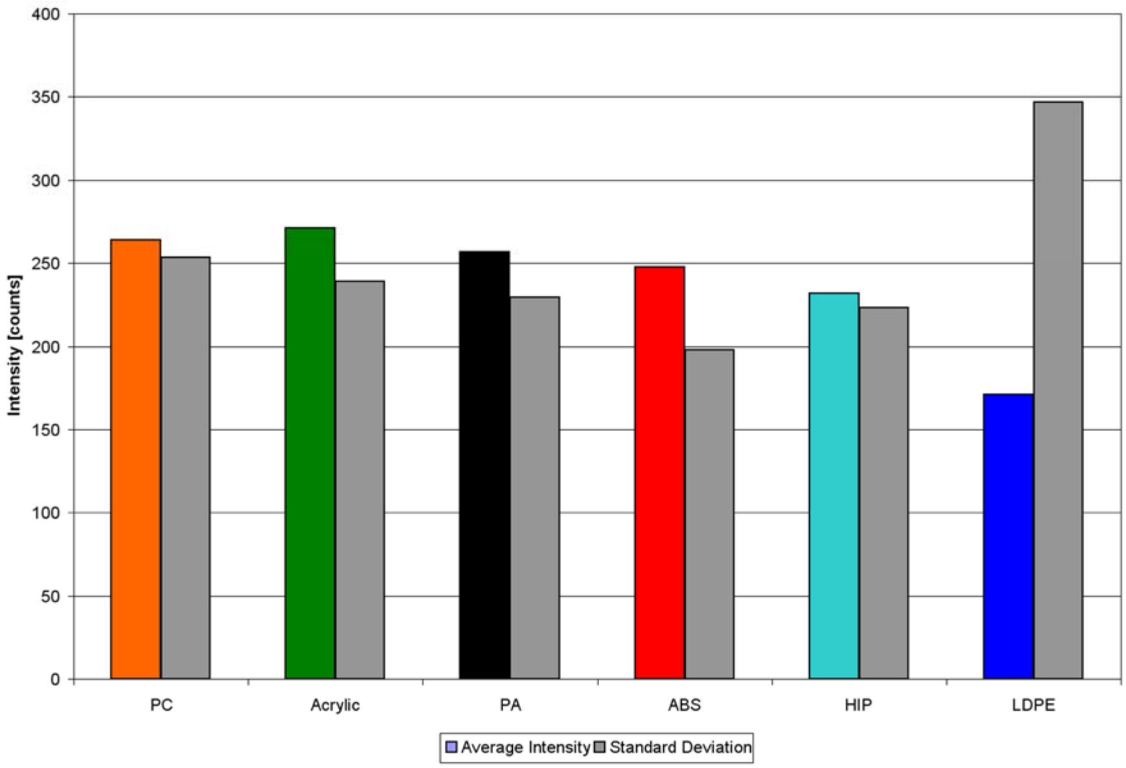

Figure 6. Average intensity and standard deviation of various plastics.

Differences in Diffraction Based on Orientation Angle

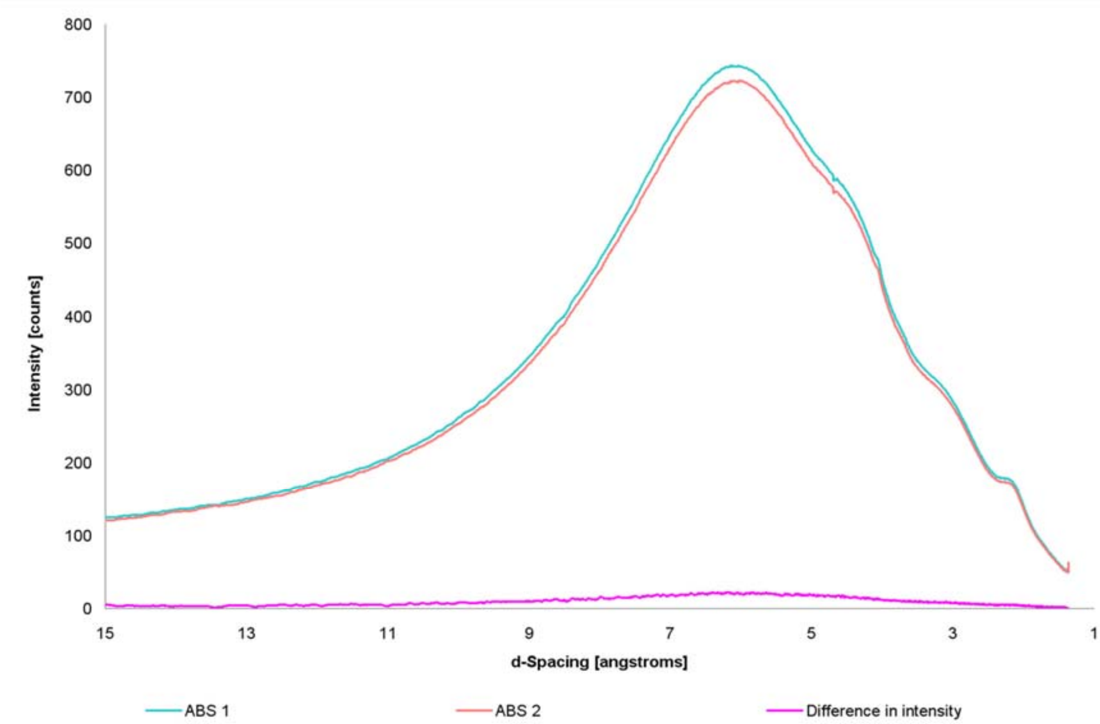

Figure 7. Average intensity versus d-spacing for ABS measured at two different orientations 90 degrees apart, showing that there is not a significant difference in intensity with orientation angle.

It was determined that the orientation of the plastic did not have a significant effect on the scattering pattern. This is demonstrated in Figure 7 for ABS plastic. Given the uncertainty in the experiment, the two curves in are nearly identical.

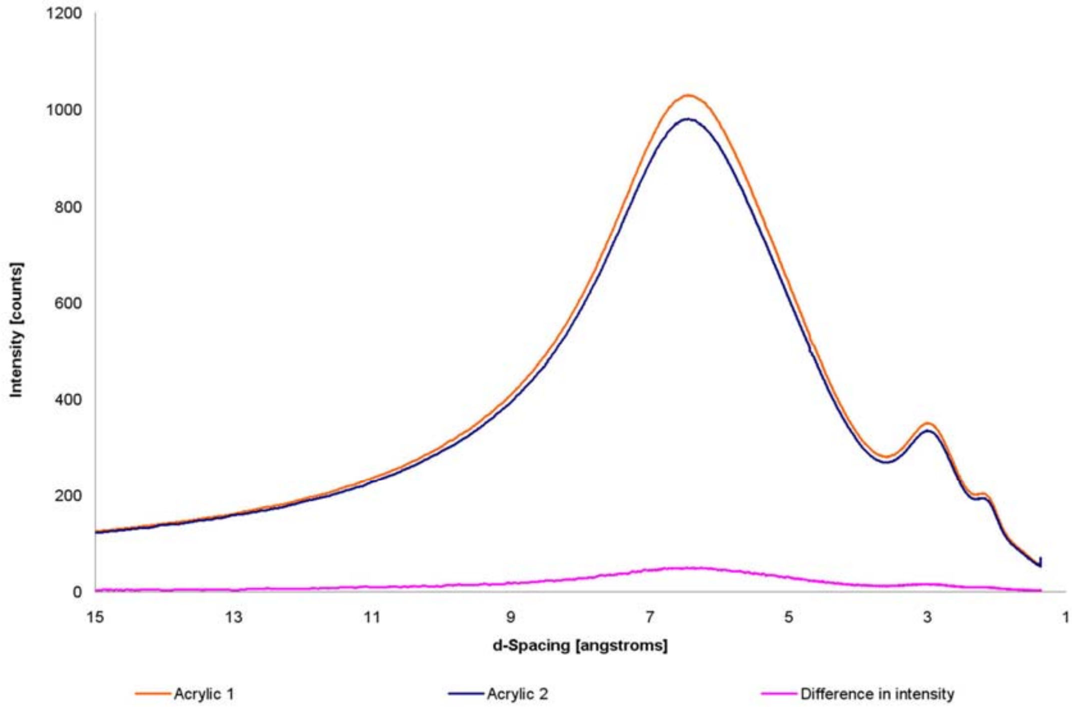

Figure 8. Average intensity versus d-spacing for Acrylic measured at two different orientations 90 degrees apart, showing that there is not a large difference in intensity with orientation angle.

Visual and Optical Clarity

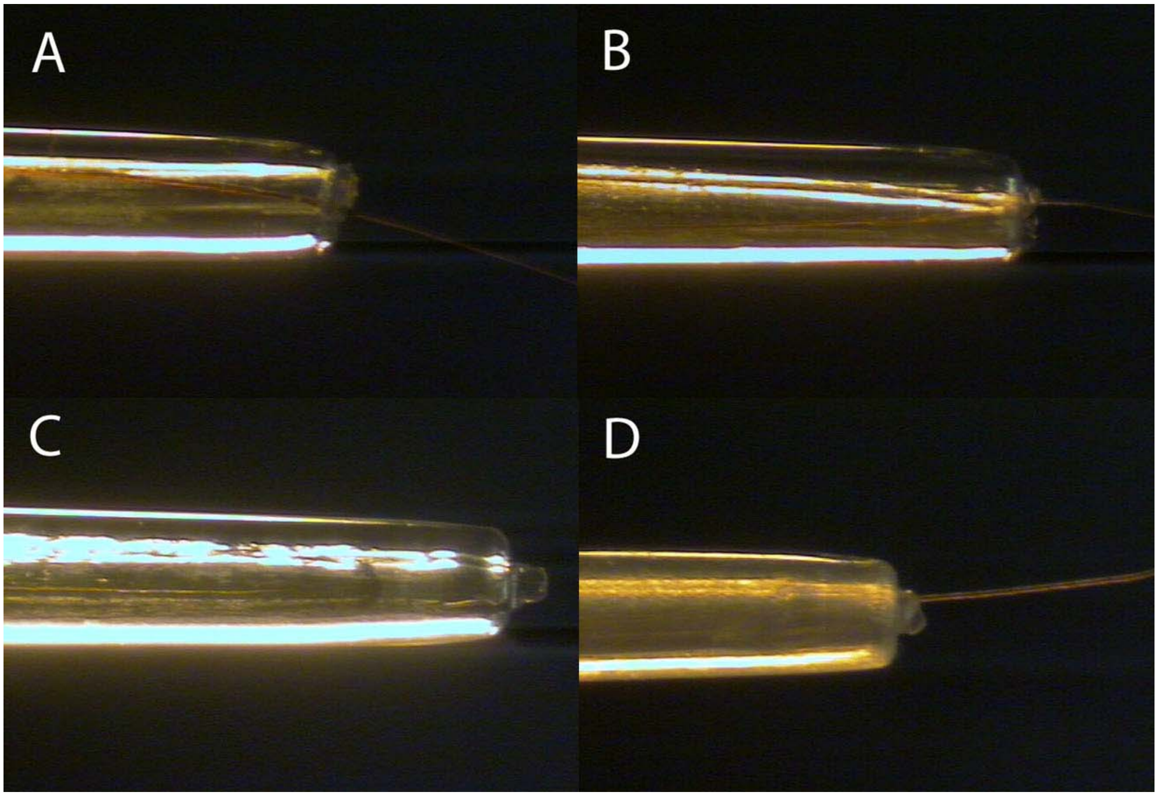

Figure 9. Room temperature image of a 100 micron human hair inside capillaries of different plastics: A) PA, B) PC, C) Acrylic, and D) LDPE is shown.

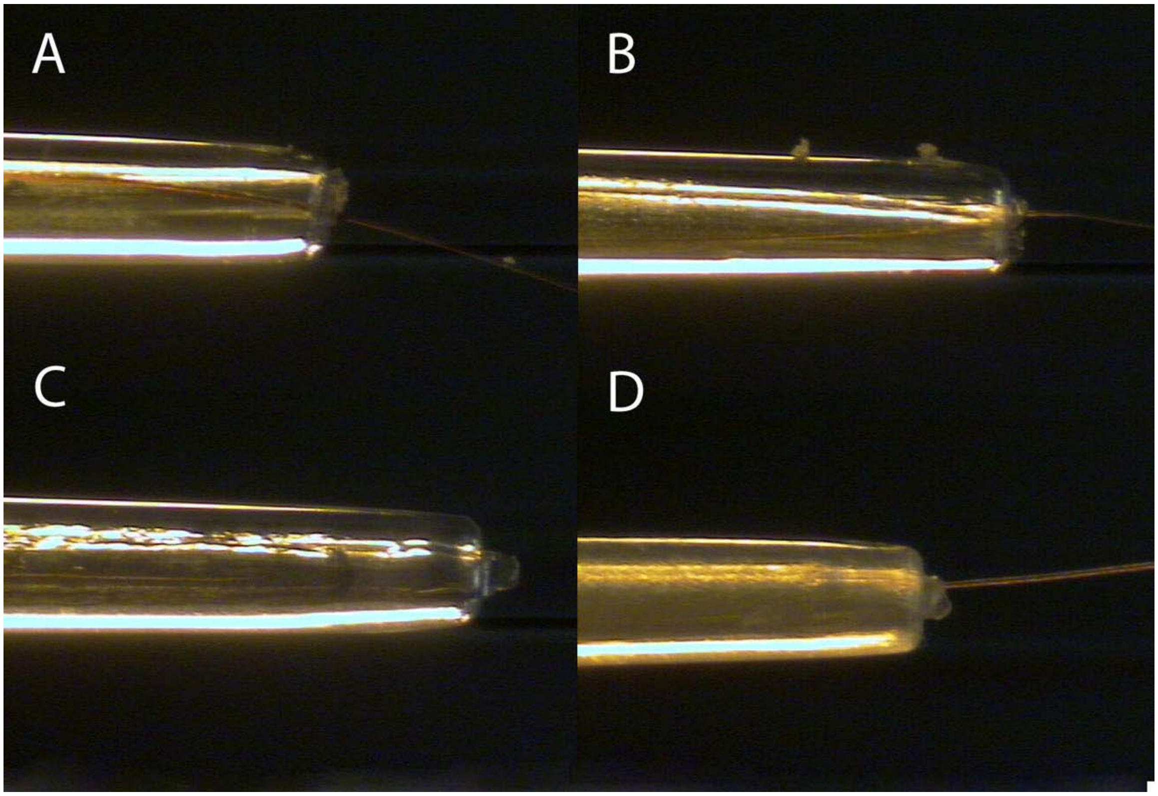

Figure 10. Image of a 100 micron human hair inside capillaries of different plastics at 100 K: A) PA, B) PC, C) Acrylic, and D) LDPE is shown.

Table 1. Grade and qualities of various plastics at room temperature and 100 K.

Room Temperature		
	Visual Clarity at Room Temperature	Grade at Room Temperature
ABS	No distortion; not reflective; hard to see through;	Good
Acrylic	Heavy distortion; highly reflective; transparent	Poor
PA	No distortion; slightly reflective; transparent	Very Good
HIP	Distortion; highly reflective; transparent	Fair
LDPE	Distortion; not reflective; cannot see through	Poor
PC	Distortion; highly reflective; transparent	Good
Cryogenic Temperatures		
	Visual Clarity at 100 K	Grade at 100 K
ABS	No distortion; not reflective; hard to see through	Very Good
Acrylic	Heavy distortion; highly reflective; transparent	Poor
PA	No distortion; slightly reflective; transparent	Very Good
HIP	Distortion; highly reflective; transparent	Fair
LDPE	Distortion; not reflective; cannot see through	Poor
PC	Distortion; highly reflective; transparent	Good

Table 1 lists the optical quality of the different plastics tested at room and 100 K. Figure 9 and 10 show the images of the two best and worst plastics in terms of optical quality. A) PA receives a high score due to its transparency and low distortion. B) PC receives a high score due to its transparency, but loses points based on its high reflectivity. C) Acrylic receives a low score due to its high reflectivity and heavy distortion. D) LDPE receives a low score due to its poor transparency and heavy distortion. The optical properties of all the plastics was relatively constant with temperature (when determined visually with the beamline camera). Of the plastics tested, the best plastics to use as a container for diffraction data collection are ABS and PA. Both of these plastics have no distortion, zero to low reflective properties and are reasonably clear, which sets them apart from plastics such as Acrylic or HIP. Unlike LDPE, ABS and PA also have the best clarity.

Conclusion

Our preliminary results indicate that ABS would be the ideal plastic for purposes of crystallography. However, there is still an abundant amount of future work that needs to be carried out. Some future projects include testing more plastics for crystallography purposes and analyzing how ABS functions with known crystals. Perhaps most important, effort should go into determining how to lower the wall thickness of the plastic capillaries. Given that most of the plastics perform similarly, lowering the thickness of the plastic will most significantly decrease the scattered intensity from the plastic container. Nonetheless, the prospects of using pipette tips to load samples and collect data are positive and further research should be done.