

## INSTALLATION FOR SHORT TIME HEAT TREATMENT AT HIGH TEMPERATURE

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**ABSTRACT.** The design of a simple installation for short time annealing of nanomaterials at high temperature is presented. The installation is capable of heating rates of over 20 °C/s and cooling rates of up to 80 °C/s with a maximum attainable temperature of 1050 °C.

**Keywords:** *short time annealing, temperature measurement, controlled atmosphere, high heating rate*

### INTRODUCTION

Many scientific efforts today focus on nanomaterials and nanostructured materials [1-3]. Some of these materials need to be annealed in order to tune their structure and/or microstructure; however annealing processes must be short in order to maintain the desired length scales [4-6]. Therefore it is essential to reach the annealing temperature as fast as possible and then quench the samples as fast as possible. To this end we have constructed an installation capable of heating rates of up to 20 °C per second and cooling rates of over 80 °C per second. Therefore a sample can be heated to 950 °C and brought back to room temperature in 65 s.

### EXPERIMENTAL

The annealing instalation consists of a furnace, quartz tube, vacuum and gas system and temperture sensor.

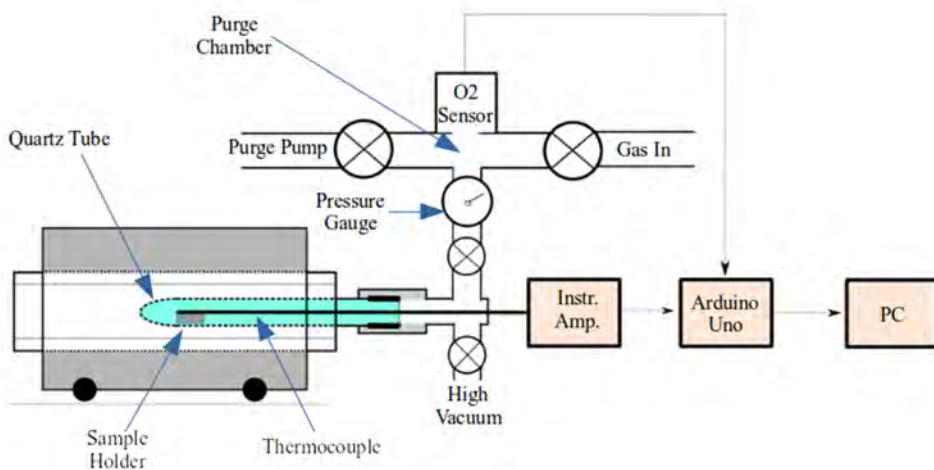
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The furnace is a small carbolite tube furnace with a maximum temperature of 1200 °C. The homogenous heating area of the furnace is 5 cm.

The quartz tube is used as the sample chamber, as quartz has great thermal shock resistance and can withstand high vacuum. For our specific tubes, the vitreous transition was found to be at approximately 1100 °C, therefore heating was limited to 1050 °C in our case to avoid melting the tube.

The vacuum system consists of a Pfeifer turbo pumping station isolated which can be isolated with a gate valve. The gas system is also isolated from the tube system by a valve, therefore we can easily switch between gas and vacuum. The gas system has a purge chamber, equipped with an oxygen sensor, so that the gas can be run through it until the O<sub>2</sub> concentration falls below 2 ppm. The O<sub>2</sub> sensor also monitors oxygen levels during annealing.



**Fig. 1** Schematic Representation of Annealing Installation

For the temperature measurement, an S-type thermocouple has been passed through an air-tight metal plate (sealed with epoxy) and affixed to the sample holder (a thin tantalum cylinder). As S-type thermocouples give a low voltage signal, the output is amplified by a simple instrumentation amplifier using the LM358N operational amplifier chip. The amplified signal is then read by an Arduino Uno [7] microcontroller board and transmitted to a Raspberry Pi 2 [8]. In order to reduce most of the electronic noise, an average is taken every 100 measurements. As the Arduino chip does not have a timekeeping function, after setting up the measurement

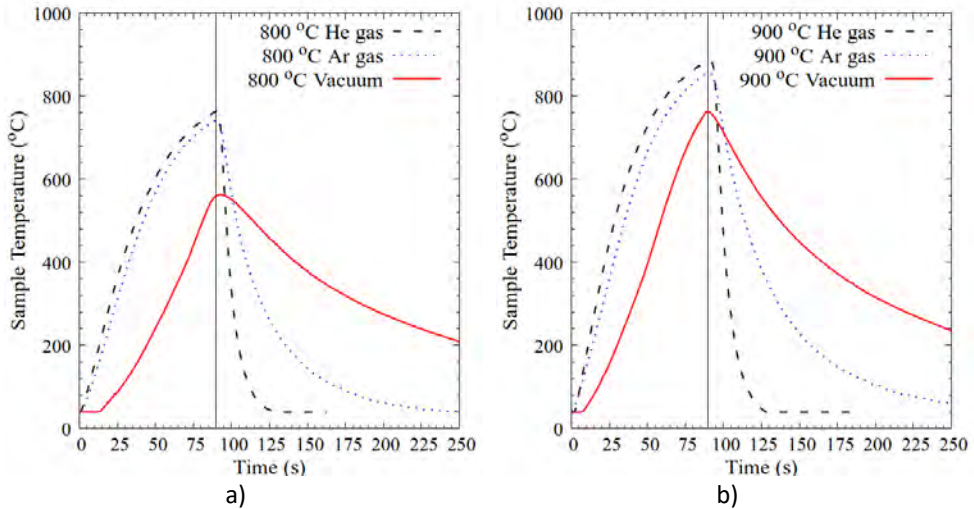
loop we let it measure for an hour and calculated the time per cycle in order to determine the time in seconds. The computer software (written in Python [9]) takes the output from the arduino and converts it into physical values which it writes to a data file. The accuracy of the temperature measurement is limited to  $\pm 2$  C. Real time data monitoring is done with Gnuplot [10].

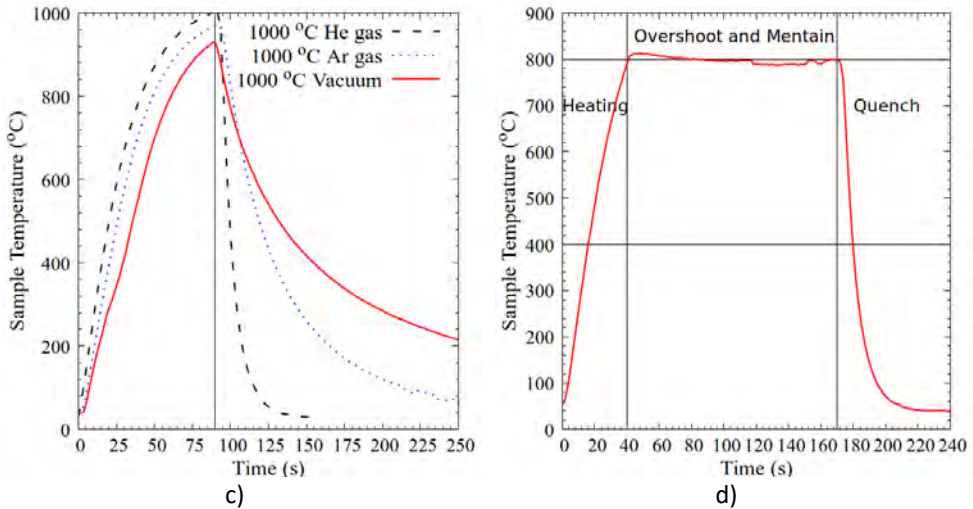
The installation functions by inserting the prepared quartz tube into the pre-heated furnace.

## RESULTS AND DISCUSSION

The performance of the setup was tested in high vacuum ( $10^{-5}$  mbar), high purity He and high purity Ar atmosphere. Heating and cooling rates are shown in Fig 2.

Of course, the heating and cooling rates are slow in high vacuum, even when the quartz tube is immersed in cold water Fig. 2a, 2b and 2c. The introduction of Ar gas significantly improves the heating and cooling speed, however the rates are not linear. Switching to He gas does little to improve the heating rate, however quenching is much faster in this configuration. The lack of a significant benefit when using He while heating is very likely due to the large mass and thermal inertia of the quartz tube, which is less of a factor when it is plunged in cold water. Preheating the tube as well, and inserting the samples with a thin manipulator rod might improve performance significantly.





**Fig. 2** Example heating and cooling curves with the furnace pre heated to a) 800, b) 900 and c) 1000 °C and an example heating and holding curve d)

We should also note the shape of the heating curves, it is only natural that our heating rates go down as we approach the set temperature of the furnace, therefore we see a much steeper curve at low temperature when compared to high temperature, still we can achieve average heating rates of about 20 °C/s up to 900 °C, which is not insignificant. The setup is capable of reaching 950 °C in 65 s, 900 °C in 50 s, 800 °C in 38 s and 700 °C in 27 s etc. Fig. 2c. The cooling speed is approximately 80 °C / s when using He as the heat transfer gas.

Some ramp and hold annealings can also be performed, Fig. 2d, by changing the sample position inside the furnace. However this procedure induces a temperature gradient of about 5 °C, and is very hard to execute as swift and precise movements of the furnace are required.

## CONCLUSIONS

A rapid annealing installation capable of average heating and cooling rates of 20 °C/s and 80 °C/s respectively, was constructed. The setup can run under high vacuum, purified Ar or He gas atmosphere and the oxygen concentration (when gas is used) can be monitored during the annealing process.

Future modifications which could improve heating rates could consist of: (i) a thin manipulator arm for inserting the sample at high temperature, (ii) replacing the quartz tube with one with a higher melting point so that the furnace set temperature can be increased, (iii) lowering the He pressure to improve heat transfer from the tube to the samples and therefore increase the cooling rates.

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