

Abstract

There are some capillary cells for high pressure experiments that are used to measure samples with x-ray [1,2], mostly made of fused silica or sapphire. However those cells are not optimized for powder diffraction characterization. Spinning to improve particle statistics, and so diffraction data quality, has not been implemented so far, and the mechanism to load the samples is slow and does not permit to analyze early stages of reactions. The aim of this poster is to present the design of a capillary cell for synchrotron X-ray powder diffraction characterization, capable of producing conditions of pressure and temperature from room up to 200 bars and 200 °C. Two key features will be enabling rapid injection of the sample, and allowing spinning up to 600 r.p.m. These features must be attained in a very compact design to fit most powder diffraction end stations sample environments. The design will raise the problematic of spinning a capillary at high pressures and will show different approaches to the motorization in order to fulfill the required small size and easy tunability.

Introduction

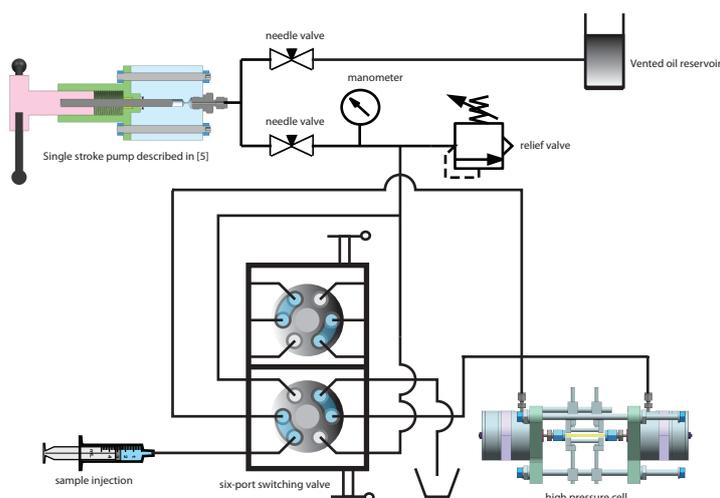
Powder diffraction is a scientific technique that consists in the diffraction of X-rays, neutrons or electrons for the characterization of the internal structure of the microcrystals of a material in polycrystalline form. The reason to use powder is to obtain an equal orientation of the microcrystals in all directions of the space, which provides that the three-dimensional reciprocal space of diffraction of a single crystal is projected on one single dimension. To provide this the sample can be also rotated so the average of equally oriented crystals is improved.

So far no high pressure cell to measure with this technique is able to spin.

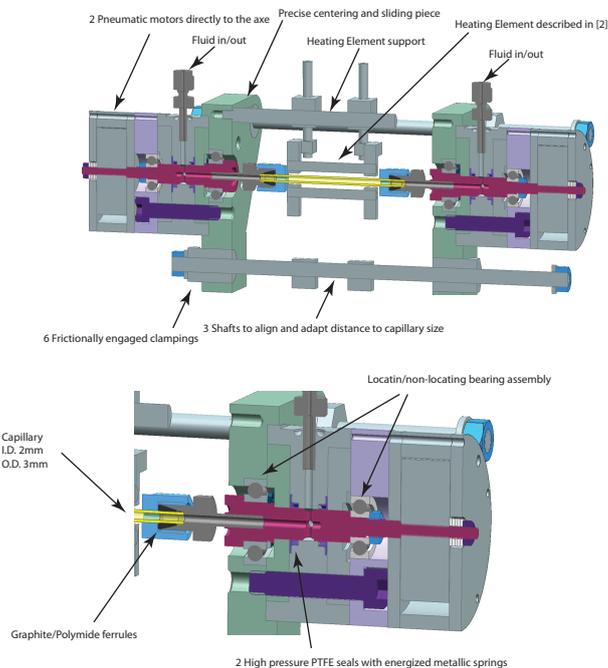
Requirements

- Capillary capable to contain large grain size powder (~10µm)
- Conditions of pressure and temperature up to 200°C & 20MPa
- Compact design to fit powder diffraction end stations
- Spinning to improve powder diffraction data quality
- Capillary material compatible to measure x-ray diffraction

System design



Cell detailed design



Future work

- Manufactured the cell and commission it
- Check the resistance of the capillaries as it is highly dependent on internal faults due to fabrication process and the tensile strength is difficult to determine [3]. Several materials that meets theoretical requirements in terms of resistance and are x-ray compatible will be tested (sapphire, borosilicate glass, quartz glass) .
- Produce a pneumatic air vane motor witch is ideal for this purpose as it can be integrated in the design and it has demonstrate to be much more compact [4] compare to stepper and brushless motors.
- Optimize the sample injection and the pressure increase timing

References

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- [2] P. J. Chupas, K.W. Chapman, et al. "A Versatile Sample-Environment Cell for Non-Ambient X-ray Scattering Experiments." J. Appl. Crystallogr. 41, 822-824. (2008)
- [3] M.R. Arnold, H.R. Kalbitzer, W. Kremer, High-sensitivity sapphire cells for high pressure NMR spectroscopy on proteins, J. Magn. Resonance 161 (2003)
- [4] M. Carfagni, R. Furferi, L. Governi, Y. Volpe "A vane-motor automatic design procedure" IJDeM, 3, 147-157 (2013)
- [5] W. B. Holzapfel, N. S. Isaacs "High-Pressure Techniques in Chemistry and Physics, a Practical Approach" 1st edition, Oxford University Press, Oxford (1997)