MSE 528 - Phase Transformation of Steel

Orton dilatometers are designed to measure the linear dimensional changes of ceramics, glasses, metals, carbon composites, cermets, minerals, and polymers as a function of temperature. The dilatometer records reversible and irreversible changes in length (expansion and shrinkage) during heating and cooling. Samples are measured for determining firing ranges and firing schedules, measuring thermal expansion ranges for glaze fits, and measuring thermal expansion ranges for R&D, QC or product certification. Orton standard dilatometers are used for ASTM E-228, ASTM C-372, ISO 7991, and other testing procedures to measure the Coefficient of Thermal Expansion (CTE), softening point, glass transition temperature, Curie point, crystalline transformation, phase transition, shrinkage, warping, bloating, sintering rate, isothermal creep, stress relaxation.

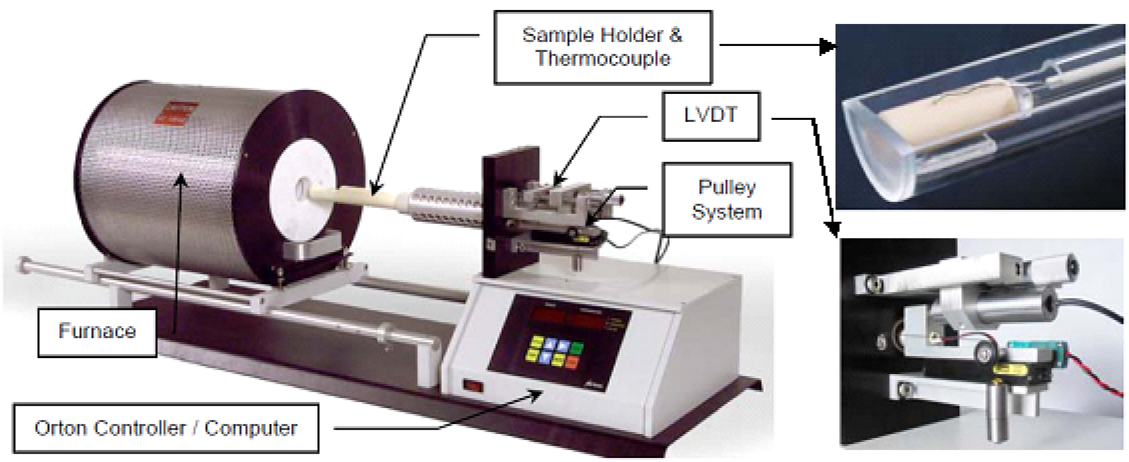

Figure 1 – location of basic components on a standard Orton/Harrop dilatometer.

The standard Orton dilatometer (Figure 1) is a digital, horizontal, single sample, compact, table top system, comprised of a furnace (for a variety of temperature ranges, including sub-ambient); a sample holder system (fused quartz or high alumina); a control/sample thermocouple; a sample displacement measuring system (probe rod and LVDT sensor); a user-adjustable counterweighted pulley system to provide a constant and uniform contact load on the test sample; the Orton control board for furnace control and data acquisition; and the Orton dilatometer software. All Orton standard systems were factory calibrated against a 1" rod of high purity, platinum, thermal expansion standard. A 1" rod of high purity, polycrystalline, commercial grade, 99.8% high alumina is included to be used as a secondary calibration standard.

Principle of Operation

The sample is placed in a high purity alumina sample holder, in contact with a freely suspended alumina probe rod. The holder and sample are inserted in the furnace. A **control thermocouple is located in close proximity to the heating element** for temperature control. **Another thermocouple, located in the probe rod**, senses the temperature of the sample. As the sample expands or contracts, the change in length is transmitted by the probe rod to the core of a **linear variable differential transformer**

(LVDT). The LVDT converts the displacement into a proportional voltage signal which is recorded as the Delta-L curve. The operator can adjust the magnitude of the electrical signal so that expansion is displayed on the graph in convenient units regardless of the length of the sample. **A slower heating rate is usually preferred to a faster one,** for the interior of the sample to be equal in temperature to the exterior. Heating too fast will cause inhomogeneous temperature and erroneous expansion. Typically values of 3**°C/minute for an alumina sample holder are useful**.

The computer software communicates with the dilatometer. The operator enters the critical run parameters and the software sends the information to the controller board inside the dilatometer. The Harrop controller board inside the dilatometer controls the thermal cycle of the furnace and collects the time, temperature, and **percent linear change** (**PLC**) data. The software extracts data from the dilatometer controller board during the run so the operator can monitor the run in real time. Upon completion of the run, the software extracts the data from the dilatometer and creates **<u>a data file for post testing review and analysis</u>**. The operator can view and analyze the past run on the same PC, or can transfer the data file to another PC for independent viewing and analysis. The software collects and displays time, temperature, and percent linear change data, and stores it in a binary file. PLC data is displayed on the PC monitor in temperature or time based modes. Data can be printed graphically or in tabular form, or exported as an ASCII file. Software features include comparisons against temperature or time of up to six runs; zoom into part of the curve; display differential or alpha CTE curves; T_G (between 400 and 850°C) softening point temperatures; α-β quartz transition temperature, and coefficient of expansion calculation for any temperature range.

<u>**Orton Dilatometer** software:</u>
Click on <u>Set Up Experiment</u>
<u>Run mode:</u> ◙ Test samples without baseline
<u>Data File:</u> enter a filename
<u>Acquisition Time</u>: enter total test time (60 minutes for steel sample)
<u>Acquisition interval</u>: how frequently data is acquired: 5 seconds
<u>Sample length</u>: measure the length of a steel sample and record
<u>Enter data and continue</u>

Place steel sample in ceramic cylinder with thermocouple directly above sample.
Rotate **knob** (located on side near switch) to position LVDT rod in contact with sample.
Close <u>furnace chamber</u> (push metal basket toward sample).
In Orton software click on **<u>Start Test</u>**.
Using the vernier potentiometer (silver dial) at the end to zero the digital reading.

The <u>**Harrop temperature** controller</u> is programmed for the following ramp rates by creating a recipe with 2 stages:
Ramp 1: 26 °C to 1000°C in 40 minutes.
Ramp 2: 1000°C to 600°C in 20 minutes.
After the temperature controller is programmed, the computer is clicked to start and the program is put in run mode. The **<u>powermax</u>** mode may have to be engaged to maintain

current and set point temperature; **powermax** may have to be set as high as 75% or the system will lag behind and not reach 1000°C.

Software collects data and plots (ΔL) the change in length vs. time and temperature.

For the memo report, plot the data from the dilatometer in Excel, explain the changes in the curve with regard to phase transformations and calculate the thermal expansion coefficient.