A capillary furnace designed for variable-temperature X-ray diffraction

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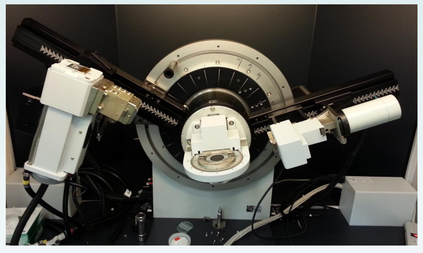
Introduction

In the present work, we build an inexpensive capillary furnace to be used in an X-ray diffraction machine. The basis of the furnace comes from work done by Lavigueur and colleagues. The purpose of this project is two-fold, namely, design a furnace that is inexpensive and conduct variable-temperature X-ray diffraction on air-stable samples. Capillary temperatures can reach temperatures up to 1000 K. We attach the furnace to a standard goniometer head, which is then mounted in the X-ray diffraction machine. The simplicity inherent to the furnace allows for easy building and operation.

Objectives

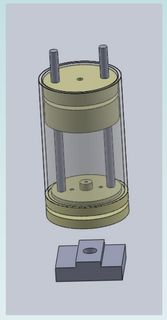
- **Design a furnace that is capable of reaching 1000 K and can be installed in an X-ray diffraction machine.**
- **X-ray diffraction machine:**

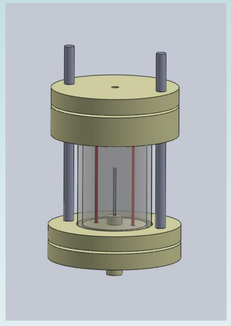


- **Inexpensive: under $1,000**
  - **Furnace quote from a company: $68,800**
    - **More advanced furnace**
- **Easy operation:**
  - **Loading capillaries**
  - **Assembling furnace**
  - **Replacing worn heating elements**
- **Vacuum capability**
  - **Next step in furnace advancement**

Methods and Materials

- **Solidworks 2011 was used to design the furnace**
- **MACOR ceramic used to make furnace**
  - **Max temperature: 1000˚ C**
  - **Sustainable at 800˚ C**
- **Heating Element: Nickel Chromium (Nichrome) wire**
  - **Melting Temperature: 1350˚ C**
- **Two models were created:**
  - **Model A and Model B:**





- **Why we chose model B:**
  - **Reduce chances of short circuiting**
  - **Improved quartz casing seal – minimize heat loss**

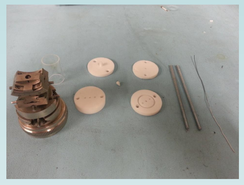
Prototype

- **Goals:**
  - **Verifying the literature**
  - **Testing various gauges of Nichrome wire**
    - **Tested: 20, 24, 26, and 28 gauge wire – chose 24**
      - **24 gauge offered best temperature control, wire controllability, and amperage usage.**
- **Design:**

- **Quartz tube used to withstand temperatures**
- **Ceramic insulators to reduce chances of wood igniting**
- **Shortcomings:**
  - **Crimping device finicky – resistance varied**
  - **Poor seal between quartz and wood – loss of heat after thermal cycling**
  - **Melting temperature of Nichrome**
    - **In the future use platinum or tungsten**

Final Design

- **Furnace unassembled:**



To left, 5 ceramic pieces, goniometer head, quartz tube, 2 threaded bolts and nichrome wire

- **Furnace Assembled:**

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References

Christine Lavigueur, E. Johan Foster and Vance E. Williams. A simple and inexpensive capillary furnace for variable- temperature X-ray Diffraction. *Applied Crystallography*, November 2007

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